

INNOVATIONS IN PRACTICAL WORK

AT THE TERTIARY LEVEL

by

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A Thesis submitted in part fulfilment of the requirements for the degree of Doctor of Philosophy in the University of Glasgow.

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April, 1977

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## A C K N O W L E D G E M E N T S

I wish to express my sincere gratitude to the supervisors of this work, Professor D.W.A. Sharp and Dr. A.H. Johnstone, for their guidance and assistance throughout this research. I am particularly indebted to Dr. Johnstone whose advice and encouragement proved a constant and valuable inspiration.

My thanks are also due to other members of the Science Education Research group, in particular Mr. Norman Reid, for many valuable discussions, and to the students and staff of Glasgow University who participated in the material produced for this thesis, and to Glasgow University for their financial assistance.

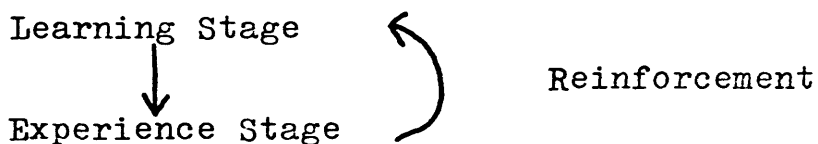
Finally, I would like to thank Mrs. Moira Carter for preparing the typescript.

## A B S T R A C T

A survey of the aims, philosophy and assessment techniques in practical work since 1805 was carried out.

Evaluation of the practical courses at Glasgow University revealed two main weaknesses in the present system. These were that the effectiveness of learning in the laboratory was low and that the student was given no opportunity to think for himself.

A two stage laboratory model was devised to overcome these weaknesses.



The aim of the learning stage is to teach the skills unique to the laboratory such as manipulative skills and other ancillary skills such as graph drawing and to provide the student with practice in mastering the techniques.

The aim of the experience stage is to reinforce the previously learnt techniques and to provide the student with an opportunity to think for himself in the laboratory.

To evaluate practical work three types of assessment were used. The first method was that of self-report techniques where the student evaluates his own performance. Both questionnaire and interview approaches were used to collect this information and an interview schedule based on the two-stage laboratory model was developed. The use of paper and pencil tests to assess the students' knowledge of practical procedures was investigated and the results analysed. A third method of assessment which involved analysing student performance in the laboratory was developed to assess the effectiveness of two films.



In the learning stage of the laboratory model three approaches were examined which were intended to increase the effectiveness of learning. These were, the development of two films, one on the use of the burette and the other on the use of the pipette, the introduction of pre-laboratory exercises and the adoption of a group participation approach to present selected experiments.

In the experience stage two methods were examined. These were the use of open-ended experiments and, secondly, the use of projects.

Suggestions for further work have been proposed which may lead to further improvement of practical courses and of assessment methods.



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CHAPTER 1

A Review of the Aims, Philosophy and Assessment  
Techniques of Practical Work

## CHAPTER 1

A Review of the Aims, Philosophy  
and Assessment Techniques of Practical Work

## 1.1 The Origins of Laboratory Instruction

The origin of modern laboratory instruction<sup>1, 2, 3</sup> is credited to Friedrich Stromeyer who at the University of Göttingen in 1806 started the first teaching laboratory. His guiding principle<sup>1</sup> was that chemistry could only really be learned through laboratory practice and that the students must be given an opportunity to carry out analyses on their own. From this sprang the method of using individual laboratory practice for learning chemistry.

However, at the beginning of the nineteenth century only a few teaching laboratories were started. Notably the one begun by Liebig in 1824 to whom many falsely credit the first teaching laboratory and the first student participation laboratory in America started by Amos Eaton<sup>4</sup> at the Rensselaer Polytechnic Institute in 1825. Eaton's philosophy was similar to Stromeyer's in that he felt that the laboratory gave the student a chance to 'learn by doing'.

In Britain the first laboratory for students was set up by Thomas Graham at the Royal Technical College in 1830.<sup>5</sup> Practical classes at Glasgow University were started by Lord Kelvin, then William Thompson, in 1845 in an old wine cellar.<sup>6</sup>

However, in Britain the growth of practical work is credited to Edward Frankland<sup>5</sup> who throughout his life did much to encourage the introduction of laboratory instruction. Largely due to his efforts by 1876 there were one hundred and fifteen laboratories in operation in Britain, most giving instruction at a very elementary level.

At the beginning laboratory work was restricted to /

to the instructor and his assistants<sup>7</sup> and apart from a few pioneering institutions it was not until later in the nineteenth century that individual laboratory classes in the sciences were generally available.<sup>8, 9</sup>

It was as late as the 1890's before there was a period of rapid growth in the establishment of student laboratories.<sup>9</sup>

During this period of expansion educators in America advocated the individual laboratory method as a solution for the problems of the scientific age and as a revolution in education. At this time universities such as Harvard made laboratory instruction a college entrance requirement<sup>6</sup> and President Eliot at Harvard detailed a list of forty experiments in physics which should have been completed by a pupil wishing to be considered for admission. This list continued to be used right up until the middle of the twentieth century at many American institutions and became known as the 'Harvard 40'.

Therefore, by the turn of the century individual laboratory instruction in science had been generally adopted. It was during this period, however, that the first criticism was voiced against individual laboratory instruction, and from then on up till the Second World War a debate raged in the literature about the particular merits of individual instruction where the student performed the experiments himself, usually from a detailed list of instructions, versus the demonstration method which Kiebler and Woody<sup>10</sup> defined as a method where 'the instructor with the aid of one or more students, demonstrates the experiment and the students get its import through observation and discussion rather than through actual performance'.

As early as 1900, Barber<sup>9</sup> had questioned the effectiveness of the individual laboratory method in physics /

physics and chemistry. Barber maintained that his students mastered the work well, if not better, by the demonstration method and he soon reduced the number of experiments done by the student to a few which required relatively simple apparatus.

In 1905 the National Education Association Department of Science Educators<sup>11</sup> reported that physics was becoming less popular among high school students partly because the student was asked in the laboratory to deduce laws, many of which he knew before, from data that cannot be made to prove anything and to apply these laws to a set of problems that have no apparent relation to his immediate scientific environment or to the questions that he is so anxious to have answered.

In 1910 Mann<sup>12</sup>, a physicist, concluded that 'laboratories had not solved the problems of science teaching .... we do not know how to use laboratories most effectively'. Once the debate had begun into the aims and styles of laboratory teaching, many researchers wished to compare the individual laboratory versus the demonstration method.

The first recorded attempt was by Wiley<sup>13</sup> in 1918. His results on comparing the performance of students following the two methods tended to favour the individual method of laboratory instruction. However, his results are only interesting for historical reasons as both his methodology and sample sizes (eight in each group) tended to make his results rather meaningless.

In the next ten to fifteen years at least twenty more investigations were published and listed in review papers by Downing<sup>8</sup> and Payne.<sup>14</sup>

The conclusions reached tended to favour the demonstration method. See for example, Kiebler and Woody.<sup>10</sup> The advantages often claimed were that the retention /

retention of information in the short and long terms was superior, and it was more efficient in both time and money.

However, for the acquisition of manipulative skills the individual laboratory method was still thought superior.

Knox<sup>6</sup> after reviewing the literature stated that previous investigations had pointed to the superiority of the demonstration method so far as those outcomes can be measured by a written test. However, he emphasised that a method must be devised for measuring the educational product unobtainable without the laboratory method.

The results obtained by all the investigators besides Horton<sup>15</sup> were by using paper and pencil tests. Horton suggested that there were some outcomes of the individual method which the written tests have difficulty in detecting. Horton then went on to use performance tests. These tests covered skills in handling apparatus and the ability to do manipulations where he thought the other possible outcomes might lie. The construction of the tests firstly involved him in determining what skills and manipulations were required for a particular experiment. To measure them objectively, he constructed a checklist and the students were assessed by a teacher who ticked the sheet as the student completed each step. The results of this investigation indicated that students who completed a laboratory course by the individual method did consistently better in the performance tests. He suggested that the paper and pencil test measured only one possible outcome of laboratory work and that was the acquisition of information. Horton also concluded that the individual method was superior for the highly intelligent student while the demonstration method may be somewhat better for students at the lower /



lower intelligence levels.

Adams<sup>16</sup> also stressed the need for further research into the possible outcomes of laboratory work.

Not all the literature, however, was involved in comparing these two methods. Many researchers put forward hybrid schemes involving different degrees of student participation. For example, Barger<sup>9</sup> describes a method known as the 'class-participation' method where the students were more actively involved by being asked to take readings for themselves.

Cooke<sup>17</sup> describes a system which involved two students taking full responsibility for the performance of an experiment watched by the rest of their group and a demonstrator. The students rotated for each experiment giving each student a chance to perform in front of the others. The demonstrator only offered help if it was needed. The advantages Cooke saw were that it gave the student a chance to learn techniques and also that it avoided sloppy work because of the scrutiny of fellow students and the staff.

Jameson<sup>18</sup> allowed groups of students to work on experiments with the demonstrator moving between groups offering help. He claimed that this group experiment method was superior to conventional laboratory work in acquisition of knowledge and the saving of time and money.

The success of these courses appeared to be very highly related to the motivation and personality of the staff involved. Again, some teachers had a more flexible approach in that they chose a method to suit a particular experiment. Among the suggestions put forward was that of Horton<sup>15</sup> who suggested that experiments involving complex apparatus, those in which it is difficult to obtain the correct results and those which may have an element of danger were best done by a demonstration /

demonstration method.

Before the War much energy was spent on trying to prove the superiority of particular methods. Due to the nature of the investigations most of the results were inconclusive and indeed the results were challenged for not testing for the correct outcomes.

## 1.2 Post-War Developments in Practical Work

The effect of the War was to give an impetus for the re-examination of the purpose of laboratory work. There was a call for larger numbers of technically educated men and women<sup>19</sup> and this raised the questions of:- 1) how to produce them efficiently, and 2) how to assess their practical ability.

Adams<sup>16</sup> reported on a survey carried out by Shearer, who sent a questionnaire to 299 colleges and universities in America, asking lecturers what they thought laboratory work should achieve. Not all institutions replied, but the pattern that developed was that chemical laboratory instruction should:-

- a) develop the ability to make observations, interpret and draw conclusions from observed facts;
- b) develop the ability to use simple scientific instruments and manipulate apparatus;
- c) develop the ability to keep a record and write a satisfactory report;
- d) develop the attitude of drawing conclusions only from observable or accepted data;
- e) develop the habits of accuracy, honesty, self-reliance, cleanliness and orderliness in the laboratory;
- f) satisfy the student's curiosity and provide experience to develop latent interests;
- g) provide opportunity for instruction.

This list which was the first attempt to detail possible laboratory /

laboratory outcomes was designed to help in the construction of possible tests for assessment of practical ability (see Section 1.4).

In the next few years many such lists were devised.<sup>20, 21</sup> Kruglak<sup>20</sup> in 1951 produced a list of objectives in even greater detail for a course in elementary electricity. This list was grouped under six categories:-

- a) Instrumental skills : e.g. manipulative etc.;
- b) Skills in the use of the controlled experiment :  
e.g. recognise adequacy of controls;
- c) Problem-solving;
- d) Miscellaneous skills : ability to follow  
directions, ability to make a good graph etc.;
- e) Functional understanding of principles : to be  
able to recognise the generalisation which is  
being verified;
- f) Habits : neatness, caution, safety.

This list of behavioural objectives was also developed as a guide for the construction of laboratory performance tests (see Section 1.4).

In 1947 Noll<sup>21</sup> produced a detailed list of objectives which he considered necessary for the successful study of science. Noll defined two main areas in practical work:- 1) instrumental skills, and 2) problem solving.

In the area of instrumental skills it was felt that students should have the ability to:-

- a) perform fundamental operations with reasonable  
accuracy;
- b) perform simple manipulatory activities with  
science equipment;
- c) read graphs and tables etc. to interpret them;
- d) make accurate measurements, readings, titrations  
etc.

In the /

In the second area of problem solving skills the student should have the ability to:-

- a) sense and define the problem;
- b) test a hypothesis proposed by an experiment or other means and reject the hypothesis on the basis of the conclusions drawn.

The discussion also covered how these objectives were to be achieved.

In a survey of teaching methods in twenty-five colleges and universities in America, Brown<sup>22</sup> discussed the problems of the vastly increased number of students doing introductory physics laboratory courses and stated that the teaching methods had not kept pace. He criticised the trend of using printed laboratory manuals which specified in far too much detail what procedures were to be followed. He also reported that various institutions had tried to put the student on his own but large numbers tended to make this system fail. Overbeck<sup>23</sup> echoed Brown but placed the emphasis for improvement on the training of demonstrators. Brown<sup>24</sup> in another survey of elementary laboratory instruction this time in English universities reported a very great emphasis on laboratory instruction and on training in manipulative technique. He noted a further difference in that since the emphasis is on manipulative technique, no attempt was made to keep the experimental work in step with any course work, as opposed to the trend in America<sup>25</sup> that most institutions attempt to correlate their laboratory with the lectures and recitations. Another contrasting feature was in preparation where in England the use was made of background readings and the emphasis was on the student to "learn on his own", as opposed to the American tradition of expecting the student to prepare in detail and be tested before entering the laboratory.

Brown<sup>26</sup> carried out a series of investigations in practical /

practical work in physics and found that after one year only 41% of students recognised that they had studied experiments they had performed previously.

His conclusion was that laboratory work must have as its goal the teaching of the scientific point of view, and the intellectual challenge of the experimental method, rather than the training of students in particular or specific techniques or in carrying out particular experiments since the details of these are so obviously lost in a very short space of time.

Many researchers had also questioned the purpose of the laboratory. Among these was Owen<sup>27</sup> who had asked how well the ordinary laboratory experience contributed to the development of skill in applying the scientific method and of developing desirable habits and attitudes that should go with it. He complained that the normal experiment provided:-

- a) too much information for the student, and
- b) was too abstract, i.e. beyond the normal student's experience.

Not all experiments, he said, should be designed to develop scientific method but those that were should give the minimum of information and let the student find out for himself. These experiments would allow the student to

- formulate questions
- recognise assumptions
- apply general principles
- interpret data
- make hypotheses
- test hypotheses.

Mallinson and Buck<sup>28</sup> stated that there was no critical thinking done in the laboratory, usually just 'cookbook manipulations' where the student followed a printed list of instructions. They said that laboratory experiments /

experiments have changed very little and that students were naive about the implications of the term 'scientific method'. They quote Schlesinger<sup>29</sup> who stated that students in the laboratory develop the habit of getting the expected results.

To overcome this problem they wanted an inductive approach, i.e. use observed data to arrive at a more general principle as opposed to a deductive approach where you start with a principle and make observations or perform experiments to verify it.

Blick<sup>2</sup> also realised the limitations of the 'cook-book approach' but advocated an inductive-deductive approach where students determine general principles and then test them by a specific experiment. Blick makes the point that the scientific method does not follow a series of steps but consists of the use of innumerable and almost unclassifiable techniques. Young<sup>30</sup> suggested discarding the laboratory manual after the student learnt the basic skills. He suggested that laboratory work had three aims in an elementary chemistry course:-

- 1) to teach elementary facts and principles;
- 2) to train the student how to use laboratory equipment; and
- 3) to help the student to think.

He advocated letting students solve problems and devise experiments to test their hypotheses.

Nedelsky<sup>25</sup> postulated that the elementary physics laboratory was well suited for acquainting the student with the 'process of inquiry'. This he believed was the central laboratory objective. For this to occur he stated that hard thinking must take place in the laboratory.

In this period the discussion had moved away from the /

the different methods of practical work to the more fundamental question of purpose.

The laboratory system was criticised for its many shortcomings and there was a growing feeling that there was too much 'cookbook chemistry' which was not of such lasting value to the student as teaching him the 'process of inquiry'.

### 1.3 Developments in Practical Work since 1960

In 1962 Michels<sup>31</sup> agreed with Nedelsky<sup>25</sup> that the laboratory should acquaint the student with the 'process of inquiry'. He stated that the laboratory was the only place where a student could experience physics as it developed. He therefore advocated that the laboratory should be open-ended. By open-ended, he meant an experiment where the student was posed a problem which he was expected to solve. Michels also saw several disadvantages of an open-ended approach in that -

- a) more time, energy and experience was required;
- b) it allowed students some choice;
- c) space and apparatus requirements would be greater;
- d) extension of single study may leave the student unfamiliar with normal techniques.

However, at this time many courses were being developed which incorporated degrees of open-endedness in experiments.

Young<sup>32</sup> proposed that laboratory work should be more than manipulating apparatus and that the failure of practical work was that no one had tried to discover if students were getting anything more. He thought that the method of presenting the student with a detailed experimental plan to work through was valid for teaching principles and techniques. But from the first year onwards this method should be supplemented by an approach which /

which allowed the student to make his own investigations. Young suggested six objectives which he claimed could be understood by the student and at least qualitatively evaluated by the staff. These were:-

- 1) to acquire directly some descriptive chemical knowledge and organisation of this knowledge with other descriptive information obtained from books;
- 2) to manipulate reagents and apparatus so that a reliable measurement or observation can be made;
- 3) to observe critically;
- 4) to interpret data;
- 5) to present a clear exposition of the interpretation of the data;
- 6) to plan and carry out further laboratory work which will extend and amplify this data and its interpretation.

It was his hypothesis that emphasis upon the sixth objective would strengthen the achievement of all of the others and that by this emphasis the student became aware of and accepted the challenge to initiate and carry out a piece of laboratory work of his own design.

Therefore, the nature of the process tended to be cyclic. For a freshman this could be composed of two cycles;

- 1) a brief cookbook demonstration, and
- 2) an experiment of his own design. This was a further exposition of his paper in 1957.<sup>30</sup>

This paper suggested a feasible design by which the student could be taught basic skills and still get experience.

Venkatachalam and Rudolph<sup>33</sup> used these ideas to produce a learning/challenge cycle of laboratory work. In the learning cycle the student was given a reading assignment which was discussed in the laboratory and followed by a 'cookbook' experiment which was intended /



intended to familiarise them with techniques and equipment. The challenge cycle was composed of an open-ended experiment based on a variation of the cookbook experiment.

In a further paper<sup>34</sup> they stated that their programme had been evaluated on a larger scale and had proved effective with students from various disciplines and with different background experience.

Like Young<sup>32</sup> they felt a major benefit was the sense of direction which was communicated to the students. Their systems also had the advantage that it could work with large numbers of students.

D'Auria, Gilchrist and Johnstone<sup>35</sup> devised a three-stage program also very similar to Young's.<sup>32</sup> This involved -

- 1) development of basic techniques and principles;
- 2) performance of some interesting and chemically orientated studies designed to illustrate the role of chemists; and
- 3) a self-chosen project on any topic not necessarily related to any of the previous experiments. Only a quarter of the time was spent on the third stage.

This course was for non-scientists and had a low staff: student ratio (4:1). The course was enthusiastically received by the students although this could possibly have had something to do with the small numbers.

Friedmann,<sup>36</sup> Wehry,<sup>37</sup> and Silbermann and McConnell<sup>38</sup> all reported on courses they had devised which progressively gave the student more freedom after initial instructions in basic techniques had been given. All these reporters indicated that student interest was generated, though Silbermann and McConnell found that in their open-ended experiments the students were initially /

initially apprehensive but eventually calmed down and began working well.

The courses were run with fairly low numbers (50-60) and involved standard experiments - all procedures supplied - as an introduction to the course.

However, not all courses were so structured. Wilson<sup>39</sup> after an introductory talk which involved a course on how to use the library, started students off with a set inorganic synthesis but then allowed them to devise their own experiments which were loosely based on an aspect of the original synthesis.

Newman and Gassman<sup>40</sup> in a course devised for chemistry majors allowed students to devise their own experiments based on objectives which were discussed in laboratory lectures. This course tried to develop a research atmosphere and techniques were taught as they were required.

King<sup>41</sup> reporting on the Final Report of the Committee on Curriculum Content Planning (Massachusetts Institute of Technology, 1964) suggested a similar approach to Newman and Gassman. The report suggested that the student early in his physics undergraduate career was exposed to two twelve-hour project laboratories where the idea was not to learn specifics or demonstrate principles but to give the students a chance to work on an extensive experiment thus receiving experience of the experimental approach and getting in and out of difficulties by himself. Doyle and Mungall<sup>42</sup> in extolling the virtues of individualised projects which they reported as resulting in greater student enthusiasm, developing self-confidence and providing a greater variety of experience allowed their students to become involved in an original research project on the synthesis of an elusive human sex attractant.

In Australia Murray and Westward<sup>43</sup> and Brennan and Fletcher<sup>44</sup> reported on a course developed at Flinders University /

University for first and second year physics undergraduates. The students were encouraged to treat experiments as small research experiments and apart from one or two specialised demonstrations were taught techniques when required. However, the courses were expensive in setting up and the intake was very restricted.

Beard<sup>45</sup> reported on similar trends to make laboratory courses 'open-ended' or research orientated in this country and quoted examples from engineering, physics, medical and chemistry courses. She claims that student interest was aroused and that the staff also got more satisfaction in running these courses. However, in this country trends towards project work at tertiary level had not been so pronounced. One notable exception to this has been the Degree by Thesis introduced at the University of Sussex. Here students after two terms at the University took up a research project which from then on is their primary commitment. Their final assessment is based on their performance in the lab. Eaborn<sup>46</sup> suggested that this type of course could develop the student's originality, individuality and creativity. In a further report<sup>47</sup> after one year of the course Eaborn found that some of the difficulties foreseen by critics had not materialised. Students had not proved to be incompetent or even dangerous in a research laboratory but had accepted their limitations and approached matters with care and caution. Furthermore the students had developed confidence and a feeling of belonging to a research group.

Mathias<sup>48</sup> in an evaluation of the course found that the students involved positively enjoyed their course and thought that it was a truer reflection of their ability and potential. The students were able to undertake original research successfully and had developed qualities of independence and motivation. Few students abandoned the course. However, of those who did there were two reasons quoted:-

1)

- 1) chemistry was not their main subject and they regretted taking it; and
- 2) some students wished a more structured course.

One side-effect of the trend towards open-ended and research orientated experiments has been a blurring of traditional distinctions within subjects.

Coyne<sup>49</sup> pointed out that this resulted because an examination of a substance or principle with respect to a specific technique could not succeed in presenting experimentation as an unfolding, open-ended, creative and thus highly personal process. Classical experiments had robbed the students of the excitement of research and an appreciation of its complexity.

This led to an integrated course being developed where the work was covered in modules which consisted of techniques grouped on a natural or essentially non-classical basis, e.g. the synthesis of an inorganic compound would be followed with characterisation by appropriate physical techniques and by measurement of its reactivity.

Many examples of integrated laboratory courses can be found in the literature. For example, the papers presented at the Renesselaer Polytechnic conference on laboratory instruction in chemistry report on a variety of these.<sup>50</sup>

In her article, Coyne also suggested group experiments which could be interdisciplinary. Portions of the problem could be allocated to students with particular interests. She also thought that the introduction of modules would mean that a core laboratory could be developed which consisted of structured exercises where essential techniques and concepts could be introduced. For students intending to study chemistry further optional assignments, possibly designed in part by the student, could be added where desirable.

Valeriotte<sup>51</sup> described an extension of Coyne's approach which would allow for self-pacing. The major aim of this course was to allow for differences in student background and to minimise these.

Each term students had to do a certain number of set experiments and if they wished optional extra experiments designed to reinforce the techniques covered in the set experiments. Each set experiment also involved a pre-laboratory assignment which had to be completed satisfactorily before commencing.

The conclusion to this experiment suggested that students decided to use their time more efficiently and liked the flexibility. The results were no less satisfactory for the self-paced as opposed to the normal groups.

The continued trend towards project work, open laboratories and independent studies designed to satisfy the need of the individual student made it more difficult to instruct large numbers in techniques at one time. In overcoming this problem some laboratory institutions developed teaching packages.

Long<sup>52</sup> developed 'laboratory learning modules' which allowed a student to teach himself techniques which were presented on audio- or video-tape.

Price and Brandt<sup>53</sup> evolved a completely self-instructional laboratory which also made wide use of audio- and video-tapes. This freed the student from the demonstrator and allowed him to 'walk-in' to the laboratory at any time. This approach was another method of allowing for self-pacing.

An audio-tutorial approach to laboratory work has been introduced in a Biochemistry course for medical students at Dundee University by Garland.<sup>54</sup> Students were guided through the practical course at their own pace /

pace by means of audio-tapes and other illustrative or explanatory material. The course was split into units and the students were expected to achieve mastery.

In Canada at McMaster University similar arrangements have been in operation for many years.<sup>55</sup>

Brooks<sup>56</sup> while approving of the audio-tutorial method noted the necessity of high initial investment in equipment and renovations for sizeable freshman classes. He instead developed a station system where each student was assigned a bench space. Each station area was used every week by ten or more students.

This method, however, appeared to restrict students to set experiments and did not allow scope for open-ended experiments.

Brubaker Jnr., Schwendeman and McQuarrie<sup>57</sup> suggested a way to cope with large numbers of students not majoring in physical sciences. In this program laboratory sections alternated between laboratory practice and filmed experiments.

In the filmed session the students were first briefed and then shown the film. A discussion was then held and a repeat showing of the film given. They saw this as a way of forestalling the abandonment of classes in laboratory work in general chemistry.

Ben-Zvi, Hofstein, Samuel and Kempa<sup>58, 59</sup> discussed the results of a project on the effectiveness of filmed experiments in High School Chemical Education.

Two groups were formed one of which carried out the experimental work as 'normal' and the other who were subjected to filmed experiments.

Both groups were extensively tested and the results indicated that both groups achieved similar results with /

with students who received the normal course achieving slightly better on the manipulative components.

In a further paper Ben-Zvi et al<sup>59</sup> looked at the attitudes of the two groups and found that students rated the educational value of the medium-based approach to laboratory work as being distinctly less than that of personalized experimental work.

These approaches<sup>57 58</sup> were developed in response to logistic and economic problems. Coyle and Servant<sup>60</sup> reported on a different use of films in laboratory practice. Their idea was to attempt to develop some of the higher level skills which are often unreached in normal practical courses.

Harding<sup>61</sup> has pointed out that not all the features of the scientific method could be reached even in project work. An approach to achieving these features had been suggested by Nedelsky<sup>62</sup> who postulated that more abilities could be tested through criticism of the performance of another person.

Coyle and Servant showed a film on Noble gases which showed the synthesis of krypton fluoride. Students were expected to produce a write-up. Results showed that students had achieved a greater critical understanding of the experiment than the control group. They suggested that an occasional exercise of this nature would be useful in developing higher abilities.

It is interesting to note that Epstein<sup>63</sup> had exposed students to research papers and conditioned students to pose questions about the content and future approaches to the work. His idea was to show how a scientist works but it also helped to develop critical abilities, and was trying to achieve similar ends.

Modern technology has produced the means of developing another possible aid to teaching in the laboratory and that is the computer.

Asycough<sup>64</sup> listed three areas where he believed that the computer, used in a teaching role, had advantages over our more traditional forms of instruction. These were in -

- 1) the provision of a method of self-testing and remedial instruction on the background to the experiment to be performed;
- 2) the involvement of the student in planning the experiment or sequence of experiments; and
- 3) the simulation of certain types of experiment where the collection of sufficient data is unnecessarily time-consuming or where the data is experimentally inaccessible in the laboratory situation.

Thus a computer can give a measure of individualised instruction taking into account student background. The use of a computer may also free the instructor from many chores.

Asycough and co-workers used a 'strand' system for helping the student to acquire knowledge of the essential background theory of an experiment and in planning an experiment. The 'strand' method essentially means that the computer always leads, by means of a sequence of questions, to which the student must respond. The alternative to the 'strand' method is the 'file' method where information of various kinds is stored in files which are structured and from which the student can draw the information which he requires. This latter method has proved more difficult for students.

Kenzie<sup>65</sup> discussed a method by which computer-assisted instruction (CAI) might be used to make actual laboratory experience more meaningful for the student. He thought that chemical problems could be formulated and pursued in the abstract but the student could not acquire the full meaning until he can think about it in terms of laboratory instruction.

This /



This led him to listing several phases of inquiry practice:-

- a) establishing an intuitive grasp of the theoretical hypothesis - student could query computer for more information on terms requiring explanation;
- b) translating theoretical hypotheses into laboratory-tested propositions;
- c) deciding on general features of laboratory procedure;

In phases b) and c) student could request standard categories of information, e.g. directly measurable qualities and materials, apparatus from the computer.

- d) gaining prerequisite laboratory experience;
- e) designing a detailed experimental procedure.

The student then proceeds to carry out the experiment and interpret results.

The third use of computers was to simulate data. Computer simulated experiments can provide a student with practice in manipulating individualised experimental data after he has received practice in experimental technique.<sup>66</sup>

Schwendeman<sup>67</sup> has used computer technology to generate individual experimental data for students and to introduce random errors in the data.

The present philosophy of laboratory work at tertiary level can be categorised under two main headings:-<sup>68</sup>

- 1) Specific objectives - basic skills, techniques applying skills to novel situations, design of experiments, recognizing data needed for a specific problem, communication about the purpose, approach results and dependability of the experiment;
- 2) General objectives - acquisition of a feel for how chemical knowledge is obtained and how a chemist /

chemist thinks and works. A firm understanding of the methods of science.

Nearly all innovations directed towards achieving these aims were designed to -

- a) aid the learning process; and
- b) to let students discover that the method of science is exciting, challenging and intellectually rewarding.

Many methods of achieving these aims have been developed over the last decade and a half. Some of these have been due to technological advances and others through an attempt to get the student to think for himself.

#### 1.4 Assessment of Practical Work

Many different methods have been devised to assess practical work. At the tertiary level there are six common methods used, usually not exclusively. These are:-

- 1) grading of laboratory reports;
- 2) assessment of 'in vitro' performance in laboratory;
- 3) laboratory performance tests;
- 4) paper and pencil tests;
- 5) quiz - written or oral;
- 6) dissertations or theses.

Commonly a student's performance on a practical is based partly on the student standard at the bench and his final written report. This has been criticised as being too subjective.

Pickering and Kolks<sup>69</sup> report on a wide variation in teaching assistants' (demonstrators') abilities to grade students' performance. Teaching assistants were asked to grade students during a practical examination. Results showed very little correlation between their /

their grades and the grade as determined by the examination. In fact, in one case in six, the teaching assistants' grades were negatively correlated with the marks on the practical examinations. They point out that a grading system is only as good as its poorest markers.

Millington and Russell<sup>70</sup> collected and photocopied ten student reports and presented them to five demonstrators to grade. The demonstrators used marking guides. They found that on a scale of ten marks there was an absolute variation of four. The mean range was 2.4. There was also a poor correlation between ranking of reports, on one experiment it was only 0.71. They found that students were unclear as to the purpose of the assessment of reports.

Eglen and Kempa<sup>71</sup> compared three methods of assessing manipulative skills in the laboratory:-

- 1) a detailed checklist which required a simple yes/no decision;
- 2) open-ended - teacher assessed student on a grade from 1 - 5 without reference to achievement criteria;
- 3) intermediate - teacher assessed student on four sub-categories of manipulative skills which were -
  - i. methodical working;
  - ii. experimental technique;
  - iii. manual dexterity;
  - iv. orderliness.

Eighty-eight teachers evaluated students' performances which had been recorded on film. Mean grades were highest for the checklist and lowest for the intermediate modes. Divergence of grades was highest for the open-ended mode. Results for the other two methods were similar. Variances even by the checklist method were found to be far from zero.

Another /

Another form of assessment is that of laboratory performance tests. This type of test involves a student carrying out a prescribed piece of work, normally a skill or manipulative technique but sometimes more involved, such as using chemical facts in a laboratory situation. This differs from assessment of a student in a practical session as it is carried out under examination conditions.

Horton<sup>15</sup> first devised these tests, to evaluate possible outcomes of laboratory work not detected by paper and pencil tests. He first determined what skills and manipulations were required in the experimentation and then devised methods of measuring them objectively. He selected two experimental topics and with the help of 'competent' teachers produced a sequence of twenty items for each experiment which represented the actual steps in the performance. Students were evaluated by ticking off the items on the checklist as they were completed. The reliability of the test was high.

Hendricks<sup>72</sup> listed reasons why performance tests had not caught on. This was due mainly to the length of time required to administer the tests and the time required for setting up and marking the tests. The practice in these tests had been for a student to be marked by a single member of staff. Hendricks suggested that tests could be constructed which would allow groups of students to be assessed by a single member of staff. The use of standard instead of special equipment would also, Hendricks felt, overcome some of the criticisms.

Wall, Kruglak and Trainor<sup>73</sup> in physics quoted three advantages of performance tests:-

- 1) gave a spread of results;
- 2) presented a challenge to students;
- 3) could be used as a tool to measure effectiveness of different teaching methods, viz. individual versus demonstration method.

The tests /

The tests that they devised consisted of short items such as focussing cross-hairs on a telescope and longer items such as calibrating instruments and using them to measure variables.

Assessment, as in the case of Horton, was by a detailed checklist.

Kruglak<sup>74</sup> published the results of an intensive study of laboratory performance (achievement) tests. He suggested that a detailed list of objectives was a necessary pre-requisite as they made it easier to evaluate outcomes. Kruglak constructed and tested items on samples of over two hundred students. His results indicated -

- a) a high reliability when a detailed key was provided; and
- b) high discrimination for performance items.

Because of numbers these tests had to take place over a period of a week but he found that leakage of information was only a problem on items which had a high theoretical content and was minimal on items with a high performance component. Thus, he suggested, performance tests were measuring more than acquisition of knowledge.

A method which has been in use for a long while is that of paper and pencil tests. These tests measure the students' knowledge of practical procedures. They are frequently objective in style, i.e. posing a question such as asking for the reading on a burette and posing four alternatives from which the student has to choose.

Hendricks<sup>75</sup> found that these tests were less time-consuming than performance tests which disrupted the time schedules of teachers and students.

Kruglak<sup>76</sup> noted two limitations of paper and pencil tests:-

- 1) /

- 1) they cannot measure creative aspects; and
- 2) they cannot measure psychomotor skills, only the knowledge of them.

However, Kruglak also noted certain advantages in that:-

- a) they could cover a large number of topics;
- b) they could evaluate many aspects of a single topic;
- c) they were a research instrument for comparing different teaching methods;
- d) they gave supplementary evidence of achievement.

He also noted certain disadvantages:-

- a) they were artificial;
- b) students' thought processes, although they may be valid and original, are not apparent as they are not covered in distractors.

In a further paper Kruglak<sup>76</sup> said that paper and pencil tests designed to measure specific laboratory outcomes had few elements in common with laboratory performance tests measuring more general and difficult tasks.

Kruglak<sup>77</sup> investigated further the relationship between paper and pencil tests and performance tests. He found low correlations between the two and concluded that paper and pencil tests were poor substitutes for performance tests.

The fifth method of assessing practical work is that advocated by Secrist<sup>78</sup> which was to evaluate students by means of a short written quiz. This he suggested would motivate the student and do away with the problems of grading written reports. For large numbers written quizzes were thought more practical than orals. An oral is useful, however, when a student is expected to define a point of view or to think creatively about points raised by his work.<sup>79</sup> This type of objective can more easily be covered in a free ranging discussion.

The last method of assessment is to ask the student to submit a thesis or dissertation. This approach is normally used where a student has completed a project or research problem and in conjunction with an oral. See, for instance, Mathias.<sup>48</sup>





## C H A P T E R 2

A Review of Practical Work at Glasgow University  
and Development of a Model to aid in the Design of  
Laboratory Courses

## 2.1 Different Approaches to Practical Work

There are many different approaches to practical work. The following list is a summary of these:-

- a) Traditional : A traditional experiment is one in which the student is expected to carry out the experiments in a pre-determined way. The experiments contain detailed instructions on how to proceed. This type of approach is often described as 'cookbook chemistry' because a student can follow the instructions like a recipe, often without thinking. Experiments like this are often used to -
  - i) illustrate or reinforce theory;
  - ii) verify principles;
  - iii) illustrate techniques.
- b) Open-ended experiments : There are many ways of introducing open-endedness into a course and the degree of open-endedness can vary considerably. The open-endedness can be introduced as a problem to be solved at the end of a set experiment or as a project where a student or group of students, given very little help, are told to investigate a problem.
- c) Unit laboratory : This is a laboratory course based on a particular topic. This usually involves teaching the student skills and giving him practice in them and then setting the student a problem. A group discussion or tutorial may also be included.
- d) Demonstrations : Where the student observes a lecturer carrying out an experiment and is expected to learn about the techniques and methods used.
- e) /

- e) Audio-tutorial : The student is guided through the practical course at his own pace by means of audio-tapes and other illustrative or explanatory material.
- f) Keller plan : The student proceeds through a sequenced course at his own rate and is only allowed to progress to a new experiment when he has mastered the previous one.
- g) Research project : An original investigation of a chosen topic usually stretching over many weeks.
- h) Group participation experiments : Where a member of staff or demonstrator leads students through an experiment explaining practical procedures and theoretical background. However, students are expected to participate, i.e. take readings, make up solutions and where possible get practice in the techniques described. This could also be described as a guided experiment.  
A variation of this -
- i) Integrated laboratory : Where lectures and practicals are tied together over several hours. If a lecturer arrives at a point which needs practical illustration, he can do a demonstration which can include student participation. The results are then used in the rest of the lecture.

## 2.2 A Description of the Laboratory Courses in Chemistry at Glasgow University

The type of course varies from laboratory to laboratory and therefore it is best to describe each course briefly in turn.

First year : Class size :- normally over 500  
Time commitment - 3 hours a week

This course runs for three terms and is designed:-

a) /

- a) to enable students to acquire and become competent in the basic skills of practical chemistry;
- b) to illustrate, by set experiments, some of the theoretical concepts which have been introduced in the lecture course.

The first term is devoted to a skills course and a chance is given to the student to undertake remedial work to improve his skills.

The experiments in the second and third term are designed to allow students to use the skills acquired in the first term to make experimental observations pertaining to the lecture material. Objectives are quoted for each experiment.

All the experiments are traditional in nature and a report is required before the student leaves the laboratory. The assessment scheme is a simple  $\alpha$ ,  $\beta$ ,  $\gamma$ .  $\gamma$  is seldom given and indicates that the student's work is well below standard.  $\beta$  denotes an average performance and  $\alpha$  an excellent performance. Because the report is required on the same day only experimental data and a brief summary of results are expected.

Demonstrators preface each experiment with a short talk which should cover important practical and theoretical points. The students are expected to have revised the material prior to entering the laboratory. During the laboratory period demonstrators, each responsible for a particular experiment, circulate around the students, correcting technique, replacing broken apparatus, and helping students who are having difficulties.

The class is split into five sections which meet in two laboratories with fifty students per laboratory. Normally experiments are set up in each laboratory and the students move round these in a cycle. When the students have completed the experiments in one laboratory they move over to the other laboratory. Students work /

work in pairs.

Second year : Class size :- approx. 200 students  
Time commitment - 6 hours in 2 three-  
hour periods

The second year laboratory is split into three main courses:-

- 1) physical;
- 2) organic;
- 3) inorganic.

In addition to this, in the first and second terms there are two 'one-week' courses on spectroscopy and theoretical chemistry respectively, which are time-tabled to follow on from the completion of the lecture courses on the same topics.

1) The physical course is split into three three-week cycles involving electrochemistry, kinetics and thermodynamics. The students are required to complete a certain number of experiments in each cycle. These experiments vary in length. The aims are similar to the first year course.

A formal laboratory report is required and this is examined for, among other things, grammar and style. A five point marking system is used ranging from I - very good, to V - unclassified.

Demonstrators and staff allocate new experiments to students when they hand in laboratory reports. The students work in pairs.

The experiments are given in some detail although simple procedures, such as making a solution of a known concentration, are assumed to be known.

Demonstrators are available for consultation and will point out errors in technique, etc.

2) /

2) The organic course is run concurrently with the lectures although the experiments are not exactly in step.

The students work individually through a series of experiments where the emphasis is on superior work and not quantity.

Assessment is by submitting a written report which is then discussed with the member of staff or demonstrator. Samples of products are also obtained from the students. Marks are awarded out of ten for both results and understanding of the theory.

Again, demonstrators circulate round the laboratory to help the students.

The course consists of nine experiments. These are:-

1. Nitration of an aromatic compound - this is designed to give the student experience in dealing with aromatic compounds.
2. Mixture separation and aromatic bromination - designed to give the student practice in separating a mixture of two compounds, one of which is then brominated in the second half of the experiment.
3. The Sandmeyer Reaction - designed to give experience in an important type of reaction, which has to be carried out within a specified temperature range.
4. Identification of an unknown organic compound - determine the structure of the compound and prepare a derivative. This experiment is fitted in when time is available - during quiet spells in other experiments.
5. The Grignard Reaction - designed to give experience in another important class of reactions, which involves working in a water-free environment.
6. /

6. Condensation Reactions - dimerone synthesis - many students do not reach this experiment which again illustrates a class of reactions.
7. Unknown identification - involving two functional groups - this experiment is fitted in while the other experiments are being carried out.
8. The Beckmann rearrangement.
9. Extraction of piperine from black pepper.

The last two experiments are not normally reached.

At all times the students have to produce pure samples which have been checked by T.L.C. and melting point determination.

3) The Inorganic course - students again work in pairs. Three of the experiments involve the use of physical methods like diffraction and determination of magnetic moments.

Other experiments involve a study of a transition metal or a selected main group element. These experiments are usually divided into three parts:-

1. a study of some properties or reactions of the element;
2. an analysis involving the element; and
3. a synthesis of a compound containing the element.

Demonstrators are deployed as before and experiments last varying lengths of time. Although the experiments are intended to be completed within a three-hour period, they usually last longer.

Assessment is given out of twenty.

