

THE DEVELOPMENT OF AN INVESTIGATIVE LABORATORY PROGRAM  
FOR GRADE ELEVEN CHEMISTRY  
IN MANITOBA

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the Faculty of Education  
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Master of Education

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by  
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Thesis Abstract

THE DEVELOPMENT OF AN INVESTIGATIVE LABORATORY PROGRAM  
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The purpose of this study was to develop an investigative laboratory program suitable for the teaching of grade eleven chemistry in the University Entrance Course for the province of Manitoba. In this study the term investigative was applied to those laboratory experiments which simulated the activities of scientific inquiry within the limits of the high school situation.

The significance of this problem has been reflected in the graduation of high school students with little appreciation of the science of chemistry as an experimental or

investigative method of inquiry because the existing traditional chemistry laboratory program, as it is conducted in most schools, contributes little towards the learning of chemistry as a process of inquiry. The scientific advances of the past decade have demanded an education different in kind; one which is geared to change and which provides for self-direction in living. This modern view of science education diminishes the importance of the particular subject matter employed at the same time that it elevates the spirit of inquiry and the role of the laboratory. From a review of science education literature, it was noted that the following summary reflects the shift in emphasis in the broad aims of science education in the past decade:

Modern course content should:

1. Provide a logical and integrated picture of contemporary science: the theories, models and generalizations that picture the unity of science.
2. Illustrate the diverse processes that are used to produce the conclusions of science and show the limitations of these methods: the ways of inquiry and the structure of scientific knowledge.
3. Enable the student to reach at some point the shadow of the frontier: to experience the meaning of "we just don't know," and to be aware of the progress of science.



From a review of various statements of objectives of the chemistry laboratory nineteen specific objectives emerged as those which reflected the shift in emphasis towards these modern aims of science education. These nineteen specific objectives served to guide the evaluation of the existing traditional laboratory program, and the development and subsequent evaluation of the investigative laboratory program.

Since most traditional experiments are not excursions into the unknown, but are thoroughly described in the text, this type of program robs the student of initiative at the same time that it fosters several undesirable practices in the laboratory--practices which make a mockery of the word experiment and ridicule the painstaking work of the scientist. The need for an improvement in the existing traditional laboratory program was established through an evaluation made by twenty-seven chemistry teachers in the Greater Winnipeg area. Based on the nineteen specific objectives of the modern chemistry laboratory, the teacher ratings for the existing program ranged from four to thirty-five out of a maximum of seventy-six. This evaluation revealed the

failure of the existing program to reflect the change in emphasis in the aims of modern science education towards the spirit of scientific inquiry.

A careful analysis of three laboratory programs, the MCA, CBA, and CHEM Study, which have been developed within the broad framework of the modern aims of science education, revealed that each program contributes greatly to instilling the spirit of scientific inquiry into its students, although each has an approach that is unique. The many new concepts in laboratory teaching which emanated from this analysis were combined with the suggestions of independent writers. From this pool of ideas the following characteristics emerged as the most desirable and the most adaptable to the existing high school situation in grade eleven chemistry.

1. The laboratory and the classroom should be closely integrated through an inductive-deductive approach.

2. The laboratory should provide experiences in the exploration and development of ideas, preferably quantitative in nature and place emphasis on methods as well as results. The evaluation of such experimental results should be made within the limitations of measurement.

3. A proper balance between student investigation and teacher guidance should be maintained throughout the program with the student being led progressively to a point at which his techniques and insight are developed to the extent that, once the problem has been crystallized, he will be able to set up his own experimental procedures.

4. Through a pre-laboratory discussion and the student's pre-laboratory preparation an atmosphere for investigation should be created before the actual laboratory period. Cooperative planning during these discussions should be employed to crystallize the problem and to develop independent procedures.

5. No experiment should be assumed completed until the student, through a post-laboratory discussion led by the teacher; has the opportunity to correlate and interpret the quantitative results of the class as a group. It is here that the student will develop an appreciation for the variations in measurement and for the advantages of group methods of investigation.

6. Student laboratory reports should be functional. In place of the repetition of printed directions, the flow sheet should be employed, since it is both functional and

time-saving. The essay or formal report should be employed only when the student is developing a portion or all of his procedures independently. The student's pre-laboratory preparation should be a truly functional part of every report.

7. A laboratory notebook, similar to the type introduced by the CBA and CHEM Study, should be used in order to facilitate the logical tabulation of data and to encourage graphical analysis. With adequate pre-laboratory preparation the laboratory reports should be completed and the carbon copies turned in at the end of the laboratory period. This type of notebook permits the student to keep a permanent record of his activities and immediately enables the teacher to check the carbon copy for an indication of the student's progress. The procedure outlined encourages greater independence and self-direction at the same time that it reduces opportunity for copying among students.

Experiments, which met the established criteria and which were related to the existing grade eleven chemistry curriculum, were selected from a review of thirty-one laboratory manuals. The experiments that were selected from modern sources were re-written to attain a uniformity of

approach, whereas those experiments selected from traditional sources were re-examined and reconstructed to illustrate not only concepts of chemistry but also processes of scientific inquiry. The resulting laboratory manual was pilot-tested by the grade eleven chemistry students at Churchill High School in Winnipeg during 1962-63 and 1963-64. For the second year of pilot-testing, a complete revision of the laboratory manual was made in order to incorporate the improvements suggested in the first year of trial.

A comparative evaluation of the existing traditional chemistry laboratory program and the investigative laboratory program was made by the investigator. To obtain a valid evaluation, quantitative judgments were made on each individual experiment rather than on each program as a whole. No single experiment was expected to satisfy all of the criteria but a quantitative summation of the judgments on the individual experiments provided a more objective evaluation of each total program than could be obtained through a direct consideration of each program as a whole.

Rating higher in seventeen out of nineteen areas, being surpassed slightly in only the areas of safety habits and techniques development, the investigative laboratory

program, it could be concluded, is more effective in meeting the aims of modern science education than the existing traditional laboratory program and is, therefore, educationally more desirable. Through an evaluation of individual student reports it became evident that students who experienced the investigative experiments were stimulated to critical thinking and as a result of their experiences were made more aware of problems and more proficient in dealing with them.

Although the approach of this investigative laboratory program provides for progressive development in self-direction and disciplined thought, it does not directly provide for totally independent investigations. However, the extent to which this laboratory program can be investigative is limited by the experience and the mental maturity of the grade eleven chemistry student.

## CHAPTER I

### INTRODUCTION

#### I. THE PROBLEM

##### Statement of the Problem

The purpose of this study was to develop an investigative laboratory program suitable for the teaching of grade eleven chemistry in the province of Manitoba, a program which is:

1. consistent with the objectives of science of education.
2. compatible with the University Entrance Course.
3. stimulating to the student.

##### Definition of Terms Employed

In this study the experiments are described as investigative if they simulate the activities of scientific investigation within the limits of the high school situation. Since chemistry is an experimental science, the chemists use the laboratory as a chief source of learning, a similar function should be served by the laboratory in the teaching of chem-

istry. Accordingly, those laboratory experiments which provide for the discovery of ideas and for development in self-direction and in disciplined thought are described as investigative.

Traditional laboratory experiments are those presently performed in secondary schools of Manitoba. Typically, these experiments require the student to start with known facts and principles, and to make observations or perform an activity to verify the predetermined result.

#### Significance of the Problem

Secondary school education has fallen progressively behind the accelerating pace of development in the science of chemistry itself. This lag between the high school classroom and the university laboratory has reached such serious proportions that a pair of writers have recorded the following observation, "While our knowledge of chemistry has been doubling every decade since the nineteen-twenties many of our high school textbooks on chemistry have barely emerged from the nineteenth century."<sup>1</sup> As a result, numerous high

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<sup>1</sup>Arthur H. Livermore and Frederick L. Ferris Jr., "The Chemical Bond Approach Course in the Classroom," The Welch Physics and Chemistry Digest, XLV (No. 1), p. 13.



school graduates have been attending universities or have been completing their formal education with "little appreciation of the science of chemistry as an experimental or investigative method of inquiry."<sup>2</sup>

As the chemistry program is now constituted, up to forty percent of the time allotted to the teaching of grade eleven chemistry in Manitoba high schools has been set aside for the laboratory. The traditional high school laboratory, as it is presently conducted in most schools, in Manitoba and elsewhere in Canada and the United States, contributes little towards the learning of science as a process of inquiry. In many cases the laboratory experiment has degenerated to a mere "cookbook" exercise, which has, in turn, fostered undesirable practices such as, the student being told what to look for, and arriving at the "right" answer by recording observations of "what should have happened" or by rearranging the data. The time devoted to this type of laboratory program cannot be justified educationally because such practices

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<sup>2</sup>Ibid., p. 13.

have no parallel in scientific work nor do they stimulate the student in any profitable manner.

Since 1956, three notable projects have been organized in the United States in order to correct this undesirable situation in the high school chemistry laboratory. This study undertakes to integrate the desirable outcomes of these projects into a grade eleven program suitable for the University Entrance Course in Manitoba.

## II. PROCEDURES

### Establishment of the Objectives of Science Education and an Evaluation of How These Objectives Are Presently Being Met

Any laboratory program should be consistent with the broader aims of science education. A survey of science education literature produced various statements of objectives from which the major aims were selected and organized into a rating form. By rating the extent to which each of the objectives was achieved, twenty-seven high school chemistry teachers from Greater Winnipeg evaluated the existing, traditional laboratory program. This evaluation revealed both its strengths and weaknesses.

A Study of Modern Chemistry Laboratory Programs and The Selection of Criteria for the Investigative Laboratory Program.

The laboratory programs of the Manufacturing Chemists' Association (MCA), the Chemical Bond Approach Project (CBA), and the Chemical Education Materials Study (CHEM Study) have all been developed within the framework of modern objectives. A careful analysis of these programs, including participation in both CBA and CHEM Study summer institutes for teachers, revealed many new concepts in laboratory teaching, such as pre-laboratory preparation and open-ended experiments. Together with these modern concepts in laboratory teaching, the suggestions of independent writers were examined; a selection was then made of those desirable features that could be adapted to local high school conditions.

The Development and Evaluation of Investigative Experiments

Experiments which met the established criteria were selected through a review of thirty-one laboratory manuals. These manuals, which included several first year university manuals for general chemistry, varied in approach from the very traditional to the most modern. The experiments that

were selected from modern sources were re-written to attain a uniformity of approach, whereas those experiments selected from traditional sources were re-examined and reconstructed in terms of modern objectives. During the 1961-62 academic year these experiments were pilot-tested by a total of one hundred and fifty grade eleven students of Churchill High School in Winnipeg. As a result of the evaluation of student performance many of the experiments were revised for further trial during the following year, 1962-63. Through the employment of the rating form by the investigator, the extent to which each of the objectives of laboratory teaching was achieved by each experiment in the existing traditional program and the proposed investigative program was rated. Thus a comparative evaluation of the overall traditional and investigative programs was effected.

### Conclusions

Following a summary of the problem and the procedures, the results obtained in the comparative evaluation of the proposed investigative and the existing traditional laboratory programs are thereupon presented and discussed. The discussion

brings into focus the comparative strengths and weaknesses of the two laboratory programs. The limitations of this study are indicated; and, in an appendix, the product of this study--the investigative laboratory manual is included.

## CHAPTER II

### OBJECTIVES OF THE HIGH SCHOOL CHEMISTRY LABORATORY

#### I. MODERN OBJECTIVES OF SCIENCE EDUCATION

##### Introduction

The objectives of any laboratory program must be consistent with the broader objectives for the teaching of science. The importance of the laboratory program for the success of any science course was strongly supported by the Committee for the Thirty-first Yearbook of the National Society for the Study of Education.<sup>1</sup> The objectives of science education which have been published since then demonstrate an increasing emphasis upon the place of the laboratory in the teaching of high school science.

This increasing emphasis on the laboratory phase of science teaching has reached a climax during the past decade. In order to accommodate the advances in science, its break-

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<sup>1</sup>"A Program for Teaching Science," Thirty-First Yearbook of the National Society for the Study of Education. (Chicago: University of Chicago Press, 1932), pp. 370-88.

throughs and its growing unity, there has come a need for an education different in kind from any previously offered young people in high school.<sup>2</sup> The demands of this modern education have been outlined by Hurd:

Science as a field of study is characterized by a moving frontier and an ever increasing amount of knowledge. Young people need to acquire those skills and abilities which enable them to assume responsibility for expanding their own learning.<sup>3</sup>

In other words, this must be an education that is geared to change and at the same time provides for self-direction in living.<sup>4</sup> Both Schwab<sup>5</sup> and Polykarp<sup>6</sup> support the need of a

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<sup>2</sup>Teachers of modern science courses, such as PSSC and CHEM Study, are required by the Manitoba and Saskatchewan departments of education to attend summer institutes in order to orientate themselves to both the content and the philosophies of these courses.

<sup>3</sup>Paul Hurd, "Summary of Objectives--Rethinking Science Education," Fifty-Ninth Yearbook of the National Society for the Study of Education. (Chicago: University of Chicago Press, 1960), p. 33.

<sup>4</sup>Paul Hurd, "The New Curriculum Movement in Science," The Science Teacher, XXIX (February 1962), pp. 6-7.

<sup>5</sup>Joseph Schwab, "The Teaching of Science as Enquiry," Teaching of Science (Cambridge: Harvard University Press, 1962), pp. 4 et seq.

<sup>6</sup>Jusch Polykarp, "A Nobel Physicist Describes What Today's Student Needs to Know About Science," Science and Maths Weekly Teacher's Edition, II (October 12, 1962), p. 1.

science education which is different from any previously offered. They agree that science should be presented as a continuous process of inquiry. This modern view of science education diminishes the importance of the particular subject matter employed at the same time that it elevates the spirit of inquiry and the role of the laboratory.

#### Objectives of Modern Science Education

In the Fifty-ninth Yearbook of the National Society for the Study of Education,<sup>7</sup> Hurd set forth the following aims of modern science education.

Understanding Science. "There are two major aspects of science teaching; one is knowledge and the other is enterprise." He suggests that science is more than a static collection of assorted facts. To be meaningful these facts must be woven into generalized concepts. While learning about the character of scientific knowledge, how it was developed and how it was used, the student must see its dynamic quality;

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<sup>7</sup>Hurd, op. cit., pp. 33-37.



that is to say, in time this scientific knowledge is quite likely to shift in meaning and status.

Problem solving. "Science is a process in which observations and their interpretations are used to develop new concepts, to extend our understanding of the world. . . and to provide some predictions of the future." Science teaching should help students develop this ability to make careful observations, seek the most reliable data, and then use rational processes, to organize the data and to suggest possible conclusions or further investigations. At higher levels the student should be able not only to establish relationships from his findings but also to predict future observations.

In addition to these major aims of science education, Hurd lists the following objectives:

Social Aspects of Science . . . . A student should understand the relation of basic research to applied research, and the interplay of technological innovations and human affairs . . .

Appreciations. A student with a liberal education in science should be able to appreciate:

1. The importance of science for understanding the modern world.
2. The methods and procedures of science for their value in discovering new knowledge and extending this meaning to previously developed ideas.

3. The men who add to the storehouse of knowledge.
4. The intellectual satisfaction to be gained from the pursuit of science either as a scientist or as a layman.

Attitudes. The knowledge and methods of science are of little importance if there is no disposition to use them appropriately. Openmindedness, a desire for accurate knowledge, confidence in the procedures for seeking knowledge and the expectation that the solution of problems will come through the use of verified knowledge, these are the scientific attitudes.

To understand the scientist is also to understand some of his attitudes, such as the desire to know and to discover, a curiosity about the world, the excitement of discovery and the desire to be creative.

Careers. Science instruction should acquaint students with career possibilities in technical fields and in science teaching. A continuous effort should be made to identify and motivate those who develop special interests. They should be given opportunities for some direct experience of a professional nature, and a perspective of the fields of science.

Abilities. Science as a field of study is characterized by a moving frontier and an ever increasing amount of knowledge. Young people need to acquire those skills and abilities which enable them to assume responsibility for expanding their own learning. Some of these are:

1. Reading and interpreting science writings.
2. Locating authoritative sources of science information.
3. Performing suitable experiments for testing ideas.
4. Using the tools and techniques of science.
5. Recognizing the pertinancy and adequacy of data.
6. Making valid inferences and prediction from data.
7. Recognizing and evaluating assumptions underlying techniques and processes used in problem solving.

8. Using the knowledge of science for responsible social action.
9. Expressing ideas qualitatively and quantitatively.
10. Seeking new relationships and ideas from known facts and concepts.<sup>8</sup>

Similar broad objectives of modern science education were advanced by Schulz, representing the National Science Teachers' Association:

Science as Knowledge

Objective: To develop quality understanding of systematically selected concepts, principles and generalizations of science and an increasing skill in applying them.

Science as a Mode of Inquiry

Objective: To learn to respect the tentative nature of scientific data and conclusions . . . Science students are rarely exhorted to question the present state of scientific knowledge. Every high school science student should have the opportunity to explore at least one conceptual scheme so intimately that he begins to sense the limitations of what we know and observe about natural phenomena . . .

Objective: To develop laboratory and communication skills . . . Learning to know is not the whole of laboratory work; acquiring the ability to feel what the scientist feels is equally important. In the laboratory, the student should be "a scientist for a day." He should encounter the joys and sorrows of experimenting, the elation and the despair. He should come upon the unexpected, run up blind alleys, and work himself out of tight places. He should experience the sights and sounds and smells and emotions of the laboratory. Having had such experiences,

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<sup>8</sup>Ibid.

the student can claim a kinship, however distant, with the scientist and will have an insight into the scientific enterprise which no amount of lecturing can give him . . .

Science classes should also contribute to the development of oral and verbal communication . . . By expecting the students to work at a mathematics level that is consistent with their prior mathematical experiences they should also be brought to recognize and appreciate the importance of quantitative communication in all branches of science.

Objective: To practice critical thinking through problem-solving activities and to recognize the applications and limitations of such procedures in non-science problems . . .

#### Science as Human Endeavor

Objective: To appreciate the interrelationships of science. . . . There is little real appreciation or understanding of the relationships among the sciences or of relationships between science and nonscience activities. The historical and stereotyped boundaries between biology, chemistry and physics disappear at the frontier of science. Above all, youth must be helped to appreciate that science is a human enterprise and that both scientists and nonscientists share a responsibility for using scientific discoveries constructively and humanely . . .

Objective: To explore science for new interests, continually evaluating science experience for career and recreational opportunities.

Objective: To develop hospitable attitudes toward initiative, resourcefulness, imagination, curiosity and creative ability.<sup>9</sup>

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<sup>9</sup>Richard Schulz, "Quality Science for the Senior High School," The Bulletin of the National Association of Secondary School Principals, XXXIV, (December 1960), pp. 77-88.

Teaching the Method (s) of Science

In a report on a survey of forty-two syllabuses for science courses from thirty-seven states, Brandwein points out the general agreement among science educators to teach "the scientific method" in the laboratory. It was found that each one of the forty-two syllabuses proposed this objective.<sup>10</sup> "To cultivate the scientific method of investigation," is listed as one of the objectives of the new University Entrance Course in Manitoba.<sup>11</sup> Despite this widely accepted agreement, there is much controversy as to the concept of "the scientific method" as distinguished from the diverse processes of scientific inquiry which produce the conclusions of science. It would seem that this controversy is responsible for the absence of the term, "the scientific method," from the preceding statements of objectives of modern science education.

Over the last few decades there has developed a point of

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<sup>10</sup>Paul Brandwein, Fletcher G. Watson, and Paul E. Blackwood, Teaching High School Science: A Book of Methods, (New York: Harcourt Brace and Company, 1958), p. 11.

<sup>11</sup>"Excerpts of Interest to Science Teachers From the Initial Report of the University Entrance Course Seminar," The Manitoba Science Teacher, V, (November-December 1963), p. 11.

view that there was "a scientific method" with definite steps to be followed in sequential order. These steps are commonly found in textbooks in the following order:

1. to recognize a problem.
2. to gather relevant data.
3. to formulate a working hypothesis.
4. to test the hypothesis.
5. to accept, modify, or discard the hypothesis.<sup>12</sup>

Consequently, this is the concept of "the scientific method" as presently held by high school students.

Among leading science educators and practicing scientists there are many who will accept the concept of "a scientific method." James B. Conant, an outstanding chemist and educator, points out that the traditional interpretation is not acceptable and advances the term, "tactics and strategies of science" in its place.<sup>13</sup> The scientist does his research in a manner best suited to his particular temperament. Some, who have a scholarly temperament, sort out their ideas and plan their experiments with extreme care before taking a single step in the laboratory; others, who are less patient, but have an equally clear idea of the problem, prefer to attack it directly

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<sup>12</sup>Brandwein, et. al., op. cit., p. 12.

<sup>13</sup>James B. Conant, On Understanding Science (New York: New American Library, Mentor Book, 1951), pp. 17-32.

in the laboratory.<sup>14</sup> Hurd is of the opinion that because the methods employed are so highly flexible, the details of investigation are seldom the same for any two problems. Furthermore, he asserts, "There is no one scientific method; in fact, there are almost as many methods as there are scientists and problems to be solved."<sup>15</sup>

The increasingly popular view in this controversy is described by Elmore, Keeslar, and Parrish:

No one step-by-step procedure in attacking a problem can be singled out as the Scientific Method, although, certain elements of the method have been found to be common to a variety of scientific problem-solving procedures (recognizing and clarifying problems, gathering appropriate information, setting up hypotheses and testing them experimentally if possible, isolating the experimental factor by means of a control, running check experiments, making careful measurements, organizing and interpreting data, drawing suitably qualified conclusions). The number of steps involved and the order in which various elements of methods may occur in any situation will depend on the nature of the problem and the background of experience and insight the problem-solver brings to it.<sup>16</sup>

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<sup>14</sup>Ernest Grunwald and Russell H. Johnsen, Atoms, Molecules and Chemical Change (Englewood Cliffs, N.J.: Prentice-Hall, Inc., 1960), p. 6.

<sup>15</sup>Hurd, Fifty-ninth Yearbook of the NSSE, op. cit., p. 35.

<sup>16</sup>E. Elmore, O. Keeslar, and C. Parrish, "Why Not Try the Problem Approach?" The Science Teacher, XXVIII (December 1961), pp. 32-37.

This point of view is in accord with the opinions of Hurd and Conant.

The widespread adoption of the traditional interpretation of "the scientific method" may be explained by examining the papers in modern scientific journals. Here one typically finds that the reports are similar and logically organized in the presentation of the problem, the hypotheses, the experiment, the data and the conclusions. Robinson suggests that anyone without much personal experience in scientific research tends to assume that such reports reflect the method by which scientific problems are solved.<sup>17</sup> Furthermore, Robinson states that in the writing of such a report, any "steps" of "the scientific method" are easier to find in retrospect. In addition, he writes that it is apparent that the traditional concept of "the scientific method" is the most understandable to the inexperienced teen-aged student.<sup>18</sup> In accord with the above writers, Hurd believes that presenting

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<sup>17</sup>Jack H. Robinson, "How Should We Teach 'The Scientific Method'?" Part I, Science and Maths Weekly--Teacher's Edition, XII (December 5, 1962), p. 2.

<sup>18</sup>Jack H. Robinson, "How Should We Teach 'The Scientific Method'?", Part II, Science and Maths Weekly--Teacher's Edition, (January 16, 1963), pp. 1-2.



problem-solving as a series of logically ordered steps is merely a technique to isolate the critical abilities and skills in order to give them special attention in teaching.<sup>19</sup> The inference seems to be that there is a greater need for the teacher to be conscious of the steps in formal analysis of scientific investigation than for the student to memorize them.<sup>20</sup>

Since there are still so many practicing scientists who are in disagreement over "the scientific method," Robinson advances the theory that educators have been preoccupied with the wrong question: "What is the Scientific method?" He encourages them to pursue a more fruitful question, such as, "What are some of the ways scientific discoveries have been made?" This redirection can be achieved by the repetition of varied experiences in the tactics and strategies used by scientists in solving different problems. Robinson feels that it is this emphasis on various aspects of the question which

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<sup>19</sup>Hurd, Fifty-ninth Yearbook of the NSSE, loc. cit.

<sup>20</sup>L. Mills and P. Dean, Problem Solving Methods in Science Teaching, Science Manpower Project Monographs, (New York: Bureau of Publications, Columbia University, 1960), p. 5.

has led to the adoption of varied terms, such as "critical thinking," "tactics and strategies of science," "problem solving," and "the processes of scientific inquiry," in an attempt to avoid the use of "the scientific method." Through varied experience in the tactics and strategies of science the student will have sufficient background to make his own judgments about the nature of science and its growth. An authoritarian presentation, on the other hand, of a single scientific method is diametrically opposed to one of the great traditions of science.<sup>21</sup>

### Summary

Numerous statements of the objectives of science education have been recorded in scientific literature since 1930. An examination of these statements reveals several similarities to the statements of modern objectives. The change in emphasis in the modern objectives, which would distinguish them from the objectives published previous to this past decade, is reflected in an interpretative summation by Hurd, who states that modern course content should:

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<sup>21</sup>Robinson, loc. cit.

1. Provide a logical and integrated picture of contemporary science, the theories, models and generalizations that picture the unity of science.
2. Illustrate the diverse processes that are used to produce the conclusions of science and to show the limitations of these methods, the ways of inquiry, and the structure of scientific knowledge.
3. Enable the student to reach at some point the shadow of the frontier; to experience the meaning of "we just don't know," and to be aware of the progress of science.<sup>22</sup>

## II. OBJECTIVES OF CHEMISTRY LABORATORY INSTRUCTION

### Objectives of Chemical Education

The role of the laboratory in chemical education cannot be considered separately from the objectives of teaching chemistry and the broader objectives of science education as a whole. The most recent statement of objectives for the teaching of chemistry has been made by the Chemistry Curriculum Revision Committee of Saskatchewan:

It was agreed that the general aim of the matriculation course should be to provide students with a thorough, up-to-date, fundamental course in chemical science which would:

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<sup>22</sup>Hurd, "New Curriculum Movement in Science," loc. cit.

- (a) be a challenge to all students especially those with academic talent.
- (b) acquaint students with scientific methods of study through the use of an experimental approach and through explanation of observations in terms of theoretical principles and models.
- (c) emphasize knowledge of theoretical principles rather than the accumulation of encyclopedic knowledge of apparently unrelated facts.
- (d) encourage a quantitative rather than a qualitative approach to chemical science.
- (e) make students aware of the importance in their lives of chemical science and technology.
- (f) encourage broadening of scientific interests and knowledge by suggesting outside reading and experimentation.
- (g) give students an understanding of chemical science on which further study may be based.<sup>23</sup>

A second Canadian source lists the following as student-teacher goals for any chemistry course:

1. The student should learn CONCEPTS through organized thinking.
2. The student should develop some PROBLEM-SOLVING ABILITY.
3. The student should learn those particular SKILLS which are specific to chemistry laboratory work.
4. Laboratory work should INCREASE OBSERVATIONAL POWERS and call attention to hidden meanings to be discovered in laboratory experimentation.
5. Students should see the PRACTICALITY, versatility, and great future of chemistry; APPRECIATE its impact

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23A. B. Van Cleave, "A New Chemistry Course for Saskatchewan High Schools," Report of the Chemistry Curriculum Revision Committee, Regina (mimeographed) 1962, p. 1.

on national economy, and be, in some cases, influenced to make chemistry a vocational choice.<sup>24</sup>

It must be noted that the preceding statements of the objectives of chemical education reflect the broader goals of science education.

### Objectives of Instruction in the Chemistry Laboratory

Most science educators feel that a science course without a laboratory phase is not worthy of the name, science. Support for this belief has been given in a report by the National Academy of Sciences on the function of the laboratory in high school science:

. . . It is of some significance to conclude from studies on concept development that an effective approach to science education would be to involve the student as much as possible in the procedures followed by a scientist at work to give the student some insight into the methodology of scientific inquiry, of the acquisition and interpretation of data, and of the sense of excitement that comes from discovery.<sup>25</sup>

Hurd proposes a similar role for the laboratory in science education:

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<sup>24</sup>Sister Ernestine Marie, "High School Chemistry Today," The Science Teacher, XXVI (October 1959), p. 426.

<sup>25</sup>Guidelines for Development of Programs in Science Instruction, National Academy of Sciences--National Research Council, Publication 1093 (Washington: Government Printing Office, 1963), p. 8.

Science is based on experiments; and so must be the study of science. The scientists use the laboratory as a primary source of learning; a similar function should be served in the teaching of science. The purpose of laboratory work is that of acquainting students with the processes of inquiry as a means for exploring and developing ideas. The student is concerned with: What questions should be raised? What data are relevant? How should the data be ordered for interpretation? Data known and analyzed, do not give a "conclusion." Theories or models are also needed. They help to synthesize the data, tell whether the experiment meant anything, and describe the conditions which permit predictions.<sup>26</sup>

There is general agreement among science educators that to become aware of what science is like, students require direct experience, with the factors, preferably even unnamed, involved in creating some tentative order in the complex world.<sup>27</sup>

Stollberg sets up the aims of the laboratory program in the following manner:

1. to increase critical thinking.
2. to increase powers of observation.
3. to develop keenness of initiative and versatility of resourcefulness.

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<sup>26</sup>Hurd, op. cit., pp. 8-9.

<sup>27</sup>Fletcher Watson, "The Place of Experiment in Science Education," The Bulletin of the National Association of Secondary School Principals, XXXVII (January 1953), p. 99.

4. to gain deeper insight into the work of a scientist.
5. to acquire improved understanding of basic concepts and principles.
6. to increase proficiency in useful skills such as organizing data.
7. to develop interest and curiosity in science-related areas.<sup>28</sup>

For the laboratory in introductory college chemistry, Sanderson and Bennett propose the following goals:

1. Some familiarity with the general nature and appearance of many elements and compounds, including substances most important in the laboratory.
2. Some experience in measurement of weights and volumes, . . . filtration, fractional distillation, recrystallization, precipitation. . . .
3. An awareness of the direct relationship between principle and practice.
4. An appreciation of the quantitative nature of chemistry.
5. Experience in following instructions, with practice in self-direction.
6. A concept of the limitations of physical measurement.
7. Thorough training in observing and reporting accurately and clearly.
8. An enlightened view of what chemistry is really like.<sup>29</sup>

The chief function of the laboratory, as discussed

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<sup>28</sup>Robert Stollberg, "Learning in the Laboratory," The Bulletin of the National Association of Secondary School Principals, XXXVII (January 1953), p. 100.

<sup>29</sup>R. T. Sanderson and William E. Bennett, A Laboratory Manual for Introduction to Chemistry (New York: John Wiley and Sons, Inc., 1955), preface.

previously, is to provide for the development of skills in scientific problem solving, or inquiry. Several analyses of the problem solving objectives have been published. The following outline is based upon these.<sup>30, 31, 32</sup>

1. Sensing and defining problems
  - (a) Sensing situations involving personal and social problems where scientific skills can be used.
  - (b) Recognizing specific problems in these situations.
  - (c) Stating the problem in concise language.
  - (d) Analyzing the problem into its essential factors.
  
2. Collecting evidence on the problems
  - (a) Learning to recognize valid evidence.
  - (b) Drawing upon past experiences, both personal and those reported in the literature.
  - (c) Isolating elements common in experience and problem.
  - (d) Using experimental procedures suitable to the solution of a problem.

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<sup>30</sup>"Science Education in American Schools," Forty-Sixth Yearbook of the National Society for the Study of Education, (Chicago: University of Chicago Press, 1947), p. 145-147.

<sup>31</sup>Oreon Keeslar, "A Survey of Research Studies Dealing with the Elements of Scientific Method as Objectives of Instruction in Science," Science Education, XXIX (October 1945), pp. 214-216.

<sup>32</sup>L. Mills and P. Dean, Problem Solving Methods in Science Teaching, Science Manpower Monograph, (New York: Bureau of Publications, Columbia University, 1960), pp. 84-86.



- (1) Devising experiments appropriate to the solution of the problem.
  - (a) Deciding upon the evidence which should be collected.
  - (b) Selecting the main factor in the experiment.
  - (c) Allowing only one variable.
  - (d) Setting up controls for the experimental factor.
  - (e) Recognizing the efficiency of both group as well as individual attack.
- (2) Carrying out the details of the experiment.
  - (a) Identifying effects and determining causes.
  - (b) Testing the effects of the experimental factor under varying conditions.
  - (c) Performing the experiment a sufficient number of times to ensure reliable evidence.
  - (d) Determining and recording qualitative and quantitative data.
  - (e) Being aware of the limitations of quantitative data.
  - (f) Generalizing on the basis of the data.
- (3) Manipulating the laboratory equipment needed in solving a problem
  - (a) Selecting the most suitable equipment and materials.
  - (b) Practicing to gain skill in manipulation in order to secure accurate results and to gain an understanding of its function.
  - (c) Appraising scales and divisions of scales on measurement instruments to become aware of their limitations.
  - (d) Avoiding hazards and consequent personal accidents.

- (e) Locating source materials such as handbooks of chemistry and physics.
  - (f) Using source materials.
  - (g) Developing skills in notetaking.
  - (h) Evaluating information pertinent to the problem.
  - (i) Solving mathematical problems necessary in obtaining pertinent data.
  - (j) Making observations suitable for solving a problem.
4. Making the best tentative explanation or hypothesis
- (a) Selecting important factors related to the problem.
  - (b) Identifying the different relationships which may exist between the factors.
  - (c) Recognizing the assumptions which must be made in formulating a hypothesis if one goes beyond the known facts.
5. Selecting the most likely hypothesis
- (a) Analyzing, selecting, and interpreting relevant data.
  - (b) Judging pertinency or significance of data for for the immediate problem.
  - (c) Recognizing and developing logical sequence of data.
  - (d) Recognizing inconsistencies in data.
  - (e) Using resourcefulness in proposing new hypotheses.
6. Testing the hypothesis by experimental means
- (a) Checking hypothesis with recognized authorities.
  - (b) Devising experimental procedures suitable for testing hypothesis.
  - (c) Applying the hypothesis to the problem to determine its adequacy.
  - (d) Making quantitative measurements of experimental results and estimating the probable error of such measurements.
  - (e) Recording the results with strict adherence to standard definitions and usage of scientific terms.
  - (f) Rechecking data for errors in interpretation.

- (g) organizing data in tables and graphs so that it may be analyzed.
- 7. Running check experiments involving the same experimental factor to verify results
  - (a) Study the conditions of the experiment to detect any omissions, defects, or errors, particularly those errors which might have been introduced in the experimental results by coincidence or chance.
  - (b) Recognizing and, if possible, checking further the validity of the assumptions involved in setting up the experiment.
- 8. Accepting or rejecting the hypothesis and testing other hypotheses
- 9. Drawing a conclusion
  - (a) Arriving at a solution to the problem based on an honest, unbiased appraisal of the data.
  - (b) Suspending judgment when results are not conclusive.
  - (c) Calling attention in the conclusion to those basic assumptions which it has been necessary to maintain through the procedure.
- 10. Using the conclusion as a basis for generalizing in terms of similar problem situation

Since not all problems are solved by following all of the above steps, nor by following them in the proposed order, the sequence suggested is to be regarded primarily as one of convenience.

#### Summary of Objectives for Laboratory Instruction

The following outline of specific objectives for chemistry

laboratory instruction is one which incorporates the aims from the literature cited and reflects the recent shift in emphasis towards improved quality in laboratory work.

1. To give the student an opportunity to state the problem in his own words.
2. To provide for adequate student preparation before entering the laboratory.
3. To encourage the formation of hypotheses, or predictions based upon predicting recorded observations.
4. To guide students in the planning and developing of their own procedures.
5. To direct students in selecting the kinds of equipment and materials that they will require, and in improvising apparatus.
6. To provide for the logical organization of recorded data.
7. To provide for training in processing and analysing data by graphing methods and solution of mathematical problems to obtain secondary data.
8. To develop an awareness of the limitations of measurements.

9. To encourage the evaluation of the experimental procedures on the basis of the results obtained; to emphasize methods as well as results.

10. To provide for experience in cooperative planning, evaluation of group data, and in other aspects of "team research."

11. To cultivate good laboratory habits of safety and cleanliness.

12. To develop proper techniques in manipulating chemical apparatus and handling materials.

13. To provide for individual differences among students by encouraging the students to extend an investigation beyond the basic experiment.

14. To develop critical thinking on the part of the student.

15. To frequently employ the investigation of an "unknown" material or chemical system.

16. To indicate practical applications for the procedures followed in the experiment.

17. To require laboratory reports that are functional in that they emphasize communication skills rather than needless repetition of printed instructions.

18. To provide for individual student growth in knowledge, independent thought and self-direction.

19. To give the student an insight into the actualities of scientific investigation.

The objectives listed above, which are consistent with the broad goals of modern science education and the more specific objectives of chemical education, will be employed as the guidelines for further development of the investigative chemistry laboratory program.

## CHAPTER III

### AN EVALUATION OF THE EXISTING LABORATORY PROGRAM IN MANITOBA

#### I. THE TRADITIONAL LABORATORY PROGRAM

##### The Traditional Laboratory Approach

The traditional laboratory programs are those programs that for a number of decades enjoyed widespread acceptance across the continent, but which are gradually being replaced, following the bold steps in chemistry curriculum revision undertaken by the Manufacturing Chemists' Association, the Chemical Bond Approach Project, and the Chemical Education Materials Study. The traditional chemistry laboratory program is described by Blick as being deductive-descriptive in approach since the experiment typically follows the classroom discussion on the topics. The student, therefore, starts with the facts and principles and makes observations or performs experiments to verify what is already known.<sup>1</sup> For each experiment, the

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<sup>1</sup>David Blick, "Purpose and Character of Laboratory Instruction," Journal of Chemical Education, XXXII (May 1955), p. 264.

object, apparatus, procedure and methods for treating data are completely described in the manual. Since the topic has been studied in the classroom, the student can usually read a description of the observations and the conclusions from the text. The only steps which are left for the student to follow are the performing of the activity and the gathering of the data.<sup>2</sup> The main outcome of the traditional laboratory experiences is the development of skills in manipulating apparatus.<sup>3</sup> The existing grade eleven chemistry laboratory program,<sup>4</sup> for the University Entrance Course, is traditional in its approach.

#### Criticism of the Traditional Laboratory Program

Numerous criticisms of the traditional approach to the laboratory may be found in science education literature of

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<sup>2</sup>Milton O. Pella, "The Laboratory and Science Teaching," The Science Teacher, XXVII (September 1961), p. 29.

<sup>3</sup>E. Pierce, Modern High School Chemistry, A Recommended Course of Study, Science Manpower Project Monograph (New York: Bureau of Publications, Columbia University, 1959), p. 5.

<sup>4</sup>Frank Harder, Outline of Laboratory Experiments, Chemistry II.



the last decade. The chief criticism is that this type of laboratory provides little experience in the ways that chemists arrive at knowledge through scientific inquiry. So much emphasis is placed on qualitative exercises such as routine preparations and verification of chemical properties that students are not likely to learn much about the actual work of the chemist.<sup>5</sup> This decided emphasis on the qualitative aspects of chemistry results in an almost complete omission of instruction in proper methods of organizing data and determining of experimental error, at the same time that it permits quantitative verification of physical laws on a single set of inaccurate measurements. As the experiments offer little or no opportunity to discover any of the basic principles of chemistry, they present no challenge because they provide no opportunity or need for individual initiative.<sup>6</sup> Hurd summarizes the objections of many science educators to the traditional laboratory program when he writes:

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<sup>5</sup>E. Pierce, loc. cit.

<sup>6</sup>Harvey Pollack, "The High School Physics Laboratory Approach," Science Teacher Achievement Recognition Program 1958 (Washington: National Science Teachers Association, 1958), p. 44.

An experiment is useless in learning science if the result is known in advance. The traditional school "experiment" set to give predetermined data is unknown to the scientist. An experiment should be an exercise in disciplined thinking, not a routine without chance for error. How can a student develop confidence in his own learning when the unexpected is always a "wrong answer"? If there are no opportunities to question experimental results, no opportunities for critical thinking exist for the learner.<sup>7</sup>

In brief, the chief criticism of the traditional laboratory experiments is that they emphasize techniques which are a part of the tactics of science, but do not emphasize the strategies or processes of science.

In a reference to traditional chemistry laboratory manuals in the United States, Campbell reports that they typically contain blank spaces to be filled in by the students. He further asserts that fifty to ninety per cent of such entries require only perusal of the textbook with no reference to laboratory work.<sup>8</sup> Such an insertion of words into a prefabricated sentence permits the student to do a minimum of thinking, thus further reducing his initiative. The latter

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<sup>7</sup>Paul Hurd, "The New Curriculum Movement in Science," The Science Teacher, XXIX (February 1962), p. 6.

<sup>8</sup>J. A. Campbell, "Chemistry--An Experimental Science," The School Review, LXX (Spring 1962), p. 52.

criticism of the traditional laboratory program is the only one which definitely does not apply to the existing laboratory program for grade eleven chemistry in the University Entrance Course in the province of Manitoba because the essay type of report is employed.

#### Undesirable Practices Resulting From the Traditional Approach

Since most traditional experiment are not excursions into the unknown but are thoroughly described in the text, this program fosters several undesirable practices in the laboratory. Science educators have reported the observation of the blind obedience of students to printed directions,<sup>9</sup> when the students read the laboratory manual and do what it directs, one line at a time.<sup>10</sup> It is because of such observations that the practices of the traditional chemistry laboratory are commonly referred to as "cookbook" chemistry. Other undesirable practices reported in science education

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<sup>9</sup>Howard Nechamkin, "Laboratory Meetings Should Teach Too," Journal of Chemical Education, XXIX (February 1952), p. 531.

<sup>10</sup>R. W. Lefler, "Teaching of Laboratory Work in High School Physics," School Science and Mathematics XXXXVII (June 1947), p. 531.

literature are:

1. The students talk about irrelevant things while the experiment is "cooking."<sup>11</sup>
2. The students record the phenomena that they know should happen rather than what actually does happen.<sup>12</sup>
3. The students often work backward from the expected answer to acceptable data.<sup>13</sup>

Such practices which are outcomes common to the traditional laboratory programs make a mockery of the word "experiment" and ridicule the painstaking work of the scientist.

## II. EVALUATION PROCEDURES

One measure of the effectiveness of the traditional chemistry laboratory program is an evaluation of the extent to which the program is meeting the objectives of modern chemical education. The application of an inventory or check-list of objectives, for the evaluation of a laboratory program,

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<sup>11</sup>Ibid.

<sup>12</sup>Sister Ernestine Marie, "Inductive Teaching at the Secondary Level," Journal of Chemical Education, XXXV (January 1958), p. 46.

<sup>13</sup>Nechamkin, loc. cit.

has been suggested by Mills and Dean.<sup>14</sup>

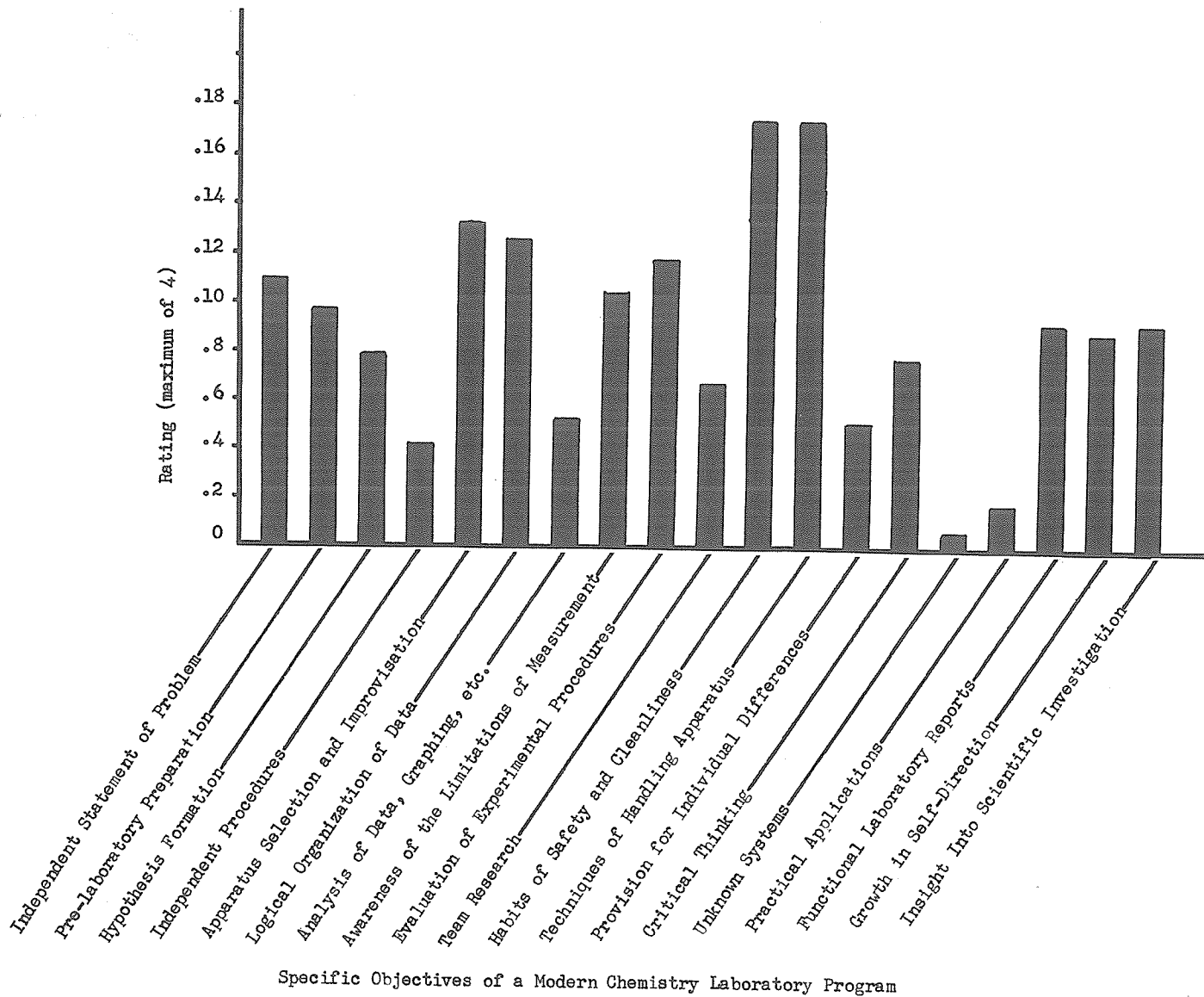
As an outgrowth of Chapter II of this investigation, nineteen specific objectives of a modern chemistry laboratory program were arranged in the form of a check list or rating form. Copies of this rating form were mailed to sixty chemistry teachers in the Greater Winnipeg Area. Rather than checking whether each specific objective was being attained, the teachers rated each objective in its attainment as either excellent (4), very good (3), good (2), fair (1), or poor (0).<sup>15</sup> Hence, every specific objective had a maximum rating of four, thus providing a maximum total for the program of seventy-six. A compilation was made of the data contained in the twenty-seven forms that were returned.<sup>16</sup> This compilation indicates that the range in rating the total program was from four to thirty-five. A summary of this data in Figure 1 reveals the assets of the existing traditional

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<sup>14</sup>L. Mills and P. Dean, "Problem Solving Methods in Science Today," Science Manpower Project Monograph, (New York: Bureau of Publications, Columbia University, 1960), p. 84.

<sup>15</sup>infra. Appendix A.

<sup>16</sup>infra. Appendix A.



Specific Objectives of a Modern Chemistry Laboratory Program

FIGURE 1

SUMMARY OF AN EVALUATION OF THE EXISTING TRADITIONAL  
LABORATORY PROGRAM IN MANITOBA

laboratory are the development of habits of safety, and proper techniques in the handling of apparatus. At the same time, the summary points out the specific areas of the program that, according to the objectives of modern chemical education, are weak. These areas of weakness include, (1) the employment of unknown systems for investigation, (2) the indication of practical application for the procedures to be followed, (3) the provision for individual differences by encouraging the student to extend an investigation beyond the basic experiment, (4) the encouragement of hypothesis formation, (5) provision for direction in planning and developing independent procedures, (6) the provision for training in processing and analyzing data, and (7) the employment of group techniques for investigation.

### III. CONCLUSIONS

Although the maximum rating for the existing laboratory program was seventy-six, no chemistry teacher gave it a rating above thirty-five; the mean rating being sixteen. Thus it would appear that the existing, traditional chemistry laboratory program does not meet the objectives of modern chemical education. The shift in emphasis in the objectives of the

chemistry program during the last decade is not reflected in the existing traditional program since the weaknesses include the failure, (1) to employ the investigation of unknown systems, (2) to give instruction on the processing and analyzing of data, and (3) to provide the opportunities for the development of independent procedures.

Further in support of the conclusion that the existing traditional laboratory program does not meet the objectives of modern science education, the Chemistry Curriculum Revision Committee for the province of Manitoba has recommended the adoption of the CHEM Study Program for grade eleven chemistry in the University Entrance Course.<sup>17</sup>

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<sup>17</sup>Sam Doctoroff, "The Latest Developments in Manitoba Curricula Changes," The Manitoba Science Teacher, V (March-April 1964), p. 41.



## CHAPTER IV

### RECENT LABORATORY PROGRAMS

#### I. INTRODUCTION

Never in the History of American education have so much talent, energy and money been invested in improving science education as in the latter half of the last decade. A notable feature of this movement has been the extensive involvement of practising scientists. After long years of indifference to problems of education, many of them have come out of their laboratories, and personally or through their professional societies, have voiced their interest and have shown their willingness to assume leadership. Practising scientists in many universities now give time and energy in order to sit on committees concerned with professional preparation of teachers and on those dealing with curriculum studies and revision. Although some programs for curriculum improvement have been initiated and supported by private foundations and industry, the majority of the support has come from the United States government through the National

Science Foundation.<sup>1</sup>

As a result of the interest shown in improving science education, many projects in course-content improvement have been undertaken in recent years in all areas of high school science education. It would be unrealistic to assume that all this curriculum revision is unified; however, there are at least two common features which give a new direction in science teaching: the provision of a logical and integrated picture of contemporary science, and the illustration of the ways of scientific inquiry and the limitations of such methods. With inquiry and generalizations as goals of science teaching, the laboratory phase has become a more intimate part of the total learning activity that has been characteristic of previous science programs. In the area of high school chemistry three such independent projects have made outstanding contributions to teaching chemistry from a chemist's point of view. These projects are:

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<sup>1</sup>Frederick Jackson, "Key Role of Scientists in Curriculum Change," Science Education News, The American Association for the Advancement of Science, December 1961, p. 24.

