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Institution: Oklahoma State University

Location: Stillwater, Oklahoma

Title of Report: IMPROVING CHEMISTRY INSTRUCTION

Pages in Report: 57 Candidate for Degree of Master of Science

Major Field: Natural Science

Scope and Method of Report: Reading research in the university library was done to try to determine the role and importance of chemistry in the high school curriculum. Laboratory research was employed to learn how standard chemistry demonstrations and experiments can be conducted successfully and effectively. Analytical procedures as given in high school laboratory manuals were surveyed and tested in the laboratory.

Findings and Conclusions: The high school chemistry course is accepted as a necessary part of the curriculum in all schools except very small ones where lack of personnel and equipment prevent its inclusion. Seemingly minor changes in procedures and techniques can produce noticeable improvement in an experiment. Suggested changes are included in the conclusions following the various experiments. Analytical procedures are an important part of the high school laboratory work in chemistry. A recommended minimum amount and variety of such procedures is made by the writer.

ADVISER'S APPROVAL ______________________

# IMPROVING CHEMISTRY INSTRUCTION

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Submitted to the faculty of the Graduate School of
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fulfillment of the requirements
for the degree of
MASTER OF SCIENCE
August, 1958

# IMPROVING CHEMISTRY INSTRUCTION

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# Preface and Acknowledgements

The purpose of this report is the same as that of the National Science Foundation Teacher Training Program. That of course is to improve the teaching of science and mathematics in our schools. A more specific purpose is to increase my efficiency as a teacher of high school chemistry.

It is not uncommon to find reports or articles relating to quickie demonstrations to be used in chemistry classes. However little work seems to have been done to revise and improve experiments which are used as an integral part of every high school chemistry course.

This report is a summary or survey of work done in the laboratory and reading done in the library aimed at promoting more successful and/or more effective demonstrations and experiments in my future chemistry classes.

I deeply appreciate the suggestions made by Dr. James H. Zant to guide me in the preparation of this report.

I also gratefully acknowledge the help of Dr. George Gorin and Mrs. Margaret Butler in connection with the laboratory work upon which this report is largely based.

The writing of this report has been made possible by the financial assistance provided by the National Science Foundation.

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# CHAPTER I

## CHEMISTRY IN TODAY'S HIGH SCHOOL

One of the most intriguing aspects of high school science teaching is the constant change in subject matter resulting from new discoveries, new inventions and especially new truths which are revealed. In a field like chemistry facts can never be treated as more than mere statements of our present status of knowledge.[1] Science as a whole and especially certain disciplines are changing so rapidly that the teacher who doesn't keep up with current advances not only fails to present current information but has no pool of knowledge on which to draw to meet the specific and changing interests of his class.[2] In no field of science does knowledge which one gains in college remain adequate for any substantial length of time. To coast on that knowledge provides totally inadequate background for the student being instructed.

It is alarming to professional school people as well as to laymen to read reports about the inadequate training of some of those now teaching high school science. The quality of our science teachers is crucial and so is their number.[3]

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[1] Frederick B. Dutton, "Editors Outlook," Journal of Chemical Education, 34:2, January, 1957.

[2] Alden H. Emery, "The Education Team," The Science Teacher, 22:10, February, 1955.

[3] Ibid.

A popular hypothesis regarding science teachers as reported by Harlow is that "high school teachers are and have been inadequately trained in science; the enrollments in high school science would have remained high if adequately trained science teachers could be retained in the schools and enrollments can be increased if effective on-the-job training in science is provided for science teachers."[4]

It is no doubt true that many high school science teachers are inadequately trained in science. It is certainly not true that all science teachers are inadequately trained. There is ample evidence that many teachers recognize a deficiency and desire additional training in science. An outstanding example is the large number of applicants for the National Science Foundation Training Program for Teachers.

To the laymen and even to non-science teachers it may seem inconsistent that science teachers work for advanced degrees in education rather than in science. An examination of the prerequisites for admission to courses granting graduate credit shows one good and sufficient reason that they do.[5] The preparatory work of the science teacher encompasses a wider base and less depth at any one point than that of other teachers.

