THE UNIVERSITY OF MANITOBA

A DEVELOPMENT OF CRITERIA FOR THE PREPARATION OF A GRADE XII CHEMISTRY LABORATORY MANUAL

BEING A THESIS SUBMITTED TO THE COMMITTEE
ON POST-GRADUATE STUDIES IN PARTIAL
FULFILMENT OF THE REQUIREMENTS
FOR THE DEGREE OF MASTER OF
EDUCATION

BY

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WINNIPEG, MANITOBA

JANUARY, 1954

The author wishes to acknowledge his indebtedness to Dr. G. Maccia of the Faculty of Education at the University of Manitoba for his patient and wise counsel during the development of this problem. He is also indebted to Dr. J. Brown, Director of Curriculum. Department of Education of the Manitoba Government, for the use of laboratory manuals belonging to that Department. He has profited greatly also from discussions with his colleagues in the field of chemistry education, particularly from those with Mr. A. Ryckman, formerly of the Daniel McIntyre Collegiate Institute, Winnipeg.

A Development of Criteria for the Preparation of a Grade XII Chemistry Laboratory Manual: Frank Harder, Winnipeg, January 1954.

This study gleans from related areas of education criteria that may be useful in constructing a chemistry laboratory manual. Its survey begins with the broad aims of general education and passes through ever-narrowing fields: secondary education, science education, chemistry education, to laboratory instruction in chemistry.

The basis aim of education in the western nations is training for participation in a democratic society. This training takes the form of a modification of individual growth, both from the social and egoistic viewpoint. Historical evidence suggests that the social viewpoint has been neglected. Involved are factual knowledge, skills, habits, attitudes, and ideals. At the secondary school level, the emphasis is on attitudes. Science education including chemistry, contributes by its emphasis on the scientific attitude. Chemistry makes its unique contribution by presenting sub-surface phenomena of sufficiently subtle a nature to permit the development of discriminating enquiry. Discriminating enquiry requires abstract thinking. In order to emphasize this, the aims of chemistry education have been stated at three increasingly abstract intellectual levels:

(a) An understanding of the composition of matter and the changes it undergoes, and the manner in which this knowledge aids man in the control of his environment.

(b) A revelation of the organization in chemical change.

(c) An appreciation of the elusive nature of truth, and original cause, and an appreciation of the power of directed imagination.

Laboratory education in chemistry serves to provide concrete raw data which to manipulate according to the scientific method; it also serves to broaden the sensory impact of a learning situation. It should employ the simplest situations and skills in order not to confuse its main functions.

This study surveys several current chemistry laboratory manuals to discover what is common practice. From such common practice a number of criteria pertaining to format and content arise. Among the more significant are the following: (a) Manuals are commonly of the fill-in type emphasizing individual performance, (b) For an experiment the object is clearly stated, the method is fully and precisely prescribed, any necessary preview of theory is provided. The emphasis is on clarification of principles.

The culmination of this study is the application of the various criteria in the construction of a laboratory manual, which is included in the appendix to the study.

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CHAPTER I

THE PROBLEM

The Setting of the Problem

The Manitoba Matriculation Course at the secondary school level includes a choice of either one or two sciences from chemistry, physics, or biology. The choice made covers a two-year course beginning in Grade XI (Junior Matriculation), and finishing in Grade XII (Senior Matriculation, equivalent to First Year of Arts or Science at the University of Manitoba). The first year of chemistry introduces most of the fundamental generalizations of inorganic chemistry and provides the descriptive chemistry of a number of common elements and their compounds. The second year of chemistry pursues the study of basic principles more thoroughly, adds a few new studies of such basic principles as the periodic law and oxidation-reduction reactions, introduces some elements of organic chemistry, and expands the descriptive chemistry to include a larger number of industrial applications.

Each year of chemistry is accompanied by a laboratory course, integrated according to individual teacher ability with the regular classroom work in chemistry but taught in specially equipped rooms. The requirements adjust themselves in practice to one 60-90 minute period of laboratory work a week. Governmental inspection assures that this requirement is met in Grade XI; as well, in Grade XII the student is expected to submit to an inspectoral test on laboratory work, for which he receives standing apart from that granted in theory.

The laboratory courses have changed very little in the past twenty years. To the knowledge of this investigator, there has been no official attempt scientifically to appraise laboratory manuals in order to discover how they could serve better the aims of chemistry education. In both years a course of individual laboratory exercises is prescribed and guided by an approved manual. No systematic examination of the nature and content of these manuals had taken place for about twenty years, until two years ago a change in the chemistry text for Grade XI brought about a change in the manual.[1] The Grade XII manual

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[1] Harder, Frank, Outline of Laboratory Experiments for the Province of Manitoba, to be performed as part of the prescribed course in Chemistry II (XI). (Altona, Man.: D. W. Friesen and Sons, 1951).

has not been revised since a reprinting in 1936.[1] Not only does this situation warrant reappraisal of the manual, but criticisms over the years demand it, and indicate the need for a new manual.

Such criticisms for the most part come from the classroom teacher, who is concerned primarily with the practicality of the guidance offered by the old manual. The old (and still current) Grade XII manual does not clearly define the purpose of an experiment. The method of procedure is outlined sparsely; it assumes either laboratory competence on the part of the pupil, or considerable guidance on the part of the teacher in laboratory skills and manipulations. No instruction is given in the simpler skills, and very few precautions are given that might assure both safety and good housekeeping. Quantities are stated in metric units, when for approximations a more familiar unit might avoid misinterpretation. Frequently, the quantity is stated without indicating the degree of precision of its measure. This fault is particularly apparent in the instructions pertaining to the volume of a liquid. There is little guidance of student observations by means of questions.

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[1] Moore, Andrew, <u>Outline of Experimental Work in Practical Chemistry</u>. Winnipeg, Man.: Manitoba Text Book Bureau, reprinted 1936; two pamphlets, one each for Grades XI and XII. The Grade XI pamphlet is now out of print.

As a rule, the conclusion is left entirely to the student, who often does not know what is expected of him. Diagrams are small, and not amplified with associated description. The experiment is never preceded by a review of the essential theory, in the light of which student performance could have meaning.

The Problem

The need expressed for a new manual to guide Grade XII laboratory chemistry in the Province of Manitoba provides the motivation of this investigation. Criticisms have tended to be disorganized. Before they can effectively guide the construction of a new manual they must be organized about sound criteria based on modern educational aims and practice. It is the purpose of this investigation to discover what modern educational aims and practice are, insofar as they have a bearing on laboratory chemistry in the secondary schools, in order to derive therefrom criteria which may serve as guides in the construction of a laboratory manual for Grade XII chemistry.

Limitation of the Problem

Laboratory chemistry, like any other educational effort, is, or should be, so integrated with general education that any widely accepted aim or practice will in some manner have an effect on it. Since it is not practical or even desirable to study all possible factors that could effect it, this study selects only those areas of modern educational effort that seem to impinge most directly on laboratory chemistry, and attempts to derive therefrom certain criteria that will help in the construction of a manual. It will attempt to answer the following questions:

1. What criteria arise out of the study of the aims of education?

2. What criteria arise out of an analysis of the nature and function of laboratory work?

3. What criteria arise out of the study of the effect of guidance on the nature and function of laboratory work?

4. What criteria arise out of a study of common practice in some current laboratory manuals?

5. Of the criteria that may be formulated, which will be selected as most applicable to the construction of a laboratory manual for Manitoba schools?

It has been shown that the situation in Manitoba is ripe for a new Grade XII chemistry laboratory manual. The time lapse since the last revision of the present manual, together with the criticism since that revision both point to the need of a new manual based on modern aims and practice. Such a manual cannot be undertaken until a survey has been made in that area of education directly related to laboratory chemistry, in order to establish criteria that will guide in the construction of the new manual. The survey must, if it is to be reasonably comprehensive, start with those broad aims that guide education in general, and work its way inwards towards narrower fields until it establishes those aims that apply particularly to chemistry education.

CHAPTER II

CRITERIA ARISING OUT OF THE AIMS OF EDUCATION

The Aims of General Education

The basic aim of education has been stated in many ways, the majority of which tend to emphasize a training for participation in a democratic society. In the exposition of the basic aim, stress is laid on individual growth both from the egoistic and the social viewpoint. The basic aim has been analyzed by the Commission of the Reorganization of Secondary Education[1] to include the following:

1. Command of health, both physical and mental,
2. Command of fundamental processes,
3. Worthy home membership,
4. Vocational training,
5. Citizenship training,
6. Worthy use of leisure time,
7. Cultivation of an ethical character.

Ruediger[2] approaches the analysis in a slightly different manner. He considers that the aims of

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[1] Cardinal Principles of Secondary Education, Bulletin No. 35, 1918. Washington: U. S. Department of the Interior, Bureau of Education.

[2] Ruediger, W.C., Teaching Procedures. New York: Houghton Mifflin Co., 1932.

education are to produce certain desirable changes in the individual, and believes that these changes may be viewed from the standpoint of their purpose. His aims may be summarized in the following breakdown of the changes he hopes to achieve:

- Subjective Changes (egoistic)
  - Physical...health development, etc.
  - Mental
    - in Intellect..knowledge, procedures.
    - in Feeling..tastes, ideals, attitudes.
    - in Motor Responses..habits, skills.
- Objective Changes (social)
  - General
    - Social
      - Citizenship..political responsibility.
      - Home Making..social responsibility.
    - Personal..diversions, avocations.
  - Vocational

The achievement of these changes in the individual represents the aim of education at all educational levels.

The Aim of Education in the Secondary Schools

At each educational level there will be a shift of emphasis in aims. For example, at the elementary level the stress is placed on the acquisition of skills and tools, the establishment of the fundamental processes among varied individuals in a manner sufficiently uniform to permit social integration. At the secondary school level the shift of emphasis appears to be towards attitudes and ideals. Kilpatrick's Philosophy of Education illustrates this new emphasis:

> As the individual grows in maturity he must more and more accept the conscious why of what he does; otherwise intelligent self-direction is not being built. (We must, however, not expect younger children to think much of why they act. This kind of learning comes but slowly; to push it too far is to hinder, not help.)[1]

Of secondary education he says:

> No longer should students in the high school or college be evaluated simply as to their knowledge, however full or exact; the real concern should be behaviour--character and behaviour. Knowledge, to be sure, is a highly essential part of the effective character, but it is not sufficient; it is not true that to know what to do suffices to bring about right behaviour. Education then must aim at the whole personality, the all-round inclusive character. In this character, thinking and feeling--disposition to think and to act on thinking, attitudes to dispose thinking and acting to constructive ends--must suffuse and interpenetrate each other and together penetrate all conscious action.[2]

And again:

> Only on the basis of strongly built habits and attitudes can effective behaviour be expected.[3]

John Dewey expresses the same view:

> Our schooling does not educate, if by education be meant a trained habit of discriminating enquiry and

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[1] Kilpatrick, W. H., Philosophy of Education. New York: The Macmillan Co., 1951, p. 366.

[2] Ibid., p. 220.

[3] Ibid., p. 427.

> discriminating belief, the ability to look beneath the surface to detect the conditions which fix the contours of that surface, and the forces which create its waves and drifts.[1]

Other general aims of secondary education have been proposed. The goal may be worker and consumer competence, mental health, or citizenship. Yet, however the goal is expressed, it resolves itself into an emphasis on attitudes and ideal. The narrow interpretation of consumer competence as an aim, for example, suggests little more than the acquisition of useful habits and skills, outcomes that are emphasized in the elementary school and that describe a method of procedure rather than a system of thinking. In the broader sense, however, such a socio-economic aim does imply a system of thinking, for which attitudes and ideals are the guides. Mental health as an educational aim suggests an acceptable balance of attitudes. Citizenship as an educational aim suggests attitudes such as co-operation, tolerance, alertness to weakness in social institutions, etc. Thus, perusal of secondary education indicates that the basic aim is the cultivation of desirable attitudes.

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[1] Dewey, J., Character and Events. New York: Holt and Co., 1929, Vol. II, p. 779.

Aims of Science Education

Since science education is a core study of the modern secondary school curriculum it too must subscribe to the aim of cultivating desirable attitudes. That it has not always done so can be seen by examining the history of science aims. During the nineteenth century, when the theory of formal discipline held sway, science was taught mainly for its informational value. By about 1875 (in the United States) colleges began to dominate the curricula of secondary schools. There followed a period, from which we are not yet free, when science was taught for its preparatory value. With the beginning of the twentieth century there came a period of affluence, when millions instead of thousands flocked to the high schools, and when attention was given to the psychology of learning; the laws of learning, transfer of training, individual differences, etc. Preparation for college could no longer be accepted as an adequate aim. There followed during the first two decades of the century a stress on the practical applications of science, whether these occurred in the individual's environment or not, and then a gradual shift to those which occurred in the individual's environment. From about 1920 and during the next ten years the aim of science was to interpret the individual's environment. The Commission on the Reorganization of Sciences[1] indicated how science interpreted environment from the aspects of the Cardinal Principles laid down by it. The civic aspect of environment began to receive more stress, as well as the vocational. Since it proved difficult to foretell into what vocation an individual might be inducted, or for the future environment for which he should be prepared, the trend of science aims took another direction.

It was the report of the Committee on the Teaching of Science, National Society for the Study of Education in its Thirty-First Yearbook,[2] which marked this new trend. It based its aim of science education on the principle that child psychology was the same as adult psychology, that any differences were relative, not absolute. It stressed two main objectives for all science teaching:

1. A functional understanding of the major principles and generalizations of science.
2. A scientific attitude.

However important the first aim may be, it was stress on the second that marks the new trend. The new school

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[1] Op. cit.

[2] National Society for the Study of Education, Thirty-First Yearbook, Part I: A Program for the Teaching of Science. (Bloomington, Ill.: Public School Publishing Co., 1932).

of thought shifts the emphasis from inculcation of facts and habits to the acquisition of attitudes and ideals.

Curtis, one of the members of the committee, analyzed the scientific attitude to include the following:

1. Conviction of basic cause and effect relations, making untenable
    (a) Superstitious beliefs in general,
    (b) Unexplained mysteries,
    (c) 'Beats all' attitude.
2. Sensitive curiosity concerning reasons for happenings, coupled with ideals
    (a) of careful and accurate observations or of equally careful and accurate use of pertinent data,
    (b) of patient collecting of data,
    (c) of persistence in the search for adequate explanation.
3. Habit of delayed response, holding views tentatively for suitable reflection.
4. Habit of weighing evidence with respect to its
    (a) pertinence,
    (b) soundness,
    (c) adequacy.
5. Respect for another's point of view, an open-mindedness and willingness to be convinced by evidence.[1]

But a prescription of attitudes was not of itself considered sufficient. Not only must there be a mental set (in the form of scientific attitude), it must express itself by means of the most efficient system of thinking. This is the scientific method, in practice probably inseparable from the scientific

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[1] Ibid., p. 56.

attitude. The Thirty-Fourth Yearbook of the National Society for the Study of Education presented an analysis of this method by Curtis.[1] He suggested that an individual must obtain power in:

1. Locating problems,
2. Making hypotheses from facts,
3. Recognizing errors or defects in the conditions or experiments described,
4. Evaluating data or procedures,
5. Evaluating conclusions in the light of facts or observations,
6. Planning and making certain new observations to find out whether certain conclusions are sound,
7. Making inferences from facts and experiments,
8. Inventing check experiments,
9. Using controls,
10. Isolating the experimental factors.

Yet even in the reports of the Thirty-First Yearbook[2] there was already apparent divergence of thought that spelled out a further shift of emphasis on aims. In the analysis of the scientific attitude by Curtis, already quoted,[3] the emphasis is laid on the cultivation of egoistic attitudes, i.e. development within the individual by himself and for himself. All except the last category, "Respect for another's point of view", tend to develop the individual without any particular reference to his relations with his fellow man. A

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[1] F. D. Curtis, "Diagnosis and Remedial Treatment in the Field of Science," Thirty-Fourth Yearbook, National Society for the Study of Education (Bloomington Illinois: Public School Publishing Co., 1935), pp. 331-345.

[2] Op. cit., p. 12, this study.

[3] See page 13 of this study.

successful society depends as much on such egoistic development as it does on the development of desirable social attitudes. It is even possible that such development will be accompanied by a desirable change in the social attitudes, but there is no assurance that it will be. The history of World War II has revealed that man and societies may develop a sound scientific attitude without acquiring a desirable social attitude. The results are such that education is seeking to mend its fences by a greater emphasis on the latter. In the same report in the <u>Thirty-First Yearbook</u>[1] Craig showed that this emphasis had not been overlooked even then by at least come of the members of the committee. He suggests that a scientific attitude calls for an acceptance of the following appreciations:

1. Man's concept of truth changes.
2. Orderliness prevails in nature.
3. The formation of hypotheses and the testing of them by experimental study are essential steps in the scientific method.
4. Attainments in science have bred confidence in the scientific method.
5. Much knowledge remains to be revealed.

Inherent in this analysis is an emphasis of social attitudes. Items 1 and 5 imply an attitude of tolerance; item 2 implies an attitude of co-operation; item 3 implies an appreciation of directed imagination that may result in a sympathetic attitude towards the

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[1] Op. cit., p. 12 of this study.

creative arts and religion.

Currently this emphasis on social attitudes is indicated by I. I. Rabi, Professor of Physics at Columbia University, and 1944 Nobel Prize Winner for his work in atomic physics:

> What does the tradition of science teach us? It teaches us moderation and tolerance of ideas, not because of lack of faith in one's belief, but because every view is subject to change, and every truth we know is only partial. The strange thought or custom may still prevail.
>
> It teaches us co-operation, not only among people of the same kind, but also among the most diverse origins and cultures. Science is the most successful co-operative effort of mankind.
>
> Science inspires us with a feeling of hopefulness and of infinite possibility. Science shows that it is possible to foresee and to plan, and that we can take our future in our hands if we but rid ourselves of prejudice and superstition.
>
> The tradition of science teaches us that no vested interests in institutions or systems of thought should escape continual re-examination merely because they have existed and have been successful. On the other hand it teaches us to conserve what is operative and useful.
>
> Science teaches us self-discipline. One must continually look for the mote in one's eye. The history of science shows us that it is always there.[1]

Aims of Chemistry Education

Chemistry education, as one area of science education, must subscribe to the aims of science education in general, with the same emphasis on the cultivation of desirable egoistic and social attitudes. But

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[1] Rabi, I. I., "Faith in Science", The Atlantic Monthly, Vol. 187, No. 1, January 1951, p. 30.

it makes its own unique contribution towards the achievement of these aims. At the high school level, chemistry more than physics deals with those subtle phenomena that are underneath the surface and that mould the surface contours, which, as Dewey says, call for a "trained habit of discriminating enquiry and discriminating belief."[1] Biology at the high school level, on the other hand, is either too shallow or extensive to offer much exercise for "discriminating enquiry and belief", or it presents sub-surface phenomena that are too subtle for anyone but the specialist. Discriminating enquiry may occur at any level of observation; it is the degree of subtlety of discrimination that appears to distinguish between the educational values of the different physical sciences at the high school level.

The aims of science education, and therefore of chemistry education, have been so comprehensively stated by other, more competent, authorities that basically they are probably fixed. They appear to be directed toward producer-consumer competence and the development of a scientific attitude. A re-assessment and restatement of these aims is defensible mainly because there is evidence that the modern community needs a strengthening of its social attitudes. A restatement of aims of

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[1] See page 10 of this study.

chemistry education may succeed in swinging the emphasis towards cultivation of those attitudes which aid in the social integration of the individual.

In order to provide the shift of emphasis indicated, the following aims for chemistry education are proposed:

(1) An understanding of the composition of matter and the changes it undergoes, and the manner in which this knowledge aids man in the control of his environment.

(2) A revelation of organization in the changes of the composition of matter.

(3) An appreciation of the elusive nature of cause and of truth, and an appreciation of the power of directed imagination.

It appears implicit in these aims that a scientific attitude will be the means of their achievement. Of themselves they do not differ much from previously stated aims, and would no more stress social attitudes as mentioned by Rabi and others than do the latter. The recognition that they represent three different intellectual levels of approach is the significant contribution of their presentation. It is submitted that such recognition makes possible a direction of emphasis towards desirable attitudes, by placing in a subsidiary rank the knowledge and skills that also will be outcomes of chemistry education. The three intellectual levels of approach in a chemistry lesson may be considered the general objectives; they are described as follows:

(a) The lowest level of intellectual approach is factual description and knowledge. It is represented by the first aim. It is necessary in order to provide the raw material from which to generalize. It provides interest by relating the new areas of investigation to older, more familiar concepts. It presents new vistas to the enquiring mind. Rather frequently the bulk of text-content consists of material furthering this level of approach. Its outcomes will be knowledge, skills, and habits. Of itself it does not provide for the development of attitudes and ideals.

(b) The next higher level of intellectual approach involves organization, which makes generalization possible. It is represented by the second aim stated. Elementary organizing ability is probably inherent, but attitudes of discrimination, tolerance, co-operation call for a more complex organizing ability that must be developed. Attitudes and ideals grow from, among other things, understanding and appreciation. Complete understanding requires the realization of relationships, expressed as generalizations. If the greater emphasis is placed on the generalizations of chemistry (the laws and theories), and less on the raw data from which these arise, chemistry becomes a tool capable of developing attitudes and ideals.[1]

In the development and use of chemical generalizations the need for discriminating enquiry is so apparent, the consequences of a lack of intellectual honesty so immediate, that chemistry lends itself well to the cultivation of these attitudes. When the chemistry student sees that the sum total of our knowledge

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[1] That this is recognized by local educational authorities is indicated by the long-term trend in the Manitoba Department of Education Matriculation Examinations for Chemistry XI. A study of these examinations covering a span of forty-two years results in the conclusions:

> <u>There has been a strong upward trend in mark allotment to questions concerning intelligent interpretation</u>...Descriptive chemistry and memory equations are tending to receive fewer marks since about 1935. Mathematical problems likewise have tended to receive fewer marks, although there is a suggestion of a reversal of this trend during the past few years. Language shows no particular trend. <u>Questions on the laws and theories show the strongest trend towards an increase of mark allotment</u>...There is some indication that a similar increase of emphasis is beginning with respect to other items stressing logical response, namely chemical shorthand which requires intelligent manipulation, and scientific logic other than that dealing with the laws and theories.[a]

[a] Harder, Frank, "A Study of Trends in Test Standards as Revealed by the Matriculation Tests in Chemistry XI issued by the Manitoba Department of Education" (Unpublished course study, Faculty of Education, University of Manitoba, 1952).

concerning the atom resulted from the combined efforts of Jews, Germans, Russians, Americans, Japanese, and English; when he sees that the co-operative efforts total more than the sum of the individual efforts, then he has been brought to a state of readiness for such attitudes as co-operation and tolerance. When he sees that even the most revered figures in science made errors, and that these were revealed in a spirit of unemotional enquiry, he is likely to appreciate and accept the spirit of discriminating enquiry into the most holy of his traditions, and thereby lay the foundation for intellectual independence.