1. The Scientific Chemistry Experiments of the Manufacturing Chemists' Association (MCA).
2. The Chemical Bond Approach (CBA) Project.
3. The Chemical Education Materials (CHEM) Study.

## II. THE MANUFACTURING CHEMISTS' ASSOCIATION

### Background and Planning

The Manufacturing Chemists' Association aid-to-education program was officially started in December of 1956. At the initial conference, four of the leading science educators in the United States reviewed the status of high school chemistry. Deciding that one of the major weaknesses was found in the use of laboratory time, the group suggested that chemistry laboratory work would be more fruitful, especially for able students, if more challenge were built into the experiments. "Open-ended" experiments which could be carried out only through true methods of scientific investigations were recommended. The stated specific objectives of the MCA Program were:

1. To help teach the principles of chemistry and their application to industry, agriculture, and everyday living.

2. To make the high-school chemistry laboratory a highly interesting and challenging experience.
3. To capture or to develop further the enthusiasm of able students for careers in science.
4. To foster intellectual activity by posing questions to be answered through laboratory experiences.<sup>2</sup>

The first steps to bring the spirit of scientific inquiry into the high school laboratory were taken when a review committee consisting of nine chemistry teachers from the Northeastern United States approved a plan to prepare the experiments, to test them and to distribute them to the schools. Procedures for the open-ended experiments were written by chemistry teachers from across the United States. After it was edited, each set of directions, together with the accompanying information for the teacher, was sent out to at least five different schools for trial and possible improvement.

#### Progress and Present Status

By 1958, thirty-one open-ended experiments had been

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<sup>2</sup>Charles L. Koelsche, "The Course in Chemistry," The Bulletin of the National Association of Secondary School Principals, XXXIV (December 1960), p. 110.

compiled by the MCA, and distributed without charge in classroom quantities to teachers of chemistry. By August 1959, more than half of all the American high schools offering chemistry had requested enough copies to equip at least one laboratory class in each school. Requests were also received from fifteen foreign countries.<sup>3</sup>

The Manufacturing Chemists' Association's aid-to-education program, the primary purpose of which is to conceive, develop and test new ideas useful in science education, gave Holt, Rinehart and Winston, Incorporated, the rights to continue the publication of the experiments in 1959. Since that time the MCA staff has organized an additional group of thirty experiments, to provide a laboratory program of open-ended experiments that could fulfill the requirements of a full year's course.<sup>4</sup>

#### The Laboratory Program

The open-ended experiment. These experiments are

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<sup>3</sup>MCA Staff, Scientific Experiments in Chemistry--Teacher Book, (New York: Holt, Rinehart and Winston, Inc., 1962), p. iii.

<sup>4</sup>Ibid.

distinctive in that students cannot anticipate the observations or conclusions beforehand. Although purposes and procedures are described clearly, the results, which are omitted from the student guide, can be obtained only through direct laboratory experience. In addition, on the basis of his laboratory experience, the student is asked to make predictions and then verify or disprove them. In order to answer the question, "How do oxides behave in water?", the student records careful observations on several oxides. He then combines his observations with those of other class members. From these observations the student is required to search for some pattern by referring to the periodic table, the activity series and the heats of formation of the compounds involved. Once the pattern or conclusion is established, it is then verified or disproved by prediction of the behavior of an oxide which was not previously studied; the prediction is then tested.<sup>5</sup> Since the results of such open-ended experiments are not necessarily uniform throughout the class, the teacher and student becomes prepared for the unexpected. Sometimes unexpected results can stimulate

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<sup>5</sup>MCA Staff, Scientific Experiments in Chemistry (New York: Holt, Rinehart and Winston, Inc., 1962), pp. 14-15.

independent projects and fruitful investigation. Open-ended experiments such as these may be described as research problems in miniature.

Practical applications. At the end of each experiment, the principles or techniques, which are involved in arriving at a conclusion, are related to real-life situations; therefore, the "What's it good for?" question has many interesting answers for the student.

Student reports. Each student is asked to make a report on the experiment in his own manner, and to include evidence for his conclusions. The requirements are not rigorously prescribed, but the report to the teacher should resemble the report of a research chemist to the group leader. The report need not repeat the directions in the student guide, but it should show that the student understands the purpose and the method of the experiment. The data collected from the experiment should be recorded as part of the report; this information is then used to arrive at a conclusion. In general, the report should answer what the student has learned and should contain the supporting evidence for his conclusions.<sup>6</sup>

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<sup>6</sup>MCA Staff, Scientific Experiments in Chemistry - Teacher Book, p. 20.

Teacher's guide. A companion Teacher Book has been designed for chemistry teachers with limited academic preparation or experience. This volume contains suggestions for the utilization of the experiments and the precautions to be taken in carrying out difficult experiments. Samples of typical student results from a trial class are also included. Owing to the nature of open-ended experiments the Teacher Book is an invaluable guide for the average chemistry teacher.

Flexibility and adaptability. In spite of the fact that the MCA experiments have not been written for any specific course they can be adapted to any course in high school chemistry. Although many of the experiments are designed to be performed individually by the student, some are particularly adaptable to group work so that different groups within a class can gather data on different aspects of the same problem. The results are then tabulated and compared, so that a class answer is reached. By following such an approach the class functions as a research team. These experiments have also been adapted for the use as projects for clubs and informal

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<sup>7</sup>Koelsche, loc. cit.



groups of students. Regardless of the way that the MCA experiments are fitted into a chemistry program, they are found to be effective not only in illustrating some of the processes of scientific inquiry, but also in challenging the student to further investigation.

## II. THE CHEMICAL BOND APPROACH PROJECT

### History and Organization

The Chemical Bond Approach (CBA) Project originated in 1957 when a group of college and high school chemistry teachers met for a two week conference at Reed College in Portland, Oregon. The following objectives were proposed for chemistry courses in high school and first year college:

1. To present the basic principles of chemistry as an intellectual discipline and to achieve an appreciation of chemistry as a creative pursuit of human knowledge.
2. To develop facility in analytical, critical thinking - especially thinking which involves logical and quantitative relationships.
3. To develop scientifically literate citizens through an understanding of, (a) methods of science, and (b) the role of chemistry in society and in everyday living.
4. To stimulate interest in chemistry, to identify promising students, and to provide adequate

preparation for further scientific studies.<sup>8</sup>

It was recognized that these objectives could be attained by more than one method of presentation, but the conference concluded that chemistry could be taught better if the course had a central theme.

Following a similar conference in the summer of 1958 at Wesleyan University in Connecticut, a group of high school and college teachers agreed to try out experimentally a course based on the chemical bond as a central theme. With this theme throughout the text and laboratory program, most of the topics normally included in high school texts were to be developed, "not as individual entities but rather, as interconnected threads in the total fabric of chemistry."<sup>9</sup>

The National Science Foundation began to support the program in 1959 and has continued to do so. In the summer of 1959, a writing conference, in which nine high school and nine college teachers prepared the first text and laboratory manuals, was held. Beginning in the fall, evaluation was made through

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<sup>8</sup>"Reed College Conference on the Teaching of Chemistry," Journal of Chemical Education, XXXV (January 1958), p. 54.

<sup>9</sup>"CBA Summer Institutes" (descriptive brochure), January, 1962, p. 1.

weekly reports sent in by teachers on the text materials and student laboratory notes on each experiment, visits to schools by CBA staff members, three series of regional meetings from small groups of teachers, and a testing program. The text and laboratory manual have been revised continuously to reflect the experience of teachers employing them.<sup>10</sup>

#### Progress and Present Status

Since the initial writing conference, the texts and laboratory manual have undergone three trial revisions. Publication of the first commercial edition of the text, "Chemical Systems," has been completed in early 1964 by the McGraw Hill Book Company. Interest and participation in the project have grown steadily as a result of the National Science Foundation Summer Institutes which provided chemistry teachers with a background appropriate to teaching CBA Chemistry. Table 1 indicates the increasing participation in the program over a three year period.

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<sup>10</sup>Ibid.

TABLE I  
ENROLLMENT IN THE CHEMICAL BOND APPROACH  
COURSE SINCE 1959<sup>11</sup>

Academic year	Teachers (No.)	Students (No.)
1959-60	10	850
1960-61	83	5,500
1961-62	200	10,000

#### The Laboratory Program

Intent and approach. The CBA Project staff feels that the best way to give a student an appreciation of chemistry is to get him to perform the functions characteristic of chemists, for only then will he see chemistry as a powerful process for uncovering and extending natural phenomena. Towards this end the organization of the CBA laboratory program has been developed to aid the student in his study of the interplay of concepts with observations and experiment. The student not only collects data in the laboratory but also applies ideas to his data. The

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<sup>11</sup>Arthur H. Livermore, and Frederick L. Ferris Jr., "The Chemical Bond Approach Course in the Classroom," The Welch Physics and Chemistry, XIV, p. 15.

data are fitted into a logical scheme based on a set of assumptions and often some mental model. Such logical reasoning leads to a reasonable solution to the problem. Because the experiments do not lead to a predetermined result, it is this ability to follow and even to construct a line of argument that is the real indication of good student work. The importance of reproducible quantitative work is not minimized, but it is not made a goal in itself. The treatment of experimentation in the text gives sufficient leeway to allow the student to interpret his own experimental data without being forced to accept conclusions which may inconsistent with the data. No unusual observation is considered wrong if the student makes it honestly, CBA takes a scientific attitude to unusual results and encourages students to seek out reasons for such results. This type of laboratory is truly research-centered.<sup>12</sup>

Student Introduction to the laboratory. The student becomes aware of the emphasis on the interplay between ideas,

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<sup>12</sup>"Chemical Bond Approach Summer Institutes," (descriptive brochure), January 1962.

observation and experiment in the very first experiment he encounters. This is the "black box" experiment<sup>13</sup> in which each student is given a sealed box containing an unknown object and is asked to develop a mental model of the object. His report is judged not on his ability to guess the contents of the box correctly, but on the way he organizes the evidence that he has collected through observation and inference. Though seemingly far-fetched, this experiment gives the student an appreciation of the problems involved in constructing a mental model of atoms and molecules.

Organization of experiments into groups of increasing difficulty. Various conceptual themes such as stoichiometry, and the relationship between structure and properties, are developed gradually in the laboratory through the design of experiments which become progressively more complex as the year progresses. The forty-one experiments are divided into three groups of increasing difficulty. Group I experiments have been selected not only to provide experimental information

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<sup>13</sup>CBA Laboratory Development Committee, Chemistry Laboratory (third rev.) (Lebanon, Pennsylvania: Wilt Offset, 1961), p. 1.

related to the introductory material in the text but also to introduce the student to many of the basic laboratory techniques which he will use throughout the course. It is important to note that the learning of manipulations is not treated as an end in itself, but also as an incidental step to the solution of a particular problem. For example, an introductory experiment which is used to illustrate precipitation and filtration techniques requires the student to find out which of the two reacting solutions is in excess.<sup>14</sup> Hence this experiment becomes an investigation rather than merely a manipulative exercise. These Group I experiments are designed to provide sufficient information to enable the student to solve for himself the problems posed. It is here that the student gains confidence in his ability to obtain meaningful results in the laboratory.<sup>15</sup>

The experiments included in Group II involve the investigation of various aspects of specific materials presented in

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<sup>14</sup>CBA Laboratory Development Committee, Chemistry Laboratory, pp. 10-12.

<sup>15</sup>CBA Laboratory Development Committee, Teacher's Guide to the Chemistry Laboratory, (Annville, Pennsylvania: CBA Laboratory Development Center, 1962), pp. 16-17.

the text. The assistance which was previously given to the student has been withdrawn so that the student now has the opportunity to suggest an experimental procedure to be used in the investigation. A brief introduction in the laboratory manual and the pre-laboratory discussion provides the student with a start in a useful direction. Typical laboratory directions in this level of experiment are:

Identify each of the substances contained in the six test tubes assigned to you. Although you will be given the specific names of these substances, the names will not be associated with the specific numbers on the test tubes assigned to you. The problem is to be solved by using only the substances contained in the test tubes. This can be done by mixing a portion of two of the substances and comparing the resulting observations with the characteristics of the various possible products reported in the literature or given in the text.<sup>16</sup>

Prior to the experiment the student must study the characteristics of the various substances assigned and organize an original flow sheet for qualitative analysis. With such experiences the student is made to feel that it is within his power to learn facts and to originate ideas.

A number of more complex, research-centered problems, which require investigation over a number of laboratory periods

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<sup>16</sup>CBA Laboratory Development Committee, Chemistry Laboratory, p. 43.



make up Group III. Here, the problems posed for investigation involve chemical systems in which several factors contribute to the general properties of the system. A typical problem is posed in this way:

Determine the order of bond strengths of AX, AY, and AZ, using data obtained from the aqueous solutions of the ionic compounds, designated AB, CX, CY, and CE, assigned to you.<sup>17</sup>

No outline of procedure is given. It is left to the student to decide what information he needs to solve the problem.

Ideally, some information should come from the laboratory and some from the literature. The student is asked to submit the design of his procedure for approval and then to perform all the required steps independently, employing the ideas and techniques accumulated in the process of investigating the other problems in Groups I and II. The entire sequence of experiments is so designed to provide the necessary background for such research-centered problems as found in Group III.

Extensions. Prominent in the philosophy of CBA chemistry, students are encouraged, wherever possible, to extend their

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<sup>17</sup>Ibid., p. 53.

investigation beyond the immediate experiment and try to collect experimental data to answer problems which they have raised. Therefore, the laboratory is used as a convenient and useful springboard for converting student questions into an experiment rather than giving the student a direct answer. Several suggested extensions are given for most experiments. These extensions are found to be most useful in providing for the needs of the gifted student who will complete the basic experiment before the majority of the members of the class do.

Pre-laboratory and post-laboratory discussion. A pre-laboratory discussion, preceding the laboratory period by at least one day, allows the teacher to set the scene for the laboratory work which follows. Here, the students will learn why they are doing a particular experiment and also with guided questions from the teacher, members of the class may design the procedure for a Group II experiment through group discussion. They may also receive a start in a useful direction for designing a procedure individually. To make the pre-laboratory discussion of real value, the teacher must be willing to allow the students to try their proposed procedures as long as this can be done safely, even though he may feel

sure they will not work. The skill of the teacher in leading the discussion of the proposed work will determine to a large extent the attitude of the student upon entering the laboratory and will be reflected in the caliber of the work accomplished. Also vital to the success of the laboratory is the post-laboratory discussion. Its chief advantage is to provide an opportunity to correlate and interpret the results obtained by the students.<sup>18</sup>

The laboratory notebook. Laboratory notes are kept in a blank notebook consisting of one-quarter inch graph paper with alternate white and yellow sheets. The original notes written in the laboratory during the experiment are made on white sheets and a carbon copy on the yellow sheets. The carbon copies are torn out and handed in at the end of the laboratory period for checking by the teacher. The student keeps his original record. During the testing program, these carbon copies were sent to the project headquarters for evaluation and revision of experiments.

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<sup>18</sup>CBA Laboratory Development Committee, Teacher's Guide to the Chemistry Laboratory, p. I-9.

The requirements of these laboratory reports are that they be concise but complete enough so that they can be read and understood by others. Each report is expected to include the following:

1. A statement of the problem being investigated.
2. An account of all procedures which are not already described in the Student's Guide . . .
3. A detailed record of observations.
4. A precise record of weights and measures.
5. A graphical or tabular presentation of data when appropriate.
6. Generalizations or conclusions based on actual laboratory observations.<sup>19</sup>

For some experiments the report might contain such additional information as the appropriate calculation of secondary data, a treatment of errors, equations for reactions, and suggestions for further laboratory work.

To facilitate the recording of experimental information, a flow sheet is employed. A flow sheet is a graphical representation of procedures to be followed, the observations noted,

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<sup>19</sup>Ibid., p. iv.

and a record of the data collected while the student is actually conducting an investigation in the laboratory. The time spent in the organization of the flow sheet as a pre-laboratory assignment will ensure that the student understands the procedures to be followed in the laboratory where he will find it a useful reference as the experiment proceeds and a convenient means of recording observations as they are made.<sup>20</sup>

Teacher's guide book. Because the experiments are truly investigative, or research-centered, the students will often arrive at results which are not anticipated by the teacher. To prepare the teacher with an average academic preparation in chemistry for such an atmosphere, the accompanying teacher's guide to the laboratory contains 354 pages of essential background information. Theoretical discussions of each experiment, background information, suggestions on possible pre-laboratory and post-laboratory discussions, information regarding the general philosophy of the program, and the intended progressive development of the experimental techniques and

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<sup>20</sup>CBA Laboratory Development Committee, Chemistry Laboratory, pp. A-13 to A-16.

The Reaction of Sodium Chloride and Silver Nitrate  
To Determine Which Solution Is in Excess

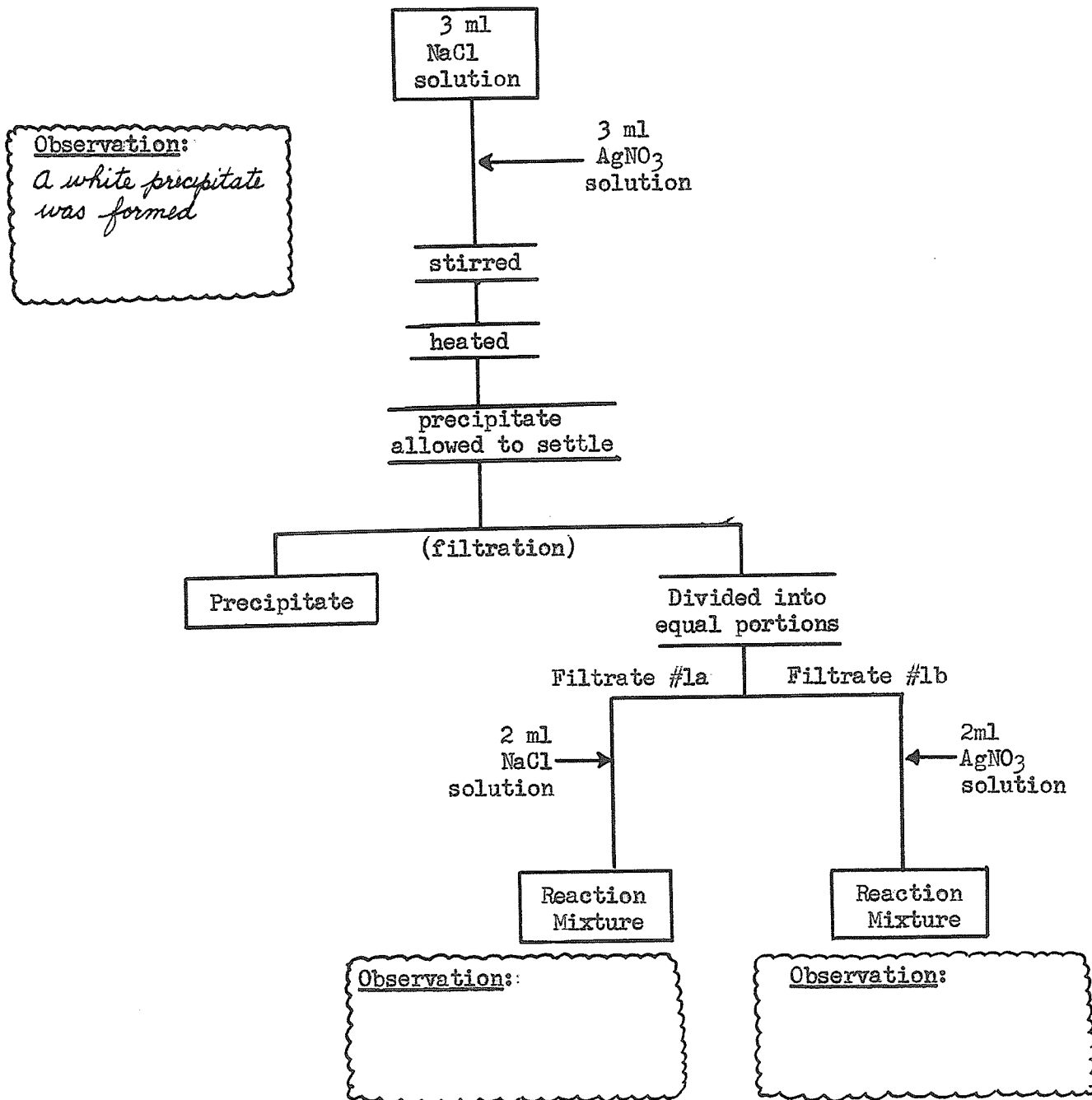


FIGURE 2

STUDENT FLOW SHEET OF EXPERIMENTAL PROCEDURE

concepts are all contained in teacher's guide for the laboratory.

Correlation between text and laboratory program. The laboratory program and the text parallel one another in terms of topics and reinforce one another considerably. In some cases direct use of the student's experimental data as a part of the text development are effective, but this order of presentation is not essential to the development of student understanding. Integrating the laboratory and text in this manner permits a flexible use of the laboratory program with either inductive or deductive development. This dual approach reflects the ways in which science develops.<sup>21</sup>

#### IV. THE CHEMICAL EDUCATION MATERIALS STUDY

##### History and Organization

The Chemical Education Materials Study, (CHEM Study), grew out of suggestions by a committee appointed in 1958 by the American Chemical Society to study the possibility of

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<sup>21</sup>Earle S. Scott, "The Necessity for Variety," CBA Newsletter, May 1962, pp. 1-2.

revising high school chemistry. This committee recommended a revision and suggested in general what the course should contain. In 1960 a grant was obtained from the National Science Foundation and a steering committee of college professors and high school teachers was assembled to investigate what could be done to produce the most effective high school course possible. The following specific objectives were proposed:

1. To diminish the current separation between scientists and teachers in the understanding of chemical science.
2. To stimulate and prepare those students whose purpose it is continue the study of chemistry at a university.
3. To encourage teachers to undertake further study of chemistry courses that are geared to keep pace with advancing scientific frontiers and thereby improve their teaching methods.
4. To further in those students who will not continue the study of chemistry after high school an understanding of the importance of science in current and future human activities.<sup>22</sup>

The steering committee set out to identify the irreducible minimum of fundamentals that could be taught in a high school course.

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<sup>22</sup>A. B. Van Cleave, "A New Chemistry Course for Saskatchewan High Schools," (mimeographed), Regina 1963, p. 2.



In the summer of 1960 a group of nine college professors and nine high school teachers met at Harvey Mudd College in California for a writing conference. As a result of their labors, a textbook and a laboratory manual were prepared. Later in the summer, these materials were studied by twenty-three teachers selected mainly from the California area. During the 1960-61 academic year, twenty-four high schools used CHEM Study materials, with thirteen hundred students involved in the trial. Through weekly meetings, where problems and changes were discussed, close contact was maintained between the staff of CHEM Study and the teachers. The reported experience of the teachers became the basis for the revision incorporated in the Second Trial Edition and the Teacher's Guide developed to accompany it.<sup>23</sup>

#### Progress and Present Status

During 1961-62 the original twenty-four teachers were joined by about one hundred more who had completed a period of preparation in the National Science Foundation Summer

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<sup>23</sup>L. Sibling, "The CHEM Study Approach to Introductory Chemistry," School Science and Maths, LXI (February 1961), p. 114.

Institutes. Over twelve thousand students were involved in the second year tryout. The third trial edition of the text and laboratory materials was exposed to about forty-five thousand students in five hundred and sixty high schools. With the addition of these numbers, the evaluating facilities were expanded and six new centers were established to facilitate the compilation of progress reports from participating teachers.<sup>24</sup> The hardbound edition of the CHEM Study textbook, "Chemistry: An Experimental Science" and its accompanying laboratory manual and teacher's guide were published in the summer of 1963 by the W. H. Freeman Company. It has been estimated that over one hundred and ten thousand students were using CHEM Study Materials during 1963-64. This figure constitutes between ten and fifteen per cent of the United States high school chemistry students during this academic year.<sup>25</sup>

#### The Laboratory Program

Intent and purpose. The CHEM Study approach is based upon the philosophy that the high school laboratory should be

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<sup>24</sup>CHEM Study Newsletter, October 1962, pp. 1-2.

<sup>25</sup>CHEM Study Newsletter, February 1964, p. 4.

a place where the student makes and records careful observations on a system, seeks patterns or regularities in what he observes, and then develops tentative explanations or mental models to rationalize his observations. The forty-four experiments are quite specific in terms of procedure, but open-ended as to expected results and interpretation. Students are led to discover many generalizations for themselves. "It is hoped that, as well as learning chemistry, students will experience the thrill of discovery," writes Merrill,<sup>26</sup> and learn first hand how scientific enterprise moves forward."

Uncertainties of measurement. Because many of the fundamental concepts developed in the course are quantitative, many of the experiments are quantitative also. An important area of emphasis in the CHEM Study laboratory is that of the uncertainty of scientific measurements and the dynamic aspect of science. The student learns three common methods of expressing the uncertainties of measurement and the advantages of

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<sup>26</sup>Richard L. Merrill, "Chemistry--An Experimental Science," The Science Teacher, XXX (April 1963), pp. 26-27.

using each method. This variability in measurements is clearly illustrated to the student by the pooling of class results to obtain averages, deviations and graphs of group data. When the result obtained by each student represents a point on a graph, critical evaluation of results follows readily. The student begins to understand and appreciate the limits of accuracy in measurement and, therefore, is made cognizant of the tentative nature of the conclusions based upon these measurements.<sup>27</sup>

Student introduction to the laboratory. After the first week at school the student realizes that the title of the course is not misleading; chemistry is indeed an experimental science. From the very first day, almost every period is spent in the laboratory and the student is not given the text-book until after the third day of school. The very first experiment requires the student to make and record as many observations as he can of a seemingly simple, yet deceptively

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<sup>27</sup>A. B. Van Cleave, "Young Wandering Minds in the Chemistry Class," CIL Oval, Spring 1963, p. 16.

complex chemical system, a burning candle. The student then compares his list, which usually contains about a dozen observations, with the list of a professional chemist which contains fifty-three observations. The difference in the number and the quality of the observations comes as a shock and revelation to the student. The student who states that he observed carbon dioxide and water vapor being given off by the flame is directed to recognize the distinction between observation and assumption. The first experiment sets the stage for a series of investigations related to the candle. The student studies melting-point behavior, investigates the chemical changes that occur when a candle burns, and identifies the products of the reaction. The investigation of the candle is culminated when he measures the heat of combustion of candle wax and heat of fusion of the wax. During this series of experiments with simple apparatus the student is encouraged to generalize on concepts such as melting point behavior and energy changes associated with physical changes and those associated with chemical reactions.<sup>28</sup>

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<sup>28</sup>J. A. Campbell, "Chemistry--An Experimental Science," School Review, LXX (Spring 1962), pp. 55-56.

Laboratory reports. Although few specific requirements have been laid down by the CHEM Study staff, certain skills which are associated with the gathering of data and arriving at valid conclusions are emphasized. These skills include: the careful recording of observations, the tabulating and graphing of data, the indicating of the limitations of each measurement recorded and each subsequent calculation of secondary data, and the supporting of conclusions through written discussion. Each laboratory report is written in a blank notebook consisting of graph paper with alternate white sheets and a carbon copy on the blue sheets. The carbon copy of the report is torn out and handed into the teacher for checking and the student keeps his original as a permanent record.

Teacher's guide. As stated in its preface, the program guide is designed for optimum day-to-day and background support of the lone teacher with somewhat less than ideal training, teaching in a poorly equipped school or working with other limiting factors. Therefore the guide is a detailed and voluminous directive. For each chapter in the text there is a section pointing out the intent and approach, the outline,

new concepts, schedule of related materials, development, supplementary materials including a full discussion of each laboratory experiment, background discussion and answers to problems for that chapter.<sup>29</sup>

Integration of text and laboratory materials. The CHEM Study student is allowed and encouraged to discover for himself ideas which through class discussion and text treatment will lead him to the understandings that the course seeks to develop. Consistent with such a philosophy, the experiments are closely integrated with topics in the text, but generally precede the textual development. Frequent reference is made in the text to experiments which the student is expected to have performed and thought about. The laboratory work is so closely correlated with the text that it would be almost impossible for the student to profit from the CHEM Study course without participating in the laboratory work.<sup>30</sup>

Although the chief approach of the laboratory is

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<sup>29</sup>CHEM Study Staff, Teachers Guide for Chemistry-- An Experimental Science, (San Francisco: W. H. Freeman and Company, 1963), p. 3.

<sup>30</sup>A. B. Van Cleave, CIL Oval, loc.cit.

inductive, many of the later experiments involve unknowns where the student is encouraged to predict chemical properties. Hence the overall approach of the laboratory program is inductive-deductive with an early emphasis on the inductive reasoning. Once the student compares his predictions with experimental results, he experiences a feeling of success in applying the general concepts of chemistry that he has discovered earlier in the course.<sup>31</sup>

#### V. CONCLUSIONS

The MCA experiments constitute an independent laboratory program which can be adapted to any high school chemistry course, whereas the CHEM Study and CBA experiments are closely integrated with their textbooks in an inductive-deductive manner, with the former emphasizing an inductive approach. The chief difference between them is the amount of direction provided for each student; MCA and CHEM Study experiments generally give the student specific direction but do not suggest the conclusions to be drawn, whereas the CBA experi-

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<sup>31</sup>Campbell, op. cit., pp. 59-60.



ments provide a minimum of direction but stress independent investigation. In each program the laboratory contributes greatly to the teaching of science as a mode of inquiry by illustrating the diverse processes that are used to produce the conclusions of science and the limitations of these methods.

All three of these programs are now exerting a strong influence on curriculum planning. Although these courses, which are based on the same broad aims of modern science education, have been developed over a number of years by leading scientists and educators, both CBA and CHEM Study staffs agree that it is neither possible nor desirable to join forces and produce a single "best" course which would result in a national curriculum. On this topic, one CBA staff member writes:

No single course can achieve all of the legitimate objectives of a high school course in chemistry in the best way for all of the different situations in which such courses are taught.

. . . Acceptance of a single course, or of a single limited set of criteria by which students and courses are evaluated, customarily leads to a standardization of practice which inhibits innovations, hence retards improvement.<sup>32</sup>

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<sup>32</sup>Earle S. Scott, "The Necessity for Variety," CBA Newsletter, May 1962, p. 1.

The aim of both the CBA Project and the CHEM Study is not to sell books, but to provide models--to educators, publishers, textbook writers--of what competent research chemists, and university and high school teachers think are good ways of teaching chemistry at the high school level. With the copyright of each program held by Earlham College and the University of California respectively, rather than by the publishers, these programs hope to encourage writers in preparing their own materials after reviewing the CBA and CHEM Study materials. In this way the area of chemical education at the secondary level will be accordingly enriched.<sup>33</sup>

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<sup>33</sup>Statement by Richard L. Merrill in an address to the CHEM Study Institute at the University of Saskatchewan, July 1963.

## CHAPTER V

### THE SELECTION OF DESIRABLE CHARACTERISTICS FOR AN INVESTIGATIVE LABORATORY

#### I. INTRODUCTION

If a major objective of chemical education is to have students attain an internal awareness that chemistry is really a dynamic science, then the laboratory will have a prominent place in the chemistry program and the experiments will be a challenge directed to the discovery of concepts unknown to the student rather than a mechanical demonstration and repetition of known facts. More specifically, the laboratory program should include the following objectives:<sup>1</sup>

1. To give the student an opportunity to state the problem in his own words.
2. To provide for adequate student preparation before entering the laboratory.
3. To encourage the formation of hypotheses or predictions based on recorded observations.
4. To guide students in the planning and developing of their own procedures.
5. To direct students in selecting the kinds of equipment and materials that they will require, and in improvising apparatus.

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<sup>1</sup>Supra, pp.22-23.

6. To provide for the logical organization of recorded data.
7. To provide for training in processing and analyzing data by graphing methods and solution of mathematical problems to obtain secondary data.
8. To develop an awareness of the limitations of measurement.
9. To encourage the evaluation of the experimental procedures on the basis of the results obtained; to emphasize methods as well as results.
10. To provide for experience in cooperative planning, evaluation of group data, and in other aspects of "team research."
11. To cultivate good laboratory habits of safety and cleanliness.
12. To develop proper techniques in manipulating chemical apparatus and handling materials.
13. To provide for individual differences among students by encouraging the students to extend an investigation beyond the basic experiment.
14. To develop critical thinking on the part of the student.
15. To frequently employ the investigation of an unknown material or chemical system.
16. To indicate practical applications for the procedures followed in the experiment.
17. To require laboratory reports that are functional in that they emphasize communication skills rather than needless repetition of printed instructions.
18. To provide for individual student growth in knowledge, independent thought and self-direction.
19. To give the student an insight into the actualities of scientific investigation.

In the pursuit of these objectives, specific characteristics of a laboratory program must be examined in order to select those characteristics deemed most desirable for the students in Manitoba. Such desirable characteristics were selected through a review of

the recommendations made by independent writers in science education, and then combined with the desirable features of the modern chemistry laboratory programs.

## II. CHARACTERISTICS OF A GOOD HIGH SCHOOL LABORATORY

The critical topic of learning in the laboratory has been reviewed by Robert Stollerg in the NSSP Journal where he lists some characteristics of good high school science laboratory experiences:

1. Classroom and laboratory work are closely integrated.
2. Flexibility of the laboratory schedule is highly desirable.
3. The function of the laboratory is to seek answers and to find information.
4. Real problems are most desirable for laboratory learning.
5. Practical applications should be found for theoretical situations.
6. Cooperative planning by the students and teacher is desirable.

7. Improvisation of apparatus is encouraged.
8. Laboratory manuals are used merely for reference.
9. The laboratory should be an approach, not merely a place.
10. Long-term laboratory activities should be encouraged.
11. Note-taking, data recording, and sketching should be truly functional.
12. Required reports should have purpose and meaning.<sup>2</sup>

In concluding his discussion of the characteristics of good laboratory experiences, Stollberg points out that no one science teacher, nor one school need display all these features in the organization of laboratory learning. In many school situations it is difficult to try to make progress along more than a few of these lines at one time.<sup>3</sup> With the latter statement in mind, Stollberg's characteristics of a good laboratory program were reviewed in this investigation and integrated with the

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<sup>2</sup>Robert Stollberg, "Learning in the Laboratory," The Bulletin of the National Association of Secondary School Principals, XXXVII (January 1953), pp. 103-108.

<sup>3</sup>Ibid., p. 108.

ideas of other writers, in order to arrive at the characteristics most suitable to the Manitoba high school situation.

#### Close Integration of Classroom and Laboratory

The laboratory should be thought of as an approach, as a method--not merely a place; therefore, classroom and laboratory should be made "mutually supporting aspects of the same enterprise with no set rule as to which should precede the other."<sup>4</sup> At no time should the classroom and laboratory experiences be divorced to the extent that they appear as separate courses, or that the students can profitably take one without the other. Rigidly scheduled periods for laboratory experience make it difficult to integrate classroom and laboratory learning and to pursue, in the laboratory, problems which arise spontaneously in the classroom. With the laboratory scheduled on prescribed days, it becomes out of phase with the classroom and bears little psychological relation to it.<sup>5</sup> As desirable as such a flexible schedule might be, few schools

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<sup>4</sup>Ibid., p. 103.

<sup>5</sup>W. Burnett, Teaching Science in the Secondary School (New York: Rinehart and Co., Inc., 1957), p. 190.

in Manitoba have the physical facilities to accomodate it; consequently, other means of integrating the classroom and laboratory must be developed.

If it is believed that the primary function of the laboratory is to transmit the factual heritage of a civilization or that deductive reasoning is more important, the laboratory will be used as a place of verification. Here the laboratory phase follows teaching. This traditional approach is referred as deductive or deductive-descriptive. However, if the students are to learn how to employ the inductive methods of inference, they must be provided with opportunities to make inference from facts; the laboratory is one place where facts are collected in a variety of ways. Hence, the inductive laboratory is planned early in the teaching-learning sequence and implies the use of observed data to arrive at a general principle. If the principle thus discovered is applied in solving a more specific problem, then the approach may be referred to as being inductive-deductive.<sup>6</sup> A comparative

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<sup>6</sup>Milton O. Pella, "The Laboratory and Science Teaching," The Science Teacher, XXVIII (September 1961), p. 31.



study of the deductive-descriptive and inductive-deductive approaches to laboratory instruction in high school chemistry has been reported by Boek. Two comparable groups exposed to the opposing approaches were evaluated on their ability to perform in the laboratory with resourcefulness, using sound techniques. Because of the fact that the inductive-deductive class achieved as well as or better than the deductive-descriptive class in the attainment of the general outcomes of a high school chemistry course, but secured significantly superior results with respect to the crucial problem of attaining knowledge and ability in the use of the methods of science with an accompanying scientific attitude, the general acceptance of the inductive-deductive approach by modern laboratory programs appears to be justified.<sup>7</sup>

Problem situations experienced in the inductive laboratory often lead to productive classroom discussion. Through this approach the student gains a feeling of personal participation in the discoveries of science. The student can then be

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<sup>7</sup>Clarence H. Boek, "Inductive-Deductive Compared to Deductive-Descriptive Approach to Laboratory Instruction in High School Chemistry," Journal of Experimental Education, XIX (March 1951), pp. 247-53.

encouraged to predict chemical properties on the basis of the generalizations he has discovered, and check his predictions with experimental results. The inductive approach followed by the inductive-deductive has been successfully applied by the CHEM Study laboratory program. In the inductive-deductive approach followed by the CBA laboratory program, the experiment gives depth and meaning to the text; the text in helping to interpret the experiment, leads back to the laboratory.

"Each type of learning," writes Stollberg," should support the other, knit together with other types of activities to constitute an integrated set of learning activities."<sup>8</sup> A balance between inductive and deductive approaches, therefore, insure the close integration between classroom and laboratory; it also emphasizes the interplay between theory and experiment, which is a characteristic of the development of science.

The Function of the Laboratory: Exploring and Developing Ideas

The function of the laboratory work is to acquaint students with the processes of inquiry as a means for exploring and developing ideas. The laboratory should develop a sound

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<sup>8</sup>Stollberg, op. cit., p. 103.

approach to critical thinking and a knowledge of how to proceed to conclusions that are defensible.<sup>9</sup> It should also embrace the spirit of discovery. An experiment should, therefore, be an exercise in disciplined thinking, where unexpected results are not regarded as a wrong answer. To be a correct answer to the situation, the hypothesis need not be true; it need only be logical and well-founded.<sup>10</sup> Since the nature of scientific enterprise is found in the methods through which problems are attacked, more attention should be directed to the processes or methods of seeking answers rather than to the too frequent emphasis on finding exact answers.<sup>11</sup>

This shift in emphasis away from the deductive-descriptive approach virtually forces a change from a qualitative to a

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<sup>9</sup>L. Mills and P. Dean, Problem Solving Methods in Science Teaching (Science Manpower Project Monograph), (New York: Bureau of Publications, Columbia University, 1960), p. 1.

<sup>10</sup>Ralph W. Lewis, "How to Write Laboratory Studies Which Will Teach The Scientific Method," Science Education, XXXI (February 1947), p. 16.

<sup>11</sup>Paul Hurd, "Summary of Objectives, "Re-thinking Science Education," Fifty-ninth Yearbook of the National Society for the Study of Education, (Chicago: University of Chicago Press, 1960), p. 35.

quantitative laboratory. The laboratory should, therefore, provide insight into the subtleties of measurement such as the limitations of measurement and the need for independent measurements, which are so basic to successful data collecting in the laboratory.<sup>12</sup> However, it is also in the quantitative laboratory, when the experiment is designed with the expectation that all students get accurate results from a single determination using crude apparatus, that the open-minded spirit of investigation can be cramped. Elmore, Keeslar and Parrish propose:

The precise right answer then is recognized as a hypothetical point more to be abjured than striven for in evaluating one's results in gathering scientific data, and we should look askance at exactly right answers when measuring instruments are crude and chance errors are inevitable.<sup>13</sup>

The laboratory should provide students which opportunities to see the variability in measurement through the pooling of class results. Once the teacher and student are aware of the limitations of measurement, neither will expect results which are beyond the precision of the measuring instrument. In such a

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<sup>12</sup>Paul D. Merrick, "Secondary School Chemistry--A Laboratory Approach," The Science Teacher, XXIX (March 1962), p. 11.

<sup>13</sup>C. Elmore, O. Keeslar, and C. Parrish, "Why Not Try the Problem-Solving Approach," The Science Teacher, XXXVIII (December 1961), p. 3.

quantitative laboratory an open-minded spirit of scientific investigation can be sustained.

The maintenance of a proper balance between student investigation and teacher guidance is emphasized by the committee for the Forty-sixth Yearbook of the NSSE.<sup>14</sup> In pursuit of the proper balance, all possible degrees of independence or freedom in student investigations should be examined. Five such degrees of student independence, as proposed by Pella, are outlined in Table II. In procedure one the purpose, apparatus, and method are completely described by the teacher or the laboratory manual. The method of treating the data is also described and the pupil generally reads the conclusion from the text. The only steps in the total procedure left to the student are the performing of the activity and the gathering of data. Pella raises an objection to the formulation of the conclusion by either the teacher or the laboratory manual. He points out that the statement of the problem very often provides a statement of the conclusion,<sup>15</sup> for example, "To demonstrate

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<sup>14</sup>"Science Education in American Schools," Forty-sixth Yearbook of the National Society for the Study of Education. (Chicago: University of Chicago Press, 1947), p. 236.

<sup>15</sup>Pella, op. cit., pp. 29-31.

TABLE II  
FIVE DEGREES OF STUDENT INDEPENDENCE IN  
LABORATORY INVESTIGATIONS

Steps in the Procedure	Procedures				
	1.	2.	3.	4.	5.
1. Statement of the problem	T*	T	T	T	S
2. Formation of hypotheses	T	T	T	S	S
3. Working plan	T	T	S	S	S
4. Performance	S**	S	S	S	S
5. Data gathering	S	S	S	S	S
6. Conclusion	T	S	S	S	S

\* T - teacher                      S\*\* - student

that one gram molecular weight of a gas occupies 22.4 liters at standard conditions."<sup>16</sup> Procedure two may be achieved by providing activities concerned with problems that are without answers in the text. The employment of procedures one and two would provide for the development of laboratory skills and methods and might serve as an elementary introduction to the laboratory. Procedure three will provide opportunity for

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<sup>16</sup>Frank Harder, Outline of Laboratory Experiment--Chemistry II, p. 15.

students to exercise some creativity in the development of procedures for testing hypotheses. Here, the student must decide what controls and variables to have in his experiments, what kinds of data to collect and how the data is to be treated. Of course the teacher must serve as a guide so that any procedures dangerous to the physical welfare of the students are avoided. Procedure four demands further creativity of the student and leads him to expect confusion as a natural and necessary state which usually precedes good thinking. The latter two procedures give considerable stress to the methods of scientific inquiry. The steps of independent research would be paralleled in procedure five, when the student is confronted with raw phenomena. At this stage he must define the problem and then over a number of laboratory periods, arrange the various facets of the problem into a logical sequence so that they may be attacked successfully.<sup>17</sup>

Sooner or later, therefore, the high school student must be weaned from total dependence upon a set of explicit laboratory instructions. He must be led progressively to a

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<sup>17</sup>Pella, loc. cit.

point at which, once the problem has been crystallized, his techniques and insight are sufficiently developed so that he will be able to set up his own experimental procedures.<sup>18</sup> At present the grade eleven student in the University Entrance course is undergoing his first formal experience in the laboratory; therefore, the problems to be investigated should be adapted to both the student's experience and maturity. The student must possess the related techniques and information needed for the solution, or he knows how to get this information. Success in dealing with a few problems will increase the student's confidence as he undertakes others. Too great a complexity early in the year may lead to escapist activities such as daydreaming and quasi-solutions.<sup>19</sup> "The problem, then, is to provide initial laboratory experiences (or to build on previous experiences) which do result in student involvement in an emotionally and intellectually satisfying manner."<sup>20</sup> Since the grade eleven student is gaining in experience and

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<sup>18</sup>Merrick, loc. cit.

<sup>19</sup>L. Mills and P. Dean, op. cit., p. 13.

<sup>20</sup>Guidelines for Development of Programs in Science Instruction, National Academy of Sciences--National Research Council, Publication 1093, (Washington: Government Printing Office, 1963), p. 7.



maturity as the year progresses, it appears reasonable to expect him to demonstrate growth in self-direction and independent thinking, although he may not yet be capable of truly investigative research after his first year of chemistry. Therefore, it would appear that an investigative laboratory program for grade eleven chemistry could be introduced through procedure two; once the student has acquired some experience and confidence, along with a knowledge of some basic techniques, the program could be progressively advanced to include experiences in procedures three and four.

#### Real Problems--Cooperative Planning

The provision for experiences in the solutions of real problems arising out of classroom discussion, project work or out of the community and not restricted to the manual source, as suggested by Stollberg,<sup>21</sup> pre-supposes a flexibility of the laboratory schedule, superior facilities with ample storage space for long-term investigations, a teacher with above-average background and preparation time, along with students who have sufficient skills and knowledge to proceed intelligently. Despite

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<sup>21</sup>Stollberg, op. cit., p. 106.

the inflexibility of the laboratory schedule, inadequate physical facilities, an authorized manual, and the limitations of student experience in grade eleven, this aspect of laboratory work can still be pursued in this province by encouraging students to return to the laboratory in their free time.

Individual students or small groups should be permitted to carry out laboratory investigations in which they are most interested and for which their talents are best suited. Since this matter of problem solving is highly individualistic and cannot be regimented effectively, it is unrealistic to expect all students to proceed at the same rate and arrive at an answer together on any given day. Some students will be able to solve problems involving subtle distinctions while others may learn only to recognize minor differences.<sup>22</sup> The experiments should be so designed to have an initial, basic part which all students can complete. Other students, proceeding at their own pace should be encouraged to investigate the more advanced problem which is an extension of the initial investigation. Besides providing for individual differences in the

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<sup>22</sup>L. Mills and P. Dean, op. cit., p. 10.

laboratory, this procedure stimulates a number of students to go into interesting, related investigations on their own time.

According to two prominent authorities, it matters little if the problems which the student attempts to solve have been problems which students before him have worked upon, and the answers to which the teacher knows. The important point is that the student investigates a problem, the answer to which he does not know.<sup>23</sup> However, a problem for the student is not necessarily inherent in any given question, interesting and stimulating as it may seem to the teacher. Expanding on this point Keeslar, Elmore and Parrish write:

The problem as such must spring to life in the mind of the student; and the sensitive teacher can do more to create situations which challenge and tantalize, leaving questions unanswered perhaps, and manipulate and encourage the student's thinking to the point where he accepts the problem as his own.<sup>24</sup>

The atmosphere or climate in which this problem solving is pursued is, therefore, important. This has to do with the motivation of the student investigator as well as the physical

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<sup>23</sup>John S. Richardson and G. P. Cahoon, Methods and Materials for Teaching General and Physical Science (New York: McGraw-Hill Company, 1951), p. 30.

<sup>24</sup>C. Elmore, O. Keeslar, and C. Parrish, op. cit., p. 32.

setting. It is the pre-laboratory discussion which sets such an atmosphere, where the student becomes psychologically involved and where the relationship of the experiment to the course work is understood. To ensure that the student returns to the laboratory the following day, still psychologically involved in the problem, he should be expected to think about the design of the experiment and about questions, the answers for which are to be found in the experimental design.<sup>25</sup> It can be concluded, therefore, that if the student is to accept a problem as his own, both a pre-laboratory discussion and a pre-laboratory student preparation are essential.

An able teacher can lead and direct the pre-laboratory discussion in such a way that a class is lead spontaneously to reach conclusions pre-determined by the teacher. Battino describes the use of such a discussion for teaching a class how to carry out an experiment from beginning to end. The following is an outline of his method for cooperative planning:

1. Presenting the problem in general terms.
2. Presenting the specific problem.

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<sup>25</sup> Frank Brescia, et al., Instructor's Manual: Laboratory Studies in General Chemistry, (New York: Academic Press, 1961), p. iii.

3. Finding out what quantities and qualities must be determined.
4. Setting up an experimental procedure.
5. Choosing a practical chemical system.
6. Designing the experimental apparatus.
7. Pointing out the available equipment, reagents to be used, and the results to be reported.<sup>26</sup>

During the course of such a discussion the student answers to stimulating questions are discussed on their merits and occasionally questions are left unanswered. The teacher's role in this type of discussion is one of guidance, of seeking the measure of the student's thinking, and calling his attention to unrecognized sources of error, false assumptions and other deviations from good practice.<sup>27</sup> Further to the topic of cooperative planning, the student's experimental data from suitable experiments should be made available to the group; as a result, more reliable answers are likely to come from the shared efforts of the group than from a single effort. Since no experiment is complete without an evaluation of results and procedures, the teacher should also lead a

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<sup>26</sup>Rubin Battino, "Laboratory by Discussion," Journal of Chemical Education, XXXVII (May 1960), p. 257.