Third year : Class size :- 25-40 students

Time commitment - substantial, averaging  
16-20 hours a week

Again the courses are along classical lines - organic, inorganic, physical.

1) /

1) Organic - The overall aim is to produce a student at the end with the competence of a technician. To achieve this the course has several sections:-

- a) techniques course;
- b) multi-step synthesis;
- c) unknown compounds to identify;
- d) short project in groups of two or three students.

Assessment is similar to the second year organic course.

2) Inorganic - The students carry out experiments where possible of their own choosing. They are expected to complete about ten to twelve experiments out of a possible thirty-two. The experiments are placed into two categories; 'A' experiments which cover techniques and handling of data such as interpretation of mass spectra. Some of the 'A' experiments are compulsory. 'B' experiments are subjectively reckoned to be harder and more time consuming. A student usually completes fewer 'B' experiments.

Assessment is out of twenty, the marks allocated being split evenly between results and write-up.

Demonstrators are used as before.

3) Physical - week long sessions involving different branches of physical chemistry such as spectroscopy and theoretical chemistry. The sessions vary from performing a single experiment to an intensive techniques course.

Fourth year : Class size:- 25-40 students  
Time commitment - varies

First term : The student chooses three technique courses, one in each of physical, organic and inorganic.

Second term : Student undertakes a short research project under the guidance of a member of staff.  
Normally /

Normally an attempt is made to give the student some choice. The student eventually submits a thesis which is counted towards his final grade in the honours examinations.

### 2.3 A Discussion about Problems with Practical Work in the first two years.

All the practical courses in the first and second years, with the possible exception of the second year inorganic laboratory, have two main aims. These are:-

- 1) to teach the students techniques and skills; and
- 2) to illustrate the theory covered in the lectures.

The purpose of this section is to ask to what extent do the courses achieve these aims, and if they don't, to examine where they go wrong.

To determine whether the courses are being successful, it is possible to construct a list of six criteria, which would need to be satisfied before a course would have a reasonable chance of achieving these aims. Each criterion will be examined in turn and questions will be raised about points which warrant further investigation. To satisfy the above aims the course will:-

- 1) have to use a standardised approach for teaching techniques which the student can clearly see;
- 2) have to provide adequate tuition and opportunity so that the student can learn the skills to the standard required;
- 3) need to ensure that the student is familiar with the background theory to an experiment;
- 4) need to ensure that the student can relate what he is doing in the laboratory to the theoretical background, i.e. link together the theory and practical;
- 5) /



- 5) use assessment procedures sensitive enough to detect where students are going wrong and be consistent;
- 6) have to develop the correct attitudes in the student to encourage him to learn.

Let us examine each of these criteria in turn.

#### Criterion 1)

To avoid confusing the student each demonstrator has to teach the skills and techniques in the same way. To this end, the demonstrators need to be instructed to teach the same method and where this is not feasible it should be made clear to the student what alternatives are permissible.

In the first and second year courses no such instruction is given to the demonstrators on how to teach the skills. For example, skills involved in the handling of the burette and pipette can be taught in many ways and demonstrators may have developed their own idiosyncracies. For instance, some demonstrators point out to students that they should not take two hands to manipulate the stopcock of a burette, others use this method all the time. This can be a problem when a student has two different demonstrators, emphasising different methods on consecutive weeks.

The confusion, caused by this approach, in the student's mind may lead to the development of bad habits. Once bad habits start they can be difficult to eradicate especially with psychomotor skills which tend to become automatic and instinctive once taught. There is a need to develop a common approach to teaching skills in the laboratory.

#### Criterion 2)

In the whole of first and second year courses there is only one 'teaching package' in use and that is a first year tape/slide programme on how to use a chemical /

chemical balance. All other instruction is either by members of staff or demonstrators. This instruction is given by:-

- a) a talk usually before the student begins an experiment ;
- b) practice experiments - see section 4);
- c) reinforcement in future experiments.

a) First year and some second year experiments; the talk is given to a group of students which can be anything up to eighteen students.

In first year this talk is given at the beginning of the experiment. This can lead to problems:-

- i) as students may not have settled down or indeed arrived and many have not prepared beforehand for the experiment;
- ii) due to the size of the group, not all students may be able to see the equipment being demonstrated.

In second year, in many experiments where the demonstrator is only teaching at most four students, the problems from first year are lessened.

b) Practice experiments - returned to later.

c) If the reinforcement in future experiments is effective then it would be reasonable to assume that students would feel that they are mastering the techniques. To examine this the students were asked about their performance on three skills taught in first and second year organic laboratory courses. These were thin-layer chromatography (T.L.C.), recrystallisation, and use of melting-point apparatus.

In a survey of the second year organic laboratory (see Chapter 3) students were asked if they had -

- A) mastered this technique;
- B) needed further practice;
- C) /

- C) did not achieve the standard required - needed further practice before they could approach the standard required.

This was conducted by interviewing a sample of students for the first three experiments of the course.

A) Mastery

Experiment	1	2	3
T.L.C.	(56) 19	(45) 10	(43) 10
Recrystallisation	(38) 13	(50) 11	(43) 10
Melting Point	(-) -	(77) 17	(87) 20

B) Needed further practice

Experiment	1	2	3
T.L.C.	(38) 13	(41) 9	(41) 9
Recrystallisation	(50) 17	(45) 10	(52) 12
Melting Point	(-) -	(23) 5	(13) 3

C) Failed to achieve objective

Experiment	1	2	3
T.L.C.	(6) 2	(14) 3	(17) 4
Recrystallisation	(12) 4	(5) 1	(5) 1
Melting Point	(-) -	(5) 1	(-) -

Figures in brackets are percentages.

Sample sizes:- Expt. 1 - 34 students  
 Expt. 2 - 22 students  
 Expt. 3 - 23 students  
 Expt. 4 - 22 students

These results indicate that a simple technique such as using melting-point apparatus is soon mastered. However, the results for the other two skills suggest that even with frequent practice, they are not being mastered quickly. These three experiments are chosen because they are straightforward and should, therefore, give the student confidence.

The two skills of handling a T.L.C. and recrystallisation are essential to an organic course. The problem exists /

exists of how the student is to acquire these skills more effectively.

Criterion 3)

It is administratively impossible to ensure that students always receive lectures on the background theory before they begin an experiment. This occurs due to shortage of equipment which forces students in all courses apart from the second year organic course to perform experiments in a cycle. (See Section 2.2)

In extreme cases this means that the student carries out the experiment well before the appropriate lectures or a long time afterwards. Thus the theory contained in the lectures and the experiments in the practicals are normally out of phase.

Therefore, it is essential for a student to prepare for each experiment, so that he can understand what he is doing. The responsibility lies with the student as no check is made on his level of preparation.

In the second year organic laboratory the students do not perform experiments in a cycle but work through a series of experiments independently. A lecture course is running at the same time although no attempt is made to correlate exactly the laboratory and lectures. Indeed this would be impossible since students work at their own pace.

In the survey of this laboratory students were asked two questions to discover the level of preparation.

- 1) Before you began this experiment did you understand the background theory such as the reaction mechanisms? Yes / No / Not sure

Experiment	1	2	3
Students who claimed to understand theory	11 (33%)	6 (27%)	13 (56%)

Many /

Many students were not clear about the theory before they started. This may be due to the time lapse between lectures and experiments.

Students were then asked:-

- 2) Did you revise the material before you started the experiment? Yes / No

Experiment	1	2	3
Revised before starting	4 (12%)	2 (9%)	4 (17%)

Even with a poor understanding of the theory students do not appear to revise the theory and procedures before starting. In experiment one this may be because the students were keen to start the course but this does not apply to experiments two and three.

Criterion 4)

A further question arises because of section 3). Do the students try to relate the background theory to the practicals contained in the laboratory manual or do they follow the experiments like a recipe in a 'cookbook'? In the second year organic laboratory we asked the students about this.

Question:- Did you understand why you followed the sequence outlined in the laboratory manual for this experiment? Did you manage to link together the theory and the practical? Yes / No / Not sure

If the students answered 'No' they were then asked:- Were you just following what was outlined in the laboratory manual like a recipe? Yes / No

Experiment	1	2	3	4
Yes	15 (44%)	13 (59%)	10 (43%)	7 (32%)

This means that almost half the students interviewed for experiments 1 and 3 and over half in experiment 2 admitted to following the experiment 'cookbook' fashion. They learnt the background theory at the end of the experiment and did not attempt to understand it as they went along. This approach is encouraged by giving detailed /

detailed instructions for the student to follow.

On the other hand experiment 4 made students think more about what they were doing. This is not surprising as the students had to solve a problem. However, 27% of the sample claimed that they attempted to understand the procedures but felt that some of the tests and possible derivatives used theory with which they were not familiar. Thus they were forced to follow instructions without understanding why.

These results suggest that to get the student to think about what he is doing while in the laboratory, we may have to free him from too detailed instructions.

In the laboratory we need to ensure that:-

- a) the student is prepared for an experiment; and
- b) look for approaches which encourage him to think about what he is doing while in the laboratory.

#### Criterion 5)

Assessment is necessary for two reasons:-

- 1) to provide feedback for the staff as to the efficiency of the course and to put students in order of merit;
- 2) to provide feedback for the student so that he knows where he has gone wrong.

A closer examination of assessment procedures in the first and second years can show to what extent these aims are met. If we examine the assessment methods for each year in turn we can get a clearer idea of the success of the methods used.

Millington and Russell<sup>70</sup> examined the marking system for the first year laboratories at the time when a mark was given out of ten. They found wide discrepancies in demonstrators' marking with as much as a four mark variation between demonstrators on the same student's /

student's report. They also found poor agreement between demonstrators on the ranking of reports. This system has since been replaced by a three-point scale -  $\alpha$ ,  $\beta$ ,  $\gamma$ . This allows for less precision and thus more scope for disagreement about rankings. The laboratory report is now handed in on the same day and therefore is required to be only a summary of results as the student does not have time to elaborate.

A demonstrator also has to give an assessment of a student's practical abilities as shown in the laboratory. No clear directions are given on what points to look for. And, if demonstrators stress different points of a technique as being important, a student could be marked down by one demonstrator for practical technique and marked up by another demonstrator. This is shown by Pickering and Kolks.<sup>69</sup>

Thus the grading is very subjective and cannot adequately meet either of the two aims of assessment.

In the second year more time is taken in evaluating the student's performance as the student is questioned on his report. The report is expected to contain an introduction, experimental, results and discussion sections. The student is also given an idea about what the mark he has been given means.

Thus there is a greater probability of the assessment being fair and providing useful information for both staff and students.

However, one effect which tends to reduce the accuracy of results is what can be termed a 'grapevine effect' where students for many reasons swap results and laboratory reports. In some laboratories this is more widespread than others. Thus the student's results may not be original.

Marking of laboratory reports and grading of students tends to be subjective. If a practical 'objective' alternative /

alternative could be found this would at least provide extra information on which to base an assessment.

Criterion 6)

There is one final ingredient for a successful laboratory course and that is the attitude or motivation of the student. His attitude may be determined by many factors:-

- a) his feelings towards the practical course;
- b) " " " " chemistry in general;
- c) " " " " staff and demonstrators;
- d) " " " " relevance of what he  
is doing;
- e) how he feels on the day;
- f) his assessment of his performance;
- g) his view of the worthwhileness of the work.

Many of these may be outwith the control of the laboratory but some courses can be designed to ensure the greatest probability of his developing the right attitudes. By satisfying the other five criteria the laboratory course can go a long way towards getting the correct attitude.

However, the student needs to develop correct attitudes towards learning, safety, cleanliness and honesty. All four may be developed as a by-product of the course. However, let us examine the student's attitude towards learning more closely.

In the second year organic laboratory interview survey students were asked on completion of an experiment if they found any particular features of interest, such as the theory, practical techniques, etc. The results were:-

Experiment	1	2	3	4
'Interest'	8 (24%)	4 (18%)	9 (39%)	13 (59%)

Experiment 4 'identification of an unknown organic compound' /



compound' appeared to the students as the most interesting. On further questioning students stated that this was because it presented them with a challenge; or a problem that they had to solve. This experiment also stimulated curiosity in the chemistry as some found interest in looking for suitable tests or derivatives.

Students who were interviewed for experiment 4 were asked if they had enjoyed working on their own.

15 students (68%) replied Yes

2 students (9%) replied No

2 students (9%) replied 'No difference'

3 students (14%) did not answer

(sample size 22)

The two students who did not like the approach complained that it introduced a 'competitive spirit' into the laboratory.

A survey of the first year laboratory course was conducted earlier by McCallum and Johnstone<sup>81</sup> who found that:-

- a) 39-40% of the students found discovering facts for themselves rewarding; and
- b) 42% would have chosen to be taught by projects.

If this open-ended or project approach to practical work interests the student this is reason enough for trying to introduce some work of this nature in all years of an undergraduate course. The problem arises of how this can be done in first year when there are over five hundred students, limited equipment and a restricted supply of demonstrators and staff.

## 2.4 A Model for Laboratory Teaching

The criticisms of the laboratories outlined in the preceding section can be grouped under two main headings. /

headings. These are:-

- 1) there is little emphasis on learning skills and ensuring a minimum standard of competence. This occurs through the teaching methods and the assessment procedures used.
- 2) there is too much 'cookbook' chemistry which does very little to stimulate anyone but the most motivated student into thinking.

It is necessary to redefine the aims of the laboratory courses to overcome these criticisms and to produce a course which can teach the required skills efficiently and stimulate the student to think for himself.

There are then two aspects involved in laboratory teaching in these years:-

- 1) a learning component, and
- 2) an experience component - where the student gets experience of thinking for himself and reinforces his previous learning.

This poses two questions:-

- 1) What should the student learn? What are the objectives?
- 2) How do we get the student to think for himself? What are the objectives?

The answer to what the students should learn will to some extent depend on the course.

In the learning cycle the student will need to learn:-

- a) skills involved in using equipment efficiently -
  - 1) to identify various pieces of laboratory equipment by name;
  - 2) to state the function of each piece of laboratory apparatus;
  - 3) to manipulate scientific apparatus to a stated accuracy;
  - 4) /

- 4) to select the correct apparatus for the required accuracy;
  - 5) to assemble scientific apparatus to achieve a required function;
  - 6) to realise if the apparatus is not functioning properly;
  - 7) to keep equipment clean and chemicals pure.
- b) skills involved in observing and recording -
- 1) to follow written or oral instructions;
  - 2) to observe materials under investigation;
  - 3) to observe changes in materials under investigation;
  - 4) to observe readings to the required accuracy;
  - 5) to record measurements and to present this information in tabular and/or graphical form;
  - 6) to collect and classify data;
  - 7) to assess validity of data.
- c) the requisite background theory which will allow him to understand what he is doing.

All the objectives involve three components -

A knowledge of how to	}	achieve the objective.
An ability to		
A willingness to		

What skills does the student require to work efficiently for himself?

The student will need to gain practice in applying what he has learnt previously. This involves in addition to previous objectives, allowing students to:-

- a) plan their own work;
- b) carry out their plans;
- c) evaluate the results;
- d) which may lead to further work and modification of the original plan.

Involved in a), planning an experiment, are such objectives as:-

- 1) /

- 1) identification of the problem;
- 2) obtaining relevant information - maybe using a library;
- 3) recognising problems capable of practical solution;
- 4) devising an appropriate experiment;
- 5) devising/selecting an appropriate technique;
- 6) making and defending decisions.

b) performance of an experiment, is the efficient use of equipment and observational skills.

c) evaluation of an experiment, has such objectives as:-

- 1) analysis and interpretation of data;
- 2) drawing of justified conclusions;
- 3) acceptance/rejection of hypothesis.

d) has as objectives:-

- 1) suggested improvements in experimental approach;
- 2) presentation of report.

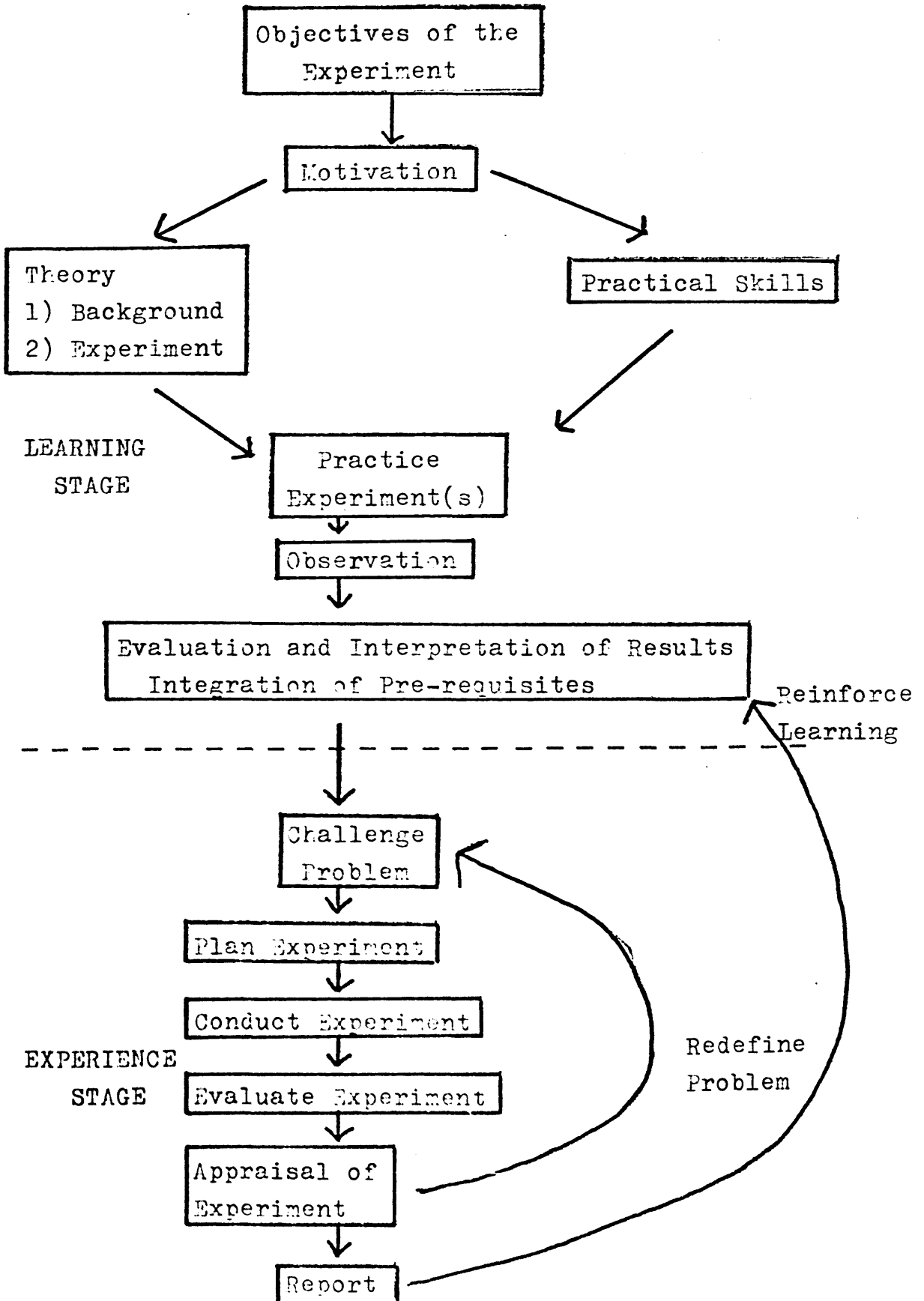
Other skills which a chemist needs to develop are:-

- 1) ability to make compromises;
- 2) to see interconnections across subject barriers;
- 3) to critically evaluate previous work;
- 4) to discriminate between fact and opinion;
- 5) to work as part of a team;
- 6) to be able to communicate with other students;
- 7) to see interconnections within a subject.

These skills may be needed at any time and would normally arise as by-products of the laboratory courses. However, it may be possible to teach for these through games and simulations.

The above lists of objectives suggest a model for laboratory work to achieve the twin aims of learning and experience. See figure 1.

Figure 1 : Proposed laboratory model to achieve the twin aims of learning and experience. (First and second years)



The laboratory model shows the different stages required to achieve the twin aims.

The first requirement is that the student is motivated. Without motivation no course can succeed. It is hoped that this model will develop motivation through -

- a) increased emphasis on learning - so that students know the standards required and see how they are expected to attain them;
- b) the use of open-ended experiments or projects to develop independence.

To begin an experiment the student will be expected to be familiar with the theory and may be tested on his understanding. Instruction will be given on skills by standardising techniques and use where necessary of teaching packages. The two components of theory and skill will be linked together by a practice experiment where the student may work as part of a group or on his own. In either case the student will be allowed to handle equipment to get experience. Practice in observation will similarly be given.

At this point, the student should have reached the stage where he can understand both theoretical and practical aspects of the experiment and can manage to link the two together.

To reinforce the learning and to present the student with a challenge a further stage can be added.

Here the student on his own or as part of a group is required to solve a problem. This can be introduced in two ways:-

- 1) as an extension of the practice experiment - open-ended; or
- 2) as a small project based on an application of the skills covered in the learning stage.

Due /

Due to the constraints already mentioned (see section 2.3) the choice of method and duration of the problem are limited especially in first year but it is felt that students will still receive sufficient stimulation to allow them to reinforce the skills already learnt and to make them think for themselves.

This model can be used in a similar fashion to that proposed by Rudolph and Venkatachalam<sup>33</sup> which was a learning/challenge cycle. This approach has the advantage of letting the students apply their newly learnt skills immediately. However, a possible disadvantage is that students will become bored by the repetition.

A more flexible model is thus to be preferred where the first stage and the second stage may not follow in consecutive laboratory periods but be separated, with the second stage being covered several weeks later.

In the third and fourth years and maybe second year further stages can be added which could invoke group discussions of results and of research papers covering a similar field.

However, at any time in the course, special exercises like the Alkali Industry case study could be introduced to help the students develop further skills. These skills could involve:-

- a) development of a theory - to show scientific method;
- b) communication exercises - presenting a paper to a group of students;
- c) observation exercises;
- d) library exercises - one such exercise for third year students has already been developed.<sup>82</sup>

## 2.5 The Research Plan /

## 2.5 The Research Plan

The aim of the research embodied in this thesis has been to investigate methods of solving the problems posed in preceding sections.

In Chapter 3 we will examine self-report techniques for evaluating a laboratory course.

Chapter 4 - paper and pencil tests.

Chapter 5 - development of teaching packages for use in the laboratory, including assessment.

Chapter 6 - a method of checking that students are prepared for the practical classes - pre-lab exercises.

Chapter 7 - group participation experiments.

The above are all methods used to aid the process of learning in the laboratory.

In the final section of the research work we will look at ways of giving the student experience.

Chapter 8 - open-ended experiments and project work.

In the last chapter, Chapter 9, we will summarise the previous chapters and pose some ideas for future work.



In the first place, the student should be given a clear and concise statement of the purpose of the study. This should be done in a way that is understandable to the student and that is consistent with the objectives of the study.

In the second place, the student should be given a clear and concise statement of the procedures to be followed. This should be done in a way that is understandable to the student and that is consistent with the objectives of the study.

In the third place, the student should be given a clear and concise statement of the results to be expected. This should be done in a way that is understandable to the student and that is consistent with the objectives of the study.

## CHAPTER 3

### Self-Report Techniques

Self-report techniques are a class of research methods in which the subject provides information about his or her own thoughts, feelings, and behaviors. These techniques are used to study a wide variety of phenomena, including attitudes, personality, and social behavior.

There are several different types of self-report techniques, each with its own strengths and weaknesses. The most common types are surveys, questionnaires, and interviews.

Surveys are a type of self-report technique in which a large number of subjects are asked to complete a questionnaire. Surveys are often used to study attitudes and opinions.

Questionnaires are a type of self-report technique in which a subject is asked to complete a questionnaire. Questionnaires are often used to study personality and social behavior.

Interviews are a type of self-report technique in which a subject is asked to provide information about his or her own thoughts, feelings, and behaviors. Interviews are often used to study a wide variety of phenomena, including attitudes, personality, and social behavior.

Each of these techniques has its own strengths and weaknesses. Surveys are often easy to administer and can provide information about a large number of subjects. However, surveys are often subject to response bias and may not be representative of the population.

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## CHAPTER 3

Self-Report Techniques

## 3.1 Introduction

In this chapter different methods of collecting and assessing student responses to questionnaires and interviews are recorded. Information of this nature is sought since it can give an insight into the students' perceptions of the laboratory. For instance, this information can reveal:-

- a) the student's attitudes towards particular experiments;
- b) what the student perceives as the objectives of experiments; and
- c) how the student thinks he has fared in achieving these objectives.

There are two different methods for collecting this data - the questionnaire and the interview approach. With the questionnaire approach the advantages are that it is easy to administer, it is not difficult to obtain good sampling and it can be a quick method taking as little as a couple of minutes to complete. However, the reliability of questionnaires issued 'en masse' to students may be suspect since not all students may give the questions careful consideration.

On the other hand interviewing students is more reliable as students tend to take more time to consider answers when faced with an interviewer. However, interviews will normally last longer and it may be more difficult to obtain a good sample.

Two surveys of the second year organic laboratory course were conducted, one using questionnaires and the other using interviews. The results of both are analysed

### 3.2 Review of questionnaire techniques used in this thesis

In constructing a questionnaire many techniques can be used to collect information from students. Where possible a battery of approaches or techniques are used as this allows cross-checking of a student's replies and assessment of their reliability.

In this thesis three techniques are used. They are:-

- 1) Likert method;
- 2) Semantic Differential method; and
- 3) Objective Rating method.

1) The Likert method<sup>83</sup> was first developed in 1932. This method consists of a list of statements beside which is a scale ranging from strong agreement to strong disagreement on which a student can indicate his judgement. Usually the scale has an odd number of points so that the student need not commit himself if he is undecided.

Originally each point on the scale was given a value, e.g. strongly agree +3 ; undecided/neutral 0 ; strongly disagree -3 , and these scores were added up to give an overall attitude score. However, this may not be valid as it assumes that:-

- a) the statements are measuring the same dimension;
- b) the frequency of responses from agree to disagree form a normal distribution;
- c) the intervals are equal.

However, it is possible by this technique to recognise variations in attitudes and opinions.

2) The second technique is that developed by Osgood, Suci and Tannebaum<sup>84</sup> in 1957. Here students are asked to judge a concept, e.g. how you feel about practical work, by judging where their opinion lies. Usually this is entered on a seven point scale between a pair of bipolar adjectives such as good/bad, interesting/boring.

This /

This method is very rapid and easy to construct, however, it assumes that:-

- a) word pairs are opposites when, in fact, the interpretation of the meaning of a word may vary from student to student;
- b) adverbs such as extremely, very, fairly, slightly etc. do not mean the same to all people, i.e. the interval between extremely enjoyable and very enjoyable need not necessarily be the same as the interval between very enjoyable and fairly enjoyable;
- c) scores are on interval scales when they are in fact on ordinal scales, i.e. a student who strongly agrees with a given statement and is given a score of 3 does not necessarily agree three times more strongly than a person who slightly agrees and is given a score of 1. Such scores provide only an indication of the relative strengths of attitudes between different people.

3) A further technique for assessing student opinion is that of objective rating. Here a student is asked to judge how he thought he performed on an objective.<sup>84</sup> This can be done in various ways:-

- a) The simplest method of student self-assessment of objectives is to limit the choice to a straightforward alternative, e.g. Yes/No; Achieve/Did not achieve, etc. There is a danger with this method that the student's assessment of his performance may not fit into either of the categories although objectives which are not being achieved can be quickly spotted by the staff.

To make a student think more about his answers, it is possible to rephrase objectives. For instance, an objective in the second year organic course where no objectives are specified to the students /

students for any experiment is:-

"A method of separating out different isomers by shaking with ice-cold methanol"

This can be rephrased to:-

"A method of separating isomers"

This makes it more difficult for a student faced with a list of objectives for several experiments to guess at the objectives of a particular experiment. Thus he has to realise what he has done in the experiment before he can decide whether he has achieved it.

Comparison between the prescribed list of objectives and what the students have ticked as achieved can yield information about which objectives are being transmitted to the student. This technique was used by McGuire<sup>86</sup> to investigate the success of experiments at secondary level. However this technique can have two drawbacks:-

- i) objectives may not be ticked as they appear too trivial; and
- ii) the original objective specifies a standard. A rephrased objective may not give so much information about standards thus allowing students to set their own.

b) Give the student three choices as to how he fared on an objective:-

A : You felt that YOU MASTERED this objective - feel confident that you would be able to repeat it to the same standard in future experiments.

B : You felt that you DID NOT QUITE ACHIEVE MASTERY but with a little more practice you could.

C : You felt that you DID NOT ACHIEVE THE OBJECTIVE and that you would need MORE PRACTICE before you could APPROACH THE STANDARD REQUIRED.

This method allows the student three distinct choices. /

choices. The pattern of results achieved by this method will be discussed in the second of the two surveys.

- c) Give the student four choices - the maximum number of distinct categories which can be specified with accuracy. The categories are:-
- A : If you felt that you completely mastered the objective then place a tick in column A.
  - B : If you felt that you did not quite master the objective and were not completely clear about either the theory or the technique place a tick in column B.
  - C : If you felt that you learnt very little about the theory or technique place a tick in column C.
  - D : If you felt that you learnt nothing about the theory or technique place a tick in column D.

With methods b) and c) the objective can be used in its original form, which specifies the required standard.

### 3.3 A Questionnaire approach to Evaluating Experiments

For list of experiments see section 2.2.

The aim of this survey was to try to find a general method of evaluating experiments. The course which was evaluated was the second year organic laboratory course. A questionnaire was devised which asked the students about -

- a) their attitudes to each experiment; and
- b) their assessment of their achievement of the practical objectives for each experiment.

No attempt was made to evaluate cognitive outcomes. This was because to assess these objectives accurately would be a lengthy process and could result in losing the goodwill /

goodwill of the students towards filling in their questionnaires.

The questionnaire which was developed was in two parts (see Appendix 3.1).

Section A : Likert-type questions. Nine statements - the student was given a choice of three alternatives to choose from:- True, Fairly True, Untrue. This was similar to the method used by Gunning<sup>87</sup> for evaluating opinion at the secondary school level.

Section B : A list of objectives for the course. The objectives were rephrased using McGuire's<sup>86</sup> technique (see page 58 ) and put in a random order. (See Appendix 3.1 and 3.2.)

In 1975 there were one hundred and sixty-three students in the second year organic course. A questionnaire was issued to each student for each experiment. All questionnaires were collected but only the replies from benches with an even number were analysed. Sample sizes decreased as fewer questionnaires were completed as the course progressed (see Table 3.1).

Expt.	Sample size	% of total population
1	80	49
2	79	48.5
3	72	44
4	69	42
5	50	31

Table 3.1  
Sample Sizes

Not all students had time to complete experiment five. The sampling of the total population was unbiased.

The results of this survey were not considered very /

very reliable as it became apparent that many students did not take much care in filling the questionnaires. Therefore the results are not included in this thesis.

However, the survey was of use in indicating the limitations of this approach and was also valuable in learning questionnaire research techniques.

There were two main criticisms of this method of evaluating experiments:-

- 1) The choice was too limited for university students. This applied especially in section B where the method of objective rating was too restrictive and left the student to set his own standards of achievement.
- 2) A survey is dependent on the goodwill of students. It is stretching this to ask students to complete a questionnaire for each experiment when there are so many other demands and pressures on them in the laboratory.

### 3.4 An Interview-based Approach to evaluating Experiments

The course which was evaluated was the second year organic laboratory course. For list of experiments see Section 2.2. For list of objectives see Appendix 3.2. The course had not been altered since the previous survey. Because of the drawbacks of the questionnaire approach it was decided to devise an interview technique with the same aims.

The interview schedule was in two sections:-

- A objectives
- B attitudes and opinions

Section A : The objectives were detailed for each experiment. The student was given a card with three choices, A, B, C, (see p. 58 method b ) and asked to make /



make a choice for each objective. (The objectives for each experiment are detailed in Table 3.4, p. 69 .)

Section B : The questions in the interview were based on the laboratory model devised in section 2.4, p.50 . Thus students were asked about (see Appendix 3.3):-

- a) the theory and their preparation for the experiment: understanding of background theory; prior revision; how understanding of theory was altered by experiment;
- b) the practical: new practical techniques, confidence. Two questions, one about opinion of laboratory work and their results to determine if anyone had a soured opinion of the experiment because of a poor performance or a bad assessment.
- c) how they managed to link together the theory and the practical: did they think about what they were doing; did the experiment make sense of earlier lectures or experiments; any features interesting: and finally a subjective impression of the experiment.

Thus the schedule was structured as below (see Figure 3.1).

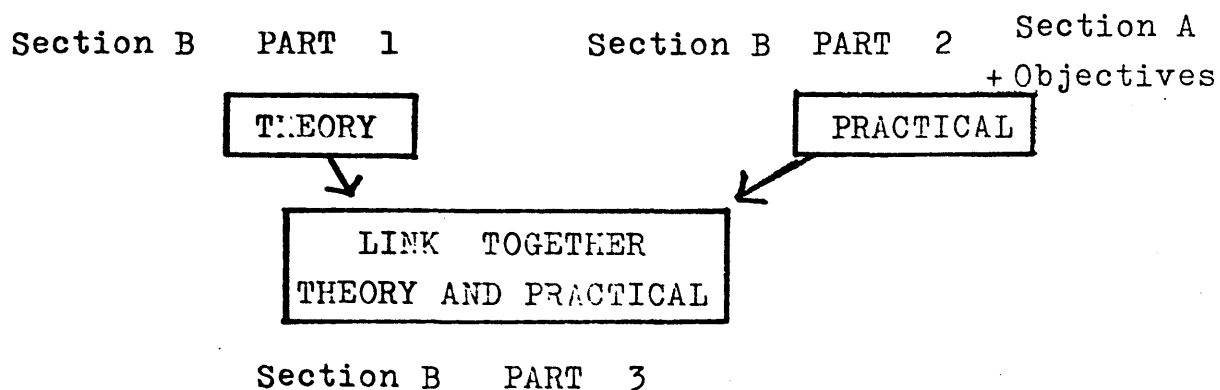


Figure 3.1

The interview took about five minutes per student and students were interviewed only after they had the experiment marked. Each student was interviewed only once. The target sample for each experiment was a fifth /

fifth of the total population of one hundred and sixty-eight students. This was achieved only for experiment one (see Table 3.2).

Expt.	Sample size	% of total population
1	34	20
2	22	13
3	23	13
4	22	13

Table 3.2

The decrease in sample size occurred for various reasons:- students were ill, some had withdrawn from the course, some reports not handed in till the end of term, etc.

To ensure a fair sample every fifth student was interviewed for an experiment, i.e. for experiment one students 1, 6, 11, etc. on the class list were chosen. For experiment two, students 2, 7, 12, etc. on the list were chosen.

Experiment five had to be excluded from the survey as not enough time was available to interview a sufficient number of students.

It was necessary only to make one change in the schedule after experiment one. This was due to misinterpretation by the students of objective five which was concerned with using melting-point apparatus.

The students' attitude towards the survey was very good and every student appeared to give fair and considered answers. No students interviewed had a soured attitude towards the experiment due to a poor performance or 'unfair' assessment and therefore the results give a fair reflection of student opinion about the course.

The /

The results are summarised in Tables 3.3 and 3.4, p.67. The results are quoted in percentages to aid comparison of results.

In Section A the method used to monitor student feedback on objective assessment appeared to give students a fair choice. The students had more choice than previously and this resulted in a more meaningful analysis.

An example can be seen in comparing the replies to the objective in experiment two on 'how to separate a mixture of two solid compounds'. In the questionnaire survey 86% of the students felt that they had achieved this (or at least recognised it as an objective). In the interviews it came to light that only 50% of the students felt that they had mastered this technique with the other 50% feeling that they needed more practice.

Results showed that students could quickly master objectives such as filtration under vacuum, weighing of samples etc. However, mastery of more complex tasks such as recrystallisation and T.L.C. was slower and students were still having difficulty at the end of the course.

In Section B of the schedule students were asked about whether they had learnt any new techniques. Apart from experiments one and two where about half said yes, the students claimed to have learnt no new techniques. Students who answered yes to this question were asked which objectives were new to them. In experiment one 27% (9 students) said the method used to separate isomers was new. This is low for a new objective. However, the other 'new' objective to be quoted frequently, recrystallisation 21% (7 students), should have been learnt in first year - students appeared to have forgotten this!

In the theory part of section B several trends showed /

showed up. Most of these have been discussed previously (see section 2.3, p. 37). However, they will be mentioned here for the sake of completeness:-

- a) Few students understood the theory prior to starting experiments one and two. For experiment three 59% of the students understood the theory. This figure probably arose due to the fact that the reaction was straight from the lectures.
- b) Very few students prepared for an experiment by revising the material before starting.
- c) Completing an experiment does have a positive effect on the understanding of the theory but this effect decreases with time. This may be due to a decrease in enthusiasm the longer a course continues.

In the third part of section B students were asked questions about how they managed to relate the theory and the practical. Two experiments managed to succeed in avoiding 'cookbook chemistry'. These were experiments three and four. It should be noted that students thought that experiment three (15; 65%) and experiment four (16; 73%) were 'good' and the level of interest shown by the students also increased to (9; 31%) and (13; 59%) respectively for these experiments.

This increase occurred for different reasons.

In experiment three the students could relate the theory and the practical because the theory was straight from the lecture where this type of reaction is well-covered. In experiment four students found it interesting because they were left to work on their own and to operate efficiently they had to understand what they were doing. This forced students to think for themselves.

In summary:-

Experiment one : a good experiment for revision of practical /

practical techniques but the students were poorly prepared for the theory.

Experiment two : poorest of the four experiments. 13 (59%) students followed it like a recipe and prior understanding of the theory was poor.

Experiment three : students seemed to understand the theory. The practical was lightweight and many students missed the point of controlling the reaction temperature.

Experiment four : presented challenge to students who seemed to enjoy the experiment.

The consequence of the results of these surveys has previously been discussed (see section 2.3, p. 37 and 2.4, p. 46 ). Although the questionnaire approach was more unreliable there was some agreement with the results from the interviews.

The method evolved for the second of the surveys yielded more useful and usable information although small samples meant care had to be taken in assessing results.

A variation of the questionnaire technique may be useful in first year where sample sizes would render an interview approach impractical.

Table 3.3

Figures in brackets are percentages (A - answer)

1 THEORY	Expt. 1	Expt. 2	Expt. 3	Expt. 4
1. Understanding of background theory				
A - Yes	11 (33)	6 (27)	13 (56)	-
A - Not sure	1 (3)	-	-	-
2. Prior revision for experiment				
A - Yes	4 (12)	3 (9)	4 (17)	4 (18)
3. Understanding of theory altered				
Improved	27 (81)	14 (64)	13 (57)	12 (55)
More confused	-	1 (4)	-	-
2 PRACTICAL				
1. Learnt new practical techniques				
A - Yes	16 (48)	9 (41)	1 (4)	3 (14)
3. Lab. mark				
Better than normal	-	3 (14)	6 (26)	4 (19)
Average	-	14 (64)	14 (61)	16 (73)
Worse than normal	-	5 (23)	3 (13)	2 (9)
4. Results - yields, M.Pt. etc.				
Good	-	3 (14)	11 (48)	6 (27)
Average	-	8 (36)	12 (52)	10 (46)
Bad	-	11 (50)	-	5 (23)
5. Became more confident in your approach to practical work				
A - Yes	20 (60)	10 (45)	10 (43)	11 (50)
A - Not sure	6 (18)	5 (23)	4 (17)	6 (18)
3 /				

	Expt. 1	Expt. 2	Expt. 3	Expt. 4
3				
1. Linking together of theory and practical				
A - Yes	18 (53)	9 (41)	10 (43)	9 (41)
Followed like a recipe	15 (44)	13 (59)	10 (43)	7 (32)
In-between	1 (3)	-	3 (14)	6 (27)
2. Experiment clarify earlier experiments or lectures				
A - Yes	20 (60)	11 (50)	12 (52)	11 (50)
Clarified lectures	16 (48)	9 (41)	12 (52)	11 (50)
Clarified experiments	-	1 (4)	-	-
Clarified expts. and lectures	-	1 (4)	-	-
Not sure	4 (12)	-	-	-
3. Did any particular features interest you?				
A - Yes	8 (24)	4 (18)	9 (39)	13 (59)
4. Experiment				
Good	18 (54)	8 (36)	15 (65)	16 (73)
Bad	2 (4)	2 (9)	1 (4)	-
Average	14 (52)	12 (55)	7 (30)	6 (27)
5. Working on your own - like it?				
A - Yes	-	-	-	15 (68)
A - No	-	-	-	2 (9)
A - No difference	-	-	-	2 (9)
No answer	-	-	-	3 (14)

Table 3.4

## Section A

Figures in brackets are percentages.