(e) At the highest intellectual level of approach, chemistry education strips man of his conceit by revealing the limitations of his understanding, and the elusive nature of the truth he seeks. It reveals and exalts the value of directed imagination. The difference between the rank-and-file laboratory technician and the genius who explores the unknown lies in this creative spark, this directed imagination. It reveals to him the need for at least qualified acceptance of the intangible, as in the case of the graviton and the meson known to man only by their secondary manifestations. It reveals to him that science, like religion, seeks that ultimate theory which will explain all phenomena. Such revelation makes possible at least a tolerance for, and perhaps a considerable sympathy with, the creative arts, religion and philosophy.

The chemistry teacher must attempt to approach any lesson at all three levels. The extent to which each will be emphasized depends on the capacity of his students, which he, among educationaists, is best fitted to judge. At the General Science level of Grade X it is probable that he will not go much beyond the lowest intellectual level; the scarcity of data about any focus prevents the development of comprehensive generalization. The Grade XI level, at which chemistry instruction is begun in Manitoba, will receive instruction at the first two intellectual levels, with emphasis on the second. The third intellectual level will have limited application in Grade X, occasional application in Grade XI, and more frequent application in Grade XII. It is perhaps apparent that this third level of approach will never be fully explored even at the university level. Yet the teacher must be alert for opportunities to work at this highest level whenever possible, since it is at these upper two levels of approach that gains may be made in developing personal and social attitudes and ideals.

No facet of chemistry instruction can be free of the influence of its aims. But one facet, such as laboratory work, may stress only a portion of the total aim. Chemistry, along with the other physical sciences, possesses the somewhat unique merit of being able to present to the individual in the laboratory the concrete raw data at an intellectual level that permits early departure into the abstract. A laboratory course that presents isolated facts, properties, applications, and stops there, is not serving to its full extent the general aims of chemistry education.

That abstraction is one of the aims in general education is indicated by the British Columbia educational authorities:

> A child's system of thought becomes more and more independent of immediate concrete objects as he matures. The ability to work in the imagination and to build up a system of ideas is essential in education. To limit the curriculum to that which has immediate practical application is to overlook the truth. Projects form the starting point of learning, but they are never the goal.[1]

Laboratory exercises constitute the project-work mentioned by the British Columbia authorities, valuable as a starting point of learning; but the achievement of a generalization is the goal. This view is supported by John Dewey when he says:

There is no science without abstraction and abstrac-

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[1] Department of Education, British Columbia, Programme of Studies for the Senior High Schools of British Columbia, Bulletin 1, 1941, p. 12.

> tion means fundamentally that certain occurrences are removed from the dimensions of familiar practical experience into that of reflective or theoretical enquiry.[1]

The background for chemistry education has been outlined, albeit in a sketchy fashion. Nevertheless, certain criteria stand out that may serve in guiding the aim of a chemistry course at the high school level. These are:

1. The basic aim of education in Canada and the United States is training for participation in a democratic society.

2. This training is brought about by changing or cultivating skills, habits, ideals, and attitudes.

3. At different levels in the school system different aspects of this basic aim receive emphasis. At the secondary school level the emphasis appears to be on the cultivation of desirable attitudes.

4. Science education in the secondary school contributes to this cultivation of desirable attitudes by its emphasis on the scientific attitude.

5. The changes in attitude may emphasize the egocentric or social nature of these. History suggests that the egocentric attitudes, although desirable, have tended to crowd out the cultivation of social attitudes.

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[1] Dewey, John, The Sources of a Science of Education (New York: Liveright, 1929), pp. 16-18.

An attempt should be made to create greater social consciousness by emphasizing the social attitudes.

6. Science education can so direct its training in scientific attitude as to emphasize its social implications.

7. Chemistry is one facet of science education that presents phenomena that are neither too obvious not too subtle for discriminating enquiry and belief at the secondary school level. Discriminating enquiry and belief are functions of a socially-centred scientific attitude.

8. Discriminating enquiry and belief are functions of abstract thinking. In order to emphasize this, the aims of chemistry education are expressed at three increasingly abstract intellectual levels, thus ranking teaching items in order of their educational significance:

(a) An understanding of the composition of matter and the changes it undergoes, and the manner in which this knowledge aids man in the control of his environment.

(b) A revelation of organization in the changes of the composition of matter.

(c) An appreciation of the elusive nature of cause, and of truth, and an appreciation of the power of directed imagination.

In chemistry education the emphasis should always be placed on the highest intellectual level that the teaching situation will permit.

9. In laboratory chemistry the emphasis must be on discriminating enquiry (which implies intellectual honesty), and on generalizations that emphasize abstract thinking.

Having developed the broad base on which all chemistry education will rest, it is possible now to direct attention towards the narrower field of laboratory chemistry. The nature and function of this area of secondary education have at times been confused with those of other areas, with the result that claims have been made for it that either could not be upheld, or which could be achieved more economically by other means. For example, the Nova Scotia Handbook to the Course of Study after listing such habits as carefulness and skill in manipulating apparatus; accuracy of measurement; careful observation; system, order, and neatness in the arranging of apparatus; accurate and methodical arrangement in reporting; legible writing; clear and neat drawings; states:

> Laboratory instruction is the great agent in developing these habits. It had been said that the calibre of the chemistry instructor is indicated by the manner of the student's 'housekeeping' in

the laboratory.[1]

If this is the chief function of laboratory chemistry the question might arise whether some other means, less costly in time and money, might not achieve the same results. Certainly, before attempting to establish further criteria for laboratory guidance it is essential to define, at least for the purpose of this study, the nature and function of laboratory work.

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[1] Department of Education, Nova Scotia, Handbook to the Course of Study, 1935, reprinted as a wartime edition, pp. 644-645.

CHAPTER III

CRITERIA ARISING OUT OF THE NATURE AND FUNCTION OF LABORATORY WORK

The Nature of Laboratory Work

What is the essence of laboratory procedure? Is it individual manipulation of raw data? If so, the term seems to have been usurped by other educational areas, e.g. whole schools are organized on a Dalton Laboratory Plan. Thomas M. Risk analyzes the meaning of the term 'laboratory method' thus:

> The term laboratory method has been variously interpreted and variously applied to almost the whole gamut of school subjects in the secondary field. According to Webster, the word laboratory was originally applied to the workroom of a chemist, a place devoted to experimental study in natural science...to testing, analyzing, and preparing various chemical materials, etc. Hence the term laboratory method was first widely used in the physical sciences to characterize a teaching procedure making use of experimentation with apparatus and materials in order to verify physical laws and other facts, as well as to study relationships and to discover new facts if possible. Such procedure was introduced in many American secondary schools as early as 1880, but it was not used extensively until about 1900.
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> Following the early introduction of laboratory work in the physical sciences, the idea of applying the same philosophy of dealing directly with materials in the classroom was soon proposed in other fields. For example, the source method of teaching history was proposed in the 80's and was put into practice in a considerable number of schools in the

> 90's. In 1896, Dewey, in his book "The School and Society" set forth his philosophy: 'We learn by doing', thereby giving impetus to the laboratory attack not only in home economics and manual training, but in all subjects. Since that time, various adaptations of laboratory procedure have been tried out in many subjects, more particularly in the physical and biological sciences, the social sciences, English, mathematics, and the vocational and commercial subjects.
>
> We may define the laboratory procedure as planned learning activity dealing with the original or 'raw' data in the solution of problems. The laboratory idea means first-hand experience with materials or facts derived from investigation or experimentation, for the purpose of solving some problem or answering some question, instead of hunting for a ready-made book answer. The method was first interpreted to apply to the use of raw data handled experimentally or obtained experimentally, but the meaning was gradually broadened to include work with any material so long as the laboratory technique was used. This interpretation makes it akin to supervised study, and as often used now, this is really what it amounts to.[1]

This development of the term 'laboratory procedure' emphasized the application of the scientific method to raw data in order to solve a problem or answer a question. The physical sciences have the special advantage of permitting the extensive use of concrete raw data in the laboratory, as contrasted with extensive use of source material in the form of printed records. It must be noted, however, that the high school chemistry laboratory is not involved exclusively with problem solving in the true sense. What are termed

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[1] Risk, Thomas M., Principles and Practices of Teaching in the Secondary Schools (New York: American Book Co., 1947), pp. 475 ff.

experiments are frequently exercises in which the individual demonstrates to himself the chemical characteristics of an element or compound, thus broadening out the sensory impact to result in a better assimilative situation. As has been indicated,[1] such exercises should, where possible, lead to generalization.

The Function of Chemistry Laboratory Work

The Thirty-first Yearbook of the National Society for the Study of Education is the first comprehensive report by that body in the field of science education. Judging by the frequent references to it in curriculum construction it may be assumed to be a highly authoritative source. In it Ralph E. Watkins suggests certain objectives that may be attained by laboratory instruction in the high school:

> 1. The development of simple laboratory techniques, such as weighing, glass-bending, microscopic manipulation, etc.
> 2. Proving and establishing for the pupil himself principles which have long since been well established and generally accepted.
> 3. Using the laboratory as an instrument of object or 'thing' teaching, according to the historical concepts of Pestalozzi, Comenius, and Basedow.
> 4. Using the laboratory for the purpose of developing better understanding and interpretation of the principles of science, as a means of better illustration.
> 5. To produce training in the scientific method.
> 6. As a means of possible training in the experimental solution of the pupil's own problems.
> 7. The use of the laboratory as a workshop for the

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[1] See page 24 of this study.

> study of science problems which arise in the science class or in the life of the pupils.[1]

It is particularly regrettable, for the sake of clarity of aim, that a high authoritative source of this nature should initiate a tendency to list as of equal merit the various objectives of an educational effort. An examination of the objectives quoted suggests that there is little difference between objectives 2 and 4; objectives 6 and 7 are less significant corollaries or extensions of objectives 2 and 4. Objective 1 is a means towards the other objectives; it cannot be considered an objective itself in the light of the criteria for chemistry education as previously established in this study. The set of objectives quoted might offer superior guidance to chemistry education if it were rearranged to emphasize the relative importance of the different objectives. According to the criteria already developed, this rearrangement might be somewhat as follows:

1. Using the laboratory for the purpose of developing better understanding and interpretation of the principles of science, as a means of better illustration.

('Proving and establishing for the pupil him-

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[1] Watkins, Ralph K., "Instruction in the Physical Sciences in the Secondary Schools", Thirty-first Year-Book, National Society for the Study of Education (Bloomington, Ill.: Public School Publishing Co., 1932), p. 279.

self principles which have long since been established and are generally accepted' is merely the inductive approach to this same objective. This objective is fundamental to the acquisition of desirable attitudes, according to the criteria already established.)

2. To produce training in the scientific method.
3. Using the laboratory as an instrument of object or 'thing' teaching according to the historical concepts of Pestalozzi, Comenius, and Basedow.

As secondary objectives, where the situation permits, the following should be noted:

4. As a means of possible training in the experimental solution of the pupil's own problems.

   This is an extension of the objective, 'to produce training in the scientific method'.

5. The use of the laboratory as a workshop for the study of science problems which arise in the science class or in the life of the pupil.

   This is very similar to objective 4 just preceding. These two objectives represent the type of extension of laboratory function that increases the cost of the laboratory work both in time and in money.

The acquisition of laboratory skills should be kept at a minimum in order not to confuse the major objectives.

The general direction of chemistry education in the laboratory becomes less clear-cut if the objectives are not ranked in order of importance. The result sometimes is a growth of expensive and elaborate laboratory workshops whose exact function is no longer certain. That this has taken place is indicated by the criticisms of the laboratory by Henry C. Morrison:

> That our elaborate laboratories can be justified as appurtenances of general education is scarcely open to doubt; they have no purpose in general education. They belong to the period of specialized training in which the primary purpose is to develop investigators, that is, professional people who will know how to settle moot points which arise in professional practice...Laboratory materials of the science type in the secondary school should be limited to what the student can use. The use of such materials in the assimilation period is limited to the need of putting the pupil within reach of experience which can arise out of physical contact with concrete appliances and observation of processes in their physical manifestation. Assimilation is learning from experience, and in science the most illuminating experience often arises out of the contemplation of processes at work. The only justification for assigning a laboratory exercise to be worked is an affirmative answer to the question Will the exercise proposed make better assimilative material than a demonstration which the teacher can present or a certain series of pages in the assigned reading? If the apparatus assigned for us is so elaborate that the pupil is obliged to learn a difficult task in manipulation, it is extremely unlikely that any particular assimilative value on the unit itself will be contributed.[1]

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[1] Morrison, Henry C., <u>The Practice of Teaching in the Secondary School</u> (Chicago: University of Chicago Press, 1931), pp. 288-289.

Morrison's condemnation is of the elaborate laboratory employing elaborate laboratory skills. He makes it plain that in his opinion the acquisition of laboratory skills is of secondary importance. It is further evident that to him the laboratory is of itself no end, but rather merely another tool by means of which assimilative material may be presented to the individual.

Authoritative opinion has been cited in order to show that the nature and function of laboratory work are governed by the following criteria:

1. Chemistry laboratory work emphasizes the application of the scientific method to first-hand, concrete, raw data, in order to solve a problem or to answer a question.

2. The problems or questions to be dealt with should be related to some generalization of chemistry, and should help to clarify it.

3. Chemistry laboratory work also serves to broaden the sensory impact of a learning situation, so that it may result in a better assimilation.

4. The acquisition of laboratory skills is a means towards the more important ends just stated.

5. Laboratory work should employ the simplest procedures and skills that will suffice to illustrate or clarify a problem.

These criteria carry the implication of guidance and even of restraint at the high school level. It is difficult to imagine the average high school student able to direct his efforts efficiently and economically towards the goals suggested without outside guidance. How is this outside guidance to function, and what must be its nature, to avoid that extreme of restraint that ends in puppet-performance on the part of the student? It is logical that the next phase of this study shall deal with the effect of guidance on the nature and function of laboratory work, in order to seek criteria for such guidance that will strike a balance between the extremes of no guidance and over-guidance.

CHAPTER IV

CRITERIA ARISING OUT OF THE NATURE OF GUIDANCE AND ITS EFFECT ON LABORATORY WORK

Laboratory work involves either problem solving or illustration. In either case the problem or illustration must be isolated from a mass of irrelevant data before it can be handled intelligently. This task is so difficult that usually it must be under teacher or manual guidance. The degree of defining the problem determines to a large extent the degree of student initiative and student thinking. Furthermore, after the problem has been limited, student performance must still be guided for sake of economy, efficiency, and clarity of thought. This guidance strengthens the discipline necessary for discarding interesting and perhaps worthwhile side-issues that might detract from the main purpose of the laboratory exercise.

The Dilemma in Laboratory Guidance

The extent of guidance strongly influences the thinking, the spontaneity, and the initiative of the individual. Indeed, in an average class it may reduce the effort of the less aggressive individual to a puppet-like performance under the close guidance by teacher or manual. Under such conditions education is replaced by training. This is the dilemma which faces the author of the manual: either to provide a situation with minimum guidance where the sink-or-swim policy will produce for some students a truly assimilative experience at the expense of number other casulaties; or to provide close guidance in order to prevent these numerous casualties, at the risk of stultifying student ingenuity and initiative. Where classes are uniformly small it is possible, of course, to give individual treatment that avoids both extremes, but in mass education a choice must be made.

The <u>Forty-Sixth Yearbook</u> of the National Society for the Study of Education discusses this dilemma thus:

> Problem solving activities are an integral part of science teaching and learning, and the science laboratory is a natural place for the pupils to engage in these activities. To avoid the "cook-book" type of laboratory work, the following approaches are suggested:
>
> <u>Use laboratory work to give the pupils practice in raising and defining worthwhile problems</u>. Each experiment should contribute to the solution of a problem. Pupils should be encouraged to share in the stating of problems and in the planning of their solutions. A co-operative approach may be used in which the teacher opens up the field of investigation, draws upon the experience of pupils to suggest where problems exist, and then encourages them to state the problem in their own words. The teacher should guide the pupils in stating problems clearly and concisely. Each problem should present a single, clearly defined question which the class can reason-

> ably be expected to solve on the basis of data which they obtain. Throughout the laboratory period the teacher should make every effort to see that the pupils keep the object of the experiment continually in mind.
>
> Conduct laboratory work in such a way that the pupil will learn the meaning and use of controls in experimentation. Pupils should understand clearly the necessity of permitting only one variable in an experiment. Frequently by wise questioning and discussion the teacher can get pupils to state the purpose of the experiment, to suggest the experimental factor, and to plan the necessary controls.
>
> Use laboratory work to test hypotheses and interpret data. Skill in interpreting evidence and drawing inferences in the solution of problems depends, in large measure, on the ability to organize, arrange, and analyze data. Pupils need to be given considerable opportunity for practice in analyzing data from problem situations. Too frequently in laboratory work we pass immediately from data to conclusions. More time should be given to proposing and testing hypotheses, as these abilities are exceedingly important in modern living.
>
> Maintain a proper balance between student exploration and teacher guidance. Many teachers assign laboratory manuals or workbooks to guide the student in the completion of laboratory work. The opposite extreme of this practice is the 'sink-or-swim' method in which the teacher provides the material but expects the student to invent his own method of investigation. With the large number of pupils most teachers have to teach, and with the great variability in interest and intelligence found in science classes, it seems likely that a happy medium between these two extremes may be found.[1]

The keynote in these suggestions is a proper balance between teacher or manual guidance and student exploration. This balance is affected by the ability of the individual teacher and by the nature of the

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[1] Heiss, Elwood D., "The Role of the Laboratory in Teaching Science", Forty-Sixth Yearbook of the National Society for the Study of Education, Part I: Science Education in American Schools (Chicago: University of Chicago Press, 1947), pp. 235-236.

individual situation. It cannot be assumed that the individual teacher is always competent to give thorough guidance. A printed manual is of necessity rigid, and predetermined to fit what is thought to be an average situation, not necessarily the individual situation.

A way out of the dilemma would be to construct a manual that provides fairly thorough and complete guidance, then augment this, as much as the individual situation will permit, by teacher guidance. The laboratory period could begin, without reference to the manual, by a co-operative development and discussion of the problem. Where the situation permits it might lead to some individual exploration. The experiment would then be undertaken by the class as a whole, using a manual with rather complete guidance. When the data have been accumulated, there could then be a class discussion on their use, falling back later on the manual directions, in the case of the bright student for corroboration of the thought developed by himself, and in the case of the dull or disinterested student for a channelling of his thought.

A survey of some recent manuals[1] indicates that the choice commonly made is to provide extensive and

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[1] Harder, Frank, "An Investigation of the Curricular Content of Several Chemistry Manuals" (Unpublished course study, Faculty of Education, University of Manitoba, 1953).

close guidance directed at the average individual; superior individuals are provided for by extra exercises with minimum guidance, provided either by the teacher or the manual. This is the criterion which will be adopted in the construction of a manual during this study.

Teacher Demonstration vs. Individual Experiments

Depending on the extent of guidance, there may be very little difference between teacher-operated and student-operated experiments, except in the actual manipulation. If the aim is closely to direct student performance and thinking it might be wiser to remove from his path all extraneous obstacles. Let the teacher manipulate the raw material, perform the experiment, and at each step in the performance ask for observations, invite suggestions for further procedure, conclusions, etc. The student mind may be kept from wandering, and might be directed more effectively into desirable channels. This, however, raises the question of student participation. Thorndike's Law of Effect emphasizes the preference of student participation in order to stimulate interest and to make more vivid and impressive the learning situation. In large classes student participation is more apt to assure the close attention of the majority of students. Yet student participation is normally at the expense of time, teacher effort, and money. The situation has given rise to a running debate on the relative efficacy of teacher or lecture-desk demonstrations vs. student-operated experiments.

Available literature lacks reports on investigations designed to discover whether some other method of presenting assimilative material, perhaps less expensive in time, effort and money, can replace laboratory work; it is, however, rich in reports on investigations undertaking the comparison of the lecture-desk demonstration with the student-operated experiment. The results, on the whole, of these investigations show no conclusive evidence of the superiority of one method over the other. Perhaps Morrison's criticisms of these investigations explains the seeming futility of attempting to prove either method superior:

> The method is commonly that of the controlled experiment. Mr. A. selects two groups of pupils of as nearly as possible equal ability, and he industriously eliminates all the variables he can think of, but he does not validate his own classroom technique, his material of instruction, his test material, (save as he secures a statistical validation on the basis of unvalidated assumptions) or his theory of education. He eventually 'proves' ordinarily a negative, that is, he proves that there is not much difference between two procedures...lecture table and laboratory in this case. The sad thing is that we do not know whether Mr. A can teach by any method. The value of ten thousand such experiments would be wiped out if a single Mr. B should show that an expensive and elaborate high school laboratory could be made to produce results quite beyond what lecture-table demonstra-

> tions can do, provided Mr. B could analyze his results and show why his facts are what they are. A single issue would then arise: Is Mr. B's procedure essential to the educative process? If the answer be in the affirmative, then we must all learn somehow to do what Mr. B does and find the money for the equipment.
>
> The specific question is typical of questions which cannot be arrived at experimentally. The problem is historical and analytical, and experimentation is valuable only for the purpose of fact-finding on specific points in the analysis.[1]

Francis D. Curtis, who made a comprehensive survey of investigations attempting to compare the two methods of presentation, sums up his findings in the Thirty-First Yearbook of the National Society for the Study of Education:

> 1. Each method offers training in certain knowledges, skills, and habits not offered by the other.
> 2. In the interest of economy both of time and money, it seems desirable to perform more laboratory exercises by the demonstration than by the individual method.
> 3. At the beginning of every laboratory course there should be a sufficient use of the demonstration method to acquaint the pupil with apparatus and some of the accepted methods of experimentation. Following this period of orientation the pupil should be allowed to perform some exercises individually in order to acquire, early in the course, desirable manipulatory skills and laboratory techniques and habits.
> 4. The time saved in each course by the use of the demonstration method should be used for other types of learning exercises, which might include additional laboratory exercises, reading projects, individual investigations, observations, and drill upon essentials.
> 5. Among the exercises to be demonstrated in each course should be those which are dangerous if per-

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[1] Op. cit., p. 289.

> formed carelessly or unskilfully, at least some of those requiring delicate manipulation and accurate observation, and those requiring special expensive apparatus.
> 6. In general, especially with the more elementary courses and the younger or less capable students, demonstrations by the teacher are likely to prove more effective than those by the pupils, unless the teacher supervise the demonstrations very closely.[1]

This advocacy of teacher demonstrations applies particularly to elementary courses and to younger and less capable students. The situation for which criteria are being developed is a second year course in Grade XII chemistry; the same emphasis on teacher demonstrations need not apply. It appears a useful criterion to leave to the classroom instructor the choice, in the light of his particular situation, of performing the experiment himself or of having his pupils perform it.

The criteria affecting the nature of guidance that have been developed may be summed up thusly:

1. Pupils should be encouraged to share in the stating of problems, and in the planning of their solutions.

2. The interpretation of data should, at the secondary school level, be a co-operative effort by pupil and teacher.

3. For the sake of maintaining interest and

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[1] Op. Cit., p. 91.

attention the majority of the laboratory experiments should be performed by the pupils. There is little evidence of the superiority of either the teacher demonstration or of the student-performed experiment.