<sup>27</sup>C. Elmore, O. Keeslar, C. Parrish, op. cit., p. 33.

post-laboratory discussion, in which individual data and conclusions are available to the group. The group would be led to consider the variations in the results and the reasons for them, and to consider the possible modification and improvement of experimental procedures. Through these considerations the student gains respect for the work of a team and learns that the best way to decide an issue is to have both individuals and groups consider the facts with all views presented.<sup>28</sup>

#### Use of the Laboratory Manual

Stollberg suggests that the use of the laboratory manual be confined to its value as a reference, with students developing their own procedures by consulting a variety of such manuals for suggestions without following any one manual specifically. Further, Stollberg states, "In using such manuals, it is well for the teacher to remember that, almost by definition they are written for use by the average teacher,

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<sup>28</sup>"Teaching for Critical Thinking in Chemistry," Three Reports of Summer Conferences for Science Teachers, (Washington: National Science Teachers Association, 1958), p. 25.

with average background, ideas, inspiration and equipment."<sup>29</sup>  
In Manitoba, however, every teacher is limited because he is authorized to use only one laboratory manual. Difficulties arising from this situation may be mitigated somewhat through exploration with the student of the plan for laboratory work as set forth in the authorized manual, such as, the basis for choice of techniques and materials, and the reason that a given procedure in the manual has been selected for the solution of a certain problem. Furthermore, this exploration may assist in making the student sensitive to further problems arising from his laboratory work.<sup>30</sup>

#### Functional Laboratory Reports

Reports of laboratory activities should have purpose and meaning. The purpose of such a report is either for communication to other people or for future study and reference by the experimenter. Stollberg doubts whether the educational value, in itself, of writing a laboratory report is equivalent to the actual laboratory experience which the report writing

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<sup>29</sup>Stollberg, op. cit., p. 106.

<sup>30</sup>Richardson and Cahoon, op. cit., p. 32.

displaces. He proposes varied methods of reporting, such as oral, written, demonstrational or graphic.<sup>31</sup> Richardson and Cahoon, on the other hand support the formal report with the four traditional headings; problem, procedure, observation and conclusion. As a supplement to the report, or if carefully planned, as a replacement of the report, they also suggest the possible inclusion of a short test or quiz to evaluate student understanding.<sup>32</sup>

An experimental study of the relative effectiveness of two methods of recording laboratory experiences was reported in the Thirty-first Yearbook of the NSSE. Method A was the preparation of a conventional or formal essay type of report with the inclusion of a statement of the problem, complete verbal description of the method, observations, and the conclusion, accompanied by a labelled diagram of the apparatus used. Method B was the preparation of an abbreviated, graphical report which included a statement of the problem, a motion-picture method of describing the procedure consisting of a series of labelled diagrams showing various stages of manipu-

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<sup>31</sup>Stollberg, op. cit., p. 107.

<sup>32</sup>Richardson and Cahoon, loc. cit.



lation and an indication of observations during the progress of the exercise; and a statement of the conclusion. The investigators found slight, though not statistically significant advantages in favor of the graphical method. They concluded that the graphical method has an advantage over the conventional method, since it effects at least as good learning of subject matter in considerably less time.<sup>33</sup> Hence it would appear that briefer methods of reporting laboratory activities such as the motion-picture method or the flow sheet method may be substituted for the formalized essay type of report with equal effectiveness in certain respects and with a considerable saving of time. However, because each method of reporting offers the student unique and essential values, every laboratory program should offer opportunities for reporting by both graphic methods such as the flow sheet and by the formalized essay type of report.

Furthermore, Lefler recommends the placing of emphasis

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<sup>33</sup>Moore, Dykehouse and Curtis, cited in "A Program for Teaching Science," Thirty-first Yearbook of the National Society for the Study of Education (Chicago: University of Chicago Press, 1932), p. 93.

on only certain individual elements of the laboratory report for each experiment. He is of the opinion that the preparation of a laboratory report presents an opportunity for the student to analyze his method of solution and express himself clearly and accurately. To require a report with equal emphasis on each of its elements for every experiment would demand so much work of the student that he would become disinterested to such a degree that the report would become mechanical. The student would then simply prepare something which he hoped would be acceptable to the teacher. To overcome this difficulty, different elements of the laboratory report may be emphasized for each experiment. The student must keep a neat record of what he does when he does it. Only occasionally should the experiment be written up as a formal report. This report should be as simple as possible without needless repetition of printed directions, but should include all the original data recorded as observed, and graphs of all relationships which are meaningful, and a paragraph of conclusions and discussion of error.<sup>34</sup>

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<sup>34</sup>R. W. Lefler, "Teaching of Laboratory Work in High School Physics," School Science and Mathematics, XXXXVII (June 1947), pp: 531-38.

Lefler's view of reporting has been adopted by all three modern chemistry laboratory programs.

In reporting, therefore, stress must be placed on description of procedures, data recording and drawing of apparatus assemblies, if they are truly functional. Data must be recorded when recording helps to clarify them, or when they are needed for future reference. When the data is recorded it must be tabulated in a way which facilitates direct calculation from the table. Diagrams must be made when they are actually needed or when they are not readily available. Since it is functional and time-saving, the flow sheet may be employed rather than a repetition of printed procedures. The essay or formal report may best be employed when the student is developing a portion or all of his laboratory procedure. A truly functional part of this student report is the pre-laboratory preparation which arises from the pre-laboratory discussion and prepares the student for the laboratory, and thus ensures a problem-solving atmosphere.

The laboratory report is to be written in a blank notebook consisting of graph paper with alternate white sheets and a carbon copy on blue sheets so that the carbon copy of the

report may be torn out and handed to the teacher for checking at the end of the laboratory period. The student must keep his original copy as a permanent record. With adequate pre-laboratory preparation, most experiments may be completed before the end of the laboratory period; if not, a copy of the student's data must be turned in so that the teacher can immediately check the student's progress. The use of one-quarter inch graph paper facilitates tabulation and encourages the graphing of data whenever it is functional. The use of carbon paper and the completion of most experiments within the laboratory time, further encourages greater self-direction and independent thought on the part of the student, and at the same time reduces the opportunity for copying among students.

### III. SUMMARY

Many desirable features of a laboratory program have been reviewed with the following characteristics emerging as the most desirable and the most adaptable to the existing high school situation in grade eleven chemistry:

1. The classroom and the laboratory are to be closely integrated. There is no set rule as to one preceding the

other; each type of learning should support the other by means of an inductive-deductive approach in order to present an integrated learning situation. The laboratory approach is to be extended into the classroom so that the laboratory is thought of as an approach and not just as a place.

2. The function of the laboratory is to provide experiences in exploring and developing ideas which are preferably quantitative in nature. Emphasis is to be placed on methods as well as on results; the results being evaluated within the limits of instrument precision. Initially the laboratory should provide the student with experiences in techniques of manipulating apparatus and in obtaining meaningful results. Once having gained some confidence the student may be progressively advanced so that his experiences include more independent activities involving hypothesis formation and subsequent experimental verification, and the development of independent procedures and their evaluation.

3. Students will become psychologically involved and be led to accept laboratory problems as their personal challenge through effective pre-laboratory discussion and preparation. For some experiments, through cooperative planning in the pre-

laboratory discussion, the students will be led to develop their own procedures. In order to evaluate their procedures and results, group techniques will be employed in post-laboratory discussions. Students will always be encouraged to go onto interesting, related investigations on their own.

4. In the reporting of experiments, stress shall be placed on functional activities. Diagrams will be made when they are actually needed or when they are not readily available. Because it is functional and time-saving, the flow sheet will be employed in place of the repetition of printed directions. The essay or formal report will be employed when the student is developing a portion or all his laboratory procedure independently. A truly functional part of every report will be the student's pre-laboratory preparation.

5. The students will write their laboratory reports on graph paper and make a carbon copy which will be handed in completed at the end of the laboratory period. This method will facilitate data tabulation and graphing; it will also encourage greater independence and self-direction.

## CHAPTER VI

### DEVELOPMENT OF THE LABORATORY MANUAL AND EVALUATION OF THE PROGRAM

#### I. DEVELOPMENT OF THE LABORATORY MANUAL

Following the establishment of the desirable characteristics of the grade eleven chemistry laboratory program, thirty-one chemistry laboratory manuals, including several first year university manuals for general chemistry, were examined. These manuals varied in approach from the very traditional to the most modern. During this examination, particular attention was directed to approaches which could be adapted to the established criteria of the investigative laboratory and to experiments which were compatible with the grade eleven chemistry curriculum.

To comply with the established criteria, experiments which developed important concepts in the grade eleven curriculum were chosen, not only for the phenomena which they illustrated but also for what they taught about the processes

of scientific inquiry.<sup>1</sup> At first, experiments were selected only from the modern sources; they were then re-written to attain a uniformity of approach and often combined features of several manuals into one experiment. As suggested by Lewis, the traditional approach to the laboratory was ignored during the early stages of preparation. However, once the mutant idea of the investigative laboratory was developed, considerable use was made of the traditional material, which in the presence of the mutant, took on new and useful characteristics.<sup>2</sup> Similarly, in the development of the CHEM Study Laboratory Program, the staff of writers took no special pains to develop totally original experiments. After deciding what concepts were to be discovered by the student, many available, traditional experiments were re-written by the CHEM Study Staff to emphasize either the inductive or inductive-deductive approach.<sup>3</sup>

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<sup>1</sup>Jack H. Robinson, "How Should We Teach 'The Scientific Method'?" (part II) Science and Maths Weekly--Teacher's Edition, XII (January 16, 1963), pp.1-2.

<sup>2</sup>Ralph W. Lewis, "How to Write Laboratory Studies Which Will Teach the Scientific Method," Science Education, XXXI (February 1947), p. 15.

<sup>3</sup>Robert Merrill, personal letter, August 26, 1963.



In the present investigation, a re-examination of traditional laboratory manuals produced several experiments which were reorganized to fit into the established framework. Obourn writes that even the most commonplace topic in science-- with a slight shift in emphasis, can be made into a challenging lesson. Such a change of approach will utilize the concept or principle of the topic, and will, in addition, give practice in using some of the processes of inquiry.<sup>4</sup> The following are examples of the reorganization of traditional experiments:

1. As a means of introducing the techniques of filtration, a traditional experiment common to many laboratory manuals involves the preparation and separation of a precipitate. This manipulative exercise was reorganized by the CBA Laboratory Development Staff into an investigation in which the student is required to determine which of the two reacting solutions is in excess.<sup>5</sup> To avoid misleading observations, the

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<sup>4</sup>Ellsworth S. Obourn, Suggestions for Starting Process Teaching in Science, A Bulletin of the Office of Education, U. S. Department of Health, Education and Welfare, (Washington: Government Printing Office, April 1963), p. 1.

<sup>5</sup>CBA Laboratory Development Committee, Chemistry Laboratory, (third revision, Annville, Pennsylvania, 1962), pp. 10-12.

student must now employ more careful techniques.

He is also required to do some critical thinking in order to arrive at an answer to the problem.

2. In order to distinguish between physical and chemical changes, the traditional laboratory employs the study of changes in physical properties of known chemical systems which have been previously described in the text. However, if after some practice is obtained in making observations on known chemical systems, the student is required to study unknown systems, he will then be participating in an investigation. One such unknown system which may be employed is hydrochloric acid and sodium hydroxide solution. The identification of the type of change taking place upon mixing equal volumes of these nameless solutions must come from visual observation and measurement with a thermometer. The answer is not available in the textbook. This investigation can also be extended by a superior student to include the identification of the types of change following a study of the chemical properties of a chemical system, as well as from an observation of the physical properties.

3. The determination of the percentage of water in a hydrate can be made to fit within the investigative framework through the use of an unknown hydrate, the independent organization of the flow sheet and data table by the student, the emphasis on reproducible results within the limits of precision of the balances used, and a guided discussion of possible errors and their effects upon the experimental determinations.

To produce this shift in emphasis towards the processes of inquiry, a similar pattern of reorganization of traditional experiments was followed for the remaining experiments in the program; however, two traditional experiments were reorganized, only slightly, in order to illustrate the use of the experiment for reproduction of the work of other scientists. For the preparation of two gases, oxygen and hydrogen, and the study of their properties, the experiments were designed chiefly to illustrate the distinction between a verification and an investigative type of experiment.

A most desirable outcome from the examination of modern laboratory manuals was a method of discussing possible errors and their effect on the experimental results following a

quantitative experiment in which the instructions to the students are given in the following manner:

In Part A, state the error (+, -, 0) of each of the following on your determined molecular weight: (Dumas Method)

- a) The stopper absorbs some vapor.
- b) The condensed liquid contains substances extracted from the stopper.
- c) The vapor does not displace all the air in the flask at the elevated temperature.
- d) During the cooling process, some of the vapor diffuses out of the flask.
- e) The temperature of the flask does not reach that of the bath.<sup>6</sup>

By requiring the student to discuss portions of this list of possible errors, he is faced directly with some of the subtleties of experimental error and is challenged to do some critical thinking. It is only after experience of this nature that the student can independently and intelligently discuss the sources of errors and their effects on his experimental results. This approach to discussion of error was readily adapted to the majority of quantitative experiments in the investigative laboratory program.

Since the varied processes of inquiry and methods of reporting are to be stressed in this investigative laboratory program,

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<sup>6</sup>Frank Brescia, et al., Laboratory Studies in General Chemistry, (New York: Academic Press, 1961), pp. 45-46.

a detailed introduction to reporting was prepared<sup>7</sup> to insure that the student will be familiar with the elements of reporting such as the organization and interpretation data, the different methods of graphing data, the distinction between observation and assumption, the concept of a negative conclusion, and directions for the organization of a flow sheet. Through this introduction to reporting, the student is also lead to appreciate the importance of a laboratory notebook and the necessity of his pre-laboratory preparation.

In this laboratory program definite emphasis has been placed on the processes of inquiry and reporting; therefore, steps have been taken to ensure familiarity with chemical apparatus, the development of proper manipulative techniques, and habits of safety and cleanliness. To introduce the student to chemical apparatus, descriptions of their structure and uses were adapted from Hoffman and Sanderson.<sup>8</sup> In order to intro-

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<sup>7</sup>Phillip Goldstein, How to Do an Experiment, (New York: Harcourt, Brace and World, 1957), pp. 28-46.

<sup>8</sup>Emil Hoffman, Basic Principles of Experimental Chemistry, (Notre Dame, Indiana: University of Notre Dame, 1955), pp. 5-10; R. T. Sanderson and William E. Bennett, A Laboratory Manual for Introduction to Chemistry, (New York: John Wiley and Sons, 1955), pp. 5-10.

duce the student to the importance of proper techniques and the steps required, detailed descriptions and diagrams were adapted from several manuals.<sup>9</sup> The description of each type of apparatus, technique, and element of reporting was given a code number such as:

- A-20. The Bunsen burner.
- T 5. Reading scales.
- T-7. Weight measurements.
- R-7. Analyzing and interpreting data.

As part of his pre-laboratory preparation the student is encouraged to review all those references which pertain to his immediate experiment.

## II. AN EVALUATION OF THE LABORATORY PROGRAM

### Pilot-testing and Revision

Following the pre-testing of the experiments in the laboratory by this investigator, the first laboratory manual was

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<sup>9</sup>Harper W. Frantz and Lloyd E. Malm, Essentials of Chemistry in the Laboratory, (San Francisco: W. H. Freeman and Co., 1961), pp. x - 27. CHEM Study Staff, Laboratory Manual for Chemistry An Experimental Science, (San Francisco: W. H. Freeman and Co., 1963), pp. x-xi et. seqq., 10, 11, 23, 24.

prepared for the academic year 1962-63. One hundred and fifty grade eleven chemistry students of Churchill High School in Winnipeg participated in the pilot-testing. Once the students of the first class performed each experiment and turned in their laboratory reports at the end of the period, these reports were carefully evaluated with special attention given to the difficulty and the lack of clarity in the questions asked in the manual or the discrepancies arising out of the outlined procedures. Students with their unexpected answers pointed the way for much revision; these revisions were often made in time for pilot-testing by another class on the following day. A colleague who participated in the pilot-testing without the preconceptions of the investigator was also a rich source of suggestions for revision. Owing to a lack of available time only a limited number of experiments could be pilot-tested by the Grade XI students during the first year. For this reason, the remaining experiments were pilot-tested by the same students the following year in grade twelve, or by the grade eleven Chemistry Club. For the academic year 1963-64, the laboratory manual was completely rewritten in order to incorporate all the revisions made during the previous year,

to achieve a greater uniformity of approach, to organize the experiments in approximate order of difficulty, and finally, to include a more detailed introduction to laboratory techniques and methods of reporting. Over a period of two academic years three hundred students participated in the pilot-testing of the laboratory program.

### Evaluation Procedures

To obtain an overall outline of the investigative laboratory program, a summary was prepared which included such relevant information as, (1) correlation with the authorized text for grade eleven chemistry in the University Entrance Course of Manitoba,<sup>10</sup> (2) the laboratory time required for each experiment, (3) the laboratory techniques employed, (4) the concepts developed, (5) the intended purpose of each experiment, and, (6) the sources of ideas for each experiment. Such an outline was useful as an overview prior to taking steps in the formal evaluation of the investigative laboratory program.

The rating form, previously used by twenty-six chemistry teachers to evaluate the existing traditional laboratory pro-

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<sup>10</sup>Dull, William Brooks and Clark Metcalfe, Modern Chemistry, (New York: Henry Holt and Company, 1950),



gram, was applied in the evaluation of the investigative program. In order to facilitate a comparison of the relative effectiveness of the two programs, a variation of the original method was employed. In the evaluation of the traditional laboratory program by the teachers, the range was from four to thirty-five out of a maximum of seventy-six. It would therefore appear that the teachers applied varied interpretations to the specific objectives used in the rating form. Taking this into account, the investigator evaluated both programs independently in order to obtain a degree of uniformity required for comparison.

The method of evaluation was varied slightly so that the investigator made quantitative judgments on the relative attainment of specific objectives by each individual experiment.<sup>11</sup> Owing to the wide scope of the objectives, no single experiment was expected to measure up to all of the criteria, but a quantitative summation of the judgments on the individual experiments provided a more valid evaluation of the total programs than that which could be obtained through a direct consideration of the programs as a whole.

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<sup>11</sup>Arthur C. Murdock, "Evaluation of Experiments in High School Science," The Science Teacher, XXVI (December, 1959), p. 546.

### Results of the Comparative Evaluation

A comparison of the judgments on the individual experiments in Tables 3 and 4 indicate that many of the experiments show a consistent weakness in specific areas which is reflected in the total effectiveness of the programs in meeting the established criteria. The quantitative summation for the existing traditional laboratory program was 176 as compared to 540 for the investigative program. The graphical summary of the comparative evaluation in Figure 3 points out that the traditional laboratory program is slightly more effective in the areas of inculcating habits of safety and cleanliness and in providing for experiences in proper techniques of manipulating apparatus. The investigative laboratory program, in contrast, is shown to be more effective in meeting all other criteria of modern chemical education, especially in those areas which reflect the shift in emphasis towards scientific inquiry, such as adequate pre-laboratory preparation, logical organization of data, analysis of data, evaluation of experimental procedures, experiences in critical thinking, and growth in self-direction.

TABLE III

AN EVALUATION OF THE EXISTING GRADE ELEVEN LABORATORY PROGRAM  
EMPLOYING THE EVALUATION OF INDIVIDUAL EXPERIMENTS

No.	Title of Experiment	Independent Statement of Problem	Pre-laboratory Preparation	Hypothesis Formation	Independent Procedures	Apparatus Selection and Improvisation	Logical Organization of Data	Analysis of Data, Graphing, etc.	Awareness of Limitations of Measurement	Evaluation of Experimental Procedure	Team Research	Habits of Safety and Cleanliness	Techniques of Handling Apparatus	Provision for Individual Differences	Critical Thinking	Unknown Systems	Practical Applications	Functional Laboratory Reports	Growth in Self-direction	Insight Into Scientific Investigation	Total
1.	Construction and Operation of the Bunsen Burner	0	0	0	1	0	0	0	0	0	1	1	0	0	0	0	1	1	1	1	6
2.	Preparation and Separation of a Precipitate	0	0	0	0	0	0	0	0	1	0	1	0	0	0	1	0	0	0	0	3
3.	Mixtures and Compounds	0	0	0	0	0	0	0	0	0	2	1	0	0	0	0	2	0	1	0	6
4.	Physical and Chemical Changes	0	0	0	0	0	0	0	0	0	2	2	0	0	0	0	1	0	1	1	6
5.	Oxygen--Preparation and Properties	0	0	0	0	0	0	0	1	0	2	3	0	0	0	0	2	0	1	1	9
6.	Water and Oxides	0	0	0	0	0	0	0	0	0	2	3	0	0	0	0	1	0	1	1	4
7.	Hydrogen--Preparation and Properties	0	0	0	0	0	0	0	1	0	3	3	0	0	0	0	2	0	1	1	10
8.	Identification of Products from Experiment Seven	0	0	0	0	0	0	0	0	0	1	2	0	2	1	0	1	0	1	1	8
9.	Graham's Law of Diffusion	0	0	0	0	0	1	0	0	1	2	1	0	2	0	0	2	0	2	2	11
10.	Boyle's Law	0	0	0	0	0	1	0	0	2	0	2	0	1	0	0	2	0	2	2	10
11.	Treatment of Water Supplies	0	0	0	0	0	0	0	2	0	1	1	0	1	0	0	1	0	1	1	4
12.	Study of Solutions	0	0	1	0	0	0	0	0	0	0	2	0	0	0	0	1	0	1	1	5
13.	Per cent Water of Hydration	0	0	0	0	1	1	0	1	0	1	3	0	1	0	0	2	0	2	2	12
14.	Hydration of Salts	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	1	1	2
15.	A Study of Destructive Distillation	0	0	0	0	0	0	0	0	0	1	1	0	0	0	0	1	0	1	1	4
16.	Gram Molecular Volume of Hydrogen	0	0	0	0	0	1	1	0	2	0	2	0	1	0	0	2	0	1	1	10
17.	Carbon Dioxide--Preparation and Properties	0	0	0	0	0	0	0	0	0	0	2	0	0	0	0	1	0	1	1	4
18.	Study of Acids	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	1	0	1	1	3
19.	Neutralization	0	0	0	0	0	0	0	0	0	1	2	0	0	0	0	1	0	1	1	5
20.	Ammonia--Preparation and Properties	0	0	0	0	0	0	0	1	0	1	2	0	0	0	0	1	0	1	1	6
21.	Allotropic Sulfur	0	0	0	0	0	0	0	0	0	1	1	0	0	0	0	1	0	1	1	4
22.	Sulfur Dioxide--Preparation and Properties	0	0	0	0	0	0	0	0	0	2	2	1	0	0	0	2	0	1	1	8
23.	Sulfuric Acid--Study of Properties	0	0	0	0	0	0	0	0	0	1	1	0	2	0	0	1	0	2	2	7
24.	Chlorine--Preparation and Properties	0	0	1	0	0	0	0	0	0	2	3	0	2	0	0	2	1	2	2	13
25.	Hydrogen Chloride--Preparation and Properties	0	0	0	0	0	0	0	1	0	1	1	0	0	0	0	1	0	1	1	5
26.	Identification Tests	0	0	0	0	0	0	0	0	0	2	1	0	1	2	0	2	1	2	2	11
		0	0	2	1	0	3	3	0	10	1	27	43	1	12	3	1	35	3	31	176

TABLE IV  
AN EVALUATION OF THE INVESTIGATIVE LABORATORY PROGRAM

No.	Title of Experiment	Independent Statement of Problem	Pre-laboratory Preparation	Hypothesis Formation	Independent Procedures	Apparatus Selection and Improvisation	Logical Organization of Data	Analysis of Data, Graphing, etc.	Awareness of Limitations of Measurement	Evaluation of Experimental Procedures	Team Research	Habits of Safety and Cleanliness	Techniques of Handling Apparatus	Provision for Individual Differences	Critical Thinking	Unknown Systems	Practical Applications	Functional Laboratory Reports	Growth in Self-direction	Insight Into Scientific Investigation	Total
1.	Scientific Observation	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	2	0	1	4	
2.	Measurement and the Metric System	0	2	0	1	0	2	3	3	0	0	3	4	2	0	0	3	2	3	31	
3.	Density--An Identifying Property	0	1	0	0	0	1	1	0	3	0	1	2	2	2	0	3	1	2	19	
4.	Using the Bunsen Burner	0	4	0	2	0	1	2	0	2	1	2	2	2	0	0	4	3	3	29	
5.	The Melting Point of a Pure Substance	0	2	0	1	0	1	3	0	1	0	1	2	1	1	1	3	1	3	23	
6.	Investigation of Physical and Chemical Changes	0	1	0	0	0	1	0	0	0	0	1	1	2	2	2	1	0	1	13	
7.	Heating Effects of Physical and Chemical Changes	0	1	0	0	0	2	2	0	2	1	1	1	1	1	0	3	1	2	18	
8.	Processing of Raw Data	0	0	0	0	0	0	3	0	0	0	0	0	2	0	0	3	1	1	10	
9.	Formation of a Precipitate--A Qualitative Investigation	0	2	1	0	0	1	0	0	0	0	1	2	2	1	0	3	1	3	19	
10.	Oxygen--A Verification Experiment	0	2	0	0	0	1	0	0	0	0	3	2	0	0	0	2	0	2	14	
11.	Hydrogen--A Verification Experiment	0	1	0	0	0	1	0	0	1	0	3	3	1	0	0	2	0	1	15	
12.	Construction of a Logical Model	0	0	2	2	0	1	1	0	2	0	0	1	3	2	1	3	2	3	23	
13.	Distillation	0	2	0	0	0	1	0	0	1	0	2	2	2	0	1	2	1	2	18	
14.	Separation of a Mixture	0	4	1	3	1	1	0	0	2	0	2	0	1	2	0	4	3	3	27	
15.	Making a Solubility Curve--Team Research	0	2	2	0	0	3	3	1	3	3	1	2	1	1	0	3	1	3	29	
16.	Making a Solubility Curve--Independent Investigation	0	1	0	0	0	1	2	0	2	0	1	1	1	1	0	3	0	2	14	
17.	An Investigation Into the Properties of Crystals	0	3	3	1	0	0	0	0	2	1	1	0	0	2	0	3	2	3	21	
18.	Water of Hydration in Crystals	0	1	0	0	0	1	0	0	0	0	1	1	1	2	0	2	0	1	10	
19.	Percent of Water of Hydration	0	2	0	0	0	2	1	0	3	0	2	3	1	2	2	3	1	2	24	
20.	Reaction of Oxides and Water--Team Research	0	1	2	0	0	2	2	0	1	2	1	1	3	1	0	3	3	4	27	
21.	Identification of Five Unknown Solutions	0	0	0	1	0	1	2	0	0	0	1	1	3	4	0	4	3	3	24	
22.	Molecular Weight Determination	0	0	0	0	0	2	2	0	3	0	1	2	1	2	2	3	1	2	21	
23.	Determination of the Volume of a Gas Produced by a Reaction	0	3	0	0	0	2	2	0	2	0	1	2	0	0	0	2	1	2	19	
24.	Analysis of an Unknown Mixture	0	2	0	0	0	2	2	0	3	0	1	1	2	2	1	3	2	2	25	
25.	Preparation of a Soluble Salt	0	4	0	3	2	2	2	0	3	0	1	1	0	3	0	4	3	4	32	
26.	Determination of the Density of Oxygen.	0	4	0	2	0	2	2	0	4	0	1	1	0	4	0	0	3	4	31	
		0	45	11	16	3	34	35	4	43	8	26	36	32	50	19	4	76	36	62	540

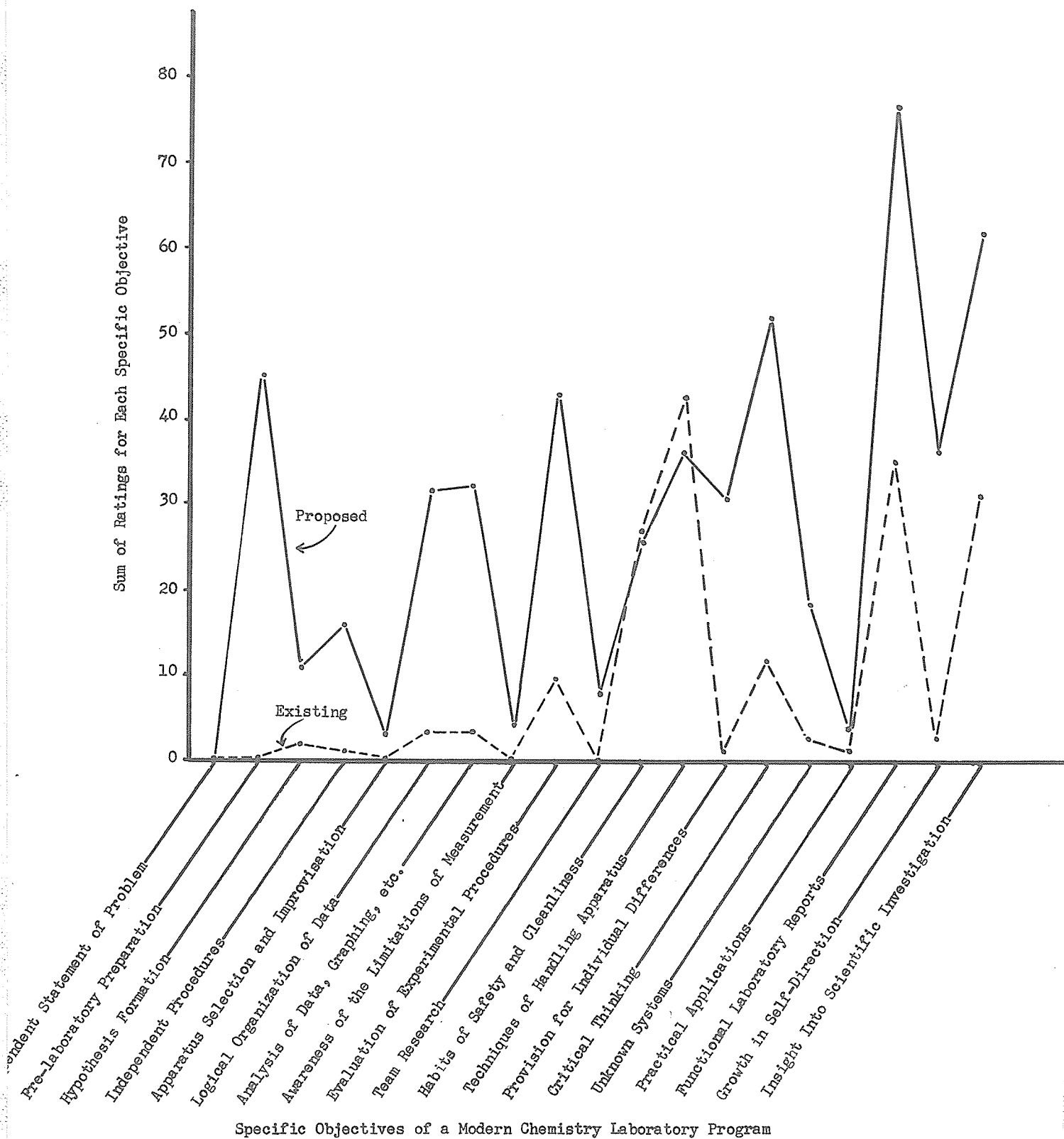


FIGURE 3

SUMMARY OF A COMPARATIVE EVALUATION OF THE  
EXISTING TRADITIONAL AND THE PROPOSED  
INVESTIGATIVE LABORATORY PROGRAMS

## CHAPTER VII

### SUMMARY AND CONCLUSIONS

The purpose of this study was to develop an investigative laboratory program suitable for the grade eleven chemistry in the University Entrance Course in the province of Manitoba. For this study, the term investigative was applied to those experiments which simulated the activities of scientific inquiry within the limits of the high school situation. The significance of this problem is reflected in the graduation of high school students with little appreciation of the science of chemistry as an experimental or investigative method of inquiry because the existing traditional laboratory program, as it is conducted in most schools, contributes little towards the learning of science as a process of inquiry. Since 1958 three notable projects have been organized, independent of each other in the United States, in order to correct the undesirable situation in the high school chemistry laboratory. This study undertook to integrate the signally desirable or unique features of each project into an investigative laboratory program suitable for grade eleven

chemistry in the province of Manitoba. The topic for this study is justified, despite the outstanding contributions of the projects reviewed, because no single course can achieve all of the legitimate objectives of a high school course in chemistry in the best way for all of the different situations in which such courses are taught.

Any laboratory program, however, should be consistent with the broader objectives of science education. From a review of science education literature, it was noted that the following summary reflects the shift in emphasis in the broad objectives of education in the past decade. As stated previously, the modern science course should:

1. Provide a logical and integrated picture of contemporary science: the theories, models and generalizations that picture the unity of science.
2. Illustrate the diverse processes that are used to produce the conclusions of science and show the limitations of these methods: the ways of inquiry and the structure of scientific knowledge.
3. Enable the student to reach at some point the shadow of the frontier: to experience the meaning of "we just don't know", and to be aware of the progress of science.

The scientific advances of the past decade have demanded an education different in kind; one which is geared to change and which provides for self-direction in living. This modern view of science education diminishes the importance of the

particular subject matter employed at the same time that it elevates the spirit of inquiry and the role of the laboratory. From a review of the various statements of objectives of the chemistry laboratory made by science educators, the following specific objectives emerged as those which best reflected the shift in emphasis towards the modern objectives of science education:

1. To give the student an opportunity to state the problem in his own words.
2. To provide for adequate student preparation before entering the laboratory.
3. To encourage the formation of hypotheses or predictions based on recorded observations.
4. To guide students in the planning and developing of their own procedures.
5. To direct students in selecting the kinds of equipment and materials that they will require, and in improving apparatus.
6. To provide for the logical organization of recorded data.
7. To provide for training in processing and analyzing data by graphing methods and solution of mathematical problems to obtain secondary data.
8. To develop an awareness of the limitations of measurement.
9. To encourage the evaluation of the experimental procedures on the basis of the results obtained; to emphasize methods as well as results.
10. To provide for experience in cooperative planning, evaluation of group data, and in other aspects of "team research."
11. To cultivate good laboratory habits of safety and cleanliness.
12. To develop proper techniques in manipulating chemical apparatus and handling materials.



13. To provide for individual differences among students by encouraging the students to extend an investigation beyond the basic experiment.
14. To develop critical thinking on the part of the student.
15. To frequently employ the investigation of an unknown material or chemical system.
16. To indicate practical applications for the procedures followed in the experiment.
17. To require laboratory reports that are functional in that they emphasize communication skills rather than needless repetition of printed instructions.
18. To provide for individual student growth in knowledge, independent thought and self-direction.
19. To give the student an insight into the actualities of scientific investigation.

These nineteen specific objectives served to guide the development and the evaluation of the investigative laboratory program.

Although these objectives are more readily attained by an inductive-deductive laboratory approach, the existing traditional chemistry laboratory program for grade eleven in Manitoba is deductive-descriptive in approach since the experiment characteristically follows the classroom discussion of topics. Since most traditional experiments are not excursions into the unknown, but are thoroughly described in the text, this type of program robs the student of initiative at the same time that it fosters several undesirable practices in the laboratory--practices which make a mockery of the workd experiment and ridicule the painstaking work of the scientists. The

need for an improvement in the existing traditional laboratory program was established through an evaluation made by twenty-seven chemistry teachers in the Greater Winnipeg Area. The rating form, an outgrowth of Chapter II, which the teachers completed, contained the list of nineteen specific objectives of a modern chemistry laboratory program. According to one of five quantitative categories, the extent to which each objective was met was rated by each teacher. In summarizing the results of the forms, it was noted that the ratings ranged from four to thirty-five, out of a maximum of seventy-six. Besides revealing the many weaknesses of the traditional laboratory program, the evaluation pointed out its two strengths-- the development of laboratory techniques and habits of safety and cleanliness.

Within the broad framework of the modern aims of science education, the laboratory programs of the MCA, the CBA Project and the CHEM Study have all been developed. A careful analysis of these programs, including participation in both CBA and CHEM Study summer institutes, revealed that each program contributes greatly to instilling the spirit of scientific inquiry into its students, although each has an approach that is unique.

The many new concepts in laboratory teaching which were revealed in this analysis were combined with suggestions of independent writers. From this pool of ideas the following characteristics emerged as the most desirable and most adaptable to the existing high school situation in grade eleven chemistry.

1. The laboratory and the classroom should be closely integrated through an inductive-deductive approach. By an extension of the laboratory into the classroom, the laboratory is thought of as an approach and not just a place.

2. The laboratory should provide experiences in the exploration and development of ideas, preferably quantitative in nature and place emphasis on methods as well as results. The evaluation of such experimental results should be made within the limitations of measurement.

3. A proper balance between student investigation and teacher guidance should be maintained throughout the program with the student being led progressively to a point at which his techniques and insight are developed to the extent that, once the problem has been crystallized, he will be able to set up his own experimental procedures.

4. Through a pre-laboratory discussion and the student's pre-laboratory preparation, an atmosphere for investigation should be created before the actual laboratory period. Co-operative planning during these discussions should be employed to crystallize the problem and to develop independent procedures.

5. No experiment should be assumed completed until the student, through a post-laboratory discussion led by the teacher, has the opportunity to correlate and interpret the quantitative results of the class as a group. It is here that the student will develop an appreciation for the variations in measurement and for the advantages of group methods of investigation.

6. Student laboratory reports should be functional. Diagrams should be drawn only when they are actually needed or when they are not readily available. In place of the repetition of printed directions, the flow sheet should be employed, since it is functional and time-saving. The essay or formal report should be employed only when the student is developing a portion or all of his procedure independently. The student's pre-laboratory preparation should be a truly functional part of every report.

7. A laboratory notebook, similar to the type introduced by CBA and CHEM Study, should be used in order to facilitate

the logical tabulation of data and to encourage graphical analysis. This type of notebook permits the student to keep a permanent record of his activities and immediately enables the teacher to check the carbon copy for an indication of the student's progress.

8. The laboratory reports should be completed and the carbon copies turned in at the end of the laboratory period. With adequate pre-laboratory preparation the laboratory report, for most experiments, will be completed in this time. This procedure encourages an investigative atmosphere in the laboratory and greater self-direction and independent thought on the part of the student.

Experiments, which met the established criteria and which were related to the existing grade eleven chemistry curriculum, were selected through a review of thirty-one laboratory manuals. These manuals varied from the very traditional to the most modern in approach. Those experiments that were selected from modern sources were re-written to attain a uniformity of approach, whereas those experiments selected from traditional sources were re-examined and re-constructed to reflect the shift in emphasis in the objective of science education towards scientific inquiry in the

the laboratory. During the academic year 1962-63, the experiments were pilot-tested by a total of 150 grade eleven students of Churchill High School in Winnipeg. Following an evaluation of student performance, many of the experiments were revised, either for the following class or for the following year. The laboratory manual was then re-written to incorporate these revisions and expanded to include a detailed introduction. Pilot-testing was completed during the academic year 1963-64.

The success of the investigative laboratory program was also measured by evaluating its effectiveness in meeting the established criteria. This was followed by a comparison with the evaluation of the existing traditional laboratory program. The investigator evaluated both programs independently in order to obtain a degree of uniformity required for the comparison. Quantitative judgments were made on each individual experiment rather than on each program as a whole. No single experiment was expected to satisfy all of the criteria but a quantitative summation of the judgments on the individual experiments provided a more objective evaluation of the total programs than could have been obtained through a direct consideration of the programs as a whole.

Rating higher in seventeen out of nineteen areas, being surpassed slightly in only the areas of safety habits and technique development, the investigative laboratory program, it could be concluded, is more effective in meeting the established criteria than the existing traditional laboratory program and is, therefore, educationally more desirable. In the final rating the traditional laboratory program received 176 points in contrast with the 540 points assigned to the investigative laboratory program. Through an evaluation of individual student reports it became evident that the students who experienced the investigative experiments correlated with their grade eleven text were stimulated to critical thinking and as a result of their experiences were made more aware of problems and more proficient in dealing with them.

Greater objectivity could have been attained in the evaluation procedures through a long range study of comparable groups exposed to the opposing programs and by comprehensive testing on all the desirable outcomes of chemical education. Such an evaluation procedure, however, was beyond the scope of this investigation.

The evaluation procedure which was employed not only pointed out the areas of comparative strength in the investi-

gative laboratory program, but also served to point out areas of weakness, (1) the independent statement of the problem, (2) an awareness of the limitations of measurement, (3) an indication of practical applications, (4) experiences in group techniques, (5) independent selection and improvisation of data, and (6) the encouragement of hypothesis formation. These areas might be further strengthened by re-writing some of the experiments and shifting the emphasis towards these areas. Since no laboratory program can pursue all these desirable areas simultaneously, a choice would be required here to determine which of these areas could be further strengthened without correspondingly weakening other areas. Although the approach of the investigative laboratory program provides for development in self-direction and disciplined thought, it does not directly provide for totally independent investigations; therefore, one of the established criteria, that is, the independent statement of the problem by the student, has not been met. The extent to which this laboratory program can be investigative is limited, however, by the experience and the mental maturity of the grade eleven student.



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APPENDIX A

SAMPLE RATING FORM EMPLOYED BY CHEMISTRY TEACHERS

An Evaluation of the Traditional Grade XI Chemistry Laboratory Program

The following rating scale has been organized in conjunction with the development of a M. Ed. thesis topic. It contains characteristics of a high school laboratory program which are in accord with the modern objectives of science education.

Rate the existing Grade XI Chemistry laboratory program which you have taught in the past year according to these characteristics:

	<u>Excellent</u> 4	<u>Very Good</u> 3	<u>Good</u> 2	<u>Fair</u> 1	<u>Poor</u> 0
) Gives an opportunity for the student to state the problem in his own words.	( )	( )	( )	( )	( )
) Provides for adequate student preparation before entering the lab.	( )	( )	( )	( )	( )
) Encourages the formation of hypotheses, i.e., making predictions from observations.	( )	( )	( )	( )	( )
) Directs students in planning and developing their own procedures.	( )	( )	( )	( )	( )
) Directs students in selecting the equipment and materials that they will require, and in improvising apparatus.	( )	( )	( )	( )	( )
) Develops the logical organization of recorded data.	( )	( )	( )	( )	( )
) Gives training in processing and analyzing data, e.g., graphing and solving mathematical problems to obtain secondary data.	( )	( )	( )	( )	( )
) Develops an awareness of the limitations of measurement.	( )	( )	( )	( )	( )
) Encourages the evaluation of the experimental procedures on the basis of the results obtained, i.e., emphasizes methods as well as results.	( )	( )	( )	( )	( )
) Provides experience in cooperative planning, evaluation of group data, and in other aspects of "team research"	( )	( )	( )	( )	( )

	<u>Excellent</u>	<u>Very Good</u>	<u>Good</u>	<u>Fair</u>	<u>Poor</u>
	4	3	2	1	0
k) Cultivates good laboratory habits, e.g., safety and cleanliness.	( )	( )	( )	( )	( )
l) Develops proper techniques in manipulating chemical apparatus and in handling materials.	( )	( )	( )	( )	( )
m) Provides for individual differences among students with opportunity to extend an investigation beyond the basic experiment.	( )	( )	( )	( )	( )
n) Develops critical thinking on the part of the student.	( )	( )	( )	( )	( )
o) Frequently employs the investigation of an "unknown" material or chemical system.	( )	( )	( )	( )	( )
p) Indicates practical applications for the procedures followed.	( )	( )	( )	( )	( )
q) Requires laboratory reports that are functional in that they emphasize communication rather than needless repetition of printed instructions.	( )	( )	( )	( )	( )
r) Provides for individual growth of the students in knowledge, independent thinking and self-direction.	( )	( )	( )	( )	( )
s) Gives insight into the actualities of scientific investigation.	( )	( )	( )	( )	( )

Any comments in relation to the rating of the existing laboratory program are welcomed below.

ANALYSIS OF RATING FORM RETURNS FROM TWENTY-SEVEN  
CHEMISTRY TEACHERS

	<u>Excellent</u> 4	<u>Very Good</u> 3	<u>Good</u> 2	<u>Fair</u> 1	<u>Poor</u> 0	<u>Mean Rating</u> (4 maximum)
) Gives an opportunity for the student to state the problem in his own words. *	( 0 )	( 2 )	( 5 )	( 13 )	( 7 )	1.08
) Provides for adequate student preparation before entering the lab.	( 0 )	( 4 )	( 7 )	( 8 )	( 8 )	.96
) Encourages the formation of hypotheses, i.e., making predictions from observations.	( 0 )	( 2 )	( 4 )	( 7 )	( 14 )	.67
Directs students in planning and developing their own procedures.	( 0 )	( 0 )	( 1 )	( 9 )	( 17 )	.41
Directs students in selecting the equipment and materials that they will require, and in improvising apparatus.	( 0 )	( 4 )	( 4 )	( 16 )	( 3 )	1.33
Develops the logical organization of recorded data.	( 0 )	( 3 )	( 8 )	( 9 )	( 7 )	1.26
Gives training in processing and analyzing data, e.g., graphing and solving mathematical problems to obtain secondary data.	( 0 )	( 0 )	( 2 )	( 5 )	( 20 )	.33
Develops an awareness of the limitations of measurement.	( 0 )	( 3 )	( 7 )	( 9 )	( 8 )	1.18
Encourages the evaluation of the experimental procedures on the basis of the results obtained, i.e., emphasizes methods as well as results.	( 0 )	( 0 )	( 11 )	( 11 )	( 5 )	1.18
Provides experience in cooperative planning, evaluation of group data, and in other aspects of "team research"	( 0 )	( 0 )	( 5 )	( 7 )	( 15 )	.67

\*The numbers in parentheses indicate the rating distribution among 27 teachers.