Objectives for EXPERIMENT 1	A	B	C
1. You know how to remove crude product by filtration under vacuum using a Buchner funnel.	(91) 31	(9) 4	- -
2. You can separate out different isomers by shaking with ice-cold methanol until the TLC shows no impurities remaining.	(41) 14	(44) 15	(15) 6
3. You can run a TLC to show the efficiency of purification using the most suitable solvent system.	(56) 19	(38) 13	(6) 2
4. You can recrystallise the crude sample of a product until you obtain crystals of the pure product as shown by the Melting Point or TLC.	(38) 13	(50) 17	(12) 4
5. You can record the M.Pt. - of the pure sample accurately to $\pm 1^{\circ}\text{C}$ of literature value.	(53) 18	(38) 13	(9) 3
6. You can calculate the percentage yield correctly.	(88) 30	(9) 3	(3) 1
7. You can record the Rf value from a TLC plate correctly to two decimal places.	(59) 20	(35) 12	(6) 2
8. You can weigh the final product to the required number of decimal places.	(91) 31	(9) 3	- -
EXPERIMENT 2			
1. You can separate a mixture of two solid compounds by acid/base extraction until the TLC shows none of the other solid present.	(50) 11	(50) 11	- -
2. You can purify the crude sample of a product until you obtain crystals of the pure product as shown by the M.Pt. of TLC.	(50) 11	(45) 10	(5) 1
3. /			



	A	B	C
3. You can run a TLC to show the efficiency of purification using the most suitable solvent system.	(45) 10	(41) 9	(14) 3
4. You can record the Rf value from a TLC plate correctly to two decimal places.	(64) 14	(36) 8	(-) -
5. You can calculate the percentage recovery of each compound.	(77) 17	(14) 3	(9) 2
6. You can record the M.Pt. of a pure sample.	(77) 17	(23) 5	(-) 1
7. You can weigh the final product to the required number of decimal places.	(100) 22	(-) -	(-) -
<b>EXPERIMENT 3</b>			
1. You can filter off a precipitate under vacuum using a Buchner funnel.	(87) 20	(13) 3	(-) -
2. You can purify the crude sample of a product until you obtain crystals of the pure product as shown by the M.Pt. or TLC.	(43) 10	(52) 12	(5) 1
3. You can weigh the product to the required number of decimal places.	(95) 22	(5) 1	(-) -
4. You can calculate the percentage yield correctly.	(100) 23	(-) -	(-) -
5. You can record the M.Pt. of a pure sample.	(87) 20	(13) 3	(-) -
6. You can check the purity of the product by TLC using the most suitable solvent systems.	(43) 10	(41) 9	(17) 4
7. You can record the Rf value from a TLC plate to two decimal places.	(83) 19	(17) 4	(-) -
8. You can carry out a reaction within a specified temperature range and ensure that the temperature is maintained within this range.	(64) 14	(30) 7	(9) 2

EXPERIMENT 4	A	B	C
1. You can crystallise the derivative until the M.Pt. is constant.	(64) 14	(27) 6	(9) 2
2. You can use distillation equipment to obtain a boiling-point accurately. (Not everyone attempted this - C)	(18) 4	(18) 4	(64) 14
3. You can check the purity of a product by TLC using the most suitable solvent system. (2 did not answer)	(32) 7	(55) 12	(5) 1
4. You can identify the nature of the functional group(s) present.	(73) 16	(23) 5	(5) 1
5. You can select suitable test(s) to check your hypothesis.	(45) 10	(55) 12	(-) -
6. You can record the results of the test(s) accurately and immediately.	(77) 17	(18) 4	(5) 1
7. You can record one R <sub>f</sub> value from a TLC plate to two decimal places. (1 did not answer)	(68) 15	(27) 6	(-) -
8. You can identify the nature of the carbon skeleton.	(64) 14	(36) 8	(-) -
9. You can determine the molecular formula given the approximate Molecular Weight and % elemental composition.	(91) 20	(9) 2	(-) -

## Appendix 3.1

Original Form of Questionnaire

University of Glasgow - Science Education Research  
Group

Throughout this term the Chemistry Department wish to monitor your reactions to the experimental work. Therefore we would like you to complete the following questionnaire EACH time you FINISH an experiment. Your returns will be treated in confidence, and therefore, you may be as frank in answering as you wish. We thank you for your co-operation.

Please complete the following:-

Experiment No.      Lab.      Bench No.      Lab Days  
-----

SECTION A - Please tick one of the alternatives to each question.

As a result of completing this experiment, I have -	A TRUE	B FAIRLY TRUE	C UNTRUE
1. become more interested in chemistry.			
2. become aware of new practical techniques.			
3. increased my knowledge of the theory covered by the experiment.			
4. become aware of the importance of safety procedures.			
5. become aware of the need for careful recording of results.			
6. gained confidence in my approach to practical problems.			
7. become aware of the limitations of practical work.			
8. reinforced my existing practical skills.			
9. increased my knowledge of the applications of chemistry to other subjects.			

SECTION B /

SECTION B - Will you please place a tick beside any of the statements which YOU believe you have achieved by completing THIS experiment.

By completing this experiment, I have learned -

1. A method of separating a mixture of two solid compounds.
2. Method(s) of testing for functional groups.
3. How to check the purity of a product.
4. A method of separating isomers.
5. How to measure a boiling-point.
6. A method of obtaining a constant melting-point.
7. To control the acidic properties of the reaction.
8. Methods of identifying the carbon framework of a molecule.
9. To use melting-point apparatus to obtain the melting-point of a compound.
10. A technique for drying the final product.
11. A method of separating the impurities from the final product.
12. How to calculate the percentage yield.
13. How to conduct a search for relevant chemical information.
14. To check the efficiency of product separation.
15. How to filter off a precipitate.
16. A technique for maintaining an experiment within a specified temperature range.
17. How to use distillation equipment.
18. Methods of avoiding contamination by water.
19. How to record a Rf value.
20. A method of determining molecular formula.
21. To confirm a proposed structure by spectroscopic methods.

## Appendix 3.2

List of Practical Objectives for each Experiment  
Numbers to the left correspond to the position of the objective in Section B for the original version of questionnaire.

(1) or (2) after an objective indicates that it was taught in first or second year.

Second Year Organic Laboratory - Objectives of Experiments

Experiment ONE - Nitration of an Aromatic Compound	
11	To remove crude product by filtration under vacuum. (1)
4	To separate out the different isomers by shaking with ice-cold methanol. (2)
3	To check the purity by TLC. (2)
19	To record the R <sub>f</sub> value from a TLC plate. (1)
6	To recrystallise the crude sample until the pure product is obtained and good crystals are obtained. (1)
9	To record the melting-point of the pure sample to within $\pm 1^{\circ}\text{C}$ of literature value. (1)
12	To calculate the percentage yield in grams. (1)
Experiment TWO - Mixture Separation and Aromatic Bromination	
1	To separate a mixture of two solid compounds by acid/base extraction. (2)
7	To purify the separated components by recrystallisation. (1)
14	To check the efficiency of separation by TLC. (2)
19	To record the R <sub>f</sub> value from a TLC plate. (1)
9	To record the M.Pt. of the pure sample to within $\pm 1^{\circ}\text{C}$ of literature value. (1)
Experiment THREE - The Sandmeyer Reaction	
15	To filter off a precipitate on a Buchner funnel. (1)
10	To dry the final product by using filter paper. (1)
6	/

- 6 To recrystallise the crude sample of a paper product until the pure product is obtained. (1)
- 12 To calculate the percentage yield in grams. (1)
- 9 To record the M.Pt. of the pure sample to within  $\pm 1^{\circ}\text{C}$  of the literature value. (1)
- 3 To check the purity of the product by TLC. (2)
- 19 To record the Rf value from a TLC plate. (1)
- 16 To carry out a reaction within a specified temperature range. (2)

Experiment FOUR - Identification of an Unknown Organic Compound.

- 2 To identify the nature of the functional group(s) present. (2)
- 8 To identify the nature of the carbon skeleton both aliphatic and aromatic. (2)
- 13 To use reference books to find relevant information. (2)
- 6 To crystallise the derivative until the M.Pt. is constant. (1)
- 5 To use distillation equipment to obtain a boiling-point recording. (2)
- 17 To check the purity of the product by TLC. (2)
- 3 To record the Rf value from a TLC plate. (1)
- 21 To use the IR spectra to collaborate structure. (2)
- 10 To determine molecular formula given approximate Molecular Weight. (2)

Experiment FIVE - The Grignard Reaction

- 18 To carry out a reaction in a water-free environment. (2)
- 9 To record the M.Pt. of the pure sample to within  $\pm 1^{\circ}\text{C}$  of the literature value. (1)
- 12 To calculate the percentage yield in grams. (1)
- 6 To check the purity of the product by TLC. (2)
- 19 To record the Rf value from a TLC plate. (1)

Experiment SIX - Condensation Reactions - Dimedone Synthesis

- 15 To collect a pure product by filtration. (1)
- 10 /

- 10 To dry the final product by using filter paper. (1)
- 6 To recrystallise the sample, until the pure product is obtained. (1)
- 9 To record the M.Pt. of the pure sample to within  $\pm 1^{\circ}\text{C}$  of the literature value. (1)
- 12 To calculate the percentage yield in grams. (1)
- 7 To maintain a constant pH environment. (2)
- 6 To check the purity of the product by TLC. (2)
- 19 To record the Rf value from a TLC plate. (1)
- 21 Confirm structure by using IR and UV. (2)

As stated above, the number on the left corresponds to the number on the questionnaire. These objectives were then rephrased to make them less obvious.

## Appendix 3.3

Second Year Organic Laboratory Questionnaire - 1976

Date: \_\_\_\_\_ Bench No. \_\_\_\_\_ Lab Days: \_\_\_\_\_

## 1. THEORY

1. Before you began this experiment, did you understand the background theory such as the reaction mechanisms? Y/N

N Was the theory behind this experiment new to you? Y/N

2. Did you revise the material before you started the experiment? Y/N

3. Was your understanding of the theory altered by doing this experiment? Y/N

Y Did the experiment improve your understanding? Y/N

N Did it make you more confused? Y/N

## 2. PRACTICAL

1. Did you learn any practical techniques that were completely new to you? Y/N

Y What were these?

N None of the objectives were new to you? Y/N

2. What was your laboratory mark? \_ \_ \_ \_

3. Was this better or worse than normal? \_ \_ \_ \_  
(laboratory mark)

4. What did you think of your results - yields, M.Pts., etc.?  
Did you think they were good, bad or average for you?

5. Did you think that you became more confident in your approach to practical work? Y/N

Not sure



3. 1. Did you understand why you followed the sequence outlined in the laboratory manual for this experiment? Did you manage to link together the theory and the practical?
- Y/N  
Not sure
- Y Everything made sense - you understood why you had to carry out all the procedures outlined? Y/N
- N Were you just following what was outlined in one laboratory manual - just like a recipe? Y/N
2. Did this experiment help you to make sense of any earlier experiments or lectures?
- Y/N  
Not sure
- Y Which?
3. Did any particular features of this experiment interest you? Y/N
- Y Which?
4. What did you think of this experiment? Was it good, bad or average?
- (g) In what sense?  
(b) In what sense?  
(av) In what sense?
5. (Unique to experiment 4)  
Do you enjoy working on your own? Y/N  
No difference

## CHAPTER 4

### 'Paper and Pencil Techniques' - Assessment of Practical Work by Written Examinations

The use of written examinations to assess practical work has been a subject of much discussion and controversy. It is often argued that written examinations are the only feasible means of assessing practical work, as they are the only method which can be used to assess the knowledge and understanding of the student. However, it is also argued that written examinations are not a valid means of assessing practical work, as they do not assess the student's ability to perform practical work. This chapter will discuss the advantages and disadvantages of using written examinations to assess practical work, and will provide some suggestions for how to design written examinations which are valid and reliable measures of practical work.

## CHAPTER 4

'Paper and Pencil Techniques' - Assessment  
of Practical Work by Written Examinations

## 4.1

A second method of assessing practical work is that of paper and pencil techniques. Here we try to assess a student's practical ability indirectly by his performance on a written test.

This method is only valid if you assume a high correlation between the student's performance on the test and his practical ability in the laboratory. In questions covering skills like graph drawing, treatment of errors, this is a reasonable assumption. However, in skills involving manipulations, handling of apparatus etc., this assumption may not always be valid. For example, knowledge of how to drive a car does not infer that you can drive the car. However, the advantage of this method is that it allows testing of a large number of students.

It was decided to introduce this method at the first year level for two reasons:-

- 1) because of the inadequate assessment scheme ( $\alpha, \beta, \gamma$ ), students needed more accurate feedback on their performance. Paper and pencil tests provided the only feasible means of producing this information.
- 2) it was felt that the introduction of these tests would produce a positive effect on the amount of learning achieved in the laboratory. Students would be encouraged to think about possible questions that could be posed.

A question paper was designed for first year. Because of the large number of students involved (over five hundred) the examination was composed of fixed-response questions with the student answering on a computer card. (See Appendix 4.1)

#### 4.2 First Year Practical Examination, December, 1975

The examination consisted of thirteen questions covering various aspects of the techniques course. The questions and discussion of the results are included in the item analysis.

#### Content of the Examination

Content	Question	Level
Reading of balance	1	Comprehension
Reading of balance	2	Comprehension
Safety	3	Comprehension
Burette reading	4	Comprehension
Errors	5	Comprehension
Chromatography	6	Comprehension
Titrations	7	Knowledge
Indicator	8	Application
Graph drawing	9	Application
Equipment usage	10	Knowledge
Equipment usage	11	Knowledge
Interpretation of data	12	Comprehension
Partition coefficients	13	Application

Questions were shredded by other members of staff and some were discarded before the final choice of questions for inclusion was reached.

In thirteen questions it is difficult to cover the whole course adequately. However, the examination was felt to be the optimum length, as it contained about the same number of questions as a standard objective (diagnostic) test on the lecture material which was given in a similar time period.

The questions were set to elicit information about the students' knowledge of facts through to application of procedures and principles learnt in the course. Questions set at the knowledge level include:-

a) /

- a) knowledge of facts;
- b) knowledge of the ways of dealing with facts; and
- c) knowledge of general principles.

The comprehension level represents the lowest level of understanding; i.e. when one knows and can make use of the material communicated without necessarily relating it to other material or seeing all its implications. The questions set at the application level involve abstracting from a particular situation and applying the abstraction in other ways.<sup>88</sup>

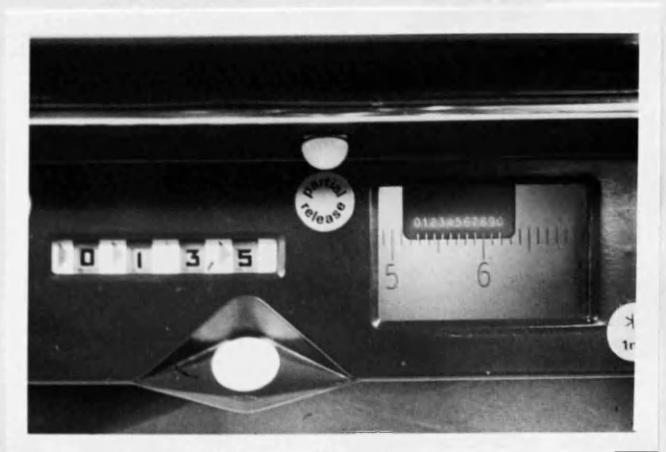
The questions covered items like reading of scales (Q. 1, 2, 4), treatment of errors (Q. 5), interpretation of data (Q. 6, 12, 13), usage of equipment (Q. 10, 11), safety (3), titrations and indicators (Q. 7, 8) and graph drawing (Q. 9). These questions covered basic non psychomotor laboratory skills and also points arising from particular experiments. For the results see Table 4.1 (p. 83).

The mean mark for the examination was 7 and the Standard Deviation was 1.68. Only one student got all thirteen questions correct. The examination was set at the right level as it was not too hard for the students, and achieved a spread of results from top to bottom.

### Item Analysis

Q.1 What is the weight of the substance being measured on the Stanton CL 41 balance?

- (A) 13.5527 gm
- (B) 13.5528 gm
- (C) 13.5530 gm
- (D) Can't tell



F.V. 0.85  $T_1-T_3$  0.21 Key B Level : Comprehension  
High discrimination considering level of facility value.

Q.2 /

Table 4.1

Results of First Year Chemistry Practical Examination

Q. No.	%	Correct Answer	$T_1-T_3$	Frequency of Answers						
				A	B	C	D	E	NA	BM
1	85.17		0.21	28	293*	18	4	0	1	0
2	11.05		0.17	68	206	31	38*	0	0	1
3	65.70		0.25	32	7	226*	75	0	3	1
4	68.90		0.26	237*	85	13	9	0	0	0
5	51.16		0.36	0	113	176*	55	0	0	0
6	63.37		0.49	9	42	71	218*	1	3	0
7	15.70		0.03	31	256	54*	1	0	2	0
8	49.42		0.23	41	40	90	170*	0	3	0
9	43.02		0.41	66	148*	121	8	0	1	0
10	74.42		0.42	85	2	256*	0	0	1	0
11	67.44		0.37	76	30	232*	5	0	1	0
12	85.47		0.18	14	294*	35	0	0	0	1
13	26.45		0.33	62	117	64	91*	1	9	0

Number of candidates - 344

\* Denotes correct answer

E - Error

NA - No answer

BM - Blunder markings

$T_1$  - Students in top third of class ranked by performance on test as a whole

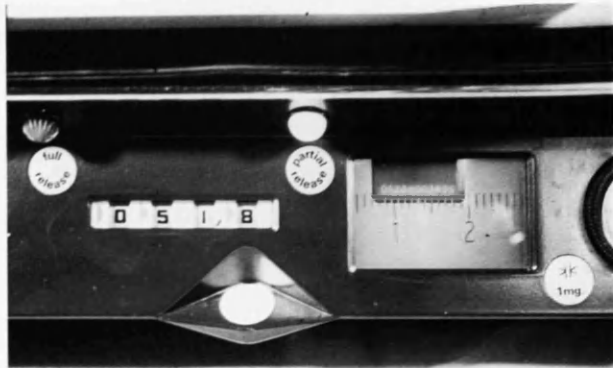
$T_3$  - Students in bottom third of class ranked by performance on test as a whole

$T_1-T_3$  - Discrimination index

Facility value F.V.  $\equiv$  percentage correct

Q.2 What is the weight of the substance being measured on the Stanton CL 41 balance?

- (A) 51.8088 gm
- (B) 51.8087 gm
- (C) 51.8179 gm
- (D) Can't tell



F.V. 0.11  $T_1-T_3$  0.17 Key D Level : Comprehension  
Balance on partial release thus the correct answer is 'can't tell'. Discrimination quite high as only 3% of students in the bottom third of the class got the correct answer. This was a trick question.

Q.3 In which of the photographs A, B, C, D is the student creating the greatest potential safety hazard?

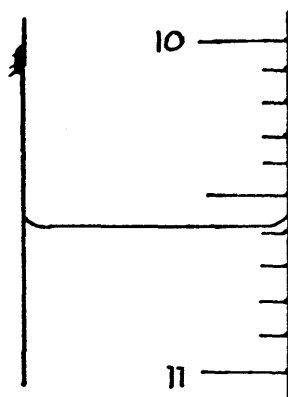


F.V. 0.66  $T_1-T_3$  0.25 Key C Level : Comprehension  
The safety hazard was student filling a burette with concentrated alkali without using safety glasses. Two-thirds of the class got the right answer. Most students /

students distracted by answer D. The other distractors contained errors in technique but none which constituted a real safety hazard.

Q.4 What is the reading on the burette?

- (A)  $10.59 \text{ cm}^3$
- (B)  $10.60 \text{ cm}^3$
- (C)  $11.41 \text{ cm}^3$
- (D)  $11.40 \text{ cm}^3$



F.V. 0.69  $T_1-T_3$  0.26 Key A Level : Comprehension

Facility value may have been low due to the quality of the diagram. The meniscus was slightly askew but was clearly above the  $10.60 \text{ cm}^3$  mark at one end. Twenty-two students were still going for distractors C and D which indicated that they were still reading the burette from bottom to top. This was after a techniques course which used burettes frequently. These answers should be treated as serious misapprehensions as students were told that this examination would form part of their practical assessment.

Q.5 A student has carried out four titrations. The results of these were:-  $16.32, 16.81, 16.87, 20.42 \text{ cm}^3$ .

What is the average value that you would use for the end-point in subsequent calculations?

- (A)  $16.58 \text{ cm}^3$
- (B)  $16.66 \text{ cm}^3$
- (C)  $16.84 \text{ cm}^3$
- (D)  $17.60 \text{ cm}^3$

F.V. 0.51  $T_1-T_3$  0.36 Key C Level : Comprehension

The method of calculating the average end-point for a titration was emphasised throughout the course.

Despite this only half the class got the correct answer. The commonest distractor was B (the average of the three /



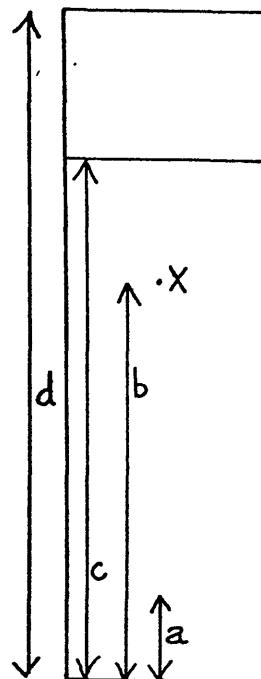
three values around  $16 \text{ cm}^3$  mark). They missed the point that only readings within  $\pm 0.1 \text{ cm}^3$  could be used. Fifty-five students took the average of all four readings thus missing the point of the question completely.

Q.6 What is the Rf value of the spot X

where  $a = 1.0 \text{ cm}$   
 $b = 5.0 \text{ cm}$   
 $c = 6.5 \text{ cm}$   
 $d = 8.5 \text{ cm}$  ?

The Rf is:-

- (A) 0.53
- (B) 0.58
- (C) 0.61
- (D) 0.72



F.V. 0.63  $T_1-T_3$  0.49 Key D Level : Comprehension

This calculation should have been straightforward with the information given. However, the discrimination index was extremely high indicating that poor students were not doing well on this question. The reason could be that the top students were either better at recalling the formula and/or had worked harder at the course, therefore having a greater working knowledge.

Q.7 In which of the following techniques would a mistake near the end-point least affect the accuracy with which you could determine the end-point?

- (A) Carrying out the reaction using an indicator.
- (B) Carrying out the reaction using a pH meter.
- (C) Carrying out the reaction using a conductivity bridge.

F.V. /

F.V. 0.15  $T_1-T_3$  0.03 Key C Level : Recall

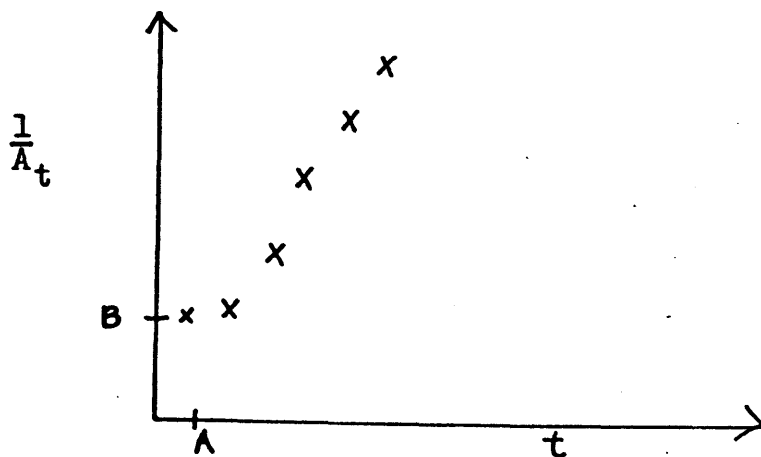
This question was recall of an assignment from an experiment on acid/base titrations by electrical methods. Thus the question should have been straightforward. Most students chose distractor B with few students going for distractor A. From the assignment most students should have realised that an error near the end-point using a conductivity bridge was less serious.

- Q.8 Which indicator would you use to follow the reaction between ethanoic acid,  $\text{CH}_3\text{COOH}$  and  $\text{NaOH}$  ?
- (A) Methyl red pH range 4 - 6.
  - (B) Bromothymol blue pH range 6 - 7.6
  - (C) Phenol red pH range 6.8 - 8.4.
  - (D) Phenolphthalein pH range 8.3 - 10.0.

F.V. 0.49  $T_1-T_3$  0.23 Key D Level : Application

Testing students about the use of indicators. This question may not have been easy for the students as many will not have covered the theory since third year at school and many students will have forgotten about it.

- Q.9 Which of the options listed below would give the 'best-fitting' line to the points on the graph?



- (A) Straight line cutting X-axis at point A.
- (B) Straight line through origin.
- (C) Curve cutting Y-axis at point B.
- (D) Curve passing through origin.

( $A_t$  = concentration of reactant A at time t.)

F.V. /

F.V. 0.43  $T_1$ - $T_3$  0.41 Key B Level : Application

Students should have known the answer from the theory which had just been covered in lectures. Option C distracted most students which could have been due in part to the quality of the drawing on the test paper. High discrimination could indicate that top students realised from the theory what the shape should be.

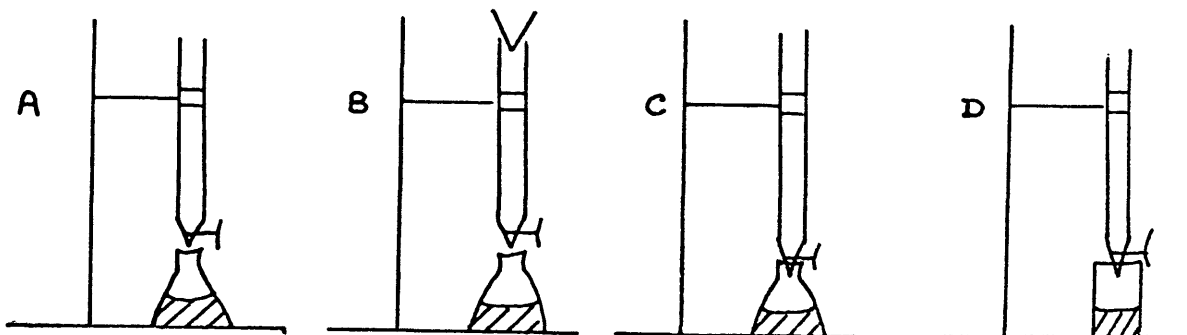
Q.10 You are required to add accurately  $50 \text{ cm}^3$  of hydrochloric acid to a reaction vessel. Which of the following pieces of equipment would give the most accurate measurement of the volume?

- (A)  $50 \text{ cm}^3$  burette
- (B)  $50 \text{ cm}^3$  measuring cylinder
- (C)  $50 \text{ cm}^3$  pipette
- (D)  $50 \text{ cm}^3$  beaker

F.V. 0.74  $T_1$ - $T_3$  0.42 Key C Level : Recall

Simple question about choice of apparatus. Almost all of the students in  $T_1$  got the answer correct. However, only slightly over half of the students in the bottom third ( $T_3$ ) got the correct answer.

Q.11 From the four diagrams below select the one that is correctly set up for carrying out a titration.

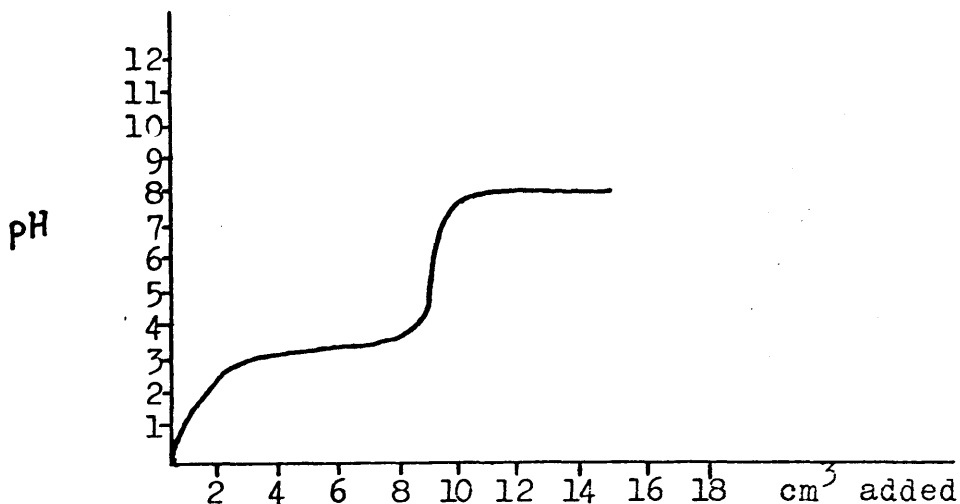


F.V. 0.67  $T_1$ - $T_3$  0.37 Key C Level : Recall

Commonest distractor was A. This occurred because it was found later that a demonstrator had taught this as the correct technique. The burette in this diagram is above the neck of the conical flask to prevent chipping of the jet (see discussion section).

Q. 12 /

Q.12



What is the pH value at the end-point?

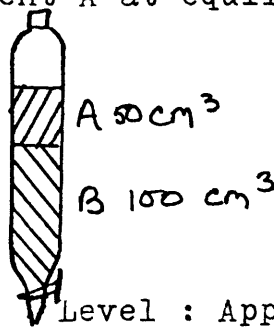
- (A) 3.15
- (B) 5.20
- (C) 7.75

F.V. 0.85  $T_1-T_3$  0.18 Key B Level : Comprehension

The theory behind this question had been covered either in the laboratories or at school. Most students got the correct answer. The discrimination index again showed that the top students were doing much better.

Q.13 0.1 gm of substance X is dissolved in  $100 \text{ cm}^3$  of solvent B. Solvent B is then shaken with  $50 \text{ cm}^3$  of solvent A. Given that the partition coefficient of X in the system A:B is 4, how many grams of X will be in solvent A at equilibrium?

- (A) 0.013 gm
- (B) 0.025 gm
- (C) 0.050 gm
- (D) 0.067 gm



F.V. 0.26  $T_1-T_3$  0.33 Key D Level : Application

This question involved using an equation covered in the experiment on partition coefficient. The discrimination was high indicating that the brighter students had a greater grasp of the course or could remember how to use the formula. Many students chose distractor /

distractor B showing that they did not really understand all the computations involved in the question.

### Discussion

Several points arose from the analysis of the first year examination.

Only skills, procedures, that are taught by a standard approach can be examined by this method. This point is highlighted by Question 11, where demonstrators taught a technique in different ways. Thus if this method of testing students is introduced generally then teaching methods must be standardised to ensure that the examinations are fair to all students. This might have the side-effect of raising teaching standards.

The results indicate that very simple procedures like reading a burette (Q. 4) and knowing which piece of volumetric apparatus to use (Q. 11) are not mastered by all students even after a 'term-long' techniques course. There is no way that students can operate meaningfully in the laboratory without mastering such topics.

Question 6 also illustrates this point. Simple calculations like this, presented in a lecture course would be quickly mastered as possible examination questions. However, in practical courses students do not seem to pick up a working knowledge of 'practical facts' as quickly. This appears to be a feature of practical work and results from the assessment procedures which allows students to write-up using a laboratory manual. Thus the students do not have to commit the facts to memory. The student realises that these facts are not going to be tested in an abstract or in an examination situation and thus does not place too much emphasis on learning them.

A practical examination of the type described in this /


this chapter will increase the possibility of students learning in the laboratory as they realise that they could be tested on the material covered.

Another important point arising from these examinations is that staff can receive information on the course and points that are causing difficulties. This information can lead to an improvement in the course or at least re-examination of the methods employed in teaching.

However, the introduction of 'paper and pencil' tests on a regular basis may have a detrimental effect on the student's attitude towards the laboratory. This may cause him to forsake long-term goals such as ability to plan and organise an experiment and to concentrate on cognitive abilities.

Thus the introduction of these examinations should be approached with caution.

Appendix 4.1

CANDIDATE'S NUMBER	SECOND CARD												QUESTION NUMBER	EXAMINATION ANSWER CARD INSTRUCTIONS
	29	30	31	32	33	34	35	36	37	38	39	40		
C01C02C03C04C05C06C07C08C09C10C11C12C13C14C15C16C17C18C19C20C21C22C23C24C25C26C27C28C29C30C31C32C33C34C35C36C37C38C39C40C41C42C43C44C45C46C47C48C49C50C51C52C53C54C55C56C57C58C59C60C61C62C63C64C65C66C67C68C69C70C71C72C73C74C75C76C77C78C79C80C81C82C83C84C85C86C87C88C89C90C91C92C93C94C95C96C97C98C99C00	<p>1. FILL IN BOXES LIKE THIS </p> <p>2. DO NOT MAKE ANY OTHER MARKS</p> <p>3. USE A <u>SOFT BLUNT PENCIL</u></p> <p>4. ERRORS MUST BE ERASED</p> <p>5. DO NOT BCND THIS CARD</p> <p>6. WRITE DETAILS BELOW</p>													
<p>QUESTION NUMBER</p> <p>43 44 45 46 47 48 49 50 51 52 53 54 55 56</p>														
<p>NAME .....</p> <p>CANDIDATE'S NUMBER .....</p> <p>DATE .....</p>														

PDC 6811

THE UNIVERSITY OF GLASGOW





## CHAPTER 5

The Development and Assessment of Two Teaching Packages for Use in the Laboratory

## 5.1 Introduction

There are several drawbacks to be overcome in using demonstrators to teach skills in the laboratory. These occur because there is no standardisation of the approach adopted to teach techniques. Thus demonstrators may teach their own variation of a technique when demonstrating to students. In addition to this, a demonstrator's introductory talk may not be the same every day, i.e. he may not place the same emphasis on the important steps and he may forget to mention some steps altogether.

One way to overcome these problems is to introduce teaching packages. Teaching packages can come in many different forms, for example, audio programmes, audio-visual programmes, films, loop cassettes, computer programmes (computer assisted learning) etc. The type of programme developed depends on the nature of the material. For instance a teaching package to show manipulative technique is best demonstrated by a film which can show the motion. A teaching package showing the preparation of a Nujol Mull for use in a spectrophotometer would be better covered by an audio-visual programme which would show the sequence of operations and illustrate important steps.

Teaching packages can be used in two ways:-

- 1) as initial instruction where the teaching of a theory, procedure or technique is done in the first instance by a teaching package; or
- 2) as remedial instruction where, if a student has failed to master a technique, he can be directed to the remedial programme.

## 5.2 The need for Teaching Packages on the Use of a Pipette and Burette

Two of the most frequently used pieces of laboratory apparatus are the burette and the pipette. It is extremely important that a student achieves proficiency in their use as soon as possible.

However, it can no longer be assumed that these techniques are taught at school to the standard required in university. Results of a questionnaire given to all students in October 1975 (see Table 5.1) show that an appreciable number of students have had extremely little practice with the burette and the pipette.

	A	B	C	D	E
Burette	40.8	40.5	9.3	2.1	3.3
Pipette	44.6	37.4	7.2	3.3	3.8

Sample size - 419

Key:- Used apparatus

A) very often.

B) several times.

C) once.

D) never and never seen this demonstrated.

E) never and have seen this demonstrated.

Table 5.1

Because of the importance of these techniques it is essential that these skills are correctly taught from the beginning so that the student does not develop his own idiosyncracies or bad habits as these can be difficult to eradicate. Therefore it was decided to develop two teaching packages to instruct the students how to use a burette and a pipette.

As previously mentioned, the best method for demonstrating manipulative techniques is that of film. It was decided to produce two films to demonstrate the techniques involved in using a pipette and a burette. A soundtrack was added to the films to emphasise important steps. In addition, loop cassettes were made of the two films, which were available in the laboratory for a student /

student to replay the cassettes until he had satisfied himself and his teacher. The loop cassette projectors have a 'freeze' button which allows a student to stop the film at any step if he wanted to.

### 5.3 Design of Programmes

Scripts were produced for both films and circulated around several members of staff for criticism. It was sometimes difficult to get complete agreement as technique varied among the staff. However, a final script was prepared which was used to construct a 'story board'. The spoken commentary is in Appendix 5.1 and the films as produced on loop cassette are contained in the flap at the back of this thesis.

The films were made in 16 mm film, in colour, and with a spoken commentary. Each film lasted for just over four minutes.

To produce the loop cassettes the films had to be shortened as the maximum length of a cassette is four minutes. An additional technical point occurred as the speed of the film was twenty-four frames a second and this had to be reduced for the loop cassette to sixteen frames a second. Thus the action on the loop cassettes is slower. The editing was done by shortening the introductory sequence which in the films was used to identify the different parts on a pipette and burette.

Loop-projectors with the cassettes were placed in the first and second year laboratories and were available to students to revise their techniques. A brief printed summary of the points made were available beside the projectors (see Appendix 5.2).

### 5.4 Assessment

In designing the assessment for the films on the burette /

burette and the pipette it seemed inappropriate to issue questionnaires or 'paper and pencil' tests as these methods focus the students' attention on to what he can recall. As Schwartz<sup>89</sup> points out this may be too narrow an assessment. A more appropriate assessment would be an examination of the effect of the film on the student, that is, his performance in the laboratory while he is using the equipment. In other words if after seeing the film a student can put into practice what he saw, then the film can be judged to have been effective. Thus an analysis of a student's performance will yield evidence as to the effectiveness of the film as an instructional package. However, evidence that a point made in the film is not getting across can be interpreted in two ways. Firstly that the film is not effective, or secondly that when that point was being made there was a break in the student's attention span.<sup>90</sup>

The question then arises of how to assess the performance of students. A suitable approach is that of direct observation. This type of assessment can come in many forms covering assessment using a detailed checklist such as that used by Horton<sup>15</sup> to a subjective impression of a student's performance with all other methods being placed on a continuum between these extremes. However, there are two main problems with this type of assessment:-

- 1) the need to use common criteria to judge a performance. To be fair to the student all judges must place a similar stress on all points.
- 2) the lack of reproducibility of results.

To overcome these problems it was decided to record the performances of the students on video tape. This provided a permanent record and allowed for repeated checking of results until a consistent analysis was produced which could be vetted by independent judges.

The analysis /

The analysis applied used a checklist which broke down the performance into criteria to which one could obtain a Yes/No answer (see Appendix 5.3).

One criticism of such a detailed list is that a student's performance is more than just the sum of the individual actions which go to make it up. This 'gestalt' approach is not applicable to this analysis as we are looking to see if points made in the film are being transmitted to the students. Thus the checklist approach is valid.

All the filming of students had to be completed within a week as this was the period for which the camera was available. We were supplied with a remote control camera, complete with a zoom lens, video-tape equipment and a monitor set.

The camera was set up, to point down the centre of a bench. This allowed students on both sides of the bench to be viewed simultaneously (see Figure 5.1). The zoom lens facility made it difficult for students to realise when they were being filmed. Students were filmed in three groups of four as indicated in the diagram for periods of twenty minutes each.

The picture obtained was of sufficient clarity to allow examination of each of the four students in turn.

It is difficult to make the appearance of a camera unobtrusive and thus students were told beforehand that they would be filmed sometime during the experiment. However, students quickly forgot the camera and their actions appeared to be natural.

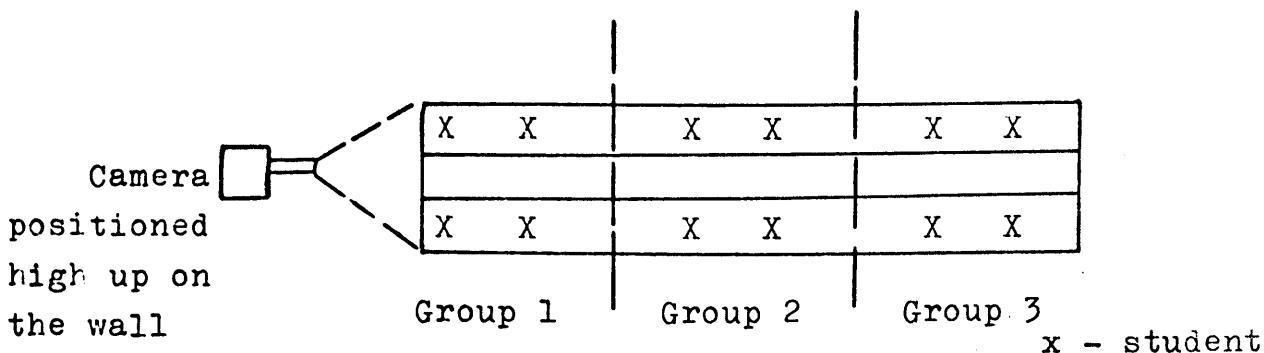


Figure 5.1

In general, students in the first group did not complete all the actions described in the films within the first twenty minutes. The most productive group as far as the recording went, was the second group as by the time the third group were reached, some students had finished the practical.

All the students' performances with the burette and pipette were analysed and the results were checked by both the author and another observer independently.

The observations were found to be consistent.

### Sample Sizes

The students were filmed for five laboratory days. Each day the target population was twelve students. Three days were designated experimental days giving a total of thirty-six students who along with their colleagues on that day, were shown the two instructional films before beginning the experiment. The other two days on which the students were filmed formed the control group and the target population was twenty-four students. Students in the control group were given an introductory talk by a demonstrator covering the points made in the film.

Sample sizes varied from item to item on the checklist but always were lower than the target populations.

### Summary of Experimental Strategy

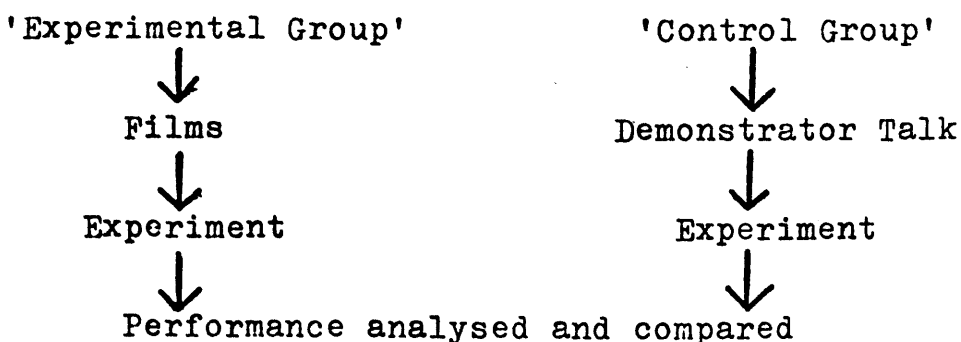


Figure 5.2

## Analysis of performance

Certain procedures were followed in the analysis of the students' performances.