4. It is advisable to leave to the teacher the choice of demonstration experiments.

5. A laboratory manual should provide fairly complete guidance. Its use should be augmented by class discussions that will develop the problem, explore pertinent background, suggest means of solving the problem, and weight the evidence. A judicious balance between the use of the manual and teacher guidance of class discussion can provide the opportunity for the brighter student to exercise his initiative, ingenuity, and thinking.

It will be shown in the following chapter that the majority of these criteria function in the common practice found in several current laboratory manuals.

CHAPTER V

CRITERIA ARISING OUT OF COMMON PRACTICE IN SOME CURRENT LABORATORY MANUALS[1]

The practical execution of the criteria developed is the next logical stage in this investigation. Yet, in order that this execution may not reflect too completely the idiosyncrasies of an individual, it was considered that practical execution should be preceded by at least a limited survey of common practices in current manual construction.

Fifteen laboratory manuals were available for the investigation. All except one had been published or revised since 1940. All except two were of the fill-in type; of these two, one had been published prior to 1940, and one was identical in content with a companion-manual of the fill-in type published by the same authors. These two were discarded from the investigation. One manual was such a thorough fusion of theory instruction, work-book, and laboratory manual that it was eliminated because of the difficulties of

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[1] Harder, Frank, "An Investigation of the Curricular Content of Several Chemistry Manuals" (Unpublished course study, Faculty of Education, University of Manitoba, 1953).

isolating its laboratory components. Four manuals revealed, either by their content or by the text which they served, that they were essentially for university students; these were also eliminated from the investigation. This left eight manuals, numbered 3, 4, 5, 6, 7, 10, 11, and 13 for purposes of discussion and so listed in the bibliography. These manuals, with two exceptions, are first editions published since 1940. One of the exceptions had been published in three prior editions; the other exception had been published in five editions, the first in 1921 and the last in 1949. Of these two manuals the final edition was investigated.

There is immediate indication of certain common practice. The eight manuals retained for study are all of the fill-in type. All clearly indicate the purpose of each experiment. All use large, clear diagrams. None of them are demonstration-centred, and most of them leave to the laboratory instructor the choice of whether any experiment will be a teacher demonstration or a pupil experiment.

The Choice of Evidence

Certain aspects of laboratory manuals are easier to discover, and seem to be better guides to the chemistry teacher than others in the preparation of a new manual.

In order to evolve criteria for the construction of a laboratory manual the writer attempted to discover how common practice in the eight manuals being surveyed answers the following questions:

(a) What is the stated function of the laboratory?

(b) Which experiments are basic to high school laboratory courses?

(c) What is the nature of basic laboratory experiments, and what is the distribution of the different types of experiments?

(d) To what extent is there systematic introduction to the laboratory course?

(e) How completely is the necessary theory reviewed for each experiment?

(f) How completely is the method of procedure outlined for the student?

(g) What degree of freedom of choice is there for the teacher or student?

To clarify this part of the investigation it is essential that certain terms used are clearly understood.

An experiment is a situation in which a new problem is raised, a new procedure is indicated, or different apparatus is needed. However, the definition is not too precise. In general, the titles used by the authors are each taken to represent one experiment. Only occasionally, where the presentation makes it obvious that one title covered several experiments, was a breakdown attempted. Lacking precision of definition, a degree of subjectivity enters into the use of this term. In general, where the new problems, procedures, or apparatus are very simple or elementary, they are not rated as separate experiments. For example, to show the difference between a compound and a mixture one might (a) draw a magnet through the mixture, (b) shake it with carbon disulphide and filter. Although these are different procedures they are of such a simple nature that they are not rated as separate experiments.

Basic experiments are those which occur in at least three of the eight manuals being surveyed. For the purpose of simplification they are listed under generic headings; thus, if it is found that all eight manuals have experiments under the generic heading 'Oxygen' it does not follow that there is identity between the different experiments so classified.

The characteristics that define the nature of an experiment for the purpose of this study are taken to be:

(a) Qualitative or appreciative experiments: These involve only the handling of simple laboratory apparatus, with no fitting together of individual items. They demand no mathematical calculations, and the minimum of laboratory skills. They demand mainly student observation and interpretation.

(b) Complex apparatus experiments: These involve the fitting together of several items of common laboratory equipment. They demand, besides student observation and interpretation, a measure of laboratory skills.

(c) Quantitative experiments: These demand either precise measurement, or precise measurement and mathematical calculation. Besides demanding observation, interpretation, and possibly laboratory skills, they call for maximum precision and accuracy.

(d) Demonstration experiments: when so labelled by the author.

(e) Demonstration experiments by Manitoba standards: Since some authors do not indicate which experiments are demonstrations, but do suggest in the preface that such should occur in the course, a classification is made of experiments which by Manitoba standards would likely be demonstrations. The criteria of choice were:

i) if the time required exceeds ninety minutes and the experiment cannot be done in sections;

ii) if the equipment is costly or rare;

iii) if the techniques involved are too advanced for first year students of about age sixteen;

iv) if experiments call for the handling of considerable quantities of noxious gases. (Few Manitoba laboratories are equipped with individual fume hoods, and often there is a lack of proper ventilation.)

v) if the experiment is considered unsafe for individuals working in pairs in large classes, with minimum supervision.

The first three definitions of experimental types make it clear that the investigator considers them roughly in ascending order of difficulty. Other terms will be defined as they are introduced.

General Procedure

Individual experiments from the eight manuals selected were appraised by the standards indicated, and arranged under generic headings. Thus, experiments like 'Preparation of Oxygen', 'Finding the Molecular Weight of Oxygen', 'Combustion', were all listed under the generic heading, 'Oxygen'. All generic headings (themes) or experiments occurring in fewer than three of the eight manuals were discarded. The remaining themes were arranged in descending order of frequency by manuals to make the Master List. In order to show the degree of uniformity of occurrence of different themes or of different experiment types in the different manuals an analysis was made of the Master List. This is given in Table 1. It will be noted that another category, student-operated qualitative experiments, was devised. This was obtained by subtracting from the total number of experiments the quantitative and demonstration experiments. It was also noted that five of the eight manuals left the choice of demonstration material to the instructor. For this reason subsequent mention of demonstration experiments refers to both demonstrations so designated by the author and demonstrations so judged by the standards proposed.

The Master List

The themes listed are stated in descending order of frequency of occurrence by manuals. The number following the theme indicates the number of manuals out of the eight studied that deal with this theme; for example 'Oxygen (8)' indicates that this theme is dealt with by all eight manuals. Under each theme is a brief description of the purpose of the experimentation included under that theme. Facing each theme is a tabulation of the frequency of occurrence of each type of experiment in all manuals combined, according to the categories defined earlier. Ql = qualitative, C = experiments using complex apparatus, Qn = quantitative experiments, D = demonstration experiments. The frequency of each category refers to the total number of experiments found for this theme. Thus, C 21/32 reads that out of a total of 32 experiments of this theme 21 employ complex apparatus. The range indicates the spread in the number of experiments per manual. Thus, a range of 1-7 means that for this theme the number of experiments found per manual varies from one to seven.

Oxygen (8) Preparation, properties, source of, & in air, molecular weight and gm. mol. volume	Range 1-7 Ql 22/32 C 21/32 Qn 10/32 D 2/32
Hydrogen (8) Preparation and properties	Range 1-3 Ql 12/12 C 9/12 Qn 0/12 D 3/12
Reducing action of hydrogen (8)	Range 1 Ql 7/8 C 7/8 Qn 1/8 D 8/8
Carbon dioxide (8) Preparation and properties	Range 1-2 Ql 12/13 C 9/13 Qn 1/13 D 2/13
Chlorine (8) Preparation and properties	Range 1-2 Ql 9/9 C 9/9 Qn 0/9 D 2/9

Sulphides (8)

Preparation and properties,
usually of hydrogen sulphide

Range 1-3
Ql 10/10
C 10/10
Qn 0/10
D 8/10

Sulphuric acid (8)

Preparation, properties,
tests for sulphates

Range 1-3
Ql 13/13
C 2/13
Qn 0/13
D 2/13

Ammonia (8)

Preparation and properties

Range 1-2
Ql 9/9
C 9/9
Qn 0/9
D 0/9

Nitric acid (8)

Preparation, properties,
test for nitrates

Range 1-2
Ql 11/11
C 11/11
Qn 0/11
D 1/11

Ionization (8)

Conductivity, electrolysis,
degree of ionization

Range 1-5
Ql 15/15
C 7/15
Qn 0/15
D 12/15

Acids, bases, salts (8)

Preparation, properties

Range 1-3
Ql 16/17
C 0/17
Qn 1/17
D 1/17

Titration (8)

Compare strengths of acids, bases,
find per cent acid in vinegar

Range 1-2
Ql 0/12
C 0/12
Qn 12/12
D 0/12

Destructive distillation (8)

Of wood and coal

Range 1
Ql 8/8
C 8/8
Qn 0/8
D 8/8

Reducing action of carbon (7)	Range 0-2 Ql 7/8 C 4/8 Qn 1/8 D 0/8
Hydrochloric acid (7) Preparation, properties	Range 0-1 Ql 7/7 C 6/7 Qn 0/7 D 0/7
Hydrolysis (7)	Range 0-1 Ql 7/7 C 0/7 Qn 0/7 D 0/7
Sulphur dioxide (7) Preparation, properties	Range 0-1 Ql 7/7 C 6/7 Qn 0/7 D 0/7
Hardness in water (7) Preparation, testing, softening	Range 0-1 Ql 6/7 C 0/7 Qn 1/7 D 1/7
Physical and chemical change (7)	Range 0-2 Ql 8/8 C 1/8 Qn 0/8 D 0/8
Halogens (7) Preparation, properties of Cl, Br, I, emphasis on relative activity	Range 0-4 Ql 16/16 C 3/16 Qn 0/16 D 1/16
Food Nutrients (7) Tests	Range 0-1 Ql 7/7 C 0/7 Qn 0/7 D 0/7

Nitrogen (6) Preparation, properties	Range 0-2 Ql 6/7 C 3/7 Qn 1/7 D 4/7
Halogen hydrides (6) Relative activity, stability	Range 0-2 Ql 7/7 C 1/7 Qn 0/7 D 0/7
Water of crystallization (6) Determining percent content	Range 0-2 Ql 0/7 C 0/7 Qn 7/7 D 0/7
Purifying water (6) Distillation, filtration	Range 0-2 Ql 6/6 C 6/6 Qn 0/6 D 0/6
Solutions, hydrated salts (6)	Range 0-4 Ql 16/17 C 0/17 Qn 1/17 D 1/17
Sulphur (6) Forms and properties	Range 0-1 Ql 6/6 C 0/6 Qn 0/6 D 0/6
Equivalent weights (6)	Range 0-2 Ql 0/7 C 4/7 Qn 7/7 D 1/7
Colloids (6) Preparation, properties	Range 0-1 Ql 6/6 C 1/6 Qn 0/6 D 0/6

Carbon (5) Properties	Range 0-1 Ql 5/5 C 2/5 Qn 0/5 D 0/5
Nitrogen oxides (5) Preparation, properties	Range 0-2 Ql 7/7 C 4/7 Qn 0/7 D 0/7
Metals gain weight on heating (5)	Range 0-1 Ql 3/5 C 2/5 Qn 2/5 D 0/5
Elements, mixtures, compounds (5) Contrasting characteristics	Range 0-1 Ql 5/5 C 0/5 Qn 0/5 D 0/5
Flames and flame tests (5)	Range 0-2 Ql 6/6 C 0/6 Qn 0/6 D 0/6
Silver halides (5) Preparation, differences, Behaviour to light	Range 0-3 Ql 8/8 C 0/8 Qn 0/8 D 0/8
Calcium compounds (5)	Range 0-1 Ql 5/5 C 0/5 Qn 0/5 D 0/5
Identifying textiles (5)	Range 0-1 Ql 5/5 C 0/5 Qn 0/5 D 0/5

Equilibrium, reversible reactions (5) Common ion effect, completed reactions	Range 0-2 Ql 10/10 C 1/10 Qn 0/10 D 0/10
Silver group of metals (5) Qualitative analysis	Range 0-1 Ql 5/5 C 0/5 Qn 0/5 D 0/5
Activity of metals (5)	Range 0-1 Ql 5/5 C 0/5 Qn 0/5 D 0/5
Saponification (4)	Range 0-2 Ql 5/5 C 0/5 Qn 0/5 D 0/5
Dyeing and mordants (4)	Range 0-1 Ql 4/4 C 0/4 Qn 0/4 D 0/4
Baking powders (4)	Range 0-2 Ql 4/5 C 3/5 Qn 1/5 D 1/5
Determining a formula (4)	Range 0-2 Ql 0/5 C 0/5 Qn 5/5 D 0/5
Hydrocarbons (4)	Range 0-3 Ql 6/6 C 5/6 Qn 0/6 D 0/6

Esters (3)

Preparation, properties

Range 0-1
Ql 3/3
C 0/3
Qn 0/3
D 0/3

Borax bead tests (3)

Range 0-1
Ql 3/3
C 0/3
Qn 0/3
D 0/3

Hydrogen peroxide (3)

Preparation, properties

Range 0-1
Ql 3/3
C 0/3
Qn 0/3
D 0/3

Types of chemical change (3)

Range 0-1
Ql 3/3
C 0/3
Qn 0/3
D 0/3

Interpretation of the Evidence

A. The Master List.--The Master List represents the common core of experimental material in the manuals surveyed. Each theme in it consists of experiments found in at least three of the eight manuals. Forty-one of its fifty themes are found in five or more of the eight manuals. Or, expressing it in another way, the fifty themes of the Master List represent an average of about fifty-two experiments per manual, of which 4.7 experiments are found in fewer than five manuals. The actual experimental content of a manual varies from 39 to 112 experiments, with five manuals contributing

TABLE 1

PER CENT DISTRIBUTION AMONG MANUALS OF DIFFERENT TYPES OF EXPERIMENTS

Manual	No. of Expmts.	% All Ql	% Student Ql	% C	% Qn	% D	% DI	% All D
6	48	85	60	44	15	2	23	25
5	48	79	65	33	20	0	15	15
4	32	81	72	47	19	0	9	9
7	56	86	75	43	14	4	7	11
3	44	90	80	38	9	0	11	11
10	67	87	78	34	13	0	9	9
11	59	92	78	36	9	0	14	14
13	55	91	82	33	7	2	9	11
Total No. Expts.	409	356	303	155	53	4	49	53
Ave.	51	86	74	39	13	1	12	13
Range in %		79-92	60-82	33-47	7-20	0-4	7-23	9-25

50 ± 6 experiments each. Such an analysis of the Master List and its sources corroborates the belief that it represents a common practice with regard to content that is fairly uniform in all manuals.

What part is this basic core of the total number of experiments listed in the manuals? There is an average of seventy-five chemical experiments per manual, not counting introductory non-chemical experiments. It should be noted that this average includes optional and alternative experiments, and, at least in one manual, a whole series of experiments comprising a unit on qualitative analysis that is not on the Master List. Yet, on the average, the core material makes up about two-thirds of the total content of the average manual.

The authors of the manuals are from nine different states: New Jersey, New Hampshire, District of Columbia, Massachusetts, New York, Ohio, Michigan, Kansas, and Wisconsin. To the extent that these authors reflect the educational trends in their states it may be said that the Master List also indicates the common practice in these states with regard to choice of core material.

The tendency of the core material is to emphasize the interpretation of principles, i.e. group behaviour. Of the fifty themes only eight deal with the preparation and properties of elements, and eleven with the preparation and properties of compounds, as isolated substances requiring the minimum of generalization. The remaining thirty-one themes emphasize principles, e.g. ionization, acids, bases, salts, the halogens, physical and chemical change, solutions, etc.

Table 1 indicates the nature of the experiments included in the Master List, and gives their distribution in each manual. The percent is given as of the total contribution of the manual in question to the Master List. From this table it is seen that the core material consists largely of qualitative experiments. Except for manuals 5 and 6, the distribution of different experiment types if fairly uniform for different manuals. In manuals 5 and 6, 15% and 25%, respectively, of core experiments were judged to be demonstration experiments, and only 65% and 60%, respectively, were student-operated qualitative experiments. The implication is that these two manuals set a higher standard of performance. The greatest uniformity of distribution is found in simple student-operated experiments of the qualitative type, and the least in quantitative experiments.

Certain criteria suggest themselves as a result of the common practice surveyed thus far.

1. Laboratory manuals are commonly of the fill-in type.

2. The purpose of each experiment must be clearly indicated.

3. Apparatus should be illustrated with large, clear, two-dimensional diagrams.

4. Laboratory experiments should emphasize the interpretation of principles.

5. The majority of experiments will be student-operated qualitative experiments.

6. Experiments will involve only elementary skills.

7. Quantitative and demonstration experiments should constitute only about 10-15% of the laboratory course each.

The second phase of this survey involves studying each manual as a whole. Each manual was examined for the following:

(a) The stated function of laboratory work.

(b) The manner of introducing the new student to laboratory work.

(c) The extent of theoretical review for an experiment.

(d) The extent of guidance with respect to the method of performance in an experiment.

(e) The freedom of choice for instructor or student.

The results of this phase of the survey are summarized in the succeeding paragraphs; greater detail is available in the original report.

B. The Stated Function of Laboratory Work.--All manuals except one (manual 5) state the function of the laboratory. Such statements are brief. Half of the authors (manuals 3, 4, 7, 10, 13) specifically stress individual performance. The remainder indicate in other ways, such as in instructions to students, that individual performance is sought. The lack of specifically labelled demonstration experiments also suggests this stress. Such stress of individual performance is hardly a significant discovery, since a course favouring teacher demonstrations would not likely make use of student manuals. This student participation may be on a group basis, since only manual 4 stresses the acquisition of laboratory skills. The emphasis of all manuals is on some manner of participation, on careful observation, and on clear thinking. Manual 6 aims at the understanding of everyday applications. Manual 13 emphasizes the understanding of principles. The scientific method of thinking is emphasized by all.

In conformity with common practice, the criterion for stating laboratory function might emphasize student participation, careful observation, and clear thinking according to the pattern of the scientific method.

C. Introduction to Laboratory Work.--Introduction to the laboratory work by means of instructions and non-chemical exercises is common practice. Such introduction consists of fairly uniform steps. The first step in the introduction is to prescribe the general attitude the student is to adopt to assure success in his laboratory ventures. The scientific attitude is stressed. Other general attitudes that receive emphasis are: self-reliance; preliminary preparation to understand the background, the purpose, and the plan of the experiment; following instructions without deviation; and recording promptly.

The prescription of general attitude is usually followed by rules that favour safety and good housekeeping. There is no sharp distinction between these two sets of rules, as one complements the other. Three of the manuals also provide First Aid rules concerning minor cuts, scalds, burns, poisoning, fainting, and shock. Two manuals (3, 13) emphasize techniques by describing common laboratory techniques and precautions in handling apparatus. All manuals then have from one to nine non-chemical introductory exercises in which the student becomes familiar with common laboratory skills. These generally deal with: use of the bunsen burner, measuring weights and volumes, handling glass tubing, filtering, and heating liquids.

In constructing a new manual the introduction to laboratory work, if it is to conform to common practice, should include the following steps:

(a) Prescription of general attitude.
(b) Rules for safety and good housekeeping.
(c) Description of common laboratory techniques.
(d) Non-chemical introductory experiments.

D. Theory Overview preceding Experiments.--There is considerable variation in the practice of presenting theory overviews, both between different manuals and between different experiments in the same manual. Two manuals (4, 13) provide none, manual 6 has extensive and thorough overviews, while manual 11 has such a complete articulation of theory and experiment that the manual is only part of a chemistry workbook.

Most manuals give little overview in the case of single, unique substances, e.g. preparation of oxygen, hydrogen, chlorine, etc. The overview, if present, is found preceding experiments that illustrate a principle (e.g. ionization) or group behaviour (e.g. the halogen hydrides).

Theory overviews are usually condensed into from four to six lines of print. The exception is some uncommon principle, like that of pH, which may receive one-half to three-quarter page of overview.

It may be concluded as a criterion for the construction of a manual that theory overviews will be brief, that they should precede experiments involving the illustration of some principle, and that they are not essential for experiments dealing with unique substances.

E. Presentation of Method.--The manuals are fairly uniform in describing with considerable detail the manner of performance. Some manuals add to this, besides a line diagram of apparatus, pertinent hints on setting up the apparatus.

Greater variation occurs in emphasizing precautions. These fall into two categories, those that are concerned with safety, and those that are concerned with good performance. Three manuals (6, 7, 13) either neglect to stress precautions in handling inflammable or noxious gases, or neglect to stress precautions such as: sweeping air out of assembled apparatus, manner of heating small quantities of liquids, mixing of water and acids.

The prescription of quantity shows the greatest lack of uniformity, and is the least well-done according to the standards suggested.[1] It is common practice in

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[1] See pages 3-4 of this study.

the manuals surveyed:

(a) To give some indication of quantity.

(b) With the exception of liquid volumes, to state whether the quantity is approximate or precise. (There is variation in this respect even within a manual, lapses being fairly frequent where no mention is made of the degree of precision of measure.)

(c) To state most weight approximations in metric units. (The exception to this is that frequently small quantities are indicated in terms more familiar to the average beginning student, e.g. drops, or inches in a test tube or flask.)

(d) To state volumes without specifying either the degree of precision or the manner of measurement.

Illustrations are common, consisting generally of clear two-dimensional line diagrams that avoid irrelevant detail.

This phase of the study suggests the following criteria:

1. The manner of performance should be described in considerable detail.

2. Precautions concerning safety and good performance should be included in the prescription of method.

3. Prescription of quantity should indicate the degree of precision, and, in dealing with liquid volumes, the manner of measurement. Approximate quantities should

be stated in terms that are familiar to the average student, e.g. drops, inches in a test tube, etc.

F. Freedom of Choice.--Freedom of choice is indicated in the manuals by the fact that (a) the author mentions it, (b) the author prescribes basic experiments but provides extra ones, (c) the author encourages omissions, (d) the author fails to designate which are demonstration experiments, yet suggests that some experiments are demonstrations.

The evidence is abundantly clear that another criterion of the new manual must be that it will have sufficient experimental material to permit local choice of experiments, and of demonstrations.

This chapter has revealed criteria which have reference to content, the stated function of the laboratory, the manner of introducing laboratory work, theory overview, prescription of method, and freedom of choice. These criteria, along with others developed in earlier chapters, will now be summarized, and, if it is considered necessary, modified to meet the Manitoba situation.

CHAPTER VI

SUMMARY OF THE CRITERIA USED IN THE CONSTRUCTION OF A CHEMISTRY LABORATORY MANUAL

Orientation

The criteria considered necessary for the construction of a chemistry laboratory manual have been developed. They may be classified thus:

A. Criteria based on the aim of chemistry education:

1. The basic aim of education in Canada and the United States is training for participation in a democratic society.

2. This training is brought about by changing or cultivating skills, habits, attitudes, and ideals.

3. At the secondary school level the emphasis of this training is on desirable attitudes.

4. Science education, through its emphasis on the scientific method and attitude, contributes to this emphasis on desirable attitudes.