	<u>Excellent</u> 4	<u>Very Good</u> 3	<u>Good</u> 2	<u>Fair</u> 1	<u>Poor</u> 0	<u>Mean Rating</u> (4 max.)
k) Cultivates good laboratory habits, e.g., safety and cleanliness.	( 2 )	( 4 )	( 8 )	( 11 )	( 0 )	1.74
l) Develops proper techniques in manipulating chemical apparatus and in handling materials.	( 2 )	( 5 )	( 10 )	( 8 )	( 2 )	1.74
m) Provides for individual differences among students with opportunity to extend an investigation beyond the basic experiment.	( 0 )	( 0 )	( 2 )	( 10 )	( 15 )	.52
n) Develops critical thinking on the part of the student.	( 0 )	( 0 )	( 5 )	( 11 )	( 11 )	.78
o) Frequently employs the investigation of an "unknown" material or chemical system.	( 0 )	( 0 )	( 0 )	( 2 )	( 24 )	.07
p) Indicates practical applications for the procedures followed.	( 0 )	( 0 )	( 0 )	( 5 )	( 23 )	.18
q) Requires laboratory reports that are functional in that they emphasize communication rather than needless repetition of printed instructions.	( 0 )	( 2 )	( 5 )	( 11 )	( 9 )	.93
r) Provides for individual growth of the students in knowledge, independent thinking and self-direction.	( 0 )	( 0 )	( 6 )	( 9 )	( 11 )	.78
s) Gives insight into the actualities of scientific investigation.	( 0 )	( 0 )	( 8 )	( 6 )	( 12 )	.82

## OVERVIEW OF THE INVESTIGATIVE CHEMISTRY LABORATORY PROGRAM

NO.	TITLE	CORRELATION WITH TEXT	TIME (periods)	LABORATORY TECHNIQUES (*introduced)	CONCEPTS	INTENDED PURPOSE	SOURCE	NO.
1	SCIENTIFIC OBSERVATION AND DESCRIPTION	introduction (first day of school)	1		1. quantitative and qualitative observations	<ol style="list-style-type: none"> <li>To develop skill in making and recording scientific observations.</li> <li>To emphasize the importance and value of careful observations.</li> <li>To illustrate how many observations can be made on a seemingly simple system.</li> <li>To distinguish between observation and assumption.</li> <li>To illustrate the importance of quantitative as well as qualitative observations.</li> </ol>	CHEM Study, p. 1	1
2	THE METRIC SYSTEM AND MEASUREMENT TECHNIQUES	Chapter 1	2	<ol style="list-style-type: none"> <li>measurement length* weight* (.1g balance) volume* (graduate)</li> </ol>	<ol style="list-style-type: none"> <li>precision</li> <li>accuracy</li> <li>limitations of measurement</li> <li>per cent error</li> </ol>	<ol style="list-style-type: none"> <li>To give practice in measurement techniques and graphing.</li> <li>To give practice in estimating to 0.1 of the smallest division.</li> <li>To illustrate the importance of independent measurements.</li> <li>To distinguish between precision and accuracy.</li> <li>To illustrate the value of, including the range of the data with the average result.</li> </ol>	<p>Three Reports, NSTA, p. 29</p> <p>Youden, pp. 24-32</p> <p>Weisbruch, pp. 15-17 and 21-23</p>	2
3	USING DENSITY AS AN IDENTIFYING PROPERTY	Chapter 2	1	1. measurement weight (.01 balance)*	1. density	<ol style="list-style-type: none"> <li>To show the limitations of a single physical property such as density, when used as a means of identification.</li> <li>To calculate the density of an unknown substance from its volume (displacement of water) and its mass.</li> </ol>	<p>CBA, p. 3</p> <p>Sienko and Plane, pp. 31-33</p> <p>Weisbruch, pp. 27-29</p> <p>Sanderson, p. 38</p>	3
4	USING THE BUNSEN BURNER	Chapter 4	2	<ol style="list-style-type: none"> <li>measurement temperature* length time*</li> </ol>	<ol style="list-style-type: none"> <li>controls</li> <li>variables</li> </ol>	<ol style="list-style-type: none"> <li>To discover the height at which a beaker of water should be placed above the burner to obtain the maximum temperature from the flame.</li> <li>To organize independent procedures for controlling the conditions for the experiment.</li> <li>To illustrate the importance of arriving at conclusions which are within the scope of the data collected.</li> </ol>	<p>MCA, pp. 5-6</p> <p>Weisbruch, pp. 2-4</p> <p>Sanderson, pp. 17-18</p>	4
5	MELTING POINT OF A PURE SUBSTANCE	Chapter 2	2	<ol style="list-style-type: none"> <li>measurement temperature time</li> <li>heating test tube*</li> </ol>		<ol style="list-style-type: none"> <li>To make a quantitative study of a phase change.</li> <li>To illustrate the value of graphing experimental data.</li> <li>To discover the similarity of the flat portions of the heating and cooling curves and to identify the melting and freezing points.</li> </ol>	<p>CHEM Study, pp. 4-6</p> <p>MCA, pp. 10-11</p>	5
6	AN INVESTIGATION INTO PHYSICAL AND CHEMICAL CHANGES	Chapter 3	2	<ol style="list-style-type: none"> <li>measurement temperature volume (pipette)*</li> <li>handling reagents</li> </ol>	<ol style="list-style-type: none"> <li>physical changes</li> <li>chemical changes</li> </ol>	<ol style="list-style-type: none"> <li>To exemplify the association of various types of laboratory observations with chemical changes.</li> <li>To extend the skill in identifying chemical changes into the study of "unknown" systems.</li> <li>To develop further skill in recording observations.</li> </ol>	<p>Taylor, et al, pp. 17-20</p> <p>CBA, pp. 4-5</p>	6

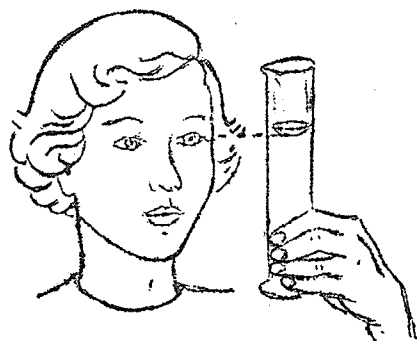
NO.	TITLE	CORRELATION WITH TEXT	TIME (periods)	LABORATORY TECHNIQUES (*introduced)	CONCEPTS	INTENDED PURPOSE	SOURCE	NO.
7	HEATING EFFECTS OF A PHYSICAL AND CHEMICAL CHANGE	Chapter 3	2	1. measurement temperature weight	1. calorimetry 2. magnitude of energy associated with physical and chemical changes	1. To contrast the energy involved in a phase change and in a chemical change. 2. To provide further experience in quantitative investigation. 3. To compare class data in order to arrive at a generalization.	CHEM Study, pp. 9-13	7
8	THE PROCESSING OF RAW DATA	Chapter 2	homework		1. stoichiometry 2. Law of Definite Proportions	1. To show in the reactions of a solid and a gas the relationship between the weight of the gas remaining and the initial volume of the gas. 2. To give further experience in processing and interpreting data.	CBA, pp. 8-9	8
9	THE FORMATION OF A PRECIPITATE--A QUALITATIVE OBSERVATION OF A CHEMICAL CHANGE	Chapter 2-3	2	1. filtration* 2. heating (beaker)* 3. measurement volume	1. stoichiometry 2. flow sheet	1. To illustrate that when two solutions react to form a precipitate, any excess of either will not react. 2. To discover which solution was in excess. 3. To introduce the flow sheet as a means or recording laboratory procedures and observations.	CBA, pp. 13-15	9
10	OXYGEN--A VERIFICATION EXPERIMENT	Chapter 4	2	1. gas generation and collection* 2. heating 3. inserting glass tubing*	1. verification	1. To study the preparation and collection of oxygen. 2. To study the function of manganese dioxide in the preparation of oxygen. 3. To verify the properties of oxygen. 4. To appreciate the value and the place of the verification experiment.	Weisbruch, pp. 71-73 McGill and Bradbury, pp. 63	10
11	PROPERTIES OF HYDROGEN --A VERIFICATION EXPERIMENT	Chapter 5	2	1. gas generation and collection 2. inserting a thistle tube into a stopper*		1. To study the preparation and collection of hydrogen gas and to verify some of the properties of the gas. 2. To study the function of a particular piece of apparatus, the thistle tube.	Taylor, et al, pp. 75-77 McGill and Bradbury, p. 64	11
12	CONSTRUCTION OF A LOGICAL MODEL	Chapter 9-10	1		1. logical models 2. black box	1. To illustrate the method of the construction of a logical model from indirect observations and assumptions. 2. To illustrate that chemistry is as much a way of thinking and handling observations as of manipulating tubes and reagents. 3. To give practice in evaluating data and discarding inconsistent items.	CHEM Study, p. 65 CBA, pp. 1-2	12
13	DISTILLATION	Chapter 7	2	1. assembling apparatus 2. use of bunsen burner	1. fractional distillation	1. To give examples of the distillation process. 2. To discover the concept of fractional distillation 3. To illustrate the importance of cleanliness of apparatus.	McGill and Bradbury, pp. 71-72 Weaver, pp. 69-70 Black, pp. 33-34	13
14	SEPARATION OF A MIXTURE INTO ITS COMPONENTS	Chapter 2 and 7	2	1. filtration	1. analysis 2. hypothesis formation	1. To devise an independent procedure for the separation of a mixture into its dry components. 2. To test and evaluate this procedure.	Garrett, p. 7 Weisbruch, pp. 63-64 Dull, pp. 11-12 Sulcoski, pp. 13-14	14

NO.	TITLE	CORRELATION WITH TEXT	TIME (periods)	LABORATORY TECHNIQUES (*introduced)	CONCEPTS	INTENDED PURPOSE	SOURCE	NO.
15	MAKING A SOLUBILITY CURVE--TEAM RESEARCH	Chapter 8	2	1. measurement weight temperature	1. solubility 2. team research 3. interpolation	1. To determine quantitatively the effect of temperature upon the solubility of an unknown salt. 2. To provide experience in organizing all data tables and graphs independently. 3. To illustrate the value of team research and the plotting of group data.	MCA, pp. 32-33 Hall, et al, p. 60 Sulcoski, pp. 35-36	15
16	MAKING A SOLUBILITY CURVE--AN INDEPENDENT INVESTIGATION	Chapter 8	2	1. measurement weight volume temperature	1. solubility 2. saturated solution	1. To determine quantitatively the effect of temperature upon the solubility of an unknown salt. 2. To evaluate the different procedures applied in Experiment 15 and 16.	Brescia, et al, pp. 29-30 Dorf and Lempkin, pp. 48-49 Sienko and Plane, p. 109	16
17	AN INVESTIGATION INTO THE PROPERTIES OF CRYSTALS	Chapter 8	2	1. heating	1. hypothesis 2. water of hydration	1. To devise and test a method for detecting whether a crystalline compound has water chemically or physically bound to it. 2. To evaluate the proposed method and to suggest improvements.	Weisbruch, pp. 97-99 Dull, pp. 55-56	17
18	AN INVESTIGATION OF WATER OF HYDRATION IN CRYSTALS	Chapter 8	1	1. measurement temperature	1. efflorescence 2. deliquescence 3. water of hydration	1. To investigate the behavior of crystals upon exposure to the atmosphere. 2. To investigate the concept of water of hydration.	Watt and Morgan, pp. 75-76	18
19	THE PERCENTAGE OF WATER OF HYDRATION	Chapter 8, 12	2	1. measurement weight	1. heating to constant weight 2. per cent error	1. To determine the percentage of water of hydration in an unknown crystalline salt. 2. To introduce the concept of heating to a constant weight	numerous sources	19
20	REACTIONS BETWEEN OXIDES AND WATER--TEAM RESEARCH	Chapter 7, 19	2		1. regularities of chemistry 2. verification of a conclusion 3. negative conclusion	1. To discover which oxides react with water; where a reaction does occur, to determine the type of compound formed. 2. To organize the data and to seek regularities in it by reference to the Periodic Table, the Activity Series, etc. 3. To verify the conclusion by prediction and checking.	MCA, pp. 14-15	20
21	THE IDENTIFICATION OF FIVE UNKNOWN SOLUTIONS	Summary of descriptive chemistry	1	1. drop reactions	1. qualitative analysis	1. To identify five unknown solutions by investigating their chemical properties.	CBA, p. 43 CHEM Study, p. 36 McGill and Bradbury, pp. 309 DeBruyne, pp. 265-6	21

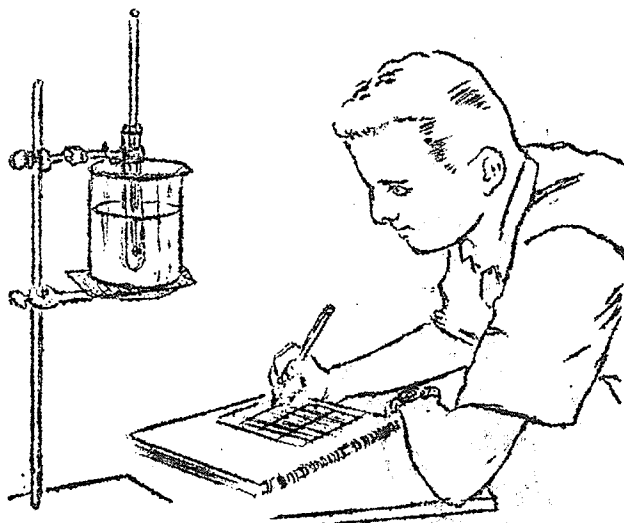


NO.	TITLE	CORRELATION WITH TEXT	TIME (periods)	LABORATORY TECHNIQUES (*introduced)	CONCEPTS	INTENDED PURPOSE	SOURCE	NO.
22	MOLECULAR WEIGHT DETERMINATION	Chapter 13	2	1. heating 2. measurement volume weight	1. the mole	1. To determine the molecular weight of an unknown volatile liquid by the Dumas method.	Sienko and Plane, pp. 85-87 Garrett, et al, pp. 85-86 Hoffman, p. 129 Brescia, et al, pp. 43-46 Sanderson, pp. 59-60	22
23	DETERMINATION OF THE VOLUME OF GAS PRODUCED BY A REACTION	Chapter 14	2	1. collection and measurement of gases* 2. measurement of barometric pressure*	1. vapor pressure	1. To make a quantitative investigation of a chemical reaction. 2. To determine the volume of hydrogen gas produced in the reaction between magnesium and hydrochloric acid. 3. To discover the concept of vapor pressure correction.	Brescia, et al, pp. 63-64 CHEM Study, pp. 27-30	23
24	ANALYSIS OF AN UNKNOWN MIXTURE	Chapter 14	1	1. heating measurement weight	1. heating to constant weight 2. stoichiometry	1. To calculate the percentage by weight of potassium chlorate in a mixture of potassium chlorate and potassium chloride. 2. To illustrate the value of weight relations from chemical equations.	Sienko and Plane, pp. 67-70 Black, pp. 39-40 MCA, p. 13	24
25	PREPARATION OF A SOLUBLE SALT	Chapter 14, 19	2	1. evaporation 2. measurement weight volume	1. stoichiometry 2. per cent yield	1. To provide experience in devising an independent method for the preparation of a given weight of sodium chloride from either a carbonate or a bicarbonate. 2. To test and evaluate the proposed method, suggesting improvements.	Brescia, et al, p. 65 Garrett, et al, MCA, p. 52	25
26	A DETERMINATION OF THE DENSITY OF OXYGEN	Chapter 13	2	1. heating 2. assembling apparatus 3. measurement volume weight temperature	1. apparatus design	1. To determine the density of oxygen gas. 2. To provide experience in the investigation of the significance of different aspects of experimental procedure and apparatus design.	Hoffman, pp. 123-125 Brescia, et al, pp. 33-36 Dull, pp. 35-36 Sulcoski, pp. 44-46 Malm and Franz, pp. 99-102	26

APPENDIX C



# INVESTIGATIONS IN CHEMISTRY



CHURCHILL HIGH SCHOOL  
WINNIPEG

SECOND TRIAL EDITION  
SEPTEMBER 1963

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## AN INTRODUCTION TO THE LABORATORY

The purpose of any introductory laboratory work is either to begin the real training of a potential scientist or to help the student to understand what science is really like. For all of you, this is an opportunity to learn chemistry from a point of view which you could never obtain from class discussions, observing demonstrations, or watching films. This laboratory provides you with the opportunity to achieve an understanding of chemistry which all the books in the world could not provide, the chance to see with your own eyes, and to work with your own hands, in order to gather information which will lead to the solution of a problem through scientific thinking. For those of you who are undecided about the future this experience will clarify your thinking as to your possible role in the advancing scientific world.

All scientists are intelligent people, especially those who are chemists. No doubt, you have already observed that if a person is intelligent he or she can usually earn a rather good salary while working at a job that is pleasant. Also, you have probably heard that chemistry is difficult. Why then should an intelligent person undertake the study of a difficult subject, such as chemistry, when he or she could more easily master a simpler subject and eventually earn as much money and live just as comfortably? The answer is simple; although chemistry is difficult, it is fascinating. Particularly in the laboratory and in the thinking associated with the laboratory work, it is an enjoyable experience.

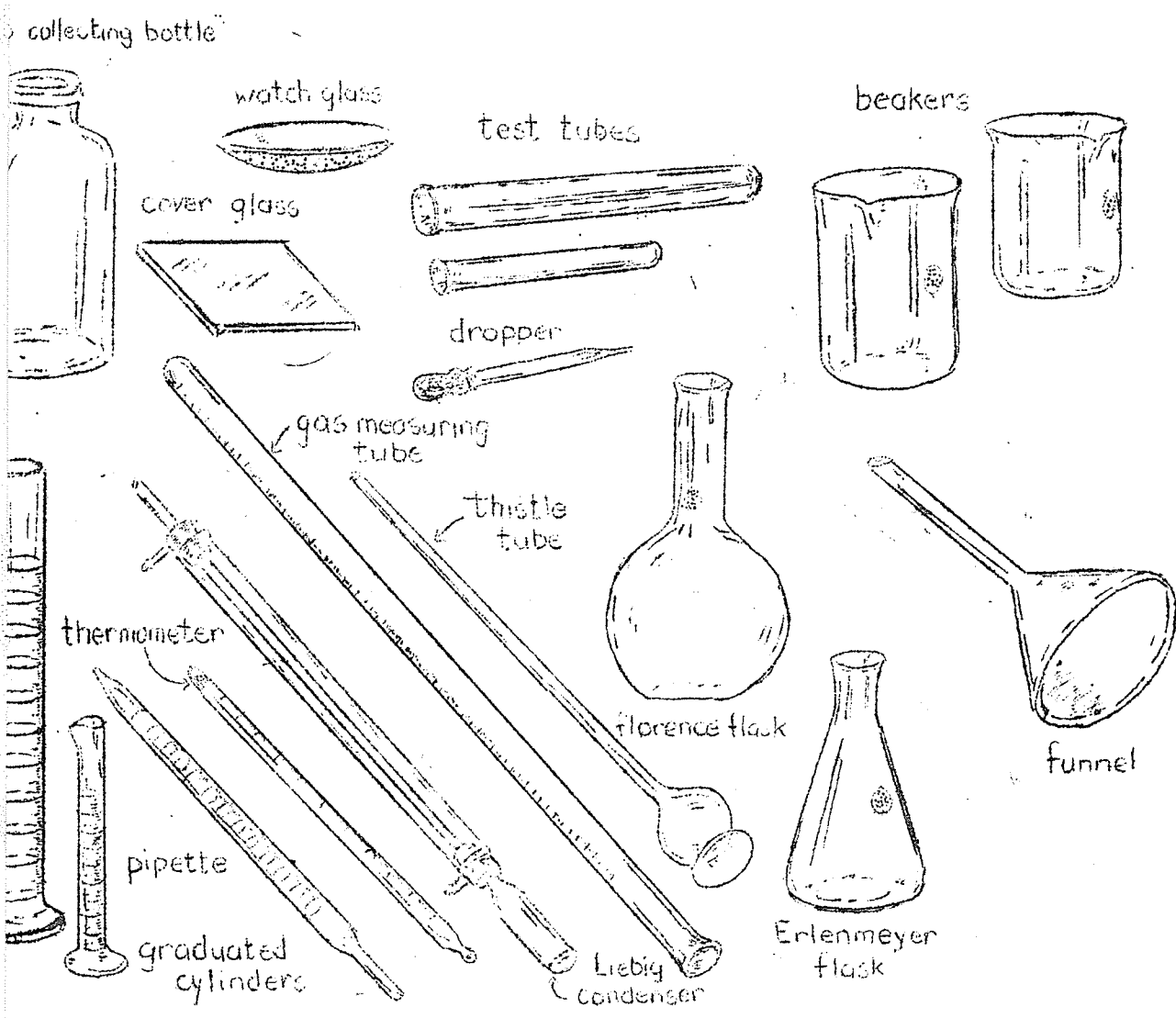
It is hoped that the experiments in this manual will cause you to think out problems, perform experiments to find answers, and get some fun out of the process. Work hard and enjoy your laboratory experiences!

AN INTRODUCTION TO LABORATORY APPARATUS

During the course of the year you will use, very likely, most of the common laboratory apparatus shown in the accompanying figures and described in the following paragraphs. Such apparatus is so generally useful in chemistry that it is found not only in high school laboratories but also in the laboratories at the universities and in all kinds of industrial laboratories as well. Familiarize yourself with the name, design, and use of each piece of apparatus.

GLASSWARE

Laboratory glassware is made of soft or hard (Pyrex) glass, according to the purpose for which the piece of apparatus is intended. Because pyrex glass softens at a higher temperature than soft glass and because pyrex glass withstands rapid changes in temperature, only glassware made of pyrex glass should be heated.



Reaction Vessels. Beakers, test tubes and flasks are primarily used as reaction vessels and as containers. Their transparency of course makes it possible to view the materials within from all sides. The specific piece of apparatus used for any particular operation depends on the nature and size of the substances involved. The volumes indicated below the pyrex trademark on these pieces of apparatus are rough approximations and are to be used only in crude estimations of volume.

A-1. Beakers. These are the most widely used articles in chemistry laboratories. They come in a wide range of sizes from a few ml to several liters capacity. Their smoothness and regular shape make them easy to clean; their relative wideness makes the materials inside easily accessible and their indented lip provides for convenient pouring.

A-2. Test tubes. Perhaps these are the most familiar of all scientific equipment. They are simply small glass tubes, closed and rounded at one end and usually flared slightly at the other. They are appropriately named since they are conveniently used to contain very small quantities of materials for small scale tests or experiments.

A-3. Florence flasks. Having miscellaneous uses, these vessels serve as containers of liquids, and as reaction vessels for many chemical reactions. They are both with round and flat bottoms. The former are mechanically stronger and more suitable for operations requiring addition of heat since liquids are less likely to "bump" in round bottom flasks.

A-4. Erlenmeyer flasks. These vessels have many uses in chemistry. They offer a wide base which can be uniformly heated, and which makes them unlikely to be tipped over when filled. They are useful for long boiling operations when it is desired to retain as much solution as possible; the tapered walls help to condense and to return the vaporized liquid. They are especially effective for mixing when adding some reagent drop by drop; the mixing action is one of swirling rather than shaking.

Volume Measuring Devices. Volume measuring devices are usually not pyrex; therefore, they should neither be heated nor, ordinarily, used as reaction vessels.

A-5. Graduated cylinders. The volumes of liquids are commonly measured in cylindrical glass vessels with graduated scales on their sides. They are often called "graduates", and are obtained in various capacities. The scale etched in the glass shows the volume in milliliters (or cubic centimeters). Some have a single scale in which the divisions are numbered from the bottom upwards, enabling the observer to measure the volume of liquids poured into the graduate. Others have a double scale, the divisions in the second scale being numbered from the top downward, thus making it possible to measure the volume of the liquids poured from the graduate.

A-6. Pipettes. The pipette is a glass tube tapered to a point at one end; it is used for more accurate measurements of volume. A transfer pipette has a bulb-like enlargement near its center and one etched mark near the top. It is calibrated to deliver a specified volume of liquid at the temperature indicated and is convenient for measuring out a number of samples of the liquid which are identical in size. A measuring pipette is graduated, usually in tenths of a milliliter and is used for the transfer of exact volumes of liquid. It is particularly convenient when the samples of liquid vary in size.

A-7. Gas measuring tubes. These are long cylindrical devices which are used to measure volumes of gases which are produced by a reaction. These tubes are usually of 50-100 milliliter capacity and are graduated to a tenth of a milliliter.

A-8. Dropper. This device provides a means of adding small quantities of liquids dropwise, rather than from any test tube or beaker. It may be used as a dropping pipette for small-scale (semi-micro) tests, once the number of drops which are equivalent to one milliliter has been determined.

#### Miscellaneous Glassware

A-9. Funnels. In the process of filtration these are used to support a porous paper called filter paper, which allows liquids to pass freely through the pores but retains the particles of solid which are not dissolved. The size of a funnel is indicated by the cross sectional diameter at the top and by the angle of the cone.

A-10. Thistle tubes. The long slender stem with an open bulb at the top is indispensable for the addition of reagents into air-tight vessels for the generation of gases. The gases generated are prevented from escaping up through the thistle tube by the weight of the column of reagent in the stem.

A-11. Thermometers. Although many devices are available for measuring temperature, the mercury-filled thermometer is the one most commonly used in an introductory laboratory.

A-12. Watch glasses. These are used as covers for beakers, as vessels in which to weigh solids, or as shallow evaporating vessels. A watch glass may be warmed gently, but thermal stresses caused by uneven heating or more intense heat may cause them to break suddenly and fly apart. Never apply a flame directly to one.

A-13. Gas Bottles. These are used as containers in which to collect gases liberated during chemical reactions. They may also be used as containers where soft glass vessels would be satisfactory.

A-14. Glass plates. These pieces of glass serve as a "cover glass" for sealing off a gas bottle, as surfaces on which to mix powders or pastes, or as surfaces on which to rest the bottles of corrosive reagents.



A-15. Condenser. This is a tube which is cooled on the outside, in order to remove heat from, and condense, vapors admitted to the inside. The condenser shown is a simple water-jacketed (Liebig) condenser. A stream of cold water is run through the jacket to carry away the heat. (The whole process of boiling or evaporation, letting the vapor flow elsewhere and then condensing it, is called distillation.) The condensed vapor is called the distillate.

REFRACTORIES are materials not damaged by high temperatures.

A-16. Crucible. The crucible is a small cup made of porcelain, a special clay which had been heated until the outside melted and became like glass. It will stand higher temperatures than pyrex glass and will not soften at the highest Bunsen burner temperature. It is commonly used for high temperature reaction of solids and as containers in which to fuse (melt) solids.

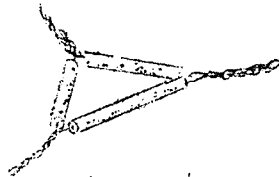
A-17. Evaporating dish. This dish is usually made of porcelain. It is used as a vessel in which to evaporate a solution to smaller volumes or to dryness.

A-18. Clay triangle. Usually placed across the ring on a lab stand, it serves as a crucible support during heating; and as a support for funnels.

A-19. Asbestos pads. Asbestos being a good insulator, these pads are used as surfaces on which to rest hot glass tubing or vessels.



Crucible + lid



clay triangle



evaporating dish

### IRONWARE

A-20. Bunsen burner. This type of burner is almost indispensable as a source of heat and is used in most laboratories. Various models are available, but all are so constructed that air is admitted into the gas stream and, therefore, a mixture of air and gas can be ignited at the top of the vertical barrel. The nature and temperature of the flame are controlled by the relative volumes of gas and air admitted.

Each type of burner the same parts in common; although specific differences exist.

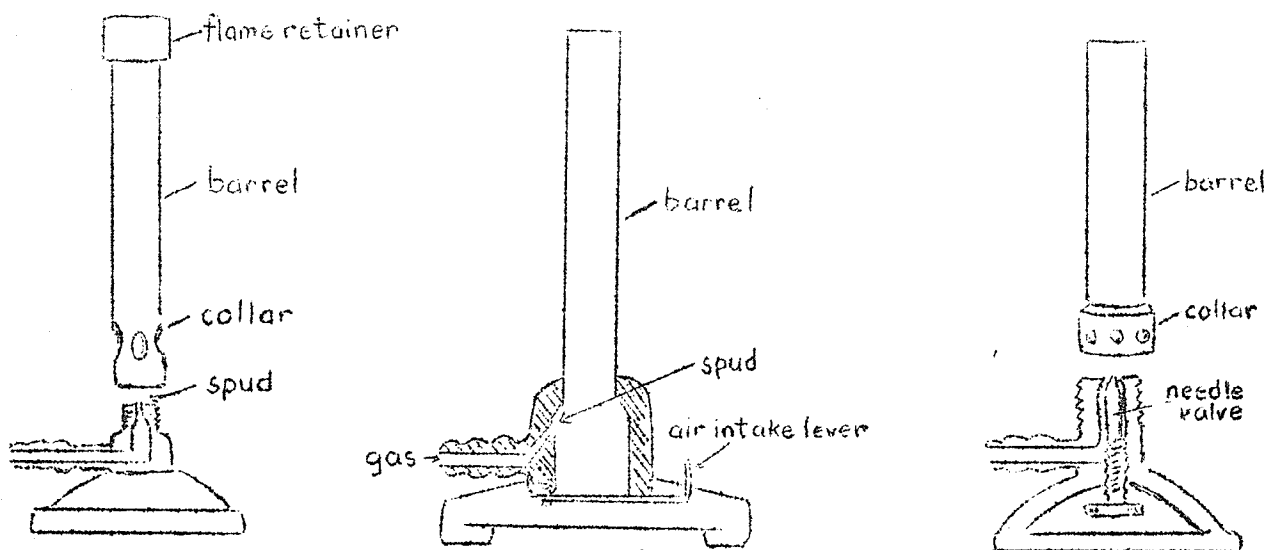
The orifice or spud is a small opening through which the gas enters. The collar with the air holes can be turned up or down, thus controlling the air intake.

The barrel permits the mixing of the air and gas before burning at the top.

The flame retainer bleeds off some of the air-gas mixture and feeds the flame from the outside. This serves to prevent the flame from

blowing out.

## Various Types of Burners



A-21. Wingtop or flame spreader. It fits on the top of your burner and provides a wide spread flame which makes it easier to heat a length of glass tubing. Its use is generally restricted to glass bending.

A-22. Test tube holder. As the name implies, this device is used to support a test tube. Frequently the application of heat to the bottom of a test tube is desired. Glass is not a good conductor of heat so it is possible to hold the top of the tube in the fingers and the bottom in the flame. However, the heat does travel upward, and especially if the liquid contents suddenly boil over, or if vapors rise and condense on the upper part of the tube, a test tube holder is much more convenient and desirable to use. Furthermore, such a holder is useful when pouring corrosive liquids into or out of a test tube, where slight awkwardness may result in spilling down the outside the tube.

A-23. Crucible tongs. These are used for the handling of hot crucibles.

The iron stand, iron ring clamp, wire gauze, and the utility clamp, in combination, form structural supports for many of the common laboratory operations.

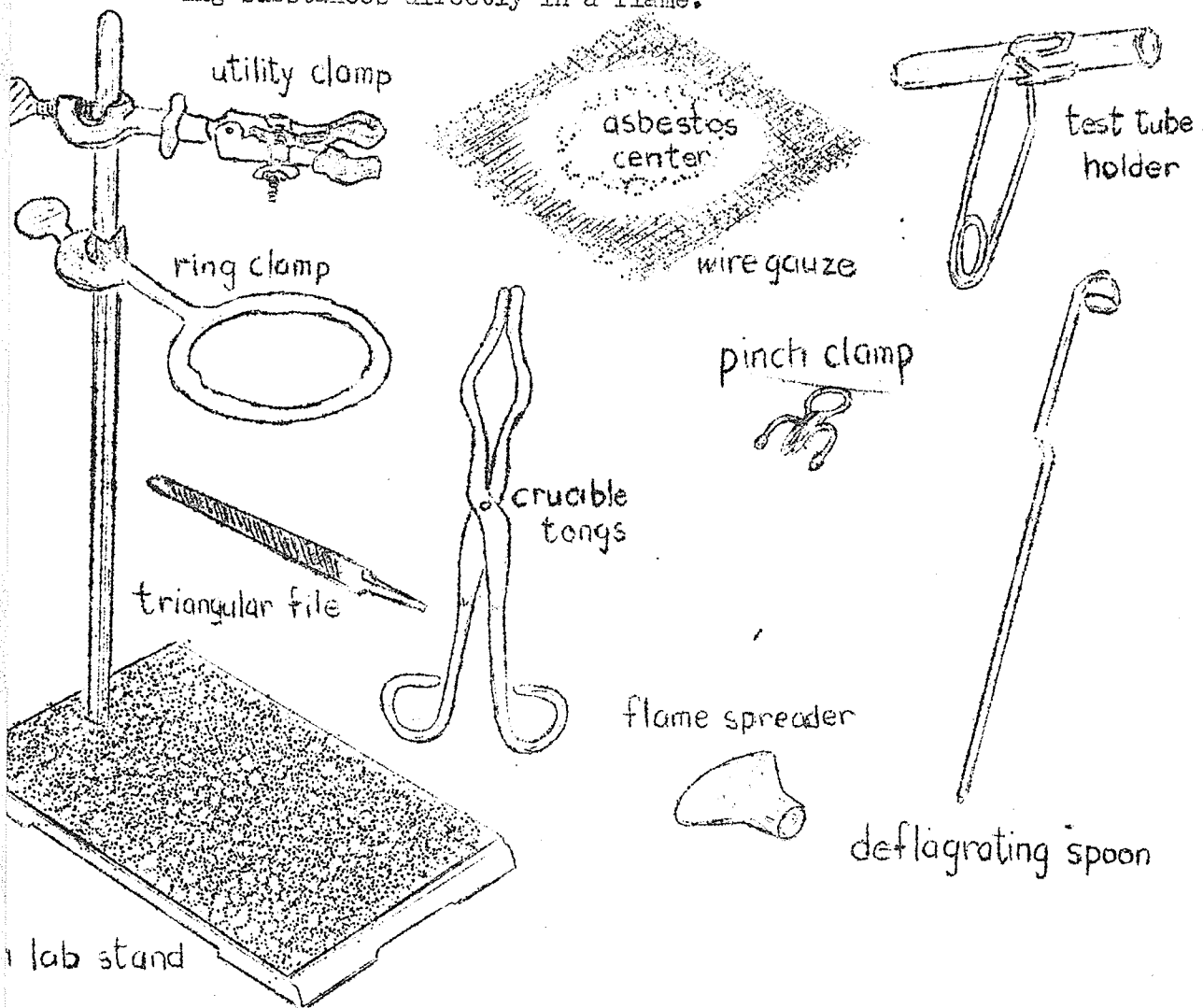
A-24. Wire gauze. Even though pyrex glass is quite resistant to thermal stress, it is too much to expect a beaker to stand the full heat of a gas flame suddenly applied to one spot on its bottom. For this reason, a small square of wire screen or gauze is usually placed between the beaker and the flame--the beaker resting on the gauze--which spreads the heat more uniformly over the entire bottom surface of the beaker and lessens the chance of breakage from uneven heat. The gauze, of course, is in turn supported on an iron ring attached to the iron stand.

A-25. Utility clamp. There are several sizes and kinds of clamps. One of the more useful is the utility or universal clamp, so called because it can be adjusted over a wide range, in direction as well as jaw opening. Most clamps require a clamp holder to support them on an iron stand, but the utility clamp has its own built-in clamp holder for this purpose. The clamp jaws are covered with rubber or asbestos in order to support firmly and yet without danger of cracking the glass of test tubes, etc.

A-26. Iron file. The triangular file is primarily for cutting glass.

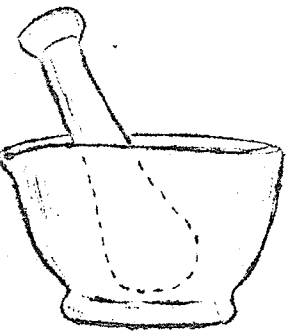
A-27. Pinch clamp. It is used to regulate the flow of a fluid through rubber tubing.

A-28. Deflagrating spoon. Also known as a combustion spoon, it is used for heating substances directly in a flame.

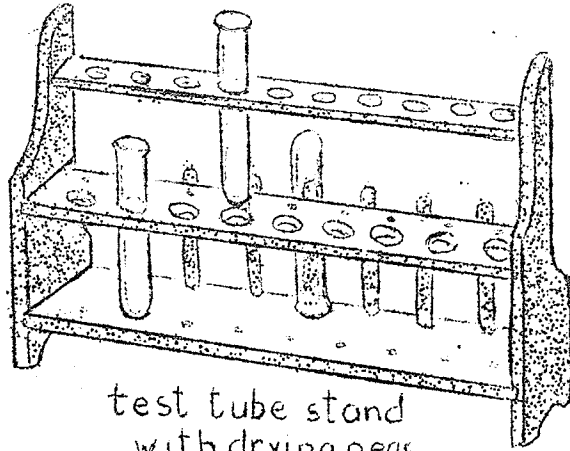


MISCELLANEOUS APPARATUS

- A-29. Mortar and pestle. Composed of porcelain or glass, this apparatus is used for grinding solids to a powder.
- A-30. The test tube rack. This is a wooden support for your test tubes while they contain materials being studied. Some racks also provide for the storage of test tubes upside down to allow them to drain dry after cleaning.
- A-31. Corks and rubber stoppers. Both kinds of stoppers find wide use in chemical experiments. Corks are cheaper, more easily drilled for inserting glass tubing and various apparatus and insoluble in ordinary liquids. However, they are badly attacked by many corrosive chemicals, and they are not entirely gas-tight. Rubber stoppers, on the other hand, may be made gas-tight and generally they are more resistant to chemical attack although some materials attack them easily. Organic solvents, such as ether, are more likely to be absorbed by, or to soften, or even dissolve some of the rubber, whereas cork is more resistant. Neither cork nor rubber will stand excessive heat. Both will decompose or burn. Rubber stoppers are usually better at staying firmly wedged in place. Stoppers are classified according to size and number of holes.
- A-32. Rubber tubing. This is indispensable for making flexible passages between fixed tubes and for connecting two fixed tubes together. You may use it for conducting the gas to your burner, the cold water to your condenser, and making gas-tight interconnections in your experimental apparatus. It is of course susceptible to the same kind of chemical and solvent attack mentioned for rubber stoppers.
- A-33. Glass tubing and rod. Miscellaneous pieces of glass tubing -- which of course is hollow -- are extremely useful if not indispensable in experimental work in a chemistry laboratory, serving as pipelines for the flow of fluids. Tubing is not to be confused with rod, which is solid and has more limited application, e.g., stirring rod.
- A-34. Centrifuge. This is a device which speeds up the rate at which a precipitate settles to the bottom of a solution. As a motor spins the centrifuge, the test tube and solution are going around in a circular path at high speed. This motion produces a force (centrifugal) that acts on the particles of the precipitate in a direction away from the center of the circular path. In an ordinary laboratory centrifuge with a radius of about ten centimeters and a speed of 1800 to 2000 revolutions per minute, the centrifugal force is from 350 to 450 times stronger than the force of gravity. Thus a precipitate which would take three hours to settle if left standing, will settle out in 25 to 30 seconds in the centrifuge.



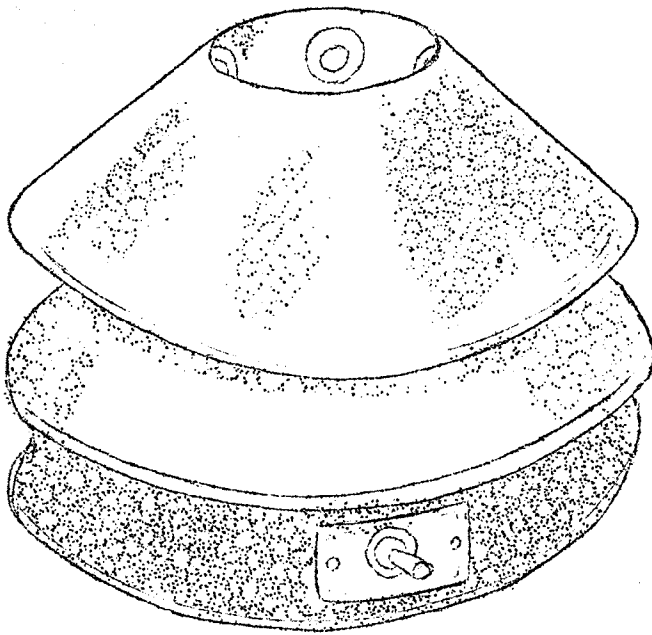
mortar + pestle



test tube stand  
with drying pegs



test tube  
brushes



centrifuge

Refer to this section of the laboratory manual often to familiarize yourself with the name, design and use of each piece of apparatus.

## AN INTRODUCTION TO BASIC LABORATORY TECHNIQUES

In this portion of the manual, information is given relative to some of the basic techniques which are used in a chemistry laboratory. A technique is a particular method of performing a mechanical operation or manipulation. Basic laboratory techniques have been established by thousands of chemists as the result of many years of experience. Observe these techniques carefully; take pride in them.

In the laboratory it is almost always more convenient and more efficient to experiment with quantities as small as can be handled with the apparatus available. It is also much safer. Consequently experiments in chemistry are performed on as small a scale as possible. However this calls for greater skill and care than would be necessary for larger scale work. If you measure out twenty-five pounds of a substance, you can usually afford to overlook a teaspoon or so that may spill. On the customary laboratory scale of experiment, however, you may use much less than a teaspoon of all the materials put together. Accordingly, chemists have developed special techniques for studying small amounts of material exactly. During the course of the year you will study many of them.

This information has been included to supplement the instructions given in the actual experiments. For most of the techniques, the reasons for using certain procedures are stated. Possible difficulties are mentioned and suggestions are made as to how they can be avoided and corrected. You can profit by their imitation, not only by learning to avoid personal hazard, but also by learning to obtain the largest amount of information in the shortest period of time and with the least amount of effort expended.

Reference will be made to the particular techniques required in each experiment. Refer back to these pages frequently in preparation for each experiment until such time when the techniques are mastered.

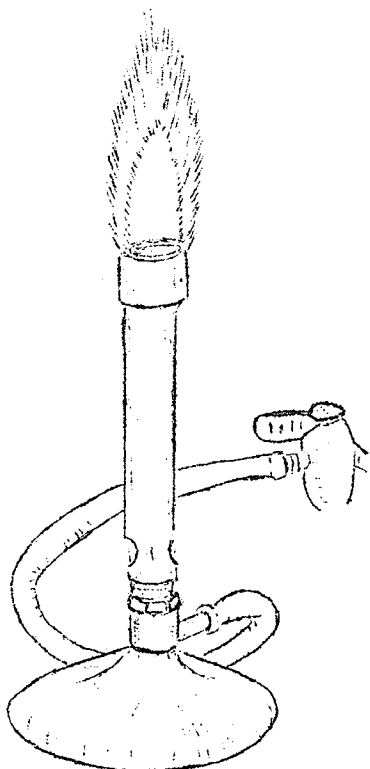
### T-1. MANIPULATION OF A BUNSEN BURNER

#### Lighting the burner

Adjust the air supply near the base of the bunsen burner so that the holes are about one-quarter open. Then turn on the gas. Hold the flint lighter and strike it by applying an upward pressure on the moveable arm by means of the thumb. Adjust the height of the flame by turning the gas cock. The best flame is one of moderate height which is getting as much air as can be supplied without it beginning to flutter or roar.

#### Non luminous and luminous flames

When the combustion of the air-gas mixture is complete, the flame burns almost noiselessly and is almost non-luminous with two distinct cones being apparent. When the combustion is incomplete the incandescent particles of carbon are formed which produce the luminous flame and which are deposited on any apparatus held in the flame. Incomplete combustion of the mixture causes the flame to burn with a bright yellow color. This flame is cooler than the ideal flame of the complete combustion.



This condition can be corrected by increasing the concentration of air in the mixture; i.e. screwing collar or the barrel away from the base of the burner until the flame approximates the ideal one.

Strike back. A high concentration of air in the mixture causes the flame to burn noisily in the barrel of the burner and, in extreme cases to strike back. If the flame strikes back, or burns inside of the barrel near the air adjustment, turn the gas off for a moment. Do not touch the hot barrel. Allow the burner to cool, reduce the air intake and relight the burner.

A flame may burn "off" the burner if the gas intake speed should be too high. This can be corrected by reducing the gas intake.

Flame-spreader. To use the flame spreader, place it on the burner when the gas is turned off. Light the burner and adjust the flame until it is about two inches high. The flame should have a distinct inner cone, and the upper edges should be even, rather than saw-toothed.

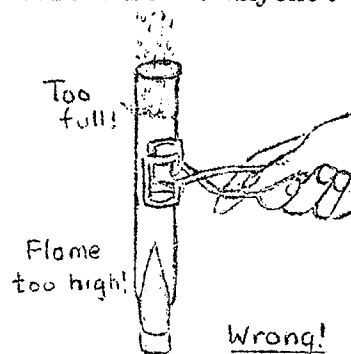
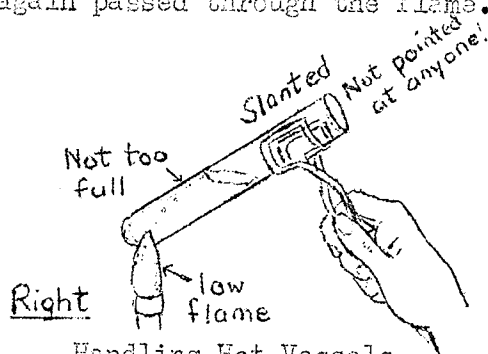
## T-2 HEATING TECHNIQUES

Liquids can be heated in test tubes, flasks or beakers, however, the vessel used should have a capacity of two to three times greater than the volume of the liquid to be heated.

Vapors generated during the heating process may cause bumping; that is, noisy and sometimes violent motion often accompanied by expulsion of the liquid from the container. This may be prevented or reduced by constantly swirling the liquid as it is being heated (as in a test tube) or by placing boiling chips (small pieces of clay plate or glass beads) in the liquid).

Large containers such as beakers and flasks are placed on a wire gauze held on a lab stand. A gentle non-luminous flame is used. The level of the vessel in the flame is adjusted beforehand in order to conserve time and fuel. No boiling vessel is left unattended. Direct contact of the flame with the part of the vessel about the liquid level should be avoided. Once the water is heated to boiling, the flame is adjusted so that the water continues to boil gently.

Test-tubes are heated using a gentle non-luminous flame. The outside of the test tube is dried before heat is applied. Supported by a test tube holder, the tube is passed back and forth through the flame at an angle so that the flame strikes it from the side rather than directly underneath. Heating the portion of the tube above the liquid level is to be avoided. Once boiling begins, the frothing is allowed to subside before the tube is again passed through the flame. Never point the test tube at anyone!



#### Handling Hot Vessels

A test tube holder should be used in pouring a hot liquid from a test tube; or it may be used in handling a hot flask.

A good method for pouring boiling water from a beaker is to fold a cloth or paper towel and wrap it around the beaker in one turn. Grasp the two loose ends of the cloth (or towel) in the fingers of one hand in such a way that a pull is exerted between the cloth. This improvised holder enables the experimenter to pour boiling liquid from a beaker into a test tube.

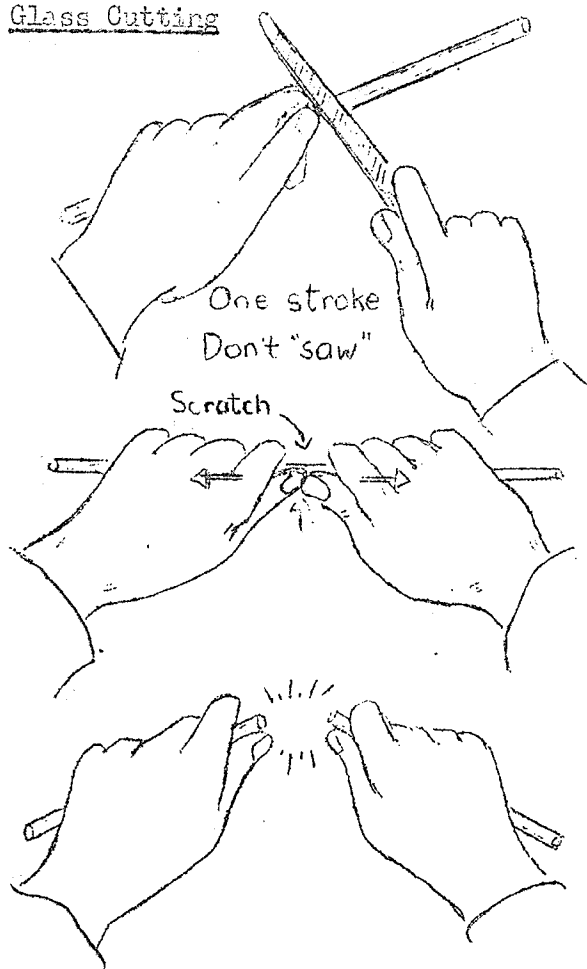
Heating to constant weight: to get a reproducible weight free from error caused by unknown content of moisture or other variable volatile impurities, it is necessary that the vessels and contents be heated to a constant weight. After heating to the desired temperature, the vessel and contents are cooled and weighed. The vessel is then reheated, for a shorter period, cooled and reweighed. This process is repeated until the weight remains constant within limits of the required precision.

### T-3 GLASSWORKING

Glass is not a true crystalline solid and therefore doesn't have a definite melting point. In this respect it more closely resembles an extremely viscous (thick) liquid which gradually softens when heated. It is this property which makes glassworking possible.

Glass used in chemistry laboratories is of two main types: soda-lime or soft glass and pyrex glass. The kind provided for your use in the form of tubing and rod is soft glass. Your gas burner does not provide a high enough temperature for working pyrex glass.



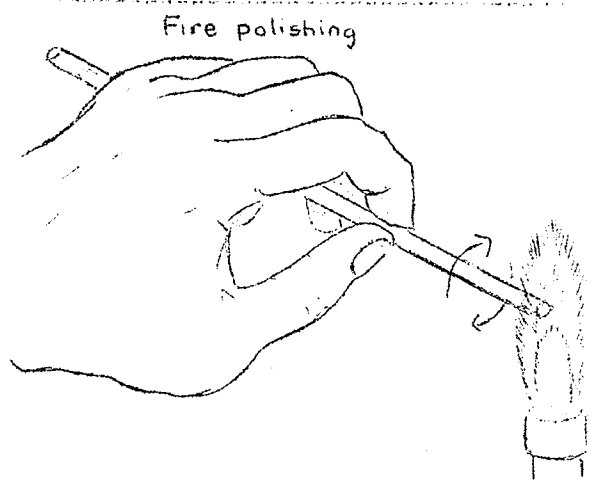
Glass Cutting

Glass tubing up to the size of your finger can be cut in two (actually, smoothly broken) very easily. A sharp file is needed. Lay the tubing flat on the bench and with a single firm motion make a sharp scratch at the desired point of cutting, with the file. Do not attempt to saw the glass. Quickly moisten the scratch with a little saliva. This sets up a strain in the glass and enables a cleaner break to be made.

Next, hold the tubing in both hands with the file mark away from you, and your thumbs one on each side of and opposite to the file mark. With your elbows close in at your sides, gently push with your thumbs and pull back and apart with your hands.

Fire Polishing

The tubing edges at the point of cutting are always very sharp and should be smoothed before using the tubing, especially when you need to insert the glass into rubber tubing or through a hole in the stopper.



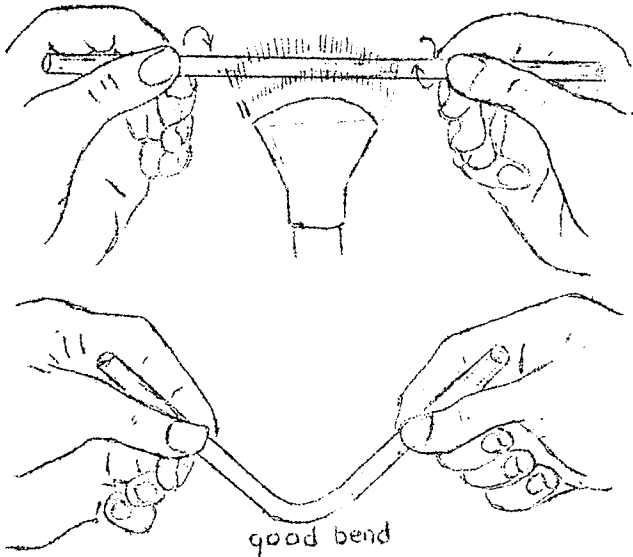
To fire-polish the freshly cut edges, hold one end of the glass tubing in the hottest part of the flame and rotate the tube back and forth between your fingers until the glass is soft enough to smoothen out by surface tension and the edges are rounded. Do not fire-polish so long that the end of the tube begins to close.



Caution:

Hot glass looks identical to cool glass. Place the hot glass on a wire gauze or asbestos pad to cool. Think before grasping a piece of worked glass, remember that hot glass cools very slowly.

Never put a piece of wet glass tubing into the flame. If the piece doesn't shatter then and there, it probably will later, when the air inside cools and the water condenses on the piece of glass that is still hot.

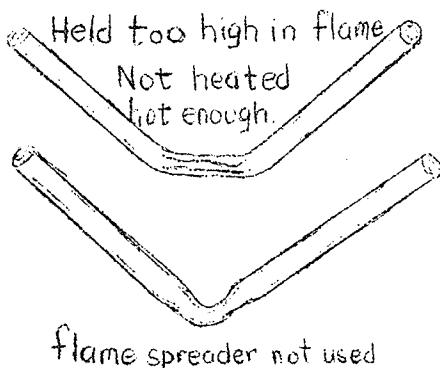
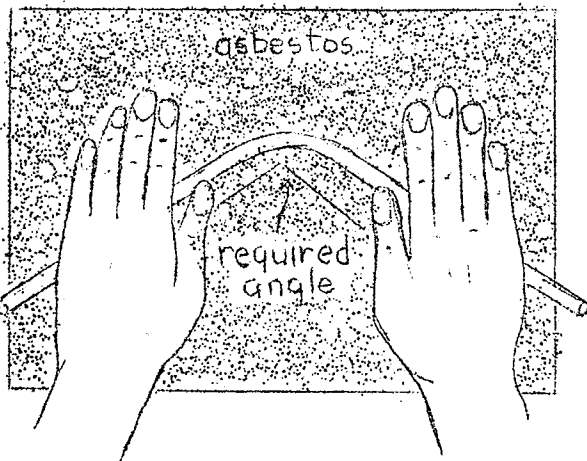
Glass Bending

The glass tubing is held in the spread flame at the point where is desired. Rotate the tube evenly at moderate speed to provide uniform heating. Continue until the flame is quite bright and the tubing begins to sag or be pliable. (The secret of successful glass bending is to get the glass sufficiently hot.) Remove the tubing from the flame and bend the glass by raising the ends to obtain the desired angle. Since the hot glass tends to sag, bending it in this manner produces a uniformly smooth bend.