- a) Where the action was not detectable, i.e. hidden by hands or not distinguishable, e.g. removal of the final drop from a pipette, then no score was given. This accounts for many low sample sizes.
- b) No action was assumed to have been completed. Thus a procedure like cleaning the pipette was frequently not caught on film as most students had cleaned the pipette or burette only at the beginning of the laboratory period. Where the actions were not recorded on film no score was given.
- c) In a twenty minute period only the students' first recorded attempt was analysed. If the equipment broke down analysis was continued from the point where the student first had trouble.
- d) Not all students completed a titration within the twenty minutes. Some students in the final group of four had departed by the time the camera was fixed on them.
- e) If a demonstrator intervened to correct a student then the student was marked wrong for that step.

Two graphs were constructed showing a point by point analysis for each procedure. Comparisons were made between the control and experimental groups. Raw scores were converted to percentages to aid comparison. Although this conversion is not valid for low sample sizes it did allow a 'quick' comparison of results to be made.

The comparison is between the frequency of two groups of students completing an action correctly.

For results see Tables 5.2 to 5.5, and Graphs 5.1 to 5.2.

Experimental Group  
Pipette - Checklist

Item No.	Yes	No	Item No.	Yes	No
1			13	22	1
2	7		14	23	-
3	7		15	23	-
4	2	2	16	9	5
5	5	1	17	21	-
6	7	-	18	19	-
7	9	-	19	15	3
8	9	-	20	-	-
9	6	-	21	13	5
10	8	1	22	18	-
11	-	-	23	12	-
12	18	1	24	-	-
			25	3	13

Table 5.2

Control Group  
Pipette - Checklist

Item No.	Yes	No	Item No.	Yes	No
1	-	-	13	21	-
2	1	-	14	21	-
3	1	-	15	21	-
4	1	-	16	9	3
5	2	-	17	21	-
6	1	1	18	16	-
7	1	1	19	14	-
8	1	1	20	-	-
9	2	-	21	16	2
10	2	-	22	17	-
11	-	1	23	5	-
12	17	-	24	-	-
			25	6	9

Table 5.3



Control Group  
Burette - Checklist

---

Item No.	Yes	No	Item No.	Yes	No
1	4	-	16	15	2
2	-	-	17	-	-
3	11	1	18	17	-
4	-	-	19	12	5
5	-	7	20	7	9
6	2	-	21	11	5
7	-	-	22	16	-
8	13	-	23	-	-
9	14	-	24	12	-
10	8	4	25	-	-
11	-	-	26	8	4
12	-	-	27	8	1
13	16	-	28	8	-
14	11	4	29	10	1
15	13	2	30	-	-

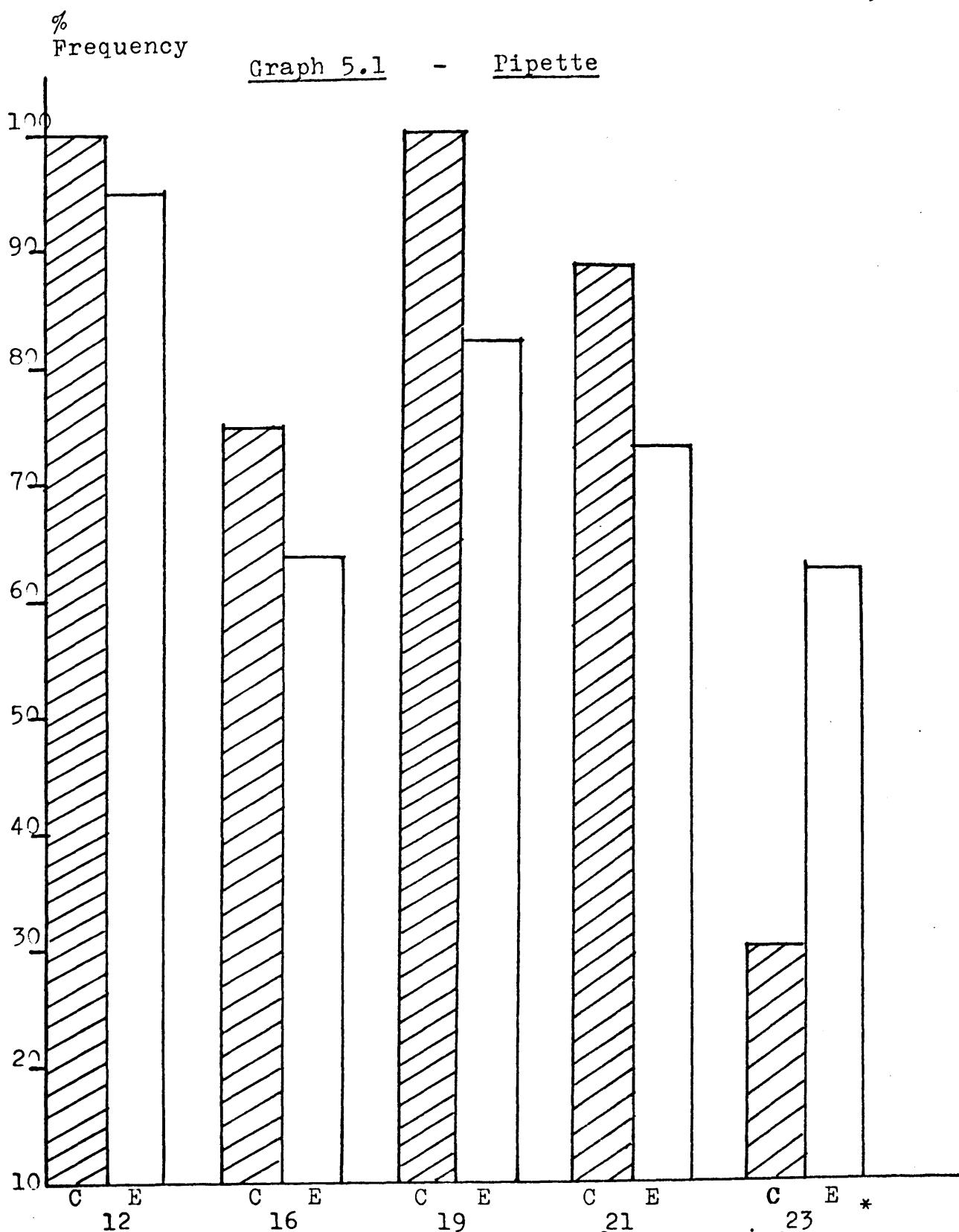
Table 5.4

Experimental Group  
Burette - Checklist

---

Item No.	Yes	No	Item No.	Yes	No
1	5	-	16	17	2
2	1	-	17	-	-
3	14	-	18	22	-
4	4	-	19	18	4
5	4	3	20	11	9
6	5	-	21	19	1
7	-	-	22	21	-
8	21	-	23	-	-
9	17	2	24	15	-
10	19	3	25	-	-
11	-	-	26	9	2
12	-	-	27	8	2
13	21	-	28	9	1
14	18	2	29	12	1
15	20	-	30	-	-

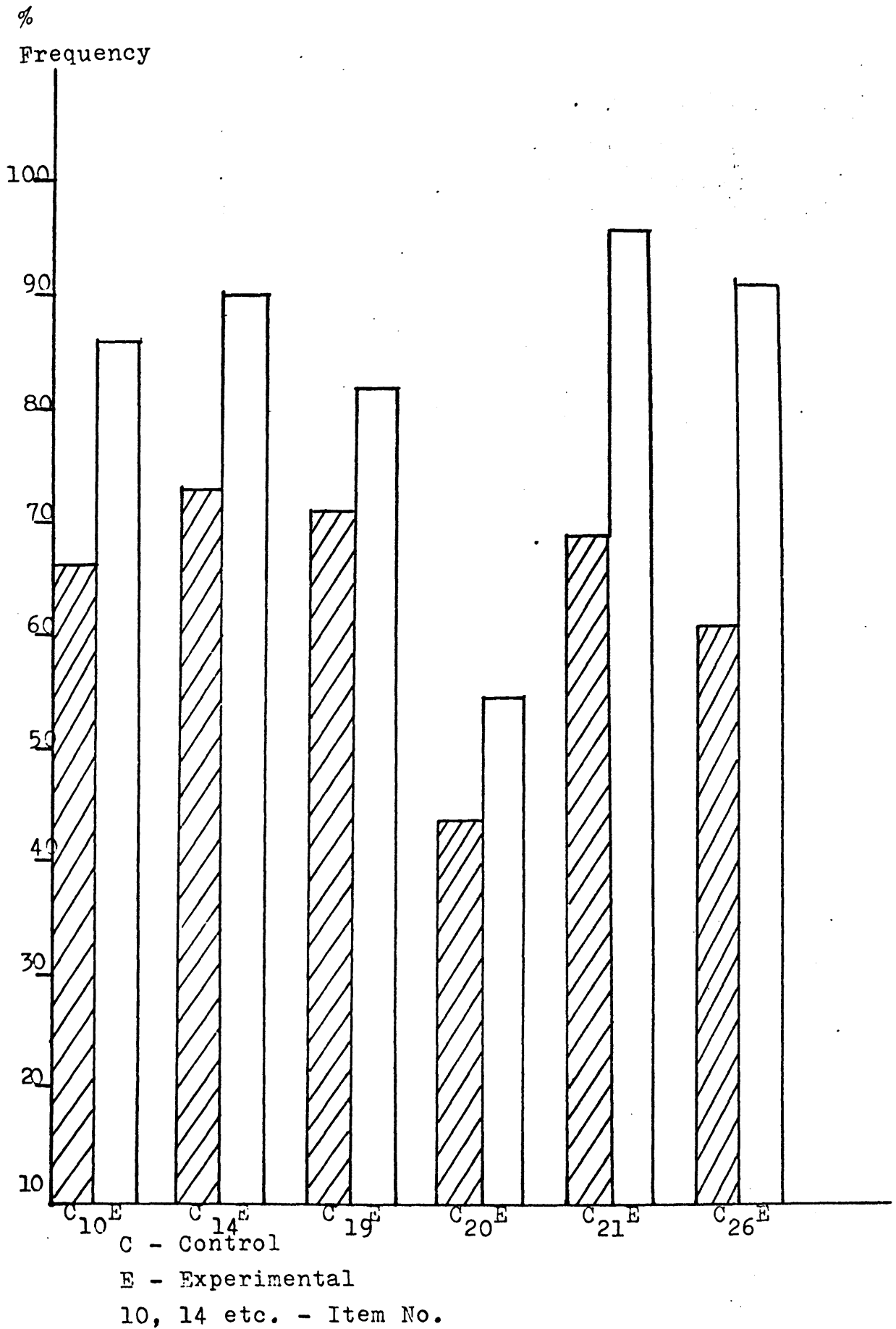
Table 5.5



N.B. Numbers usually so small that there is no statistical significance.

C - Control ; E - Experimental ; 12, 16 etc. - Item No.

\* An alternative approach to discharging the contents of a pipette was frequently adopted by students. This was simply to remove the pumpette. This method was more obvious even to students who had seen the film. However, more of experimental group have used this method.

Graph 5.2 - Burette

In both analyses the experimental group did at least as well as the control group. However, due to the sample sizes no statistical significance can be claimed.

However, the films had certain advantages over the demonstrator approach, in that the film consistently showed the same technique without forgetting to mention any points which can happen with a demonstrator talk. In addition to this, the film always laid the same stress on each point whereas a demonstrator may unconsciously vary the stress he places on points from talk to talk.

The adoption of a standard approach, not without some debate, lead to an increase in teaching standards as demonstrators all had to use the same approach. The students benefitted from this in not being told different methods by different demonstrators.

Thus the advantages in producing the two films on the burette and the pipette were considerable and justified the effort.

The analysis of the students took a considerable time (one week) as the students' technique was examined and re-examined.

Despite the time involved in the analysis, it was felt that a fairer assessment of the impact of the films resulted as their potency as teaching packages was scrutinized at the most crucial level; their effect on the students' performance. This method of assessment is the only method capable of determining this effect as both the other approaches (questionnaire and paper and pencil) are indirect methods of assessment and the assumption has to be made that there is a strong correlation between their attitude or cognitive attainment and their manipulative skill in the laboratory. This assumption may not always be valid.

Assessment of the nature described in this chapter can provide for a more meaningful assessment of instructional packages involving practical skills.

Appendix 5.1

BURETTE		
Speech No.	Meter Reading	Script
1	10	A burette is used to deliver any specified volume of liquid up to its stated capacity. In the burette the volume of liquid delivered is the difference between the initial and final readings.
	21	The burette should be clamped vertically and at a convenient height.
	22	A burette has a delivery tip at one end and a stopcock for controlling the flow of liquid.
	32	The stopcock is lubricated and therefore if the seal is broken and starts to leak the stopcock will require to be cleaned, dried and relubricated.
	34	
	35	The stopcock should be operated with one hand, in this position, the natural tendency to pull out the stopcock is reduced.
	40	
2	60	If any bubbles appear remove them by gently tapping the tip.
	64	
3	73	Your eye should be level with the bottom of the lower boundary of the meniscus. All readings should be recorded to two decimal places - the second decimal place is estimated.
	80	
4	83	When carrying out a titration a white tile is sometimes placed under the conical flask to allow you to detect small colour changes.
	89	
	92	Sit down in a relaxed position, then grip the conical flask by the neck - this enables you to swirl the contents with the minimum of effort.
	98	
	104 /	

Speech No.	Meter Reading	Script (cont'd)
	104	When the colour change takes two seconds to disappear slow down the rate of addition.
	107	
5	113	Before taking the final reading remember to allow time for drainage.
	116	
	119	Take the reading to two decimal places as before.
	122	

PIPETTE

Speech No.	Meter Reading	Script
1	12	A pipette is a precision instrument which delivers fixed volumes of liquid. After delivery there is always a drop left in the pipette which is taken into account and should not be removed.
	17	
2	28	The end of the pipette which is tapered is called the jet. Unlike the burette the pipette only has a single graduation. As the warmth of your hand can expand glass you should only hold the pipette above the graduation mark.
	39	
3	40	You must use a pumpette because of the dangers of pipetting by mouth.
	43	
	46	Press in the fingerplate - this opens the jaws for the pipette to be slipped in.
	50	
	50	Leave the fine adjustment screw about a $\frac{1}{4}$ " out to allow for later adjustment.
	54	
	54	Depress the bulb to expel air - cover the hole on the coarse control valve and dip the pipette into the liquid.
	58.5	
	59 /	

Speech No.	Meter Reading	Script (cont'd)
	59 62	The pipette must be cleaned and rinsed before using.
4	71 75	Always pipette from a beaker - never from a stockbottle as you could contaminate the contents.
5	92 96 99 100 101 105 110 113 117 121	When the liquid level is slightly above the graduation mark stop pipetting. To get the exact level rotate the fine adjustment screw. Your eyes must be level with the graduation mark. Dry the outside carefully with a tissue. Press the coarse control valve taking care not to obstruct the air vent. Allow the pipette to drain before removing the final drop by touching it against the surface of the liquid.

## Appendix 5.2

### Burette Checklist

The following is a brief summary of the points covered in the loop cassette:-

- Volume - a burette can deliver any specified volume up to its stated capacity to an accuracy of  $\pm 0.05 \text{ cm}^3$ .
- Stopcock - used to control flow of liquid and should be operated with one hand as shown - using the method shown you are less likely to pull out the stopcock and ruin your experiment.
- Cleaning - rinse out with tap water and pyroneg - and then rinse with distilled water and some of the liquid which is to be used.
- Titration - use a white tile - grasp conical flask by the neck - as you approach end point (colour takes a couple of seconds to fade) slow down rate of addition - titrations should agree to within  $\pm 0.1 \text{ cm}^3$ .
- Reading - take all readings to two decimal places and remember to allow time for drainage before taking the final readings (for a  $50 \text{ cm}^3$  burette allow one minute). Your eyes must be level with the meniscus.
- Stockbottle - do not return excess contents of the burette to the stockbottle but dispose of as directed.

(Please note that the person in the film is left-handed.)

### Pipette Checklist

The following is a brief summary of the points covered in the loop cassette:-

- Handling - hold above graduation mark to avoid the warmth of your hands expanding the glass.
- Pumpette /



Pumpette - insert - press in fingerplate - and push pipette firmly in.

Clean - press in rubber bulb - tip under water - draw liquid up - rotate - avoid splashing into rubber bulb - discharge by removing pumpette or press in coarse control valve (not covering air vent) - dry outside of pipette.

Stockbottle - pour sufficient contents into a beaker - pipette from beaker (do not return content of beaker to stockbottle) - press in bulb etc. - adjust fine adjustment screw till =  $\frac{1}{4}$ " out. When liquid level slightly above graduation, stop - rotate fine adjustment screw till to bottom of meniscus on graduation mark.

Reading - eyes must be level with the graduation mark to avoid parallax.

Dry outside carefully with a tissue.

Discharge - as before.

Allow time for drainage - depends on size - for 50 cm<sup>3</sup> allow one minute. Remove final drop by touching tip of pipette against the surface of the liquid.

(Please note that the person in the film is left-handed.)

Appendix 5.3Checklists for pipette and burette

The order of points on the two lists is flexible as it is possible to perform procedures in different sequences.

Problems arise in checking several points. With the pipette these are:-

- a) With the fine adjustment screw, the point was not ticked unless student was seen to adjust it before pipetting; (16)
- b) Detecting if the final liquid level is at the graduation mark; (18)
- c) Checking if the student's eyes are level with the graduation mark when checking the final level; (21)
- d) Final drop removed correctly. (25) Assumed that if student made motion, i.e. dipped pipette before removing it from the beaker, then he was aware of significance of final drop without necessarily having removed it correctly.

With the burette these are:-

- a) Not always possible to check if students had examined for leaks; (2)
- b) Not always possible to tell if the burette reading aid is correctly adjusted or right way up; (14,15)
- c) Impossible to tell if rate of addition was slowed down when the colour change took two seconds, as video-equipment was black and white. (23)

DATE \_\_\_\_\_ LAB DAY \_\_\_\_\_

BENCH No. \_\_\_\_\_ CONTROL/EXPERIMENTAL \_\_\_\_\_

BRIEF DESCRIPTION OF ACTIONS \_\_\_\_\_

METER READINGS \_\_\_\_\_ TO \_\_\_\_\_

PIPETTE - CHECKLIST - 1st Year

YES NO

- |  | YES | NO |
|--|-----|----|
| 1 pipette held above graduation mark   |     |    |
| 2 Pumpette - press in fingerplate<br>insert pipette - firmly<br>(check fine adjustment screw<br>- $\frac{1}{4}$ " out) |     |    |
| 5 cleaning - water   |     |    |
| 6 depress bulb   |     |    |
| 7 press in coarse control valve  |     |    |
| 8 release bulb   |     |    |
| 9 repeat until liquid level - middle of bulge  |     |    |
| 10 rotate pipette - avoid getting liquid into<br>pumpette  |     |    |
| 11 discharge - remove pumpette or press<br>coarse central valve<br>- do not cover air vent                             |     |    |
| 12 pipetting - pour liquid from stockbottles<br>into beaker  |     |    |
| 13 pipette from beaker   |     |    |
| 14 press in fingerplate  |     |    |
| 15 insert pipette - firmly   |     |    |
| 16 (check fine adjustment screw<br>- $\frac{1}{4}$ " out)  |     |    |
| 17 depress bulb  |     |    |
| 18 repeat until liquid level - about<br>graduation mark  |     |    |
| 19 use fine adjustment screw to get<br>correct level   |     |    |
| 20 dry outside with a tissue   |     |    |
| 21 eye - should be level with<br>graduation mark   |     |    |
| 22 pipetting - insert into conical flask   |     |    |
| 23 /   |     |    |

- 23 discharge - remove pumpette or  
press in coarse control valve -  
not obscuring air vent
- 24 allow time for drainage
- 25 remove final drop

DATE \_\_\_\_\_ LAB DAY \_\_\_\_\_  
 BENCH No. \_\_\_\_\_ CONTROL/EXPERIMENTAL \_\_\_\_\_  
 BRIEF DESCRIPTION OF AC \_\_\_\_\_ LAB DAY \_\_\_\_\_  
 METER READING \_\_\_\_\_ CONTROL/EXPERIMENTAL \_\_\_\_\_

BURETTE - CHECKLIST - 1st YEAR

YES      NO

- 1 Clamp burette - vertically
- 2 Check grease - seal tip
- 3 Insert funnel at top
- 4 Pour in liquid to clean
- 5 Handle stopcock with one hand - palm of hand  
not touching stopcock
- 6 Allow liquid into delivery jet
- 7 Drain burette of cleansing liquid - allow  
time for drainage
- 8 Add titrant - to required level
- 9 Allow titrant into delivery jet
- 10 Remove funnel
- 11 Remove bubbles by gently tapping
- 12 Remove drop by touching tip against beaker
- 13 Take initial reading
- 14 Burette reading aid
- 15 Burette reading aid - adjusted properly
- 16 Eyes level with liquid
- 17 Take reading to two decimal places (check lab  
reports)
- 18 Place conical flask beneath burette
- 19 Adjust height of burette until inside conical  
flask
- 20 /

- 20 Grip stopcock
- 21 Other hand on neck of flask
- 22 Add titrant
- 23 Colour change takes two seconds slow down  
rate of addition
  
- 24 Add dropwise near end point
- 25 Allow time for drainage
- 26 Final reading - burette reading made
- 27                   - correct way up
- 28                   - adjusted properly
- 29                   - eyes level with reading
- 30 Take reading to two decimal places.

## CHAPTER 6

### Pre-Laboratory Exercises

## CHAPTER 6

Pre-Laboratory Exercises

## 6.1 Introduction

Before starting an experiment students are requested both in the manual and by staff members to read the laboratory manual and familiarise themselves with the theoretical background involved. At present it is assumed that students will be adequately prepared to start an experiment. Any evidence that we have (see for instance Section 2.3, p. 37) suggests that the opposite is, in fact, true. Poor preparation reduces the chances of a student understanding what he is doing and tends to reduce the experiment to the level of a cookbook exercise.

This problem is not new and has been recognised for years. Many instructors have developed techniques to ensure that the student reaches a minimum level of familiarity with the experiment before beginning. In America the method most commonly used is that of a 'recitation' period before an experiment begins. Here students are quizzed about the background theory and procedures to be followed. Barger,<sup>9</sup> Horton<sup>15</sup> and Brown<sup>22</sup> all describe this technique. However, this method has not caught on in Britain<sup>24</sup> where the responsibility has been placed on the student to prepare himself for an experiment.

In Holland at the Technical University of Arnhem the approach adopted to solve the problem of lack of preparation has been to issue 'preparation tasks' to students one week before they are due to start the experiment.<sup>91</sup> These tasks include topics that the student is expected to revise and a small test has to be completed and handed in for marking a day before beginning the experiment. The questions cover both theoretical and practical aspects of the experiment. However, there is /

is nothing to prevent a student copying from a friend.

Another approach to preparing students, which involves the use of computers, has been devised by Asycough.<sup>64</sup> This has the advantage of being flexible in that the computer can ask supplementary questions to check that students have grasped points. However, initial costs are high and programming the computer to allow for possible answers can be time-consuming.

At Glasgow University in first and second year laboratories it was decided to experiment with pre-laboratory exercises. These exercises were to be designed to be completed before a student started any practical work and to ensure that the students had reached a minimum level of competence in both the background theory and the practical aspects of the experiment. To be feasible these exercises had to -

- a) be easily marked so that demonstrators would not waste time assessing long written answers which might be only regurgitations of a textbook;
- b) be designed so that a demonstrator could quickly identify where a student was going wrong and be able to help him by issuing remedial material;
- c) last for not more than twenty minutes at the beginning of a laboratory period otherwise the students might panic and rush through the experimental thus ruining the effectiveness of the exercise.

One technique which seemed well-suited to our requirements was that of multiple-completion questions where students were asked to indicate whether they thought statements were true or false. The statements were presented in the form of an Answer Grid. For example see Figure 6.1.



Please complete the grid on the Answer Sheet by writing in each box whether you think the statement is TRUE or FALSE.

<p>1. The units used for a 1st order rate constant are <math>1 \text{ mol}^{-1} \text{ sec}</math></p>	<p>2. The decomposition of ozone is an example of a unimolecular process.</p>	<p>3. A bimolecular reaction step will always be second order.</p>
<p>4. The order of the following reaction is</p> $\text{H}_2\text{O}_2 + 2\text{HI} \rightarrow \text{I}_2 + 2\text{H}_2$ <p>is three.</p>	<p>5. In the expression for the rate of a reaction <math>k[\text{A}]^x[\text{B}]^y</math> the sum of the exponents of the concentration terms is called the Order of The Reaction.</p>	<p>6. A reaction involving two species e.g.  <math>\text{A} + \text{B} \rightarrow \text{products}</math>  <math>2\text{A} \rightarrow \text{products}</math>  must be second order.</p>
<p>7. The integrated rate law shows how the concentration of the reagents depends on time.</p>	<p>8. For a reaction <math>(a\text{A} + b\text{B} \rightarrow c\text{C} + d\text{D})</math> the rate is:</p> $\frac{-1}{a} \frac{d(\text{A})}{dt} = \frac{1}{b} \frac{d(\text{B})}{dt}$ $= k(\text{A})^x(\text{B})^y$ <p><math>x</math> and <math>y</math> need not equal <math>a</math> and <math>b</math>.</p>	<p>9. The order of an elementary process or reaction step is predictable.</p>

Figure 6.1

The grid is designed to examine several topics, each topic being covered by several statements which are randomly distributed on the grid. This means that it is difficult for a student to guess all the answers to a topic correctly. If a student makes a mistake, he is issued with a discussion section which points out where he went wrong and may pose further questions to test his understanding.

Thus the procedure adopted was that a student entering the laboratory was issued with a pre-laboratory exercise which he was allowed twenty minutes to complete and hand to the demonstrator for marking. The demonstrator will then mark his responses and issue him if necessary with the appropriate discussion sections.

If, after completing the discussion section(s), a student still fails to satisfy the demonstrator, he can get extra help from the demonstrator. The procedure can be summarised (see Figure 6.2).

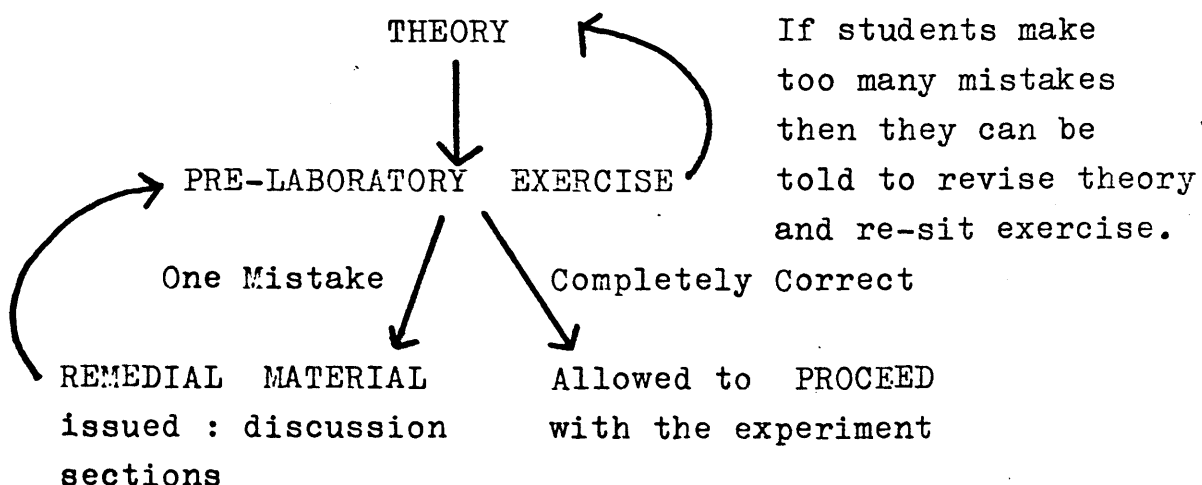


Figure 6.2

The demonstrator's role is changed when this type of exercise is being used as his introductory talk is virtually eliminated, being only included if there are changes in the procedures or if points about safety have to be stressed. His role is to issue and mark the exercise./

exercise. However, this method has the advantage of letting him identify 'poor' students quickly and therefore he can organise his work more effectively for the rest of the period to give his help where it is most needed.

The design, implementation and assessment of two exercises is described in the rest of this chapter.

## 6.2 Pre-Laboratory Exercise : First Year Inorganic Experiment

This experiment involved the determination of the formula of the cuprammonium ion by a partition coefficient technique. This is done by shaking a solution of copper ions with excess ammonia solution with the result that some ammonia molecules become attached to the copper ions. If chloroform is added two immiscible layers are formed with excess ammonia partitioning itself between the water and chloroform layers. By titration the total concentration of ammonia can be found and the formula deduced. For the full text of the experiment see Appendix 6.1.

A pre-laboratory exercise was devised for this experiment to test the students' comprehension of the theory behind the experiment.

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### Pre-lab Exercise for Experiment 8

Determination of the formula of the cuprammonium ion

Read the following information and then attempt the questions. On a separate piece of paper, write down your answers and hand these to a demonstrator for checking.

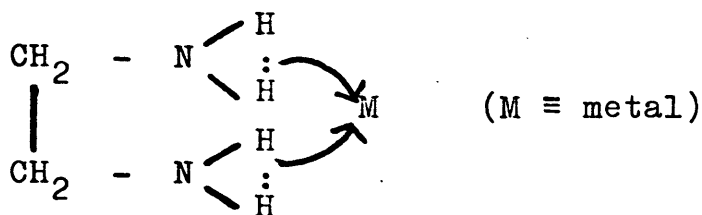
For the following exercise it is assumed that you know /

know that a ligand is an electron donor. For instance, in the complex molecule  $\text{Ti}(\text{H}_2\text{O})_6^{3+}$  the water molecules are called ligands because they donate electrons from the lone pair on the oxygen to the titanium ion forming six titanium-oxygen bonds. A ligand like water is called neutral as it carries no charge.

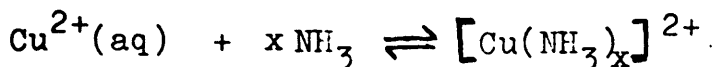
Therefore, to calculate the charge on the titanium ion in  $\text{Ti}(\text{H}_2\text{O})_6^{3+}$ , you do not consider the water molecules.

Therefore, the charge on the titanium ion is 3+. For the case of a ligand which carries a charge, e.g.  $\text{Cl}^-$  then to calculate the charge on the metal ion in  $\text{Co}(\text{Cl}^-)_6^{4-}$  you must consider the charge on the chloride ion. The charge on the Co ion is 2+ as there are 6  $\text{Cl}^-$  ligands and the overall charge is 4-.

If there is only one bond between the ligand and the central ion, the ligand is called UNIDENTATE, e.g. water in  $\text{Ti}(\text{H}_2\text{O})_6^{3+}$ . If there are two bonds between the ligand and the metal ion, then the ligand is called bidentate, e.g. 1,2 diaminoethane.



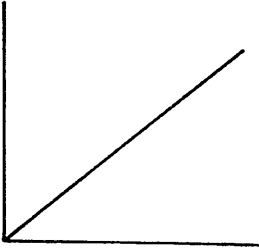
An equilibrium between the ligand and the metal ion also exists -



Thus an ammonia molecule, once it becomes attached to the metal ion, is not bound irreversibly but can be exchanged with ammonia molecules from the surrounding solution. The rate at which this happens differs according to the transition metal and the ligand and varies considerably from system to system.

Having /

Having studied the information in the laboratory manual and having read the above information, please write down, on a separate sheet of paper, the number of any statement which you believe to be TRUE .

<p>1. <math>\text{Cu}^{2+}</math> ions exist in an aqueous solution as free ions.</p>	<p>2. <math>\text{NH}_3</math> is a neutral bidentate ligand.</p>	<p>3. conc. of <math>\text{NH}_3</math> in <math>\text{H}_2\text{O}</math> = 23            conc. of <math>\text{NH}_3</math> in <math>\text{CHCl}_3</math>            The meaning of the above equation is that for every free <math>\text{NH}_3</math> molecule in the <math>\text{CHCl}_3</math> layer there are 23 free <math>\text{NH}_3</math> molecules in the aqueous layer.</p>
<p>4. The copper ion in the cuprammonium ion has been oxidised by the ammonia.</p>	<p>5. The value for a partition coefficient is dependent on the temperature.</p>	<p>6. <math>\text{Cu}^{2+}(\text{aq}) + \text{NH}_3 \rightleftharpoons \text{Cu}(\text{NH}_3)^{2+}</math>            Once an ammonia molecule becomes attached to a <math>\text{Cu}^{2+}</math> ion it cannot be displaced by another <math>\text{NH}_3</math> molecule.</p>
<p>7. There is exchange of free ammonia between the aqueous layer and the organic layer.</p>	<p>8. In <math>\text{Cu}(\text{NH}_3)_x^{2+}</math> where x is the number of ligands attached to each <math>\text{Cu}^{2+}</math> ion then x can be found by the following equation:  <math display="block">\frac{\text{combined } \text{NH}_3 \text{ in moles litre}^{-1}}{\text{conc. of } \text{Cu}^{2+} \text{ ions in moles litre}^{-1}} = X</math></p>	<p>9.             Vol. aqueous layer ml.            The value of the partition coefficient is given by the gradient of the graph.</p>

The following instructions were issued to the demonstrators.

### Pre-Lab Exercise for Experiment 8

#### INSTRUCTIONS

If the student makes more than one mistake, then, without giving him any indication of which of his answers are wrong, ask him to re-read the theory and attempt the questions again.

The answers to the question are:-

TRUE : 3, 5, 7, 8, 9

FALSE : 1, 2, 4, 6

If the student is completely correct, then give him permission to start the experiment. If the student makes only one mistake, or after further attempts is still making mistakes, then use the following guide and issue him with the appropriate remedial material.

#### ANSWER GUIDE

If the student has included any or all of the following:-

1, 2, 4, 6 (a)

If the student has omitted any or all of the following:-

3, 9 (b)

If the student has omitted any or all of the following:-

5, 7 (c)

If the student has omitted the following:- 8 (d)

Answers to questions given in discussion sections:-

(a) 1. E; 2. E; 3. C

(b) A

(c) D

(d) A

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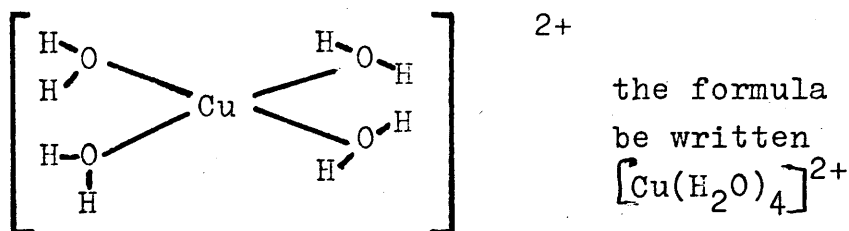
Pre-lab Exercise for Experiment 8

DISCUSSION SECTION (a)

Read /

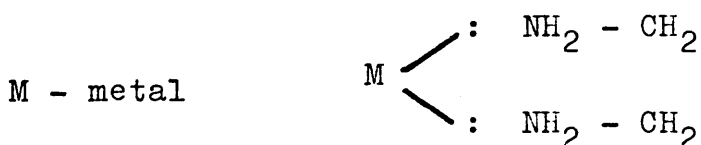
Read the following material and then answer the questions.

In water, the copper ion is hydrated. Therefore, it is surrounded by water molecules which are bonded to the metal atom by a lone pair of electrons on the oxygen.



Ammonia, like water, is a neutral ligand, i.e. it has no charge. Therefore, replacement of a water ligand by an ammonia ligand will result in no change in the overall charge of the complex cation.

Ligands are electron donors and they donate electrons to the metal centre. Ammonia reacts with the metal centre by donating its lone pair of electrons. Ammonia is a unidentate ligand because it forms only one bond between itself and the metal ion. A bidentate ligand has two bonds between the ligand and the metal centre, i.e. 1, 2, diaminoethane  $\text{NH}_2\text{CH}_2\text{CH}_2\text{NH}_2$



The bonding of the ammonia to the copper is reversible, i.e. bound ammonia can be exchanged with free ammonia from the surroundings. Thus the ammonia is being continuously exchanged around the metal centre. Likewise, in  $[\text{Cu}(\text{H}_2\text{O})_4]^{2+}$  the water molecules attached to the metal are being exchanged all the time. The rate of the exchange will, however, vary depending on the central metal atom, and for some transition metal complexes, i.e. Cr (III) complexes the rate of exchange is /

is extremely slow.

QUESTIONS : Please tick the alternative which you believe to be correct.

1. The charge on the cobalt ion in  $\text{Co}(\text{H}_2\text{O})_6^{2+}$  is -

A. -2 ; B. -1 ; C. 0 ; D. +1 ; E. +2

2. The charge on the iron ion in  $\text{Fe}(\text{CN})_6^{4-}$  is -

A. -2 ; B. -1 ; C. 0 ; D. +1 ; E. +2

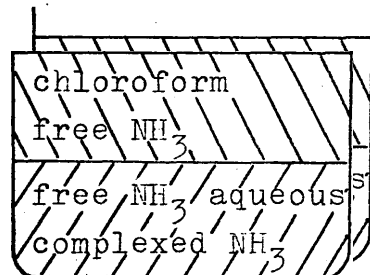
3. Labelled ammonia,  $^{15}\text{NH}_3$ , is added only in the complexed form  $\text{Cu}(^{15}\text{NH}_3)_2^{2+}$  to the aqueous layer then the  $^{15}\text{NH}_3$  will be found only -

A. in the aqueous layer.

B. distributed equally among the two layers.

C. distributed according to the partition coefficient.

D. still complexed to the copper.




---

Pre-lab Exercise for Experiment 8

DISCUSSION SECTION (b)

Read the following material and then answer the question.

Only the free ammonia, (that is, ammonia which is not bound to the copper), will distribute itself between the aqueous and the organic layers. Therefore, the value of the partition coefficient is the value for the ratio of the concentration of free  $\text{NH}_3$  in the chloroform layer.

The value for the partition coefficient can be calculated by finding the gradient of the line. In most cases this will be a straight line which should always pass through the origin.

QUESTION : Given that  $\frac{\text{conc. of } \text{NH}_3 \text{ in aqueous layer}}{\text{conc. of } \text{NH}_3 \text{ in chloroform}} = 23$

(at /



(at room temperature) and that the concentration of  $\text{NH}_3$  in the aqueous layer is  $0.5 \text{ moles litre}^{-1}$ , then the concentration of  $\text{NH}_3$  in the chloroform layer is -

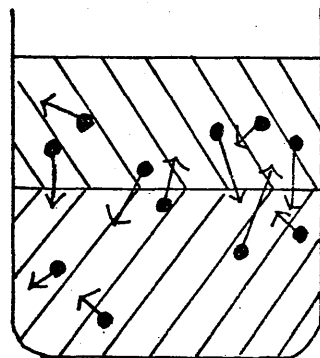
- A.  $0.022 \text{ moles litre}^{-1}$
- B.  $0.44$  " "
- C.  $12.50$  " "
- D.  $46.00$  " "

### Pre-lab Exercise for Experiment 8

#### DISCUSSION SECTION (c)

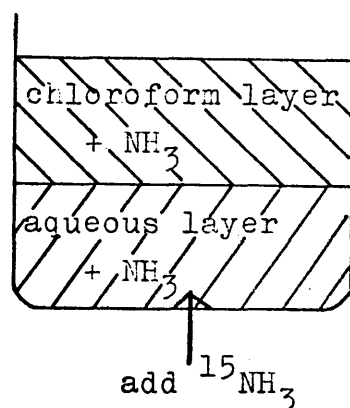
Read the following material and then answer the question.

There will be an exchange of 'free' ammonia between the aqueous and the organic layers. Eventually an equilibrium is produced where the rate of  $\text{NH}_3$  molecules leaving the organic layer is equal to the number of  $\text{NH}_3$  molecules entering the organic layer. This is an example of dynamic equilibrium.



The value of any equilibrium is affected by temperature and therefore, the value of the partition coefficient is temperature dependent. When quoting a value for the partition coefficient, you should always state the temperature.

QUESTION : Labelled ammonia  $^{15}\text{NH}_3$  is added only to the aqueous layer of the system shown. 'Free' ammonia is present in both layers. After a suitable time span the labelled ammonia will be -



- A. only in the aqueous layer.
- B. only in the organic layer.
- C. equally distributed between the two layers.
- D. distributed according to the partition coefficient.

## Pre-lab Exercise for Experiment 8

## DISCUSSION SECTION (d)

Read the following material and then answer the question.

To find the value of  $x$  in  $[\text{Cu}(\text{NH}_3)_x]^{2+}$  you have to find the ratio of the number of bound ammonia molecules to the number of copper ions.

$$\frac{\text{Conc. of combined ammonia moles litre}^{-1}}{\text{conc. of Cu}^{2+} \text{ moles litre}^{-1}} = x$$

QUESTION : In a beaker you have a solution of hexachloro-cobalt (II). Given that the ratio of cobalt to chloride ions is 6:1 and that the concentration of cobalt ions is  $0.035 \text{ moles litre}^{-1}$ , then the concentration of free cobalt ions is -

- A. 0 ; B. 0.058 ; C. 0.210 ; D. 0.420

---

### Experiment Design

We decided to examine the effects of the pre-laboratory exercise on a group of students. As this 'cuprammonium' experiment was running in two different laboratories it was decided to use one of these for the 'experimental group' and the other for the 'control group'. Since no particular laboratory day had a representative mix of students, days were chosen at random to ensure a fair sample. On each day, an average of approximately nine students attempted the experiment in each laboratory. This number varied from six to twelve. The pre-laboratory exercise was issued on five occasions with the groups of students taking on average approximately thirty-five minutes to complete the exercise. The control group conducted the experiment as normal.

When the students had finished the cuprammonium experiment /

experiment both groups were given a questionnaire to complete.

### Assessment

The assessment was in two sections (Appendix 6.2):-

- 1) Attitude survey consisting of Likert statements on a three-point scale;
- 2) Objective rating on a four-point scale.