5. Education in the scientific attitude may be directed so as to emphasize its social implications.

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[1] See pp. 24 ff. of this study.

History shows that this emphasis is necessary.

6. Chemistry presents phenomena that readily permit practice in discriminating enquiry and belief at the secondary school level; discriminating enquiry and belief are functions of a socially centred scientific attitude.

7. Discriminating enquiry and belief require abstract thinking. In order to emphasize abstract thinking, the aims of chemistry education are stated at three increasingly abstract intellectual levels:

(a) An understanding of the composition of matter and the changes it undergoes, and the manner in which this knowledge aids man in the control of his environment.

(b) A revelation of the organization in the changes of the composition of matter.

(c) An appreciation of the elusive nature of cause, and of truth, and an appreciation of the power of directed imagination.

Most chemistry lessons involve at least the two lower levels of approach. It must be the aim to teach at the highest intellectual level that the situation will permit.

8. In laboratory chemistry the emphasis will be on discriminating enquiry (which implies intellectual

honesty), and on the ability to generalize (which requires abstract thinking).

B. Criteria based on the nature and function of laboratory work:[1]

1. Chemistry laboratory work emphasizes the application of the scientific method to first-hand, concrete, raw data, in order to solve a problem or to answer a question.

2. The problems or questions to be dealt with should be related to some generalization of chemistry, and should help to clarify it.

3. A second function of laboratory work is to broaden the sensory impact of a learning situation, in order that better assimilation may result.

4. The acquisition of laboratory skills is a means towards the ends just stated.

5. Laboratory work should employ the simplest procedures and skills that will suffice to illustrate or clarify a problematic situation.

C. Criteria based on the nature of guidance:[2]

1. Pupils should be encouraged to share in the

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[1] See page 34 of this study.

[2] See pages 43-44 of this study.

stating of a problem, and in the planning of its solution.

2. At the secondary school level the interpretation of data should be a co-operative effort by pupil and teacher.

3. To maintain interest and attention the majority of the laboratory experiments should be performed by the pupils.

4. The choice of demonstration experiments should be left to the teacher.

5. The laboratory manual should give fairly complete guidance. It should be augmented by class discussions that will develop the problem, suggest means for its solution, and weight the evidence.

D. Criteria based on common practice in some current chemistry laboratory manuals:[1]

1. Laboratory manuals are usually of the fill-in type.

2. The purpose of each experiment must be clearly stated.

3. Apparatus should be illustrated with large, clear, two-dimensional diagrams.

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[1] See pp. 61 ff. of this study.

4. Laboratory experiments should emphasize the interpretation of principles.

5. The majority of the experiments should be student-operated qualitative experiments.

6. Quantitative and demonstration experiments should constitute only about 10-15% each of the total laboratory course.

7. Experiments should involve only elementary skills.

8. The common statement of function of laboratory work emphasizes student participation, careful observation, and clear thinking according to the pattern of the scientific method.

9. Introduction to laboratory work includes the following steps:

   (a) Prescription of general attitude.
   (b) Rules for safety and good housekeeping.
   (c) Description of common laboratory techniques.
   (d) Non-chemical introductory experiments.

10. Theory overviews should be provided for at least those experiments involving the interpretation of some principle. They should be brief.

11. The manual should prescribe the manner of performance in some detail.

12. Precautions concerning safety and good performance should be included in the prescription of method.

13. Prescription of quantity should indicate the degree of precision of measurement, and, in dealing with liquid volumes, the manner of measurement. Approximate quantities should be stated in terms that are familiar to the average student, e.g. drops, inches in a test tube, etc.

14. The content of the manual should be such as to permit some latitude of choice with respect to both teacher demonstrations and student-operated experiments.

Manual Structure

The survey in the preceding chapter reveals a rather uniform manual structure. It generally consists of:

1. Suggestions to the instructor.
2. Suggestions to the student.
3. Safety and good housekeeping hints.
4. Illustrations of common apparatus assemblies for later reference.
5. Description of simple techniques.
6. Non-chemical introductory experiments.
7. The main body of experimental work.

Manual Content

It might be supposed that, having constructed a Master List of content that reflects a good measure of common practice, it would be necessary only to materialize this into concrete experiments. However, several criteria arise which demand that this list of average content be modified to fit the needs of a particular situation, perhaps thereby suffering mutilations and additions.

1. Laboratory work must conform closely to the classroom studies which it is designed to complement.

> This is of greater importance than that the content of the laboratory course reflect closely the content and nature of the Master List. The manuals on which the Master List is based appear to provide for a one-year course; the local situation calls for a two-year course, of which the second year is under consideration in this study. The content of the Grade XII manual would be the total experimental content of the two-year course minus those experiments already performed in Grade XI, providing always that such omissions do not violate the principle that laboratory must closely parallel classroom work. Thus it will happen that certain study-areas touched on in Grade XI are again encountered in Grade XII. In such cases it becomes a

judicious choice which Grade XI experiments to repeat in Grade XII, and in what manner. Then too, a two-year course may cover a wider area of study than a one-year course. For this reason it may be necessary to include experimental topics which receive little or no mention in the Master List.

2. A laboratory course must take cognizance of local limitations in laboratory equipment.

In the average Manitoba high school laboratory equipment is usually of a simple nature, frequently not including precision balances or fume cupboards. Thus it is impractical to insist on quantitative experiments that require delicate weighings, or preparations that involve quantities of noxious gases.

Table 2 lists the broad experimental themes of the Master List. It shows which of these themes are currently dealt with in the existing Grade XII laboratory course, and which of these themes will be dealt with in the laboratory course as outlined in the proposed manual.

Table 3 shows the general content of the current Grade XII chemistry course by chapter headings from the text,[1] together with experimental work suitable for these areas of study and which will be included in

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[1] McPherson, W. et. al., Introduction to College Chemistry (Boston: Ginn and Co., 1946).

the proposed manual.

3. A laboratory course must have its experiments arranged in the order that will match the sequence of classroom approach.

It will be noted from Table 3 that the subject matter in the current text has been rearranged for teaching purposes. The reasons for this rearrangement bear reviewing, since they apply to the order of experimentation as well. Yet the order of experimentation must be sufficiently flexible that it may be adapted to a different order of approach.

Sulphur and its compounds, and ammonia, form the subject matter for the earliest studies in Grade XII chemistry because they provide a detailed area of study in descriptive chemistry. They do this without drawing heavily on basic generalization, which are yet to be studied. At the same time they permit several examples of industrial applications, which provides a concrete approach to the study. They are suitable for early laboratory experimentation, and provide numerous exercises in equations. They provide sufficiently difficult situations whose clarification demands a deeper

TABLE 2

THEMES OF MASTER LIST COMPARED WITH THEMES OF THE PRESENT AND THE PROPOSED LABORATORY COURSES IN GRADE XII CHEMISTRY

Themes of the Master List	Present Course	Proposed Course
Oxygen (8)		
Hydrogen (8)		
Reducing action of hydrogen (8)		
Carbon dioxide (8)		
Chlorine (8)	x	x
Sulphides (usually H_2S) (8)		
Sulphuric Acid (8)	x	x
Ammonia (8)		x
Nitric acid (8)	x	x
Ionization (8)	x	x
Acids, bases, salts (8)		x
Titration (8)	x	x
Destructive distillation (8)		
Reducing action of carbon (7)	x	x
Hydrochloric acid (7)		x
Hydrolysis (7)	x	x
Sulphur dioxide (7)	x	x
Hardness in water (7)	x	x
Physical and chemical change (7)		
Halogens (7)	x	x
Food nutrients (7)		
Nitrogen (6)	x	
Halogen hydrides (6)	x	x
Water of crystallization (6)	x	
Purifying water (6)	x	
Solutions, hydrated salts (6)		x
Sulphur (6)		x
Equivalent weights (6)	x	x
Colloids (6)	x	x
Carbon (5)		
Nitrogen oxides (5)	x	
Metal gains wt. on heating (5)		
Elements, mixtures, compounds (5)		
Flames and flame tests (5)	x	x
Silver group analysis (5)	x	x
Silver halides (5)	x	x

TABLE 2--Continued

Themes of the Master List	Present Course	Proposed Course
Calcium compounds (5)	x	x
Identifying textiles (5)	x	
Equilibrium reactions (5)	x	x
Activity of metals (5)		
Saponification (4)	x	x
Dyeing and mordants (4)	x	
Baking powders (4)		
Determining a formula (4)		x
Hydrocarbons (4)		
Esters (3)		
Borax bead tests (3)	x	
Hydrogen peroxide (3)		
Types of chemical change (3)		
<u>The following themes are not in the Master List</u>		
The gas laws	x	x
The solution laws	x	x
Heats of neutralization or sol'n.	x	
Phosphorus	x	
The iron compounds	x	x
Diffusion of gases		x
Gram molecular volume and/or wt.		x
Commercial processes (Ostwald and Solvay processes)		x

TABLE 3

OUTLINE OF GRADE XII CHEMISTRY COURSE WITH PROPOSED LABORATORY WORK TO ACCOMPANY IT

Page	Chapter	Subject Matter
254	19	Sulphur and its compounds. ..Preparation of the allotropes of S. ..Preparation and properties of SO_2. ..Preparation and properties of H_2SO_4.
287	21	Ammonia. ..Preparation and properties of NH_3.
113	10	Atomic structure and its implications.
293	21	Nitric acid, nitrogen oxides. ..Preparation and properties of HNO_3. ..Oxidation-reduction. ..The Ostwald process.
63	6	The gas laws; the molecular theory. ..Demonstrating Boyles Law. ..Demonstrating Graham's Law of Diffusion.
83	7	Hydrogen peroxide; laws of definite and of multiple proportions.
103	9	Valence, Molecular and Atomic Weights, Formulae, Equations, Equation problems. ..Determining molecular wt. of CO_2. ..Determining the formula of tin oxide.
173	14	Solutions. ..Factors affecting solutions; hydrated salts; types of solution. ..Effect on boiling point of concentration.
186	15	The theory of ionization, salts, neutralization. ..Conductivity in solution; strength of acids. ..To show difference between atom and ion. ..Titration.

Page and chapter numbers refer to the prescribed text; see page 76 of this study.

TABLE 3--<u>Continued</u>

Page	Chapter	Subject Matter
200	16	Chlorine, sodium, hydrochloric acid, sodium hydroxide, etc. ..Preparation and properties of chlorine. ..Preparation and properties of HCl.
276	20	Rates of reaction, equilibrium. ..Chemical equilibrium; common-ion effect.
221	17	The periodic classification.
240	18	The halogens. ..Study of the elements, their hydrides, and their silver salts.
254	19	Review of the sulphur family as such.
305	22	Phosphorus family. ..Hydrolysis.
320	23	Silicon and boron compounds.
338	24	Colloids. ..Preparation and properties of colloids. ..Preparation and properties of emulsions.
<u>Organic Chemistry</u>		
363	26	Hydrocarbons, petroleum products, etc.
376	27	Fuels and flames. ..Flame tests for metal ions.
390	28	Carbohydrates, alcohols, ethers.
405	29	Organic acids, coal tar compounds. ..Saponification.
<u>The Metals</u>		
427	30	Alloys, metallurgy, metallic salts. ..Reduction with carbon. ..Analysis of the silver group of metals.
451	32	Some very active metals: Na, K, Ca. ..The Solvay process. ..Preparation of calcium compounds.
489	34	Water treatment. ..Testing for, and removing hardness in water.
510	35	Iron, its metallurgy, its compounds. ..Preparation and properties of ferrous and ferric salts; their oxidation and reduction.

and more abstract study of basic principles.

The theory of atomic structure is studied next, as a natural consequence of the need to explain the chemical changes already studied (just as in Grade XI it arises from the study of oxygen and hydrogen). At an early stage the student is familiarized with the most basic principle of chemistry. It may be argued that such a principle should be left till much later, when the student has been more adequately induced into the level of abstract thinking. However, popular interest in subatomic science and its products is so great that the early study of this theory is more apt to stimulate than to discourage the student interest. It provides early opportunity to work at the highest intellectual level of approach, to demonstrate the limited accomplishments of science and the elusive nature of truth.

Nitric acid and the oxides of nitrogen are studied next because they afford opportunity to illustrate the electronic principle of oxidation-reduction.

Although the gas laws and the molecular theory are frequently omitted from chemistry courses they are included in the Manitoba course. The theory provides the most basic principle for explaining physical change at the high school level. As dealt with at this level, it is comparatively simple, and is taught at this stage to offset any possible student fears concerning the difficulty of theory that may have arisen through the study of the modern atomic theory. Also, the presentation of these laws permits early and simple mathematical problem work.

Having acquired the theoreticl background to understand valence, formulae, and chemical change, the student is now ready to undertake practice in the chemical shorthand: the writing of correct formulae and the balancing of equations. He is also in the position by now to glean from the formula or equation all the implied information. It follows that he can now undertake the solution of problems based on chemical change. He has till now had four changes of pace: from descriptive chemistry to theory to shorthand to problems. This is considered good pedagogical practice.

The remaining arrangement of subject matter (see Table 3) appears pedagogically sound, and is not tampered with except that the study of equilibrium is undertaken immediately following the study of hydrochloric acid and the theory of ionization. This places it where it may have immediate application, and so that it precedes the study of the halogens, where its application will again be desirable.

CHAPTER VII

EVALUATION OF THE PREPARED MANUAL IN THE LIGHT OF THE CRITERIA DEVELOPED

Evaluation

Criteria based on the aims of chemistry education have been stated.[1] They call for a socially-centered scientific attitude requiring abstract thinking. Yet the directives of a laboratory manual do not indicate them or their subtleties. The final form, the detailed pattern of the educational effort must be built by the teacher around the somewhat stark skeleton of directives found in the laboratory manual. Yet this skeleton must permit such building. The prepared manual[2] emphasizes two attitudes: intellectual honesty and scientific attitude, which are inseparable. It emphasizes the first by demanding an accurate report, and by suggesting a sheet of Extra Conclusions on which any deviations from the expected results may be discussed and perhaps explained. It emphasizes the second attitude by stressing abstract intellectual effort

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[1] See page 69 of this study.

[2] See Appendix.

through the use of 'how' and 'why' questions.

The criteria based on the nature and function of laboratory work[1] have served in part to channel this investigation along a prescribed course. But their influence will be seen in the manual. It emphasizes experiments designed to clarify principles, but it also uses the laboratory for object or 'thing' teaching.[2] Skills are not considered of primary importance. It keeps apparatus, procedures, and skills simple.

The criteria developed from an examination of common practice in current laboratory manuals[3] have been adopted almost completely. The object of each experiment is stated and there is a brief review of pertinent theory. The body of experimental work is preceded by an introductory section that prescribes general attitude, instructions concerning safety, housekeeping, laboratory techniques, and common laboratory assemblies. The chemical experiments are preceded by three introductory experiments that give exercise in the commonest laboratory techniques.

Laboratory assemblies of common equipment are not prescribed; rather, it is expected that the stu-

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[1] See Page 71 of this study.

[2] See also page 30 of this study.

[3] See page 72 of this study.

dent select these from the list of common apparatus assemblies, relying on his knowledge of the reagents involved and of their properties. This, it is hoped, will emphasize a discriminating attitude, and lessen somewhat the danger of 'cook-book' performances.

The method of performance has been prescribed in greater detail than that found in the current laboratory manual in use in Manitoba schools. Care has been taken to state whether a prescribed quantity is approximate or precise, and, in the case of volumes, to indicate the manner of measurement. Approximate quantities are usually expressed in terms familiar to the student.

As shown in Table 2, the criteria designed to fit laboratory content in common practice to the local situation[1] have considerably mutilated the Master List. It is hoped that this will result in closer harmony between laboratory and classroom work, and in a sounder pedagogical approach. The Master List has served as a temporary guide towards a body of content that resembles it only remotely.

Common practice is followed by including in the manual more experiments than will likely be performed by the average class, and by not designating

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[1] See pp. 74 ff. of this study.

demonstration experiments. This permits freedom of choice to fit the local situation.

Recommendations

The prepared manual is an outgrowth of the criteria developed by this study. Whether it is a good fit remains to be determined. Only after a period of extensive use will it be possible to investigate its merits fully.

This study has stirred up a number of questions which either it has not attempted to answer, or it has not been able to answer within the limits of the set problem. Investigation is necessary, for instance, in the following areas:

1. Which has greater educational value, the individually written report or the fill-in type of report? Can the former be so revised that it gains the merits of the latter and loses its present defects? The prepared manual attempts to bridge the two forms by encouraging student discussion and student Extra Conclusions.

2. Will the attempt to have the student deduce the proper apparatus assemblies cause more confusion than its merits warrant? Only actual practice will reveal this. Several weeks of practice suggest that its merits outweigh any resulting confusion.

3. Is the choice of experimental material suitable? If students are apt to obtain inconclusive data for an experiment, then that experiment would be better replaced by another. Again, only actual laboratory practice will reveal such weaknesses fully. More experimental material seems necessary for the introductory organic section of the course. Difficulties in compiling or obtaining source material, together with current limitations of laboratory equipment in the average Manitoba high school, have kept this material at a minimum.

4. Should the ultimate objective be a combined workbook-manual? The integration of theory and practice could be more complete, and it would seem possible to stress still more the thinking, rather than the doing aspect of laboratory work. This may not be possible until schools have workrooms where, under the same teacher, laboratory and classroom exercises are no longer distinguished from each other.

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Laboratory Manuals

In current use in Manitoba:

Harder, Frank. Outline of Laboratory Experiments for the Province of Manitoba, to be performed as part of the prescribed course in Chemistry II (XI). Altona, Man.: D. W. Friesen and Sons, 1951.

Moore, Andrew. Outline of Experimental Work in Practical Chemistry. Winnipeg, Man.: Manitoba Text Book Bureau, reprinted 1936. (Two pamphlets, one for each of Grades XI and XII. Only the Grade XII pamphlet is at present authorized.)

In current use elsewhere, surveyed for common practice:

Ref. No.

13 Bayles, Ernest E. et al. *Laboratory Experiments and Exercises to Accompany Basic Chemistry.* New York: Macmillan Co., 1947, 1st edition.

11 Fowler, George Winegar. *Workbook in Chemistry.* New York: Ginn and Co., 1949, 3rd edition.

6 Garrett, Alfred B., et al. *Introductory Chemistry for the Laboratory.* New York: Ginn and Co., 1943, 2nd edition.

4 Hogg, John C., et al. *Laboratory Manual to Chemistry.* New York: Van Nostrand Co. Inc., 1945, 1st edition.

5 Holmes, Harry N. *Laboratory Manual of General Chemistry.* New York: Macmillan Co., 1949, 5th edition.

3 Price, William E. *Laboratory Chemistry.* New York: World Book Co., 1947, 1st ed.

7 Rawlins, George M. *Chemistry in Action in the the Laboratory.* Boston: D. C. Heath, 1948, 1st edition.

10 Weaver, Elbert C. *Laboratory Introduction to Chemistry.* New York: McGraw Hill Book Co., 1947, 1st edition.

APPENDIX

TO THE INSTRUCTOR

The Department of Education has prescribed the minimum requirements of the Grade XII Chemistry Laboratory course. Within the framework of this requirement it is your responsibility to select wisely, in order to meet the needs of your particular situation, the individual and demonstration experiments that will constitute your laboratory course.

Laboratory work is of little value if it fails to clarify problems encountered by the student in the course of his regular chemistry classes. Where possible, a generalization should develop from the laboratory exercise. The exercise should require mental effort on the part of the student, not merely manipulation. Close guidance may defeat this requirement. In order to maintain a balance between guidance and individual effort it is advisable to begin each experiment with a class discussion. During this discussion the student is encouraged to suggest means by which the purpose of the experiment may be achieved. He should, in most cases, be able to suggest the apparatus assembly that will be necessary for the development of the experiment. He should be able to suggest some of the points at least for which he will be on the alert. When the experiment is begun it would be wise for the instructor to move about the room to advise on mechanical procedures; these are of secondary importance and should not be permitted to befog the main issues. Partway through the experiment it may be wise to stop proceedings for a brief discussion on the observations. This is particularly important if the experiment does not proceed as expected. Interim conclusions may be developed at this stage. Reports should be written up as the experiment proceeds. Each student should be encouraged to insert a blank sheet at the end of each experimental report on which he should state extra conclusions. These may include definitions of new terms that he has encountered, relevant observations that have not been touched on by the manual, and explanations. He must be encouraged to report honestly what he observes, even if it is not what either he or his instructor expected. If the time permitted he would normally check his findings by repeating the experiment. Since this is often impractical he should state in his Extra Conclusions what he expected to get, and, where possible, he should discuss the probable causes of his deviation. If this plan is followed, there is opportunity for individual initiative, and less likelihood of 'cook-book' performance in the laboratory.

The instructor will note that in each preparation the student is expected to review the sheet of Common Apparatus Assemblies, and from his knowledge of the chemical reaction and the properties of the substances involved, to deduce the proper equipment he will need to assemble. This may be developed during class discussion, with the possibility of variation from the suggested equipment.

It will be further noted that several experiments consist of sections which probably can not all be performed in one laboratory period. These may be done in different periods, and, in effect, rate as separate experiments.

TO THE STUDENT

Your laboratory work provides you with the opportunity of developing within yourself some of the characteristics of the scientist: complete honesty and accurate,intelligent appraisal of your findings. Honesty means not only that you must refrain from copying, it means alos that you will not let your knowledge of what to expect interfere with your observations. Report what you find-even if you think it is wrong, BUT use a blank sheet as the last page of your report,on which to explain what you expected to get,why you expected it, and perhaps why your results are different. This critical appraisal may be the most important part of your report.

Some people are more alert to notice things than are others;try to develop this characteristic.

Having made a number of observations it is often possible to draw certain general conclusions from them. This is the chief value of making observations: to learn some principle from them.

To make sure that your report is honest and accurate write it as you do the experiment,not at some later date.

In order to arrive at a successful conslusion to a laboratory experiment:

1. Make sure that you understand clearly what the purpose of the experiment is, and do not lose sight of this. Prepare yourself by reading for understanding any theory or description that has a bearing on the experimental problem.
2. Do not deviate from the instructions of the experiment without first consulting your instructor; dangerous unforeseen situations might arise.
3. Even when working with a partner,take a personal part in the experiment. Be alert. Be self-r reliant.
4. Report your work immediately,even if only in a rough form.

GENERAL LABORATORY PRECAUTIONS

Equipment

1. Apparatus and surroundings should be kept clean. Before using any apparatus be sure it is clean.
2. When finished with your equipment dismantle it, wash it, sort it out, and return it to your instructor.
3. Leave your workbench clean and tidy.