If an exact angle is required, the hot glass may be bent while resting on an asbestos pad, where the desired angle was previously measured and marked. This method would also ensure that both arms of the bend are in the same plane.

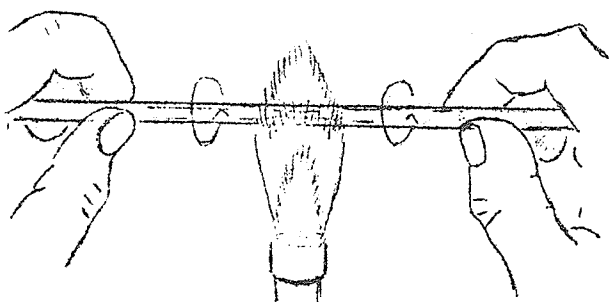
If the bend has been made correctly, it will be smooth and the bore of the tubing will be the same throughout its length. An uneven or constricted bend may result if

1. The tubing is bent before the glass has been sufficiently heated.
2. The tubing is lengthened as it is bent.
3. Both ends of the tubing are not rotated at the same rate while the glass is being heated.
4. The flame is concentrated on too narrow a portion of the tubing, as when no flame spreader is used.

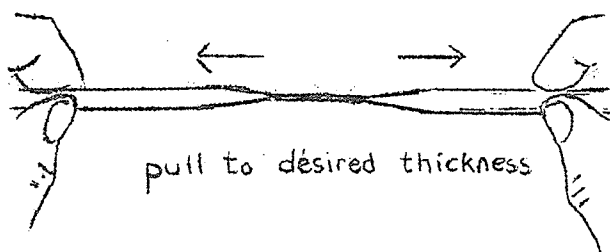


Drawing out tubing

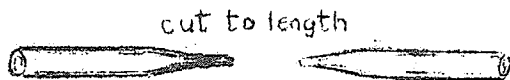
To draw out tubing to a smaller diameter, as to form a jet tip, a regular capillary or a medicine dropper, simply rotate the tubing at the correct point in the regular burner flame until it becomes pliable. Allow the tube to become shorter as the walls thicken to about twice their normal thickness. Then remove from the flame and pull apart carefully to the desired diameter. Place the hot glass on the wire gauze or asbestos pad to cool. Cut the glass in the middle of the drawn out portion and fire-polish the sharp ends.



no flame spreader



pull to desired thickness



cut to length

fire polish

Making a dropper

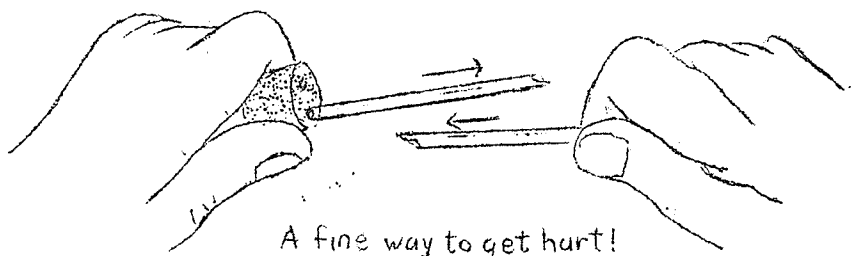
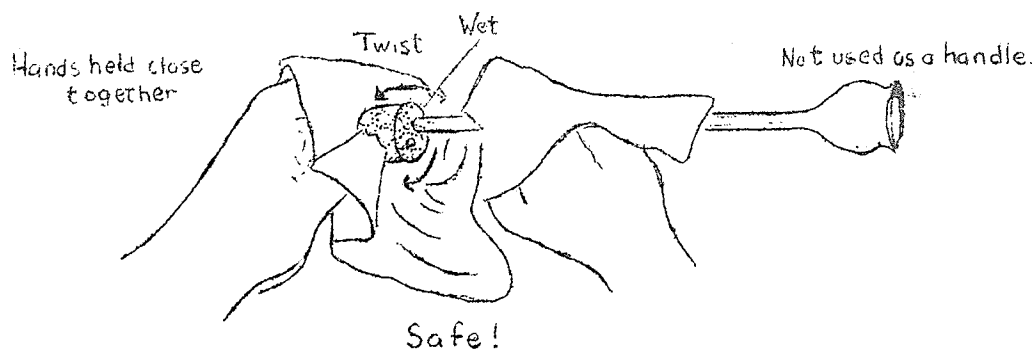
Heat the wide end of one of the tubes, and when the glass is soft, place the tip of your iron file against the inside of the glass tube and slowly rotate the tube. Reheat the tube and gently press the edge against an asbestos pad until the flange is even. When the tube is cool, a rubber bulb can be placed over the flange and the dropper may be tested with water. (This dropper may be used as a dropper pipette if you determine the number of drops that are equivalent to one milliliter. If you do so, label it for future use.)

T-4. INSERTING GLASS TUBING INTO STOPPERS

Caution: Injury, always painful, and sometimes severe, comes from improper techniques in inserting glass tubing through holes in the rubber. Read these instructions carefully. Don't become a casualty.

(a) The end of the glass tubing must be fire-polished so that no rough or sharp edges remain to catch on the rubber.

(b) The hole in the stopper must be almost as large as the tubing. If it is larger the joint will leak. If it is not large enough, do not try to stretch the rubber even though it may be possible. Either get tubing to fit the holes snugly, or get a stopper with holes to fit the tubing, or enlarge the stopper holes with a round file or cork borers until the fit is right.



- (c) Always lubricate the glass-rubber surface with water or glycerine.
- (d) Hold the glass as close to the end going into the stopper as possible, to avoid leverage or breakage. Never force a thistle tube or funnel into the stopper by grasping the large end.
- (e) Insert the tube with only a minimum of direct forcing together. Use most of the force (and very little should be needed) in rotating the tubing or stopper. Remove the tubing from a stopper in a similar way. Never push glass tubing or thermometers through a hole in a stopper or remove it by brute force.
- (f) Always hold your hands so that they cannot be a backstop for broken glass. Exert a uniform small force only on the tubing which is directly entering the hole, never on a part such as a bend which is not in line. If any possibility of breakage exists, wrap both hands in a towel to protect them.
- (g) If you experience any difficulty consult your instructor.

## MEASUREMENT

Chemistry is distinctly an experimental science. The establishment of the truth of laws and the behavior of matter depends on careful measurements of various quantities: volume, weight, length, temperature, time, etc. Measurement is the comparison of an unknown quantity with a standardized or known quantity.

A major feature of measurement undertaken by a chemist is the need for two or more sets of results in which the measurements are in close agreement; that is reproducible. The more sets of results which closely approximate each other the higher the degree of precision. This close agreement among separate sets of results is a basic requirement whenever definite conclusions are to be drawn. Keep this fundamental feature of measurement in mind even though a lack of time in the laboratory period may occasionally prevent you from doing so.

Accuracy is a term that expresses how closely the measurement comes to the true or accepted value. A measurement may be extremely reproducible, giving the same results each time, yet it may not measure that which it is supposed to measure. In this case the accuracy of the result is uncertain. The inaccuracy of a result is usually expressed as

$$\% \text{ error} = \frac{\text{the difference between the experimental and accepted value}}{\text{the accepted value}} \times 100$$

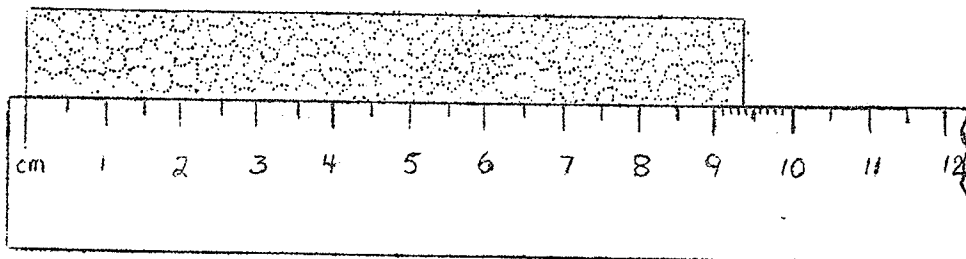
Thus, if the experimental result for the volume of a mole of oxygen were 22.1 liters, and the accepted value were 22.4 liters, the percent error of the result would be  $\frac{22.4 - 22.1}{22.4} \times 100\% = 1\%$

Strive for both precision and accuracy, since the former leads to the latter, and accuracy is the aim of the competent experimenter. Check your measurements carefully. Don't be satisfied with the first measurement, even experts using more precise instruments make mistakes. If possible check every measurement before you accept it. Often it may be possible to check it by a different method.

### T-5. READING SCALES

The most common source of error in measurement is in reading scales. The scale should be studied carefully before a start is made in an experiment. The value of each division should be worked out and the extent to which estimation can be made should be considered. In reading an instrument it is standard practice to take all readings to the nearest tenth of the smallest division on the scale being used, unless otherwise instructed.

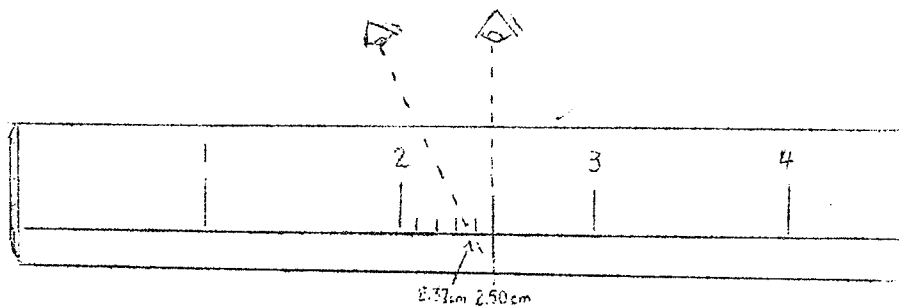
Since the smallest scale division on a meter stick represents a millimeter (mm) or a tenth of a centimeter (cm), your readings should be expressed to the nearest tenth of a millimeter or hundredth of a centimeter. In the example below, the edge of the object measures 9.37 cm. The last figure of the reading was obtained by estimating, in tenths, the distance between two millimeter marks. This figure is not completely precise but it is better than no estimation at all. For example, a reading of 0.19 cm states that the



length probably lies between 0.185 and 0.195 cm with a leeway of 0.01 cm out of 0.19 cm; the uncertainty of this reading is therefore one part in nineteen. What would have been the uncertainty of the reading if the last figure had not been estimated and the reading had been taken as 0.2 cm? Thus, although the last figure is only an estimation, it is a significant figure. Do not omit the last, significant figure even when it is a zero, that is the reading falls exactly on a millimeter mark. What is the difference in uncertainty of a reading of 4.7 cm and one of 4.70 cm?

Therefore, when recording the value of a measurement, one must indicate, in some way, the reliability of the measurement. This is done by using only those figures which are significant for the measurement. The numbers which are significant are all those which are known with certainty plus the first number that can be estimated. If you do not know how to determine the number of significant digits, study the section in the appendix.

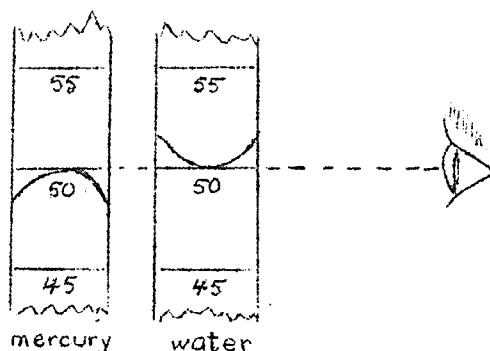
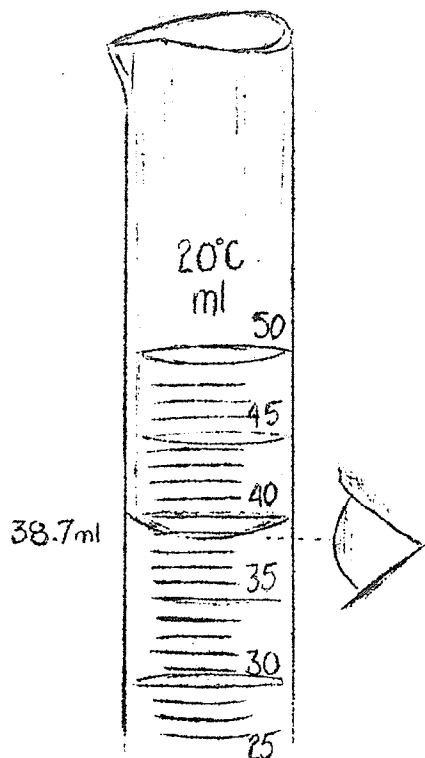
When readingscales, the eye should always be in line with the position on the scale. As you move your head the reading is also changed as errors of parallax are introduced. (Parallax is the relative motion of two objects which are at different distances from the observer, when the observer moves.) The errors introduced in this way can be studied below.



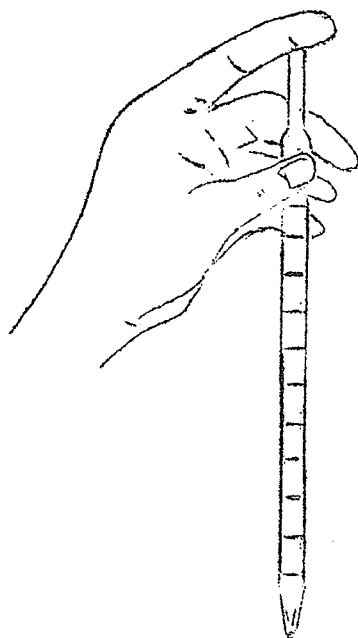
T-6. VOLUMETRIC MEASUREMENTS

When measuring volumes always read the level of the liquid so that

the eye level is directly parallel to it. You will notice that the liquid "wets" the glass, as in water and aqueous solutions and a curved "meniscus" results. The level is read at the bottom of the meniscus. Mercury does not wet the glass so the meniscus has a different shape. In this case, the level is read at the top of the meniscus.



Graduated cylinders. A graduated cylinder has a rather large diameter. For this reason, the quantity of liquid needed to fill it exactly cannot be precisely determined. Therefore do not use graduated cylinders for very precise experiments. You may fill the graduate to contain a predetermined volume, or you use it to measure the difference, i.e., filled completely and then emptied of the desired quantity.



Pipettes. These measuring tubes are used for more accurate volumetric measuring out a volume of liquid with a measuring pipette.

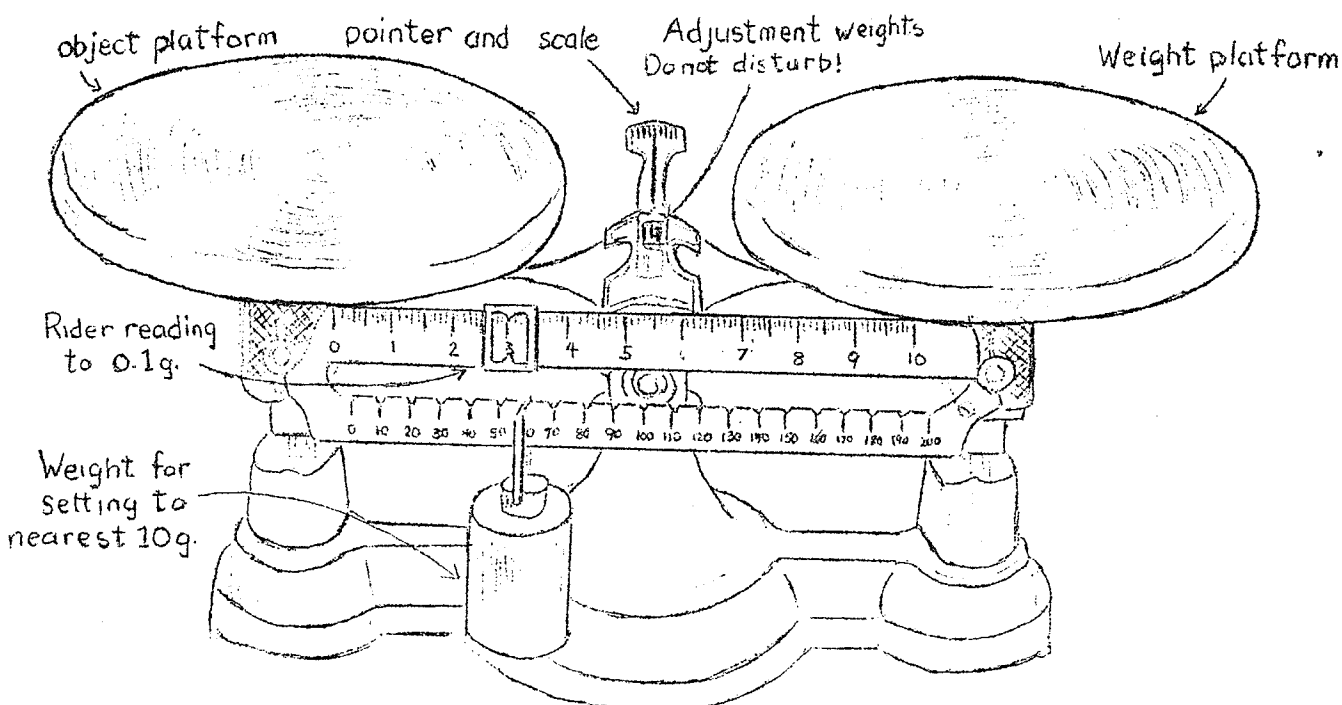
- (a) Be sure that the pipette is clean.
- (b) Draw the liquid into the pipette by using a suction bulb, or if the liquid is not dangerous, by sucking it up by the mouth.
- (c) When the meniscus of the liquid is above the top (zero) graduation mark on the scale, place the index finger over the mouth of the pipette. This will hold the liquid in the instrument.
- (d) Allow the liquid to drain until the meniscus is even with the zero mark of the scale. This is done by gradually releasing the pressure exerted by the finger.

(e) Measure out the desired volume of liquid by again releasing the pressure exerted by the finger until the meniscus is even with the line representing the volume. Remember, the zero line is usually the uppermost line; all lines below it refer to the volumes of liquid delivered.

(f) In the transfer pipette, allow the last drop of solution to remain because when the pipette was calibrated "to deliver" a certain volume, that last drop was taken into consideration.

#### T-7. WEIGHT MEASUREMENTS

One of the most common operations in experimental chemistry is the determination of weight. The weight of an unknown is usually determined by comparing its weight to that of a known standard. For making your comparisons you will use two types of balances. The platform balance shown below is sensitive to 0.1 g and is used only for approximate weighings. When you desire a more accurate weight to 0.01 g, you shall use the centigram balance shown on the next page.



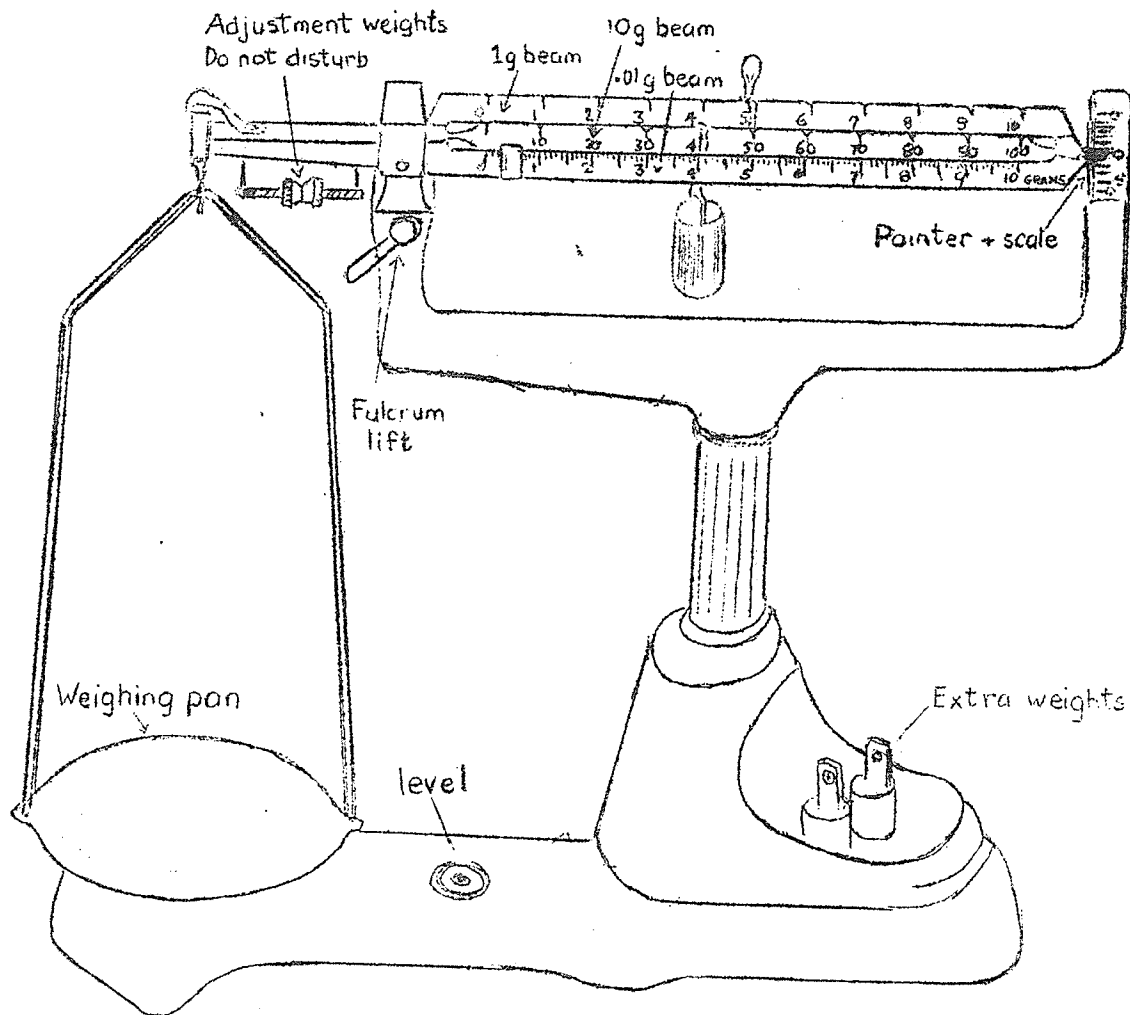
In the type of platform balance (trip balance) shown above, the unknown on the left pan is balanced by placing a large weight on the right pan and then sliding the weights attached to the cross-beam until the pointer makes equidistant swings to the right and left of the center point of the scale.



In using the centigram triple beam balance shown below, equilibrium is achieved by sliding the weight along the three beams until the pointer on the extreme right makes equal swings above and below the zero point.

This chemical balance is a delicate instrument that will weigh objects to the nearest centigram. Since the quantities of substances to be weighed are usually small, these balances have to be much more accurate than those in ordinary use in the commercial world. If you recall that one ounce is equivalent to 28.35 grams and that in quantitative experiments one has to make accurate weighings in terms of hundredths of a gram, it is obvious that this sensitive instrument must be used with a great deal of care, since it may be easily damaged by careless manipulation.

When the balance is not in use, the beams should be supported, thus relieving the knife edge of needless pressure and possible damage. To release the beams, giving freedom of movement, turn the fulcrum lift counterclockwise.



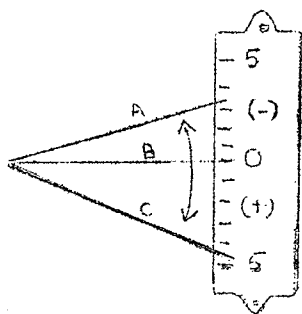
CAUTION:

A balance is a precision instrument it should not be abused by placing hot objects or chemicals on the pan. When weighing chemicals, always place a piece of paper on the pan first. Do not place any objects on the pan in such a way that the beam moves so swiftly that a banging sound is heard. Rough treatment will damage the knife-edges which are the heart of the balance. During an experiment, you must use the same balance for all your weighings since no two balances are identical in precision.

Weighing

(a) When using either balance, the first thing to do is to inspect it. Make sure the pan is clean and dry, and all the rider weights are set at zero.

(b) Next find the rest point. This is the place on the scale where the pointer will come to rest when equilibrium is established. Then the known weights equal the unknown weights. First, set the balance swinging slightly with the pointer moving from two to five divisions away from the center. It is not necessary for you to wait until the pointer comes to rest to determine the rest point, and the rest point need not be at the center of the scale. A typical determination on the centigram balance is shown below.



Close up of scale

A = furthest extent of swing above 0.

B = rest point - no load.

C = furthest extent of swing below 0.

$$-35 + 45 = +1$$

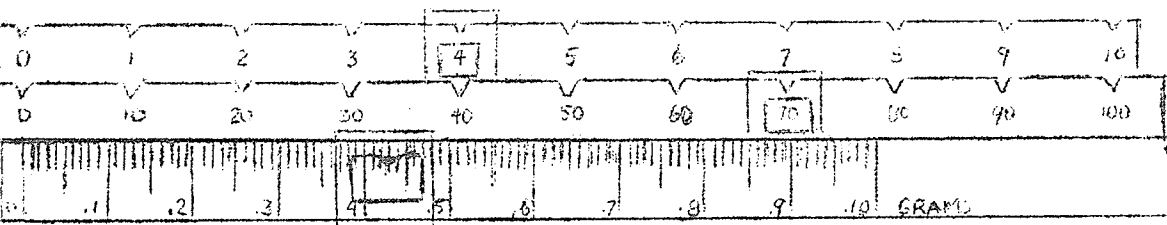
$$\text{Rest Point} = \frac{1}{2} = +0.5$$

If the rest point is more than three divisions on the scale away from the center, tell your instructor. Do not make any adjustments on the balance yourself. Never weigh an object which is warm; the convection currents will affect the rest point.

(c) Place the object to be weighed in the left pan on the balance and move the rider on the next highest beam until the beam just about balances, and then the lowest beam to obtain the final adjustment. The weight of the object has been determined when the rider weights on the beams balance the weight of the object by having the rest point at the same place it was when there were no objects on the pan and the riders were not used. Read and record the total weight.

(d) Return all the beam riders back to the zero point after weighing an object. Be sure to leave the balance and the surrounding areas as clean as, or cleaner than you found it.

Here is a close-up of a triple beam balance. What is the total weight on the balance beams?

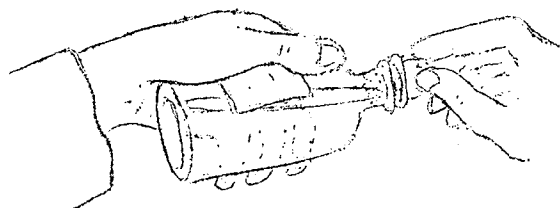


To weigh a chemical, place a piece of paper on the pan and weigh the paper, then add the chemical and weigh both. The difference in weights is equal to the weight of the chemical. The weight of the paper is tare weight. A tared beaker is one whose weight is known.

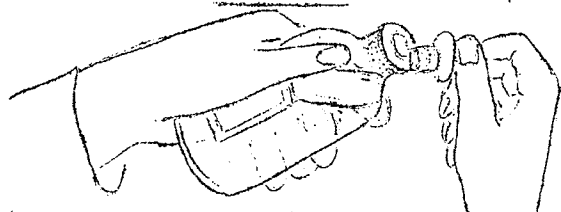
#### HANDLING AND TRANSFERRING REAGENTS

In the laboratory you betray your training, or lack of it, every time that you pick up a reagent bottle.

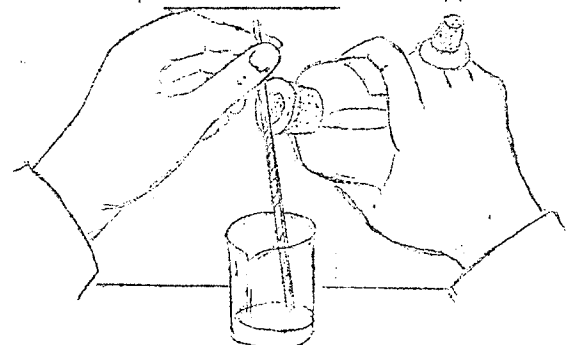
#### T-8. TRANSFERRING LIQUIDS



Hold the stopper in and tilt the bottle until the contents wet the stopper.



Moisten the inside of the neck and the lip with the wet stopper



Replace stopper and withdraw it again with back of hand. Never place the stopper down.

(a) Read the label on the bottle twice to be certain that you have the right reagent.

(b) Check whether bottles of corrosive liquids are wet outside. If so, clean with a wet sponge.

(c) Hold the stopper in and tilt the bottle until the contents wet the stopper. With the wet stopper moisten the inside of the neck and lip on the side opposite the label. Replace the stopper. The moistened neck and lip prevent the first drops from gushing out. Over a wet lip, a liquid can be poured so slowly that drops may be counted.

(d) Withdraw the stopper again by placing it between the index and middle fingers of one hand with the inserted end of the stopper pointed away from the back of the hand. Never set the stopper down. With the same hand, hold the bottle and pour the liquid from it into another vessel. Where possible use a glass rod to guide the flow of liquid and pour the liquid from the side opposite the label to avoid ruining a paper label. Always keep fingers out of the path of flowing liquid.

(e) Clean the bottle again with a wet sponge before placing down. The reagent bottle should never be placed on an unprotected desk surface. Use a glass square (cover glass) as a protective device for the desk.

(f) If too much reagent is poured out discard the excess. Never put anything back into a reagent bottle, it may carry impurities that would spoil the reagent for some future test.

Caution:

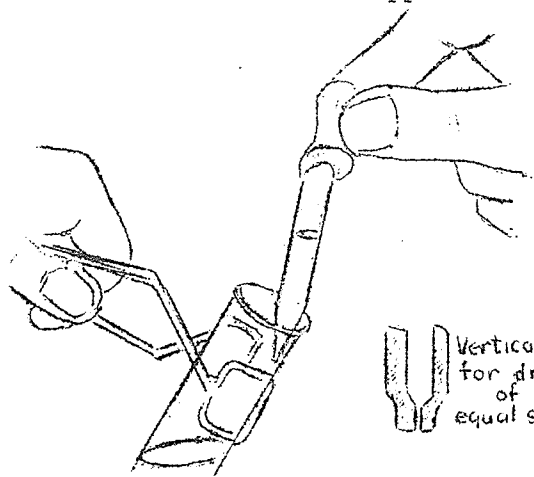
When diluting sulfuric acid, pour concentrated sulfuric acid into cold water (never water into acid) slowly, with stirring.

Using a Dropper

A dropper allows you to make a drop by drop transfer when required. Remember to rinse the dropper with water after each use. If the liquid being

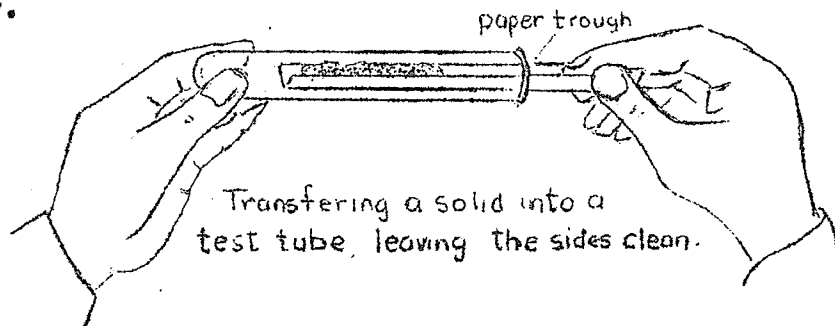
"drawn" into the tube should ever chance to enter the rubber bulb, disconnect this immediately and rinse both parts with water. The tip of the dropper must never be placed beneath the surface of the receiving liquid, thus avoiding the introduction of impurities into the dropper.

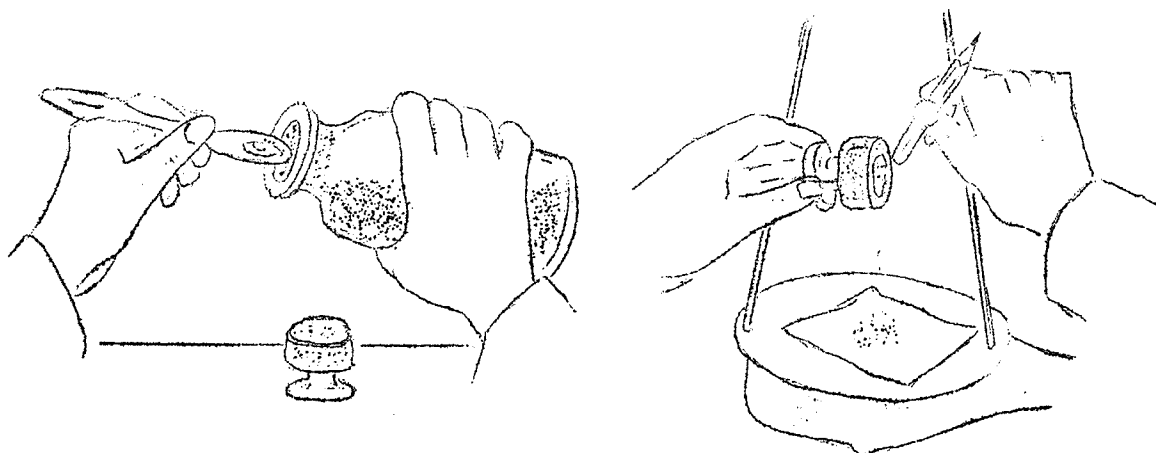
The dropper must be held in a vertical position if the drops it delivers are to be the same size. Also the lower end of the dropper must be ground square and flat.



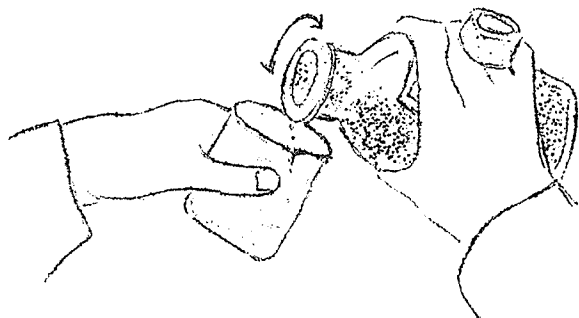
T-10. TRANSFERRING SOLIDS

Various methods may be used for transferring solids. The type you choose will vary with the situation. Some of these methods are diagrammed below.





For removing small samples, a flat topped glass stopper may be placed on the table upside down, and a little of the solid removed with a clean spatula or plastic spoon and placed in its cavity. Then still smaller portions may be parcelled out by tapping the stopper with a pencil.



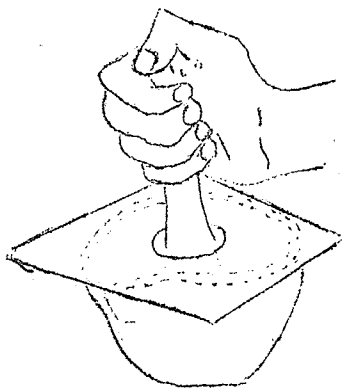
For pouring a large sample of solid into a wide-mouthed container, roll and tilt the bottle until enough of the material comes out.

For transferring solids by any of these methods the following precautions should be noted:

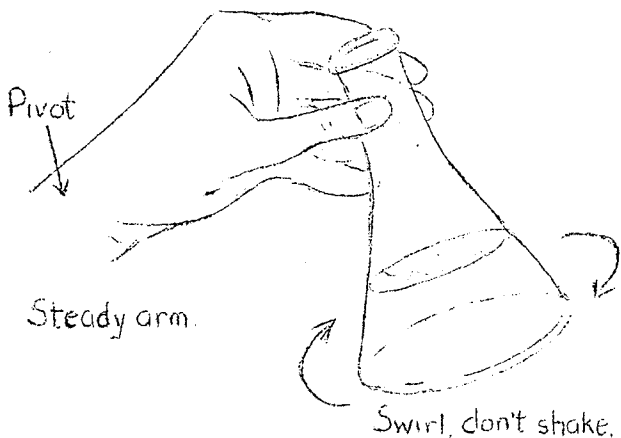
- (a) Take time to read the labels carefully. Use of the wrong materials may result in accidents and burns.
- (b) Never rest a stopper on the desk (except when placing a flat-topped stopper upside down). It is sure to pick up dirt, which may ruin some future experiment.
- (c) Never open two bottles at once to avoid interchanging their stoppers.
- (d) Make sure that the solid to be poured is in loose crystals, and not caked. Break up any cakes with a clean, empty test tube.

DISSOLVING A SOLID IN A LIQUID:

A solid may be dissolved in a liquid most quickly by (a) grinding the solid, (b) stirring, shaking, or in some way agitating the mixture, and (c) heating the liquid or mixture if necessary.

T-11. GRINDING

A mortar and pestle are used for grinding a solid into a fine powder. The solid should first be crushed and then ground into a powder. Don't pound! A paper shield may be used for corrosive or poisonous solids. The handle of the pestle may be passed through a hole in the paper.

T-12. MIXING OR STIRRING

A stir rod may be used for agitation in a wide-mouthed container, such as a beaker. However this is impractical in a flask or bottle. Effective mixing is obtained by swirling the mixture in an Erlenmeyer flask by means of a rotary wrist action.

If you are directed to shake a liquid in a flask or bottle, close this with a clean stopper (not with a finger or thumb), then shake it vigorously.

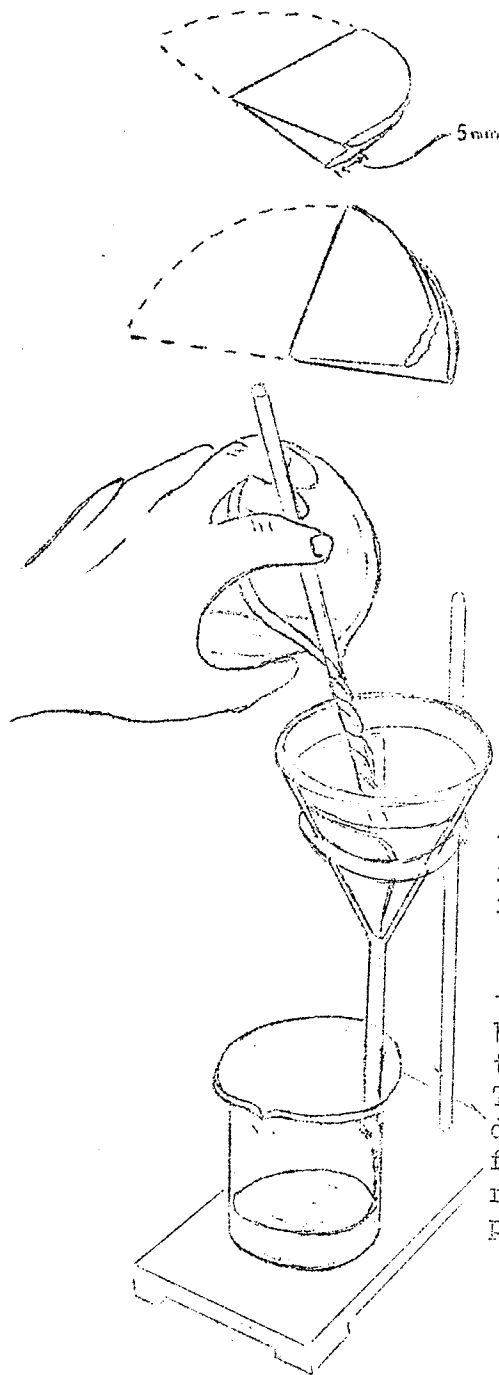
To mix liquids in a test tube, close it with the thumb and invert several times. First be certain that the tube contains no hot or corrosive liquid. Nitric acid, except when diluted, will stain your thumb yellow.

METHODS OF SEPARATIONT-13 DECANTATION

Heavy coarse residues may be separated from the liquid simply by pouring (decanting) the liquid from the residue which had previously been allowed to settle out.

T-14 FILTRATION

The preparation of a filtration apparatus is accomplished as follows:



(a) A circular piece of filter paper is folded lightly in half, and then this semi-circle is folded to give a piece approximately one-quarter the size of the original circle. The second fold is made unevenly with about 5mm of the bottom fold allowed to extend beyond the top fold.

(b) A small irregular triangle is torn from the corner of the top fold, to aid in sealing the paper against the funnel. The folded paper is opened into a cone with the torn folded edge to the outside and is placed in the funnel.

(c) A small amount of water is poured into the funnel and used to moisten the filter paper. The paper is gently pressed against the glass around the top edge of the funnel to make an airtight seal which prevents air from leaking down the fold. The moistening of the paper also discourages the absorption of large quantities of the solution to be filtered. When the funnel is filled with water and allowed to drain, the air should be washed out of the stem, leaving a solid column of water in the funnel stem, uninterrupted by air bubbles. The weight of this column of water hastens filtration. The water is emptied from the beaker and the apparatus is now ready for the separation of the mixture.

(d) The mixture is most easily transferred to the funnel by placing a stirring rod across the beaker, holding it in place with one finger and tilting both the beaker and the stirring rod. The mixture is guided by the stirring rod and directed against the side of the funnel. The filtrate that passes through the paper should run down the walls of the collecting beaker to prevent spattering.

(e) Any precipitate which remains in the beaker is washed with a small quantity (5ml) of distilled water and this mixture is transferred to the funnel. This last operation is repeated until all the solid is transferred to the funnel. The precipitate is then washed several times with small (5ml) portions of water.

#### T-15. CENTRIFUGING

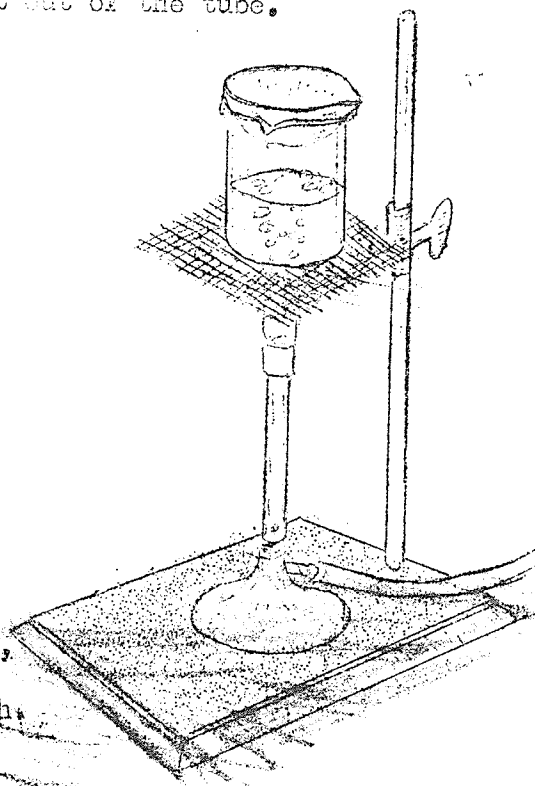
In using the centrifuge to hasten the settling of precipitates be sure to observe the following precautions:

- (a) Before starting the machine, counterbalance your test tube with another one containing an equal volume of water, in the opposite cup. Do not forget this. If you do, you will probably ruin the bearings of the motor.
- (b) Allow the centrifuge to rotate about one minute for most efficient separation.
- (c) Keep your hands away from the top of the centrifuge while it is rotating.
- (d) After turning off the motor, do not try to brake the centrifuge unless the instructor shows you how to do it. Braking is possible in most types of centrifuges, but requires skill, since too much braking will remix the precipitate with the solution and will often cause the solution to fly right out of the tube.

#### T-16. EVAPORATION

The solute can be recovered from a solution by evaporation. The rate of evaporation is controlled so that "spattering" of the contents is prevented. Once the volume of liquid is considerably reduced the final evaporation to dryness is accomplished with a much smaller flame, since small pockets of steam cause the liquid to spatter as the solution becomes more concentrated. The rubber tube leading to a burner should always pass in front of the iron stand. You can then always take the burner away without upsetting the contents, if it does spatter.

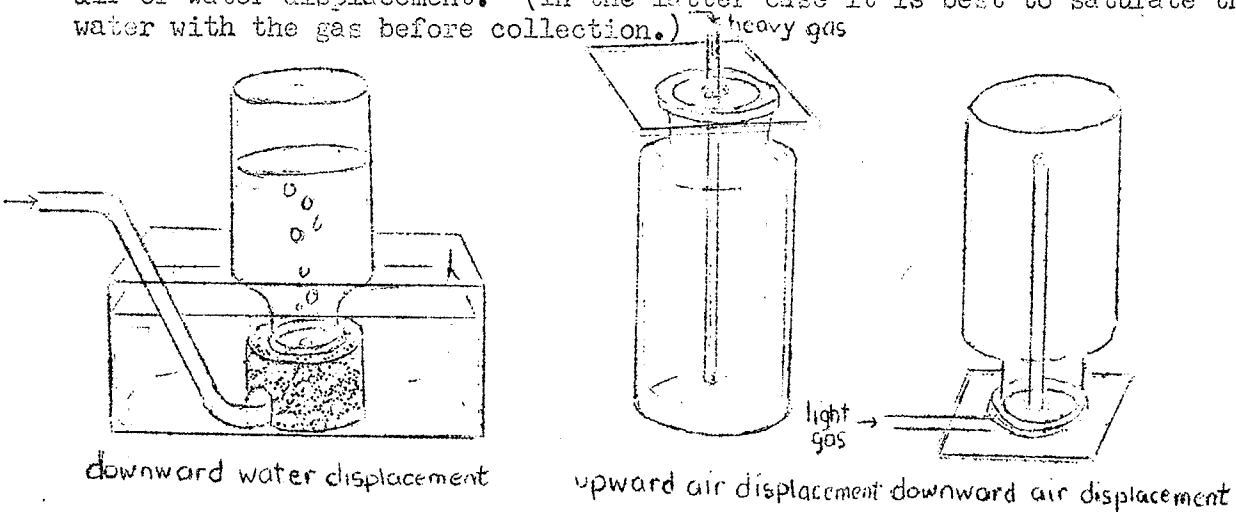
For quantitative experiments a slow, even rate of evaporation is accomplished by heating the solution over a water bath. Care should be taken to replace the water in the beaker from time to time, as a prevention of boiling to dryness.



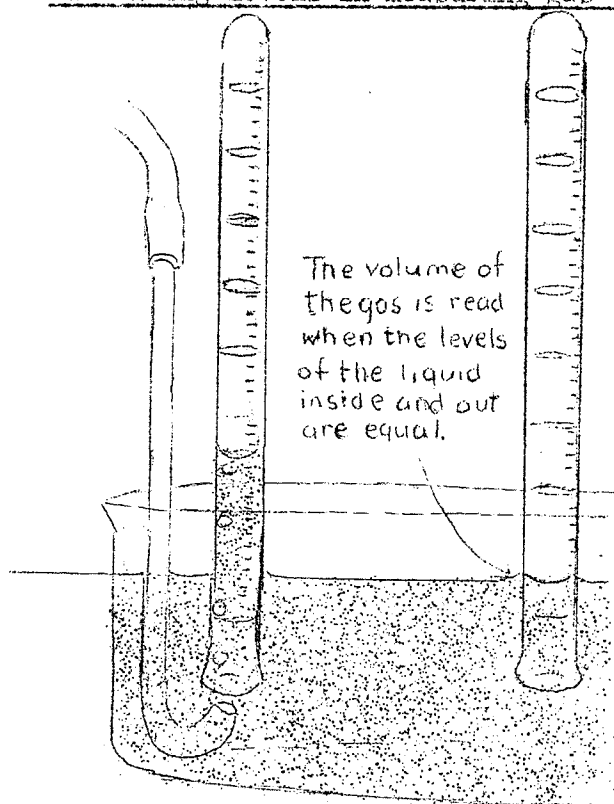


### T-17. GAS COLLECTION AND MEASUREMENT

There are three general methods of collecting gases. If the gas is insoluble or slightly soluble, in water then it is collected by downward displacement of water. A gas that is soluble in water can be collected by the displacement of air. If a gas is heavier than air, it can be collected by upward displacement of air. The delivery tube should extend to the base of the collecting bottle in both cases. Gases, such as chlorine and carbon dioxide, that are moderately soluble in water, may be collected by either air or water displacement. (In the latter case it is best to saturate the water with the gas before collection.)



### Equalizing levels in measuring gas volume



As well as measuring the volume of a given sample of gas produced by a reaction we also need to measure the conditions of temperature and pressure.

Before reading the volume of the gas, raise or lower the measuring tube until the level of the water inside the tube is even with the level of the water in cylinder. When the two surfaces are at the same level, the weight of the atmosphere pressing downward on the surface of the water on the outside cylinder is then balanced by the expansive force of the gas. In other words, the confined sample of gas with the liquid levels equalized, is under atmospheric pressure, which is read from the barometer.

Now, read the volume of the sample of gas at atmospheric pressure.

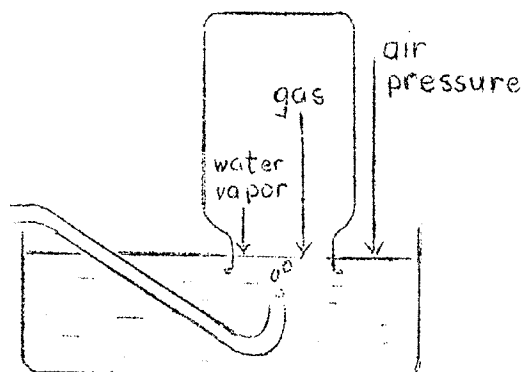
### Correction for the Vapor Pressure of Water:

When a gas is collected over water, it is a mixture of gas and water vapor which is under barometric pressure after the liquid levels are equalized. Only part of the measured pressure is due to the gas collected since the water molecules contribute to the pressure.

$$\text{Barometric pressure} = \text{the pressure of the dry gas collect} + \text{the pressure of the water vapor.}$$

The (water) vapor pressure is dependent upon temperature. Having measured the temperature of the water, you can obtain the vapor pressure at that temperature by referring to Table I in the Appendix. By subtracting the vapor pressure of the water from the barometric pressure, you can find the pressure that the gas would be under if it were dry and occupied the whole volume of the mixture alone.