In general a factor which has to be considered when analysing these results is that it has been observed that the experimental group took greater care in completing the questionnaire.

The test of statistical significance used in the results analysis is described in Appendix 6.3.

#### 1) Results (see Table 6.1)

Section A - Experiment 8 - Attitudes - Comparison of responses between control and experimental group

As a result of completing this experiment, I have -		% Control		% Expt.		Sig.
		(7)	3	(2)	1	
1. become more interested in chemistry.	T	(7)	3	(2)	1	*
	FT	(37)	16	(39)	17	
	U	(56)	24	(59)	26	
2. become aware of new practical techniques.	T	(26)	11	(25)	11	No sig.
	FT	(49)	21	(45)	20	
	U	(26)	11	(30)	13	
3. increased my knowledge of the theory covered by the experiment.	T	(51)	22	(59)	26	*
	FT	(28)	12	(34)	15	
	U	(16)	7	(7)	3	
4. become aware of the importance of safety procedures.	T	(14)	6	(5)	2	*
	FT	(33)	14	(27)	12	
	U	(53)	23	(68)	30	
5. become aware of the need for careful recording of results.	T	(35)	15	(43)	19	Sig. >0.005
	FT	(51)	22	(20)	9	
	U	(14)	6	(36)	16	

		% <u>Control</u>		% <u>Expt.</u>		<u>Sig.</u>
6. gained confidence in my approach to practical problems.	T	(40)	17	(19)	5	Sig. >0.005
	FT	(42)	18	(64)	28	
	U	(19)	8	(25)	11	
7. become aware that theoretically expected results are seldom obtainable in practice.	T	(40)	17	(59)	26	Sig. >0.10
	FT	(40)	17	(27)	12	
	U	(16)	7	(14)	6	
8. reinforced my existing practical skills.	T	(30)	13	(23)	10	No sig.
	FT	(57)	22	(61)	27	
	U	(19)	8	(16)	7	
9. increased my knowledge of the application of chemistry to other subjects.	T	(5)	2	(9)	4	*
	FT	(28)	12	(23)	10	
	U	(67)	29	(68)	30	
10. appreciated the need for cleanliness in handling equipment.	T	(37)	16	(34)	15	No sig.
	FT	(42)	18	(50)	22	
	U	(16)	7	(16)	7	

Table 6.1

Results in brackets are percentages

Size of samples:- Control - 43 students

Expt. - 44 students

Test of significance  $\chi^2$  test, see Appendix 6.3

\* Statements 1, 3, 4, 9 - test not applied as expected frequency in some classes is too small (< 5).

Three significant differences appeared:-

- a) More of the control group (sig. > 0.005) had become aware of the need for careful recording of results. This may be due to the time taken on the pre-laboratory exercise precluding a discussion on the reliability of answers obtained by this method as a range of answers was usually obtained. (statement 5)
- b) The control group felt they had gained confidence (statement 6 ; sig. > 0.005) in their ability.  
The /

The experimental group seemed more cautious in their replies to this statement. This was due in part to their care which they took to fill in the questionnaire.

Another factor may be that because of the pre-laboratory exercise the experimental group could not spend so long on the experimental and thus did not feel so confident about their practical performance.

- c) More of the experimental group (sig. > 0.10) felt that theoretically expected results were seldom obtainable in practice (statement 7). This is probably due to the same reasons as in (b).

The high significances (> 0.01) may indicate that the sampling was not completely random<sup>92</sup> and thus the results have to be treated with caution.

2) Practical Objectives. These were assessed from Section B of the questionnaire. For convenience this questionnaire was designed to be used with two experiments. In particular, objectives 5 and 6 were not relevant to this experiment. Coincidentally, this provided information about the validity of the method.

<u>Objective</u>		<u>%</u>		<u>%</u>	
		<u>Control</u>		<u>Expt.</u>	
1. Use a pipette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .	A	(85)	33	(78)	31
	B	(10)	4	(18)	7
	C	(5)	2	(3)	1
	D	-	-	-	-
2. Use a burette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .	A	(85)	33	(88)	35
	B	(8)	3	(8)	3
	C	(5)	2	(3)	1
	D	-	-	-	-
3. Carry out titrations confidently and carefully so that the end points in successive titrations agree to within $\pm 0.1 \text{ cm}^3$ .	A	(54)	21	(38)	15
	B	(33)	13	(48)	19
	C	(13)	5	(10)	4
	D	-	-	-	-

4. /

		%		%	
		<u>Control</u>		<u>Expt.</u>	
4. Carry out accurately the separation of immiscible liquids using a separating funnel.	A	(36)	14	(28)	11
	B	(26)	10	(38)	15
	C	(36)	14	(33)	13
	D	(3)	1	(3)	1
5. Use a spectrophotometer to obtain accurate values of I and I <sub>0</sub> .	A	(21)	8	(13)	5
	B	(18)	7	(15)	6
	C	(13)	5	(5)	2
	D	(15)	2	(18)	7
6. Use a spectrophotometer and a set of standard solutions to produce a calibration curve for the instrument.	A	(13)	5	(13)	5
	B	(10)	4	(15)	6
	C	(28)	11	(5)	2
	D	(8)	3	(18)	7

Table 6.2

Results in brackets are percentages

Size of samples : Control - 39 students

Expt. - 40 students

All four practical objectives for this experiment had been covered in previous experiments. However, the students had had more practice with burettes and pipettes than with separating funnels and this is reflected in the results.

The students were limited to four responses. These were -

- A. "Could do before this experiment and did not learn anything new."
- B. "Helped to improve my technique but not completely achieve this standard."
- C. "Because of this experiment I can now perform to this standard."
- D. "I could not do this before this experiment and I still have not learnt this technique."

However, there were no other trends in the results /

results for objectives 1 to 4.

The number of replies to objectives 5 and 6 show up the limitations of a questionnaire approach to collecting objective assessments as many students have not taken care to complete the questionnaire properly by using options B and C which were not valid. This is especially true of the control group. However, to obtain large enough samples there is often no practical alternative but to adopt a 'questionnaire approach'.

### 6.3 Pre-Laboratory Exercise : Second Year Physical Experiment

The aim of this experiment was to find the dissociation constant of a weak monobasic acid by conductivity measurement (see Appendix 6.4). This experiment involved the use of a conductivity bridge and the determination of the cell constant.

It was felt that this experiment was suitable for a pre-laboratory exercise as the underlying theory was crucial to the understanding of the experiment and had not been revised in lectures since the first year. Thus many students would not have a working knowledge of the theory.

The pre-laboratory exercise was designed in three parts:-

- 1) a multiple completion grid covering factors affecting conductivity;
- 2) /

- 2) a multiple completion grid covering the behaviour of ions in solution, e.g. conductance, specific and equivalent conductance;
- 3) a short question on the calculation of dissociation constants requiring a worked numerical answer.

---

Experiment 44 - Pre-laboratory Exercise

The following exercise should be completed before beginning any experimental work. You should put your answers on the sheets provided and hand them to a demonstrator, who will check your replies and, if necessary, give further material before permitting you to start the experiment. It is therefore in your interests to revise your First Year lecture notes and any relevant section in your textbooks. The exercise should take approximately twenty minutes to complete.

As this is the first time that an exercise of this nature has been attempted in the Second Year laboratory we would like you to complete a short questionnaire at the end of the experiment. Your replies to the questionnaire will, of course, be treated in complete confidence.

Thank you for your co-operation. Whenever you feel ready, then please start.

- 
1. Please complete the grid on the answer sheet by writing in each box whether you think the statement is TRUE or FALSE .

1. Ohm's Law applies to all electrolytic solutions.	2. The greater the distance between electrodes the lower the conductivity.	3. The specific conductance is the conductance across the opposite faces of a one centimetre cube.
4. /		



4. The greater the cross-sectional area the greater the resistance.	5. The temperature must be kept constant in conductivity expts. as the mobility of most ions increases with temperature.	6. An A.C. current is used in conductance measurements to <u>eliminate</u> electrolysis.
7. Dissolved carbon dioxide can be ignored when considering sources of error.	8. In comparing the conducting properties of different substances one must use equimolar quantities.	9. N <sub>2</sub> is bubbled through the solution to prevent impurities building up on the electrodes.

2. Please complete the grid on the answer sheet by writing in each box whether you think the statement is TRUE or FALSE .

1. The numerical value of the dissociation constant depends on the units used.	2. Halving the concentration of ions will halve the specific conductance of the solution.	3. The value of $\Delta_0$ for benzoic acid can be found by extrapolation to zero conc.
4. The $\Delta$ of benzoic acid will vary considerably with conc.	5. For a dilute KCl solution diluting to twice volume will have little effect on the .	6. At $\Delta_0$ the ions are independent of each other.
7. The value of $\Delta$ for KNO <sub>3</sub> will vary considerably at low concentrations.	8. For a solution of KCl the equivalent conductance will increase with dilution due to the reduction of inter-ionic effects.	9. For a solution of acetic acid the specific conductance will increase with dilution.
10. /		

10. In an ideal solution $\Delta$ would be constant.	11. The value $\Delta_o$ for KCl can be found by extrapolation to zero concentration.	12. The mobility of an ion is independent of charge.
--	---	--

3. Find the dissociation constant for acetic acid given that:-

the specific conductance is  $2.92 \times 10^{-4} \text{ cm}^2 \text{ equiv}^{-1} \Omega^{-1}$

the concentration is  $0.0316 \text{ equiv l}^{-1}$

the equivalent conductance at infinite dilution is 390.7

---

ANSWER SHEET

Matriculation No.....

Experiment 44 - Dissociation Constant by Conductivity Measurement

1. Put true or false where appropriate in the boxes:-

1.	2.	3.
4.	5.	6.
7.	8.	9.

2.

1.	2.	3.	4.
5.	6.	7.	8.
9.	10.	11.	12.

3. Calculation:

Answer

---

## Demonstrator's Answer Sheet

- |    |    |    |    |
|----|----|----|----|
| 1. | 1. | 2. | 3. |
|    | T  | T  | T  |
|    | 4. | 5. | 6. |
|    | F  | T  | F  |
|    | 7. | 8. | 9. |
|    | F  | T  | F  |

If the student has marked wrongly:

1, 2, 3 discussion section A)

4, 5, 6 discussion section B)

7, 8, 9 discussion section C)

- |    |    |     |     |     |
|----|----|-----|-----|-----|
| 2. | 1. | 2.  | 3.  | 4.  |
|    | T  | T   | F   | T   |
|    | 5. | 6.  | 7.  | 8.  |
|    | T  | T   | F   | T   |
|    | 9. | 10. | 11. | 12. |
|    | F  | T   | T   | F   |

If the student has marked wrongly:

3, 4, 6, 7, 8, 9, 10, 11, 12 discussion section D)

1, 2, 5 discussion section E)

$$3. \quad \alpha = \frac{A}{\Delta_0} = \frac{9.260}{390.7} = 0.0237$$

$$K = \frac{\alpha^2 C}{1 - \alpha} = 1.819 \times 10^{-5}$$

$$\alpha^2 C = 1.76 \times 10^{-5}$$

$$1 - \alpha = 0.9763$$

Students should be encouraged to spend no more than twenty minutes on this exercise.

---

Experiment 44 - DISCUSSION SECTION A)

In a metal wire resistance increases with length and decreases with cross-section

R /

$$R \propto \frac{L}{A}$$

$$\text{or } R = \rho \frac{L}{A}$$

where  $\rho$  is the specific resistance or resistivity which is the resistance between opposite faces of a unit cube of a conductor.

In a solution conductance is a more natural term to use than resistance since it is related to the number of ions present and to their rate of movement and to the charge on them. Conductance is the reciprocal of resistance and the reciprocal of specific resistance is specific conductance or conductivity denoted by the symbol  $K$  (Kappa). Therefore

$$R = \frac{1}{K} \cdot \frac{L}{A}$$

$$\text{or } K = \frac{1}{R} \cdot \frac{L}{A}$$

The specific conductance is the conductance across the opposite faces of a cm cube. The ratio  $\frac{L}{A}$  will be a constant for any particular cell and is known as the cell constant  $C$ .

$$K = \frac{\text{cell constant}}{R}$$

The cell constant is normally found by filling the cell with an electrolyte of accurately known resistance, e.g. 0.1M potassium chloride solution.

---

#### Experiment 44 - DISCUSSION SECTION B)

The passage of a current through a solution of an electrolyte may produce changes in the composition of the solution in the vicinity of the electrodes. Potentials at the electrodes may thus arise with the introduction of serious errors. To prevent the build-up of these potentials an alternating current is used to reverse this polarisation constantly and thus reduce it. A frequency of a 1000 cps is commonly used. Although /

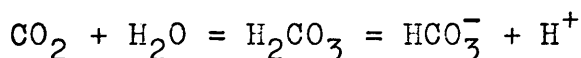
Although by this means the electrolysis is substantially reduced further measures can be introduced. Smooth platinum electrodes are sometimes coated with a thin layer of platinum black which has the effect of INCREASING the effective area for current discharge, thus reducing the local current density. In some cases, however, a thin layer of platinum black may catalyse the decomposition for example, of organic acids - which we are using.

The mobility of most ions increases by about 2% for each 1°C rise in temperature. It is, therefore, important to allow the contents of the conductivity cell to attain thermal equilibrium.

---

#### Experiment 44 - DISCUSSION SECTION C)

Carbon dioxide from the atmosphere can dissolve in water and react to form carbonic acid thus:



Carbonic acid is a weak acid and thus if present in your solution will result in you trying to measure the conductivity of two weak acids at once.

To eliminate carbonic acid, nitrogen is passed through the solution blowing out the carbon dioxide and upsetting the equilibrium. The carbonic acid will decompose thus removing this source of error.

When the CO<sub>2</sub> has been completely removed, which may take up to twenty minutes, the conductivity reading should remain constant. In comparing the conducting properties of different substances you should compare chemically composable quantities with respect to molarity and change, i.e. normalities should be the same.

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#### Experiment 44 - DISCUSSION SECTION D)

The conductance of a solution depends on the:-

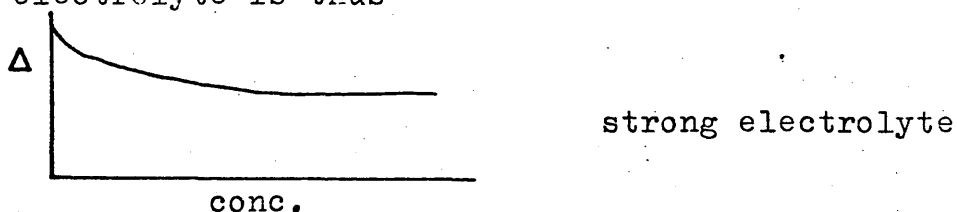
a) /

- a) number of ions present;
- b) ionic charge;
- c) ionic speeds (mobility).

Electrolytes can be divided into two categories:

- 1) Strong electrolytes, i.e. salts such as NaCl,  $\text{KNO}_3$ , NaOAc, which are completely dissociated at any concentration; and
- 2) Weak electrolytes, i.e. organic acids such as acetic acid, benzoic acid, in which the degree of dissociation increases with dilution.

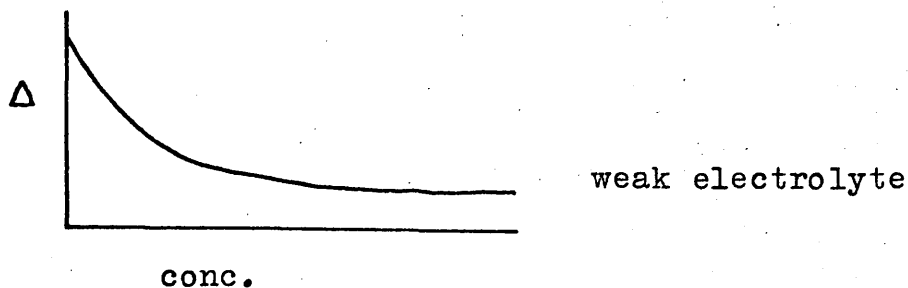
With strong electrolytes the conductivity of the solution will increase slightly at low concentrations due to the decrease in interionic attractions. These interionic attractions occur because in the vicinity of an ion there tend to be more ions of the opposite charge (ion - atmosphere). Under the influence of an electrical potential the ions will be migrating in opposite directions and will slow down the speed of the ion. At low concentrations this will not occur to such an extent. The variation of equivalent conduction for a strong electrolyte is thus -



It is possible to extrapolate the equivalent conductance back to zero concentration or infinite dilution and find the equivalent conductance at infinite dilution,  $\Delta_0$ .

For a weak electrolyte such as benzoic acid the conductivity increases considerably at lower concentrations because of an increase in the degree of dissociation. (N.B. Interionic attractions will however still apply to a slight extent.)

The variation of the equivalent conduction with concentration is thus -



It is impossible to extrapolate back to infinite dilution.

According to Kohlrausch's Law  $\Delta_o$  can be found from the sum of the individual conductivities of the ions which at infinite dilution will act independently of each other.  $\Delta_o = \lambda_+ + \lambda_-$

$$\Delta_o \text{ K Br} = \lambda_{\text{K}^+} + \lambda_{\text{Br}^-} = 74 + 78$$

$$\Delta_o \text{ K Br} = 152$$

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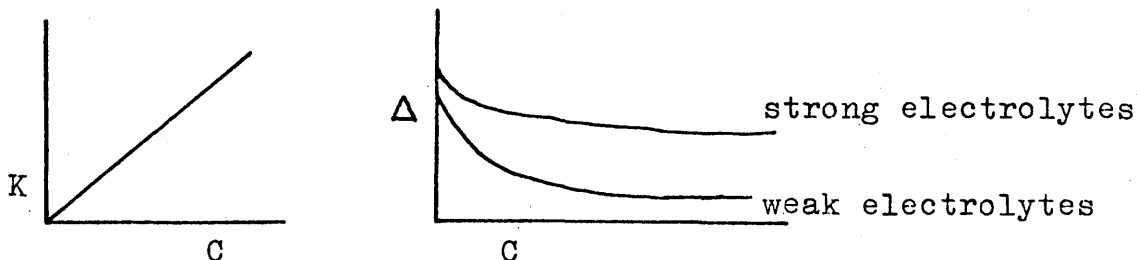
#### Experiment 44 - DISCUSSION SECTION E)

The variation of the equivalent conductance  $\Delta$  and the specific conductance  $K$  with concentration is different.

The specific conductance varies with the number of ions in the solution. Halving the concentration (diluting to twice volume) halves the specific conductance.

$$\text{The equivalent conductance } \Delta = \frac{1000K}{C}$$

$\therefore$  classically halving the concentration, e.g. for a dilute solution of KCl, has no effect since  $\Delta = \frac{1000(\frac{K}{2})}{(\frac{C}{2})}$ . Variation of  $\Delta$  is due to interionic effect (salvation sheath, ion atmosphere) in strong electrolytes and due to the degree of dissociation in weak electrolytes.



## Experimental Design

In the second year physical laboratory, students are allocated experiments depending on the availability of the apparatus and thus the students who attempted this experiment were allocated randomly. The experiment was designed to last two to three days. The students doing the experiment were split into control and experimental groups as follows, giving four weeks of the eight week course to each group.

<u>Weeks</u>		
1 and 2	Control	No pre-laboratory exercise
3, 4, 5 and 6	Experimental	Pre-laboratory exercise
7 and 8	Control	No pre-laboratory exercise

Table 6.3

Each student was issued with a questionnaire when they handed in a report.

The expected samples were around forty for each group. However, the final sample size was twenty-two for the experimental group and twenty-three for the control group. Many students simply did not hand in their questionnaires. From previous experience it was decided not to pressurise the students into completing them because this results in questionnaires not being filled in properly.

Thirty-eight students completed the pre-laboratory exercises and a record was kept of their performance.

## Assessment

The questionnaire was divided into three sections (see Appendix 6.5):-

Section A : General information about the experiment and what they thought about their performance.

Section B : A semantic differential on how they felt about the experiment.

Section C /



## Section C : Objective rating (method c) p. 59 ).

An analysis was made of the students' performance on the pre-laboratory exercise. No cognitive post-test was devised as it was felt that this would loose student co-operation.

Section A

1. How long did this experiment take you? (approx.)  
..... (afternoon)
2. Did you have any problems with the equipment which delayed you? Yes/No If Yes, please specify.

Questions 1 and 2 were general questions about the experiment to find out if they had encountered any difficulties, which may have influenced their replies. However, no students had abnormal difficulties with the experiment and all had completed it within the allotted time.

The results to Question 3 confirmed how few students normally revise the material before starting an experiment. Students were asked:-

3. Before starting any experiment do you normally revise the method, i.e. read over relevant lecture notes or textbooks?

	Control	Expt.
A. All the time	2	1
B. Only if the experiment appears difficult.	5	4
C. Occasionally.	4	3
D. Never.	2	-
E. Revise when writing up laboratory report.	4	12
F. Revise as I go through experiment.	6	2

(Figures are raw scores)

The attitude of revising before beginning an experiment /

experiment is hampered in this laboratory as students do not know the next experiment until they hand in the previous laboratory report. This is borne out by the above results.

4. When you were doing the experiment did you know precisely what you were doing?

	Control	Expt.
A. Most of the time	8	11
B. Sometimes	15	8
C. Rarely	-	3
D. Never	-	-

5. Before you began this experiment did you understand -

A. All of the theory	-	-
B. Most of the theory	5	5
C. Some of the theory	18	13
D. None of the theory	-	4

Any differences in Questions 4 and 5 could be due to small sample sizes and thus no conclusions can be drawn.

Section B The results for this section were:-

What did you feel about this experiment?

Meaningful	C $\frac{1}{2}$ : $\frac{7}{7}$ : $\frac{6}{5}$ : $\frac{4}{7}$ : $\frac{2}{-}$ : $\frac{3}{1}$ : $\frac{-}{-}$	meaningless
varied	C $\frac{-}{1}$ : $\frac{-}{2}$ : $\frac{1}{-}$ : $\frac{5}{3}$ : $\frac{7}{6}$ : $\frac{9}{8}$ : $\frac{1}{2}$	repetitive
difficult	C $\frac{1}{2}$ : $\frac{1}{3}$ : $\frac{10}{2}$ : $\frac{3}{9}$ : $\frac{4}{2}$ : $\frac{2}{4}$ : $\frac{2}{-}$	easy
worthwhile	C $\frac{-}{1}$ : $\frac{6}{6}$ : $\frac{8}{6}$ : $\frac{5}{7}$ : $\frac{2}{1}$ : $\frac{1}{1}$ : $\frac{-}{-}$	worthless
boring	C $\frac{2}{-}$ : $\frac{4}{3}$ : $\frac{1}{4}$ : $\frac{6}{9}$ : $\frac{5}{5}$ : $\frac{4}{1}$ : $\frac{-}{-}$	interesting
accurate	C $\frac{2}{1}$ : $\frac{4}{2}$ : $\frac{5}{2}$ : $\frac{5}{1}$ : $\frac{-}{8}$ : $\frac{2}{3}$ : $\frac{-}{5}$	inaccurate
pleasant	C $\frac{-}{2}$ : $\frac{2}{2}$ : $\frac{4}{1}$ : $\frac{9}{7}$ : $\frac{5}{5}$ : $\frac{1}{4}$ : $\frac{2}{1}$	unpleasant
unimportant	C $\frac{1}{-}$ : $\frac{2}{-}$ : $\frac{1}{1}$ : $\frac{4}{5}$ : $\frac{6}{7}$ : $\frac{7}{7}$ : $\frac{2}{2}$	important
useful	C $\frac{2}{3}$ : $\frac{9}{11}$ : $\frac{4}{1}$ : $\frac{4}{5}$ : $\frac{3}{1}$ : $\frac{1}{1}$ : $\frac{-}{-}$	useless

Table 6.4

Larger samples would be needed before any results could become significant. It was hoped that the semantic differential would show up trends towards the experiment being more meaningful, easy, worthwhile, interesting, useful for the experimental group. Certainly no strong trends have shown up.

The one trend which does appear is that more of the experimental group found the conductivity experiment inaccurate. After discussion with staff it appears that most students relate the accuracy of this experiment to the accuracy with which they have made up their two solutions to calculate the cell constant. It may be because of the time the experimental group have spent on the pre-laboratory exercise that they rushed the practical and experienced more trouble in making up the solutions

SECTION C Results are included in Table 6.7.

Objective

1. To be able to determine the dissociation constant  $K$  by conductivity measurement.
2. To be able to determine the cell constant  $K$  ( $\kappa$ ) to within 1% accuracy by determination with solutions of  $KCl$ .
3. To be able to operate a conductivity bridge.
4. To be able to take suitable precautions to eliminate errors arising from temperature effects.
5. To be able to take suitable precautions to eliminate errors arising from carbon dioxide effects.

A		B		C		D	
C	E	C	E	C	E	C	E
12	14	11	8	-	-	-	-
18	18	5	2	-	2	-	-
18	15	3	6	2	1	-	-
9	13	9	6	2	3	4	-
18	20	5	1	-	1	-	-

6. /

6. To be able to make, with sufficient accuracy, a solution for conductivity measurements.
7. To be able to calculate the specific conductance.
8. To be able to use K (Kappa) to determine  $\Delta$  (equivalent conductivity).
9. To be able to allow for activity factors.
10. To be able to obtain a value for the dissociation constant of an unknown weak acid.

A		B		C		D	
C	E	C	E	C	E	C	E
19	20	4	2	-	-	-	-
15	17	3	5	3	-	-	-
15	17	5	2	2	2	-	-
3	7	14	8	4	3	2	3
8	16	11	3	1	2	2	-

Table 6.5

Sample sizes:- Control - 23 students  
Expt. - 22 students.

The replies are listed under four categories, A, B, C and D which were classified as:-

- A. If you felt that you completely mastered the objective then place a tick in column A.
- B. If you felt that you did not quite master the objective and were not completely clear about either the theory or the technique place a tick in column B.
- C. If you felt that you learnt very little about the theory or technique place a tick in column C.
- D. If you felt that you learnt nothing about the theory or technique place a tick in column D.

In general, students did not use all the four categories and most students confined their replies to categories A and B.

Thus, objectives 3, 4, 9 and 10, in which all categories /

categories were used, probably tended to be viewed as difficult by the students since they seemed reluctant to use categories C and D. Apart from objective 10, there appears to be little difference between the two groups. In objective 10 more of the experimental group have claimed to master the objective, which was the main aim of the experiment. Given that the experimental group tend to be more cautious in their replies, this result may be better than it seems.

#### Analysis of Students' Performances on the Pre-Laboratory Exercise.

The replies are summarised below.

#### 1. Multiple completion question on conductivity.

1. Ohm's Law applies to all electrolytic solutions.  8	2. The greater the distance between electrodes the lower the conductivity.  15	3. The specific conductance is the conductance across the opposite faces of a one centimetre cube.  13
4. The greater the cross-sectional area the greater the resistance.  7	5. The temperature must be kept constant in conductivity expts. as the mobility of most ions increases with temperature.  -	6. An A.C. current is used in conductance measurements to <u>eliminate</u> electrolysis.  21
7. Dissolved carbon dioxide can be ignored when considering sources of error.  5	8. In comparing the conducting properties of different substances one must use equimolar quantities.  11	9. N <sub>2</sub> is bubbled through the solution to prevent impurities building up on the electrodes.  25

Table 6.6

Numbers of students who answered the statements correctly. Sample size, 38-39.

Statements 6 and 9 caused the most difficulties to students. Statement 6 was tricky but the important word was underlined, and thus students should have paused to give closer consideration to this statement. However, many students still got it wrong.

The replies to statement 9 indicated that students did not appreciate the reason why nitrogen was bubbled through the solution.

2. Multiple completion question on the behaviour of ions in solution

1. The numerical value of the dissociation constant depends on the units used.  10	2. Halving the concentration of ions will halve the specific conductance of the solution.  12	3. The value of $\Delta \circ$ for benzoic acid can be found by extrapolation to zero conc.  14
4. The $\Delta$ of benzoic acid will vary considerably with conc.  7	5. For a dilute KCl solution diluting to twice volume will have little effect on the $\Delta$ .  20	6. At $\Delta \circ$ the ions are independent of each other.  3
7. The value of $\Delta$ for $\text{KNO}_3$ will vary considerably at low concentration.  13	8. For a solution of KCl the equivalent conductance will increase with dilution due to the reduction of inter-ionic effects.  18	9. For a solution of acetic acid the specific conductance will increase with dilution.  22
10. In an ideal solution $\Delta$ would be constant.  2	11. The value $\Delta \circ$ for KCl can be found by extrapolation to zero conc.  8	12. The mobility of an ion is independent of charge.  8

Table 6.7

Number of students who answered statements incorrectly.

The three statements that have caused the students difficulty all involved the effect on conduction of diluting solutions. This appears to be an area of student misconception as this topic is also badly answered in other examinations. Statements which involved applying theory to specific examples were in general more poorly answered whereas statements of laws or theories were more often correctly answered. Thus students may have been recalling the appropriate facts without necessarily understanding them.

3. Only seven students managed to complete this question correctly. If students had a working knowledge of the theory behind this experiment then they should have been able to complete this problem which was crucial to the experiment.

#### 6.4 Discussion

In the two pre-laboratory exercises students took longer than twenty minutes to complete the exercises. In both exercises students were given no warning that they were going to be subjected to an exercise. In first year this was because days were chosen at random and in second year because students did not know their next experiment until they had completed the previous one. Thus both exercises may have unsettled the students who wished to start immediately on the experiment.

However the lack of prior warning did highlight the lack of preparation shown by students. With the pre-laboratory exercise on the determination of the dissociation constant, students took on average an hour to complete all the questions and to read the discussion sections. A majority of students were issued with all discussion sections.

Assessment of exercises of this nature is difficult as it is unrealistic to expect a single pre-laboratory exercise to produce detectable changes in students. Certainly a change in short-term cognitive gains could be detectable but this would involve a comprehensive and lengthy test. A test of this nature would produce a negative response from the student and it would be difficult to find time to fit it into the laboratory period. As can be seen from the first year exercise, the results of the objective rating section indicates that many students completed the scales without carefully considering the choices. Although this effect may be lessened with the experimental group, nevertheless, results were not considered to be reliable.

Interview techniques were considered but rejected as impractical due to the size of the samples and the time involved.

However, informal discussions were held with demonstrators, staff and students to obtain general impressions. First year students did not find the exercises enjoyable but found it useful in covering the theory. It was not possible to detect the effect of the exercise on the students' practical work.

Staff and demonstrators found that the exercises were workable and did not give them an unacceptable amount of work. The exercises were thought to be useful because they showed up the lack of preparation by students.

If for a trial period exercises of this type were introduced on a regular basis, a more realistic assessment could be attempted.

In conclusion, exercises of this type would appear to be as efficient in preparing the student as an introductory talk from a demonstrator. Pre-laboratory exercises also have certain advantages over the demonstrators/



strators in that once developed they do not make mistakes and can convey information consistently without the possibility of forgetting to mention any points. The exercises can also show up poorer students who may need extra help.

The introduction of pre-laboratory exercises places a greater emphasis on learning and increases the possibility of students preparing for the experiment.

With the use of exercises similar to the ones devised, it is possible to introduce a flexible system of examining the students' knowledge which can equal a computerised approach without the disadvantages of high initial costs and programming.

Appendix 6.1

## Experiment 8 - Determination of the Formula of the Cuprammonium Ion

Objectives

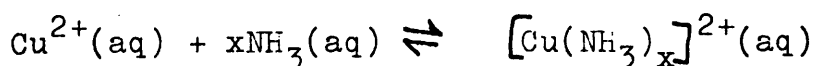
At the end of this experiment you should be able to:-

1. understand one method of determining the formula of a complex ion.
2. perform calculations on molarities, and be able to apply the answers to specific problems.
3. describe the application of partition coefficients to practical situations in chemistry.
4. list possible sources of error in the experiment.

Introduction

Last term you learned how to determine partition coefficients using graphical techniques (Experiment 2). This experiment involves using a given partition coefficient to find the value of x for the complex ion  $\text{Cu}(\text{NH}_3)_x^{2+}$  - the cuprammonium ion.

The principle is basically simple. If a solution of  $\text{Cu}^{2+}$  ions is shaken with excess ammonia solution, some of the ammonia molecules become attached to the metal ions. If chloroform is added, two immiscible layers will form. The  $\text{Cu}^{2+}$  ions with the attached ammonia molecules will remain in the aqueous ammonia layer.

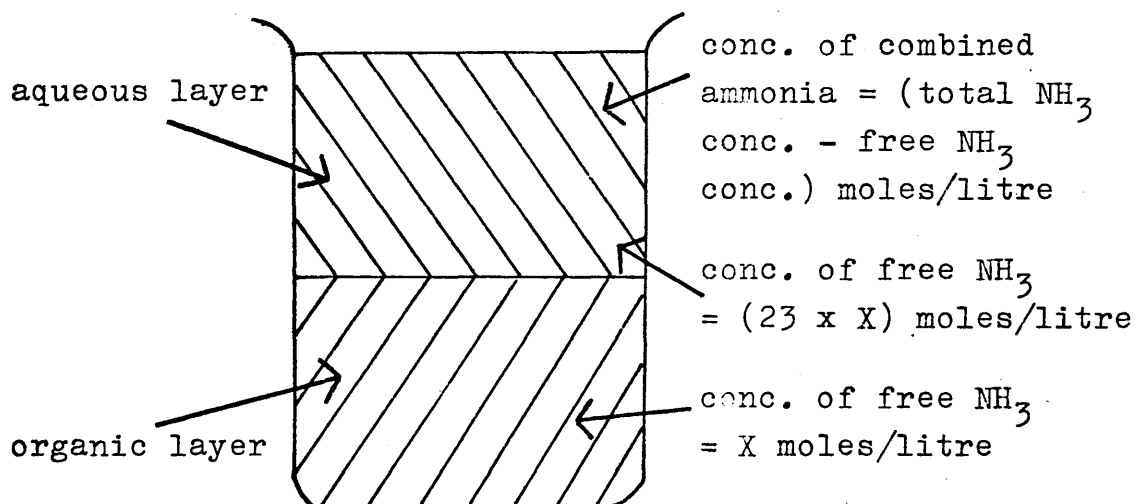


If we ensure that the total amount of ammonia is in excess of that required to form the complex ion, the excess uncombined ammonia will be found in both the chloroform layer and the aqueous layer, i.e. the excess ammonia will partition itself between the aqueous and organic /

organic layers. By titration of the aqueous layer with standard acid solution, the total concentration of ammonia in that layer (free + combined) can be found because the ammonia combined with the copper ions can be easily detached to react with the acid. By titration of the chloroform layer with standard acid solution, we can find the concentration of free ammonia in that layer, and with a knowledge of the partition coefficient, which in this case is

$$\frac{\text{concentration of NH}_3 \text{ in aqueous layer}}{\text{concentration of NH}_3 \text{ in chloroform}} = 23 \text{ (at room temperature)}$$

the amount of free ammonia in the aqueous layer can be determined. Only the free ammonia will obey the partition law and distribute itself between the two layers. Hence the amount of combined ammonia can be derived by subtraction.



Summary Diagram

The number of ammonia molecules attached to each  $\text{Cu}^{2+}$  ion is found by dividing the concentration of combined ammonia (in moles/litre) by the original  $\text{Cu}^{2+}$  ion concentration (in moles/litre). This assumes that all the copper is present as cuprammonium ion. The result obtained is a statistical average, not necessarily implying that all ions have this formula.

#### Experimental Method

Experiment (a) Place 40 ml. of approximately molar ammonia /

ammonia solution in a 100 ml. measuring cylinder, and pipette in exactly 5 ml. of 0.5 M copper sulphate solution. Stir well, and then add more ammonia solution to give a total volume of 50 ml. Next, add chloroform to make the total volume 100 ml., and pour all of the solution into a separating funnel. Stopper the funnel and shake well, for about 5 minutes remembering, periodically, to release any build up in pressure while you are shaking the funnel. Now clamp the funnel upright on a retort stand, and allow about 5 minutes for the two layers to separate.

Using a pumpette, pipette 25 ml. of the upper blue aqueous layer, and deliver it into about 100 ml. of water in a flask. Add screened methyl orange indicator (about 10 drops) and titrate with standard 0.5 M hydrochloric acid. The dark blue solution will gradually become green then violet. The end point is when the solution turns violet. Note your burette reading at the end-point.

Now, run off the entire chloroform layer into a conical flask containing about 50 ml. of water and 10 drops of methyl orange indicator. Ensure that none of the blue layer is run off also. Mix the flask contents well before titrating with 0.05 M hydrochloric acid. Note the burette reading at the end-point, i.e. when the solution just turns pink.

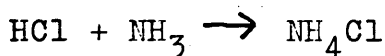
Experiment (b) Repeat the above experiment using 25 ml. of ammonia solution, 5 ml. of the copper sulphate solution, and water to give a total volume of 50 ml. Add chloroform to make the volume up to a total of 100 ml.

### Treatment of Results

Experiments (a) and (b) are the same experiment done with different concentrations of ammonia. The results from each experiment should give similar values for x in  $[\text{Cu}(\text{NH}_3)_x]^{2+}$ . Take the final value of x as the /

the average of your calculations of  $x$  from experiments (a) and (b). For each experiment, the first step in the calculation is to find the total concentration of ammonia (free + combined) in the aqueous layer. Suppose  $A$  mls. of  $0.5$  M HCl were required in the first titration.

Now, 1 mole of HCl combines with 1 mole of  $\text{NH}_3$



∴ No. of moles of HCl used in titration = no. of moles of  $\text{NH}_3$  present.

$$\frac{A}{1000} \times 0.5 \text{ moles of HCl were used}$$

∴  $\frac{A}{1000} \times 0.5$  moles of  $\text{NH}_3$  are present in 25 ml. of layer.

∴  $\frac{A}{1000} \times 0.5 \times \frac{1000}{25}$  moles of  $\text{NH}_3$  are present in 1 litre.

∴ Total concentration of  $\text{NH}_3$  in aqueous layer

$$= \frac{A}{1000} \times 0.5 \times \frac{1000}{25} \text{ moles/litre.}$$

This is the concentration of combined + free ammonia in the layer. A similar calculation can be performed for the chloroform layer, remembering that 50 ml. of the layer were used, not 25 ml. as in the case of the aqueous layer, and that the acid molarity was  $0.05$  M instead of  $0.5$  M.

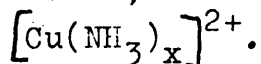
Hence, find the concentration of free ammonia in the aqueous layer using the partition coefficient, which is

$$\frac{\text{Conc. of } \text{NH}_3 \text{ in aqueous layer}}{\text{Conc. of } \text{NH}_3 \text{ in chloroform}} = 23 \text{ (at room temperature)}$$

By subtraction you can deduce the amount of combined ammonia in moles/litre.

5 ml. /

5 ml. of 0.5 M  $\text{Cu}^{2+}$  solution was diluted to 50 ml. therefore the concentration of  $\text{Cu}^{2+}$  ions is 0.05 moles/litre. Using this, and the concentration of combined ammonia, calculate the value of x in the formula



### Assignments

1. Calculate the value for x in the complex  $[\text{Cu}(\text{NH}_3)_x]^{2+}$  using both sets of results.
2. Collect results from other groups, and present them, with yours, in a table. Calculate a class value for x.
3. List the possible sources of error in the experiment, and suggest means of reducing the error.



SECTION B

Could you please study the following list and place a tick in the most appropriate column. If you wish to elaborate on any point, please write your comments at the bottom.

(Not all points are covered by this experiment.)

	A Could do before this experiment and did not learn anything new.	B Helped to improve my technique but not completely achieve this standard.	C Because of this experiment I can now perform to this standard.	D I could not do this before this experiment and I still have not learnt this technique.
1. Use a pipette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .				
2. Use a burette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .				
3. Carry out titrations confidently and carefully so that the end points in successive titrations agree to within $\pm 0.1 \text{ cm}^3$ .				
4. Carry out accurately the separation of immiscible liquids using a separating funnel.				
5. Use a spectrophotometer to obtain accurate values of I and $I_0$ .				
6. Use a spectrophotometer and a set of standard solutions to produce a calibration curve for the instrument.				

Additional Comments



Appendix 6.3Chi-squared Test<sup>92</sup>

The equation for the  $\chi^2$ -test is

$$\chi^2 = \sum_{i=1}^n \frac{(O_i - E_i)^2}{E_i}$$

The  $\chi^2$  statistic measures the closeness of the agreement between the observed frequencies and expected frequencies.

In order to apply this test to a set of given data, it may be necessary to combine some classes to make sure that each expected frequency is not too small (less than 5).

Appendix 6.4

## Experiment 44 - Dissociation Constant by Conductivity Measurement

Conductivity water must be used for all solutions, which must be made up and diluted very accurately. Electrodes must never be allowed to dry out. Rinse several times with each solution and remove the excess solution by wiping gently with Kleenex. Overnight and at the end of the experiment leave the electrodes immersed in conductivity water.

For general information see Findlay p. 240 onwards.

Read the notes on "Use of Conductance Bridge" (p. 12).

Procedure Firstly, determine the cell constant.

Make up two solutions of KCl by weighing out accurately about 0.1 g and 0.15 g, dissolving in conductivity water and making up to 100 ml. accurately. Clean and dry a flask with a side arm. Rinse a conductivity cell twice with a small quantity of the solution and then pour enough solution into the flask to immerse the cell, which is the small compartment containing the electrodes. Put the flask into the thermostat and allow to come to temperature equilibrium (about 10 minutes) as shown by a steady reading on the conductance bridge. Repeat with the second solution. Calculate the specific conductivity of the KCl solutions from the equation

$$K (\text{cm}^{-1} \text{ ohm}^{-1}) = 1.804 \times 10^{-3} g + 7.4 \times 10^{-5}$$

where  $g$  is in  $\text{g.l}^{-1}$

Calculate the cell constant for each determination from the equation,  $C = K \times R$ . If these values differ by more than 1%, make up another KCl solution and repeat the measurement.