Reagents

1. Reagent bottles must not be kept at your desk. Return them to their proper place as soon as you are finished with them.
2. Unused reagents must not be poured back into reagent bottles. For this reason exercise care in measuring out just enough reagent for use.
3. Use small quantities of reagents as specified.
4. While pouring reagents keep the stopper of the reagent bottle between your fingers; do not place it on the table to become contaminated. Replace it when you are finished.
5. Solid reagents may be poured onto a piece of clean paper, and from the paper into the test tube or flask.
6. Read labels carefully.
7. Do not mix reagents other than as specified; common reagents improperly mixed may result in very harmful reactions. There can be danger in a laboratory if you hunt for it.
8. Never mix reagents so that the open container is directed towards you or another person.
9. Handling acids:
   (a) Concentrated Acids: (especially sulphuric acid) If the container into which the strong acid is being poured is wet, add the acid slowly, swirling the contents as you do so. Enough heat might otherwise be generated so that the steam from it could blow the contents out of the container. NEVER POUR WATER INTO CONCENTRATED SULPHURIC ACID; ALWAYS MIX BY POURING THE ACID SLOWLY INTO THE WATER WITH CONSTANT STIRRING OR SWIRLING.
   (b) Heating Acids: Especially with concentrated acids, it is wise to place the acid container into an empty sandbat or other metal dish and then to heat the dish. If the container should crack you are not so likely to be spattered with concentrated hot acid.
   (c) Spill: Wipe up any spills immediately with a wet rag. If you get acid on your skin rinse it off with plenty of cold water. If it is concentrated acid you may follow this up with a risne of sodium bicarbonate solution. (The instructor will have this handy).

If you suspect that you have acid on your clothing soak the spot with sodium bicarbonate solution until there is no more fizzing. Strong bases like sodium hydroxide should be washed off with water,then neutralized with boracic acid solution.

(10) Never bring your nose close to the opening of a container to smell the fumes. Rather,waft them towards your nose with your open hand.

Waste Materials

1. Liquid wastes may be run down the sink. Strong acids particularly should be flushed down with copious amounts of water,so that they may be diluted to a harmless concentration in the plumbing.
2. Solid wastes should be emptied into the crock provided.
3. Sinks should be kept free of papers,splinters, or any other solid wastes.

General Techniques

1. When not using a burner keep it turned low.
2. Never leave a bunsen burner under a reaction or under a boiling solution when you must leave your experiment unattended.

LABORATORY TECHNIQUES

Boiling

1. In a test tube: Use a gentle non-luminous flame. Avoid heating the portion of the tube above the liquid level. Make sure the outside of the test tube is dry. Hold the test tube with a test tube holder or a folded strip of paper. Pass it through the flame back and forth at an angle so that the flame strikes it from the side rather than from directly underneath. When it starts to boil wait until the frothing subsides before again passing it through the flame.
2. In large containers: Use thin-walled vessels, dry on the outside. Beakers,flasks,etc. are placed on a wire gauze held on a ring stand. Use a gentle non-luminous flame. No boiling vessel should be left unattended. Do not boil to dryness,and avoid direct flames against the part of the vessle above the liquid level. To prevent bumping,especially where the liquid depth is considerable,place in the liquid small pieces of porous earthenware,or short lengths of capillary tubing.

Filtering

Fold the filter paper into quarters. Open it to make a paper cone. Fit the cone into the filter funnel. If it is not a close fit adjust your fold. Unless otherwise instructed,wet the filter paper so that it clings to the funnel walls. The fewer air spaces there are between paper and walls the more rapid your filtering will be;there should be no air spaces reaching from top to bottom of your filter paper. If possible let your solution stand to settle,and drain off the clear liquid into the filter first;this is called decanting. Wash the solid portions in last. Never let the solution level in the funnel rise above the edge of the filter paper.

Measuring Volumes

A liquid has a curved surface or meniscus,usually higher at the edges than at the centre. Always measure from the bottom (i.e.centre) of the meniscus. Take readings with the instrument at eye-level,since errors will result from reading at a slant. Coarse measurements of large volumes can be made by means of graduated cylinders. More precise measurments can be made either by means of a pipette or a burette.

When using a pipette suck at the top opening to draw the liquid into the instrument well past the uppermost calibration. Then quickly place the thumb or finger over the top opening. By admitting air past the thumb the exact amount of liquid can be run out. If a harmful liquid is being measured it should be drawn into the pipette by means of a rubber bulb.

When using a burette fill it with liquid well above the top calibration. See that there is no liquid on the outside to drip down. Then run out enough liquid to fill the tip or nozzle both above and below the clamp or tap. Adjust the clamp so that no liquid is leaking out of the nozzle. Now adjust the liquid level in the burette so that the meniscus is at the zero calibration. You are now ready to deliver any exact volume of liquid.

In most pipettes or burettes the calibrations do not extend to the bottom. In such cases do not drain the liquid past the lower calibration. Suppose that the bottom calibration reads 10 ml. This means that you have run off 10 mls. when the liquid level has dropped from the zero mark to the 10 ml. mark;any liquid below this level is not to be delivered.

Weighing

Since the nature and delicacy of balances will vary, your instructor will demonstrate the proper use of the balance in your laboratory. In general

1. Never place hot objects on the balance pan.
2. Protect the pans from water and active reagents. Always use a weighed container or filter paper on the pan to hold your sample to be weighed.
3. Always lower the balance pans on to their rests before placing on them the object to be weighed.
4. The object to be weighed should rest on the left pan.

Glass tubing

1. To cut a given length: Make a scratch where you wish to break the tubing. Grasp the tube at the scratch, one hand on each side of the scratch and with the thumbs together at the scratch. Pull and bend sideways at the same time.

2. To fire-polish: The end of the glass tubing may have a sharp edge. Hold it in a hot blue flame while rotating the tube until this edge melts down to a smooth edge.

3. To make a bend: Use a fish-tail top to spread the hot blue flame of your burner. Hold the glass tubing lengthwise in the flame so that about two inches of it are heated at the same time. The object is to heat it uniformly all over. To do this, rotate the tubing between your fingers, at the same time moving it back and forth lengthwise through the flame. After a while, by gentle sideways pressure test the glass for softness, but do not attempt to bend it while it is in the flame. When it is soft remove it from the flame and by gentle sideways pressure bend it slowly to shape. If you bend too rapidly, consentrate the heat too much in one spot, or get the walls too soft, the walls will collapse and make a bend whose inside diameter is flattened out.

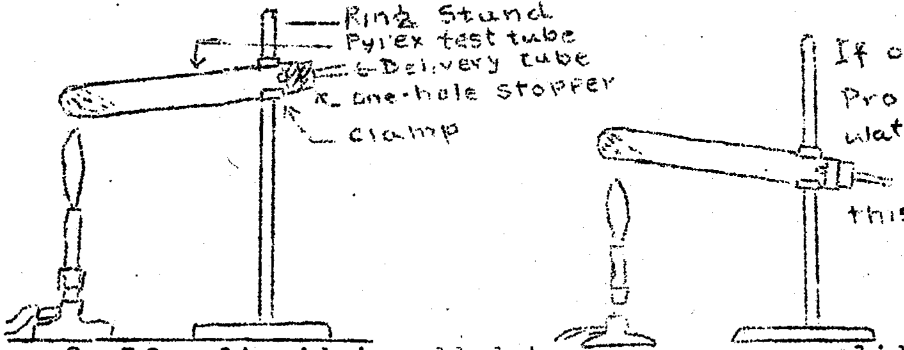
4. To insert glass tubing into a stopper: First wet both the tubing and the stopper. Then gently force the tubing into the hole with a twisting motion. Avoid holding the tubing so that you are pushing on it directly from behind. If it should break the jagged ends might cut your hand or arm. It is better to grasp it from the side, or where possible to pull on it.

COMMON APPARATUS ASSEMBLIES

Laboratory preparations usually involve two stages: the generation and the collection of the substance. In the majority of cases the substance involved is a gas. Knowing what materials will be needed to generate the gas and what its physical properties are, you should be able to deduce which combination of apparatus would serve the purpose of preparing and collecting the gas.

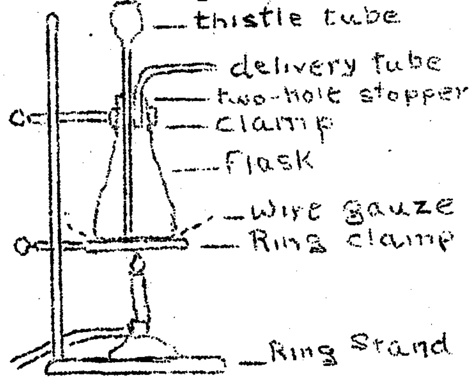
GENERATORS

1. If only solids are being heated:



If one of the Products is water tilt the tube in this manner.

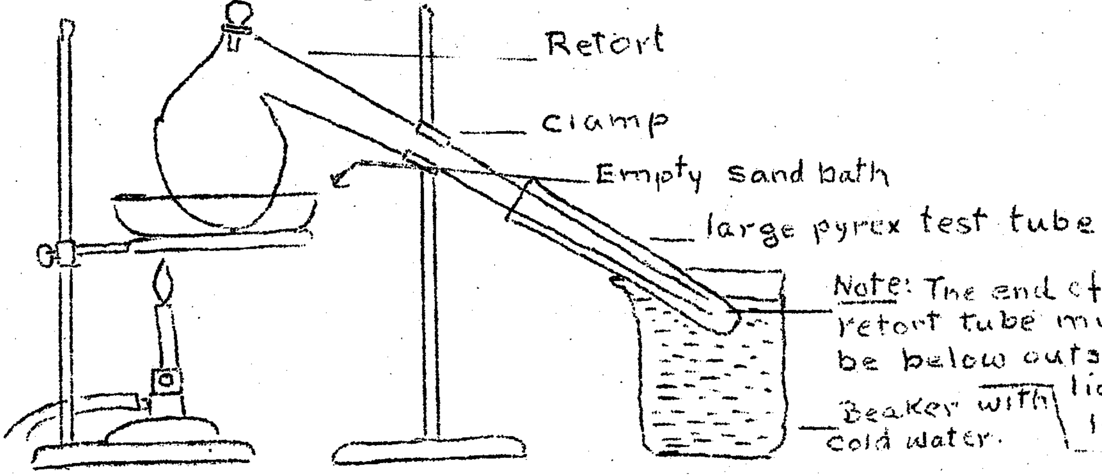
2. If a liquid is added to one or more solids:



Note: The bottom of the thistle tube must be under liquid. This may mean running in some water; use the least amount possible.

When clamping glassware protect it by placing folded paper between glass and clamp.

3. If the reagents or products act on rubber:

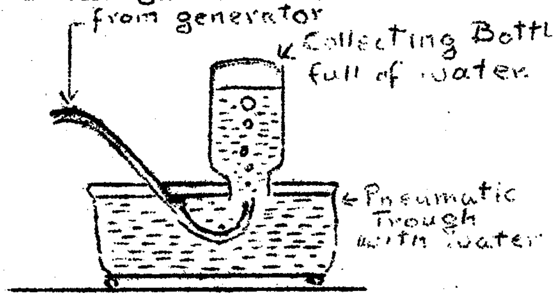


Note: The end of the retort tube must be below outside liquid level

Common Apparatus Assemblies

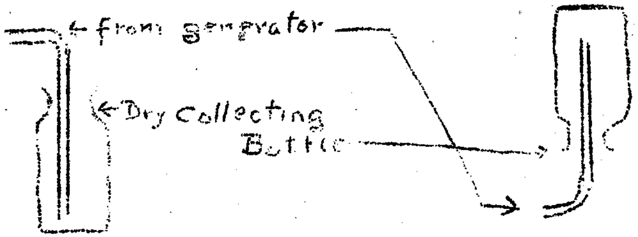
Collecting Devices

1. If the gas is insoluble in water

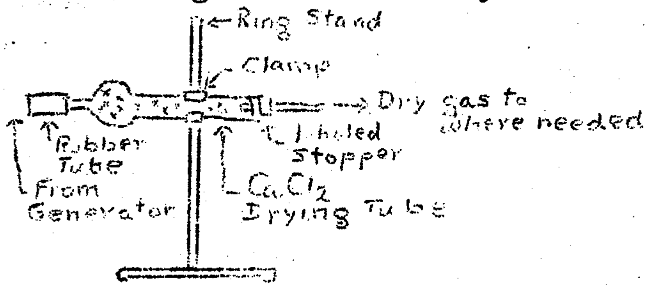


2. If the gas is soluble in water

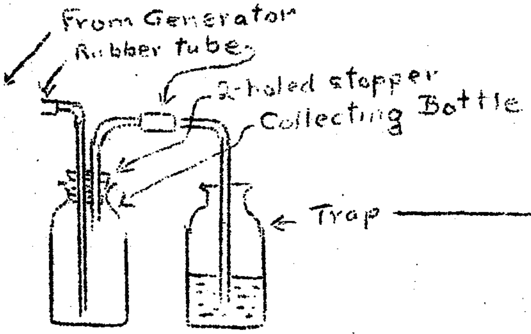
Heavier than air **Lighter than air**



3. If the gas is to be dry



4. If a noxious or unpleasant gas is to be collected without using a fume hood the following method of collection may be employed: (e.g. sulphur dioxide, chlorine).



Note: the 2-holed stopper should fit loosely. When the collecting bottle is full it can then be readily replaced by an empty bottle.

TRAP: For SO_2 it should contain water. For Cl_2 it may contain either strong sodium thiosulphate solution or strong NaOH solution.

TO INSERT GLASS TUBING INTO A RUBBER STOPPER, WET BOTH, GENTLY FORCE TUBING INTO STOPPER WITH A TWISTING MOTION. GRASP TUBING FROM THE SIDE, TO AVOID CUTS IF THE TUBING SHOULD ACCIDENTALLY BREAK.

PRELIMINARY EXERCISES

1. THE BUNSEN BURNER

Apparatus and Materials: Bunsen burner, porcelain dish, crucible tongs, splinters, cardboard.

Procedure: Examine all parts of the burner, dismantling where possible. Clean them while they are apart and reassemble. Make a cross-sectional diagram of the burner. Turn on the gas and light the burner. Manipulate the air vents and the adjustment at the base of the burner (or the gas tap), to discover the effect of each on the nature of the flame.

1.How do you adjust the height of a flame?..........................

....

2.How do you make a yellow (luminous) flame?..

DIAGRAM

3.How do you make a gentle blue flame?..............

....

4. How do you make a very hot blue flame?..........

....

Open the air vents and hold a flame near them. The gas inside will catch fire and burn at the bottom of the burner. This happens most often when the vents are wide open and reflected heat from some object over the burner heats the burner. It is called 'striking back'. It is harmful; turn off the gas whenever this happens, let cool, and light again.

5.How can you tell by sound, shape, or colour of flame whether a burner has struck back?

....

....

By means of tongs hold a cold porcelain dish over the tip of a luminous flame; then try it with a non-luminous (blue) flame.

6. What kind of flame is sooty?.....................

7. Whigh kind of flame is a reducing flame,and why?

....

Adjust to a gentle blue flame. Thrust a splinter or piece of heavy cardboard across the base of the flame and remove before it catches fire.(If it catches fire drop it into the sink). Note whether there is more scorching at the edge than at the centre. Repeat,holding the splinter higher in the flame.

8. Which is the hottest part of a flame,

The tip or the base?...............................

The inside or the edge?............................

2.DETERMINING THE DENSITY OF WATER

Apparatus and Materials: Balance,small beaker(50-150ml.) pipette,water.

Procedure: Weigh a clean and perfectly dry small beaker as accurately as your balances permit. Measure into it 15 ml.(pipette) of water and weigh again.

1. Weight of water and beaker.......... gms.

2. Weight of empty dry beaker.......... gms.

3. Volume of water (as measured) ...... mls.

4. Density of water (Weight ÷ volume)... gms./ml.

5. What factors could affect your answer?

....

3. PREPARING AND SEPARATING A PRECIPITATE.

Discussion: A precipitate is a finely divided solid formed by chemical action in solution. This is a practice in laboratory techniques;refer closely to the sheets on Laboratory Techniques. The following steps are taken by the analyst when he wishes to determine the concentration of sulphuric acid in a sample.

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1. Weight of dish and precipitate.......... gms.

2. Weight of dish alone..................... gms.
(If you have not the weight already, empty and clean the dish, warm it to make sure it is dry, cool it, and weigh.)

3. Weight of precipitate .... gms.

4. Percent of sulphate radical in the ppte.,,, %
(Obtain the atomic weights and work this from the formula).

5. Weight of sulphate radical in your ppte.,, gms

6. From the formula of sulphuric acid calculate the weight of sulphuric acid that would contain 1 gram of sulphate radical.......... gms

7. Knowing the weight of sulphate radical in your precipitate, and the weight of sulphuric acid that would contain 1 gm. of sulphate radical, calculate the weight of sulphuric acid that must have provided the sulphate of your precipitate........................... gms.

8. Knowing that you used 10 ml. of acid sample, calculate the percentage strength of the sample. (I.e. Weight of acid in 100 ml. sample)

.... %

Name...........................Class......Date.......

SULPHUR: Preparation of its allotropes. Chemical properties.

Discussion: Sulphur, like oxygen, carbon, phosphorus, and other elements, can exist in several different forms (in the same state) called allotropes. These allotropes differ in the physical properties and in the energy they contain. They are different arrangements of the same atoms.

(a) Preparation of the allotropes of sulphur.

Apparatus and Materials: Test tubes (use old ones), funnel, filter paper, beaker, powdered sulphur, carbon disulphide, watch glass, magnifying glass.

Procedure:

Rhombic Sulphur (below 96.6°C)

In a dry test tube place as much powdered sulphur as will rest on the end of a small knife blade. Pour on it roughly one-third test tube of carbon disulphide and shake well. CAUTION: CARBONDISULPHIDE IS INFLAMMABLE AND POISONOUS. KEEP IT AWAY FROM FLAMES. Filter through dry filter paper into a watch glass. Lay a piece of paper over the watch glass and set it aside to evaporate, away from flames. When evaporation is complete observe the crystals (under a magnifying glass if they are very small.) In the space provided make a diagram of the crystals and state their colour.

Monoclinic Sulphur (above 96.6°C)

Fill a dry test tube half full of powdered sulphur. Have a dry filter paper folded ready. Melt the sulphur very slowly. During the melting the tube should be about 3 inches above the tip of a gentle flame and inclined at about 45°. Rotate the tube and shake constantly until the melting is complete. During the heating the sulphur should be of the consistency of water and should remain of an amber colour. When it is melted, pour the contents of the tube into the folded filter paper held by its edge (or placed dry inside the filter funnel). Watch the sulphur as it cools. As soon as crystals form on the surface, from the edge towards the centre of the sulphur, pour the still unmelted portion into a beaker of cold water (to be discarded). Unfold the paper immediately and study the crystalline forms with a magnifying glass. In the space provided make a diagram of the crystals and state their colour.

Amorphous sulphur

Use the same test tube as you used for monoclinic sulphur. Again half-fill it with sulphur, and heat strongly until it boils. Observe closely the changes in colour and consistency as the temperature rises. Pour the boiling liquid into a beaker of cold water at once. Do not be alarmed if the boiling liquid catches fire, but do not let it burn, as the fumes are unpleasant; extinguish with water). Remove the sulphur from the beaker and examine it. In the space provided make a diagram of some amorphous sulphur; describe its colour and consistency.

Diagram of Rhombic Sulphur

Colour:........

Diagram of Monoclinic Sulphur

Colour:..........

Diagram of Amorphous Sulphur

Colour:..........

Consistency.......

(b) Chemical Conduct:

Burn a very small amount of sulphur in a spoon or on the base of a ring stand.

1. Colour of the flame.......................................

2. Odour of the gas ....

3. Formula of the gas......................................

Using the same test tube as before, melt and bring to a boil about one-third test tube of sulphur. Drop strips of copper turnings or copper foil into the boiling sulphur. The copper should turn red-hot as it unites with the sulphur.

4. Equation...

5. Is this an example of combustion?..................

6. Why?...

....

7. Sulphur is like what other element chemically?........

Name........................Class......Date.......

SULPHUR DIOXIDE: Laboratory Preparation
Properties

Discussion: This gas is used in refrigeration, preserving food, fumigating, bleaching straws and paper, and in the manufacture of sulphuric acid. Commercially it is prepared by burning sulphur or sulphide ores. In the laboratory it is made either by the action of a dilute acid on a sulphite salt or by the action of concentrated sulphuric acid on copper.

(a) Laboratory Preparation

Apparatus and Materials: The gas does not act on rubber. Study the sheet on Common Apparatus Assemblies and choose the correct generator. The gas is very soluble in water and is heavier than air. Since the odour of it is choking take care to collect it so that little escapes. Choose the collecting assembly to connect to your generator that will serve to trap escaping gases.

Procedures: Set up the apparatus you have found necessary. Be sure that you have three or four dry collecting bottles and cover glasses. As each will be filled you will cover it with a cover glass and set it aside for testing. Now place about half a test tube of sodium sulphite into the flask, and add about half a test tube of water to cover the bottom of the thistle tube. Add concentrated HCl through the thistle tube when ready to collect. If the reaction is slow, heat gently. Pass the gas through each collecting bottle for several minutes in order that you can assume it to be filled with sulphur dioxide.

1. Equation..

....

2. Colour of gas.............. 3. Odour of gas...........

4. Density..................

(b) Testing the properties of sulphur dioxide.

Apparatus and Materials: Large beaker of water, blue litmus paper, weak pot. permanganate solution, blade of green grass, splinter, hydrogen peroxide, barium chloride solution.

Procedure: Upend one of your samples in the large beaker of water. Let stand, shaking at intervals. After a while test the solution with blue litmus paper.

1. Is the gas very soluble?..........2. Is this a chemical

or a physical change?..............3. Equation.................

4. Name of the acid produced...........................

5.Can sulphur dioxide be called an anhydride?..........

6.Why?..

Into a second sample pour some weak pot.permanganate solution. Describe the colour change.

....

Sulphur dioxide or sulphurous acid bleach by reduction,i.e.,by removing oxygen from the dye to render it colourless.This is in contrast to the bleaching action of chlorine or hydrogen peroxide,which bleach by oxidation. Try the bleaching action further by placing a moist green blade of grass in another bottle along with a moist piece of paper with ink-writing on it. Let stand for a period. Describe any change.

7...

Into a third sample thrust a burning splinter.

8.Does sulphur dioxide burn?........9.Support combustion?.....

To your fourth sample of sulphur dioxide add a bit of water and shake.

10.What is formed?................To this add about a third-test-tube of hydrogen peroxide solution and shake.

11.What happens?................12.Equation.................

....

Now add a few drops of barium chloride solution and a few drops of hydrochloric acid. This is a test for the sulphate ion.

12.What do you notice?..............................

13.What have you shown?.............................

Name

.........................Class......Date.........

SULPHURIC ACID: Preparation
Properties

Discussion: Sulphuric acid is the most important commercial acid. Besides being a strong acid,when concentrated it is also a powerful dehydrating and oxidizing agent. It has a high boiling point. Many acids are made by heating their salts with concentrated sulphuric acid. When such reactions are heated the acid to be prepared is boiled off, but the sulphuric acid is not. Sulphuric acid generates a great deal of heat when mixed with water. CAUTION: CONC. SULPHURIC ACID IS VERY HARMFUL TO THE SKIN OR CLOTHING. WATER MUST NEVER BE POURED INTO CONCENTRATED SULPHURIC ACID; IF THE ACID MUST BE DILUTED POUR THE ACID SLOWLY INTO THE WATER WITH CONSTANT STIRRING. Some care must also be exercised when pouring the acid into wet containers; do this at arms length. Remember that strong acids on skin or clothing should be washed off, then treated with sodium bicarbonate solution till the fizzing stops.