The corrected volume for the dry gas at barometric pressure can be determined by applying Boyle's law, using the values for the pressure of the dry and "wet" gas. (Since the molecules of water vapor are theoretically removed, the volume of the dry gas would decrease of necessity until its pressure was again equal to the atmosphere.



### SAFETY

A first consideration in a chemistry laboratory is safety. The dangers of a laboratory are approximately the same as those which face a housewife working over the kitchen range. She may be burned by a hotplate or by a gas flame. She may burn her arms while taking hot pans from the oven. She may spill boiling water and scald herself. Droplets of hot fat may fly from the frying pan onto her face. All these dangers are easily avoided by using care. The same sort of common-sense care applies in the laboratory.

The following safety precautions are given to safeguard your interests as well as those of other members of the class. Learn them immediately and observe them at all times.

1. Protect your clothing with an apron and avoid dangling cloth sleeves.
2. Whenever an accident occurs in the laboratory, notify the instructor immediately.
3. When heating a substance in a test tube, do not point the open end of the test tube at any of your neighbors or at yourself -- it may bump.
4. When attempting to detect chemical odors exercise extreme caution. Keep your face a good distance away from the unknown substance and use your open hand to gently waft the vapors toward your nose.
5. Never taste a chemical or a solution unless directed to do so.
6. Avoid touching hot objects, such as the barrel of a gas burner. Allow worked glass to cool; hot glass looks like cool glass.
7. When diluting acids with water, always pour the acids into water carefully. Do not pour water into acids.
8. If any chemical substance is spilled or splashed on your skin, wash it thoroughly with water. Neutralize spilled acid or base as follows: Acid - use a solution of sodium carbonate. Base - use a solution of boric acid. Report the occurrence to your instructor.
9. When working with equipment made of glass, such as tubing, thermometers, and thistle tubes, be very careful to avoid breaking the glass and cutting yourself.
10. Read the label on a reagent bottle carefully before using the substance in the bottle.
11. Never mix two or more chemicals ignorantly and without permission. What you don't know will hurt you!

Neatness and cleanliness is essential for experimental work in any science, but this is particularly true in the science of chemistry. It is often difficult for a newcomer to chemistry to realize what tiny quantities of materials can show easily visible effects. One important rule in chemistry is to know what materials you are working with. If you have unclean apparatus, how can you tell whether the observations you make are due to the materials you think you are working with, or to the traces of unidentified impurities left clinging to your apparatus from a previous experiment? You can't.

### Cleaning glassware

Use plenty of water for rinsing, then scrub with a brush and detergent, and then use plenty of water for rinsing. Rinsing thoroughly with small amounts of water several times have been found to be more effective than rinsing once or twice with large amounts of water. Test your glassware for cleanliness. The glass is clean if the water drains away in a film that remains continuous leaving a glistening surface. The glass is dirty when the water film breaks into scattered droplets. If the above treatment seems inadequate, consult your instructor.

Cleaning of glassware should be done immediately after an experiment, not before. You expect to find clean, dry glassware when you enter the laboratory; other students following you into the laboratory should expect the same.

### Cleaning Apparatus and Desktops

1. Avoid spillage, but if you do have a spill, clean it up immediately. There are many corrosive solutions that are colorless like water, but here the similarity ends. It is easy to keep the desktop clean by frequent use of a damp sponge, which is rinsed after each application.

2. Throw all solids chemicals to be discarded into a waste crock; all paper should be thrown into a waste basket. Never leave splinters, filter paper, etc., in the sink.

3. Everyone should realize without being told that broken glass fragments should never be left on the bench tops, where someone may receive painful injuries, just by leaning on his hand or elbow. A separate container for broken glass will be found along with a brush and dust pan, in a designated location.

4. At the end of each laboratory period, put your own clean equipment into your drawer or tote box and return any special apparatus to its proper place. Be sure to leave your laboratory desk as clean, or cleaner, than you found it. Others must share the same work area.

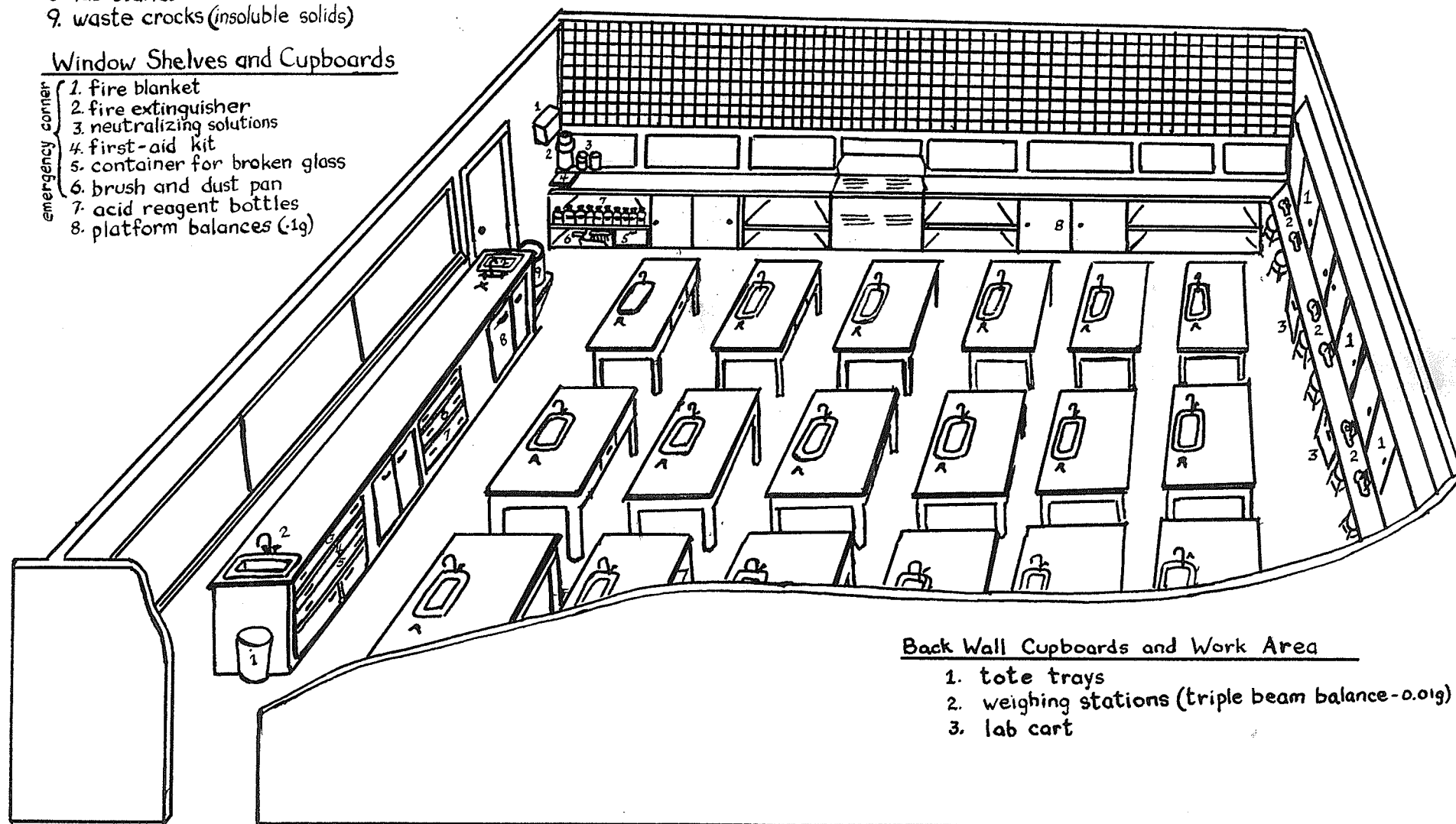
## Instructors Demonstration Table

1. waste basket (paper)
2. hot water source
3. corks and stoppers
4. burettes
5. condensers
6. utility clamps
7. ring clamps
8. lab stands
9. waste crocks (insoluble solids)

## Window Shelves and Cupboards

- emergency corner {
1. fire blanket
  2. fire extinguisher
  3. neutralizing solutions
  4. first-aid kit
  5. container for broken glass
  6. brush and dust pan
  7. acid reagent bottles
  8. platform balances (1g)

## KNOW YOUR LABORATORY FACILITIES



### Back Wall Cupboards and Work Area

1. tote trays
2. weighing stations (triple beam balance-0.01g)
3. lab cart

## AN INTRODUCTION TO METHODS OF EXPERIMENTATION AND REPORTING

All science is built upon the results of experiments. Whenever a scientist takes an object or event out of nature and studies it in his laboratory under controlled conditions, he is performing an experiment. An experiment is a situation planned and set up by the experimenter, which will provide him with the answer to a certain specific question, because all other conditions or possibilities have been eliminated. An experiment may be classified generally as being one of two types: a discovery experiment or a verification experiment.

During the course of any scientific investigation, the scientist may make an observation that is completely new to him. The curiosity of the scientist will not allow this observation to remain unknown for very long. Some of these questions will be running through his mind, "What is happening?" "How is it happening?", "Why is it happening?". On the basis of his present knowledge he will then think about possible answers to these questions. Each possible answer, or educated guess is known as a hypothesis. After selecting his most likely hypothesis, the scientist will test to find if it is correct or not. This is where experimentation begins. If the results indicate that the hypothesis is correct, further experimentation will be used to check the results. If the hypothesis is false, the scientist will form another possible solution, and proceed to test it by experiment. When used to test the truth of a hypothesis, the experiment is one of discovery.

An important characteristic of scientific facts is that they can be reproduced again and again. Before a discovery can be widely accepted in the scientific world it must be duplicated and verified by other scientists who have studied the reports of the original experiment. This type of experiment requires keen observation and good laboratory techniques. When used as an attempt to duplicate the work or discovery of another scientist, the experiment is one of verification.

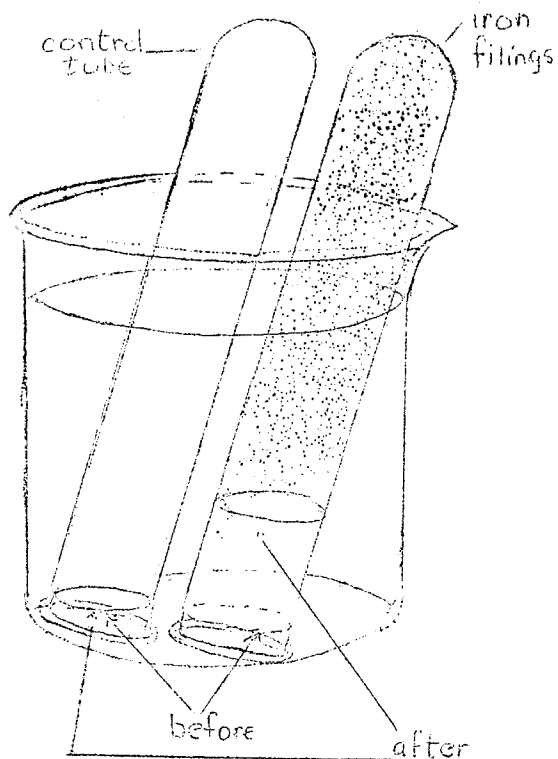
In the course of the year, you will be given experience in performing both discovery and verification experiments, with emphasis placed on the former type. You will find that the laboratory work often involves a more detailed consideration of a particular topic than the description given in the text. Occasionally, the results obtained in the laboratory will be used to introduce the discussion of a specific idea in the text.

You will experiment in the laboratory, asking nature to answer questions for you. But you should not flounder around aimlessly. An experiment answers questions only for those who know how to ask them. Therefore you must come into the laboratory prepared to work, with an understanding of the question being asked, and with some ideas of how to find the answer. You are expected to read the applicable sections of your laboratory manual, text and any other sources of information that may be of any value. You will find it essential to prepare in advance, notes on outlining the procedures and tables for data, and to anticipate trouble before it happens.

Since you have had little practice in experimentation, this manual is designed to help you set up your experiments. You will find that the instruction for some later experiments are far sketchier than those at the beginning. Since you will learn techniques, and gain some confidence in your ability as you go along, you should be able to plan more and more of the laboratory work as the year progresses. In other words, the laboratory work will give you practice in self-direction.

A well planned experiment generally has two parts, with a single difference between them. One of these parts is known as the experimental, or variable part of the experiment, and the other is called the control constant or check part. A condition is controlled when it is known, constant, and it can be varied deliberately if desired. This control is best attained in a special locale -- a laboratory. There must be only one variable -- only one difference -- between the experimental and control parts; thus there will be but one question to be answered by the experiment.

To improve the experiment, there must be a large number of trials or cases in both experimental and control parts. This means that the conclusions should not be made from the results of one trial only. The reliability of your conclusion increases with the number of trials attempted. Unfortunately, in the school laboratory, owing to a lack of time, you may be asked to arrive at a conclusion from the observations that you made in a single trial. Even if this is sometimes the case you should not forget the importance of a number of trials on the reliability of your conclusions.



You have previously experienced a controlled experiment when studying, "What happens to the air in which iron rusts?" If only the one test tube, with the iron filings, was inverted and placed into water, the results would be indefinite. Observing that water rose up into that test tube, you might conclude that part of the air was consumed by:

- (a) uniting with the iron
- (b) dissolving in the water, or that
- (c) the air was compressed as the tube was lowered into the water.

However, this situation was cleared up somewhat by the use of a control. The conditions for the control tube were identical to the experimental tube, except for the one experimental difference (variable) -- the iron filings.

By comparing the two tubes, you could conclude the change undergone by the iron filings had consumed part of the air and therefore it accounted for the rise in the water level to the greatest extent; solution in water and/or compression having affected the water level only slightly.

The conclusion that you reach must not be expected to include a wider area than your experimental materials and procedures allow. If you conclude that oxygen of the air was consumed in the rusting of iron filings, on the basis of the procedures described in the preceding paragraph and diagram, then your conclusion goes beyond the observation that you made, and is therefore not correct. Even though the water level rose upwards to about  $\frac{1}{5}$  the height of the tube, it would be more correct to conclude that "part of the air" was consumed during rusting and that the specific part consumed might be identified by further testing. If however, you inserted a burning splinter into both tubes (after covering and turning the tubes right side up), then the additional observations would permit you to use the broader conclusion which identifies oxygen as the specific part of the air that was consumed.

#### LABORATORY NOTEBOOK

One of the most important parts of scientific experimentation is the maintenance of a complete, accurate record. A man who discovers new knowledge and makes no permanent record of it, has buried valuable treasure without a map to show its location. He can neither claim credit for its discovery nor have it verified by others. The record should be written so that it would be possible, at any time after the experiment has been completed, for a competent person to duplicate the experiment exactly, with only a written record as a guide. The experiments carried out by Thomas Edison have been verified by hundreds of scientists; his laboratory notebooks (2500 of them containing up to 300 pages each) are still preserved to this day in his laboratory at West Orange, New Jersey.

Your laboratory notebook has been specially designed to encourage the immediate recording of observations and the completion of most of the experimental work before the end of the laboratory period. Your report must therefore be concise. Remember that it should be meaningful to others besides you. In order not to be wasteful of the notebook some planning is required on your part.

The use of a carbon copy permits you to have a permanent copy of your laboratory work as reference during classroom discussions and for further experiments. At the same time your instructor has a copy of your report for correction. If the experiment is not completed before the end of the period, you will be required to turn in a carbon copy of your data, and still be able to complete the calculations and arrive at conclusions from your permanent copy. A copy of the data you collected, at this time, permits the instructor to keep in close contact with your progress in the laboratory.



### R-1. Pre-Laboratory Preparation

An experimental investigation begins before the laboratory work is started. Careful thought is given to the problem that will be investigated experimentally. The procedure to be used should be reviewed and studied with reference being made to the sections on apparatus, techniques and safety. Attention should be given to the type of data to be obtained and the best method for tabulating it. For most of the experiments, special entries should be made in your notebook before beginning the laboratory work. These entries include tables for the recording of data, flow sheets illustrating the experimental procedure to be followed in the laboratory or information obtained from the textbook, or preferably from other textbooks and scientific literature. A discussion of the arrangement and content of such entries follows.

### R-2. Statement of the Problem

The statement of the problem should be simple, clear and concise. It should be written evidence that you understand what the experiment is designed to answer. Do not begin any actual experimental work until you have recorded a clear statement of the problem in your notebook.

Since you have had no practice in phrasing these statements the statement of the problem will be made for you, until you become more familiar with experimental procedures.

### R-3. Materials and Apparatus

The report of a research chemist would contain a detailed description of the types of purity of the material used, along with the type and accuracy of the apparatus employed. The results obtained from the experiment may have been greatly affected by the purity of the materials and the accuracy of the apparatus. Therefore this information should be recorded in order to explain or evaluate your results and also to allow another experimenter to duplicate your observations with no other guide but the written record.

If you are outlining your own procedure, be sure to add such information as the purity of the material or the accuracy of the apparatus. If the procedure is outlined in the lab manual you may be required to make a list of the apparatus or to represent it by means of a labelled diagram. Be aware of the importance of such information about the materials and apparatus, even though it may not be emphasized in your high school laboratory.

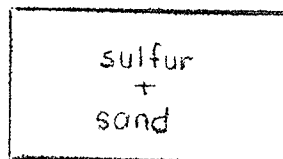
#### R-4. Laboratory Procedures

Under this heading you might begin with a general statement of how you propose to proceed. Then describe, clearly, but concisely, your exact procedure, including any special precautions to be taken. (The most acceptable form of this description is in the third person, passive voice.) This is suggested, especially for those experiments in which you will develop your own procedures, after some guidance from the instructor. If, in the course of the experiment, you meet any special problem, describe how you solved them. If the laboratory procedures are already described in the laboratory manual, never repeat these instructions; your instructor has already read them.

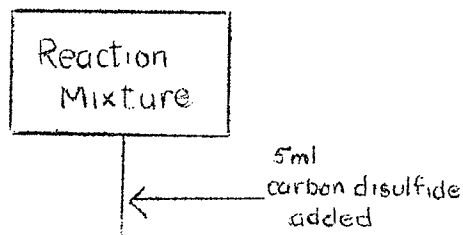
A convenient recommended device for describing laboratory procedures, in either case, is the flow sheet. This graphical representation is extremely useful as a means of establishing a record of the procedure followed, and also the observations noted and the data collected while conducting an experiment. The organization of a flow sheet is such that the recorded information can be easily read. An added advantage lies in the fact that the information can be entered in the laboratory notebook with a minimum amount of actual writing, thus saving both time and paper.

The following information describes some of the symbols which can be used when making a flow sheet.

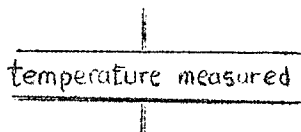
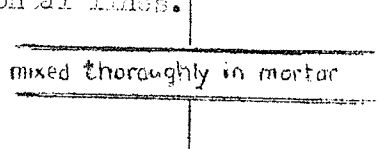
(a) When using flow sheets the starting materials are enclosed in boxes. These materials may be referred to, collectively, as the reaction mixture.



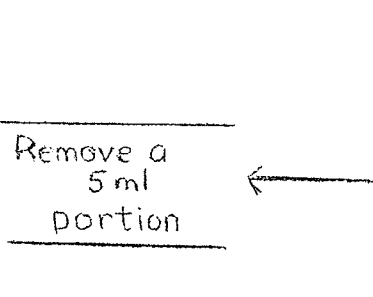
(b) The addition of a reagent to the reaction mixture, or starting materials, is indicated by the use of an arrow pointing from the reagent to the vertical line representing the reaction mixture.



(c) A manipulation of a reaction mixture which does not involve a separation of the components present in the mixture is represented by the use of two horizontal lines.

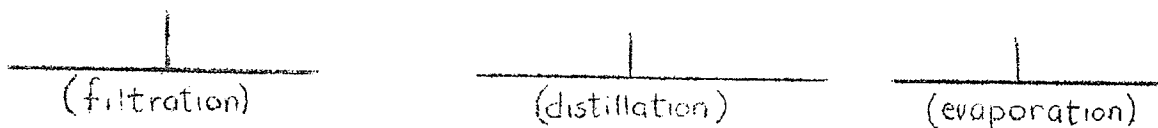


(d) The removal of a portion of the reaction mixture is indicated by an arrow pointing away from the vertical line.

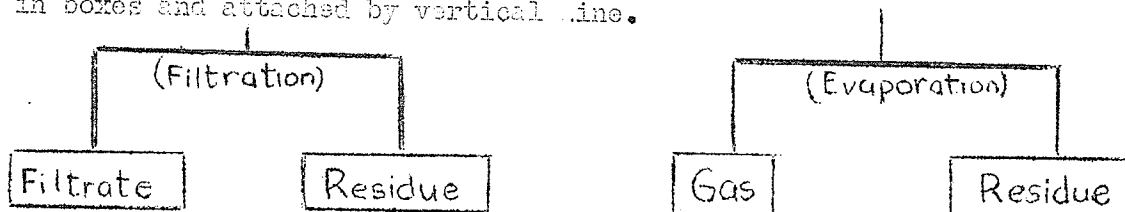


Remove a  
5 ml  
portion

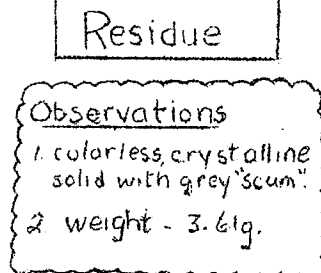
(e) Operations which involve the separation of the components of the mixture, such as filtration or evaporation, are indicated by a horizontal line. The name of the type of separation conducted is enclosed in brackets below this line.



(f) The products of the three processes mentioned above are enclosed in boxes and attached by vertical line.

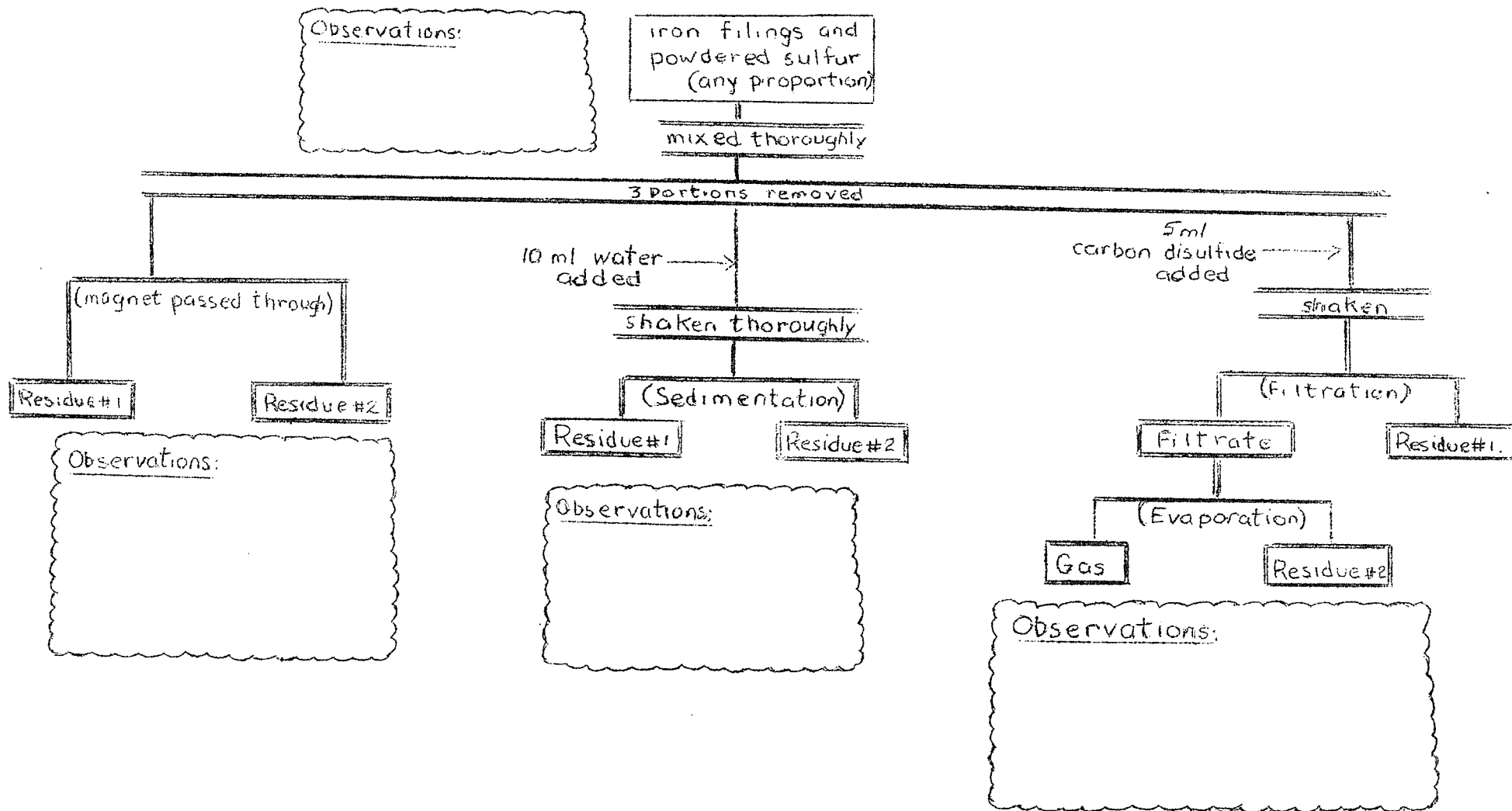


(g) All recorded observations are enclosed in a box whose sides are constructed of wavy lines. The observations are written below or to the side of a particular manipulations, but are not adjoined to any part of the diagram. Complete the recording of the observations before enclosing them with the wavy lines



A completed flow sheet is presented on the next page for your inspection.

# Comparing Mixtures & Compounds



In carrying out the procedures, be sure that you are first familiar with the operations to be followed and with the apparatus which you are using. Refer back to the description of the laboratory apparatus and the specific techniques required before each experiment in order that you may accomplish the maximum amount of laboratory work in a short period of time and that you may foresee trouble before it happens. Make certain that you know the accuracy of your measurements. Also, check your measurements carefully. Don't be satisfied with the first weighing or the first reading of an instrument. Even experts make mistakes. If possible check every measurement before you accept it. Often it may be possible to check it by a different method.

Another method of carrying out your laboratory procedures most efficiently is to work in cooperation with your partner, by sharing the duties equally. While your partner is setting up the apparatus, you might be making the first weighing, or obtaining the required reagents. While the experiment is "cooking", look ahead at the conclusions which will be required after all the data is collected. Anticipate some possible conclusions and at the same time think of errors which could have been made during the course of the laboratory procedures. Adequate planning should result in completion of the laboratory procedures within the period, leaving sufficient time to interpret your data and arrive at definite conclusions.

#### R-5. Observations

To observe means "to see", but observation extends beyond the visual sense. It includes the use of all the senses and all the available scientific instruments to learn what is occurring. Thus a blind man can observe, although he cannot see. Observations can be made by seeing, hearing, tasting, smelling, feeling, weighing, measuring, and by numerous other methods. In order to gather information about a patient's heart, a doctor will take the pulse, and count the number of beats per minute. At the same time he feels whether the pulse is weak or strong. He listens to the heart with a stethoscope. He may also record the action of the heart on an electrocardiogram so that he has a visual record. All these are methods for observing the condition of the heart.

Chemistry is built upon the results of experiments. An experiment is a controlled sequence of observations. A good experimentalist is a good observer. But even for the trained observer, his human senses are not sharp enough to make all necessary observations and often these observations may be misleading. Below is a simple example to illustrate this point. Study the two figures, then, decide which of the two lines is the longest. Check your observation by measuring with a ruler? Were you correct?



Because scientists cannot always trust their senses, they have devised instruments which make the senses more acute, or more accurate. Thus to extend vision beyond the limitations of man's senses, scientists employ the microscope, telescope and the X-ray machines. The analytical balance, the micrometer and the voltmeter are among the thousands of instruments which multiply the accuracy of the scientist's observations.

Choosing and using your language carefully, you should report and describe your observations to others. To say something so that it cannot be misunderstood requires that you be absolutely clear yourself as to what you mean and that you say it in clear unmistakable terms. These should include both qualitative observations such as color change, sudden appearance of new solid, foaming or bubbling, and quantitative observations which refer to all numerical measurements such as those of length, temperature, weight, etc. Suppose you record in your notebook that flame A is bigger than flame B, what does this mean? Does "bigger" mean taller, thicker or what? Wouldn't it be much better to say that flame A was 7.1 cm high and B was 5.5 cm high? The second statement is obviously better because it is a more objective, quantitative measurement. Remember to label or identify all numbers used. If you are the only one in the world who can tell what your written 18 is a weight in grams, rather than weight in ounces, or a temperature, or the number of times that you stirred a solution your record is worthless.

A carefully planned experiment does not guarantee a successful experiment. Even the best laid plans are useless in the hands of a poor observer. Never fail to record your observations immediately after they have been made. Do not depend on your memory. It is very easy to record in your observations only what you want to see, leaving out those things that are contrary to your own hypothesis. Don't let your hopes and expectations bias your observations and records. Remember, it is often the unexpected observation which leads to discovery. Don't fail to record anything.

#### R-6. Recording Data:

Quantitative observations should always start with "raw" data, that is the figures actually read off the instruments, and be followed by calculated data. All data should be presented in tabular form. The data recorded should be arranged in such a way that it is an aid to calculation. A data table for an experiment might look as follows:

	1st trial	2nd trial
Volume of liquid	25.0 ml	
Weight of beaker + liquid	50.0 g	
Weight of empty beaker	33.2 g	
Weight of liquid	16.8 g	

It will save time in the end to acquire the habit of recording data legibly. Note the following about the table:

- (a) All lines are drawn with a ruler
- (b) The figures are not crowded.
- (c) The units for all measurements are indicated.
- (d) The data is recorded with the decimal points kept in line.
- (e) The arrangement of the data aids in calculation. Note that in this experiment, the weight of the empty beaker is taken first, but for convenience of simple subtraction it is placed second.

### R-7. Analyzing and Interpreting Data

The value of each experiment conducted in the laboratory depends not only on the careful manipulation of chemicals and apparatus but also on the manner in which the observations and data are correlated and interpreted. The discovery of regularities permits simplification of observations. Instead of each observation standing alone, several observations can be grouped together and hence can be used more effectively.

#### a) Calculations:

In calculating the results from experimental data, it is advisable to show specifically the method of calculation. This may be done by giving a sample calculation using the numbers labelled with units or by also writing out a general expression which indicates how the experimental data are mathematically combined. For example:

$$\text{Density of the liquid} = \frac{\text{weight}}{\text{volume}} = \frac{16.8 \text{ g}}{25.0 \text{ ml}} = .670 \text{ g/ml}$$

In no case is it useful to show the detailed arithmetic. This is to be done on a scratch pad or in some inconspicuous place in your laboratory notebook.

#### b) Graphing

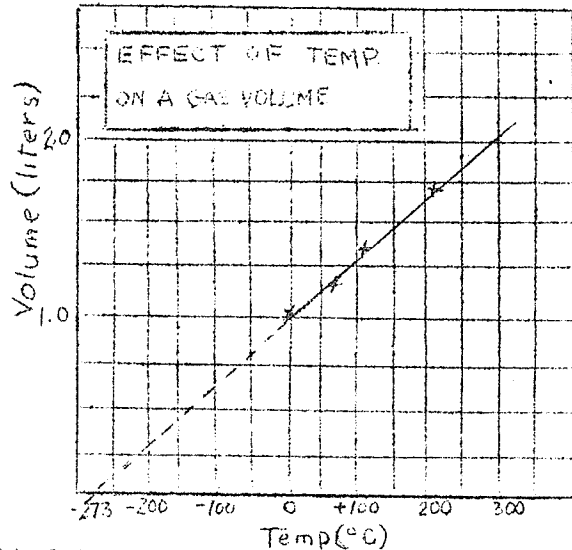
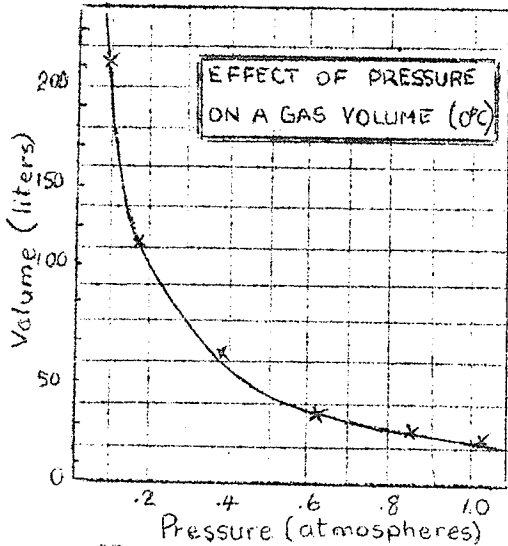
A graph is a statistical picture. It is a graphic way of summarizing and presenting the information which you record in your data table. Regularities or trends are often not immediately evident from direct examination of the data recorded in the table. However, a graphical picture of the data often establishes a regularity or trend, which correlates the data, and, at the same time, reveals the uncertainty of the experimental measurements.

In the preparation of a graph consider the ranges of the data to be plotted and then decide what scale units are most suitable for each division on the axes of your graph. You should select the number of units conveniently represented by each division on your graph paper. This should be done so that the data you plot doesn't result in a graph which is too small for accurate readings or so large that all the points can't be placed on the graph. Label (print) each axis with the appropriate property and the unit employed to measure that property. Indicate the scale on each of the axes over its entire length.

Mark a sufficient number of units so that the graph can be read easily. By convention, the horizontal axis (abscissa) represents the property that is independently varied (i.e., chosen at random), while the vertical axis (ordinate) represents the dependent property that is observed as a result. In Fig. A, various values of pressure are placed on a gas; various values of volume are observed as a result, therefore the volume is dependent on the pressure.

Plot the points with a sharp pencil. Represent each reading by a small "x" and always, maintain accuracy to a tenth of a scale division. Now draw a smooth curve. It is not necessary for the curve to pass through all the points, but it should pass through as many as possible. Uncertainties produced by experimental errors cause the data points to fall above and below the smooth curve. Hence the graphical picture reveals the reliability of the measurements involved. The smooth curve "irons out" these uncertainties and provides a convenient basis for predicting the volumes (Fig. A) at intermediate pressures which were not measured. This type of prediction is known as interpolation. If the curve (Fig. B) appears to be a straight line draw it with a ruler. When the curve is extended beyond the known, or attainable data, the type of prediction is known as extrapolation. Finally, title your graph to indicate the relationship that it shows.

Fig A



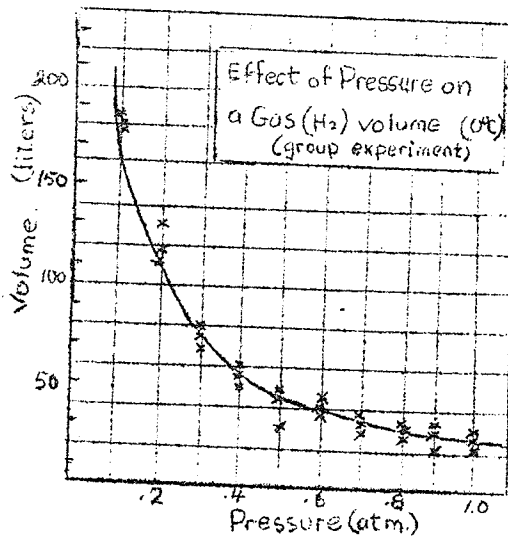
When group data is being plotted two methods may be followed.

(a) If the results recorded in the group data table are in close agreement, they may be arithmetically averaged, and these averages plotted on the graph.

(b) Usually, when the results differ markedly, all the data values are plotted on the graph (Fig. C). A smooth curve is drawn with an equal number of points located above and below the curve, to represent the general result. If some of the points disagree markedly from the others, they may be assumed to be an error and therefore ignored when the curve is drawn.



Fig. C



### R-8. Conclusions

Now you should be able to arrive at a conclusion which is related to your statement of the problem. Actually, if you have planned your investigation properly you have already anticipated certain possible results, and you have already thought about different conclusions which you might reach. Now, however, is the time to decide which of these conclusions is actually indicated by the data. It is possible that the data does not point to any of the conclusions which you anticipated. In such a case you must be ready to discard all conclusions which you thought about, and reach a new conclusion which is based on these unexpected results. In some of your experiments a statement of your numerical result will suffice here.

Don't be the "bubbling-over" type who has a run-away attitude and jumps to conclusions. You might then be like the man who said, "All Indians walk in a single file". When asked what evidence he had for this he replied, "The one I saw did." Avoid making your conclusion too broad. A conclusion is valid only for the precise conditions of the experiment, therefore do not report conclusions that you read somewhere, but only what you can conclude from your own data.

On the other hand, don't be the too-cautious type with the "lean-backwards" attitude, who avoids reaching any conclusion. It is possible that a negative conclusion may be just as important as a positive one. It is just as important to know what a substance can't do, as what it can. Also, there is nothing wrong with stating that your conclusion is a tentative one, subject to further testing and investigation. Nor is there an objection to explaining that your conclusion is limited by many conditions which you realize may make the conclusion inaccurate. In a case where you find that you did not demonstrate anything, do not hesitate to say so. Try to reach a conclusion which is the direct outgrowth of only the observed facts.

Evaluate the accuracy of your results. If these results are quantitative, you will be required to calculate the percent error. For a qualitative experiment the conclusion may be evaluated on the basis of the assumptions upon which the whole laboratory procedure is designed. Identify the sources of error in your experiment and state the effect of these errors on your experimental results; also suggest methods of avoiding such\*\*

Not all investigations turn out successfully even in the hands of trained scientists. Of course, most of us hear only about the successful experiments and this gives us the false idea that scientific research is easy. It isn't. Yet things may not be as black as they seem. "This experiment failed to answer the question for which it was intended." is a conclusion. Perhaps you may learn as much from such a "failure" as you would from a successful experiment. Ask yourself the following questions. How much of the result was my fault? How much was unavoidable? Would there be any point in repeating the experiment? What steps could be taken to avoid "failure" again? Discuss these with care in your laboratory report. Your instructor may be convinced that you have learned something from the experiment if you can answer those questions to his satisfaction. If he feels that the errors were due to lack of preparation and careless techniques, or that you still do not understand the experiment, then you will be required to repeat it.

R-9. Extensions:

Although only one experimental approach to a problem is suggested in the manual, there are often other procedures which could be used for each problem. This could involve the use of different equipment, techniques or reagents. Also, there are many ideas related to the original problem which might be investigated. Any such study would be considered an extension of the original experiment.

Suggestions for possible extensions will be given at the end of some of the experiments. After consulting your instructor about the safety of your procedure, you may begin your investigation before the end of the laboratory period, or you may return to the laboratory before school hours to pursue the extension. All the extensions that you carry out should be reported in your laboratory notebook.

\*\*errors in repeating the same experiment, or in carrying out future experiments.

## R-10. The Laboratory Report

The experimental data gathered in the laboratory should be entered directly in your notebook during the period. If it is necessary to delete or change any such entry, the item should be crossed out in such a way that the original writing can still be read. Recorded "raw" data should never be erased. Such practices will assure the establishment of recorded entries appearing in a notebook as a permanent record of the work done while carrying out a laboratory investigation.

For each experiment, the laboratory report should include such information as:

- (a) The date of writing.
- (b) The title of the experiment
- (c) A concise statement of the problem being investigated
- (d) A record of any pre-laboratory preparation required.
- (e) An account of all the laboratory procedures which are not already described in the manual.
- (f) The code letters or number of any assigned unknowns.
- (g) A detailed record of observations
- (h) A precise record of weights and measures.
- (i) A tabular presentation of data when suitable.
- (j) Quantitative information obtained from the "raw" data by suitable calculations or by graphing procedures.
- (k) Conclusions based on the actual laboratory observations.
- (l) A calculation of percent error and the location of error sources.
- (m) A discussion of the results, including suggestions for improvement.
- (n) Equations for reactions where suitable.

Note that the following information would not be required for the reporting of all experiments: (h), (j), (l), (n).

### R-11. Corrections

Immediately following the post-laboratory discussion and the receipt of the corrected copy of your report, check to see whether your work is acceptable. Read your instructor's comments carefully and then neatly insert any required corrections into your permanent notebook, or rewrite certain portions of the lab report to correct the errors. If the instructor is not satisfied that you understood the experiment and that you did not carry out the procedures with reasonable accuracy, then you will be required to repeat the experiment.

To repeat an experiment, make the necessary arrangements with your instructor and then report at 8:00 a.m. having made the necessary pre-laboratory preparation. If such an experiment is not completed before the next laboratory period (providing that a reasonable period of time has elapsed), you will not be admitted to the laboratory.

### R-12. Evaluation of Laboratory Work

Each of your laboratory reports will be evaluated from a maximum of ten marks. The following are some major factors which will determine your laboratory mark:

- (a) Self-direction. A good student will show individuality and originality of thought.
- (b) Understanding of the experiment, as checked orally by the instructor during the course of the experiment, or preceding the experiment.
- (c) The thoroughness and clarity of reports.
- (d) The accuracy of numerical results.
- (e) Techniques in handling chemicals and apparatus.
- (f) The use of good safety practices.
- (g) Cleanliness. The condition in which you leave the apparatus and work space.

You will notice that the laboratory mark is not based solely on your lab report, but also takes into consideration other important aspects of laboratory work. To achieve success, the pre-laboratory preparation cannot be overemphasized. Refer back to this section of the manual frequently, since it is important to perform techniques correctly from the beginning and to repeat them correctly when necessary. Poor lab work is the result of sloppy techniques and fuzzy thinking. Learn to think clearly, work neatly and to plan more of the experimental procedures as the year progresses and your laboratory work will be a pleasant experience.

SCIENTIFIC OBSERVATION AND DESCRIPTION

PROBLEM: to develop skill in making and recording scientific observations.

INTRODUCTION:

A carefully planned experiment does not guarantee a successful experiment. Even the best laid plans are useless in the hands of a poor observer. The desired results may occur, but they will pass unnoticed by the careless experimenter.

Everyone think of himself as a good observer. Yet there is much more to this than meets the eye. It requires concentration, alertness to detail, ingenuity and also patience. It even takes practice!

See how complete a description you can write about a familiar object such as a burning candle. Be scientific about this and do your study under "controlled" conditions. But how do we know what conditions need to be controlled? Here are some conditions that might be important in your experiment:

The windows are open.

Your lab table is near the door.

You are close enough to the candle to breathe on it.

Why are these conditions important? Do they have something in common? Yes, there is a common factor that a candle does not operate well in a draft.

LABORATORY PROCEDURES:

Examine the candle carefully and record your observations in your lab notebook, using complete sentences. Light the candle and observe it under controlled conditions. If your conditions are changed during the course of the experiment, don't fail to record it before continuing your observations.

CONCLUSIONS:

From the comparison study of your observations with those of professional chemists, summarize the important features of scientific observation and description which are necessary for successful work in any laboratory.

EXTENSIONS: (or questions for class discussion)

a) Are we really sure that things are as they appear to us? To study this question, use the library to locate information on optical illusions and the accuracy of observations made by crime witnesses.

b) How can scientists extend their senses and improve their observations along with their methods of recording observations? List some methods used by scientists to improve both aspects of observation.

THE METRIC SYSTEM AND MEASUREMENT TECHNIQUES

PROBLEM: to investigate measurement techniques and the metric system.

A. INTRODUCTION:

Chemistry is distinctly an experimental science. The establishment of the truth of the laws and the behavior of matter depends on the careful measurements of various quantities: volume, weight, length, etc.

The metric system of weights and measures is used universally in science because of the simplicity of the system. Since all units of the metric system are obtained by multiplying or dividing by ten, it is sometimes called the decimal system of weights of measures.

PROCEDURE: (a homework assignment)

Before beginning the experiment, read over the appropriate sections of your lab manual to learn the required techniques (T-5, R-6, R-7b).

Using your ruler, measure several objects around the house in both English and metric units. Record your data in a suitable table. Plot this data on a graph, where centimeters are placed along the vertical axis and inches along the horizontal axis. Draw a straight line through as many points as possible.

CONCLUSIONS:

1. From the graph, what is the ratio of cm/in? What is the ratio found by division?
2. Applications: How many cm are there in a foot? a yard? How many cm tall are you? How many inches per 100 cm?

B. INTRODUCTION:

In scientific work the skill of estimating tenths of a division on a scale and instrument dials becomes almost automatic. The only way to acquire the ability is to get some practice.

PROCEDURE (homework assignment)

Measure the thickness of the paper used in your chemistry textbook. Although a single sheet is much thinner than the smallest division on your ruler, a simple procedure will make it possible for you to determine the thickness of the paper quite accurately.

Open your chemistry text near the beginning and also the end. Pinch the stack firmly and place the scale across the edge of the stack. Measure the thickness, estimating to the nearest tenth of a millimeter. Record your data similar to the one below. Calculate the thickness of one sheet by dividing the thickness of the stack by the number of sheets.

Close the book, then reopen it at a new place and record the new data. Repeat this procedure four times. After calculating the thickness of a sheet four times, average your results. If one result disagrees greatly with the other three results are you justified in discarding it and obtaining new data?

Observation*	Page number		Pages in stack	sheets in stack	stack thickness(m)	thickness per sheet
	at front	at back				
					Total	
					Average	

#### CONCLUSIONS:

1. Calculate the range between your smallest and largest value for thickness. Give two reasons why your measurements don't agree.
2. Give a reason for closing the book at the end of each measurement instead of measuring the same number of sheets each time.
3. What are the advantages of taking four readings?

#### EXPERIMENTS:

1. Extend your understanding of measurement by summarizing the class data in a table and calculating the average thickness obtained by the group, along with the average range. You might also include a record of the scatter for each student's values, i.e., the number of readings which were above the class average and the number which were below the class average. A study of such a table should provide further insight into scientific measurement.
2. On a separate sheet of paper construct a right triangle with opposite acute angles at  $30^\circ$  and  $60^\circ$ . You will need a ruler and protractor to do this accurately. Make the base of your triangle at least 15 cm in length. If you have constructed the triangle correctly and measured the length of the lines accurately, the side opposite the  $30^\circ$  angle should be exactly half the hypotenuse. Check it.

G. PRE-LABORATORY PREPARATION:

1. Read the description of the techniques required for this experiment T-5, T-6, T-7, T-8, and R-6, R-7.
2. Prepare a table to be used for recording the data. Organize it in such a way that it will be an aid to calculation.

LABORATORY PROCEDURES:

Weigh an empty beaker. Record this weight. Measure exactly 25ml (cc) of water by means of graduated cylinder. Pour the water into the beaker and weigh again. Record this weight and obtain the weight of 25ml of water from your data.

From your experimental data, calculate the weight of 1 ml of water; the weight of one liter of water.

CONCLUSIONS:

1. What are your calculated values for the weight of 1 ml? one liter of water?

2. How accurate is your answer? Find the difference between your experimental answer and the accepted answer (given by your textbook or other reference). This is your error. To find the percentage error, divide your error by the accepted value and express the result as a percentage

3. Indicate how each of the following affects (+, -, or 0) your calculated answer:

- a) The bottom of the meniscus was read with the eye above the level of the water.
- b) The graduate was not completely emptied of water into the beaker.
- c) The wet beaker was not thoroughly dried at the beginning of the experiment.
- d) The beaker contained a solid which dissolved in the water which was poured in.
- e) The balance was not zeroed before making the weighings.
- f) Different balances were used in the course of the experiment.

EXTENSIONS:

1. Obtain objects of unknown weight from your instructor. Record your weighings. Your instructor will then check your results against his own.



EXTENSIONS:

2. Since the volume of a single drop of water is too small to measure accurately with a graduate, devise a method by which you could indirectly measure the volume of a drop of water with considerable accuracy using only your dropper and your graduate.
3. Compare the accuracy of a graduated cylinder with that of a pipette. Run exactly 1 ml of water into a dry 10 ml graduate and check the volume of the graduate. Continue with larger portions, each time checking the error between the volume delivered by the pipette and the volume recorded in the graduate. Calculate the % error for 10 ml of water. Why is the pipette more accurate than a graduated cylinder?
4. Using a method similar to the one that you devised in (2), calibrate a dropper by counting the number of drops that it requires to deliver 1 ml. Be sure to make a permanent record of this information since it is not constant for every dropper. This calibration will allow you to use the dropper as a pipette.
5. Devise a method to accurately determine the weight of one filter paper. State any assumptions made in arriving at solution.
6. Graph the relationship between the value and weight for several coins. Weigh a penny, nickel, dime, quarter, half-dollar, and silver dollar; graph the data. Examine the line drawn and describe the relationship between the value of the coins and the weights. Explain the position of the penny and nickel outside the line. From your graph find the weight that corresponds to sixty cents on your graph, then check your answer by weighing a half-dollar and a dime.
7. Check the accuracy of your balance against a set of accurate standard weights, beginning with the largest weight in the set. Record the weights in a way which will bring out the difference between the balance reading and the standard. If the standard weight is more, mark the error (+); if the standard weight is less than the balance reading, mark the difference (-).
8. Check the sensitivity of the balance: Half fill a 50 ml beaker with water and place it on the pan of the balance. Adjust the weights so that pointer is at zero. With a dropper pipette, add water, a drop at a time. Check the position of the pointer each time. Repeat until you determine the number of drops required to cause sufficient change in the position of the pointer so that a re-adjustment of the weight is necessary? Can you now convert the sensitivity into units of weight?