Secondly, /

Secondly, obtain a sample of a weak monobasic acid from a demonstrator and, given its molecular weight and  $\Delta_0$ -value, find its dissociation constant.

Make up accurately about 250 ml. of approximately 0.1 M solution of the acid. Measure the resistance of this solution. Dilute two portions of the solution accurately, say 25 ml. and 20 ml. each diluted to 100 ml. and measure the resistance of these solutions. Make up accurately about 250 ml. of 0.05 M solution and repeat the procedure. Remember to rinse the cell well with conductivity water between each measurement and also to measure the resistance of the water. Nitrogen should be bubbled slowly through the solutions and water while coming to temperature equilibrium. Consult a demonstrator before touching a cylinder. Calculate an approximate dissociation constant from the equations

$$K = \frac{\text{Cell Constant}}{R}$$

$$K(\text{acid}) = K(\text{solution}) - K(\text{water})$$

$$\Delta = \frac{1000 K}{c}$$

$$\alpha = \frac{\Delta}{\Delta_0}$$

$$K = \frac{\alpha^2 c}{1-\alpha}$$

Plot  $\log K$  against  $(\alpha c)^{\frac{1}{2}}$  and extrapolate to infinite dilution to correct for activity effects.

Discussion Why do we use a.c. and not d.c. for resistance measurements? Why cannot we obtain the cell constant by measurement of its dimensions? Why do we bubble nitrogen through the weak acid solutions and water?

Appendix 6.5

## Second Year Physical Laboratory - Questionnaire

Matriculation No. . . . . Experiment No. .44. . . .

After finishing the experiment could you please complete this questionnaire. All information supplied by you will be treated in strict confidence and will not be used in any way to alter the assessment of your performance in this laboratory course.

Thank you for your co-operation.

Section A

1. How long did this experiment take you? (approx.)  
 . . . . . (afternoon)
2. Did you have any problems with the equipment which delayed you? Yes/No  
 If Yes, please specify . . . . .

Please underline the option which most closely corresponds to your opinion.

3. Before starting any experiment do you normally revise the method, i.e. read over relevant lecture notes or textbooks?
  - A. All the time
  - B. Only if the experiment appears difficult.
  - C. Occasionally.
  - D. Never.
  - E. Revise when writing up laboratory report.
  - F. Revise as I go through experiment.
4. When you were doing the experiment did you know precisely what you were doing?
  - A. Most of the time.
  - B. Sometimes.
  - C. Rarely.
  - D. Never.
5. Before you began this experiment did you understand -
  - A. All of the theory.
  - B. Most of the theory.
  - C. Some of the theory.
  - D. None of the theory.

Section B The purpose of this section is for you to make judgements on a series of scales. For instance if you believe very strongly that this experiment was repetitive then mark the scale:-

varied \_ : \_ : \_ : X : \_ : \_ : \_ repetitive

If your feelings are neutral on this issue then you should mark the scale:-

varied \_ : \_ : \_ : X : \_ : \_ : \_ repetitive

Place a cross at the position on the scale that best suits your opinion. You have seven options varying from strongly agree through to strongly disagree.

IMPORTANT:-

1. Please place your check-marks in the middle of spaces, i.e. \_ : X : \_ not \_ : \_ X \_
2. Complete each scale.
3. Never put more than one check-mark on a single scale.

WHAT DID YOU FEEL ABOUT THIS EXPERIMENT ?

meaningful	_	:	_	:	_	:	_	:	_	:	_	:	_	meaningless
varied	_	:	_	:	_	:	_	:	_	:	_	:	_	repetitive
difficult	_	:	_	:	_	:	_	:	_	:	_	:	_	easy
worthwhile	_	:	_	:	_	:	_	:	_	:	_	:	_	worthless
boring	_	:	_	:	_	:	_	:	_	:	_	:	_	interesting
accurate	_	:	_	:	_	:	_	:	_	:	_	:	_	inaccurate
pleasant	_	:	_	:	_	:	_	:	_	:	_	:	_	unpleasant
unimportant	_	:	_	:	_	:	_	:	_	:	_	:	_	important
useful	_	:	_	:	_	:	_	:	_	:	_	:	_	useless

Section C The following is a list of objectives for this experiment. By the end of this experiment:-

- A. If you felt that you completely mastered the objective then place a tick in column A.
- B. If you felt that you did not quite master the objective and were not completely clear about either the theory or the technique place a tick in column B.
- C. If you felt that you learnt very little about the theory or technique place a tick in column C.
- D. /



## CHAPTER 7

### Group Participation Methods

## CHAPTER 7

Group Participation Methods

## 7.1 Introduction

Not all the experiments in a laboratory course are of the same standard. It is possible to identify two factors which may make an experiment more difficult than normal. These are where the theory is either new or very difficult and/or experiments which involve much information gathering or processing. In these types of experiment it is possible that the student could get lost either through not understanding what he is doing or by being overwhelmed by a mass of information.

Therefore it may be more profitable in either of these circumstances for a member of staff or a demonstrator to take a more active part in the experiment. The tutor can act as team leader to lead the students through the experiment explaining the practical procedures and theoretical background. For instance, in an experiment to determine the visible spectrum of a coloured solution the tutor could begin by discussing the reasons why some molecules are coloured and how electromagnetic radiation can cause electrons to jump from one energy level to another. The explanation can also include how to use and calibrate a spectrophotometer. After this he can call on the students to take individual readings, perform calculations, draw a graph of the visible spectrum, etc. All the time, he can point out errors in technique, and explain why certain procedures must be adopted.

The approach is really a team effort with the tutor as a guide and with the students actively involved.

This approach could probably increase the amount of learning that goes on in the laboratory because the students would be given more help than in the 'normal' laboratory /



laboratory situation. It reduces the possibility of a student attempting to follow the experimental procedures like a recipe.

It is also possible that this approach could be more 'cost effective' as large groups of students could be taught by one tutor thus saving on equipment and other materials. However, the larger the group the less chance there is for students to gain practice in the techniques covered.

Almost all the laboratory objectives outlined in section 2.4 (p. 46) could be developed by a group participation approach, apart from 'hands-on' skills such as manipulation of scientific apparatus to a stated accuracy; performance of an experiment; efficient use of equipment and observational skills, which may need individual laboratory practice for the student to achieve competence. It would not be expected that a practical course would adopt this approach exclusively, so experiments not using this approach could be used to develop manipulative skills.

To examine the practicalities of this method and its usefulness, a pilot experiment was run in the first year to discern the possible advantages and disadvantages to the group participation approach.

## 7.2 Trial experiment using Staff

A pilot experiment was chosen from the list of experiments in the first year course. The experiment chosen was entitled, 'Phosphates in detergents' (see Appendix 7.1).

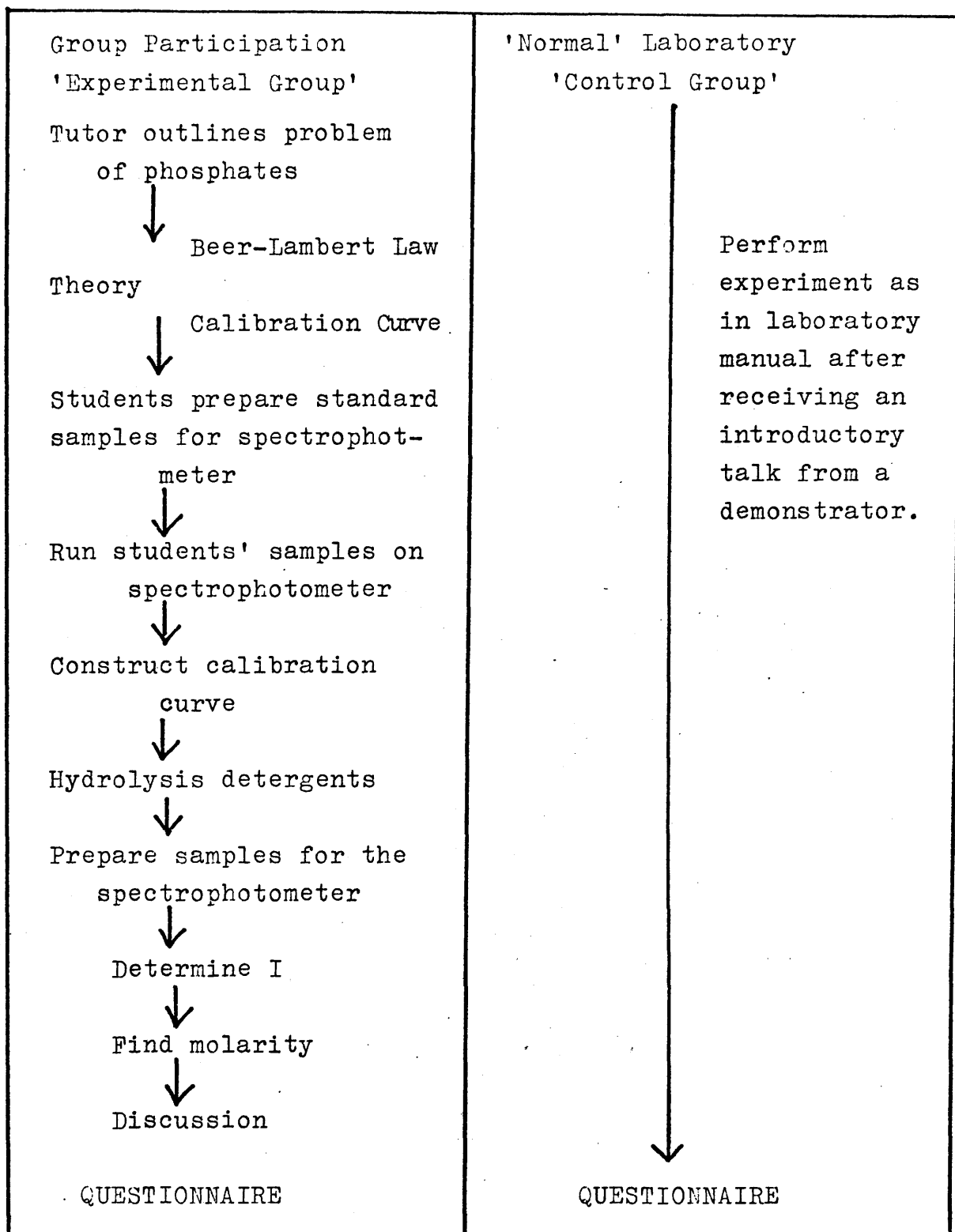
This experiment involved the construction of a calibration curve for a visible spectrophotometer to determine the concentration of phosphates in detergent samples. In this experiment the tutor began by discussing /

discussing the theory of why some solutions are coloured and the Beer-Lambert Law covering light absorption at a given wavelength and the concentration. This led to the idea of a calibration curve which the team (tutor and students) then produced. The students helped by making up solutions and running them in the spectrophotometer under the scrutiny of the rest of the students, and the tutor who could criticise the students for leaving fingerprints on cuvettes, etc.

The next part of the experiment involved the preparation of the detergent samples. Students working in groups boiled their solutions for half an hour and prepared the samples for the spectrophotometer. The team then got together and ran the samples and noted the results. Finally, there was a short discussion about errors and techniques.

### Experimental Design

As the 'phosphates' experiment was running in two different laboratories it was decided to use one of the laboratories for the 'experimental group' and the other for the 'control group'. Since no particular laboratory day had a representative mix of students, days were chosen at random to ensure a fair sample. On each day that we attempted the group participation approach, an average of approximately nine students attempted the experiment in each laboratory. This number varied from six to twelve. The reasons for using small groups of students was to minimise the disruption to the normal laboratory routine.



### Assessment

The questionnaire used for this experiment was the same as the one used to evaluate the first year pre-laboratory exercise (see section 6.2, p.120).

Results /

## Results

The results for Section A are included in Table 7.1.

Attitude results - Comparison of responses between  
Control and Experimental Group;  
Section A, questionnaire

As a result of completing this experiment, I have -		% <u>Control</u>		% <u>Expt.</u>		<u>Sig.</u>
1. become more interested in chemistry.	T	(10)	5	(19)	11	No sig.
	FT	(51)	29	(42)	24	
	U	(32)	18	(37)	21	
2. become aware of new practical techniques.	T	(30)	17	(51)	29	Sig. > 0.01
	FT	(38)	20	(35)	20	
	U	(28)	15	(16)	9	
3. increased my knowledge of the theory covered by the experiment.	T	(35)	18	(54)	31	*
	FT	(56)	29	(39)	22	
	U	(10)	5	(7)	4	
4. become aware of the importance of safety procedures.	T	(19)	10	(18)	10	No sig.
	FT	(15)	8	(25)	14	
	U	(65)	34	(58)	33	
5. become aware of the need for careful recording of results.	T	(38)	20	(46)	26	Sig. > 0.10
	FT	(52)	27	(37)	21	
	U	(10)	5	(18)	10	
6. gained confidence in my approach to practical problems.	T	(12)	6	(18)	10	No sig.
	FT	(62)	32	(54)	31	
	U	(27)	14	(26)	15	
7. become aware that theoretically expected results are seldom obtainable in practice.	T	(58)	30	(26)	15	Sig. > 0.005
	FT	(29)	15	(37)	21	
	U	(13)	7	(35)	20	
8. reinforced my existing practical skills.	T	(19)	10	(32)	18	Sig. > 0.10
	FT	(62)	32	(47)	27	
	U	(19)	10	(21)	12	
9. increased my knowledge of the application of chemistry to other subjects.	T	(29)	15	(40)	23	Sig. > 0.01
	FT	(42)	22	(46)	26	
	U	(29)	15	(14)	8	
10. /						

		% <u>Control</u>		% <u>Expt.</u>		<u>Sig.</u>
10.	appreciated the need for cleanliness in handling equipment.	T	(44) 23	(54) 37		Sig.
		FT	(27) 14	(35) 20		> 0.005
		U	(25) 13	(11) 6		

Table 7.1

Figures in brackets are percentages.

Size of samples:- Control - 52 students

Expt. - 57 students

Test of significance  $\chi^2$  squared test<sup>92</sup>

\* Statement 3; test not applied as expected frequency  
in one class is too small (< 5)

The experimental group became more aware of new practical techniques (statement 2; sig. > 0.01) and felt that they had reinforced their existing practical skills (statement 8; sig. > 0.1). The experimental group (statement 10; sig. > 0.005) were more aware of the need for cleanliness in handling equipment. This is not surprising as the lecturer was always present to supervise the students as samples were being run on the spectrophotometer. However, the control group felt that theoretically expected results were seldom obtainable in practice (statement 7; sig. > 0.005). The experimental group felt that the reverse was true. This may have been because the experimental group had been helped in getting results by the lecturer and had not tried to get results completely by their own efforts.

A further trend arose in favour of the experimental group as they felt they had increased their knowledge of the applications of chemistry to other subjects. In the introduction to this experiment in the laboratory manual there was a discussion of some of the issues raised by using phosphates in detergents. The trend may be explained because the lecturer covered this material in his presentation. The control group may not /

not have read the introduction to the experiment which covered this material.

The high significant values ( $> 0.01$ ) may indicate that the sampling was not completely random<sup>92</sup> and thus the results have to be treated with caution.

These results may also be explainable by the 'Hawthorne effect' as the students responded positively because they realised that they were part of an experimental group.

### Section B - Practical Skills - Experiment 7

The 'phosphates' experiment involved the use of a spectrophotometer. Of the practical objectives listed in Section B, 1, 3 and 4 were not applicable to this experiment. However, the students were simply asked to complete this section and were only told that not all the points listed were covered by this experiment (see Appendix 7.2).

The students were limited to four responses.

These were:-

- A. "Could do before this experiment and did not learn anything new."
- B. "Helped to improve my technique but not completely achieve this standard."
- C. "Because of this experiment I can now perform to this standard."
- D. "I could not do this before this experiment and I still have not learnt this technique."

Sample sizes:- Experimental group - 50; Control group - 44.

For objectives 1) to 6) see Appendix 7.2

- 5) Here for option (C) more of the 'experimental group' thought that they achieved this standard more frequently than the control group. However, for option (B) the control group replied more /

more frequently.

- 6) More of the experimental group felt that because of the 'phosphates' experiment they could now perform to the required standard. (Option C)

For objective 2) there were no differences as both sets of students had had equal practice before this experiment in handling a burette.

For objectives 2), 3) and 4) most students answered (A) or did not respond at all, as would be expected.

For comments on the validity of this method of assessing practical objectives see section 6.2 (p. 130).

Practical results - Comparison between Control and Experimental Group; Section B, questionnaire.

<u>Objective</u>	<u>%</u>		<u>%</u>	
	<u>Control</u>		<u>Expt.</u>	
1. Use a pipette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .	A	(80) 35	(74) 37	
	B	(16) 7	(18) 9	
	C	(-) -	(2) 1	
	D	(-) -	(2) 1	
2. Use a burette to measure variable volumes of liquid to an accuracy of $\pm 0.5 \text{ cm}^3$ .	A	(52) 23	(62) 31	
	B	(11) 5	(6) 3	
	C	(2) 1	(-) -	
	D	(-) -	(2) 1	
3. Carry out titrations confidently and carefully so that the end points in successive titrations agree to within $\pm 0.1 \text{ cm}^3$ .	A	(39) 17	(50) 25	
	B	(16) 7	(6) 3	
	C	(2) 1	(2) 1	
	D	(5) 2	(2) 1	
4. Carry out accurately the separation of immiscible liquids using a separating funnel.	A	(11) 5	(10) 5	
	B	(27) 12	(18) 9	
	C	(9) 4	(10) 5	
	D	(11) 5	(2) 1	
5. Use a spectrophotometer to obtain accurate values of I and $I_0$ .	A	(32) 14	(36) 18	
	B	(36) 16	(18) 9	
	C	(30) 13	(46) 23	
	D	(-) -	(-) -	
6. /				

		% <u>Control</u>		% <u>Expt.</u>	
6. Use a spectrophotometer and a set of standard solutions to produce a calibration curve for the instrument.	A	(20)	9	(14)	7
	B	(23)	10	(14)	7
	C	(36)	16	(60)	30
	D	(11)	5	(8)	4

Table 7.2

Figures in brackets are percentages

Size of samples:- Control - 44 students

Expt. - 50 students

Again, the trends indicated that the 'experimental group' with less practice felt more confident.

### Discussion

The results obtained for this pilot experiment tended to confirm our views on the advantages and disadvantages of this approach. It appeared that students do become more 'aware' of the practical techniques involved and of the theory behind the experiment. However, there was a feeling that students may have had a 'misplaced confidence' in their practical ability as they felt that they had mastered the techniques with less practice.

Members of staff who participated in this approach were asked to list possible advantages and disadvantages. The advantages that were listed for the group participation method were:-

1. More help to weak student;
2. Better employment of staff and demonstrators;
3. Lower costs;
4. Shows where students are making mistakes;
5. Helps nullify effects of incompetent demonstrators;
6. Bad errors can be seen and help given;
7. Direct experiment to show reasons for carrying-out procedures;
8. Teaches student to work more efficiently;
9. /



9. Makes students think;
10. Allows for more effective report;
11. Self-correcting of students' results;
12. Allows for better integration of theory and practical.

The disadvantages listed were:-

1. Demonstration apparatus must work;
2. Boring to good students;
3. Organisation;
4. Teacher prone.

The important factor to arise out of the lecturers' experience with the group participation method was that good students may find the pace too slow. This may lead to them being bored by this approach.

On the other hand, they felt that the approach was of benefit to slower students and that it helped them to point out misapprehensions quickly.

One point which was emphasised strongly in the interviews was that this method is tutor-prone, i.e. a poor tutor could destroy the advantages of this approach. This criticism also applies to many other methods of instruction.

### 7.3 Trial Experiment using Demonstrators

It was decided to test this approach using demonstrators instead of lecturers for a trial experiment. The results of this experiment would indicate whether or not this approach could be generally adopted if it was necessary. For the purposes of this trial the 'Phosphates in detergent' experiment was again chosen.

Every demonstrator was given charge of a group of ten to twelve students to guide through the experiment, in the same manner as described in the previous section.

As the background and experience of the demonstrators varied /

varied considerably their ability to guide a group in this manner successfully also varied. However, most demonstrators managed to cope, although one or two tried to revert back to their more normal approach. If this approach was used more often then it would be realistic to assume that there would always be some variability.

As all students were subjected to this approach there was no control group, instead students were issued with a questionnaire (Appendix 7.3) which asked them to compare the group participation approach to the 'normal' approach. This comparison was made on thirteen Likert type statements based on the previously listed advantages and disadvantages.

Statements could be grouped under four headings:-  
'Statements covering students' learning experience'

- a) Statements 1, 2, 3, 6, 8 and 12 were all aimed at finding out how much the students thought they had learned about the experiment.

'Statements covering possible disadvantages'

- b) Statements 4, 5 and 13 listed possible disadvantages of this approach.

'Student opinion of the group participation method'

- c) Statements 7, 9 and 11 were aimed at gauging how students had reacted to the experiment.

'A test statement'

- d) Statement 10 was included as it should show a neutral response.

## Results

The sample size was 228 students which represented over fifty percent of the possible sample size. Reasons for this size of sample were:-

- a) with demonstrators in charge this method normally lasted the full three hours and so many students did not have time to complete the questionnaire.  
Some /

Some students took the questionnaires home but few were returned the next day;

- b) some students were absent.

The results for Section I are included in Table 7.3, overleaf.

- a) 'Statements covering students' learning experience'

Students felt strongly that this approach helped them to clarify background theory (statement 1) and to see trends in experimental results (statement 2). It also helped them to understand the procedures (statement 6) and calculations (statement 8) outlined in the laboratory manual. This approach helped to make students aware of the importance of carefully recording results (statement 3). There was a positive trend indicated in the answers to statement 12 which might show that the students have realised the limitations of the techniques used i.e. the upper limits of phosphates detectable by calibration curve. However, for statements 3, 6 and 12 about half the class found no difference.

- b) 'Possible disadvantages'

About a third of the class thought that the pace was too slow (statement 4) and a quarter felt that it did not give them a chance to think for themselves (statement 5). Again, about a third of the class felt that the method was too restrictive (statement 13) as it did not allow them to work out procedure for themselves. These figures cannot be ignored and there may need to be a method found to 'siphon-off' the students who find the method too slow and leave them more on their own if this method is to be used generally. The answers to these statements were evenly distributed amongst those who agreed, disagreed or found no difference. Thus the opinion of the student population varied considerably.

- c) 'Student opinion of the group participation method'

The students were undecided if it would let them develop confidence in their practical work with half of them /

STATEMENT	Strongly agree	Agree	No difference	Disagree	Strongly disagree
<p>Comparing this method of presentation with the normal*type of lab. experiment, I found that this method:-</p> <p>* working in pairs after an introductory talk from a demonstrator</p>	12.7	40.0	34.4	10.5	0.3
<p>1. helped me to clarify the background results.</p>	8.9	40.5	39.2	12.8	0.2
<p>2. helped me spot trends in the experiment results.</p>	10.9	34.2	48.2	5.3	0.1
<p>3. made me aware of the importance of carefully recording results.</p>	7.0	23.7	29.8	28.5	12.7
<p>4. was boring as the pace was too slow.</p>	4.4	20.2	30.7	35.1	10.1
<p>5. did not give me a chance to think for myself.</p>	6.2	28.6	53.5	9.7	2.2
<p>6. helped me to understand why we followed the procedures outlined in the lab. manual.</p>	8.8	28.5	15.4	27.2	19.3
<p>7. should be used for every experiment.</p>	6.1	39.3	38.0	14.0	2.2
<p>8. helped to clarify all the calculations.</p>	5.3	23.1	50.2	11.8	9.2
<p>9. allowed me to develop confidence in my practical work.</p>					
<p>10. /</p>					

	Strongly agree	Agree	No difference	Disagree	Strongly disagree
10. made me appreciate the need for cleanliness.	2.7	22.1	60.6	11.1	3.5
11. would be boring if repeated too often.	18.3	31.7	15.2	31.3	7.1
12. helped me to appreciate the limitations of practical procedures.	4.0	28.4	54.9	11.1	1.3
13. was very restrictive - allowed me no scope to work out procedures for myself.	9.4	19.2	29.0	32.4	9.8

Table 7.3

Comparison of group participation method with 'normal' method of presentation. Figures are in percentages. Not all statements were answered by every student. Occasionally a statement was left blank.

them undecided and the other half split evenly (statement 9).

About half the students did not wish to see this approach universally adopted but a third did. This latter figure is quite large considering this was a once-off exercise (statement 7). About half of the students (statement 11) thought this approach would be boring if used too often. However, about two-fifths of the students disagreed.

d) 'A Test Statement'

As expected for statement 10 the results formed almost a normal distribution.

### Discussion

In general, the demonstrators were not so enthusiastic about this approach as the lecturers had been the year previously. This was not surprising as this approach is more demanding of the tutor and thus demonstrators with limited experience would find it more of a burden.

Students appeared to find that a group-participation approach helped them to clarify the theory, practical, calculations and to spot trends. These are the advantages that you would expect as all these points are covered more fully by the tutor. These results were consistent with the results of the pilot experiment.

However, the results of the 'phosphates' experiment also indicated some of the disadvantages of using this method. The disadvantages arise because of the nature of the groups being taught, which are mixed ability. This means that in a single group some students may have very little practical experience and others may have considerable practical experience even to the extent of having worked on their own projects. Thus, there is a danger that a tutor in setting a pace to try and suit everybody will suit no one as the pace will be too fast for /

for some and too slow for others. This is also a failing of many other approaches such as lectures.

To overcome this students with sufficient practical experience or confidence could be allowed to work on their own, although this would mean using additional equipment.

The effectiveness of this method will depend on the ability of the tutor to maximise the advantages and allow sufficient student involvement to encourage all students to participate fully. If demonstrators are to be used as tutors this may necessitate providing them with extra training. This method can be useful in experiments which students find difficult due to lack of knowledge about the theory or practical techniques.

## A P P E N D I X 7.1

Experiment 7 - Phosphates in DetergentsObjectives

When you have completed this experiment you should be able to -

- (1) use a spectrophotometer and a set of standard phosphate solutions to produce a calibration curve for the instrument;
- (2) use the calibration curve to determine the phosphate concentration in given samples of detergent powders;
- (3) maintain high standards of cleanliness in handling the spectrophotometer particularly by avoiding spillage.

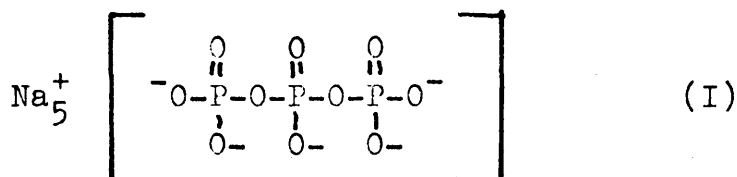
General

There has been considerable concern in recent years about the pollution of rivers and coastal waters by phosphates. The presence of phosphates in water appears to be partly responsible for a condition known as eutrophication, or over fertilisation. This causes increased growth of certain organisms at the expense of others. One example of this phenomenon is the accelerated rate of growth of algae on the surface of inland waters. The algae deplete the water of oxygen, with the result that other organisms die and the water becomes highly polluted, not least with dead fish. The presence of an excess of phosphate alone cannot result in the accelerated growth rates of algae, nevertheless, the control of phosphate pollution may go a long way to limiting the problem.

Two of the most important sources of phosphate pollution are agricultural fertilisers and industrial and domestic detergents. The detergents contain considerable /



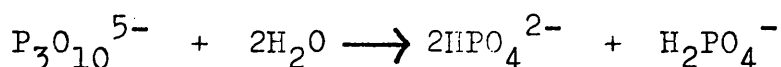
considerable quantities of sodium tripolyphosphate, (I)



This has a number of functions. It helps to soften the water by complexing  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions which would otherwise form a "scum" (soft water makes this less important in Scotland), disperses dirt in fabrics, and provides the slightly alkaline conditions necessary for the efficient operation of the "soap" in the detergent. Much effort is at present being devoted to the search for a substitute for polyphosphates in detergents, but the substitutes suggested so far appear to have more undesirable side-effects than those of polyphosphates.

### Method

In order to conveniently estimate the quantity of phosphates in a detergent the triphosphate must be hydrolysed to orthophosphate under acidic conditions.



(This step also occurs slowly in sewage or rivers.) The orthophosphate is best estimated in dilute solution by a colorimetric method. The phosphate is complexed (in this case by ammonium vanadomolybdate) to produce a coloured solution. The concentration of the phosphate in this solution may then be estimated by measuring the amount of light absorption at a specific wavelength. The instrument used is a spectrophotometer (see Experiment 6 for details) which measures the absorption of near-monochromatic light. The phosphate complex formed absorbs light in the blue region of the visible spectrum and therefore appears yellow. Accordingly, measurements of light absorption are best made in the blue region (at 470 nm in this case). The relationship between the light absorption at a given wavelength and the /

the concentration,  $C$ , of a coloured species in solution (Beer-Lambert Law) is:-

$$\log_{10} \frac{I_0}{I} = C \times \text{constant} \quad \begin{array}{l} \text{(related to the size of the} \\ \text{cell and the characteristics} \\ \text{of the complex)} \end{array}$$

(Optical density)

where  $I_0$  is the intensity of light incident on the sample and  $I$  is the intensity of light transmitted by the sample (both  $I_0$  and  $I$  are measured by a photoelectric cell connected to a galvanometer). Thus by plotting optical density against known concentration of coloured phosphate complex it is possible to estimate the phosphate concentration of an unknown solution from a measurement of its optical density.

### Experimental Method

Note that tap water may contain phosphates, therefore use distilled water only in the following operations.

Weigh, accurately, about 1 g. of detergent and place in a clean 250 ml. conical flask. Add 25 ml. of the 15% sulphuric acid solution supplied plus two drops of antifoam solution and gently boil the solution over a bunsen for 30 minutes to hydrolyse the triphosphate. Keep the volume of solution approximately constant by topping up with distilled water from time to time. Whilst the solution is boiling, you can conveniently make up a calibration scale for the spectrophotometer. Measure out 10.0 ml. of the standard phosphate solution supplied ( $3 \times 10^{-3} \text{M KH}_2\text{PO}_4$ ), add 5.0 ml. of ammonium vanadomolybdate solution and wait 5 minutes for the yellow colour to develop. Place a portion of the solution into a clean cubette (glass spectrophotometer cell) and add distilled water to the matched cuvette. With the spectrophotometer adjusted to a wavelength of 470 nm and the cuvette containing distilled water in front of the lens adjust the reading on the galvanometer to 10.0 (lower scale) using the "set zero" control on top of the galvanometer. /

galvanometer. The range switch on the galvanometer should be on  $\times 1$ . This gives  $I_0$ . Now slide the yellow solution in front of the lens and record the new reading  $I$  (which will be less than  $I_0$ , because less light is transmitted). Repeat the determination of  $I$  using solutions containing 5.0, 2.5, and 1.25 ml. of the standard phosphate made up to 10 ml. with distilled water in a graduated cylinder.

Then:-

conc. of phosphate  $\propto$  optical density

$$\propto \log_{10} I_0 - \log_{10} I$$

$$\text{or } \propto 1 - \log_{10} I$$

So that a graph of molarity of solution vs.  $\log_{10} I$  should be a straight line (if Beer's Law is obeyed).

The concentration of phosphate in the hydrolysed detergent can now be obtained by cooling and then transferring the boiled acid solution to a 250 ml. standard flask. (The soap in the detergent may not all have dissolved, but wash the conical flask out well with distilled water and transfer the washings to the standard flask.) Make the volume up to 250 ml. and mix thoroughly. Using a pipette, transfer 5.0 ml. of this solution to a 100 ml. standard flask and make up to volume; this solution should now have the correct concentration range for the colorimetric determination. Take 10.0 ml. of this diluted solution in a 100 ml. beaker add 5.0 ml. of ammonium vanadomolybdate solution as before and determine  $I$ . This will give the molarity,  $M$ , of the phosphate.

### Assignments

(1) Calculate the percentage phosphate in detergent -

$$\left( = \frac{M \times (\text{sum atomic wts. } PO_4^{3-})}{\text{wt. detergent}} \times \frac{250}{1000} \times 20 \times \frac{100}{1} \right)$$

(2) If time permits, you should also be able to estimate the phosphate concentration in the river sample, or, if negligible, estimate its upper limit.

## A P P E N D I X 7.2

University of Glasgow - Science Education Research Group

Throughout this term, the Chemistry Department wish to monitor your reactions to the inorganic experiments. Therefore, we would like you to complete the following questionnaire when you FINISH experiments 7 and 8. Your returns will be treated in confidence, and therefore you may be as frank in answering as you wish.

We thank you for your co-operation.

Please complete the following:-

Experiment No. \_\_\_\_\_ Lab \_\_\_\_\_ Lab Day \_\_\_\_\_

SECTION A

Please tick one of the alternatives to each question

As a result of completing this experiment, I have -

1. become more interested in chemistry.
2. become aware of new practical techniques.
3. increased my knowledge of the theory covered by the experiment.
4. become aware of the importance of safety procedures.
5. become aware of the need for careful recording of results.
6. gained confidence in my approach to practical problems.
7. become aware that theoretically expected results are seldom obtainable in practice.
8. reinforced my existing practical skills.
9. increased my knowledge of the applications of chemistry to other subjects.
10. appreciated the need for clean-

A	B	C
TRUE	FAIRLY	UNTRUE
	TRUE	

SECTION B

Could you please study the following list and place a tick in the most appropriate column. If you wish to elaborate on any point, please write your comments at the bottom. (Not all points are covered by this experiment.)

1. Use a pipette to measure variable volumes of liquid to an accuracy of  $\pm 0.5 \text{ cm}^3$ .
2. Use a burette to measure variable volumes of liquid to an accuracy of  $\pm 0.5 \text{ cm}^3$ .
3. Carry out titrations confidently and carefully so that the end points in successive titrations agree to within  $\pm 0.1 \text{ cm}^3$ .
4. Carry out accurately the separation of immiscible liquids using a separating funnel.
5. Use a spectrophotometer to obtain accurate values of  $I$  and  $I_0$ .
6. Use a spectrophotometer and a set of standard solutions to produce a calibration curve for the instrument.

Could do before this experiment and did not learn anything new.

ADDITIONAL COMMENTS:-

## APPENDIX 7.3

Matriculation No. \_\_\_\_\_

Today's experiment was taught by a different method where the tutor led you through the experiment and you worked as part of a group in getting results.

To discover what you thought of this method we would like you to fill in this questionnaire. As your answers will be treated in confidence we would appreciate if you would be as honest as possible.

Please place a tick in the appropriate column:-

Comparing this method of presentation with the 'normal'\* type of lab. experiment, I found that this method:-

\* working in pairs after an introductory talk from a demonstrator

	Strongly agree	Agree	No difference	Disagree	Strongly disagree
1. helped me to clarify the background theory.					
2. helped me spot trends in the experiment results.					
3. made me aware of the importance of carefully recording results.					
4. was boring as the pace was too slow.					
5. did not give me a chance to think for myself.					
6. helped me to understand why we followed the procedures outlined in the laboratory manual.					
7. should be used for every experiment.					
8. helped to clarify all the calculations.					
9. allowed me to develop confidence in my practical work.					
10. made me appreciate the need for cleanliness.					
11. would be boring if repeated too often.					
12. helped me to appreciate the limitations of practical procedures.					
13. was very restrictive - allowed me no scope to work out procedures for myself.					

## CHAPTER 8

### Open-ended Experiments and Project Work

## CHAPTER 8

Open-ended Experiments and Project Work

## 8.1 Introduction

At present in first and second year practical courses students are subjected almost exclusively to set experiments, which specify experimental procedures in great detail.

The amount of experimental detail included in laboratory manuals tends to stifle any ability that a student has to think for himself since it leaves the student no scope to work on his own.

In Chapter 2, page 50, a laboratory model was described which had two stages; the first being a learning stage where the aim was to teach the students the necessary skills and secondly, an experience stage where the aim was to give the student an opportunity to apply the skills learnt in the first stage and to develop 'non-manipulative' skills such as ability to plan experiments, to draw valid conclusions from experimental results, to think independently and to solve problems of a chemical nature.

There are two broad approaches which can be adopted to increase the possibility of giving the students the experience of thinking for themselves:-

- a) by providing fewer details for set experiments and/or providing no details at all for some problems thus making the experiments open-ended;
- b) by setting the students project work.

The first approach which could encourage independence in students is to provide less information in the laboratory manual, which will force students to work out some of the procedures for themselves. Examples of this would be to allow students to work out the molar quantities for a reaction for themselves or to work out the most suitable /



suitable molarity of acid to use in a titration.

This approach is open-ended in the sense that the student is left to make decisions for himself and need not arrive at a unique solution. With this approach the learning/challenge cycle is encapsulated in a single laboratory period as the student after initial practice is given a problem to solve using the techniques learnt. Advantages of this approach are that students are prevented from following the instructions like a recipe and there is minimum disruption caused to the organisation of the laboratory.

In the next section of this chapter the results of a preliminary investigation into this approach are discussed.

A second way to encourage independence in students is to introduce project work. Here the student is left to make his own decisions from the beginning of the experiment

There are two problems to be overcome here. The first is logistic because with a class of over five hundred students, as we have at present in the first year, any attempt at allowing free-ranging individual projects would almost certainly be faced with impossible organisational problems. The only feasible method to overcome this problem was to investigate the possibility of three-hour mini-projects which would cause the minimum disruption to the organisation as they could be fitted into available time slots.

The second problem arises because of the disparity in the practical experience of the students. Many students will never have attempted project work and therefore will be apprehensive about tackling it. To help overcome this we encouraged students to work in groups.

By basing the mini-projects on previously learnt techniques we were introducing a learning/experience cycle /

cycle which did not disrupt the organisation of the laboratory even though the complete learning/experience cycle lasted for more than one laboratory period.

Three mini-projects were developed and their assessment is described in this chapter.

a) Project 1 Phosphates in the River Kelvin followed a set practical experiment in which students learnt how to use a simple spectrophotometer to determine phosphate concentration colorimetrically. The set experiment entitled 'Phosphates in Detergents' was taught by a 'group-participation approach' and was described previously (see Chapter 7).

In this project students were provided with water samples taken from various parts of the River Kelvin and asked to analyse them for phosphate and to relate their results to the domestic and industrial environment of the river. Problems encountered were those of dilution and calibration. A short discussion was included at the end covering the effects of a total phosphate ban in the river on the social and economic environment. Therefore, students in one laboratory period had worked out a procedure to analyse the samples, carried out the analyses, investigated possible reasons for the results and finally discussed the implications of them.

b) Project 2 Chlorine in the Clyde Estuary was based on ion-exchange techniques which the students had encountered in two previous experiments. In this project the students attempted a quantitative analysis for the chloride ion in samples of water from the Clyde Estuary. The results were related to sample position, main tributaries and industry. This was the first time that the students had attempted a quantitative analysis using an ion-exchange column and students were faced with problems of preparing the resin, passing the samples through the column and titrating the eluent with acid.

c) Project 3 Citric Acid Content of Fruit Juices was based /

based on volumetric analysis. This project was related to a set experiment on determining the molarity of acids and bases. Before this set experiment the students were shown the films on the use of the burette and the pipette. This project involved determining the citric acid concentration in four commercially available fruit juices all of which were highly coloured. Students were asked to place them in order of value for money in terms of acid content.

Students had to make decisions about indicator range and suitability of colour change while problems of dilution of the fruit juice and of base had to be overcome.

## 8.2 Open-ended Experiments

To investigate the possibilities of introducing open-ended experiments into the first year course it was decided to choose one of the experiments contained in the laboratory manual and to make it open-ended by modifying it.

The experiment chosen was called 'Standard Solutions and Volumetric Analysis' (see Appendix 8.1) and in its original form involved determining the percentage of iron in an iron salt. As the laboratory manuals had already been printed containing detailed procedures, part of the experiment was altered and the students were asked instead to determine the percentage of iron in iron wire, thus ensuring that students could not use the details in their manuals. This change involved informing students to ignore sections b) and c) in the laboratory manual. In the modification the reaction was to be conducted in an air-free environment. This involved the use of a bunsen-valve which no student had used before.

Two forms of the modified experiment were prepared,  
one /

one for the control group and the other for the 'experimental group'. The control group were given a detailed list of instructions, to replace sections b) and c) (see Appendix 8.2). The 'experimental group' were given a branching programme which posed questions and suggested alternatives (see Appendix 8.3) on how to use a bunsen valve. Apart from describing the use of a bunsen valve no detailed instructions were given and students were left to make their own decisions.

### Assessment

This 'modified experiment' was run for three weeks. In the first week, which was designated the control, students were given full instructions (see Appendix 8.2). In the second and third weeks students were issued only with the branching programme (see Appendix 8.3) and demonstrators were instructed to give very little help to the 'experimental group', i.e. to answer a student's query with a question. With the control group 'normal' help was given to the students.

The number of students in the control group was approximately seventy-five and the number of students in the 'experimental group' was approximately one hundred and forty.

The assessment was designed to detect any changes in the students' attitude towards the experiment. A three-part questionnaire was devised (see Appendix 8.4) which consisted of a semantic differential, Likert-type statements and an objective ratings section, using a four-point scale.