(a) Preparation of sulphuric acid.

Commercially sulphuric acid is prepared either by the contact process or by the lead-chamber process. The latter method will be demonstrated here.

Apparatus and Materials: Set up apparatus as for making sulphur dioxide, but connect the delivery tube with a short length of rubber tubing to a calcium chloride drying tube clamped horizontally to a ring stand. The other end of the drying tube is connected by rubber and glass tubing to one hole of a two-hole stopper that fits into the neck of a very dry and clean half-liter flask. Study your sheets on Common Apparatus Assemblies. You will also need sodium sulphite and conc. hydrochloric acid (to make the sulphur dioxide), and conc. nitric acid, (preferably some that is yellowish or reddish in colour).

Procedure: Into the very dry half-liter flask drop not more than two or three drops of the conc. nitric acid. Start your sulphur dioxide generator (see your experiment on preparing sulphur dioxide), and pass into the flask a slow stream of sulphur dioxide. At first reddish-brown oxides of nitrogen are seen. Since there is some water in the nitric acid the following reaction is occurring:

$$H_2O + 2SO_2 + 2HNO_3 = 2H_2SO_4 + N_2O_3$$

Now pass in just enough sulphur dioxide to make the reddish brown colour disappear. If the flask is dry enough a frostwork of white crystals forms over its inside walls. These are the "chamber crystals" of nitrosyl sulphuric acid. The following reaction has taken place:

$$H_2O + 2SO_2 + N_2O_3 + O_2 = 2\ H(NO)SO_4$$

In the commercial process these crystals do not appear, as there is enough steam to change them into sulphuric acid and again liberate the brown oxides of nitrogen. Add a few drops of water to the flask and shake. Watch the crystals disappear with the formation of heat and a re-occurrence of the brown fumes. Now add about 50 ml. (graduate) of water and test for sulphuric acid by adding a few drops of barium chloride solution plus a few drops of hydrochloric acid. If a white precipitate forms sulphuric acid is indicated.

1.What is the purpose of the nitric acid?..............

....

2.What is the formula of the brown gas?................

3. Is it a catalyst?.......... 4.Why?...................

....

4.What is the formula for "chamber crystals"?..........

5.Describe a test for sulphate ions....................

....

6b) Properties of sulphuric acid.

Apparatus and Materials: Evaporating dish, test tubes, barium chlroide solution, sugar, copper turnings, conc. sulphuric acid.
Procedure: Put about half a teaspoon of sugar (sucrose) into the evaportaing dish and moisten it thoroughly with conc. sulphuric acid. Observe it for several minutes.

1.What changes are observed?..........................

2.Since sugar consists of C,H,and O,what does the residue consist of?..

3.What has been the action of the acid?...............

....

To half a test tube of water add conc. sulphuric acid slowly in small amounts, with constant shaking or stirring. Feel the sides of the tube.

4. What is observed?..................................

Place a small ball of copper turnings in each of two test tubes. To one add half an inch of dilute sulphuric acid.

5. Any noticeable reaction?..............................

6. Why would this be expected?.........................

....

To the other test tube add half an inch of conc. sulphuric acid and heat. Cautiously sniff the evolving gas. After a minute of heating, flush the acid down the sink, wash the copper turnings, and examine them.

7. What gas is evolved? ....

8. How has the colour of the copper changed?...........

9. Why is this?..

10. Equation...

11. What has been the action of the sulphuric acid?....

....

Name

........................Class....... Date..........

AMMONIA Preparation
Properties

<u>Discussion:</u> Commercially ammonia is prepared by the Haber process or by the destructive distillation of coal. In the laboratory it is prepared by heating any ammonium (NH_4) salt with any base. A double replacment occurs yielding ammonium hydroxide; this is quite unstable and breaks down to form ammonia and water. A water solution of it, called aqua ammonia or ammonia water, contains NH_3 molecules, H_2O molecules, NH_4OH molecules, NH_4^+ ions, and OH^- ions. Such a solution is a weak base.

(a) Preparation

From the formula for ammonia gas calculate the weight of 22.4 liters. The weight of 22.4 liters at S.T.P. of air is 29 gms. approximately. The weight of ammonia is more or less than water................ The gas is soluble im water. You will make it by heating together solid ammonium chloride and (ammon) solid calcium hydroxide.One of the products will be water.Determine from this and the Common Apparatus Assemblies the equipment you will use.

1.Equation..

Fromthe equation calculate the weights of ammonium chloride and calcium hydroxide needed to make about two liters of ammonia. The weights are

2.Ammonium chloride..........gms. 3.Calcium hydroxide......gms.

Weigh out approximately on the rough balance,and place in the pyrex test tube of your apparatus.Arrange the delivery tube with a short rubber join so that it can be directed up or down. When not collecting gas have the end of the delivery tube just touching the surface of water in a beaker.Heat gently and collect four samples.

3.Colour.....................Odour......................

(Note:Ammonium chloride fumes sometimes contaminate the sample and may affect its colour.)

While awaiting testing and during testing the samples must be kept mouth downward. Why?.......................

(b) Testing the Properties of ammonia.

Apparatus and Materials: Your gas samples,splinter,large beaker with water,litmus paper,conc. HCl.

Procedure: Lower an inverted bottle of ammonia into the beaker of water and remove cover glass. Shake gently and let stand for a while. Slip cover glass over the mouth of the bottle,remove bottle and contents,and test the contents with litmus paper.

1.Is the gas very soluble?..............................

2.Is this a chemical or physical change?................

3.Equation...

4.Is the solution acidic or basic?.....................

Into a second sample,keeping the mouth of the bottle downward,insert a burning splinter.

5.Does the gas burn?...........6.Support combustion?........

Place a few drops of conc. HCl into an empty dry bottle.Roll the bottle until thei inside is covered with the acid. Cover it with a cover glass and turn it upside down upon a covered sample of ammonia. Remove both plates at once and keep the mouths of the bottles together.

7. What do you notice?...................................

8.Equation.......................(underline product)

9.In which bottle should there be the most visible

change?..............10.Why?..........................

....
(Consider Graham's Law of Diffusion of Gases.)

Name.........................Class.......Date........

NITRIC ACID: Preparation
Properties
Test for Nitrates
Oxidation-Reduction.

Discussion: Acids contain hydrogen which may be replaced. Some oxygen acids,however,will under the proper conditions liberate their oxygen rather than their hydrogen. Then they may be said to be oxidizing agent.Such substances are at times acids,at times oxidizing agents (particularly if there is present a reducing agent that takes on oxygen readily). Nitric acid is such an acid. It has a low boiling point. It reacts with rubber. It is unstable, on heating breaking down to form the brown gas nitrogen dioxide.

(a) Preparation of Nitric Acid.

Apparatus and Materials: See sheets on Common Apparatus Assemblies,remembering that nitric acid acts on rubber. Rough balance,graduate,funnel,glass rod,sodium nitrate, (or KNO_3),conc. sulphuric acid.

Procedure: Set up the apparatus as planned. Before clamping the retort put into by means of a paper funnel about 15 gms.(rough balance) of sodium nitrate. Run in 12 ml. approximately(graduate) of conc. sulphuric acid,using a funnel. In both cases see that the materials do not enter the side arm of the retort. Complete assembly of your apparatus as planned. Heat gently by passing the flame back and forth under the sandbath.If the fumes become reddish,slow your heating.When you have at least an inch of nitric acid in your receiving test tube remove flame and let cool. When cool to the hand,first pour any remaining acid from the retort into the sink,keeping the water tap running meanwhile. Then cautiously run water into the retort,warm over a flame to dissolve contents,and flush down the sink.CAUTION:BOTH NITRIC ACID AND SULPHURIC ACID ARE VERY CORROSIVE. HANDLE WITH CARE. WIPE UP SPILLS AT ONCE. ACIDS ON THE SKIN OR CLOTHING SHOULD BE WASHED OFF WITH WATER,THEN WITH SODIUM BICARBONATE SOLUTION.

1.What is the colour of the liquid collected?.............

2.Account for this colour................................

3.Equation for the formation first of nitric oxide,then of nitrogen dioxide

....

4.Equation for laboratory preparation of nitric acid....

....

5.Why is sulphuric acid used?...........................

....

6.Why must the heating be gentle?.......................

7.What is the solid left in the retort?.................

(b) Properties of Nitric Acid

Apparatus and Materials: Your sample of nitric acid,test tubes,pieces of zinc and copper the size of a pea or smaller,porcelain dish,pipe-clay triangle,ring stand, sawdust, dilute HCl,ammonium hydroxide.

Procedure: Place a pinch of sawdust in a porcelain dish supported by the pipe-clay triangle on a ring stand. Heat until the sawdust smoulders (from above if necessary). Cautiously pour on a few drops of your sample acid.

1.What happens?...

2.What property of nitric acid is exhibited?...........

....

3.What do you conclude about the stability of nitric acid?

....

By means of a glass rod carefully place on your thumbnail a very small drop of the sample nitric acid. Allow it to remain a few seconds,then rinse off.Add a drop of ammonium hydroxide to the same spot in the same manner. Repeat,asing conc.HCl on another part of the thumbnail.

4.Describe any colour changes...........................

....

This is a test for proteins that is frequently used.

Put a small piece of zinc in a test tube and pour on it about one-quarter inch of your sample acid. Note if the gas burns.

5.What gas would you expect when an acid acts on zinc?.......

6.Explain your results.................................

....

Repeat the above with a small piece of copper .

7.Considering that copper is below hydrogen in the electrochemical series explain the reaction that occurs

....

....

8.What is the formula of the brown gas?................

(c) Test for the Nitrate Ion

Apparatus and Materials: Conc. sulphuric acid,ferrous sulphate,any nitrate as a sample,test tubes,beaker.

Procedure: Make about one-third test tube of a saturated solution of ferrous sulphate.Add to it a few drops of a strong solution of your nitrate sample. In another test tube pour about half an inch of conc, sulphuric acid.(CAUTION). Cool both solutions under the tap or in a beaker of cold water.Hold the sulphuric acid test tube in a beaker of cold water,as much at a slant as possible. Carefully run down its inner side the ferrous sulphate+ nitrate solution,avoiding stirring or mixing of the two solutions. Keep it under cold water for a few minutes.

1.What is observed?.....................................

(d) Oxidation-Reduction.

Remember that Loss of Electrons is Oxidation = LEO

$$Zn^{0} + 2H^{+},NO_3^{-} = Zn^{++}, 2\ NO_3^{-} + H_2^{0}$$

1.What has been oxidized,and why?......................

....

2.What has been reduced,and why?.......................

....

3.Which is the oxidizing agent?.........................

Name............................Class......Date...........

BOYLES LAW: To prove that the volume of a given quantity of gas is inversely proportional to the pressure, provided the temperature is maintained constant.

Discussion: Whenever two quantities are inversely proportional to each other it can be shown that their product is constant. This experiment will attempt to show that the volume of a given quantity of gas times the pressure at which it exists will be equal to any other volume of the same gas times whatever pressure the second volume exists at. I.e. $P_1V_1 = P_2V_2$

Apparatus and Materials: Hydrometer jar or other deep vessel, burette, meter stick, barometer, pipette, beaker.

Procedure:

The given quantity of gas will be the air contained in the burette when it is inverted over water in the hydrometer jar. The pressure on it is made to change by thrusting the burette down into the water, so that the pressure increases as more and more water outside the burette rests above the water level (meniscus) inside the burette. In this manner the volume of the given quantity of gas will be made to change slightly.

First note the position of the clamp on the rubber nozzle of the burette with ink, and make sure that it does not leak by thrusting the burette down in the water and noting whether the liquid level inside remains constant. Do not shift the position of the clamp hereafter. Now measure the capacity of the burette between the clamp and the nearest calibrations by filling this space with water from a pipette ;(be sure to let any water on the inside wall of the burette drain down before adding more.) Note the volume delivered by the pipette. Empty the burette.

Set the hydrometer jar into an empty pneumatic trough and fill it with water. Invert the burette, open the clamp, and push the burette down until the inside water level is just at the zero mark of the burette calibrations. (Always read levels from the lowest part of the meniscus.) Close the clamp, and again make sure it does not leak. Do not touch it from here on.

Obtain the barometer reading, and the aqueous vapour pressure for the room temperature from your instructor. Calculate the volume and pressure of the gas when the burette is in such a position that the water level both inside and outside touch the zero mark of the calibrations.

Now thrust the burette as far down in the water as it will go. The water inside the burette will have risen slightly above the zero mark. Determine to the tenth of a ml. how much it has risen by reading the calibrations. Subtract this from the first volume in order to obtain the present, second volume. Measure the depth of the inside water level below the outside water level accurately in mm. To do this shift the burette inside the hydrometer jar to the side, and hold the meter stick alongside it but outside the hydrometer jar. The total pressure will be the barometer pressure (in mm. of mercury) plus the pressure of the depth of water just measured (in mm. of water.) Mercury weighs 13.6 times as much as water. Calculate what column of mercury would exert the same pressure as the depth of the water just measured. Add this to the barometer reading to obtain the total second pressure.

1. Barometer pressure (in mm. of mercury)...............

2. Aqueous vapour pressure..............................

3. V_1........................4. P_1..................

5. P_1V_1.......................................

6. $V_2(=V_1$ - rise of water in burette)...............

7. Depth of inside water level below outside..........mm.

8. Equivalent of this in mercury.................... mm.

9. $P_2(=P_1$ + water pressure in terms of mercury).......mm.

10. P_2V_2..

If the products P_1V_1 and P_2V_2 are approximately within one percent of each other you may assume that within experimental error you have demonstrated Boyle's Law. However, you should analyze your technique for possible error and make another attempt inorder to decrease the margin of error.

Possible errors of technique:.............................

Percent error (The difference between the two products divided by P_1V_1, the result multiplied by 100).........

Name..........................Class........Date.......

GRAHAMS LAW OF DIFFUSION: The rates of diffusion of gases is inversely proportional to the square roots of their densities, temperature remaining constant.

Discussion: In the case of gases the densities are approximately equivalent to their molecular weights. When two quantities are inversely proportional their products have a constant value. Thus, if rates of diffusion are d,D, for the two gases respectively, and their molecular weights are w,W, respectively, then $d\sqrt{w} = D\sqrt{W}$. If the gases have molecular weights of 32 and 4 respectively, to find how many times as fast the first gas diffuses as the second find d/D, which equals $\sqrt{W}/\sqrt{w} = \sqrt{4}/\sqrt{32} = .38$ times as fast.

Apparatus and Materials: Dry glass tube approximately 12-18 mm. in diameter($\frac{1}{2}$-$\frac{3}{4}$ inch), and 15-20 inches long; meter stick, absorbent cotton, conc. HCl, conc NH_4OH.

Procedure: When HCl fumes and NH_3 fumes(from the ammonium hydroxide) diffuse and meet, a white solid (smoke) forms.

1. Equation..

To avoid this smoke do not bring together the two gases at your desk. Place one-quarter inch of the HCl and the ammonium hydroxide in separate stoppered test tubes; do not open near each other, or the smoke will form. Place the glass tube flat on the table. Make two cotton plugs that will fit the ends of the tube. Moisten one with conc. HCl, and the other with conc. ammonium hydroxide. Introduce the two plugs into the ends of the glass tube at the same time. Watch the tube for the appearance of a white ring where the two gases meet after diffusion. Measure the distance of this ring from either end.

2. Distance diffused: HCl.............mm; NH_3........mm.

3. Calculate Mol.Wts. HCl.............. NH_3........m

4. Calculate $d\sqrt{w}$ for HCl..........., for NH_3.............
These two products should be equal.

5. Percentage error $\left(\frac{\text{Diff. between products}}{d\sqrt{w}\text{ for HCl}} \times 100\right)$=.....%

Name..........................Class........Date.........

TO DETERMINE THE MOLECULAR WEIGHT OF CARBON DIOXIDE.

Discussion: This may be done as a demonstration experiment. One mole (or gram molecular weight) of a gas occupies 22.4 liters at S.T.P. The molecular weight of the gas is expressed by the same number as the weight in the mole.In order to determine the molecular weight of carbon dioxide:

1. Prepare and collect some dry gas in a flask.
2. Obtain the weight of this gas.
3. Obtain the volume of this gas by filling the same flask with water and weighing.(One gm. water= one ml.)
4. Determine what this volume would be at S.T.P.
5. Calculate what 22.4 liters of the gas weigh at S.T.P.
6. The number expressing the weight in grams is the Mol.Wt.

Apparatus and Materials:Apparatus for generating and drying carbon dioxide,(see Common Apparatus Assemblies: make the carbon dioxide by the action of HCl on marble chips or chalk);thermometer,barometer,balance,as large a thin-walled flask as will fit your balance pans,two-holed stopper to fit,glass tubing,rubber joints,clamps for the rubber tubing.

Procedure: Equip the clean,dry flask with a two-holed stopper carrying one tube that extends very nearly to the bottom of the flask,and a delivery tube that does not project below the stopper.(See Common Apparatus Assemblies:collecting a noxious gas;the tubing is arranged as for the collecting bottle in this case.).Attach a short piece of rubber tubing to each glass tube and put a screw clamp on each rubber tube.Weigh the flask and its attachments.

The CO_2 generator is next set up with its delivery tube attached to a calcium chloride drying tube clamped horizontally to a second ring stand. The other end of the drying tube is attached by glass tubing to the rubber join of the longer glass tube in the flask. Both clamps of the flask are opened.

CO_2 is now generated and the dry gas allowed to pass through the flask for at least five minutes.This is to assure that all the air has been swept out of the flask. Close the clamps,detach the flask from the drying tube,and weigh flask and attachments,now containing CO_2.

Obtain the room temperature (C^o) and the barometer pressure.

Attach a funnel to the longer glass tubing of the flask.Open both clamps and through the funnel fill the flask completely with water (there must be no air bubbles.).DO NOT MOVE THE RUBBER STOPPER AT ANY TIME. Dry the outside of the flask.Weigh flask and attachments, now filled with water.

1.Wt. of flask and air..............................gms.

2.Wt. of flask and CO_2..............................gms.

3.Wt. of flask and water............................gms.

4.Temperature.......................................C°

5.Pressure (barometer reading).....................mm. Hg.

Calculations:

1.Wt. of flask without air..........................gms.
(The approximate volume of your flask is stamped on it;subtract 1.29 gms.for each liter of air contained.)

2.Wt. of water,and therefore volume of flask........ml.
(Subtract wt. of flask alone,from wt.of flask+ water.)

3.Calculate volume of flask(or of CO_2) at S.T.P.

....ml.

4.Wt. of CO_2 (Subtract Wt. of flask from Wt. of flask+CO_2)............................gms.
You now know the weight and the volume of a given quantity of carbon dioxide.

5.Calculate weight of 22.4 L(22,400 ml.) of carbon dioxide at S.T.P.........................gms.

6.Experimentally determined molecular weight............
The correct molecular weight of CO_2 is 44.

7.Percentage error $\left(\frac{\text{Diff. between Mol.Wt. found and 44}}{44} \times 100\right)$

....

8.Can you suggest any sources of error?

....

....

Name..........................Class.........Date........

TO DETERMINE THE FORMULA OF TIN OXIDE

Discussion: A formula is a recipe of a molecule in terms of atoms. Thus, if we know that a molecule of steam contains two atoms of hydrogen and one atom of oxygen we can write the formula (or recipe) of its molecule as H_2O. In our laboratory we cannot find the number of atoms of each element in a compound directly. It is comparatively easy however to get the percentage composition by weight of the compound. This tells us the weight of each element for every 100 gms. of compound, but not the number of atoms. From this we can deduce the ratio between the atoms of the elements involved. For example, suppose the elements X and Y form the compound XY so that there is 50% of each in the compound. Does this mean that there will be the same number of atoms of X as of Y in the molecule? No, because it might happen that the atoms of X each weigh twice as much as the atoms of Y. Then, in order to have equal weights of the two elements we would need only half as many atoms of X as of Y. We could say then that the formula is XY_2, but it would need to be remembered that the correct formula might be $X_{10}Y_{20}$, or $X_{20}Y_{40}$, etc. What we have determined is the simplest formula only. This is what will be attempted in this experiment.

Apparatus and Materials: Small crucible, balance, clay triangle, tongs, burner, ring stand, mossy or sheet tin, conc. nitric acid.

Procedure: Weigh a clean crucible accurately. Place in it about 2 gms. of tin and weigh again. Place the crucible on a pipe clay triangle on the ringstand. Add nitric acid drop by drop until no more brown fumes come off. You may have to break up small particles with a stirring rod. Now heat, gently at first to avoid spattering, then strongly until no more brown fumes appear. The tin nitrate formed at first has broken down into tin oxide, nitric oxide gas, and steam. Allow the crucible to cool, then weigh. Calculate the weight of oxygen that has combined with the tin, then find the percent of each element in the compound. From these data calculate the simplest formula.

1. Wt. of crucible + tin..............................gms.

2. Wt. of empty crucible..............................gms.

3. Wt. of tin alone...................................gms.

4. Wt. of crucible + tin oxide........................gms.

5. Wt. of oxygen that combined with the tin........gms.
(Subtract item 1 above from item 4)

6.Wt. of tin oxide..................................gms.

7.Percent of tin in the oxide.........................%

8.Percent of oxygen in the oxide......................%

9.Divide the percents by the atomic weights of each element to get the relative number of atoms:

(a) Percent of tin in oxide ÷ 119...................

(b) Percent of oxygen inoxide ÷ 16..................

10.Simplest whole number ratio of the above(divide both by the smaller):

.......atoms of tin for every........atoms of oxygen.

11.Formula of tin oxide...............................

12.Why cannot we use the weights directly as subscripts to write the formula?

....

13. Why did we divide percents by atomic weights?

....

14.Why do our results not give exact whole-number ratios?

....

15.Is our formula necessarily the correct one? Why?

....

....

16.In order to make sure that our formula was the correct one,not merely the simplest,what further information would we need?

....

Name..........................Class..........Date........

SOLUTIONS	Factors affecting solution Hydrated and anhydrous salts Types of aqueous solutions.	Different sections will need to be done in different periods.

Discussion: A solution is a homogeneous mixture of two substances, the one in excess being called the solvent and the other the solute. The solubility of a substance is measured by the weight of it that will dissolve in a given weight(usually 100 gms.) of water or other solvent. True solution is a physical change, but we also speak of zinc dissolving in sulphuric acid when it disappears due to chemical change; this is not true solution. Even when a solute no longer appears to be dissolving, the chemist assumes that it is still dissolving but that this is just matched by the rate of dissolved material settling back on to it. Even in true solutions there may be a mild chemical action in which some water attaches itself to solute molecules to form hydrated salts. Such an action is accompanied by heat called the Heat of Hydration.