USING DENSITY AS AN IDENTIFYING PROPERTY

- PROBLEM:
- 1) to find the density of an "unknown" substance.
  - 2) to investigate the possibility of using density as a means of identifying an "unknown" substance.

INTRODUCTION:

The identification of chemical substances is based on their characteristic properties. In this experiment the property to be used for identification will be density. Density is defined as the weight of any substance per unit volume (at a given temperature.) In the metric system this ratio is expressed as grams per cubic centimeter (g/cm) or grams per milliliter (g/ml).

Weight and volume are properties of a specific object, while density is a property of the material of which any object is composed, rather than of the object itself. It is therefore independent of the size or quantity of any given sample. For example a splinter in your hand has the same density as the board from which it came although the weight and volume of the splinter are much smaller than those of the board, the ratio of weight to volume for the splinter is that for the board.

PRE-LABORATORY PREPARATION:

1. Obtain from a reference such as a chemistry handbook and record in your notebook the densities for the substances listed by your teacher. These are known as literature values.
2. Prepare a table for collecting data in the laboratory similar to the table below. Rearrange the order of your data in your table to make the calculations more convenient.

	Trial		Average
	1	2	
Sample No.			
Weight of Sample			
Volume of water in graduate			
Volume of water and sample in graduate			
Volume of sample			
Density of Sample			

LABORATORY PROCEDURES:

Weigh the unknown substance assigned to you to the nearest hundredth of a gram. Record the weight and identifying number of the unknown in your notebook.

Fill the graduated cylinder to half of its capacity. Read the volume accurately and record its value. Carefully add your unknown to the water in

the cylinder. Be sure that the sample is completely immersed in the water and that no air bubbles are trapped below it. Record the new volume of water. Thoroughly dry the sample before the second trial.

Using the data collected, calculate the volume of the sample and also its density.

### CONCLUSIONS:

Compare your calculated density (experimental value) for your unknown substance with the literature values for the group of known substances being considered. On the basis of your comparison, draw a conclusion about the identity of the unknown assigned to you.

Discuss the possible limitations of this identification including a consideration of the uncertainty of your measurements. In view of these considerations suggest a possible procedure by which additional data such as other characteristic properties could be obtained.

Discussion of Errors: Indicate how each of the following affects your calculated density. (+, - or 0). Your instructor may ask for a complete discussion of any of these possible errors.

- An air bubble is trapped under the sample in the graduate.
- The sample was not totally immersed in the water.
- You read the upper instead of the lower portion of the meniscus in the cylinder during both readings.
- You read the upper portion for the first reading only.
- You read the upper portion of the meniscus for the second reading only.
- Alcohol (density 0.79 g/ml) was accidentally substituted for water (density 1g/ml).

### EXERCISES:

- Find the density of a block of wood by a second procedure. Compare your answer to that obtained from the first procedure. Which is more precise? Explain.
- On the basis of the first experimental procedure, devise a method for determining the density of an insoluble granular solid, e.g. fine sand.
- Devise an experimental procedure by which you could find the density of a liquid. Perhaps you could determine the density of the same liquid by three different methods.
- The density of mercury is 13.5 g/ml. Draw a graph that shows how the weight of Hg (up to 1 kg) varies with volume. From your graph, find 1) the weight of 112 ml of Hg, 2) the volume of 0.315 kg of Hg.

USING THE BUNSEN BURNER

PROBLEM: to discover the height at which a beaker of water should be placed above the burner to obtain the maximum temperature from the flame.

INTRODUCTION:

The production of a flame involves a chemical reaction between two gases, a fuel gas and oxygen of the air. The temperature obtained in the reaction depends on the nature of the gases themselves, on the presence of any inert gas (e.g.  $N_2$ ) which also must be heated, and on the rate and completeness of mixing and burning of the gases.

Very often students are seen heating objects in the wrong part of the Bunsen Burner flame. Such inefficient use of apparatus results in a waste of both time and fuel. You will discover in this experiment that there is one arrangement of apparatus for so simple a job as heating water in a beaker that gives maximum efficiency.

You will place an iron ring at a given height above the top of the Bunsen burner and find the time required to boil the water under these conditions. You will then change the height from the top of the burner to the iron ring and repeat the procedure for different heights. The height providing the highest temperature, and therefore requiring the shortest time to boil the water is then found.

In order to arrive at a valid conclusion in this experiment, the starting conditions for each test should be identical. This is a controlled experiment, with only one variable - the different heights of the beaker above the burner. In this experiment there are several constants, or identical starting conditions, such as the volume of water in the beaker, the starting temperature of the water, etc. The pre-laboratory assignment is the key to the success of this experiment.

PRE-LABORATORY PREPARATION:

1. Study the description of the Bunsen burner in A-20 and T-1.
2. Carefully review the topic of controlled experiments. Then, in your lab notebook, list the conditions to be controlled. Describe how you plan to keep each of these conditions constant.
3. Prepare a data table for time and height recordings.

LABORATORY PROCEDURES:

Place the burner on the base of the lab stand. Adjust the position of an iron ring on the stand so that it is 2.5 cm above the top of the burner. Using a ruler and a pencil, mark off the present position of the center of the clamp and the successive positions to be used for each test.

Adjust the burner flame so that the inner blue cone is clearly defined. The flame should not make a roaring noise, nor should it be burning at its maximum height.

Each test should require no more than about 5 minutes. Therefore, choose a reasonable amount of water to add to the beaker so that it will reach the boiling point in the time available. During the heating, stir the water gently with a thermometer, and record the time taken to reach the boiling point.

### CONCLUSIONS:

1. As a result of examining and interpreting your tabulated data, arrive at one or more definite conclusions. At what heights above the burner does the water boil in the shortest time?
2. If your data does not provide sufficient information to arrive at such a conclusion, do not hesitate to say so. If your data is insufficient suggest reasons why this is so.
3. Evaluate your techniques in controlling the conditions of this experiment. If you were to repeat this experiment, how could your techniques be improved?

### EXTENSIONS:

1. Fill a test tube half full of water. Using a test tube holder, bring the water to a boil over the Bunsen flame. If you can do this without the water boiling out of the tube, you have mastered the technique. If not, keep repeating until you are successful.

2. You have determined the position of the hottest part of the flame by experimental means, but it is also desirable to learn how hot the Bunsen flame is. Thermometers for such high temperatures are not readily available. However, it is possible to estimate the flame temperature by testing substances with a variety of known melting points to see if they can be melted. This may be done by heating a few small crystals of the substance in a clean, dry deflagrating spoon. Some common chemical compounds and their melting points are:

ZnCl <sub>2</sub>	264°C	MgCl <sub>2</sub>	712°C	NaCl	800°C
ZnBr <sub>2</sub>	394	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	741	H <sub>2</sub> SO <sub>4</sub>	334
CuCl <sub>2</sub>	498	CaCl <sub>2</sub>	772	Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	938
NaI	651				

3. The temperature gradient throughout the length of the Bunsen flame can be obtained approximately by placing an iron wire in the flame at different levels. The color of the wire change with temperature as follows: 500°C (glow), 700°C (red), 900°C (orange), 1100°C (yellow) and 1500°C (white).

4. The temperature of the Bunsen flame may also be obtained by calorimetry. For further information refer to page 142 of the "Science and Maths Weekly", December 5, 1962. You might find it interesting to compare the results obtained by all three methods.

MELTING POINT OF A PURE SUBSTANCE

PROBLEM: to determine the melting point of a pure substance by two different methods.

INTRODUCTION:

A pure substance (element or compound) melts sharply at a definite temperature. Determination of the melting point of a substance, then, provides a convenient means to identification, since each substance has its characteristic melting point.

PRE-LABORATORY PREPARATION:

1. Review Heating techniques, T-2 and Graphing, R-7b
2. Prepare tables suitable for recording data (time, temperature, observations) for parts A and B.
3. Set up the axes of the graph and identify.

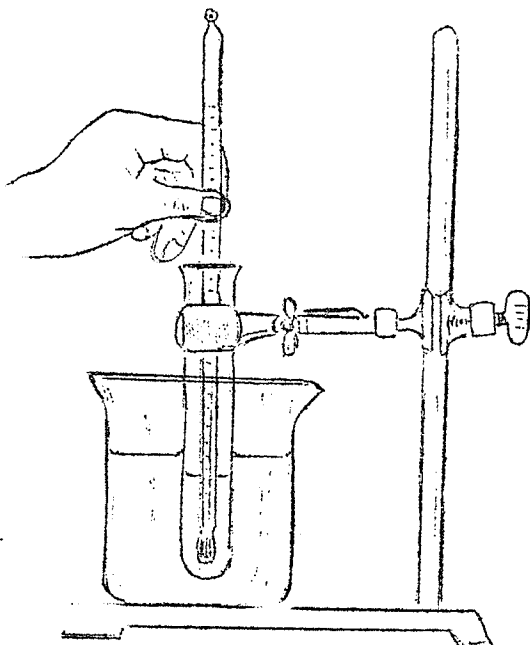
LABORATORY PROCEDURES:A. Cooling Behavior

1. Fill a 400 ml beaker to three-quarters capacity with tap water at about room temperature (25°C). Place the beaker on the base of your lab stand.

2. Obtain about 15 grams of the compound para-dichlorobenzene in a test tube from your instructor. Gently heat the test tube with the burner adjusted to a low flame. Continuously move the test tube back and forth. Gradually increase the temperature until the solid melts. Then place a thermometer in the liquid and continue heating until the temperature has reached the 65-75°C range.

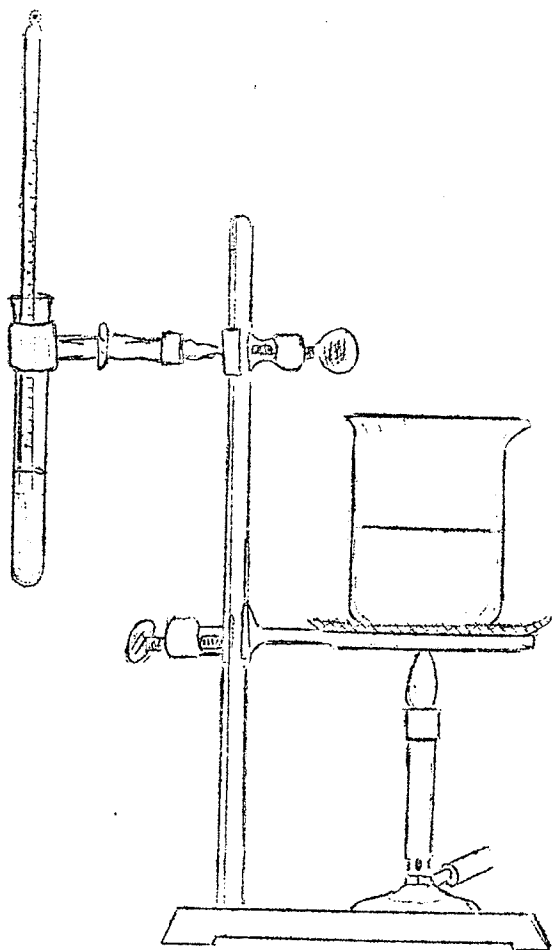
3. Clamp the test tube containing the melted compound into position above the beaker of water. Before taking the next step, be certain that you and your partner are organized: One partner should be prepared to read the time every 30 seconds and at the same time record the observations (temperature, appearance, etc.) noted by the other partner.

4. Read the temperature of the para-dichlorobenzene to the nearest 0.2°C. Record this as the temperature at 0 seconds. Immediately immerse the lower half of the test tube into the water bath and clamp the tube into position. Hold the thermometer against the side and just off the bottom of the tube so that it will be "stuck" in that position when the compound solidifies. Record the temperature every 30 seconds until a temperature in the upper thirties is reached. In your observations, note when solidification starts and when it is complete.



### B. Warming Behavior

Set up the apparatus as shown in the diagram. Raise the test tube out of the water and turn the clamp around so that the test tube is on the opposite side of the lab stand, opposite the waterbath. Support the waterbath on an iron ring and wire gauze. Heat the water to approximately  $70^{\circ}\text{C}$ . How can you save time in this last step? Then, turn off the burner, but leave it in position. During the course of the experiment, occasionally read the temperature of the water with a second thermometer to avoid cooling of the water below  $60^{\circ}\text{C}$ . Therefore be prepared to use the burner, to keep the temperature between  $60^{\circ}\text{C}$  and  $65^{\circ}\text{C}$ , if necessary.



Read and record the temperature of the solidified dichlorobenzene to the nearest  $0.2^{\circ}\text{C}$ . Immediately immerse the lower half of the test tube into the waterbath so that the level of the compound in the tube is below the water level. Record the temperature of the compound every 30 seconds, noting when it is complete.

When the solid is freed of the walls of the test tube, move the thermometer gently up and down. Record the time and temperature until the temperature of the para-dichlorobenzene is about  $60^{\circ}\text{C}$ .

### C. Processing the Data:

Plot the cooling and heating temperatures on the same graph, starting your plotting from the left edge of the graph. For each cooling temperature and its corresponding time, make a small cross. For the heating data use small circles so you can distinguish between the plotted points. Using a black pencil, draw a smooth curve to represent the heating behavior of the para-dichlorobenzene. Using a red pencil, do the same for the cooling behavior.

A graphical form of the data will help you to arrive at some conclusions. Organize your graph so that it will be the size of a full page. Which axis should be used for the time and temperature? Which of the data is independent of the other? The independent variable is plotted along the horizontal axis.

CONCLUSIONS:

1. Study the experimental results as summarized in your graph. What is the melting point of the para-dichlorobenzene. What is the freezing point?
2. In a short paragraph discuss the shape of the heating and cooling curves. If a greater amount of para-dichlorobenzene was used, what affect, if any, would this have on the shape of the curves?

EXTENSION:

Using the cooling method, investigate a series of materials such as paraffin, beeswax and naphthalene. An air bath can be substituted for the water bath to obtain quicker results. Determine which of the materials are pure substances and which are mixtures.



## Experiment 6

AN INVESTIGATION INTO PHYSICAL AND CHEMICAL CHANGES

PROBLEM: to develop skill in recognizing chemical and physical changes when they occur.

INTRODUCTION:

Regardless of whether a change is physical or chemical, there are frequently changes in the appearance of the material. The nature of these visible alterations often gives a clue as to what is happening to the materials being treated, so it is very important that you make a habit of observing carefully. Observe the substance not only at the beginning of the experiment but all the way through it, and look for changes which occur and which may give you a hint as to what is happening. It is not enough simply to make the observations; they must be recorded accurately and completely. What appears insignificant at the time may prove later to be vitally important in understanding what has taken place. Therefore make it a habit to record every observation completely and accurately.

PRE-LABORATORY PREPARATION:

1. Review your knowledge of physical and chemical changes from your text and notes.
2. Review the techniques required for this experiment, T-16, T-7,
3. Prepare in your laboratory notebook, a table similar to the one below for collecting the data to be recorded. Turn your notebook sideways and expand the table to avoid crowding your notes.

	OBSERVATIONS			CONCLUSION (type of change)
	properties before	properties during	properties after	
1. salt + water				

LABORATORY PROCEDURES:

1. Into a 25 ml of water in a beaker, dissolve as much sodium chloride as you can. From the clear solution pipette 5 ml into a clean evaporating dish. Save the remaining portion for a later test. Place the dish on a wire gauze and ring stand and heat with a gentle flame. When the solution has nearly evaporated remove the flame and let the solution evaporate to dryness from the heat that it still remains, otherwise it will spatter. Compare the taste of the residue to that of the original solution. Compare the taste of the residue to the original salt.
2. Add sulfuric acid to one inch of sugar placed at the bottom of a beaker. Note any change in temperature which may occur by using a thermometer. Also note carefully any change in appearance.
3. Examine a platinum wire. Heat the wire in the tip of a Bunsen flame, then allow it to cool. Examine it again. What was the source of heat in this experiment?

4. Clean a 10 cm strip of magnesium ribbon with sandpaper. Note its appearance and flexibility. Hold one end of the ribbon in the tongs and bring the other end to the flame. Remove once the reaction has begun. What was the source of heat in this reaction? Caution: Do not look directly at the ribbon during the reaction (to protect your eyes). Place the product on a cover glass and examine it carefully.

5. Pipette 5 ml of sodium chloride solution from part A into a test tube. Place a thermometer into the solution and read the temperature ( $^{\circ}\text{C}$ ). Add to it a few drops of silver nitrate solution. Caution: Silver nitrate solution reacts with the skin and will stain it black. Clean hands the day following this experiment indicate good laboratory techniques. Note any temperature change, and compare the appearance of the products to the original solutions.

6. Pour 20 ml of copper sulfate solution into your smallest beaker. Into this solution place an iron nail (shiny). Examine the products and note any temperature change during the reaction. If zinc powder is available, add a spatula full instead of the nail, and note the changes. Would a second beaker be useful here as a control?

7. You will be given pairs of "unknown solutions". Record the code numbers in the data table. Fill a test tube to  $1/3$  capacity with solution A. Record the starting temperature of the solution. Add a similar amount of solution B to another test tube. Will it be necessary to measure the temperature of both solutions? Mix the two solutions. Record any change in temperature and in appearance.

#### CONCLUSIONS:

1. Immediately following each change investigated, decide what type of change occurred and record, this in the appropriate column of the data table.
2. Make a list of the kinds of behavior (heat given off, etc.) which you observed during the performance of these tests and which could have given you a clue that chemical change was occurring.

#### EXTENSIONS:

1. Thus far your interpretation of the observations depended on changes in physical properties and energy changes. Another method of identification is to determine if there is a difference in the reaction of each of the reactants with some reagent, e.g., alcohol, hydrochloric acid, etc., as compared to the resulting product(s) reacting with the reagent. In other words, you are checking for changes in chemical properties. Test a small sample of magnesium ribbon with hydrochloric acid in a test tube. Hold another sample in the burner flame (with tongs), then add the product(s) to another test tube of hydrochloric acid. Has there been any change in chemical properties?

2. In a chemical change there is an alteration in the properties of the substances involved. Is there a change in weight during the reaction? Devise a method to investigate this problem.

## Experiment 7

HEATING EFFECTS OF A PHYSICAL AND A CHEMICAL CHANGE

PROBLEM: to make a quantitative investigation of the energy changes accompanying a chemical change and a physical change.

INTRODUCTION:

In the last experiment you gained experience in recognizing physical and chemical changes when they occurred. You discovered that chemical changes can be recognized by a release in heat energy. However, both physical and chemical changes may be accompanied by an intake or a release in heat energy. An example of such a physical change is a change of state or phase change, such as solidification or vaporization. In this experiment you will make a quantitative investigation of the relative amounts of heat energy accompanying a chemical change and a phase change.

The heat produced by these changes will be absorbed by water, resulting in an increase in the temperature of the water. One calorie of heat will raise the temperature of 1 g of water one degree Centigrade. Ten calories of heat will raise the temperature of 1 g of water 10 degrees Centigrade. One hundred calories of heat will raise the temperature of 10 g of water 10 degrees Centigrade. The quantity of heat (calories) is obtained by multiplying the temperature change by the weight of the water in grams.

PRE-LABORATORY PREPARATION

1. Review the techniques of weighing, T-8.
2. Prepare a data table for parts A and B.

LABORATORY PROCEDURESA. Heat of Combustion

In this part of the experiment you will determine the amount of heat liberated when a candle burns. The amount of candle used is obtained by weighing the candle before and after a part of it has been burned. The heat obtained from the candle is used to warm a weighed amount of water.

1. Fasten a candle to a tin can lid and weigh them on a centigram balance to the nearest .01 g. Record this weight in your data table.
2. Weigh an empty can (with two holes punched in opposite sides near the top) on a platform balance to the nearest 1 g. Record the weight in the data table.
3. Assemble the apparatus as shown in the diagram. The height of the small can should be great enough to prevent the candle flame from touching the bottom when it is lit. For the chimney use a large can opened at both ends and with two or three holes punched near the bottom for ventilation.

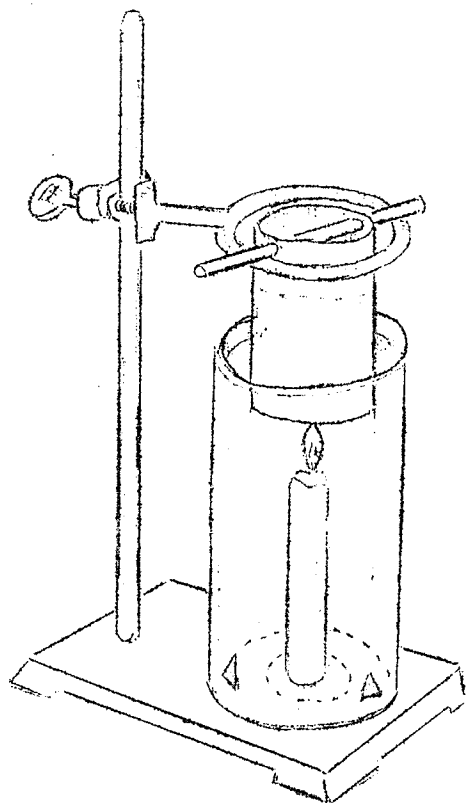
4. Fill the weighed can to about two-thirds capacity with cold tap water. The water should be about 10-15°C below room temperature. Ice may be added. Once the water is at the desired temperature be sure to remove any remaining ice.

5. Read and record the temperature of the water to the nearest 0.2°C. Ignite the candle and heat the water. Stir gently until it reaches a temperature about as much above room temperature as it was below at the start. Blow out the candle and continue to stir the water until the highest temperature is reached. Record this highest temperature, as before, to the nearest 0.2°C.

6. Weigh the candle on the same centigram balance as before. Should you discard any drippings, or weigh them along with the candle? Record the weight.

7. Weigh the can and the water on the same platform balance that was used before, and record the weight.

8. Repeat the experiment if time permits.



### Data and Calculations

Include the following data in your table: the weight of the candle before and after burning; the weight of the empty can; the weight of the can and water; the temperature of the water before and after heating; and room temperature.

Organize the above data to calculate the following directly from the table: the weight of the candle burned; the weight of the water heated; and the temperature change.

Calculate the quantity of heat required to warm the water and also the heat of combustion of the candle material (calories per gram)

### B. Heat of Solidification

In this experiment you will use some melted wax at its melting point, to produce a change in temperature of a weighed amount of water.

1. Obtain a test tube containing about 10 g of wax from your instructor. Weigh the test tube with the wax to the nearest 0.1 g. Record the weight.

2. Using the same balance, weigh a similar, dry, clean test tube, provided by your instructor, to determine the approximate weight of an empty test tube.

3. On a platform balance weigh an empty 250 ml beaker. Record this weight to the nearest 1g. Fill the beaker to about one-half capacity with cold tap water so that when the test tube is placed in the beaker the water level will be above the liquid wax level. Adjust the temperature of the water to 1 or 2° below room temperature. Weigh the beaker and water using the same balance.

4. Heat the water in the beaker to boiling and place the wax filled test tube into the boiling water bath until the wax has just melted. Avoid overheating.

5. Remove the test tube of hot wax from the water by means of a test tube holder. Allow the wax to cool until the first sign of cloudiness (solidification) is evident. While the wax is cooling, measure and record the temperature of the cold water in the water bath to the nearest 0.2°C. Quickly place the test tube containing the slightly cloudy wax into the beaker and stir the water gently with the test tube. Note the temperature of the water as you stir and observe the temperature until no further change is noted. Record the maximum temperature to the nearest 0.2°C. When reading the thermometer, immerse it in the water midway between the test tube and the wall of the beaker. If time permits, repeat the experiment.

#### Data and Calculations

Include the following data in your table: weight of test tube + wax, weight of empty test tube, weight of empty beaker, weight of beaker + water, temperature of water before, temperature of water after.

Organize the data table to enable you to calculate the following: weight of wax, weight of water and temperature change.

Also calculate the heat required to change the temperature of the water (calories) and the heat of solidification of the wax (calories per gram).

#### CONCLUSIONS:

1. Compare the class averages for the heat of combustion and heat of solidification. Calculate the heat of combustion/ heat of solidification ratio. Is this ratio typical of all chemical change / phase change energy ratios?

2. Indicate how each of the following would affect (+, -, or 0) your calculated values.

#### Heat of Combustion

- The wax drippings were not weighed along with the candle after burning.
- The starting temperature of the water in the can was 30°C.
- Some of the heat was transferred from the water to heat the tin can.
- The candle flame was in contact with the bottom of the tin can of water.

Heat of Solidification

- a) the empty test tube was smaller in the size than the test tube containing wax.
- b) The test tube of slightly cloudy liquid wax was not immediately inserted into the water bath.
- c) The temperature of the water was several degrees below the room temperature.
- d) The test tube of liquid wax was placed in the water bath so that the level of the wax in the tube was higher than the water level in the bath.

EXERCISE

Refer to physics reference text and study the topic of heat transfer and calorimeters. Then devise suitable improvements in procedure A to eliminate the chief sources of error. Test your procedure by finding the heat of combustion of another compound. Compare your result to the "accepted" value which is found in a chemistry handbook.

THE PROCESSING OF RAW DATA

**PROBLEM:** To gain practice in the processing and interpreting raw data by making a quantitative study of the reaction between magnesium and oxygen.

INTRODUCTION:

The value of each experiment carried out in the laboratory depends not only on the careful manipulation of chemical and apparatus but also on the manner in which observations and data are correlated and evaluated.

The readings recorded directly from the scales of instruments are known as raw data. Below you are confronted by the raw data recorded by a student who was making a quantitative investigation of the reaction between magnesium and oxygen.

You are asked to "stop in" at this point in the experiment, to process and interpret the data and arrive at a conclusion. Some relationship between two changing quantities or variables might be discovered by directly from data table. However this is often quite difficult. Relationships or trends are more readily discovered by summarizing and presenting the information in a graph. (Study R-7b thoroughly before tackling this problem.)

DATA:Reaction of Magnesium with Oxygen

<u>Initial Weight of Mg</u>	<u>Initial Volume of O<sub>2</sub> at 0°C and 1 atmosphere pressure</u>	<u>Weight of Product</u>	<u>Weight of O<sub>2</sub> reacted</u>
1.20	0.10 liters	1.34 grams	0.14 grams
1.20	0.20	1.49	.29
1.20	0.30	1.64	.44
1.20	0.40	1.78	.58
1.20	0.50	1.93	.73
1.20	0.60	2.00	.80
1.20	0.70	2.00	.80

PROCEDURE:

In your laboratory notebook make a graph of the above data. Plot the weight of O<sub>2</sub> used on the ordinate (vertical or y-axis) and the volume of O<sub>2</sub> on the abscissa (horizontal or x-axis). Draw a smooth curve through the points

CONCLUSIONS:

What can you conclude from the graph you constructed? What law is illustrated by this experiment? Could any useful information be obtained by plotting the weight of magnesium oxide produced against the weight of oxygen used?

THE FORMATION OF PRECIPITATES  
A QUALITATIVE OBSERVATION OF CHEMICAL CHANGES

PROBLEM: to prepare a precipitate from two solutions; separate the reaction mixture by filtration, and to discover which solution was in excess.

INTRODUCTION:

One of the evidences accompanying a chemical change is the formation of a precipitate. When a solution of sodium chloride and a solution of silver nitrate are mixed, a precipitate of silver chloride is formed. The solid silver chloride can be separated from the solution by filtration. In this process the silver chloride is held back by the filter paper; the solid is called a residue. The solution passing through the filter paper is known as the filtrate.

Your problem is then to discover which of the two solutions was in excess. Your conclusion will be a qualitative one since you will discover which solution is in excess. If you collected further data which could lead you to answer that a solution was excess by  $x$  grams per liter, the conclusion is then quantitative.

PRE-LABORATORY PREPARATION:

1. Thoroughly review the techniques of filtration (T-14)
2. Organize a flow sheet to represent the procedures you will use for this experiment. First study the information (R-7) on this subject with care. Your instructor will want to examine your flow sheet before you begin the experiment.

LABORATORY PROCEDURES:

1. Record the code number of the solutions assigned to you. Use the same solutions throughout the first trial of the experiment.
2. To 3 ml of sodium chloride solution in a test tube, carefully add an equal volume of silver nitrate solution. Stir the mixture. Place the tube and its contents in a beaker of boiling water for a few minutes to coagulate the precipitate. Allow the silver chloride to settle to the bottom of the tube.
3. Filter the cooled reaction mixture through a moistened filter paper in a funnel and collect the filtrate in a 10 ml graduated cylinder. Record this on your flow sheet.
4. Transfer all of the precipitate to the filter paper. Place the residue and the filter paper on a watch glass to dry. Expose the precipitate to direct sunlight or place it under an ultraviolet lamp. Record the appearance of precipitate before and after exposure.



5. Divide the filtrate into equal portions. Label one portion "Filtrate 1a" and the other "Filtrate 1b". To Filtrate 1a, add an equal volume of sodium chloride solution. To Filtrate 1b, add an equal volume of silver nitrate solution. Record the observed results.

6. If a precipitate formed in (a) or (b), filter it and label it "Residue 2." Determine whether this precipitate is identical to the first residue.

#### CONCLUSIONS:

Which of the reacting solutions was in excess? Defend your selection in a short paragraph.

#### EXTENSIONS:

A. Will the addition of silver nitrate solution to the solutions of other chlorides result in the formation of silver chloride? To answer this problem carefully add silver nitrate solution to an equal volume of the chloride solutions which are available. Compare the precipitates produced and determine whether they are silver chloride. Why is it essential to prepare the silver nitrate solution in your reagent bottles by using distilled water?

B. The addition of sodium chloride solution to a solution of lead nitrate produces a white precipitate. Are the two white precipitates the same chemical substance? To answer this problem, compare the two precipitates carefully, noting any change in appearance. Expose samples of both precipitates to ultraviolet light. You might also wish to consult a chemical hand book or other reference to find out the solubility of these chloride precipitates in both hot and cold water.

## Experiment 10

OXYGEN - A VERIFICATION EXPERIMENT

- PROBLEM:** 1) to study the preparation of oxygen and its collection.  
2) to verify some of the properties of oxygen.

INTRODUCTION:

You may be of the opinion that this is not an experiment at all, since you already know the physical and chemical properties of oxygen before you begin. This opinion is not entirely correct. An important characteristic of the scientific facts which are discovered from experiments is that they are reproducible. Any other scientist can read a report of the original experiment and reproduce the results. The work of this type of experiment is one of verification rather than discovery. The ability to reproduce the results of another scientist is a test of your own laboratory techniques and your powers of observation.

PRE-LABORATORY PREPARATION:

1. Review the techniques required for this experiment, T-2 and T-17.
2. Organize a flow sheet to represent the laboratory procedures that you will follow. Turn your laboratory notebook sideways and use the whole page to provide sufficient space for recording your observations.

LABORATORY PROCEDURES:A. Function of Manganese Dioxide

To study the function of manganese dioxide in the reaction mixture, place enough potassium chlorate in the bottom of a large test tube to cover the curved portion. Clamp the tube vertically on a lab stand and heat it with a Bunsen burner. When the salt begins to melt, test for oxygen with a glowing splinter. Caution: Be careful not to drop the splinter into the test tube. If this happens remove the heat and prepare a new sample.

Once the test for oxygen is obtained, remove the flame and allow the potassium chlorate to cool until the bubbling just subsides. Now add a pinch of manganese dioxide to the contents of the tube and immediately test for oxygen. Compare the results produced before and after adding the manganese dioxide.

B. Preparation and Collection

Weigh out 3 grams of potassium chlorate and 1.5 grams of manganese dioxide. Mix these two compounds thoroughly on paper. Caution: Any attempt to grind the mixture in a mortar may result in an explosion. Transfer the mixture to a test tube and set up the apparatus as shown on page 72.

Fill the collecting bottles brimful with water. Slide a cover glass over the mouth and invert each bottle into the pneumatic trough, removing the cover under water. Similarly fill 3 other bottles with water and invert these in the pneumatic trough as needed.

Gently apply heat near the surface of the mixture and as melting occurs move the flame downward. Caution: Any contact of the hot potassium chlorate with the rubber stopper may produce an explosion. Let a little of the gas escape into the air, tc. permit the generator to fill with pure oxygen, then fill the 4 bottles with oxygen by displacement of water. As soon as each bottle is filled, place the cover glass over the mouth and place it right side up on the table.

Remove the delivery tube from the water as soon as the heat is removed from the generator. Why? Record observations on the appearance and odor of the samples.

### C. Verification of Properties:

1. Line a deflagrating spoon with asbestos paper. Place in it a piece of sulfur not bigger than half a pea and ignite it over the burner. Immediately thrust the spoon into a bottle of oxygen, keeping the bottle covered. After comparing the burning of sulfur in air and oxygen, quickly extinguish the burning sulfur under tap water. Record your observations immediately, including a description of the products.

2. After cleaning the spoon, reline it and repeat the above process for red phosphorus.

3. With your tongs hold a strip of magnesium ribbon in the burner flame to ignite it. Then quickly insert it into a bottle of oxygen. Do not injure your eyes by looking directly at the brilliant light. Examine the product(s).

4. Put a little water into a bottle of oxygen to form a protective layer at the bottom. Heat some steel wool, holding it with tongs, in the burner flame until it ignites, and at once thrust it into this bottle. Be careful not to touch the sides of the bottle. Examine the product(s).

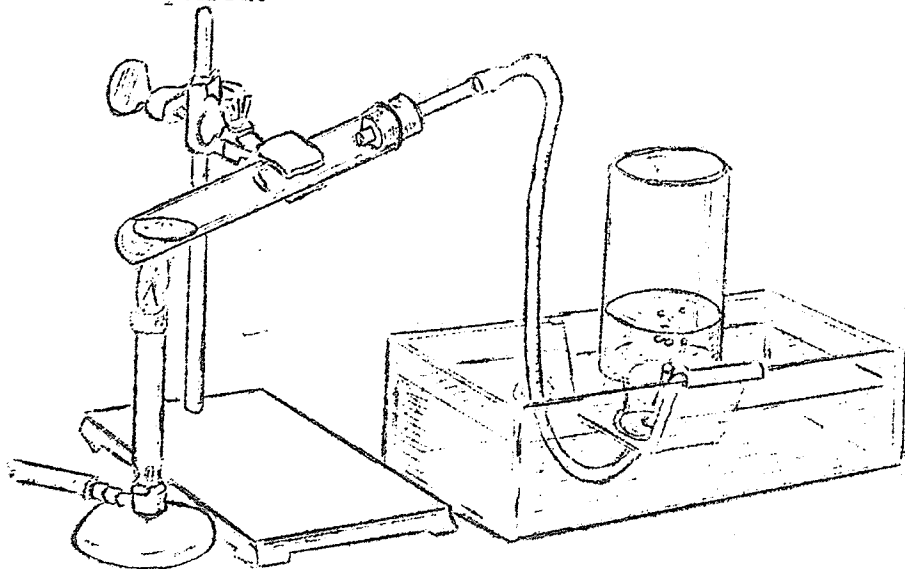
### CONCLUSIONS:

1. Describe the function of manganese dioxide in this experiment. What name is given to such a substance? Write an equation for this reaction, indicating that manganese dioxide was used.

2. By what method was oxygen collected? What property of the gas makes this method of collection possible?
3. Summarize the physical and chemical properties of oxygen that you verified in the laboratory. Include balanced equations where appropriate. Were there any properties of this gas which you could not verify? Discuss anything unusual in your observations and attempt to explain it.
4. What product results when any substance burns in oxygen? From the observations recorded what generalization can you arrive at about the states of this product?

#### EXTENSIONS:

1. You may have noted that procedure A was not a carefully controlled experiment. Devise a procedure which will more carefully control the conditions. Test your procedure, after getting your instructor's approval.
2. Is a catalyst used up in a reaction? To help you answer this problem, here are some suggestions: You can use a carefully weighed amount of  $MnO_2$  mixed with  $KClO_3$ . After decomposition that which remains in the generator is a mixture. You can separate it by making use of the property of solubility of the components. Why should the residue be dry before weighing? Check the weight of the manganese dioxide against the original weight at the start of the experiment.
3. Investigate the possibilities of substituting another substance for manganese dioxide in this experiment.
4. Prepare oxygen by a) the addition of water to sodium peroxide. b) the decomposition of hydrogen peroxide or c) the electrolysis of water. First study the method employed in a reference text and note any precautions required.



## Experiment 11

PROPERTIES OF HYDROGEN - A VERIFICATION EXPERIMENT

- PROBLEM: 1. to study the laboratory preparation and collection of hydrogen.  
2. to verify the properties of hydrogen.

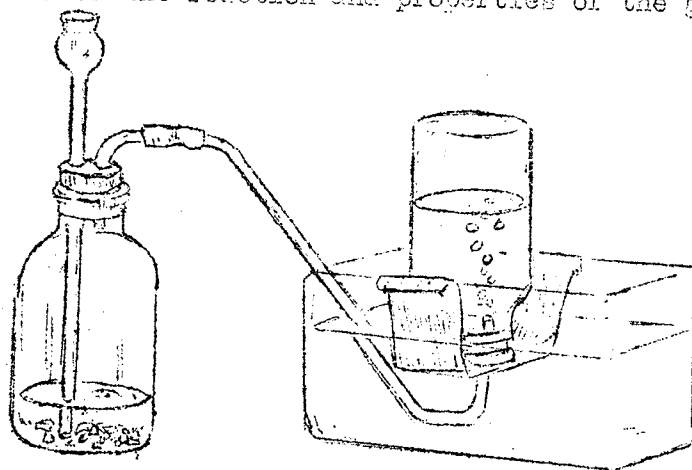
INTRODUCTION: The laboratory work of this experiment is also one of verification rather than discovery. You already know the place of such an experiment in the world of science. A further reason for its use in high school is to emphasize certain laboratory techniques and apparatus that are peculiar to the science of chemistry. In this experiment take particular care in inserting the thistle tube into the rubber stopper, then study its significance in the gas generator during the preparation of hydrogen.

PRE-LABORATORY PREPARATION:

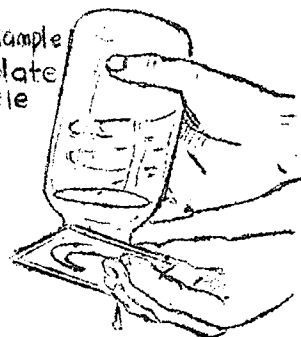
1. Study T-4 and T-9, taking special note of the safety precautions involved.
2. Carefully organize a flow sheet to represent the procedures to be followed in the experiment.

LABORATORY PROCEDURES:Preparation and Collection

1. Set up the apparatus as shown in the diagram. Slide a few pieces of zinc into the flask. Then pour enough water into the flask to seal off the bottom of the thistle tube. Test to see that the generator is air-tight by blowing into the delivery tube to that the water rises in the thistle tube. Then quickly close off the delivery tube with your finger. There are no leaks if the water level in the thistle tube does not sink.
2. Once the instructor has approved your set up, and you are certain that there are not any open flames nearby, pour a few ml of dilute sulfuric acid into the thistle tube. After the reaction subsides a little more acid may be added. Collect a sample of the gas which is equivalent to the volume of the flask. Discard this sample. Now collect four bottles of pure hydrogen. Set the filled bottles aside, mouth downwards. Record your observations of the reaction and properties of the gas, e.g. color.



To remove sample  
of gas, put plate  
under bottle



3. Dismantle and clean out the generator, rinsing the contents into a container provided by the instructor. The pieces of dark solid in this mixture are zinc. This zinc may be recovered by filtration and used again.

Verification of properties:

1. Keeping the mouth of the sample bottle downward insert a burning splinter. Withdraw the splinter slowly, watching carefully for evidence of the gas and splinter burning. Repeat this as often as gas remains in the bottle.
2. Place a bottle of hydrogen mouth to mouth on the top of a bottle of air. After a few minutes test each bottle for hydrogen. Be careful to keep the second bottle covered while testing the contents of the first bottle. Don't let the results of this test distract you from making careful observations.
3. Turn a bottle of hydrogen right side up and uncover. After about a minute, test for the presence of hydrogen.
4. Carefully lower a deflagrating spoon containing some strongly heated cupric oxide ( $\text{CuO}$ ) into a bottle of hydrogen. Observe and record any resulting changes.

CONCLUSIONS:

1. Discuss the significance of the thistle tube in the gas generator.
2. What is the source of hydrogen in this experiment? Write an equation for the reaction.
3. List the chemical and physical properties of hydrogen that you verified in this experiment. Include balanced equations where appropriate. What properties were you unable to observe or verify? Why? Discuss.
4. Carefully explain the differences that you observed in the burning of hydrogen during the first two tests.

EXTENSIONS:

1. Collect a full tube of hydrogen and pour it into a second tube containing air. How must this be done? Test the second tube for hydrogen to be sure that the transfer is successful.
2. Collect tubes in which hydrogen fills  $\frac{1}{8}$ ,  $\frac{1}{4}$ ,  $\frac{1}{3}$ ,  $\frac{1}{2}$  and  $\frac{2}{3}$  of the tube. Empty the water out and test the explosive properties of each hydrogen-air mixture. Calculate the optimum mixture of air and hydrogen from the standpoint of the oxygen concentration in the air. How does this compare with your experimental observations.

3. Devise a method for the recovery of the second product formed by the reaction between zinc and sulfuric acid. Organize your procedure in a flow sheet, then test it. When the product has been isolated in a dry state, describe some of its physical properties. Would it be correct to state that zinc dissolves in the acid? (i.e. undergoes a physical change)

4. Why would a comparison of the speed of getting hydrogen 1) from cold concentrated hydrochloric acid and magnesium ribbon and 2) from warm dilute sulfuric acid and mossy zinc not be scientific? List five variable factors that would affect the speed of getting hydrogen. Set up controls so you could know how each condition (factor) affects the speed of getting hydrogen.

5. Investigate the effect of adding copper sulfate solution to a hydrogen generator. Mossy zinc is most often used instead of zinc strips to attain faster generation of the gas. The greater surface of the mossy zinc is said to account for the greater speed. But there may be another factor involved also. Perhaps you can identify this other factor by controlling the surface area of the zinc. To each of two test tubes add equal amounts of acid (dil) and equal sized strips of zinc. Add a small amount of copper sulfate solution to only one of the tubes. Can you identify the factor that you study here?

Experiment 1.2

CONSTRUCTION OF A LOGICAL MODEL

PROBLEM: to construct a logical model of an unseen object in a sealed container or "black box".

INTRODUCTION:

Scientists explain the observed regularities in the behavior of matter by using theoretical, logical models. The theories proposed involve atoms, electrons, and chemical bonds between atoms to form molecules. None of these can be observed directly and any properties that are assigned to them must be inferred by interpreting experiments performed with instruments which can be observed.

A parallel situation in a high school laboratory would be to infer the properties of an object contained inside a sealed container or black box by making a series of external or indirect observations. A "black box" represents any physical system which we use to study without understanding its inner functions or knowing its contents.

Some of you may think that this experiment bears little relation to chemistry since it does not involve the use of standard laboratory apparatus. However chemistry is as much a way of thinking and handling observations as of manipulating test tubes and reagents. This experiment illustrates how ideas, thought and experimentation are interrelated in chemistry.

PRE-LABORATORY ASSIGNMENT:

Prepare a table similar to the one below for recording the observations noted during the experiment. Turn your laboratory notebook sideways and use the whole page to avoid overcrowding your notes.

Box #	Question	Manipulation	Observations	Tentative Conclusion (Assumption)

LABORATORY PROCEDURES:

Without opening the box or damaging the box or its contents, you are to make all the observation you can through small, controlled movements or manipulations of the box. Gather data which will enable you to make closer and closer approximations of the size, shape, and some of the physical properties of the object. Some of these controlled movements could be: tilting the box at various angles, shaking the box gently, and rotating it end for end, or side for side. The goal is not to identify the object precisely, but to describe it sufficiently so you can mentally construct a logical model of its general appearance.



In your laboratory notebook, record the number of the box assigned to you. Before subjecting the box to a given procedure or manipulation, record in your table the particular question you want to ask and a description of the manipulation that you plan to use to obtain the answer. When each procedure has been completed, record the corresponding observations. For each step of the investigation present an assumption or tentative conclusion.

#### CONCLUSIONS:

1. Evaluate each assumption or tentative conclusion which you have made by checking whether it is consistent with the other data and conclusions. Discard any inconsistent conclusions, that is, those which don't agree with the other conclusions. Support the rejection of any tentative conclusion by a written discussion.

2. Describe in writing a model which is consistent with all observations and which does not contradict any of your consistent conclusions. Include a sketch of this model in your notebook.

3. Discuss the limitations of such indirect observations. What properties of the object could not be obtained by this procedure?

4. Suggest other manipulations or procedures which might reveal additional data on the object in the box. What additional data would you expect to obtain? What scientific instruments might be used to obtain this information (without opening the container).

#### EXTENSIONS:

Ask your instructor for another black box which now contains more than one unknown object, follow the same procedures to arrive at a logical model of the unknown.

DISTILLATION

PROBLEM: to investigate the purification of water by distillation.

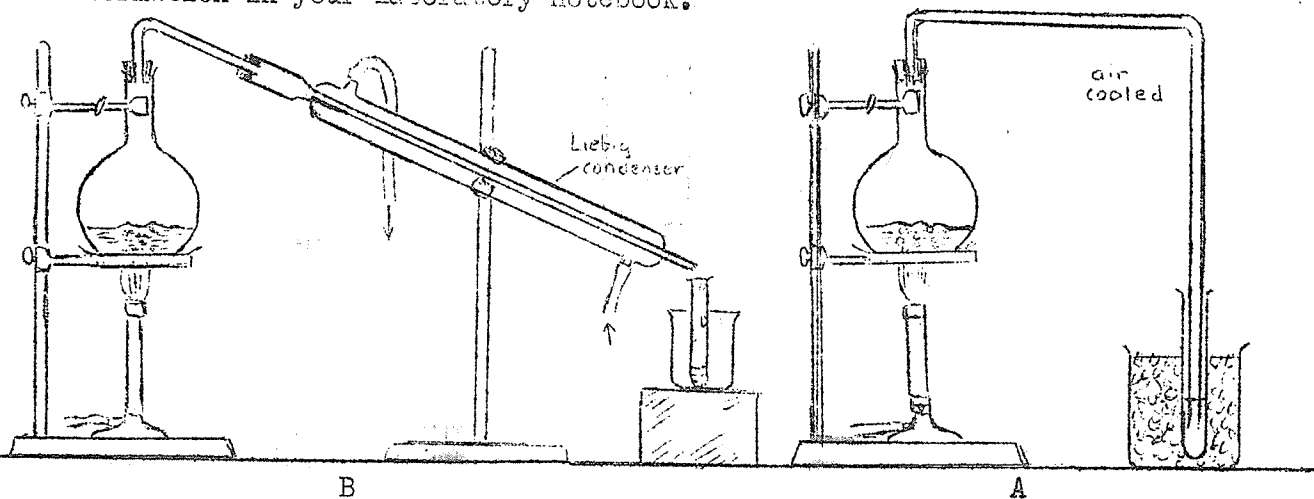
INTRODUCTION:

Impure water may contain dissolved matter that is in solution and undissolved matter that is held in suspension because the particles are too small to settle out. River water, for example, will contain both dissolved solids such mineral salts from soil and rock beside dissolved liquids from sewage, and suspended matter such as clay. Suspended solids are always removed from water to make it potable. Dissolved salts, and liquid may need to be removed if they interfere with certain industrial processes or with chemical reactions.

PRE-LABORATORY PREPARATION:

Diagram A on this page shows a simple inverted "L" glass tube, called an air-cooled condenser. Diagram B shows a Liebig (water-cooled) condenser which is the more efficient of the two. Study the details of both set-ups so you can use whichever one is available. If the air-cooled condenser is used, note its advantages. Also think through the reasons for the particular hose connection arrangement.

Chemists usually need to refer to scientific literature such as a chemistry handbook to fill in their backgrounds before attempting to carry out an experiment in the laboratory. In your experiment, you will be given either a sulfate or chloride salt to use as the soluble solid impurity and ammonium hydroxide as a volatile liquid impurity. Before you can check the effectiveness of the removal of the impurities, you will be required to fill in your own background. By referring to your text or other reference, learn how to carry out a test for (a) sulfate salts, (b) chloride salts, and (c) ammonium hydroxide (a solution of ammonia gas in water). Record this information in your laboratory notebook.



LABORATORY PROCEDURES:

1. Set up the distilling apparatus.
2. Prepare about 50 ml of "impure" water by adding about 2 grams of sodium sulfate or sodium chloride (soluble solid impurity). Stir the mixture until the salt has dissolved.
3. Pour out half a test tube of the "impure" water and place the remainder in the distilling flask. Drop into the flask one or two boiling stones (small pieces of marble or clay plate) to ensure against uneven boiling or "bumping". Begin heating. Test the sample of "impure" water for sulfate or chloride salt, whichever was assigned. Record your observations and set this tube aside to be used as a control.
4. Collect an equal volume of the distillate in a clean test tube. Again test for the presence of the soluble salt. Repeat the process until the test is negative.
5. Repeat the procedure after adding a drop of ammonium hydroxide (volatile impurity) "impure sample". Obtain a sample to be used as a control. Again collect as many tubes (well cleaned) of distillate as required to completely remove the volatile impurity. If more than one tube is necessary, be sure to keep the tubes in order. Test each sample of distillate for the presence of the volatile impurity. Note carefully any indication of different amounts of volatile impurity in the samples, as shown by depth of color. In how many samples of distillate did the volatile material show up? Which sample gave evidence of containing the greatest amount of volatile impurity?

CONCLUSIONS:

1. What type(s) of impurity are completely removed by simple distillation?
2. Why would you not collect a beakerful of distillate and divide it into equal samples for testing rather than the procedure that you followed? When would it be acceptable to collect all the distillate in a beaker? What name is given to each sample collected? What name is given to this type of distillation.
3. Why must cold water flow into the lower end of the condenser?