### Results

It would be expected that any effects resulting from changing to 'open-ended' experiments would take time to appear since the students would need to alter their approach to tackling experiments. Thus, very little /



First adjective	Positive responses		Neutral		Negative response		Sig.
	C	E	C	E	C	E	
meaningful/	56	96	10	25	9	13	>0.005
varied/	38	84	19	27	16	25	>0.005
difficult/	9	41	24	52	42	45	>0.005
worthwhile/	56	98	13	30	6	10	>0.005
boring/	15	19	18	31	42	88	>0.005
accurate/	50	74	11	27	14	37	>0.005
pleasant/	38	82	31	37	9	18	>0.005
unimportant/	10	15	13	40	51	83	>0.005
useful/	65	10	3	25	7	10	*

Table 8.2

C - control

E - experimental

\* It was not possible to apply the  $\chi^2$  test as the frequency in one class is too low (< 5)

In every case in which it was possible to assess the results statistically the significance levels were all better than 0.005. While with smaller sample sizes this might suggest that the sampling was not random, with the large sample sizes in this experiment this would seem unlikely.

The experimental group found this experiment more varied, difficult and pleasant, while the control group found this experiment more meaningful, worthwhile, accurate, important and useful.

SECTION B /

## SECTION B

I think that this experiment -	AB		C		DE		Sig.
	C	E	C	E	C	E	
1. forced me to organise and plan procedures in advance.	32	91	15	26	27	22	>0.005
2. helped me to appreciate the limitations of the methods used.	46	82	17	36	12	19	>0.005
3. made me scrutinise the procedures for possible errors.	43	85	13	31	19	23	>0.005
4. gave me too much to do in too short a time.	4	11	9	8	63	121	*
5. allowed me to develop confidence in my practical work.	47	98	18	37	11	10	>0.005
6. did not give me enough instructions to work from.	4	26	5	15	67	98	*
7. illustrated practical applications of the lab. course.	56	105	15	26	4	8	*
8. was boring as there was too much repetition.	6	14	13	25	57	98	>0.005

Table 8.3

C - control

AB positive response

E - experimental

C no difference

DE negative response

Raw scores.  $\chi^2$ -squared test<sup>92</sup>

\* Statements 4, 6 and 7: test not applied as expected frequency in some classes is too small (< 5)

The results from Section B indicate that the open-ended /





	A		B		C		D	
	C	E	C	E	C	E	C	E
2. I can weigh out a sample to an accuracy of $\pm 0.0001$ g.	60	93	14	42	2	-	-	-
3. I can use a standard flask accurately to make up a standard solution of a reagent to a specified molarity.	54	112	22	30	-	1	-	-
4. I can carry out a titration using a standard solution of an oxidising agent to determine the amount of iron (II) in a sample to the required accuracy ( $\pm 0.1$ cm <sup>3</sup> ).	51	92	21	40	3	4	1	-
5. I can handle air-sensitive chemicals by protecting them from oxidation using a bunsen valve.	47	103	22	30	7	4	-	-

Table 8.4

Figures given are raw scores. Sample sizes as before. It was not possible to apply a  $\chi^2$ -test as two of the options, C and D, were not used often enough.

In objective 1 more of the control group felt that they had achieved mastery. However, this trend is reversed for option B. This may reflect a more cautious attitude on behalf of the experimental group.

In objectives 2, 3 and 4 there were no real differences between the control and experimental groups.

For objective 5 which was the technique covered in the open-ended part of the experiment the experimental group appeared to feel that they had achieved mastery.

## Discussion

It would appear from the results that the introduction of 'open-ended experiments' would encourage students to think more about the experiment as the 'experimental group' felt that they were forced to organise and plan in advance, i.e. to think about what they were doing. They also seemed more conscious of possible errors. However, the 'experimental group' also felt that they did not get enough instructions and that they found the experiment difficult. This would suggest that the students were aware of the different approach adopted, for this experiment. However, they seemed to find the approach more varied and pleasant.

The students did not seem to mind being left on their own and in fact felt that they had gained confidence in their practical ability and more of them felt that they had mastered the technique in the open-ended part of the experiment.

The results appear encouraging as this approach involves more work on the part of the student.

### 8.3 Phosphates in the River Kelvin - Mini-Project

Schematic Experimental Design - see Figure 8.1 overleaf.

#### Introduction

A description of the 'learning stage' is included in Chapter 7. The 'experience stage' was designed to reinforce the techniques covered in the 'learning stage' and in addition to give the students a chance:-

- to work out procedures for themselves;
- to organise and plan a suitable approach to the problem;
- to draw valid conclusions from experimental results;
- to think for themselves;
- to gain experience of problem solving situations;
- to use /

## Schematic Experimental Design

Learning StageWeek 1

'Phosphates In Detergents' experiments  
taught by a group participation approach  
(see Chapter 7, p. 164)

Techniques covered

Principle and construction of calibration  
curve

Preparation of samples for use in a spectro-  
photometer

Use a spectrophotometer

REINFORCEMENT  
OF TECHNIQUES


Experience StageWeek 2

'Phosphates in River Kelvin'

Decisions to be made

How to construct a calibration curve

How to prepare samples for use in a spectro-  
photometer

How to use a spectrophotometer

Discussion:- Awareness of social and  
economic implications of  
total phosphate ban.

Figure 8.1

to use an interdisciplinary approach to tackling  
problems;

to be aware of the social implications of decisions;

to be aware that some decisions have to be reached by  
compromise.

## Assessment

To assess if any of the above objectives had been  
achieved /

achieved, a questionnaire was designed (see Appendix 8.5) which was in two parts:-

- I fourteen Likert-type questions on a five-point scale asking the students to compare the project with the normal type of experiment.
- II two questions asking the students how they had enjoyed the project and how often they would like to see it repeated.

As every student had to attempt this or the 'chlorine' project (see Section 8.4) there were no students available to form a control group.

#### Sources of Information

The information on phosphate levels and sources of pollution in the River Kelvin<sup>93</sup> used in this project were provided by the Clyde River Purification Board.

Project:- The students were given the following information.

#### Phosphates in the River Kelvin

You have to determine the concentration of ortho-phosphates at various sampling points on the River Kelvin and suggest possible explanations of the different levels. In this experiment you will work in pairs. It is up to yourselves to decide on the method of analysis and how to organise the work.

The location of the sampling points and a sketch map of the area are provided.

The samples have been reduced to a  $\frac{1}{10}$ th of their original volume by evaporation.

Express your results in mg/l.

Table 1 /

Table 1 River Kelvin

Sampling point	Distance from source (km)	
Near source	2	← Dock Burn
Twechar	6	← Glazert Water
B 757 Bridge	10.5	← Luggie Water
Torrance	14.5	← Allander Water
Balmuidy	20	
Dawsholm	25	
Partick Bridge	29.5	

Map - Sketch Map of River Kelvin

An ordinance survey map of the area will be available for consultation. However, the sketch map will allow you to see the approximate locations of the sampling points.

Sketch Map of River Kelvin - see overleaf

Experiment 1 - Phosphates in River Kelvin

## Discussion

Discuss the implications of a ban on phosphate pollution at the Burnside Industrial Estate, Kilsyth, where the source of pollution is the bus garage which cleans fifty buses a day, seven days a week!

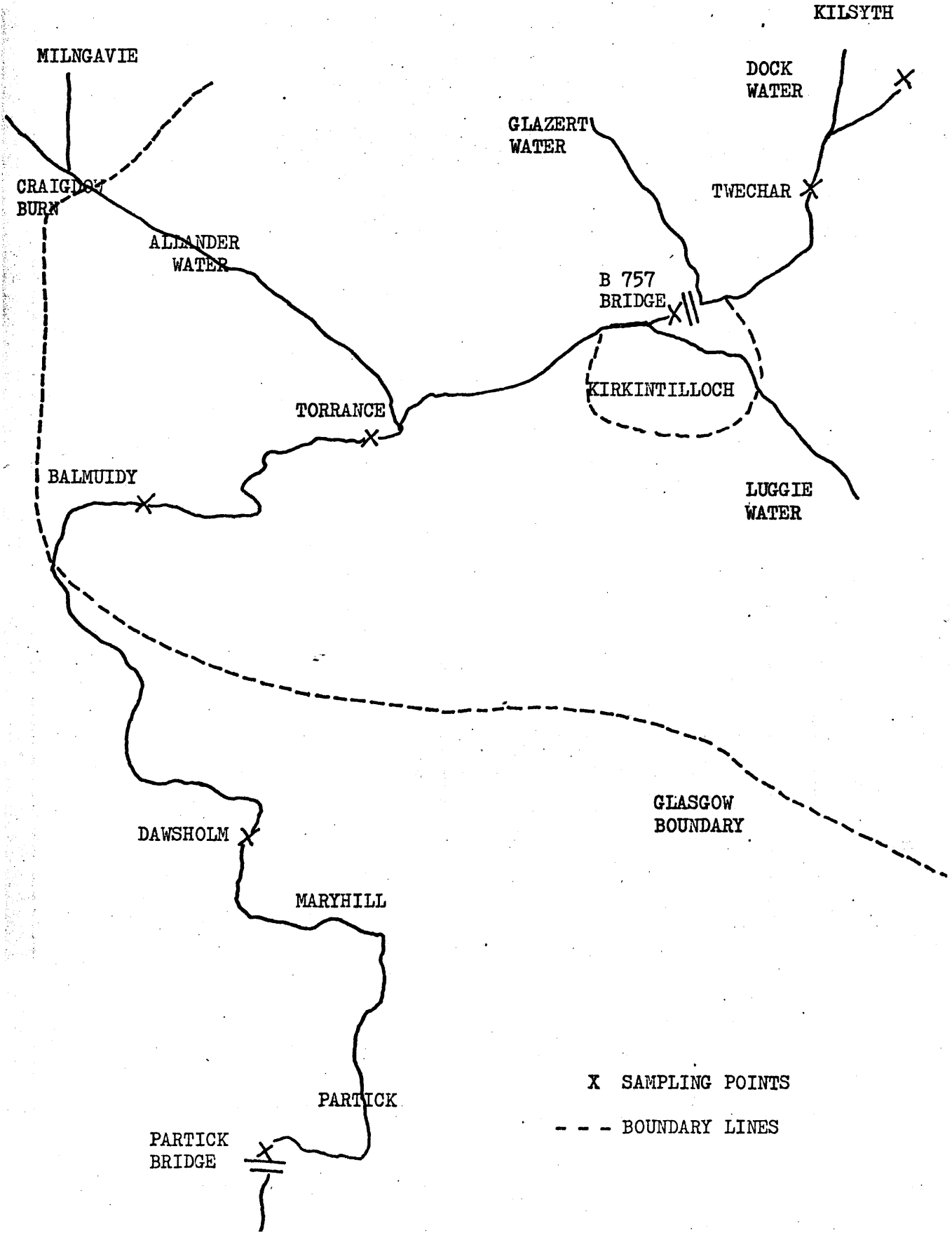
Investigate the costs of -

- a) alternative inc. 'phosphate-free' detergents; and
- b) connection of output to sewers, or alternative methods of disposing of effluent.

The demonstrator will provide you with details of the respective costs.

Demonstrators' Notes The demonstrators' notes giving details about the organisation of the exercise, phosphate levels, procedures, sources of pollution and figures costing alternative to a phosphate based detergent are included in Appendix 8.6.

Sketch Map of River Kelvin



X SAMPLING POINTS

- - - BOUNDARY LINES

Results: The results are given in Tables 8.5 and 8.6.

I think that this experiment -	Strongly agree	Agree	Undecided	Disagree	Strongly disagree	Sample size
1. allowed me to develop confidence in my practical ability.	9	37	33	19	3	153
2. gave me a lot of freedom to work out procedures for myself.	14	42	18	26	1	153
3. made an interesting change to the usual lab. experiments.	34	42	12	8	3	154
4. let me see that the lab. course had practical applications.	35	54	7	3	-	153
5. helped me to appreciate the limitations of practical procedures i.e. sensitivity or accuracy of methods.	28	46	20	5	1	154
6. forced me to organise my practical work i.e. plan procedures in advance.	10	46	27	15	1	154
7. improved my understanding of previous lab. experiments.	7	34	31	26	3	155
8. forced me to look for trends in my experimental results.	11	49	26	12	2	152
9. made me think about the experiment before I started any experimental work.	12	61	19	7	-	155
10. made me aware of the importance of carefully recording results.	12	50	29	8	-	155
11. made me scrutinise the procedures for possible errors in method.	11	46	24	18	1	154
12. /						

	Strongly agree	Agree	Undecided	Disagree	Strongly disagree	Sample size
12. allowed me to use my knowledge of other subjects to investigate the problem.	16	48	20	13	3	155
13. made me aware of the social implications of my decisions.	29	48	14	7	1	155
14. made me aware that some decisions have to be reached by compromise.	23	51	18	5	1	155

Table 8.5

(All figures as percentages)

'Phosphates in River Kelvin'

## I How enjoyable was this project?

	No. of ticks	Sample size	%
A Very enjoyable	20	138	15
B Quite enjoyable	63	138	46
C Average	42	138	30
D Unenjoyable	9	138	7
E Very unenjoyable	3	138	2

## II How often would the students like to see this project repeated?

	No. of ticks	Sample size	%
A All the time	10	138	7
B Frequently	35	138	25
C Sometimes	70	138	51
D Rarely	17	138	12
E Never	6	138	4

Table 8.6



## Discussion

Students quickly overcame any initial apprehension and tackled the project enthusiastically. The students organised themselves, distributing the work fairly, with little prompting from demonstrators. The results confirmed these observations.

Over sixty percent of the students enjoyed the project and over three-quarters of them felt that it made an interesting change to normal laboratory experiments (statement 3 ; 77%). Over eighty percent of the students stated that they would like to see similar exercises repeated at least sometimes, with a third of the sample wanting to see projects introduced frequently.

Ninety percent of the students saw that the laboratory course had practical applications (statement 4) and forty percent felt that it had improved their understanding of previous experiments (statement 7 ; 40%).

Nearly half of the students thought that the experiment had helped to develop confidence (statement 1 ; 45%) in their practical ability although a third thought there was no difference. For an approach which was completely different from anything the students had previously encountered at university and which demanded far more effort these trends are very good.

Students felt strongly that they were forced to think and organise their practical work in advance (statement 6 ; 56% ; statement 9 ; 73%) and over half also agreed that they were given a lot of freedom to work out procedures (statement 2 ; 57%). Thus students realised that they had to work through the project on their own without the normal help from the staff.

The students also seemed more aware of what they were doing during the project as they agreed that it helped them to appreciate the limitations of practical procedures (statement 5 ; 74%), made them aware of the importance /

importance of carefully recording results (statement 10 ; 62%), made them scrutinise the procedures for possible errors (statement 11 ; 57%), and forced them to look for trends in their experimental results (statement 8 ; 61%).

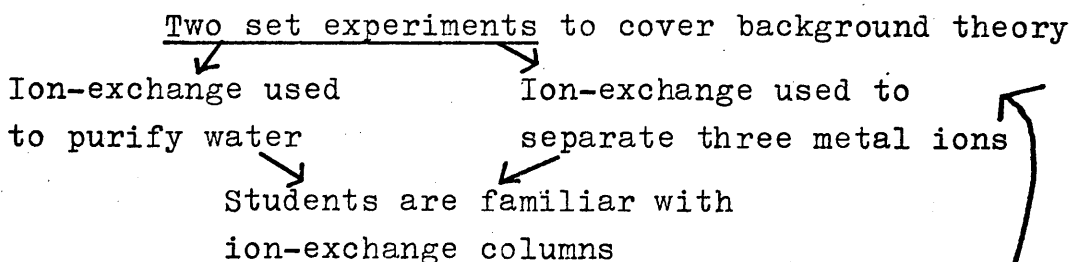
Finally, the discussion at the end of the project about the implications of a total phosphate ban in the River Kelvin seems to have been successful as in the replies to statements 12, 13 and 14 over sixty percent of the students either agreed or strongly agreed.

Thus, the 'phosphate' project was successful in achieving all the non-manipulative objectives that were set and in reinforcing the techniques taught in the learning stage.

#### 8.4 Determination of Chlorine Content in the Clyde Estuary by Ion-exchange.

##### Schematic Experimental Design

##### Learning Stage



##### Experience Stage

##### Quantitative Analysis by Ion-Exchange

##### Decisions to be made:

- How to prepare the resin
- How to pass through samples
- How to analyse the eluent
- How to calculate results

Figure 8.2

## Introduction

Two previous experiments had explained the background theory and demonstrated two possible uses of ion-exchange columns. The 'experience stage' involved the students devising a method to analyse quantitatively for chloride ions. This method was new to all but a few students who had covered the technique at school. As students were on holiday there was a gap of at least a few weeks between the learning and experience stage. However, it was felt that this project would reinforce their experience in using ion-exchange columns and ability to operate them. In addition it would force the students:-

- to work out procedures for themselves;
- to organise and plan a suitable approach;
- to draw valid conclusions from experimental results;
- to think for themselves;
- to gain experience of problem-solving situations.

There was no discussion section at the end of this experiment.

## Assessment

The questionnaire was the same as that used for the 'phosphates' project (see Appendix 8.5). Once again there was no control group and students were asked to compare the project with the normal type of experiment.

## Sources of Information

The information on chloride levels was provided by the Clyde River Purification Board.<sup>93</sup>

**Project :** The students were given the following information.

Experiment 2 : Determination of Chlorine Content in the Clyde Estuary by Ion-Exchange

You have to determine the concentration of  $\text{Cl}^-$  ions /

ions in the six samples of 'sea water' that you have been given and interpret your results. The equipment that you have been provided with is; an ion-exchange column, volumetric equipment and various concentrations of acids and bases. You are working as part of a team of six and therefore you will have to decide how to distribute the work-load among yourselves. Please remember that you have to know the efficiency of each ion-exchange column. Table 1 gives information about the location of the sampling-points.

By the end of the day you should be ready to present and explain the results that you have collected.

Table 1     Clyde River Purification Board

Samples taken from river run on 14.7.75

Sample	Situation	Miles below King George V Bridge
1	Upriver of R. Kelvin	2
2	Renfrew	6
3	Near Erskine Bridge	10
4	Cardross	16
5	Port Glasgow (Great Harbour)	20
6	Gourock	24

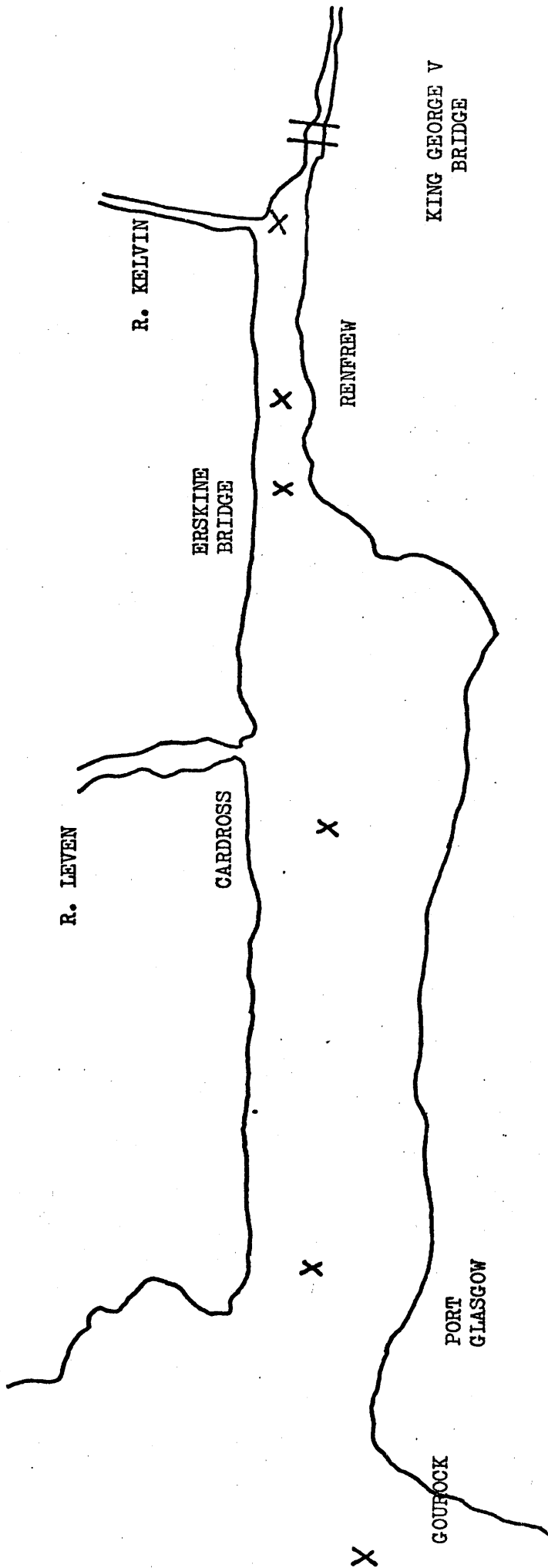
Sketch Map of Clyde between King George Vth Bridge and Gourock - see overleaf

Demonstrators' notes : The demonstrators' notes gave the details about the organisation of the exercise, chlorine levels of the various sampling points, procedures and background information (see Appendix 8.7).

## Results

The results are given in Tables 8.7 and 8.8.

Sketch Map of Clyde between King George Vth Bridge and Gourcock



X SAMPLING POINTS

Determination of Chlorine						
I think that this experiment -	Strongly agree	Agree	Undecided	Disagree	Strongly disagree	Sample size
1. allowed me to develop confidence in my practical ability.	6	51	26	15	3	98
2. gave me a lot of freedom to work out procedures for myself.	15	49	20	14	4	100
3. made an interesting change to the usual lab. experiments.	23	40	16	16	6	103
4. let me see that the lab. course had practical applications.	23	53	12	11	1	103
5. helped me to appreciate the limitations of practical procedures i.e. sensitivity or accuracy of methods.	15	38	30	16	1	100
6. forced me to organise my practical work i.e. plan procedures in advance.	29	44	11	13	5	102
7. improved my understanding of previous lab. experiments.	6	33	31	29	3	101
8. forced me to look for trends in my experimental results.	9	37	33	18	6	100
9. made me think about the experiment before I started any experimental work.	37	48	7	3	6	102
10. made me aware of the importance of carefully recording results.	9	46	22	21	3	99
11. made me scrutinise the procedures for possible errors in method.	12	48	24	14	3	100
12. /						

	Strongly agree	Agree	Undecided	Disagree	Strongly disagree	Sample size
12. allowed me to use my knowledge of other subjects to investigate the problem.	9	23	27	32	8	100
13. made me aware of the social implications of my decisions.	-	32	28	27	9	99
14. made me aware that some decisions have to be reached by compromise.	6	29	33	23	9	97

Table 8.7 (All figures as percentages)

I How enjoyable was this project?

	No. of ticks	Sample size	%
A Very enjoyable	18	96	19
B Quite enjoyable	36	96	38
C Average	28	96	29
D Unenjoyable	8	96	8
E Very unenjoyable	6	96	6

II How often would the students like to see this project repeated?

	No. of ticks	Sample size	%
A All the time	1	96	1
B Frequently	23	96	24
C Sometimes	47	96	49
D Rarely	16	96	17
E Never	9	96	9

Table 8.8

## Discussion

As in the case of the 'phosphates' project students were initially apprehensive but soon settled down to tackle this project, distributing the work load evenly among themselves.

About three-quarters of the students agreed that it made them think about the experiment before starting (statement 9 ; 85%) and that it forced them to organise and plan the procedures (statement 6 ; 74%). These figures suggest that the students found this experiment quite demanding. This would seem right as students were asked to work out a procedure with which few students were familiar.

However, over half the students enjoyed the experiment (56%) and 63% found it an interesting change (statement 3). In addition 75% of the students would like to see similar exercises repeated at least sometimes.

Nearly two-thirds of the students felt that this project was a practical application of the earlier experiments (statement 4 ; 77%) and about 40% of the students claimed that it had helped them to improve their understanding of previous laboratory experiments (statement 7 : 39%). The figure here may be low as it was not a direct application of previous experiments.

Over half the students felt that this project had helped them to develop confidence in their practical ability (statement 1 ; 57%) and only a quarter felt that it made no difference. Again these figures are encouraging for an innovation which required more effort on the part of the students.

Students seemed more aware of what they were doing during the project as it helped them to appreciate the limitations of practical procedures (statement 5 ; 53%), made them aware of the importance of carefully recording results (statement 10; 55%), made them scrutinise /



scrutinise the procedures for possible errors (statement 11; 60%) and forced them to look for trends in their experimental results (statement 8 ; 46%).

### 8.5 Comparison of the 'Phosphates' and 'Chlorine' Projects

A comparison of these two projects suggests that the assessment was reliable even though the number of completed questionnaires for both projects was around fifty percent of the total sample. The main reason why sample sizes were small was that students were short of time at the end of the laboratory period and left without filling in their questionnaires.

Statements 6 and 9 were included to act as a cross-check on the reliability of the assessment as both statements reflected the same phenomenon. The responses in each case followed the same trend.

The last three statements (12, 13 and 14) were only relevant to the 'phosphate' discussion but were included in both discussions. A chi-squared test<sup>92</sup> showed a significant difference in favour of the 'phosphate' project at the 0.1% level and the replies to these statements in the 'chlorine' assessment followed an almost normal distribution of responses.

Although the replies for statements 6 and 9 were consistent for each project there was a significant difference between the projects at the 0.1% level in favour of the 'chlorine project'. This suggests that the students found the 'chlorine project' more difficult which agrees with personal observation.

One other expected difference in the nature of the projects showed up in the responses. More students who attempted the 'phosphates' project realised the limitations of practical procedures (sig. 1%). This is explained by the fact that several of the 'phosphate' levels /

levels in river samples were too low to be picked up by the spectrophotometer. In the 'chlorine' project the method properly applied gave a result for each sample.

## 8.6 Citric Acid Project

### Schematic Experimental Design

#### Learning Stage

##### Volumetric Analysis

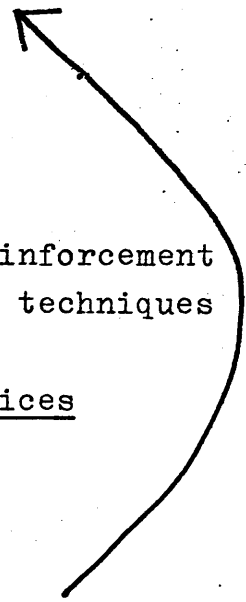
##### Set experiment (see Appendix 8.8)

Three-fifths of the students saw the two films on the use of the burette and the pipette.

Techniques covered:-

- use of pipette;
- use of burette;
- titrations;
- molarity calculations.

Reinforcement  
of techniques



#### Experience Stage

##### Citric Acid Concentration of Fruit Juices

Decisions to be made:-

- choice of indicator;
- how much to dilute fruit juice;
- how much to dilute base;
- how to calculate the molarity.

Figure 8.3

#### Introduction

A description of the teaching packages developed for the learning stage is covered in Chapter 5. The experience stage was designed to reinforce the students' ability with volumetric apparatus and to develop the same non-manipulative goals as described for the chlorine project (see previous section, p. 207).

All students performed the set experiment on volumetric analysis in the first week. Thereafter the students performed the experiments in a cycle with a new group of students tackling the project every day. Three-fifths of the students who attempted the project had viewed the two instructional films on the burette and the pipette shown in the first week. The rest of the students had received instruction from the demonstrators.

In the assessment (see Appendix 8.9) information was requested from the students about previous experience with volumetric analysis, practical experience and qualifications in chemistry. As this project was carried out early in the first term possible differences in practical experience at school level were still important - for instance, if there was a greater proportion of Sixth Year Studies students than normal this might bias the results, because of their previous experience with projects. The previous two projects had been carried out in third term and by this time a levelling out process would mean that the students' school experience would not be important. An attempt was made to compare their answers to these questions with their answers to the rest of the questionnaire. It was hoped that this would provide information about any relationship between background and how students react towards the project, i.e. the confidence with which they tackled the project. A comparison of the results obtained from students who had watched the film and those students who did not see them was also attempted.

Since students had little experience of the 'normal' type of laboratory experiment, they were asked what they thought of this experiment using a five-point Likert scale. There was no control group as all students had to attempt this project during the first term.

Project: The students were provided with the following information.

## Concentration of Citric Acid in Fruit Juices

### Objectives

The objectives of this experiment are:-

1. to give further practice in handling burettes and pipettes to the required accuracy (see experiment 1);
2. to give further practice in carrying out titrations to the required accuracy (see experiment 1);
3. to give further practice in molarity calculations;
4. to show the uses and limitations of indicators;
5. to use a standard flask accurately to make up a standard solution of a reagent to a specified molarity.

You are required to determine the concentration of citric acid in each of the four fruit drinks provided and list the drinks in order of value for money in pence per gram of citric acid. The method you devise is limited to using the volumetric apparatus and indicators provided. You will be provided with only 15 cm<sup>3</sup> of each drink so THINK BEFORE YOU START! You will need to think particularly of the colour changes of the indicators and the concentration of the sodium hydroxide. If you have any problems consult a demonstrator.

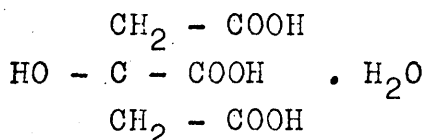
The fruit drinks you are provided with are:-

lemon squash;  
blackcurrant cordial;  
orange squash;  
lime cordial.

Other materials provided:-

volumetric apparatus - pipette, standard flask, burette;  
1M NaOH ;  
selection of indicators.

The formula of citric acid is:-



The formula /

The formula weight in the hydrated form is 210. You can assume that citric acid is the only acid in these drinks.

The following list will help you to choose suitable indicators. (Remember that you must be able to see the colour change and that citric acid is a weak acid.)

Indicator List

Common Name	pH range	Colour of acidic form	Colour of basic form
Thymol blue	1.2 → 2.8	Red	Yellow
Methyl orange	3.1 → 4.4	Red	Orange
Bromocresol green	4.0 → 5.6	Yellow	Blue
Methyl red	4.4 → 6.6	Red	Yellow
Bromothymol blue	6.2 → 7.6	Yellow	Blue
Cresol red	7.2 → 8.8	Yellow	Red
Phenolphthalein	8.0 → 10.0	Colourless	Red
Alizarin yellow	10.0 → 12.0	Yellow	Lilac

Demonstrators' Notes: The demonstrators' notes gave details about results, possible points of difficulty and organisation (see Appendix 8.10).

### Results

The results for SECTION A were (frequencies):-

- Present qualifications in chemistry of students who tackled citric acid project:-

Higher	-	165	(76%)
Sixth Year Studies	-	47	(22%)
A-level	-	4	(2%)
Others	-	2	(1%)

- Number of students who left school in:-

Fifth year	-	85	(39%)
Sixth year	-	132	(61%)

- How often, after initial instruction from your teacher, were you allowed to conduct an experiment on your /

your own?

- A) Frequently 76
- B) Quite often 72
- C) Seldom 61
- D) Never 13

4. Were you ever encouraged to devise and conduct your own experiments?

- A) Frequently 16
- B) Quite often 28
- C) Seldom 92
- D) Never 86

5. How often did you use a pipette at school?

- A) Very often 52
- B) Several times 92
- C) Once 39
- D) Never 38

6. How often did you use a burette at school?

- A) Very often 56
- B) Several times 105
- C) Once 35
- D) Never 25

7. When carrying out a titration were you asked to:-

- A) accept the value of the first readings. 35
- B) repeat the titration until successive readings agreed to within  $\pm 1 \text{ cm}^3$ . 58
- C) repeat the titration until successive readings agreed to within  $\pm 0.1 \text{ cm}^3$ . 102
- D) repeat the titration until successive readings agreed to within  $\pm 0.05 \text{ cm}^3$ . 8

The results for SECTION B are shown in Table 8.9.

I think that this experiment:-	Strongly agree	Agree	No difference	Disagree	Strongly disagree
1. forced me to organise and plan procedures in advance.	14	77	7	5	1
2. illustrated practical applications of the lab. course.	19	63	14	3	2
3. helped me to appreciate the limitations of the methods used.	19	47	25	5	1
4. did not give me enough instructions to work from.	9	19	16	45	11
5. allowed me to use my knowledge of other subjects to investigate the problem.	4	22	20	40	14
6. was boring as there was too much repetition.	4	11	18	50	13
7.* gave me too much to do in too short a time.	-	-	-	-	-
8. gave me confidence in my ability to do molarity calculations.	10	45	28	12	5
9. showed me the limitations of using indicators.	23	48	15	8	4
10. improved my ability to do titrations to the required standard.	19	55	15	5	3
11. made me scrutinise the procedures for possible errors.	6	49	28	11	4
12. allowed me to develop confidence in my practical work.	10	56	23	5	4
13. was interesting as it applied to real life.	16	48	20	13	5
14. forced me to look for trends in my experimental work.	6	25	48	17	3

Table 8.9

(Figures are given in percentages.)

The results for SECTION C are given in Table 8.10.

1. How students enjoyed this exercise:-

	%
A Very enjoyable	9
B Enjoyable	61
C Average	22
D Unenjoyable	5
E Very enenjoyable	2

2. The frequency with which they would like to see this type of exercise repeated:-

	%
A All the time	2
B Frequently	26
C Sometimes	60
D Rarely	9
E Never	4

Table 8.10

### Sampling

The sample size was 229 although a few students did not complete all responses, so the sample size for individual statements might vary. Completed questionnaires were obtained from over ninety percent of the students who attempted the project.

The replies to questions 1 and 2 indicated that the sample was representative of students entering first year science courses.

### Discussion

Students did not appear so apprehensive as the students who had attempted the two projects in the previous year. This may have been because students did not see it as an exercise different from the other laboratory experiments, because of their limited experience in the



the laboratories.

Nearly seventy percent of the students found the project enjoyable (70%) and nearly three-quarters of the students would like to see similar exercises repeated at least sometimes (77%).

Over eighty percent of the students realised that the project illustrated a practical application of earlier experiments (statement 2 ; 82%) and over sixty percent found that it was interesting as it applied to real life (statement 13 ; 64%). Although the procedures adopted were repeated for each juice the students did not find the exercise boring (statement 6 ; 62%).

Students also found that the project forced them to organise and plan procedures in advance (statement 1 ; 87%) and allowed them to develop confidence in their practical work (statement 12 ; 66%).

The students felt that they had increased their ability to carry out titrations (statement 10 ; 74%), to do molarity calculations (statement 8 ; 55%) and to understand the limitations of indicators (statement 9 ; 71%).

The replies to statements 3 and 9 were a close match as was expected as they covered the same phenomenon. This suggests that students were responding consistently. In addition, statements 5 and 14 were included as test statements. As expected few students agreed with statement 5 which was not applicable to this project. The replies to statement 14 formed a normal distribution which was expected as there were no trends in the amount of citric acid in each fruit juice.

The replies to statement 7 were not totalled because the work load was reduced after the first week as the students did not have enough time to determine the acid content of four juices. After the first week, students /

students attempted the project in groups of four with each student doing one acid determination. The replies in weeks 2 and 3 reflected this.

Statement 7 gave me too much to do in too short a time

	A	B	C	D	E
Week 1	13	18	10	26	10
Week 2	4	10	11	32	10
Week 3	4	7	1	42	15

Table 8.11

Student responses were analysed according to previous practical experience. To do this students' previous experience with the burette and pipette was compared to their attitude towards statements 4, 8, 10, 11 and 12 in Section B and questions 1 and 2 in Section C. To make the analysis simpler students who had little or no experience with either the burette or pipette (questions 3 and 6, options C and D) were compared with students who had used these pieces of apparatus often (questions 5 and 6, options A and B). The scores were compared statistically using a chi-squared test. Only one significant difference at the 5% level was found and that was for statement 4. Here students who had little experience with the burette and the pipette felt that they were not given enough instructions to work from. This difference is explained in that students who have not had much experience with this apparatus are bound to feel more apprehensive.

A further analysis was conducted to discover whether or not students who had devised and conducted their own experiments (question 4, options A and B) had a different attitude towards the project than students who had little or no experience in devising their own experiments (question 4, options C and D). However, no significant differences were found between the two groups.

A final /

A final comparison was made between students who had watched the two films on the use of the burette and the pipette before starting the project and those who had not, to examine if the films had developed confidence. There were no apparent differences in trends.

This project was successful in strengthening the students' techniques and in achieving the non-manipulative goals. It appears to have been set at the correct level being not too difficult for students with little practical experience nor too easy for students with more practical experience.

## 8.7 Conclusions

The research embodied in this chapter has shown that it is possible to introduce project work into the normal laboratory timetable without creating major organisational problems.

We have also shown that open-ended and project work is an extremely useful tool in giving the students an opportunity to reinforce previously learnt techniques and to achieve non-manipulative goals such as planning and organising an experimental approach.

A common feature of all the projects has been the level of involvement and initiative shown by the students which has been considerably higher than for 'normal experiments'. The fact that the students have enjoyed these projects is demonstrated by the number of students who wished to see exercises of this type repeated.

It is difficult to assess the extent of the 'novelty factor' in the students' responses and further work would need to be carried out in this direction. Although, it might have been expected that students faced with a situation which involved more work and effort on their part, and which to many students was an introduction to work of this nature, might have had a negative effect on the students' attitudes.

## A P P E N D I X 8.1

Experiment 3 - Standard Solutions and Volumetric Analysis

Objectives By the end of this experiment you should be able to:-

1. use a single pan balance confidently paying due attention to cleanliness and accuracy;
2. weigh out samples to an accuracy of  $\pm 0.0001$  g.;
3. using a standard flask, accurately make up a standard solution of a reagent to a specified molarity;
4. carry out titrations using a standard solution of an oxidising agent to determine the amount of iron (II) in a sample, paying due attention to the objectives of experiment 1.

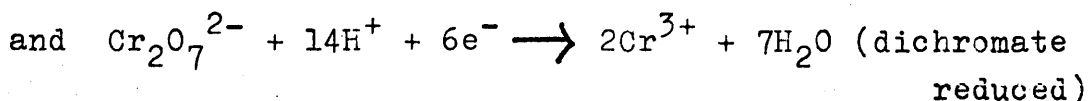
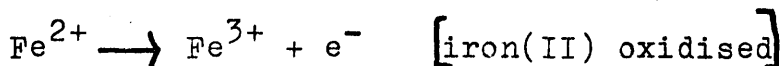
Introduction

The concentration of an iron(II) solution can conveniently be determined by oxidising the iron(II) to iron(III) using oxidising agents such as acidic solutions of the permanganate ion ( $\text{MnO}_4^-$ ) or dichromate ion ( $\text{Cr}_2\text{O}_7^{2-}$ ).

In this experiment you are required to prepare a standard solution of potassium dichromate and use this to determine the percentage of iron(II) in an unknown salt.

Theory and Calculations

The relevant equations are:-



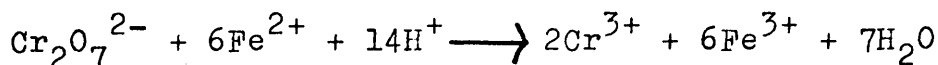
$$\therefore 1 \text{ mole of } \text{Fe}^{2+} \equiv 1 \text{ mole of } e^-$$

$$\text{and } 1 \text{ mole of } \text{Cr}_2\text{O}_7^{2-} \equiv 6 \text{ moles of } e^-$$

$\therefore /$

∴ 1 mole of  $\text{Cr}_2\text{O}_7^{2-}$  will oxidise 6 moles of  $\text{Fe}^{2+}$ .

∴ overall equation



Wt. of  $\text{K}_2\text{Cr}_2\text{O}_7$  in 100 ml of solution = A grams

Molecular weight of  $\text{K}_2\text{Cr}_2\text{O}_7$  = 294.2

∴ No. of moles of  $\text{K}_2\text{Cr}_2\text{O}_7$  in 100 ml solution =  $\frac{A}{294.2}$

∴ Molarity of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution =  $\frac{A}{294.2} \times 10 = \underline{\underline{B}}$

If z mls of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution are required for 25 ml iron(II) solution

No. of moles of  $\text{K}_2\text{Cr}_2\text{O}_7$  for 25 ml iron(II) solution

$$= \frac{B}{1000} \times z = \underline{\underline{C}}$$

∴ No. of moles of iron(II) in 25 ml of solution

$$= \underline{\underline{6 \times C}}$$

Now 1 mole of iron(II) contains 55.85 g of iron(II)

∴ Wt. of  $\text{Fe}^{2+}$  in 25 ml of solution =  $6C \times 55.85$  g

∴ Wt. of  $\text{Fe}^{2+}$  in 100 ml of solution =  $\underline{\underline{6 \times C \times 55.85 \times 4}}$  g  
=  $\underline{\underline{D}}$

∴ % iron(II) in unknown salt =  $\left( \frac{D \times 100}{\text{Wt. of unknown salt}} \right)$

### Experimental Procedure

#### (a) Preparation of Standard Solution of Potassium Dichromate

Using a weighing bottle, weigh out approximately 0.5 g of potassium dichromate and record the weight to within  $\pm 0.0001$  g. With the aid of a small funnel, transfer the solid to a 100 ml graduated flask washing the funnel with approximately 50 ml distilled water. (N.B. DO NOT wash out the weighing bottle.) Mix the contents of the graduated flask until all the solid has dissolved, (NEVER heat a graduated flask, why?) and CAREFULLY make up the solution with distilled water /

water to the volume indicated by the graduated mark on the stem of the flask. Do not "overshoot" the mark. Insert the stopper and mix the solution thoroughly by inverting the flask at least five times.

Accurately reweigh the weighing bottle and any small quantity of dichromate which was not transferred to the graduated flask. Obtain the weight of potassium dichromate in the solution by difference between the initial and final weighings. Record your results as follows:-

Wt. of weighing bottle + $K_2Cr_2O_7$	=	g
Wt. of weighing bottle + residual $K_2Cr_2O_7$	=	<u>g</u>
∴ Wt. of $K_2Cr_2O_7$ in solution	=	<u>g</u>

(b) Preparation of Standard Solution of Unknown Salt

Using the above procedure, prepare 100 ml of a solution of the unknown salt using approximately 3 g of the salt and dissolving this in about 50 ml of 5% sulphuric acid, before finally "making up" to the mark with distilled water. Mix thoroughly as before. Record your results as previously shown.