(a) Factors affecting solution:

Apparatus and Materials: Ring stand and ring, wire gauze, burner, beaker, test tubes, thermometer, balance, mortar and pestle, sodium chloride, iron filings, pot. nitrate, pot. dichromate, iodine, alcohol, chloroform.

Procedure: To separate each half full of water add approximately equal amounts (one-quarter inch in bottom of test tube) of the following finely divided materials; shake, and note which dissolve and which do not.

1. What factor affects solubility inthis case?...........

....

Add 2 or 3 crystals of iodine to three test tubes each containing about an inch of alcohol, chloroform, and water respectively. Shake and note signs of dissolving.

2. What factor affects the solubility inthis case?.......

....

Select two lumps of pot. dichromate of about equal size. Grind one to a fine powder in the mortar and pestle. Place both intest tubes nearly full of water. Shake each for the same length of time and let stand for the same length of time. Observe which has dissolved the most.

3. What factor affects the solubility in this case?......

....

Grind to a powder a small amount of pot.dichromate in a mortar andpestle. Place equal amounts (about half an inch) in separate test tubes each about two-thirds full of water. Make sure that all conditions in the two cases are alike,except that the one test tube is shaken vigorously.Observe which has dissolved the most.

4.What factor affects solubility in this case?.........

....

Weigh out roughly 12 gms. of fine sodium chloride and divide it into four piles of approximately 3 gms. each. Pour 25 ml.(graduate) of water into a test tube. To this water add one pile of sodium chloride at a time with vigorous shaking,making sure that each pile has disappeared before another is added.When no more dissolves the solution is saturated. You may not need to use all the piles. Repeat in another test tube,using roughly 60 gms. of pot. nitrate divided into four piles of 15 gms. each. Water temperature should be the same in each case and should be determined by thermometer.

5. Water temperature..............................C°

Calculate the solubility of each salt in 100 ml. of water.From solubility tables in the text determine what the solubilities should be. Now place the test tubes with their saturated solutions in a beaker of water and bring it to a boil. Continue to add portions of each solute as before until each solution is again saturated at the boiling point. Again compare with the data of the solubility tables.

	Sodium chloride	Pot.Nitrate
6.Solubility at.....C°	....	....
7.Solubility at the above temperature,from tables	....	....
8.Solubility at 100°C	....	....
9.Solubility at the above temperature,from tables	....	....

11.How does temperature affect the solubility ....

12.Does temperature affect solubility of different salts

in the same way?..

13.Define a saturated solution...........................

....

Let both solutions cool under the tap.

13.Which solute is more soluble at room temper ture?..........

14.What kind of solution have you after cooling?.............

(b) Hydrated and anhydrous salts.

Discussion: Some salts have attached to their molecules several molecules of water. They are called hydrated salts, and the water is called water of crystallization since without it their crystals crumble to a powder. Many hydrated salts cling to their water of crystallization firmly, but others lose it slowly even at normal conditions.

Apparatus and Materials: Watch glasses, porcelain dish, clay triangle, ring stand, mortar and pestle, washing soda, bluestone, sodium thiosulphate, anhydrous calcium chloride.

Procedure: On separate watch glasses expose to the air crystals of washing soda $Na_2CO_3.10H_2O$, bluestone $CuSO_4.5H_2O$, sodium thiosulphate $Na_2S_2O_3.5H_2O$. Compare these at the end of the period with similar samples set out the day before.(INSTRUCTOR NOTE).

1.Which salts become powdery at the surface?...........

2.Why ?..

3.What is this phenomenon called?.....................

Powder some bluestone with a mortar and pestle, and heat it strongly in a porcelain dish. Let cool, observe, then add a few drops of water.

4. What changes occur?...............................

5.Why?...

6.Is this efflorescence?........ Why?..................

Some anhydrous salts will at normal conditions take on moisture from the air to become visibly wet. Place a piece of anhydrous calcium chloride in a watch glass for the rest of the period and compare it with a sample previously set out by the instructor.(INSTRUCTOR NOTE).

7.What name is given to this phenomenon?.................

8.This happens with any very soluble salt.Explain why.

....

....

....

(c) Types of solutions.

Apparatus and Materials: Test tube, sodium thiosulphate, burner.
Procedure: Fill a clean dry test tube about one-quarter full of sodium thiosulphate crystals. Make sure that no crystals cling to the inside walls. Add not more than five drops of water. Heat gently until the thiosulphate dissolves. At first the liquid will be whiteish, then it will turn clear. Do not heat beyond this stage. Stopper loosely with a piece of paper and cool under the tap. Now add a small crystal of thiosulphate; observe changes, and feel the side of the test tube.

1. What kind of a solution was this?....................

2. What are properties of such a solution?...............

....

3. What kind of solution is it after crystallization finishes?

....

4. Given saturated, unsaturated, and supersaturated solutions, how could you tell them apart by a simple test?

....

....

Name.........................Class........Date.......

THE EFFECT OF CONCENTRATION ON BOILING POINT OF SOLUTIONS

Discussion: One mole of a non-volatile non-electrolyte in 1000 gms. of water, or any solution of these proportions, will have a boiling point 0.52°C higher than that of pure water at standard pressure. One mole (gram molecular weight) of any substance contains a definite number of particles (Avogadro's Number= 6.06×10^{23} molecules). Thus the B.P rise depends on the number of particles, not on their size or weight. A mole of an electrolyte will represent the same number of molecules, but since these ionize, it will represent a greater total number of particles (ions), and will consequently have a greater effect on the B.P.
Apparatus and Materials: Thermometer (one-tenth degree preferred), anhydrous calcium chloride, sucrose, ring stand and clamps, large pyrex test tube, balance, small pieces of earthenware or capillary tubing.

<u>Procedure:</u> If an ordinary thermometer is used estimate the temperature as accurately as you can to the nearest quarter of a degree. Clamp the large test tube in a vertical position. Pour into it 20 ml. (graduate) of water. Mark the level of the water on the test tube either with ink or a piece of tape; as water boils away keep adding water to keep it at this level. Hang a thermometer by a loop of wire from a clamp so that the bulb is in the middle of the water and not touching the sides. To keep the water from bumping while being boiled put in a few small pieces of porous earthenware or capillary tubing. Very gently boil the water. Note exactly the boiling point of water.

1. Boiling point of water.................................C^o

Weigh out accurately 20 gms. of calcium chloride. Remove burner and thermometer to add the 20 gms. to the water in the test tube. Mark the new liquid level, and maintain it by adding water if necessary. Heat gently with shaking until the salt has dissolved. Bring to a gentle boil and again note the exact boiling point.

2. Boiling point of calcium chloride solution..........C^o

Empty and clean the test tube. Repeat, using this time 60 gms. of sucrose.

3. Boiling point of sucrose solution...................C^o

From the formulae of the solutes calculate what one mole is in each case; then calculate how many moles per liter (1000 gms) water you have.

	<u>Rise of B.P. Found</u>	<u>Moles per Liter</u>
Calcium chloride	.............C^o	....
Sucrose	.............C^o	....

4. Calculate how many degrees the boiling point should have risen for your sucrose solution (see Discussion)......C^o

5. Similarly claculate what the rise in B.P. should have been for calcium chloride solution if it behaved like sucrose................C^o

6. Why is it different?..................................

7. Considering how each molecule ionizes, what should be the theoretical rise in B.P. for calcium chloride.......C^o

8. If your experimental findings show less than this, how could you account for this other than by experimental error?

....

Name..........................Class.....Date.......

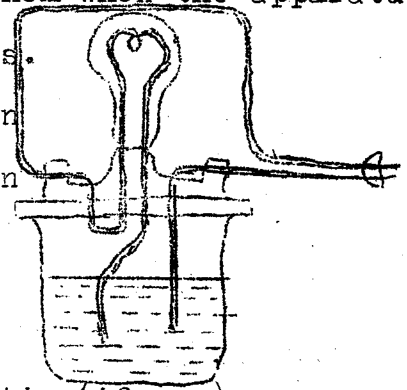
IONIZATION: Conductivity in solution
Strength of acids.

Discussion: Some substances, called electrolytes, in solution conduct electricity, undergoing at the same time a chemical change called electrolysis. It is believed that their molecules in solution break up into charged particles called ions. These obtain their charge by a transfer of electrons.

(a) Conductivity in solution

Apparatus and Materials: Conductivity apparatus of the same principle as in the diagram, several clean beakers, distilled water, dilute solutions of : glycerin, ethyl alcohol, sucrose, sulphuric acid, hydrochloric acid, acetic acid, sodium hydroxide, ammonium hydroxide, sodium chloride.

Procedure: Clean the two wires or electrodes. After each test rinse them in water. Do not touch them when the apparatus is connected to the current source. Use the same depth of solution for all tests. Place your sample in the beaker, plug in the conductivity apparatus, and set it on the beaker so that the electrodes are immersed in the solution. If the solution conducts electricity it leads current across the gap between the electrodes, the circuit is completed, and the bulb lights up. If the solution is a poor conductor, the bulb will will not glow as brightly. Record your data.



Substance	Conductivity	Ionic Equation(if any)
Dist. water	....	....
Glycerin	....	....
Ethyl alcohol	....	....
Sucrose	....	....
Sulphuric acid	....	....
HCl	....	....
Acetic acid	....	....
Sodium hydroxide	....	....
Ammonium hydroxide	....	....

1. What classes of substances are electrolytes?..........

....

2. What evidence did you notice of electrolysis?

....

3. What kind of valence holds together electrolyte molecules?

....

4. What is meant by this kind of valence?

....

5.Why are some electrolytes poor conductors?............

....

6.What ions are found in every acid?........Base?.........

(B) Strength of Acids.

Apparatus and Materials: As before,but use conc.HCl.
Procedure: Into a dry beaker pour some conc.HCl and test its conductivity as in part (a).If your laboratory acid is not very strong use glacial acetic acid. Now pour out half your sample,refilling,with stirring, with water to the former level. Again determine the conductivity.

1.If your conc. acid was strong enough not to contain

water,what would you have expected?........................

2.Why?..

3.In your experiment,which had the better conductivity,

the concentrated or the dilute solution?................

Acid molecules break up into ions.In concentrated solutions these ions are near each other,can meet easily and change back into molecules. In dilute solutions they are farther apart and are not so likely to change back into molecules.

4.Which of the solutions would have the greater degree

of ionization?..

5.What makes a strong acid,the molecule or the ion

concentration?..

6.Does a concentrated acid mean it is a strong acid?........

7.Why?...

....

....

Name............................Class.......Date...........

TO SHOW THAT THE ION DOES NOT RESEMBLE THE ATOM

Discussion: An atom behaves essentially like the molecule of the element, which in turn has all the characteristics we attribute to the element. Thus the copper atom is the same colour as copper metal. A solution of a compound has no atoms, only molecules and ions. These are in a state of equilibrium, each changing into the other. In concentrated solutions the proximity of ions to each other makes them combine readily to form molecules, so these predominate. In dilute solutions the ions are scattered so far apart that the ion condition tends to prevail. Properties of solutions are caused either by molecules or by ions.

Apparatus and Materials: Test tubes, distilled water, copper sulphate, copper chloride, copper bromide, sodium sulphate, sodium chloride, sodium bromide, bunsen.

Procedure: Dissolve about one-quarter of an inch of each salt in a test tube in the least amount of water by heating. Note colours of the solutions. Now fill each test tube with distilled water and shake. Again note the colours.

Solution	Colour concentrated	Colour Dilute
Copper sulphate	....	....
Copper chloride	....	....
Copper bromide	....	....
Sodium sulphate	....	....
Sodium chloride	....	....
Sodium bromide	....	....

1. In concentrated solution the colour is due to.............

2. In dilute solutopms the colour is due to ....

3. How do you know the colour is not due to negative ions?

....

4. What ions are common to all copper salts in dilute solution?...

5. What is the colour of the copper ion?................

6. How does it differ from the Cu atom?.................

....

Name.........................Class.........Date..........

TITRATION

Discussion: Accurate measurement of the volumes of acid and base involved in neutralization is called titration. It is accomplished by means of burettes to an accuracy of one drop or less. The acid and base solutions are run from burettes into a beaker until the exact point of neutralization(the end-point) is indicated by a colour change in an indicator (litmus,phenolpthalein etc.)in the beaker. The ratio of the volumes involved is determined. If the strength of one were known,that of the other could be calculated.

Apparatus and Materials: Two burettes,clamps,stand,beaker, indicator,funnel,HCl solution,NaOH solution.

Procedure: Study Laboratory Techniques on manipulating a burette.Thoroughly clean two burettes and clamp them to a stand.Rinse each burette with a small amount of the solution it is to contain;this is to assure that no water remains in it to dilute the sample later.See that the burettes do not leak,and that it is easy to deliver single drops with them.Fill one with NaOH solution,the other with HCl solution.Run out enough solution in each case to make sure that the burette tip above and below the clamp is full of liquid with no air bubbles.Refill burettes, if necessary,to the zero calibration. Wipe off the outside of the burettes.Run about 10 ml. of the NaOH solution into a clean beaker containing 3-5 drops of indicator. Now add HCl from the second burette until the colour is changed by addition of one drop.This is the end-point. If you do not get it the first time,add enough NaOH from the first burette to reverse the colour change,then add HCl drop by drop until the endpoint is reached. Now read both burettes accurately to the nearest tenth ml.Repeat until you have three closely checking results.

Base Burette		Acid Burette		Acid Volume
Reading	Ml.Base used	Reading	Ml.Acid used	÷ Base Volume
1......	....	....	....	....
2......	....	....	....	....
3......	....	....	....	....

Average Acid/Base ratio....................................
(All such calculations should be taken to two decimals)

Your titration has shown that on the average 1 ml. of base solution will react with ....ml.acid solution.

Equation for the reaction...

Name..........................Class........Date.........

CHLORINE Preparation Properties

Discussion: Chlorine gas,even in small quantities,is irritating and harmful to the respiratory tract. If fume hoods are not available escaping fumes should be trapped, and care must be exercised in the inhaling of fumes. Chlorine is prepared in the laboratory by the oxidation of hydrochloric acid. Any active oxidizing agent may be used,but manganese dioxide is chosen because the rate of its reaction can be more readily controlled.

(a) Preparation

Apparatus and Materials: Chlorine is soluble in water and heavier than air.See sheets on Common Apparatus Assemblies and select the appropriate equipment. Since its fumes are undesreable,arrange the collecting bottles with two-holed stoppers and fume trap as illustrated on these sheets. The fume trap should contain strong sodium thiosulphate or NaOH solution.You also need; 4 collecting bottles with cover glasses,conc.HCl,MnO_2.

Procedure: Into the flask place roughly 15 gms. of powdered MnO_2 and add about a test-tube full of conc. HCl through the thistle tube. The initial reaction will soon stop,and then the flask should be heated gently.If heated strongly the HCl will boil off.When a sample bottle is full (as noted by its colour) exchange it for an empty one and quickly cover it with a cover glass. A rag dipped in ammonium hydroxide placed nearby will tend to remove any escaping fumes.Collect four samples.

1.Equation...

2.Colour of the gas?....................................

3.What impurities might be in your sample?..............

(b) Properties

Apparatus and Materials: Large container with water,litmus paper,calcium carbide,antimony powder,turpentine,evaporating dish,tongs,filter paper.

Procedure: Invert one sample of chlorine in the container of water;let stand,with occasional shaking. After 15 minutes slip cover under bottle,and remove bottle and contents. Test with litmus paper.

1.Is chlorine quite soluble?...........................

2.Is this a chemical change?..........3.If so,give the equation..

4.Why does chlorine react with water?..................

....(be specific).

5.What change occurs on the litmus?....................

6.Why?...

Heat a small amount of antimony powder on the handle of the tongs(or spatula) and sprinkle into a second sample.

7.Describe what happens................................

8.Equation...

Add about one-quarter inch of water to a third sample and drop in a small lump of calcium carbide.

9.What happens?..

10.Equation for calcium carbide acting on water........

....

11.Reaction between chlorine and acetylene

....

12.Is this an example of combustion?...................

13.Why?..

In an evaporating dish cautiously heat about a teaspoon of turpentine (if it catches fire,cover it with a glass dish or plate).By means of tongs dip in a piece of filter paper and immediately drop it into another sample of chlorine.

14.What happens..

15.Why?..

16.With what element has chlorine a particular affinity?

....

17.What reactions have you demonstrated that depended

on this affinity?......................................

....

Name..............................Class....Date........

HYDROCHLORIC ACID: Preparation
Properties

Discussion: Hydrogen chloride gas is inactive, water soluble, and heavier than air. In solution it yields H^+ ions to produce a strong acid.

(a) Preparation

Apparatus and Materials: See Common Apparatus Assemblies for the preparation and collection of a heavy, soluble gas. Beaker, dry test tubes with stoppers to fit, sodium chloride, conc. sulphuric acid, bunsen.

Procedure: Set up the apparatus. Place about 10 gms. (or enough to cover the bottom of the flask to one-quarter inch depth) of sodium chloride in the flask. Through the thistle tube add about half a test tube of conc. sulphuric acid. Place a test tube under the delivery tube so that the tube reaches to the bottom of the test tube. Now heat gently until a reaction is noticed in the flask. Pass gas into the test tube for a few minutes, then remove test tube and stopper it. Repeat with a second test tube. Now arrange the delivery tube so that it extends to just above the surface of water (about one test tube full) in a beaker. Pass gas on to the surface of the water for about five minutes.

1.Equation ....

2.What is the general method of preparing most acids?

....

....

3.Why is sulphuric acid used, and why is it heated?

....

....

(b) Properties

Note: These are the properties of acids in general.

Apparatus and Materials: Test tubes, medicine dropper, litmus paper, zinc and copper (size of a pea or smaller), copper oxide, ammonium hydroxide, sodium carbonate, silver nitrate solution, nitric acid.

Procedure: Remove the stopper from one test tube of sample and blow across it.

1.Observation...

Remove stopper from the second sample and invert test tube over water, with occasional shaking.

2.Is the gas very soluble?.................................

3.How does this account for your observation in (1)?

....

....

From now on use the HCl solution you have made in the beaker. Take samples from it with the medicine dropper. Place one drop in the palm of your hand, dilute with at least an equal amount of water, and taste it.

4. Taste...

On separate pieces of red and blue litmus paper place drops of the HCl solution.

5. Effect on litmus....................................

On separate small pieces of zinc and copper place a drop of the HCl solution. Observe colosely for a minute.

6. Which reacts with the acid?...........................

7. Equation...

8. In terms of the electrochemical series of metals explain why there is a difference in the reactions......

....

Place a very few grains of black CuO in a quarter inch of water in a test tube. Add an equal amount of the HCl solution. Do the grains disappear? Heat if necessary.

9. Equation...

10. Is there a difference in the action of HCl on copper and cupric oxide? ....

To a test tube add a few drops of ammonium hydroxide. Dilute with half an inch of water. Drop in litmus paper.

11. What is the colour of the litmus?.......Why?.........

Add HCl solution drop by drop till the colour of the litmus changes. What kind of reaction have you

illustrated?.............12. Equation.......................

On a very small pile of sodium carbonate on a piece of paper place a drop of HCl solution. Observe.

13. Observation................Equation....................

To an inch of HCl solution in a test tube add a few drops of silver nitrate solution.

14. Observation................Equation...................

Filter off the precipitate. To the precipitate on the filter paper add ammonium hydroxide (about a test tube full)

15. Is the precipitate soluble in NH_4OH?.................

NOTE: The preceding two steps are a test for the chloride ion.

Name............................Class....Date......

CHEMICAL EQUILIBRIUM

Discussion: In a typical chemical change, substances (reactants) act on each other to form new substances (products). This is called the forward reaction. But the products, if left together, may react together and change back to the reactanta. This is called the backward reaction. If the two reactions are equal the result is chemical equilibrium, and no noticeable change is observed. If we desire to obtain the products we must slow down or prevent the backward reaction. Then we have shifted the equilibrium or brought the reaction to completion. To have a completed reaction one of the products must be removed or isolated as fast as it forms. To shift the equilibrium, one of the reactions must be speeded up without changing its opposing reaction. The result is more or less product, without having achieved a completed reaction. Equilibrium may be shifted by adding an excess of a reactant or a product. Then the reaction speeds up in the direction that will tend to remove this excess.

Apparatus and Materials: Test tubes, evaporating dish, stirring rod, zinc, dil.HCl, silver nitrate solution, phenolpthalein solution, sodium hydroxide solution, ammonium hydroxide, ammonium chloride, medicine dropper.

Procedure: In a test tube add some dil.HCl to a small piece of zinc.

1.Equation...

2.Observation..

3.Should this tend to go to completion?.................

4.Why?...

In a test tube add a few drops of silver nitrate solution to half an inch of dil.HCl.

5.Equation...

6.Observation..

7.Should this tend to go to completion?.................

8.Why?...

Place about a quarter test tube of dil. HCl in an evaporating dish with a drop of phenolpthalein solution as indicator. Add NaOH solution drop by drop until a faint colour appears. Now add HCl drop by drop until the colour just disappears. Taste the solution.

9.Ionic equation...................................

10.Observation.....................................

11.What are the smallest particles that enter into chemical reaction in this case?.......................

12.Should this tend to go to completion?................

13.Why?...

....

Shifting the equilibrium: Consider the equilibrium

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$

If the backward reaction can be speeded up, there should be less OH^- ions remaining in solution, and their effect on an indicator should be less. The backward reaction can be speeded up by adding a great excess of one of the ions.

Add 2 drops of ammonium hydroxide to half a test tube of water, then add one drop of phenolpthalein solution. Divide the solution into two test tubes. To one of the samples add as much solid ammonium chloride as will dissolve with shaking. Compare the colours of the two solutions.

14.Has the equilibrium been changed by adding the ammonium chloride?.................................

15.How do you know?..................................

16.Which ion concentration has been lowered?

....

17.Which ion was added that caused this shift in equilibrium?

....

Note that when two electrolytes having the same ion are mixed in solution the ionization of the one is reduced. This is called the Common-Ion Effect.

Name............................Class.....Date.........

THE HALOGEN FAMILY: illustrating the Periodic Law.

Discussion: The periodic law states that properties of elements depend on their atomic number, but that the properties repeat themselves at intervals called periods. The result is that if elements are arranged in a series according to their atomic numbers, at regular intervals there occur elements that resemble preceding ones. In the periodic classification these similar elements are placed under each other to form a family. Their similarities illustrate the periodic repetition of properties. But in a family there is also a noticeable gradation of properties. Thus, in the non-metal families the activity of the elements decreases as one proceeds down the family. Such a gradation illustrates that in spite of periodic repetition of properties these depend on, and change with, the atomic numbers. This series of experiments will show the similarities and gradation of properties of the halogens: fluorine, chlorine, bromine, and iodine.

(a) Preparation

Apparatusaand Materials: Test tubes, sodium chloride, sodium bromide, sodium iodide, manganese dioxide, conc. H_2SO_4

Procedure: Fluorine is difficult to prepare and will not be attempted in these experiments.