The greatest assurance of sound teaching in any field is a thorough knowledge of subject matter. It has been reported that other factors being equal effective learning is more likely to occur when the teacher has a broad background of knowledge in the particular science he is

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[4] James G. Harlow, "The Secondary School Science Problem," _Science Education_, 41:114, March, 1957.

[5] Frederick B. Dutton, "Editors Outlook," _Journal of Chemical Education_, 33:313, July, 1957.

teaching as well as in related scientific areas.[6] However, it is just as unreasonable for the science teacher to contend that anyone who knows his subject matter can teach as it is for the educational faddist to contend that anyone who can teach can teach anything.

The quantity and quality of the offerings in science and mathematics in our high schools have a very direct bearing on our supply of scientists and engineers for industrial progress, teaching, national defense and fundamental research. In most cases, persons who make a career of scientific work discovered their interest before college, with the high school science class room and laboratory probably being the most important place for the awakening of that interest.[7]

Public education as a whole has experienced declines in percentage enrollment in the physical sciences. Even though they typically exhibit well-trained staffs, the larger city school systems have experienced about the same decline as smaller systems.[8] In some of these large systems a master's degree in the subject taught is a prerequisite to employment. Graduation requirement patterns have stabilized enrollment in general science and biology in many schools of all sizes.

Several suggestions have been advanced to explain low percentage enrollments in science courses, especially the advanced courses. Among these are: (1) students feel they are too difficult compared with other fields that are easier to enter and which pay as well or

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[6] Warren Maynard Davis, "Factors of Effectiveness in Science Teaching," Science Education, 38:151, March, 1954.

[7] Will S. DeLoach, "Chemistry and Physics Enrollments in Tennessee High Schools 1954-55," Science Education, 41:197, April, 1957.

[8] Harlow, p. 114.

better, (2) counselors are rarely familiar with or enthusiastic about the sciences, and (3) students have poor study habits and are ill-prepared to master the sciences when they reach the eleventh grade.[9] Telling a student he should take chemistry and physics if he plans to go to college, as is done in many high schools, does not give him any real reason for taking these courses.

Teachers must do a better job than ever before in making science a vital part of the total education of all young people. Chemistry is only a segment but a very important segment of science education. For success and satisfaction in modern living, the citizen must understand and appreciate the scientific aspects of his environment and the role of science in the development of our civilization.[10]

The first purpose of the high school chemistry course of today is to contribute generously and effectively to the general education of all students enrolled.[11] Because the products, methods, and objectives of chemistry have such a large influence on our daily lives, it would seem no one could be considered an educated person without some knowledge of the subject.[12]

The teacher of general chemistry has both the opportunity and the obligation of helping his students to see his subject in relation to

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[9] Willard L. McRoy, "Chemistry Instruction in California High Schools," Journal of Chemical Education, 34:139, March, 1957.

[10] Bernard Jaffe, New World of Chemistry (New York, 1955), p. iv.

[11] Robert H. Carleton and Floyd F. Carpenter, Chemistry for the New Age (Chicago, 1949), p. iii.

[12] George M. Rawlins and Alden H. Struble, Chemistry in Action (Boston, 1956), p. v.

the whole field of knowledge. The teachers who show the place of chemistry among the other natural sciences and the relation of science to other disciplines can help a great deal in reducing the intellectual provincialism among his students.[13]

Science courses, including chemistry, should be so taught that they condition the behavior of those who study them. As long as the acquisition of facts is the chief purpose of chemistry teaching, other important educational values such as development of desirable habits and attitudes will come out a poor second.

The study of science should cause all students to take a new interest in some of the common factors of our environment, while some may find sufficient interest in some phase of science to pursue its study and application as a life vocation. The high school chemistry course will acquaint students with the chemical nature of many of the materials of everyday living and with chemical processes used in the manufacture of many commercial products. Most students will acquire, in some degree, an appreciation for the methods of industrial and research chemists.

As has already been stated, the first purpose of high school chemistry is its contribution to general education. A second purpose, of only slightly less importance, is to help lay the foundation needed by some students for later specialization in science fields.[14] Subject matter and activities of the chemistry course are selected for their

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[13] Edward C. Fuller, "Chemistry In Perspective," Journal of Chemical Education, 34:110, March, 1957.