(N.B. When preparing a standard solution it is not usually necessary to have a precise amount of solid. However, it is important to know accurately the weight of solid used.)

(c) Determination of Percentage of Iron(II) in the Unknown Salt

Using the "Pumpette" device, pipette 25 ml of the iron(II) solution into a 350 ml flask. Using a measuring cylinder, measure out, and add to the iron(II) solution, 100 ml 5% sulphuric acid and 5 ml 85% phosphoric acid. Add 8 drops of sodium diphenylaminesulphonate indicator. Titrate this solution SLOWLY with the standard potassium dichromate solution, stirring constantly until the solution assumes a bluish-green tint. Continue to add /

to add the dichromate solution dropwise until, at the end-point, an intense purple colour is obtained. Note the volume of dichromate required and repeat the titration twice more. Record your titration results in a similar manner to that shown in experiment 1.

## A P P E N D I X 8.2

Alteration to Experiment 5

There is a change in the experiment as written in the lab. manual. Sections B) and C) are replaced by the experiment on this sheet. However, section A) remains the same, i.e. you should still make up the standard solution of potassium dichromate.

To find the percentage of iron in iron wire

THEORY Commercial iron wire is not chemically pure although the amount of impurities in some varieties of iron is very small.

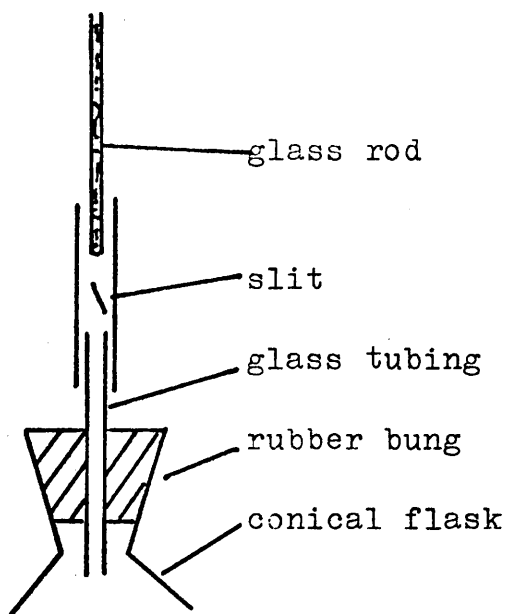
To determine the amount of iron, the iron wire has first to be cleaned to remove traces of rust and then dissolved in acid. Sulphuric acid is used to dissolve the iron because the iron produced is present only in the iron(II) state. With other acids such as nitric acid a mixture of iron(II) and iron(III) can be produced.

An additional complication in this reaction is that air will slowly oxidise the iron producing iron(III). Since potassium dichromate, which you will use to titrate the iron solution is an oxidising agent, this would result in an error.

To prevent this a bunsen valve is used.

A bunsen valve consists of a narrow rubber tube closed with a short piece of glass rod. A longitudinal slit in the rubber allows gas to escape outwards, but prevents any air getting in. Therefore the solution is protected from oxidation by the air.

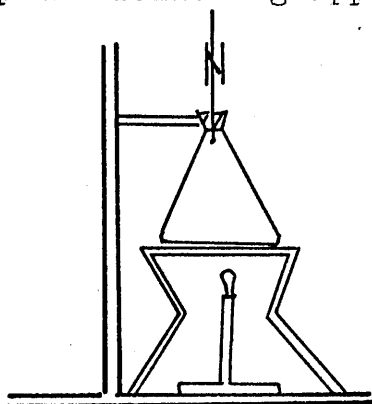




Once the iron is dissolved the solution is made up to a standard volume using a 100 ml standard flask and titrated with potassium dichromate. (See introduction to Experiment 5.) The calculation remains the same.

**EXPERIMENT** To determine the percentage of iron in iron wire break off a two inch section of iron wire and remove any traces of rust by means of the emery cloth - make sure the wire is clean! Weigh the weighing bottle provided and note the weight accurately. Place the sample of iron wire in the weighing bottle and reweigh. Calculate the weight of the iron to within  $\pm 0.0001$  g.

Set up the following apparatus:-



bunsen valve

250 ml conical flask

bunsen burner

Place the wire in the conical flask and pour in 20 - 30 cm<sup>3</sup> of 2M H<sub>2</sub>SO<sub>4</sub>. Replace the bunsen valve checking that it is fitted tightly.

Heat /

Heat the solution gently until the iron dissolves. (Minute particles of carbon sometimes remain undissolved.)

Allow the solutions to cool and then transfer to a 100 cm<sup>3</sup> standard flask. Make up to the graduation mark with distilled water. Remember to rinse the conical flask to ensure that all the solution has been removed.

Pipette 25 cm<sup>3</sup> of the solution into a conical flask and add a few drops of sodium diphenylaminesulphonate indicator. Titrate this with the potassium dichromate solution. Remember the end-points of the titrations should coincide to within  $\pm 0.1$  cm<sup>3</sup>.

## A P P E N D I X 8.3

Experiment 5

Problem: To find the percentage of iron in iron wire.

You are asked to find the percentage of iron in iron wire as commercial iron wire contains some impurities.

Before starting this experiment please read the following instructions carefully!

1. This problem replaces sections b) and c) in the lab. manual. However, you will still need to make up the standard solution of potassium dichromate as outlined in section a).
2. To help you with any procedural points that may puzzle you, you should work through the following programme which has been designed to help you.

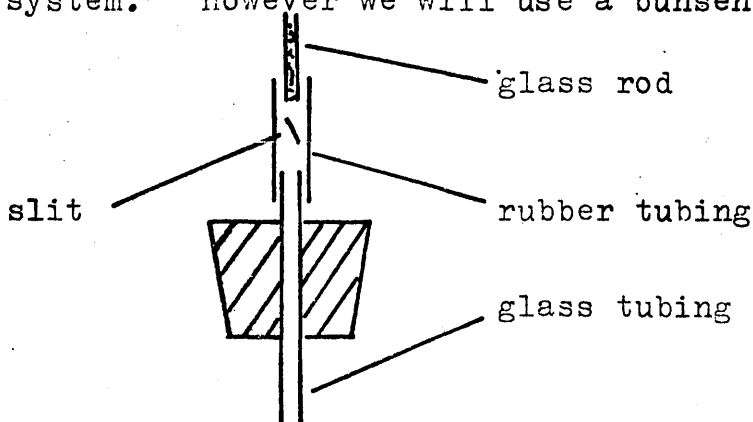
Start with Question 1). Choose the alternative which is appropriate in your case and pass on to the question which is indicated after the alternative.

- 1) At what point are you stuck?
  - a) I have no idea how to tackle the problem. → 13)
  - b) I do have some idea, but I am stuck on some experimental points. → 5)
  - c) Neither a) nor b) → consult demonstrator.

- 2) No - with warm dilute nitric acid some of the iron(II) ions are oxidised to iron(III) ions. → 7)
- 3) Right, before titrating with  $K_2Cr_2O_7$  the solution should be cooled and made up to a known volume - What piece of apparatus should you use? What indicator? Once this is done proceed with the titration. For any further information please consult the lab. manual.

4) /

- 4) No 8)
- 5) Go to the first experimental point which causes you trouble.
- A) How to measure the quantity of iron. 13)
- B) How to dissolve the iron wire. 11)
- C) The effect of air on the solution. 7)
- D) Anything else consult demonstrator.
- 6) No - air will in fact oxidise the iron to the iron(III) state and not reduce it. 10)
- 7) Yes - iron dissolves in non-oxidising acids such as dilute sulphuric acid to yield Fe(II). The  $H_2SO_4$ /Fe(II) solutions should be heated gently with a bunsen burner.
- With warm dilute nitric acid some of the iron goes to the Fe(III) state.
- What effect will air have on the solution?
- A) No effect. 8)
- B) Negligible. 4)
- C) Oxidise  $Fe^{2+}$   $Fe^{3+}$ . 10)
- D) Reduce  $Fe^{3+}$   $Fe^{2+}$ . 6)
- 8) No - the air will oxidise some of the iron Fe(II) Fe(III) oxidised.
- Thus air must be excluded from the reaction vessel. 10)
- 9) No - dichromate is an oxidising agent. Therefore it will oxidise the iron.
- Fe(II) Fe(III) 11)
- 10) Yes - the air will oxidise the iron and produce some iron(III) ions. For the redox titration all the iron needs to be in the iron(II) state. There are several ways to exclude air from a reaction system. However we will use a bunsen valve.



A bunsen valve allows air to escape through the slit in the rubber tube but prevents any air getting in.

How will you proceed once you have succeeded in dissolving the iron wire?

Can you titrate it with  $K_2Cr_2O_7$  immediately?

a) Yes → 12)

b) No → 3)

11) Correct, the iron should be in the iron(II) state.

To perform a redox titration you will have to dissolve the iron in acid and make sure that all the iron is in the same oxidation state.

In section a) of your lab. manual you were asked to make up a solution of  $K_2Cr_2O_7$ . This will oxidise  $Fe(II) \rightarrow Fe(III)$ . Therefore you will need to ensure that the iron is all in the iron(II) state.

Which acid could you use to ensure that the dissolved iron will be in the correct oxidation state?

a) Warm dilute  $H_2SO_4 \rightarrow 7)$

b) Warm dilute  $HNO_3 \rightarrow 2)$

12) No - the solution should be cooled and made up to a specified volume. A portion should then be titrated with  $K_2Cr_2O_7$ . → 3)

13) You have been asked to find the quantity of iron in iron wire. Commercial iron is not chemically pure, although the amount of impurity in some varieties of iron is very small.

How then can you measure the quantity of iron? The simplest method is to perform a redox titration.

This requires all the iron to be in the same oxidation state. Which oxidation state is necessary if you are to titrate with the solution of  $K_2Cr_2O_7$  which you made up in section a) ?

a)  $Fe(II) \rightarrow 11)$

b)  $Fe(III) \rightarrow 9)$

## A P P E N D I X 8.4

Experiment 5    1st Year Chemistry Lab.    Questionnaire

After finishing experiment 5 could you please complete this questionnaire. All information supplied by you will be treated in strict confidence and will not be used in any way to alter the assessment of your performance in the lab.

Thank you for your co-operation.

SECTION A    The purpose of this section is for you to make judgements on a series of scales. For instance, if you believe very strongly that this experiment was repetitive, then mark the scale:-

varied            \_ : \_ : \_ : \_ : \_ : \_ : X            repetitive

If your feelings are neutral on this issue then you should mark the scale:-

varied            \_ : \_ : \_ : X : \_ : \_ : \_            repetitive

Place a cross at the position on the scale that best suits your opinion. You have seven options varying from strongly agree through to strongly disagree.

- IMPORTANT:-
1. Please place your check-marks in the middle of spaces, i.e. \_ : X : \_  
not \_ : \_ X \_
  2. Complete each scale.
  3. Never put more than one check-mark on a single scale.

What did you feel about this experiment?

meaningful	_ : _ : _ : _ : _ : _ : _	meaningless
varied	_ : _ : _ : _ : _ : _ : _	repetitive
difficult	_ : _ : _ : _ : _ : _ : _	easy
worthwhile	_ : _ : _ : _ : _ : _ : _	worthless
boring	_ : _ : _ : _ : _ : _ : _	interesting
accurate	_ : _ : _ : _ : _ : _ : _	inaccurate
pleasant	_ : _ : _ : _ : _ : _ : _	unpleasant
unimportant	_ : _ : _ : _ : _ : _ : _	important
useful	_ : _ : _ : _ : _ : _ : _	useless

SECTION B /

SECTION B We would like your opinions (good or bad) about this experiment to enable us to see if we can make improvements. What we want you to do is to place in the boxes beside the statements the letter of the response that most closely corresponds to your opinion, i.e. if you strongly disagree with statement 6 place the letter E in the box.

- A Strongly agree
- B Agree
- C Undecided
- D Disagree
- E Strongly disagree

I think that this experiment:-

1. forced me to organise and plan procedures in advance.
2. helped me to appreciate the limitations of the methods used.
3. made me scrutinise the procedures for possible errors.
4. gave me too much to do in too short a time.
5. allowed me to develop confidence in my practical work.
6. did not give me enough instructions to work from.
7. illustrated practical applications of the lab. course.
8. was boring as there was too much repetition.

SECTION C The following is a list of objectives for this experiment. By the end of this experiment:-

- A. If you felt that you completely mastered the objective then place a tick in column A.
- B. If you felt that you did not quite master the objective and were not completely clear about either the theory or the technique place a tick in column B.
- C. If you felt that you learnt very little about the theory or technique place a tick in column C.
- D. /

D. If you felt that you learnt nothing about the theory or technique place a tick in column D.

1. I can use a single pan balance confidently paying due attention to
  - a) cleanliness
  - b) accuracy.
2. I can weigh out a sample to an accuracy of  $\pm 0.0001$  g.
3. I can use a standard flask accurately to make up a standard solution of a reagent to a specified molarity.
4. I can carry out a titration using a standard solution of an oxidising agent to determine the amount of iron(II) in a sample to the required accuracy ( $\pm 0.1$  cm<sup>3</sup>).
5. I can handle air-sensitive chemicals by protecting them from oxidation using a bunsen valve.

A	B	C	D



## A P P E N D I X 8.5

Matriculation No. \_ \_ \_

Today's experiment was different in many ways from previous experiments in that the responsibility for planning and carrying out the experiment was left to you. We would be very interested in receiving YOUR opinions of this type of experiment. Your answers will be treated in strict confidence but will enable us to plan similar experiments for the future and may enable us to improve present experiments.

Thank you for your co-operation.

Could you please indicate which experiment you did:-

Experiment 1	Phosphates in the River Kelvin	_ _ _
Experiment 2	Determination of Chlorine in the Clyde Estuary	_ _ _

I Comparing this experiment with the 'normal type' of laboratory experiment (working in pairs from a lab. manual after an introductory talk from a demonstrator) indicate in the box provided the letter of the following responses which most accurately represents your own personal opinion or reaction.

- A Strongly agree
- B Agree
- C Undecided
- D Disagree
- E Strongly disagree

I think that this experiment:-

1. allowed me to develop confidence in my practical ability.
2. gave me a lot of freedom to work out procedures for myself.
3. made an interesting change to the usual lab. experiments.
4. let me see that the laboratory course had practical applications.
5. /

5. helped me to appreciate the limitations of practical procedures i.e. sensitivity or accuracy of methods.
6. forced me to organise my practical work, i.e. plan procedures in advance.
7. improved my understanding of previous laboratory experiments.
8. forced me to look for trends in my experimental results.
9. made me think about the experiment before I started any experimental work.
10. made me aware of the importance of carefully recording results.
11. made me scrutinise the procedures for possible errors in method.
12. allowed me to use my knowledge of other subjects to investigate the problem.
13. made me aware of the social implications of my decisions.
14. made me aware that some decisions have to be reached by compromise.

II Tick where appropriate:-

1. How enjoyable did you find this exercise?
  - A Very enjoyable
  - B Quite enjoyable
  - C Average
  - D Unenjoyable
  - E Very unenjoyable
  
2. Would you like to see this type of exercise repeated?
  - A All the time
  - B Frequently
  - C Sometimes
  - D Rarely
  - E Never

## A P P E N D I X 8.6

Demonstrators' Notes Experiment 1 Phosphates in  
the River Kelvin

Organisation - Each pair of students should be able to construct a suitable calibration curve and obtain readings for each of the seven samples. To obtain readings the students will have to extend the calibration curve. However, in two cases the students will have to estimate the upper limit of phosphate concentration.

Procedure - Instructions for constructing the calibration curve are given in the laboratory manual. This experiment is in two parts:- 1) Experimental; 2) Discussion. Begin the discussion section with at least an hour to go - distribute the second sheet.

Laboratory report:- 1) Complete set of results.  
2) Discussion of possible errors  
in procedures.

They will not have time to write up the discussion.

## RESULTS

River Kelvin Sampling Point	Distance from source km	Conc. of orthophosphate mg l <sup>-1</sup>
Near source	2	0.18 ← Dock Burn
Twechar	6	0.63 ← Glazert Water
B 757 Bridge	10.5	0.28 ← Luggie Water
Torrance	14.5	0.39 ← Allander Water
Balmuidy	20	0.48
Dawsholm	25	0.43
Partick Bridge	29.5	0.41

Experiment 1 - Results

From the 'Phosphates in Detergents' experiment it should be known that the main sources of phosphate pollution are agricultural fertilisers and industrial and domestic detergents.

Because /

Because of predominance of sheep and dairy farming in the West of Scotland the pollution caused by fertilisers is negligible in comparison to the other pollution sources - except for the sample at the B 757 bridge where this is the main cause.

Also, pollution due to domestic detergents in the River Kelvin is negligible due to purification plants.

Therefore, the only major source of phosphate pollution is due to the use of detergents in industry which are not purified before reaching the River Kelvin. According to the Clyde River Purification Board, in whose area the River Kelvin lies, the industrial sources are mainly bus depots. The pollution results from the cleaning of buses at the depots of the Greater Glasgow Transport Executive, and W. Alexander & Sons (Midland) Ltd. The sources may be some distance from the actual river but due to burns and various other tributaries the final output is into the Kelvin.

This information will probably have to be given as it is unlikely that the students will discover this by themselves.

Sampling Point	Source of Pollution
Partick Bridge 0.41 mg/L	Maryhill Bus Depot - discharge of vehicle wash water into River Kelvin
Dawsholm 0.43 mg/L	Sources could be:- paper chemical works / heavy industry / gas works
Balmuidy 0.48 mg/L	W. Alexander & Sons - Milngavie - vehicle wash water feeds into Craigdow Burn which feeds into the Allander Water and then the River Kelvin - distance 4-5 miles
Torrance 0.39 mg/L	W. Alexander & Sons - Kirkintilloch - vehicle wash water feeds into Luggie Water and hence into River Kelvin
B 757 Bridge /	

B 757 Bridge 0.28 mg/L	Phosphates on fields being washed into Glazert Water and then River Kelvin
Twechar 0.64 mg/L	Bus Depot - Burnside Industrial Estate, Kilsyth - vehicle wash water feeding into the Dock Water and then the River Kelvin
Near Source 0.18 mg/L	No obvious explanation

NOTE: Pollution sources may be quite a distance from the River Kelvin

### Discussion

If phosphate level is high then the effect on the fish life in the River Kelvin may be serious. Maximum level should be around 0.05 mg/L (USA limit). Although concentrations as low as 0.005 mg/L have been known to promote growth of algae (*microcystis aeruginosa*). The result of a total ban on phosphate pollution at the Burnside Industrial Estate could be as follows:- (to give life in the river a chance to recover)

1) If an alternative 'phosphate-free' detergent is used (sold on market)

Detergent containing phosphates      £1 per gallon

'Phosphate-free' detergent              £50 per gallon

Cleaning 50 buses a day / seven days a week - amount of detergent used is approximately 17 gallons. Then extra cost of using phosphate is £800 a week.

Cost - approximately £41,600 a year.

2) Cost of installing sewers.

To connect to public sewers - nearest main sewer -  $\frac{1}{2}$  mile. Need to cross private land and conceal pipes. Approximate cost £100,000 less 25% government grant.

For every year delay add £25,000 to cover inflation.

Need to obtain planning permission from Local Council - may not be straightforward. Local Council may have to extend /

extend facilities at purification plant - will increase pressure on rates. Even if cost of extending plant shared, Local Council may not be too keen.

Local Council may rent out facilities = £5,000 per annum.

Instal sludge tanks - cost of construction - high as you have to excavate site, build concrete tanks and instal pumps to empty tanks when full. Need to hire firm to empty tanks on regular basis.

Costs:- Construction £50,000

Cost of emptying tanks £500 a month - £6,000 p.a.

Maintenance of tanks and supervisory labour -  
£10,000 per annum (minimum)

Company can -

- 1) spend capital reserve - low - other priorities - new buses, etc.
- 2) borrow money - 10% per annum in interest rate
- 3) cut costs - reduce labour - T.U. - effect on bus service
- 4) raise fares
- 5) mixture of 1) - 4)

Trade unions will not be happy with a cut in the size of the labour force.

Alternative:- treat effect of phosphates on river - control growth of algae - cost uncertain but expensive - boat (or hire) + staff.

Other pollutants:- can also cause damage metal ions, nitrates etc.

## A P P E N D I X 8.7

'Chlorine' Project

Organisation - minimum of six students.

Leave then to divide work load among themselves but if no group leader appears, please ensure that the work is evenly distributed.

The procedure outlined is not too lengthy and therefore it is fair to expect that each student can complete one run to check on the efficiency of the column ( and to familiarise themselves with the method) and to run through two samples. Please check that each sample is going to be checked.

Throughout the afternoon check that the work is progressing. With approximately  $\frac{1}{2}$  hour to go, gather the students together and discuss the results.

Ion-ExchangeProcedure

1. Column - Amberlite 1 RA 400.  
 Regenerate column - pass through approximately 50 ml 5M NaOH - rate 2-3 ml/min.  
 Wash through with distilled water until pH of effluent same as that of the distilled water.  
 To check efficiency of column pass through a sample of 20 ml of 0.1M NaCl
2. Before introducing sample lower the water level until it is about 1 cm above the resin. Pipette 20 ml of sample and allow it to pass through column slowly in conical flask (250 ml).
3. When level of sample solution is within 1 cm of the top of the resin add distilled water and continue to pass through column until effluent is neutral as indicated by litmus paper. 25 - 30 ml of distilled H<sub>2</sub>O is needed.
4. Titrate contents of conical flask with 0.1M H<sub>2</sub>SO<sub>4</sub> acid using phenolphthalein as indicator.  
 Calculate /

Calculate molarity of solution and multiply by the formula weight to give answer in g/l. Assume that density of sea water is approximately one and therefore express answers in g/kg.

Sample No.	Sampling Points	Miles below King George V Bridge	Conc. $\text{Cl}^-$ * g/kg.
1	Upriver of River Kelvin	2	5.30
2	Renfrew	6	7.50
3	Near Erskine Bridge	10	8.80
4	Cardross	16	12.30
5	Port Glasgow (Great Harbour)	20	15.80
6	Gourock	24	18.00

\* Assume density of sea water equals one

2) Accuracy of results:-

- a) density, temperature;
- b) low tide or high tide - high tide chlorinity higher;
- c) time of year;
- d) sources of chlorine in river which feed into the Clyde Estuary
  - human urine 1%
  - rock salt from roads during winter
  - waste from industry as HCl or NaCl.
 Chloride ion is a conservative parameter because it is unaffected by micro-organisms or mild chemical reactors in the river water, so the concentration generally increases with distance from source as shown by the results.
- e) repeat results to reduce margin of uncertainty;
- f) pollution effects.

3) Other methods of determining results:-  
gravimetric

4) Extensions of method to cations - other ions.



## A P P E N D I X 8.8

Experiment 1 - Volumetric AnalysisObjectives

At the end of this experiment you should be able to:-

1. choose the appropriate pieces of apparatus (measuring cylinder, pipette or burette) for measuring volumes of liquids;
2. use a pipette to measure a fixed volume of liquid to an accuracy of  $\pm 0.05$  ml.;
3. use a burette to measure variable volumes of liquid to an accuracy of  $\pm 0.05$  ml.;
- 4.\* carry out titrations confidently and carefully so that the end-points in successive titrations agree to within  $\pm 0.1$  ml.;
5. use the results of titrations to calculate the unknown concentration of a solution from the known concentration of a standard solution.

Introduction

Accurate measurements of volume and mass play a crucial role in modern chemistry. The need for accuracy is obvious whether you are required to estimate the amount of a very expensive material required in an industrial process, or you are to carry out a biochemical assay on which someone's life may depend.

In this experiment you are required to standardise a solution of hydrochloric acid by titration against a standard 0.05 M solution of borax ( $\text{Na}_2\text{B}_4\text{O}_7$ ) and then to use the standardised hydrochloric acid solution to determine the amount of sodium hydroxide in a solution of unknown concentration. (M is the symbol for molarity and is the number of moles of solute in 1 litre of solution.)

Experimental Method Part 1

Carefully rinse the burette and jet with a small amount /

amount of the hydrochloric acid solution and discard the washings. With the aid of a funnel, fill the burette with hydrochloric acid. Ensure that the jet is also filled by discharging a small amount of liquid from the burette. Record the initial burette reading to the nearest 0.05 ml.

Rinse a 25 ml. pipette with a small quantity of the borax solution. Pipette 25 ml of 0.05 M borax solution from a 100 ml beaker into a clean 350 ml flask and add 2 to 3 drops of methyl red indicator until a yellow colour is just observable. Place the flask on a white tile and add hydrochloric acid solution slowly from the burette. After each addition, swirl the liquid in the flask rapidly. At the end-point, the colour of the solution will change from yellow to red.

Record the burette reading at the end-point to the nearest 0.05 ml.

Repeat the titration using another 25 ml of the borax solution. Record your results as follows:-

	<u>Titration 1</u>	<u>Titration 2</u>
Final burette reading (ml)	Y	
Initial burette reading (ml)	X	
	<hr/>	<hr/>
Titre (ml)	Y-X	
	<hr/>	<hr/>

Mean titre =      ml

\* Since the two titrations must agree to within  $\pm 0.1$  ml, it may well be necessary for you to carry out three, four or more titrations until you achieve this objective. It is often worth while to do the first titration quickly in order to obtain an approximate titre, then follow this with at least two very accurate titrations.

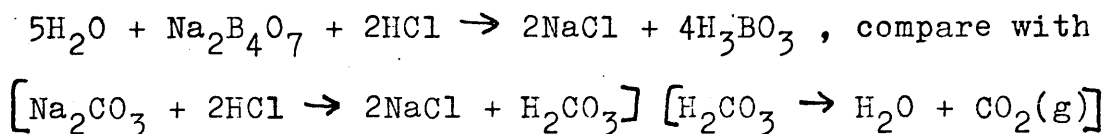
### Experimental Method Part 2

Repeat the above procedure using 25 ml portions of the /

the sodium hydroxide solution of unknown concentration instead of the 0.05 M borax solution again using methyl red as an indicator, until concordant titrations are achieved. Record your results as above.

### Theory and Calculations

Borax, the salt of a very weak acid, has basic properties in aqueous solution and reacts with hydrochloric acid:-



An end-point is reached in this titration because boric acid,  $\text{H}_3\text{BO}_3$ , is a very weak acid (like carbonic acid). Since 1 mole  $\text{Na}_2\text{B}_4\text{O}_7 \equiv 2$  moles HCl and 25 ml 0.05 M borax requires (Y-X) ml of HCl solution, then molarity of HCl solution =  $\frac{25 \times 0.05 \times 2}{(Y-X)}$

Using your results, calculate the molarity of the hydrochloric acid solution. Use this value to determine the molarity of the sodium hydroxide solution.

## A P P E N D I X 8.9

Citric Acid Project - Questionnaire

In today's practical you were given the responsibility for planning and carrying out the experiment. We would be very interested to find out what you thought of this. Your replies will be treated in strict confidence and in no way will it affect your assessment for this lab. course.

Thank you for your co-operation.

Date: . . . . . Matriculation No: . . . . .

## SECTION A

First, a few questions to determine your previous experience in chemistry.

1. What qualifications do you hold in chemistry?  
 . . . . . (O, H, SYS)
2. What year did you leave school? . . . . . (Vth, VIth)
3. How often, after initial instruction from your teacher, were you allowed to conduct an experiment on your own?
  - A) Frequently (Tick where appropriate)
  - B) Quite often
  - C) Seldom
  - D) Never
4. Were you ever encouraged to devise and conduct your own experiment?
  - A) Frequently
  - B) Quite often
  - C) Seldom
  - D) Never
5. How often did you use a pipette at school?
  - A) Very often
  - B) Several times
  - C) Once
  - D) Never

6. /

6. How often did you use a burette at school?
- Very often
  - Several times
  - Once
  - Never
7. When carrying out a titration were you asked to:-
- accept the value of the first readings;
  - repeat the titration until successive readings agreed to within  $\pm 1 \text{ cm}^3$ ;
  - repeat the titration until successive readings agreed to within  $\pm 0.1 \text{ cm}^3$ ;
  - repeat the titration until successive readings agreed to within  $\pm 0.05 \text{ cm}^3$ .

## SECTION B

We would like your opinions (good or bad) about this experiment to enable us to see if we can make improvements. What we want you to do is to place in the boxes beside the statements the letter of the response that most closely corresponds to YOUR opinion i.e. if you strongly disagree with statement 6 place the letter E in the box.

- strongly agree
- agree
- undecided
- disagree
- strongly disagree

I think that this experiment -

- forced me to organise and plan procedures in advance.
- illustrated practical applications of the laboratory course.
- helped me to appreciate the limitations of the methods used.
- did not give me enough instructions to work from.
- allowed me to use my knowledge of other subjects to investigate the problem.
- was boring as there was too much repetition.
- /

7. gave me too much to do in too short a time.
8. gave me confidence in my ability to do molarity calculations.
9. showed me the limitations of using indicators.
10. improved my ability to do titrations to the required standard.
11. made me scrutinise the procedures for possible errors.
12. allowed me to develop confidence in my practical work.
13. was interesting as it applied to real life.
14. forced me to look for trends in my experimental work.

### SECTION C

Finally, tick where appropriate.

1. How enjoyable did you find this exercise?
  - A) Very enjoyable
  - B) Quite enjoyable
  - C) Average
  - D) Unenjoyable
  - E) Very unenjoyable
  
2. Would you like to see this type of exercise repeated?
  - A) All the time
  - B) Frequently
  - C) Sometimes
  - D) Rarely
  - E) Never

## A P P E N D I X 8.10

Experiment 4 - Demonstrators' Notes

As this is the first time that most of the students will have attempted an experiment of this nature many of them will be apprehensive. Therefore, do not allow students to get stuck for too long but gently push them in the right direction. In particular, try and see in the first half hour that everybody has an idea of what they are supposed to be doing and of some of the possible snags that may crop up. However, please insist on the highest standard of techniques and do not allow students to develop any bad habits. Loop cassettes on the use of the pipette and burette will be available in the back room and students should be directed to those if necessary.

Possible points of difficulty

1. Citric acid is weak and tribasic. Although the students are given the formula some may forget this.
2. Indicator changes may be masked by other colours. Suggest quick tests to see which indicator is most useful. Remember however that they are limited to 15 cm<sup>3</sup>. Possible solution to this problem is to dilute the fruit drink. Dilute 5 cm<sup>3</sup> of the concentrate to 50 or 100 cm<sup>3</sup>. The titrations will need to be done at least twice.
3. To achieve greater accuracy they will have to dilute the M NaOH to  $\frac{M}{10}$  NaOH using a standard flask. If any students are attempting to use the M NaOH point out of the advantage of diluting the alkali to give a bigger titre. N.B. No instruction in the use of standard flasks has yet been given. Therefore help will be required to be given.
4. In calculations the M. Wt. to be used is 210, i.e. including water of hydration.
5. Check that both students in any pair are distributing the work evenly - turn about with titrations.
6. It is most important that the students complete the questionnaire when they have finished the experiment and /

and before they leave the laboratory. Could you please then collect the questionnaires, add the date and return them to Mr. A. Wham, Room 243.

7. Marking: If the student has obtained reasonable answers in about three out of the four cases / technique has been good (followed the points shown in the films) / kept within the 15 cm<sup>3</sup> allocation, then award an  $\alpha$ . To avoid discouraging students  $\gamma$  should only be given in extreme cases.

Fruit drink	Titre cm <sup>3</sup> of NaOH	Mol.	gl <sup>-</sup>	Price	In bottle	Val. g/p*	Indicator
Lemon squash	9.3	0.056	11.76	23	8.47 (720 ml)	0.368	bromo-thymol blue cresol red
Orange squash	10.6	0.064	13.4	23	9.74 (725 ml)	0.424	bromo-thymol blue cresol red
Black-currant cordial	3.0	0.020	4.20	25	2.98 (709 ml)	0.119	bromo-thymol blue
Lime cordial	19.2	0.115	26.15	25	1.75 (725 ml)	0.700	bromo-thymol blue cresol red

\* g/p = grams/pence



CHAPTER 9

Discussion

## CHAPTER 9

Discussion

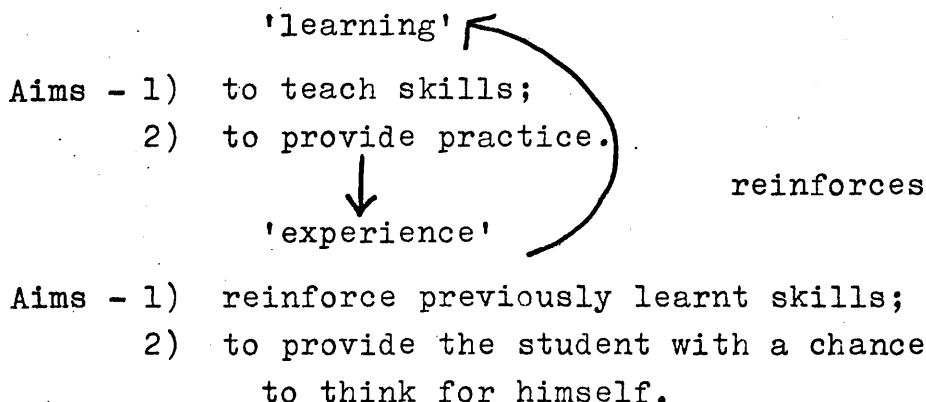
## 9.1 Summary of this Work

Part of the research constituting this work has been into the literature of aims, philosophy, methodology and assessment of practical work in science at the tertiary level. The history of practical work has been traced since its origins in 1805 and the patterns and trends analysed.

After searching the literature and assessing a second year practical course at Glasgow, six criteria were identified which should be met if a laboratory course was to be successful. The courses at Glasgow University were judged against these criteria and scope for improvement was found. Two main criticisms of the course were formulated. These were:-

- 1) not enough emphasis placed on learning; and
- 2) students were not given the chance to think for themselves.

To overcome these criticisms a two-stage model of laboratory instruction was developed.



In the first stage the emphasis is placed on learning and giving the student a chance to practice the techniques, and in the second stage the emphasis is on giving the students a chance to work on their own. It was /

was felt that this second stage would help to reinforce the techniques taught in the first stage and develop the student's ability to think for himself and to organise his practical work. It is important to deliberately design courses to include both learning and experience components otherwise the distinction between the two stages becomes blurred. Unless the experience stage is deliberately built in then it is unlikely to be reached since most experiments use procedures which are highly specified and leave no room for planning and thus are reduced to 'cookbook exercises'.

To increase the effectiveness of the learning stage we looked at three innovations, teaching packages, group participation experiments and pre-laboratory exercises. These innovations were all intended to have an impact on the effectiveness of student learning in the laboratory.

To develop the experience stage two approaches were examined: open-ended experiments and project work.

However, to assess the impact of these innovations and to evaluate present laboratory courses three main techniques were used.

The first of these was self-report techniques where the students were either interviewed or issued with questionnaires. The reliability of the questionnaire approach was found to be poor in some instances although in many cases there was no practical alternative.

An interview schedule based on the proposed laboratory model was devised and used to evaluate the second year organic course. This method was found to be reliable and yielded useful data.

The second method of assessment used was that of paper and pencil tests which sought to assess the student's knowledge of practical procedures. The introduction of this method in a fixed-response format indicated that even simple procedures, such as reading a burette or calculating /

calculating an average titre were still not mastered after a term-long techniques course.

This method, although it assumes a high correlation between the student's knowledge of practical procedures and his performance in the laboratory, was found to be feasible and its introduction would increase the pressure on the student while in the laboratory to learn more effectively.

The final method of assessment, that of direct observation of student performance in the laboratory, was used to assess two instructional films which had been developed on the use of the burette and the pipette. It was felt that the only way to measure the effectiveness of these two films was to analyse the student's performance in the laboratory. An experiment was designed in which the performance of two groups of students, the first group having watched the films and the second having only had a talk from a demonstrator, was recorded on videotape. An analysis was made of their actions using a detailed checklist and the results indicated that using these films was at least as effective as using a demonstrator to give an introductory talk. In addition, the films had the advantage of standardising the approach adopted by the demonstrators and the students benefitted by being taught in a standard manner.

The rest of the thesis examined methods of increasing the effectiveness of learning in the laboratory and ways of giving the students a chance to think for themselves.

Three methods were examined to investigate their potential as possible vehicles for learning in the laboratory.

The first of these concerned the development of teaching packages, in particular two films on the burette and the pipette which were considered as topics which would /

would benefit from a standardised approach. The assessment of these films has previously been discussed.

The second method looked at was that of pre-laboratory exercises which attempted to ensure that all students had reached a minimum competence in their knowledge of the theory and practical procedures involved in a particular experiment. The style of pre-laboratory exercise introduced, true/false multiple completion questions, was found to be easy to administer and did not produce an unacceptable workload for the students. Remedial work was issued to students who showed by their answers that they had not grasped any of the topics covered.

The assessment procedures adopted failed to detect changes in the student attitude to experiments caused by completing these exercises. This was due in part to small sample sizes and also to the fact that changes in attitude might only develop after regular exposure to exercises of this type.

One disadvantage of pre-laboratory exercises gleaned from informal discussions with students and staff was that the benefits of the exercise were sometimes nullified because students rushed through experiments to catch up on the time lost by doing the exercise. Regular use of this method would enable students to prepare in advance for the exercises and this would cut down the time spent on them in the laboratory time.

The last method examined in this section was that of group participation experiments where a tutor acting as a group leader guided the students through an experiment explaining the background theory and demonstrating practical techniques. However, there is considerable scope for interaction within the group as students will help with the preparation of samples, recording of results etc. Although the amount of practice the student gets in 'hands-on' skills is reduced he may benefit from the close scrutiny of his performance by fellow group members.

It was felt that group participation experiments may be of use where the experiment is introducing new or difficult theories, where complicated procedures are being used or where there is a mass of information to collect and process and which might result in students being overwhelmed.

Assessment of experiments run in this fashion indicated that this approach was useful in helping to clarify theories, helping with calculations, in understanding procedures and seeing trends in results. However, the main disadvantages of this approach resulted from the 'mixed-ability' nature of the groups which meant that the pace adopted did not suit everyone, a drawback common to many teaching techniques. It was felt that students may have developed a misplaced confidence in their practical ability since more of them claimed to have mastered the techniques involved with less practice.

The final part of this thesis involved the development of an open-ended experiment and three projects for use in the first year laboratory. These materials were produced to strengthen material already taught and to attempt to achieve 'non-manipulative' goals. These goals include such objectives as to organise and plan a suitable approach, to draw valid conclusions from experimental data, to think independently in a problem-solving situation, etc.

Only a preliminary investigation was carried out into adopting an open-ended approach to experiments but the results were encouraging. In particular the 'experimental' group found the open-ended approach more varied and felt that they had gained in confidence. They also claimed that it forced them to organise and plan the experiment in advance. In addition, more of the experimental group felt that they had mastered the technique in the open-ended part of the experiment.

With a class-size of over five hundred students there was a logistical problem which meant that any attempt /

attempt at free-ranging, individual projects would almost certainly have foundered on the organisational work required. However, we decided to investigate the possibility of mini-projects of three-hour duration.

Each of the projects introduced was successful in strengthening the students' confidence in their practical ability and in providing them with an opportunity to achieve the non-manipulative goals.

The assessment procedures used were reliable as various checks which were made showed that the students had answered consistently.

It is difficult to detect how much of the enthusiasm, initiative and involvement shown by students was due to a novelty effect. However, it cannot be denied that students exhibited a high level of involvement and that the method appeared a powerful tool for using in practical courses. Feedback from staff was also positive and enthusiastic.

Thus we have developed a method for introducing project work, which is aimed at achieving goals often neglected by traditional instruction, at any level in a tertiary course.

It would not be suggested that this approach is adopted universally but it should be built into a course at regular intervals to reinforce previously learnt techniques and to develop non-manipulative goals.

In this thesis a model has been developed which overcomes the criticisms of traditional practical courses by:-

- 1) placing emphasis on the student learning in the laboratory; and
- 2) gives the student an opportunity to think for himself.

Furthermore, the model developed is flexible since it is able to fit into laboratory timetables without creating problems, /

problems, and allows the inclusion of material to meet all the aims of practical work.

It is unlikely that any single laboratory innovation such as a group participation experiment or project work could be used exclusively.

Instead it is suggested that a more flexible approach is adopted by using the method which could most effectively cover the material. The most appropriate method would have to be chosen by experience or trial and error.

For example, for the learning stage of an experiment which involves analysing a mass of data, a group participation approach may be the most useful whereas in teaching manipulative skills the use of films, as instructional aids, may be of most use.

The choice of method will be dependent on the objectives and aims of the material to be covered.

## 9.2 Suggestions for Further Work

It is considered important that further critical appraisal of what practical courses are trying to achieve at all levels is carried out.

In particular, doubt has been cast on the value of using set experiments, with tightly specified procedures, and work needs to be carried out to develop more stimulating alternatives.

There are three main areas in which further research may be useful:-

- 1) in the area of assessment there is scope to refine the present assessment techniques and to develop new methods. In particular:-
  - a) to refine the interview schedule developed for use in the second year organic course;
  - b) /



- b) to examine the effect on student attitude of introducing regular 'paper and pencil' tests.
- 2) to develop new methods for use in the learning stage of the laboratory model and to refine the methods presently used. In particular to examine the use of computers:
- a) to provide self-testing and remedial instruction on background theory to the experiment to be performed;
  - b) to provide opportunities for the student to plan his own experiments;
  - c) to simulate experiments where the collection of data would be too dangerous or time-consuming or too complex to perform in the laboratory.
- 3) to examine the effect of project and open-ended experiments on the students. In particular:
- a) to assess the effect of novelty on the student attitude towards projects and other open-ended work;
  - b) to determine the frequency with which projects should be used;
  - c) to examine the implementation of project work to other years of undergraduate practical work and at the secondary school level.

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