In a test tube mix a quarter inch each of sodium chloride and manganese dioxide. Add an equal amount of conc. H_2SO_4 and heat gently. Carefully waft gas to the nose.

1.Colour of gas..............2.Odour of gas............

3.Equation..

Repeat the above, but use sodium bromide.

4.Colour of gas.............. 5.Odour of gas.............

6.Equation...

Iodine is normally a solid. Repeat the above, using sodium iodide. Hold the test tube almost horizontal, and heat gently. Watch for deposits of iodine on the cooler walls at the upper end of the test tube.

7.Colour of deposits.................................

8.Equations...

....

9.What similarity of the halogens has been illustrated?

....

(b) Gradation of Properties

Apparatus and Materials: Test tubes, evaporating dish, glass plate, wax, chlorine water, NaBr, NaI, carbon tetrachloride, CaF_2, conc. H_2SO_4, dil. HNO_3, NH_4OH, $AgNO_3$ solution.
Procedure: Dissolve about one-quarter inch of NaBr and NaI, each in separate test tubes, in half a test tube of water. Add to each half an inch of chlorine water and shake. Now add one-quarter inch of carbon tetrachloride, shake vigorously, and let stand to settle.

1.Observation...

2.Which of the halogens is the most active.............

3.Equations..

....

4.Why is carbon tetrachloride added?

....

5.Which would be more active, fluorine or iodine?........

6.Judging from the activity of the halogens, rank the following compounds in descending order of stability:

HI, HF, HCl, HBr...

7.Recall how HCl was made. How could HBr be made?

....

Try it with small portions in a test tube. Note colour and odour of escaping gases. HBr has no colour. Note if the odour of sulphur dioxide is present.

8.Observations...

9. Conc. sulphuric acid is a good oxidizing agent, while HBr, being unstable, is a good reducing agent. Write equations for the reactions that prevented you from making pure HBr.

....

....

10.Could HF be made like HCl?.........Why...............

11.Could HI be made like HCl?........Why?...............

Melt a small piece of wax on the surface of a glass plate so that it forms a thin layer over the whole surface. Scratch your initial <u>through</u> the wax. Place about half a teaspoon of calcium fluoride in an evaporating dish. Wet it thoroughly with conc.H_2SO_4,and immediately cover it with the glass plate,wax surface down. Do not sniff the fumes,they are dangerous. Let stand till the end of the period,or preferably for at least an hour. Heat the glass plate to melt off wax. Examine the plate for etching.

13.Equation for making HF.................................

14.Equation for etching..................................

Place four test tubes in a rack. Into them place 4-5 drops of NaF,NaCl,NaBr,NaI solutions respectively. To each add about two inches of water.To each add 3-4 drops of silver nitrate solution.

15.In which solutions is there precipitation?

....

16.Equations

....

....

....

Shake the solutions in which precipitation occurred,and split each into two parts in separate test tubes. To one part of each add an equal volume of NH_4OH. In which cases does solution occur?

17...

To the second part of each solution add several drops of nitric acid.Do the precipitates dissolve?

18...

19.How could you tell apart sodium chloride from sodium iodide?

....

....

Name..........................Class.....Date..........

HYDROLYSIS OF SALTS

Discussion: The reaction between the hydroxyl ion of a base with the hydrogen ion of an acid to form water which ionizes very slightly is called neutralization,and is the forward action of a very nearly complete reaction. But water ionizes very slightly to yield a few hydroxyl and hydrogen ions,which associating themselves with other ions,form a very minute amount of acid and base. This slight backward reaction to neutralization is called hydrolysis. Whenever a salt is dissolved in water hydrolysis occurs,forming an acid and a base.A strong acid or base ionizes nearly completely,a weak one very little.Thus if the result of hydrolysis is a strong acid and a weak base, there will be more hydrogen ions than hydroxyl ions set free,and the solution is acidic.The reverse will also happen. Thus it quite frequently happens that an aqueous solution of a salt,because of hydrolysis,is either acidic or basic.

Apparatus and Materials: Test tubes,blue and red litmus paper,stirring rod,the salts listed below.

Procedure: In separate test tubes dissolve not more than a quarter of an inch of each of the salts listed in half a test tube of water. Shake to hasten solution,but do not close test tube with your finger.By means of a stirring rod transfer a drop of each solution to both red and blue litmus paper.(Each paper may be used for several tests.)Rinse the rod before dipping it into another solution.Record your observations below:

Salt	Effect on Red Litmus	Effect on Blue Litmus	Acid Present	Base Present	Ion causing Colour change
$CaCl_2$					
$ZnSO_4$					
$NaC_2H_3O_2$					
NH_4Cl					
Na_2CO_3					

1.Salts giving acid reaction.................................

2.Salts giving base reaction.................................

3.Salts forming strong base+weak acid.................

4.Salts forming weak base+ strong acid.................

5.From the above make a rule by which you can predict whether an aqueous solution of a salt will be acidic or basic..

Name............................Class.....Date.........

COLLOIDS: Preparation
Coagulation
Emulsions

Discussion: A colloid is that condition of a mixture in which the dispersed phase(the material distributed in another substance) consists of particles composed of more than one molecule,yet too small to settle out. It is therefore at the borderline between a true solution and a mechanical mixture or suspension. The dispersed particles cannot draw together because by selective adsorption they have attracted ions all of the same charge. Colloidal particles are produced either by breaking down bigger pieces,or by condensing molecules to aggregations of colloidal size. Collois may be coagulated by neutralizing the charge on their particles,either by means of an electrolyte or an electric current.

(a)Preparation

Apparatus and Materials: Beaker,wire gauze,bunsen,test tubes,filter paper and funnel,stand,ferric chloride, very fine clay or slaked lime,sodium hydroxide solution.

Procedure: Heat about 100 ml.(graduate) of water in a beaker to boiling,(to remove carbon dioxide).Cool slightly, then add slowly about a quarter test tube of ferric chloride solution.Save for later.

1.Compare colours of this and ferric chloride solution

....

2.Hydrolysis has occurred;write the equation

....

3.What caused the colour of the colloid?

....

4.What keeps colloidal particles from growing big enough

to settle out?...

Filter about a quarter test tube of the prepared colloid.Does any of the dispersed phase remain on the paper?

....

5.Was the colloid formed by condensation or dispersion?

....

To about 100 ml.(graduate) of water in a flask add about 1 gm.(a quarter inch in a test tube) of finely powdered clay or slaked lime.Shake well.Filter about one-quarter test tube,and save the filtrate.Now add to the suspension about half a test tube of sodium hydroxide solution and shake vigorously.Again filter about one-quarter of a test tube.Compare the filtrates.

6.What difference do you notice?.......................

....

7.What was the purpose of the sodium hydroxide?

....

8.What name is given to a substance that acts as did the sodium hydroxide in this case?

....

9.Is this a condensation or a dispersion?

....

(b) Coagulation

Apparatus and Materials: Sample of ferric hydroxide colloid, aluminum sulphate, filter funnel and paper, gelatin.

Procedure: Place about an inch of ferric hydroxide colloid in each of two test tubes. To one add an equal volume of strong aluminum sulphate solution and shake. Set aside for 20 minutes and filter. Examine filter papers.

1.Observations...................................

2.How did the aluminum sulphate function?

....

3.Could any other salt have been used?..................

Repeat the above, only before adding the aluminum sulphate add to each test tube a few flakes of gelatin and boil.

4.Is there any noticeable difference between the two

samples this time?...............................

The gelatin is called a protective colloid.

(c) Emulsions These are colloidal suspensions of two mutually insoluble liquids. A third substance, called an emulsifying agent, is usually necessary to make the emulsion permanent.

Apparatus and Materials: Test tubes, kerosene, soap.

Procedure: To two test tubes half full of water add 4-6 drops of kerosene. To one sample add a small piece of soap (size of a pea or less). Shake vigorously and let stand for five minutes.

1.Can you notice any difference?........................

2.Why is the soap added?..............................

Name..........................Class......Date..........

FLAME TESTS FOR METALS

Discussion: Many elements may be identified by the characteristic colour their compounds impart to a gas flame. Flame tests are particularly useful in identifying metals of the alkali and alkali-earth families. Sodium imparts such an intense colour to a flame, and is so often found as a trace impurity, that its yellow tinge may be noticed even when testing other metallic salts. For more delicate work cobalt glass is used to screen out its effects. In this experiment test for it last.

Apparatus and Materials: Watch glass, platinum wire (fused into a glass handle), bunsen, conc. HCl, salts as below.

Procedure: Clean and adjust your burner to burn with a gentle non-luminous flame, only faintly blue in colour. Place some conc. HCl in the watch glass. Dip the platinum wire in and hold the wire in the flame. Repeat this until no colour is imparted to the flame. On separate filter papers get a few crystals each of the salts to be tested. Wet the platinum wire with HCl and hold it against a small crystal of salt so that it will adhere. Hold the wire in the flame just above the inner cone. Observe the flame colour. Clean the wire as before, and repeat with another salt.

Test for	Salt Used	Colour of the Flame
K	KNO_3	....
Li	$LiNO_3$	....
Ca	$CaCl_2$	....
Ba	$BaCl_2$	....
Cu	$CuSO_4$	....
Sr	$Sr(NO_3)_2$	....
Na	NaCl	....

1. Would it make any difference if different salts of each metal were used than those suggested?

....

Name............................Class.....Date.........

SAPONIFICATION

Discussion: Saponification is the manufacture of soap from organic fats or oils and a strong base, usually sodium or potassium hydroxide. A by-product is glycerin. The process is slow, taking several hours of boiling by the commercial method. The soap you will prepare will be impure, containing both glycerin and strong base.

Apparatus and Materials: Ring stand, wire gauze, bunsen, evaporating dish, beaker, test tube, stirring rod, olive oil (or cottonseed oil or lard), conc. sodium hydroxide solution, denatured alcohol, sodium chloride.

Procedure: Pour into an evaporating dish about one-half test tube of olive oil; add a test tube of alcohol. The alcohol serves only as a solvent, and takes no part in the reaction. Now add about one-half test tube of conc. sodium hydroxide solution. Heat the mixture, with occasional stirring, for 15-20 minutes. Do not allow the mixture to boil vigorously. If the alcohol ignites smother the flames by placing a beaker or glass plate over the dish. (There is no serious danger, since at worst you can put out the flame with water and start again.). Take care not to tip over the dish of hot mixture. Let the mixture cool, and while it is still warm pour it into a beaker nearly filled with saturated sodium chloride solution. Stir the mixture, collect the solid mass, and wash it several times in water. Squeeze the mass into a cake. See if it will form a lather.

1. Consider olive oil to be olein, and write the equation for the saponification of olive oil.

....

2. Name the by-product in this saponification

....

3. Soap in water forms mostly a colloidal suspension. What would be the purpose of adding to salt water as you just did?

....

....

Name.............................Class......Date........

HOW ARE METALS OBTAINED FROM THEIR OXIDES?

Discussion:Metal ores are roasted to change them into oxides. These are then reduced,usually by means of carbon. Commercially the carbon is in the form of coke (as in the blast furnace.)

Apparatus and Materials: Ring stand,clamps,hard glass test tubes,one holer rubber stopper to fit,delivery tube,bunsen,cupric oxide,charcoal,limewater,mortar a and pestle.

Procedure: Clamp the test tube to the stand;arrange stopper and delivery tube as for collecting oxygen or ammonia.Grind fine some charcoal,pour about an inch of it into the test tube,add about half an inch of cupric oxide,and shake to mix. Have the delivery tube lead into a second test tube containing limewater..Heat the mixture to dull red heat for ten minutes. What evidence is there of chemical action?

1...

2.Equation..

3.What is the escaping gas?.....................................

After the reaction seems to have ceased let the mixture cool and empty it into a mortar. Add some water,and grind with the pestle,pouring off what remains suspended in the water. Add more water and repeat this process several times. Look for copper metal in the residue remaining in the mortar.Sometimes hard grinding with the pestle will create a coppery smear on the inside of the mortar by which you can be sure you have reduced the cupric oxide to copper.

Name..............................Class.....Date.......

CALCIUM COMPOUNDS: Preparation of some common compounds of importance commercially.

Discussion: Limestone when heated changes into lime, which in turn can be changed to slaked lime by addition of water. These are used to make mortar, soften water supplies, and to make certain types of glass. Write formulae

for Chalk...........Lime..........Slaked lime...........

Apparatus and Materials: chalk, tongs, bunsen, litmus paper, calcium hydroxide, test tubes, apparatus for making carbon dioxide, conc. HCl.

Procedure: Hold a piece of chalk in the hottest bunsen flame for about five minutes, using the crucible tongs. Let it cool.

1. Equation for the reaction.............................

2. Commercially this is done in a device called a ....

Wet your finger and touch the part of the chalk that has been heated. Note any change in temperature. Touch the same part with a piece of wet red litmus paper.

3. Observations...

4. Equation...

5. When done commercially, what is this process called?

....

A solution of calcium hydroxide is called limewater. Make some by shaking about half a teaspoon of calcium hydroxide in a few hundred ml. of water (graduate), letting stand to settle, decanting and filtering the liquid. Set up apparatus to make CO_2. (Use pieces of chalk, conc. HCl) Fill a large test tube with limewater, arrange the delivery tube to reach to the bottom of this test tube. Bubble carbon dioxide into it. Equation for the reaction when the limewater turns milky:

6...

Continue bubbling CO_2 into the milky limewater until it turns clear again.

7. Equation...

Save about an inch of this solution; return the rest to your instructor who will put it aside for the experiment on the hardness in water. Boil the sample you saved, for about five minutes.

8. Observation..

9. Equation...

10. Why is this called temporary hard water?

....

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HARDNESS IN WATER SUPPLIES

<u>Discussion</u>: Hardness in water is caused by the solution of calcium,magnesium,or iron salts.If they are bicarbonates it is called temporary hardness,if not ,permanent hardness. Hardness is detected by its action onsoap;the soap forms a curdy precipitate,not a lather. The extent of hardness is measured by noting how many drops of a standard soap solution are needed to make a permanent lather.

<u>Apparatus and Materials:</u> Test tubes,medicine dropper, distilled water,soap solution,permanent hard water, temporary hard water,bunsen,filter paper and funnel,pipette.

<u>Procedure:</u> Soap solution: Dissolve 10 gms. of castile soap in 100 ml. of alcohol,dilute 20 ml. of this to 300 ml. with distilled water. Temporary hard water: Use the calcium bicarbonate solution made in the preceding experiment.Permanent hard water: Dissolve a few crystals of calcium chloride in a beaker (250 ml) of water.

(a) To 5 ml.(measure these quantities by pipette) of temporary hard water add soap solution by medicine dropper,drop by drop with shaking,until a lather forms that persists for one minute. Do not confuse a curdy precipitate that may float on top with a lather. Note the number of drops used.(b) Boil a test tube of temporary hard water for 5 minutes.Filter,and repeat test with 5 ml. of this.(c) To half a test tube of temporary hard water add limewater drop by drop with shaking until no more precipitate forms.Filter,and repeat test with 5 ml. of this. (d) Repeat test with 5 ml. of permanent hard water.(e) Boil a test tube of permanent hard water. for 5 minutes;filter,and repeat test with this.(f) To half a test tube of permanent hard water add strong sodium carbonate solution,drop by drop with shaking, until no more precipitate forms.Filter,and repeat test with 5 ml. of this.

	Drops of Soap Used		
	<u>Before Boil.</u>	<u>After Boil.</u>	<u>After Chem.Treat.</u>
Temp.Hardness	(a).........	(b).........	(c).......
Perm.Hardness	(d).........	(e).........	(f).......

1. In what ways may temporary hardness be removed?

....

2.Equation for boiling temporary hard water

....

3.Equation for limewater acting on temporary hardness

....

(Commercially slaked lime is used instead of limewater)

4.How is permanent hardness removed?..................

....

5.Equation for removing permanent hardness

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FERROUS AND FERRIC SALTS: Preparation
Tests

Discussion: Iron has the valences 2 and 3. The lesser valence is indicated by the ending ous, the greater by the ending ic. Metallic iron consists of neutral atoms Fe^0 In becoming an ion it loses 2 or 3 electrons to become either Fe^{++} or Fe^{+++} ion. This is oxidation. (Loss of Electrons=Oxidation LEO). For Fe^{++} or Fe^{+++} ions to revert to metallic iron Fe^0 they must pick up 2 or 3 electrons. This is called reduction.

(a) Preparation of Ferrous and Ferric Salts.

Apparatus and Materials: Filter paper and funnel, beakers, test tubes, stoppers, 250 ml. flask, powdered iron, nitric acid, hydrochloric acid, iron tacks or wire,.

Procedure: Place about 0.5 gms. of iron powder (about as much as would make one-quarter inch in a test tube) in a 250 ml. flask. Pour over it 5 ml. (graduate) of water, and 2 ml. (graduate) of conc. HCl. Mix the contents of the flask, heat gently, and set aside for about five minutes. The iron dissolves in the acid forming ferrous chloride.

1. Ionic equation...

2. What has been oxidized?............Reduced?...........

Now add 50 ml. of water to the flask, mix well, and filter off the undissolved iron. Nearly fill a test tube with the filtrate; add to it 4-5 drops of conc. HCl and a few iron tacks (or wire). Mark this solution A and save it for later tests. It is a solution of ferrous chloride. The acid and iron tacks generate enough hydrogen to keep it from becoming oxidized to ferric chloride.

To the remainder of the filtrate add approximately 1 ml. (graduate) of conc. HCl and heat to near boiling. Then add conc. nitric acid drop by drop with shaking until the solution, which is at first a dark-brown colour, becomes lighter in colour. The ferrous chloride has been changed to ferric chloride by the oxygen of the nitric acid.

$$4(Fe^{++},2Cl^-) + 4(H^+,Cl^-) + O_2 = 4(Fe^{+++},3Cl^-) + 2H_2O$$

3. What has been oxidized?.............Reduced?..........

Save the solution for the tests following.

(b) Tests for Ferrous and Ferric ions.

Apparatus and Materials: Test tubes, solutions of ferrous and ferric ions made i part (a), ammonium hydroxide, pot. ferricyanide, pot. ferrocyanide, pot. thiocyanate.

Procedure: Compare the action of the following reagents upon ferrous and ferric ions, adding 2-3 drops of the reagents to about one-quarter inch of ferrous and ferric solutions in separate test tubes.

Iron compounds-2

Observations:

Reagent	On Ferrous ion	On Ferric ion.
Amm. hydroxide	....	....
Pot.ferrocyanide	....	....
Pot.ferricyanide	....	....
Pot.thiocyanate	....	....

Equations:

1.Ammonium hydroxide acting on ferrous chloride

....

2. Ammonium hydroxide acting on ferric chloride

....

3.Formation of a blue ppte. with ferrous chloride

....

4.Formation of a blue ppte. with ferric chloride

....

(c) Blueprinting

Apparatus and Materials: Ferric chloride solution,oxalic acid solution(about 10%),pot.ferricyanide solution,paper beaker.

Procedure: In the presence of light the oxalic acid reduces the ferric chloride to ferrous chloride. In commercial blueprint this is done by a salt of citric acid.

In a beaker,well shaded from the light, mix half a test tube of each of ferric chloride and oxalic acid solutions.Add about one-inch in a test tube of pot. ferricyanide solution.

1.Any ppte.?........Should there be one?................

Wet a good quality writing paper with the solution,and dry it away from light. Place a key on the dry paper and expose to strong light for five minutes. Now wash the paper in water.

2.Is any change expected?..............................

3.Why?..

4.Equation..

....

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TESTING FOR METALS OF THE SILVER GROUP

Discussion: In chemical analysis it is common practice to separate the metal ions by groups or families. Silver, mercurous, and lead ions can be separated from a solution containing other ingredients by taking advantage of the fact that their chlorides are insoluble. It then becomes necessary to identify each metal ion in the group of insoluble chlorides by some further test.

Apparatus and Materials: Test tubes, filter paper and funnel, dil. HCl, pot. dichromate($K_2Cr_2O_7$), ammonium hydroxide, nitric acid, a sample solution made up of mixtures of mercurous, silver, and lead nitrates.

Procedure: Precipitate the chlorides from about half a test tube of the sample solution by adding to it about 5 ml. (graduate) of dil. HCl. Filter the mixture, discarding the filtrate. Wash the precipitate on the filter by filtering through it a few ml. of water acidified with a few drops of HCl.

1. Observations..

2. Equations..

....

....

Test for lead: Heat about a test tube of water to boiling and pour it through the residues remaining on the filter paper. Since lead chloride is soluble in hot water it will, if present, pass through the filter. Test the filtrate with a few ml. of pot. dichromate solution.

3. Observations..

4. Equations..

Test for mercurous ions Pour a few ml. of ammonium hydroxide on the precipitate remaining on the filter paper. Note change in appearance. This is caused by a complex ammonium mercurous ion. Save the filtrate.

5. Observation..

Test for silver ion: To the filtrate from the preceding step add nitric acid, a little at a time, till the odour of ammonia is gone. Observe change in appearance.

6. Observation..

NOTE: If time permits, your instructor will give you an unknown sample that may contain one, two, or three of the metal ions just tested. Using the steps above, discover what kind of ions are present in the solution.

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SOME IMPORTANT COMMERCIAL PROCESSES

It may be desireable to perform these as teacher demonstrations. Each process involves about a period of laboratory work.

Ostwald Process: the oxidation of ammonia in the presence of platinum as a catalyst. Make a coil with 11 inches of No.22 platinum wire by winding it around a pencil 8 times and bending the two ends so that the coil may be held horizontally in a beaker with its two ends hooked over the sides. In a 250 ml. beaker place enough conc. ammonium hydroxide so that the platinum wire will hang down in the beaker 1½ inches and still be 1 inch from the surface of the liquid. Heat the platinum coil to a bright cherry red and immediately place it in the position described. Cover top of beaker with a wire gauze. The coil should remain glowing, as the reaction is exothermic. Careful observation will show white fumes just above the liquid surface. This is evidence of nitric acid being formed and reacting with ammonia to form ammonium nitrate smoke.

1.Equations...

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Solvay Process: the commercial method of making sodium bicarbonate or sodium carbonate. Fit a large one-liter container with a two-holed stopper through which passes a right-angled glass tubing that extends to the bottom of the bottle. Place in the bottle 42 gms.(rough balance) of sodium chloride; add to this 100 ml.(graduate) of conc. ammonium hydroxide and an equal amount of water. Stopper the bottle and shake gently till the sodium chloride is dissolved. If necessary add more ammonium hydroxide till the sodium chloride is dissolved in the least amount of ammonium hydroxide. Now connect the glass tube with a long piece of rubber tubing to a CO_2 generator. Lift the rubber stopper and tubing out of the bottle far enough so that the lower end of the tube is just above the liquid surface in the bottle. Generate CO_2 and pass it in the bottle to fill it. Now lower the stopper and tubing in place, so that the tube extends below the liquid surface. Continue bubbling CO_2 through the liquid for about half an hour, shaking the bottle every few minutes to thoroughly agitate the liquid contents. The sodium bicarbonate formed will gradually precipitate out.

1.Equation..

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