EXTENSIONS:

1. Record the observations of what happens as the distillation apparatus condenser cools and explain why this happened. What measures can you take to prevent this?
2. In the experiment you were asked to arrive at a general conclusion after making only a single trial. For a conclusion to be valid it should be based on the results of a large number of trials. You could repeat the procedure and try reproducing your own results or substitute another impurity in your water sample. Another example of a volatile impurity is hydrochloric acid (hydrogen chloride gas in water solution).
3. Investigate the distillation of a compound which decomposes when the temperature is raised.

## Experiment 14

SEPARATION OF A MIXTURE INTO ITS COMPONENTS

PROBLEM: to separate a mixture into its components (using a method of your own design.)

INTRODUCTION:

A mixture is a sample of matter in which pure substance, elements and/or compounds, are simply intermingled in any proportion. The ingredients which make up a mixture are referred to as its components.

The mechanical or physical, method used for separating the components of a mixture or for purifying a substance depends mainly upon the physical states of the components. Two or more solids intermingled may be separated because of difference in physical properties such as susceptibility to magnetic attraction, solubility, and in some cases particle size. A solid mixed with a liquid but not dissolved in it may be separated from the liquid by decantation or by filtration. A solid dissolved in a liquid may be recovered by evaporating off the liquid and, if it be necessary to recover the liquid, by distillation.

PRE-LABORATORY PREPARATION:

Your instructor will assign you one of the following mixtures:

- Mixture #
- 1 - sodium chloride, sulfur and sand
  - 2 - sodium chloride, naphthalene and sand
  - 3 - manganese dioxide, sodium chloride and sulfur
  - 4 - potassium nitrate, sulfur and charcoal (as in black gunpowder)

by referring to your text or other source, investigate the solubility of each component in the mixture assigned to you. Is it soluble or insoluble? If it is soluble, which is the best solvent to use? If the solvent is water, would it be of any advantage to use hot water? Record this information in your laboratory notebook.

Devise a method to separate the dry components from the mixture. Illustrate the separation scheme by means of a flow sheet. Since some of the solvents may be dangerous, investigate the precautions to be taken and indicate these on the flow sheet.

LABORATORY PROCEDURES: (Testing your method)

Remove one component at a time using one of the solvents. The separation will involve filtration, (centrifuging if available) and evaporation. Remember that separation is not complete unless the components are dry. At the completion of the separation do not discard the components until the instructor has checked them.

CONCLUSIONS:

Evaluate your method of separation. Suggest ways of improving the method if you were to do the experiment again.

EXTENSIONS:

Study the physical properties of five "unknown" substances assigned to you by testing small samples with a magnet and different solvents. Record these properties for each substance. You will then be given a mixture containing three of the original substances. Separate the components. Record your procedure.

MAKING A SOLUBILITY CURVE - TEAM RESEARCH

PROBLEM: to determine the effect of temperature upon the solubility of various salts in water.

INTRODUCTION:

In this experiment you will find the solubility of a salt at several different temperatures. This is done by obtaining a weighed solution saturated at a given temperature and evaporating it to dryness. The solubility in grams per 100 g of water can be calculated after weighing the residue. From the results you will then construct a graph called a solubility curve.

This is a group experiment. Several students in the class may be finding the value at one given temperature. Others are doing the same experiment but at different temperatures. The summary of the experiment will be developed from the results of the entire class. Therefore this will be a further example of scientific teamwork in action.

PRE-LABORATORY PREPARATION:

1. Review the techniques required in this experiment:
2. Organize a flow sheet to represent the procedure that you will follow.
3. Prepare a data table for the recording of your own data and the calculation of the solubility of the salt at one given temperature. Also prepare a table for summarizing the group data.
4. Set up the axes for the graph.

LABORATORY PROCEDURES:

Your instructor will prepare about 500 ml of saturated solution of a salt such as potassium dichromate. This is done by bringing 500 ml of water to a boil and adding enough salt with constant stirring to leave excess crystals on the bottom of the container. He will then cool the saturated solution by immersing the container in the cold water until the temperature is about 80°C. The container will then be removed from the water and stirred vigorously to equalize the temperature in the solution. The temperature will be recorded.

A small group of students (1/6 of the class) will then have about 10 ml of the solution at the recorded temperature poured into their weighed evaporating dish. Be sure the evaporating dish is clean and dried before weighing so that it will not change weight when it is heated.

The solution is cooled by the instructor by 10°C steps. Students in small groups will use 10 ml portions of the solution at each temperature level.

Prepare a steam bath immediately, by filling your beaker three-quarters full with water and begin heating. Can you think of a method which will save time in this step?

Weigh the empty evaporating dish. Weigh the dish containing approximately 10 ml of solution. Then evaporate the solution to dryness over the steam bath. The final traces of water may be removed by placing the dish on a wire gauze over a small burner flame. Proceed with caution in this step to avoid overheating which may cause spattering or even decomposition of the salt. Cool and weigh the dish containing the residue.

Your data table should be organized in such a manner that from the data obtained experimentally, you can determine the weight of the dry salt and the weight of the water in the solution by two subtractions directly from the table. Once knowing the weight of water and the weight of the dry salt you can calculate the solubility (the number of grams of salt /100 g of water).

The class results will be posted on the board. Record in tabular form, all the solubilities at each given temperature. Plot the class results using the values for temperature on the horizontal axis and solubility in grams per grams of water on the vertical axis. The curve may be obtained by first averaging the group results at each given temperature. A second method would be to plot all the values and then approximating the average by drawing the curve between all these points. Is it permissible to discard or ignore any of these values by either method? Your instructor may assign either method.

### CONCLUSIONS:

1. By examination of the data and the graph, arrive at a conclusion. Can you assume that the same sort of a graph is typical of the solubility of all salts in water?

2. Discussion of possible error:

Indicate how each of the following affects your calculated solubility (+, -, 0). Your instructor will assign certain parts for discussion.

- a) The volume of the solution used was 13 ml instead of 10 ml.
- b) The saturated solution crystallized once it was poured into the dish.
- c) Some of the residue spattered during the completion of evaporation.
- d) A drop of the solution was spilled when being carried to the desk.
- e) The evaporating dish was not completely dry before the initial weighing.
- f) The residue contained was not completely dry before the final weighing.
- g) The instructor read the temperature of saturated solution but you were the last person in the group to receive a sample.

### EXTENSIONS:

1. Checking the accuracy of your solubility curve. By inspecting your graph predict the solubility of the salt for temperature that you did not use. Record this prediction. Obtain a saturated solution of the salt at

that temperature and find the solubility. Record the solubility and compare it to the value obtained from your graph. Comment on the accuracy. (Compare your results to your prediction).

2. If the salt you used in the experiment should be hydrate, what difference in the method, if any, would you use? Would you expect any difference in the results?

3. Did your salt absorb or give off energy when it dissolved? How can you tell. Can the type of energy change be used to predict the shape of the solubility curve? Investigate.

### Experiment 16

#### MAKING A SOLUBILITY CURVE - AN INDIVIDUAL INVESTIGATION

PROBLEM: to investigate the solubility of an unknown salt.

#### INTRODUCTION:

The solubility of a solid in a certain solvent can be specified by giving the weight of the solid which, when dissolved in 100 g of solvent, makes a saturated solution. The solubility, of course, depends on the temperature; the solid studied in this experiment becomes more soluble in water as the temperature increases. Instead of measuring the solubility at a given temperature, you will measure the temperatures at which solutions of known compositions become saturated. You can then plot a curve of solubility as a function of temperature, and read on it the solubility of any temperature in which you are interested.

#### PRE-LABORATORY PREPARATION:

1. Organize a data table and the axes for your graph after reviewing R-7.
2. Calculate the solubility for the first trial, assuming that you will use exactly 7.0 g of solid.

#### PROCEDURE:

1. On tared paper, weigh accurately about 7 g of unknown solid. Transfer the solid to a large test tube. Clamp the tube vertically to a lab stand.
2. Pipette 5 ml of water into the tube. Warm to dissolve. If the salt doesn't dissolve add more water, 1 ml at a time, keeping careful record of how much was added. The warming may be done by applying heat to the tube which is adjusted at an angle, or by the use of a water bath when the tube is held vertically.
3. Once the solid has dissolved, clamp the test tube vertically. Allow the solution to cool, using the thermometer as a stirring rod. Note carefully the temperature at which the first sign of crystallization was observed.



4. Repeat the above procedure with four more 5 ml dilutions.

#### PROCESSING THE DATA:

1. The weight of the water (assume the density of water is 1.00 g/ml for every temperature involved) and temperature of initial crystallization should be recorded for each trial. Calculate the solubility (g/100 g water) for each temperature.

2. Convert the data to a useable form by plotting the curve of solubility as a function of temperature.

#### CONCLUSIONS:

1. By examination of the graph, arrive at a conclusion. Can you assume that the same sort of a graph is typical of the solubility of all salts in water?

2. What is the solubility of your unknown solid at 50°C and at 30°C as indicated by your solubility curve?

3. Discussion of Errors:

If your thermometer reading is too high (positive error), your error in calculating the solubility of the unknown would have been (+, -, or 0).

If you had failed to notice the initial appearance of crystals and had waited until a large fraction of the solute had precipitated, your error in the solubility would have been (+, -, or 0).

#### EXTENSIONS:

Prepare a solubility curve for the same salt by two different methods. Evaluate the methods and decide which is more accurate.

AN INVESTIGATION INTO THE PROPERTIES OF CRYSTALS

PROBLEM: 1. to devise and test a method for detecting whether a crystalline compound has water chemically or physically bound to it.

INTRODUCTION:

Water is joined to crystalline substances in two general ways. Water may be chemically bonded to a compound. Compounds of this type are called hydrates, and they are said to contain water of hydration. Gently heating a compound containing water of hydration is enough to free the compound of its water. Water may also be physically or mechanically held by a crystalline compound. When heating this type of crystalline substance, it is said to decrepitate. Compounds that contain no water are said to be anhydrous.

PRE-LABORATORY PREPARATION:

Before the laboratory period, ponder over the problem and in your lab notebook suggest a method of detecting whether a compound has water chemically or physically bound to it. Describe the method to be used and observations you expect to make. Also include a theoretical discussion of why you would expect those observations. Your instructor will initial your hypothesis at the beginning of the laboratory period. Also prepare a table for the recording of your observations on 8 salts during the experiment.

LABORATORY PROCEDURES:A. Testing your hypothesis

You will first test a few small crystals of cupric sulfate, a compound known to hold its water chemically, and heat them gently in a clean, dry test tube, held in a horizontal position. Make careful observations of the compound and the test tube during this procedure. Repeat this procedure with sodium chloride, a compound known to have its water physically bound.

Several other compounds will be available for testing to see if  $H_2O$  is chemically or physically bound, by the class as a group. Use a clean dry test tube for each salt. Record a summary of the group observations in your table.

CONCLUSIONS:

On the basis of the results obtained, evaluate your hypothesis. In your discussion include summary of what you found to be accurate. Also include the information which <sup>was</sup> learned from the experiment and which should be included in your hypothesis to make it complete.

AN INVESTIGATION OF WATER OF HYDRATION IN CRYSTALS

- PROBLEM: to investigate 1. the behavior of salts upon exposure to the atmosphere.  
2. The concept of water of hydration in crystal formation.

INTRODUCTION:

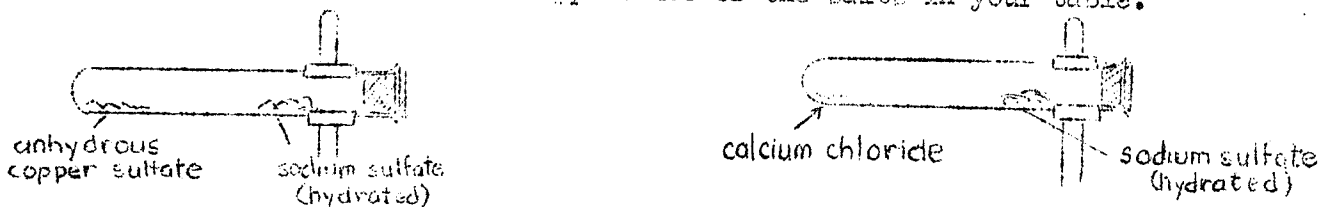
Compounds which absorb moisture from the air are said to be hygroscopic. If the compound absorbs enough water to become visibly wet (and sometimes dissolve itself) or to become a hydrate, it is said to be deliquescent. Compounds which lose their water of hydration upon exposure to the atmosphere are called efflorescent. These compounds release their water of hydration without the addition of heat.

PRE-LABORATORY PREPARATION:

1. Set-up a table for the recording of observations during procedure A.
2. Organize a flow sheet for Procedure B.

LABORATORY PROCEDURES:

A. Demonstration Your instructor will set up two demonstrations as diagramed below. Each "experimental" tube was prepared a few days before the laboratory period. It will be carefully compared to the "control" tube which was prepared immediately preceding the laboratory period. Record the differences observed in the appearance of the salts in your table.



B. Using a paper trough, place "hypo" (sodium thiosulfate) crystals in a clean dry test tube to a depth of about 5 cm. Add 5 drops of water to the tube, noting any temperature change with a thermometer. Warm the tube gently over the flame until only a clear solution remains. Cool the tube without agitation in a stream of cold water from the tap until it is approximately at room temperature. Rinse off a thermometer with water and touch it to a crystal of "hypo". (This is known as seeding a solution; the crystal of "hypo" is a seed crystal.) Leave the thermometer in the test tube. Record all observations on the temperature change and appearance in the appropriate place in the flow sheet.

CONCLUSIONS:

- A. Classify the salts investigated as efflorescent, deliquescent, or neither. Defend your classification.

- B. 1) Identify the type of solution remaining in the test tube after  
a) the solution was cooled, b) the crystallization took place.
- 2) Account for the solubility of the "hypo" crystals in such a small volume of water.
- 3) Account for the temperature change during solution and crystallization.

EXTENSIONS:

Make a quantitative investigation of the behavior of calcium chloride upon exposure to the atmosphere. Place 3 grams of anhydrous calcium chloride on a watch glass and weigh. Expose this calcium chloride to the atmosphere and check and record the weight change periodically. A large number of values are desirable. Using time and weight values for plot the points and draw the graph. Draw a conclusion from your graph and discuss whether this information is of any practical value.

## Experiment 19

THE PERCENTAGE OF WATER OF HYDRATION

PROBLEM: to determine the percentage of water of hydration in crystalline salts.

INTRODUCTION:

The percentage of water of hydration (crystallization) may be determined in the laboratory by heating hydrated crystals of a salt and driving off the water of hydration. An accurate accounting of the weights before heating and after heating will give the necessary data to make this calculation. Complete dehydration plus accuracy in weighing are factors upon which the success of this experiment depends.

A major feature of any quantitative work undertaken by a chemist is the need for two or more sets of results in which the quantitative data are in close agreement. If time permits, two trials should be made. The results of each trial should be within 1% of each other. This close agreement among separate sets of results are a basic requirement whenever definite conclusions are to be drawn.

PRE-LABORATORY PREPARATION

Weigh a clean, dry evaporating dish and record its weight. Add about 5 g of "unknown" hydrated crystals. (if the crystals are not fine, the mortar and pestle should be used.) to the evaporating dish. Weigh the dish and its contents. Record the weight.

Place the evaporating dish on a wire gauze which is supported by a ring clamp and lab stand. Warm the contents slowly with a low flame. The temperature should come to slightly over 100°C. Stir the contents carefully. Avoid spilling any of the contents or causing a loss in weight in any other way. The hydrates may stick to the stirring rod until it becomes warm. Avoid heating it to such a high temperature that decomposition takes place.

After no more change seems to take place, cool the evaporating dish and reweigh it. Record the weight. Heat the dish once more for about 5 minutes as before. Cool and weigh the dish a second time. If there is no additional loss in weight, the weight is said to be constant. You can assume that all the water of crystallization has been driven off. If there is an additional loss in weight, repeat the process until two successive weighings are the same. Record the final weight.

Data and Calculations: Your data table should contain three columns for the quantitative data: trial #1, trial #2 and Average. Indicate the operations used in making the calculations of % water of hydration by showing an orderly sample calculation. Do not clutter the calculations section with arithmetic details.

CONCLUSIONS:

- A. What is your experimental percentage of water of hydration for the given salt?
- B. Obtain the identity of your hydrates from your instructor. Calculate the theoretical % of water of hydration and then determine your percentage of error.
- C. Discussion of errors:  
Indicate how each of the following errors or conditions affect your calculated answer, ( +, -, or 0 ).
- a) The hydrated salt was efflorescent.
  - b) Some of the contents of the evaporating dish spilled during heating.
  - c) The evaporating dish was not clean at the start.
  - d) The hot evaporating dish was allowed to cool on the painted desk surface.
  - e) The salt was not heated to constant weight (to dryness)
  - f) The salt was heated to such a high temperature that it decomposed.
  - g) The salt was deliquescent.

EXTENSIONS:

Devise a method for determining the formula of a hydrate ( the number of water molecules that are chemically bonded to the compound) from a similar experimental procedure. The formula of anhydrous compound would also need to be known. Test and evaluate your method.

## Experiment 21

REACTIONS BETWEEN OXIDES AND WATER - TEAM RESEARCH

- PROBLEM: 1) to discover which oxides react with water.  
2) where a reaction does occur, to determine what type of compound is formed.

INTRODUCTION:

Although many methods are used by scientists, the following may be considered to be the four basic activities of science:

1. to accumulate information through careful observation.
2. to organize this information and to seek patterns or regularities in it.
3. to wonder why these regularities exist and devise theories to explain them.
4. to communicate the findings to others.

This experiment will provide you with experience in the first two of these basic activities.

The oxides will be mixed with water one at a time. The water will be tested with red and blue litmus to see if an acid or base is formed. Observations will be made on any other reactions and on the extent of the solubility of the oxide. Regularities in your observations will be sought by referring to the periodic table, the activity series of metals, etc. The regularities in your conclusion will be developed from the results of the entire class. Hence, this experiment will serve as an example of scientific teamwork in action.

PRE-LABORATORY PREPARATION:

Prepare a table for the recording of observations during the experiment. This table should include columns for: the formula of the oxide, the physical state, the solubility, the effect on litmus, and acid or base-forming.

LABORATORY PROCEDURES:

1. Select an oxide. With a spatula take a sample of it the size of a grain of rice and place it in the centre of a watch glass. Drop water from a dropper onto the oxide until you have made a small puddle around the sample. Stir the mixture with a glass rod.
2. Test the liquid around the oxide with both types of litmus paper. Rinse the litmus paper to be sure that any color change is not due to undissolved solid remaining on the paper. Notice any change that takes place in the oxide and record it also.
3. Repeat the test with other oxides. Be certain that the watch glass is thoroughly clean before reusing.

4. Obtain samples of the elements, each about half the size of a grain of rice. Place the element into a clean deflagrating spoon. Ignite the element by heating. Hold the burning sample in a jar containing a half-inch layer of water. Close the top with a cover glass. When the burning stops, shake the gas or smoke in the jar with the water. Remove the spoon. Using a glass rod, put a sample of the solution on both types of litmus paper. Repeat this method for the remaining elements.

5. The group data may be collected and recorded on the board by the instructor or "group captain". Summarize the group data by preparing a table which classifies the oxides under three headings: a) Acid-forming b) Base-forming c) No Reaction with Water.

### CONCLUSIONS:

1. On the Periodic Table locate the elements whose oxides formed a base with water. Also find the position of the elements whose oxides reacted to produce an acid. The long form of the Periodic Table shows groups of metals, non-metals, inert gases, transition heavy metals, rare earths, etc. Are the acid-forming elements classed in any one group? Are the base-forming elements classed in any one group? Are those elements which produce no reaction classed in any one group? If so, identify the group.

2. In the Activity Series of the Elements, find the position of those elements whose oxides you tested. See if you can establish a pattern between your results and the position of the elements in this list.

3. Reorganize your data by writing in two columns: (a) the formulas for the oxides which did give a reaction, and (b) those which did not. After each formula write down the corresponding heat of formation. Can you discover a pattern between these values and your results?

### EXTENSION:

Verifying your conclusions. A conclusion is satisfactory when it enables you to predict with reasonable success the behavior of matter which was not previously tested. On the basis of the conclusions that you drew, predict how an oxide, which you did not previously study, should react with water. Once you have recorded your prediction in your laboratory notebook, obtain a sample of that oxide and test your prediction. Report your result. Does this result strengthen or weaken your conclusions?



### Experiment 21

#### THE IDENTIFICATION OF FIVE UNKNOWN SOLUTIONS

**PROBLEM:** to identify five unknown solutions by investigating their chemical properties.

**INTRODUCTION:**

In this experiment you will be given samples of each of five unknown solutions in numbered test tubes (or in dropper bottles). The five tubes will contain solutions of five compounds whose characteristic reactions have been previously studied. Although you will be given specific names of these substances, the names will not be associated with the numbers on the test tubes. The problem is one of qualitative analysis, where you are to identify each solution by name. This can be done by mixing a portion of two of the solutions and comparing the resulting observations to the characteristic reactions of the solutions of the five compounds.

The reagents must be limited to the contents of the five tubes. No other chemicals, no other reagents, no heat and no additional amount of any of the solutions are to be used. You must work (as does the practicing chemist at times) with a limited set of materials and confine your thinking to the results obtained with them. This involves making a careful record of all that is known about the materials.

**PRE-LABORATORY PREPARATION:**

1. Before reporting to the laboratory, complete the following table in your laboratory notebook, using any source of information at your disposal. Indicate with an X where a precipitate will not form and write the color of any precipitate that will form. You should be able to indicate a precipitate in eight of the squares.

	$Pb(NO_3)_2$	$Na_2SO_4$	$K_2CrO_4$	$FeCl_3$	$AgNO_3$
$AgNO_3$					
$FeCl_3$					
$K_2CrO_4$					
$Na_2SO_4$					
$Pb(NO_3)_2$					

2. Also prepare a second table for the recording of your laboratory observations. This table should be similar in organization, except that the numbers 1 to 5 should be substituted for the formulas of the compound. With 5 different solutions, how many different tests will you need to make in order to study all the possible combinations of solutions, using two at a time. Perhaps, you can improve on the first table and avoid unnecessary duplication.

LABORATORY PROCEDURES:

A. Using test tubes. Take five numbered test tubes to the instructor and obtain 10 ml each of the five unknown solutions. Mix small volumes (1 ml), two at a time. Carefully note any reactions and record your observations in the table.

B. Using medicine droppers and glass plates. Place a drop of one solution on a clean, dry glass plate and add a drop of the second solution to it. The glass plate may be marked off in small squares so that several combinations may be tried on the same plate. Continue until you have tried all possible pairs of solutions from the five to be added. Record all your observations.

CONCLUSIONS:

Compare the observations of the reactions in the laboratory with the characteristic reaction of the solutions which are recorded in your first table. This provides you with enough information to identify by name each of the five solutions.

After having established the identify of the solutions, present, in writing, an account of how you accomplished this identification.

EXTENSIONS:

Devise a similar problem of your own by using different solutions, then test your predicted reactions in the laboratory. The characteristic reactions of the solutions need not only involve precipitation. Report your investigation in a manner similar to the one in this experiment.

## Experiment 22

MOLECULAR WEIGHT DETERMINATION (DUMAS METHOD)

PROBLEM: to determine the molecular weight of a condensable vapor.

INTRODUCTION:

You know that 22.4 liters of a gas at STP contains one gram-molecular weight of any gas. In this experiment you shall determine the volume of a given weight of a gas; then you will calculate the weight of 22.4 liters of the gas at STP. The resulting value is the gram-molecular weight.

This procedure can also be followed for a liquid that can be converted to a gas. A small volume of liquid is placed in a flask. The flask is then heated until all the air is expelled and the flask is filled with vapor of the substance being used. The flask is then cooled, causing the vapor to condense, forming a liquid. The weight of the liquid is the same as the weight of the vapor that completely filled the flask, at the elevated temperature and barometric pressure in the laboratory. You can then calculate from the gas laws the volume of this same weight of vapor at S.T.P.

PRE-LABORATORY PREPARATION:

Design a table which will include all the essential data. Organize the data to obtain the weight of the weight of the vapor (which is equal to the weight of the liquid condensed).

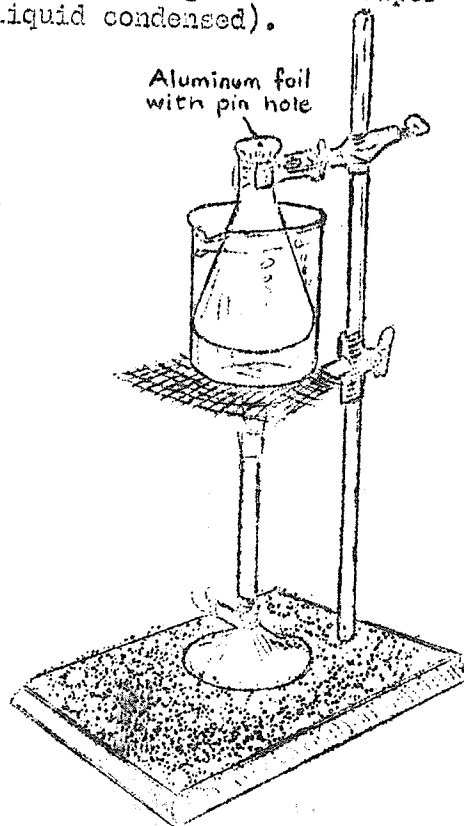
LABORATORY PROCEDURES:

1. Obtain a square of aluminum foil (approximately  $1\frac{1}{2}$  inches square), and shape a cap for your flask by laying the foil on the mouth and folding down the sides. Using a pin, poke a tiny hole in the center of the cap.

2. Determine the weight of the flask and cap to the nearest .01 g. What else are you weighing?

3. Add about 3 ml of the unknown liquid to the flask and replace the cap securely.

Clamp the flask in a water bath. Fill a 600 ml beaker with water so that the maximum flask surface is covered with water. Heat to boiling. Record the temperature of the boiling water and the barometric pressure.



Observe the apparatus carefully to decide when evaporation is completed. Do not confuse bubbles in the boiling water with those of the unknown liquid in the flask.

4. After the liquid in the flask has completely evaporated, remove the flask by means of the clamp and set it aside to cool. After the flask has cooled to approximately room temperature, wipe the flask dry. Examine the cap to make sure there are no water drops on it.

5. Weigh the flask, cap and condensed unknown liquid. What else are you weighing?

6. Determine the internal volume of the flask. First, fill the flask completely full of water and then measure its volume by pouring the water out into a large graduated cylinder. Does the marking "125 ml" on the flask indicate the exact volume, thus making it unnecessary to measure the volume of the flask?

7. Calculate the molecular weight of the unknown vapor.

#### CONCLUSIONS:

1. State your experimental value for the molecular weight of the unknown. How is the molecular weight of the unknown liquid related to that of its vapor?

2. Discussion of possible errors. Indicate how each of the following factors would influence (+, -, or 0) your determined molecular weight.

- (a) Instead of a foil cap, you use a stopper which absorbs some of the vapor.
- (b) The condensed liquid contains substances extracted from the stopper.
- (c) The temperature of the flask does not reach that of the bath.
- (d) During the cooling process, some of the vapor diffuses out of the flask.
- (e) The vapor does not displace all of the air in the flask at the elevated temperature.
- (f) In the process of weighing, the total weight obtained would include that of the flask, the cap and also the weight of the air.

#### EXTENSION:

Determine the molecular weight of a gas by displacing the air in a flask of known weight and volume with a gas at a known temperature and pressure, then recording the final weight.

DETERMINATION OF THE VOLUME OF A GAS PRODUCED BY A REACTION

PROBLEM: to make a quantitative investigation of the reaction of a metal with hydrochloric acid and to determine the volume of gas produced in the reaction.

INTRODUCTION:

First you will calculate the volume of  $H_2$  that should be evolved by a known weight of Mg reacting with an acid under laboratory conditions. Then you will determine experimentally the volume of  $H_2$  actually evolved by this weight of Mg. Finally you will compare the theoretical and experimental volumes.

RE-LABORATORY PREPARATION:

1. Carefully review the collection and measurement of gases, T-17.
2. After studying the procedure, use a series of labelled diagrams to illustrate the major steps that you will follow in conducting the experiment. Indicate any special precautions required.
3. Organize an appropriate data table.

LABORATORY PROCEDURES:

1. Obtain a piece of Mg ribbon approximately 8 cm long. Measure the length of your ribbon carefully and record this to the nearest 0.05 cm. Your teacher will give you the weight of a meter of the ribbon, and since it is very uniform in thickness you can calculate the weight of magnesium used.
2. Fold the ribbon loosely so that it can be encased in a small spiral cage made of copper wire. Leave about 5 cm of the wire to serve as a handle.
3. Set up a lab stand and utility clamp in position to hold a 100 ml gas measuring tube which has been fitted with a one or two hole stopper.
4. Incline the gas measuring tube slightly from an upright position and pour in about 10 ml of moderately concentrated HCl acid.
5. With the tube in the same position, slowly fill it with tap water poured from a beaker. While pouring, rinse any acid that may be on the sides of the tube so that the top of the tube will contain very little acid. Try to avoid stirring up the acid layer in the bottom of the tube. Bubbles clinging to the sides of the tube can be dislodged by tapping the tube gently.
6. Holding the copper handle, immediately insert the Mg metal about 3 cm down into the tube. Hook the copper wire over the edge of the tube and clamp it there by inserting the rubber stopper. The tube should be full with water up to the end of the stopper.

7. Cover the hole(s) in the stopper with your finger and invert the tube into a tall cylinder containing water at room temperature. Clamp the gas measuring tube in place. The acid, being more dense than water, will diffuse down through it and eventually react with the Mg.

8. After the reaction stops, wait for about 5 minutes to allow the tube to come to room temperature. Dislodge any bubbles clinging to the sides of the tube. Raise or lower the tube until the level of the liquid inside the tube is the same as the level outside in the tall cylinder. This permits you to measure the volume of the gases in the tube at room pressure. Record the volume of the gas to the nearest 0.05 mL.

9. Record the temperature of the water in the tall cylinder. After consulting the barometer, record the pressure in the laboratory.

10. Remove the gas measuring tube from the cylinder and pour the acid solution it contains down the sink. Rinse it with tap water.

#### DATA AND CALCULATIONS:

The data table should include the following:

- the weight of the Mg ribbon per meter; the length of the Mg ribbon.
- the experimental volume of H<sub>2</sub>; the temperature of the water; the barometric pressure.

1. Find the weight of Mg that you used from the grams per meter relationship and the length of the ribbon.
2. Calculate the theoretical volume of hydrogen at room conditions.
3. Compare the theoretical and experimental volumes and calculate the percent error.

#### DISCUSSION OF ERRORS:

1. Discuss the effect (+, -, 0) on the experimental volume of hydrogen of each of the following errors:
  - a) Some air is allowed to enter the gas measuring tube upon inversion.
  - b) Some hydrogen gas escapes.
  - c) Some Mg ribbon sticks to the side of the tube and doesn't react.
  - d) The volume of H<sub>2</sub> is read when the water level in the tube is above that of the tall cylinder.
  - e) The measured volume of gas was assumed to be hydrogen only.
2. Expanding on part (e) above, account for your large percent error in this experiment. Would a correction factor applied to your experimental volume reduce this percent error? Calculate the experimental volume of dry hydrogen, measured under laboratory conditions.

#### Pressure of Water Vapor (mm of Hg)

<u>°C</u>	<u>mm</u>	<u>°C</u>	<u>mm</u>
20	17.5	24	22.4
21	18.7	25	23.8
22	19.8	26	25.2
23	21.1	27	26.7

## Experiment 24

ANALYSIS OF AN UNKNOWN MIXTURE

PROBLEM: to calculate the per cent by weight of potassium chlorate in a mixture of potassium chlorate and potassium chloride.

INTRODUCTION:

This experiment provides an example of quantitative analysis. By investigating the weight relations obtained from an equation for the decomposition of potassium chlorate you can apply the results to analyze a potassium chlorate-potassium chloride mixture.

As you have previously noted, potassium chlorate decomposes when heated strongly. All the oxygen can be driven off leaving potassium chloride. Therefore, when a mixture of potassium chlorate and potassium chloride is heated, the weight loss is due to the oxygen released by the potassium chlorate. This information permits you to calculate how much potassium chlorate was present in the original mixture.

PRE-LABORATORY PREPARATION:

1. Prepare a flow sheet which represents the procedures you will follow.
2. Organize data table to obtain; (a) the weight of the unknown mixture and (b) the weight of the oxygen evolved, directly from the table.
3. Illustrate the weight relations of potassium chlorate by setting up a weight-weight problem.

LABORATORY PROCEDURES:

1. To a clean, dry pyrex tube add a pinch of manganese dioxide. Weigh the test tube and its contents to the nearest 0.01g. Obtain an unknown mixture from your instructor; record its number in your laboratory notebook. Add about 3 g of the mixture to the weighed tube containing Manganese dioxide, and weigh the total. Mix the contents of the tube by tapping gently.
2. Clamp the tube to a lab stand at an angle of 45°. Heat gently with a flame to avoid the appearance of fumes emerging from the tube. When the solid melts increase the heating and heat as strongly as possible for a few minutes. Allow the tube to cool and weigh the tube plus the product(s). Repeat the process of heating, cooling and weighing, until a constant weight is attained.
3. Show the calculations of (a) the weight of potassium chlorate present in the mixture, and (b) the per cent by weight of potassium chlorate in the unknown mixture.
4. Obtain a second determination by repeating the whole procedure.

CONCLUSIONS:

State the per cent by weight of potassium chlorate in the unknown mixture.

Discussion of errors: Indicate how each of the following conditions would have influenced your experimental per cent. (+, -, or 0).

- (a) The potassium chloride vaporized at a high temperature.
- (b) The mixture was not heated to constant weight.
- (c) The crystalline components of the mixture held water mechanically.
- (d) Manganese dioxide was added to the mixture.
- (e) The test tube was not dry at the start of the experiment.

EXTENSIONS:

Devise a method by which you could determine the per cent composition by weight of a mixture composed of two hydrates. This quantitative analysis permits you to apply your knowledge of simultaneous linear equations. Test your method in the laboratory.



## Experiment 25

PREPARATION OF A SOLUBLE SALT

PROBLEM: to prepare a sample of sodium chloride from either a bicarbonate or a carbonate.

INTRODUCTION:

In many of the previous experiments, you were directed toward the solution of the problem by clear-cut methods. By now you have learned enough about chemistry and laboratory techniques to prepare a specified amount of a given compound with the available equipment. For reasons of safety, however, your procedure must first be approved by your instructor. You will prepare a written plan for a series of laboratory procedures to be used to prepare a sample of sodium chloride.

Chlorides are the salts of hydrochloric acid. In this experiment you will treat sodium carbonate (or bicarbonate) with hydrochloric acid in order to produce sodium chloride. You will measure the quantity of sodium chloride obtained from the known weight of sodium carbonate (or bicarbonate) and compare this experimental value with the theoretical value.

PRE-LABORATORY PREPARATION:

1. Make a list of the apparatus and materials required for this experiment.
2. Prepare a written plan for a series of laboratory procedures to be used to prepare the sample of sodium chloride, clarifying the questions raised in the outline and indicating any special precautions to be taken.
3. Show well-organized calculation of a) the volume of the acid required; b) the theoretical yield of sodium chloride.
4. A data table for recording your weighings.

LABORATORY PROCEDURES - Outline.

1. Weigh an evaporating dish accurately on the balance. Add 1 gram of sodium carbonate (or bicarbonate) to the dish and reweigh.
2. Using your knowledge of concentrated hydrochloric acid, calculate the volume of the acid which is needed to react with your sample. Would you advise adding an excess of the acid? Will this cause an error? How about too little hydrochloric acid?
3. Add the acid to the sample. Will there be a visible reaction? What causes it? Is there danger of loss of sample at this stage? How can you minimize this danger? What means should be used to add the acid? What rate should be used for the addition of acid? How can you be sure that all the sample is exposed to the hydrochloric acid? If you decide to stir, a stirring rod is not advisable? Why not? How can you be sure when the reaction is complete?
4. How can the sodium chloride be separated from the excess liquid without any loss? What is the liquid?
5. Weigh the evaporating dish and product. How can you be sure that all the liquid has been removed?

CONCLUSIONS:

Indicate the experimental and theoretical values for the weight of sodium chloride. Calculate the per cent yield. If you were to repeat the experiment, state the points at which your technique could be improved to increase the yield of sodium chloride.

## Experiment 26

A DETERMINATION OF THE DENSITY OF OXYGEN

- PROBLEM:
1. to determine the density of oxygen experimentally under room conditions.
  2. to investigate the significance of different aspects of the experimental procedures.

INTRODUCTION:

The weight of the oxygen is determined from the loss in weight of a solid which, upon heating, yields oxygen as the only gas. The oxygen so produced is used to displace water from a bottle into a beaker. The volume of the oxygen is determined by measuring the volume of the displaced water.

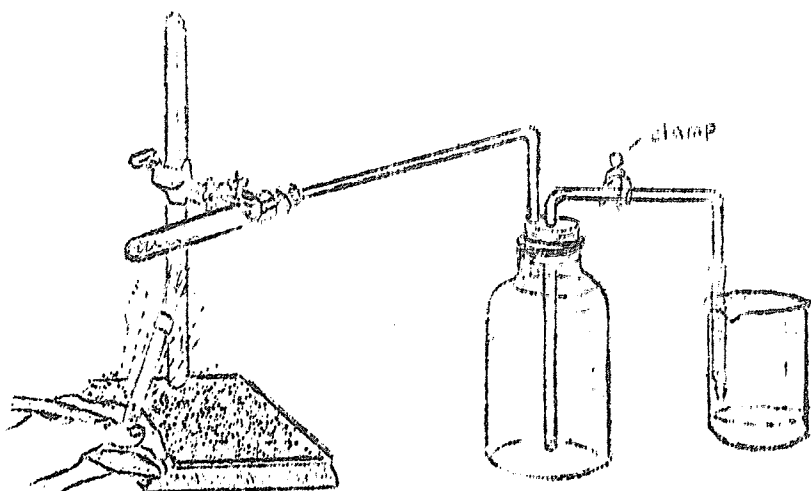
LABORATORY PROCEDURES:

1. Weigh a small test tube containing 15-20 grams of lead dioxide. Be certain that the tube is clean and dry at the start of the experiment.
2. Use a  $\frac{1}{2}$  liter bottle (a liter bottle, if possible), a 400 ml beaker, a pinch clamp, necessary glass tubing and stoppers to set up the apparatus so that during the experiment the oxygen evolved on heating the lead dioxide with force water from the bottle into the beaker. To start the experiment, should the bottle be nearly empty, half-full or nearly full of water?
3. When heating the lead dioxide move the Bunsen flame about. Do not concentrate the heat in one spot.
4. During the course of the experiment you will
  - (a) displace at least 300 ml of water.
  - (b) record the temperature of the water and the barometric pressure.
  - (c) reweigh the test tube and residue.
  - (d) measure the volume of the displaced water by weighing it, or by using a graduated cylinder.
  - (e) calculate the density of oxygen (g/ml) at i) room conditions, and ii) at S T P.

PRE-LABORATORY PREPARATION:

Design a table to include the following data: the weight of the test tube and lead dioxide, the weight of test tube and residue, the volume of the oxygen evolved, the temperature of the oxygen, barometric pressure, pressure of the oxygen. Organize the data table so that the calculation of the weight of the oxygen evolved may be facilitated.

To be a good experimenter it is important to study the value of the different procedures and to anticipate any problems before they occur. Before performing the experiment, consider and answer the following question in your laboratory notebook.



1. What is your estimate of error (+, -, or 0) in milliliters in the volume of the displaced water if:
  - a) the delivery tube is not filled with water before heating?
  - b) the pressure of the gases in the bottle is not the same at the beginning and at the end of the experiment?
  - c) the beaker containing the collected water is removed at the end of the heating period, but before the test tube has cooled to room temperature? (Lead dioxide decomposes at  $290^{\circ}\text{C}$ .)
  
2. What will happen if:
  - a) the pinch clamp is not removed before starting to heat the test tube containing the lead dioxide?
  - b) the opening of the delivery tube is very large?
  - c) the opening of the delivery tube is very small?
  - d) the delivery tube is full of water and the test tube is imperfectly sealed into the stopper?
  
3. Is there any advantage in collecting 300 ml instead of 100 ml of water?
  
4. With reference to the diagram of the apparatus, with the delivery tube also filled with water, the water does not siphon into the beaker; if it does what corrective steps, if any, should you take?

#### CONCLUSIONS:

1. State your experimental value for the density of oxygen at standard conditions. From the results of this experiment, what volume would 32 grams of oxygen occupy under standard conditions?

2. Discussion of errors: What effect would each of the factors (+, 0, or 0) have on the density of oxygen under S.T.P.?
- a) You neglected to take into account the vapor pressure of the water.
  - b) The lead dioxide did not decompose completely.
  - c) Some oxygen leaked out of the test tube.
  - d) The lead dioxide contained an inert impurity such as sand.
  - e) The lead dioxide contained an impurity such as potassium chlorate, which also gave off oxygen when heated.
  - f) The test tube contained some droplets of water before the experiment began.
  - g) The receiver beaker contained some water before the experiment started.

## AN EXTENDED INVESTIGATION -- A PROJECT

A project provides an opportunity for you to choose an area in chemistry which is of special interest to you and then to perform an investigation of your own design in this area. This investigation should be beyond the scope of the experiments which you have previously done from this manual. Nor would the performance of an extension be regarded as a project unless it raised many unanswered questions which are investigated in turn. This work should allow you to exhibit some originality in applying the principles of chemistry which you have been studying.

The choice of projects are limited only by the available facilities and good judgement, and the restrictions are few.

Although no set procedure may be outlined, a general method of approach for the setting up of an investigation is suggested.

- A. You may be able to conserve time if you first choose some narrow field of chemistry which appeals to you. Once the choice is made, go to the library and skim through all the information that you can find in this field. If this reading doesn't suggest a good investigation, you may be well advised to choose another field and start again. The ideal topic is one in which the student can gain both in knowledge and appreciation of chemistry. Be careful not to attempt investigations involving theories which are beyond your capacity to understand.
- B. After your choice is made, the project should be approved by your instructor.
- C. In the interest of safety, there are certain precautions that must be followed.
  1. Under no circumstances should the preparation or handling of explosive materials be attempted.
  2. Avoid projects requiring large amounts of poisonous substances such as chlorine.
  3. Under no circumstances should any chemical work be done in anyone's home.
- D. An important part of the experiment is the write-up. The results should be summarized in the form of a brief but well-written report. Your report should include both the summary of the literature survey preceding the experiment and the literature references. A detailed description of the work done should follow. The results obtained should be carefully recorded and the calculations included. These results should be carefully evaluated.

It is hoped that you find much inspiration and gain further interest in science as you do a project.

### AN EXTENDED INVESTIGATION OF A CANDLE

In your first experiment you learned that careful observation of a familiar object usually reveals characteristics not recognized before. Perhaps while observing the candle, you may have wondered why certain things were happening. In attempting to answer such questions, many other questions usually arise. So therefore, an extended investigation could be made of something as simple (on the surface) as a candle. (This is probably the only type of chemistry project that is safe enough to investigate at home -- with the permission of your parents)

The following are some questions which may guide your investigations:

1. What is the colorless liquid in the bowl at the top of the burning candle? Perhaps this liquid is just melted wax, or it may be another liquid? What type of experiment would help you to decide which is correct?
2. What are the products produced on burning? What tests can you use to identify them? Can you measure how much of each product is formed on burning of the candle.
3. What is the purpose of the wick? Of what is it composed? Can other materials be substituted to serve as the wick? If so what do these materials have in common?
4. Investigate the flame by gathering evidence to answer the following questions:
  - a) Why does the flame have a particular shape? Does it vary with air currents?
  - b) Why is the flame blue at the base, dark in the center, and yellow elsewhere?
  - c) Why is the flame extinguished when you blow on it?
  - d) What does the size of the flame depend on? Will a candle with a larger diameter produce a larger flame?
  - e) Can the candle burn with non-luminous flame?
5. The burning of a candle involves both chemical and physical changes, which are accompanied by energy (heat) transfer. Can you measure how much energy was transferred during these changes?
6. Are all candles made only of wax? Can you make a candle with another substance? Are the products of burning the same?

## APPENDIX I

Table I

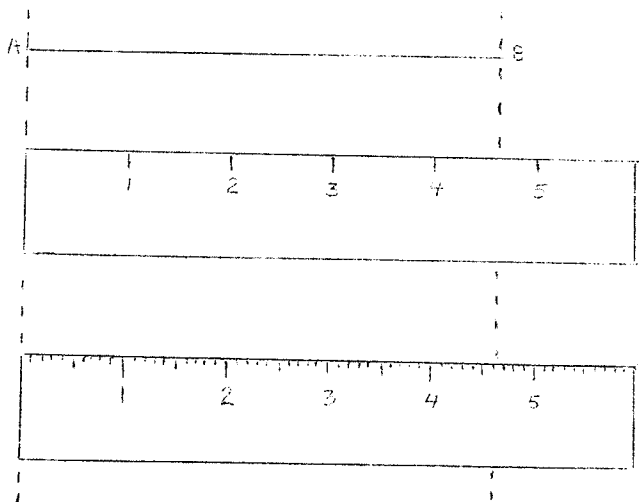
## VAPOR PRESSURE OF WATER

Temp (°C)	Pressure (mm Hg)	Temp (°C)	Pressure (mm Hg)	Temp (°C)	Pressure (mmHg)
0	4.58	18	15.5	27	26.7
10	9.21	19	16.5	28	28.3
11	9.84	20	17.5	29	30.0
12	10.5	21	18.7	30	31.8
13	11.2	22	19.8	40	55.3
14	12.0	23	21.1	60	149
15	12.8	24	22.4	80	355
16	13.6	25	23.8	100	760
17	14.5	26	25.2		

SIGNIFICANT FIGURES

Laboratory measurements usually require that you estimate the fraction beyond the smallest division on the instrument. The accuracy of this estimated figure is doubtful. It is customary to retain one doubtful figure in the result of any measurement. Numbers which express the result of a measurement such that only the last digit is in doubt are called significant figures.

The number of significant figures used to express a measured result depends on how precisely the measuring instrument is calibrated.



What can be determined by measurement, about the length of line AB? The upper ruler gives 4.6 cm; the line is certainly between 4 and 5 cm and appears to be about six-tenths of the way from 4 to 5. The lower ruler gives 4.63 cm; the line is certainly between 4.6 and 4.7. The first measurement (4.6 cm) has two significant figures; the second measurement (4.63 cm) has three significant figures. Because of the

difference in the two measuring instruments, there are two ways of expressing the length of the same line. These represent different degrees of precision. The first measurement 4.6 cm, is precise to 1 part in 46 (approx. 2%). The second 4.63 cm, is precise to 1 part in 463 (approx. 0.2%)

It is obvious that a measurement can be no better than the measuring instruments used to make it. It should be remembered also that the last significant figure is always uncertain in the number obtained from a measurement.

The following guides are useful in determining when a number is significant.

- 1) All nonzero digits are significant: 127.34g contains five significant figures.
- 2) All zeros between two nonzero digits are significant: 120.007 m contains six significant figures.
- 3) Unless specifically indicated by the context to be significant, all zeros to the left of an understood decimal point but to the right of a nonzero digit are not significant: 109,000 km contains three significant figures.
- 4) All zeros to the left of an expressed decimal point and to the right of a nonzero digit are significant: 109,000. km contains six significant figures.
- 5) All zeros to the right of a decimal point but to the left of a nonzero digit are not significant: 0.00476 kg contains three significant figures.
- 6) All zeros to the right of a decimal point and to the right of a nonzero digit are significant: 0.04060 cm and 30.00 mg contain four significant figures.



Exercise: How many significant figures does each of the following measurements contain?

- |                |              |
|----------------|--------------|
| a) 127.50 km   | e) 670 mg    |
| b) 1200 m      | f) 0.0730 g  |
| c) 90027.00 cc | g) 43.050 l  |
| d) 0.0053 g    | h) 300900 kg |

Rounding off Results of a Calculation:

Correct use of significant figures makes it possible to give as much information as is available or desirable about a measurement and no misinformation. Chemistry students generally carry out their calculations to an excessive number of decimal places. Such calculations are misleading because they create an erroneous impression of precision.

Suppose you wish to determine the volume of a block of metal. Your measuring instrument is a meter stick having 1 mm divisions. You find the sides to be 3.54 cm, 4.85 cm, and 5.42 cm, estimating the last figure in each case. The area of one surface is

$$3.54 \text{ cm} \times 4.85 \text{ cm} = 17.1690 \text{ cm}^2$$

Recognizing that the product of anything multiplied by a doubtful figure is also doubtful, and that only one such figure may be carried, the result is rounded off to 17.2 cm<sup>2</sup>. It should be obvious that the former degree of accuracy, ten-thousandths of a square centimeter could not be obtained with a meter stick graduated in tenths of a centimeter.

Multiplication and Division: Three points previously discussed should be remembered in these operations:

- 1) The rightmost significant figure in a measurement is of doubtful accuracy.
- 2) The product of any number multiplied by a digit of doubtful accuracy is also uncertain.
- 3) Only one doubtful digit is retained in the result.

Therefore, the product or quotient is accurate to the number of significant figures contained in the least accurate factor.

Addition and Subtraction: The number resulting from an addition or subtraction can have no significant figures to the right of the last significant figure of any number involved in the operation:

$$\begin{array}{r} 29.86 \text{ g} \\ + 3.0579 \text{ g} \\ \hline 32.9179 \text{ g correct sum} = 32.92 \text{ g} \end{array}$$

Exercise

1. Perform the indicated operations and express the result in the proper number of significant figures:
 

a) 8.2365 + 98.62 - 0.062	c) 24 x 12.5
b) 5.146875 + 0.0236 - 3.547	d) 52 x 0.16 ÷ 1537
2. Show how significant figures are important in calculating the density a liquid from the following data:

Weight of container	102.6 g
Weight of container + liquid	126.598 g
Volume of liquid	30.00 ml