[14] Carleton, p. iii.

potential value in promoting growth in knowledge and understanding of the more important concepts and principals of chemistry, growth in skills and abilities involved in scientific problem solving, and its development of attitudes and appreciations characteristic of the scientific habit of mind.

A group of college teachers has listed five major pedagogical problems which all high school and college chemistry teachers must face:[15]

1. Teaching systematic thinking.
2. Making students science conscious and science curious.
3. Stimulating student imagination.
4. Instilling important principles of chemistry into the minds of students.
5. Providing experience in and appreciation for experimentation.

These problems are not peculiar to chemistry, but the chemistry teacher is in an excellent position to make important contributions to their solutions.

The high school chemistry teacher or other science teacher finds his task more complicated than it would otherwise be if he recognizes the importance of working toward differentiated objectives with the two groups of students comprising the typical class. These two groups parallel somewhat the two purposes of the chemistry course, given previously. The general education aspect is an important factor with both groups, while the specialization foundation is much more important to those students who may choose a vocation closely related to science than to those who will not.

A more penetrating understanding of the tactics and strategy of the scientific endeavor is a crying need of the day—not only for those

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[15] William T. Lippincott, "College Teachers Look at High School Science," _The Science Teacher_, 23:184, May, 1956.

who would become scientists of one kind or another, but for all youth growing up in a world characterized by the philosophy of science and the impact of scientific research and its applications. The high school chemistry course should be a personal study of chemistry in its applications to the life of each student regardless of his ultimate life's work. At the same time it should present the fundamental theories and concepts which are so necessary for those who elect a career that is based on science.[16]

Even as teachers stress the general education values of chemistry, they recognize their responsibility for the "science potential" among their students. There is more urgent need today than ever before to identify, nurture and develop the science-talented youth. Unfortunately or otherwise, one cannot always identify science potential. Some so-called "better students" achieve less than mediocre success in life, while others who had to plug away to keep up with the class develop into outstanding men in the workaday world.[17]

James G. Harlow, who is well-known in science education in Oklahoma, contends that today's high demand for technical and scientific manpower is in large part due to the low birth rate of the thirties and the currently high military demand. The effects of the first of these will certainly pass, relatively soon, he says, and the second could virtually disappear overnight as the aftermath of a sharp reduction in inter-

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[16] B. Smith Hopkins et al., Chemistry and You (Chicago, 1952), p. iii.

[17] Roger J. Williams, "Forty Ways To Be Dumb," Journal of Chemical Education, 34:261, June, 1957.

national tension. These facts illuminate a serious danger in planning a broadscale educational stimulation to meet short-range increases in demand. He cautions that heavy educational emphasis based on a current demand could, in a very few years, produce an extremely damaging public view of educational activity in this field as over selling.[18]

The tremendous growth in chemical knowledge and its applications creates a problem for the teacher of high school chemistry. There is a body of principles upon which the science of chemistry has been built. Development of control over the "fundamentals" and the ability to use them in explaining and predicting phenomena must be one of the major goals of instruction in high school chemistry. These fundamentals will enable young people to interpret and explain chemical aspects of the world in which they live and to gain competence in solving their problems of adjustment in the scientific world of today. Indeed there is a large body of chemical thought and reasoning which will always remain relatively unchanged and which must be thoroughly mastered as the basis of a chemist's training.[19] Presentation of this matter is an unforgettable duty.

Successful living in today's world demands that the citizen base his everyday thinking and actions on the best information available. The basic chemistry referred to above will help him to realize why the methods of science are superior to other methods of obtaining information. In fact, there is a universally accepted conviction that the

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[18] Harlow, p. 116.

[19] Raymond B. Brownlee, Robert W. Fuller, and Jesse E. Whitsit, Elements of Chemistry (Boston, 1954), p. ii.

greatest contribution science has to offer, for both the general and prespecialization student, is its method.

Add to the fundamentals the rapidly growing areas of plastics, textiles, metallurgy, petrochemistry, nuclear energy, biochemistry and organic chemistry. The task facing the chemistry teacher would appear to be an impossible one. Following a careful examination of the situation, Wisconsin chemistry teachers reached these conclusions:[20]

1. Chemistry teachers must continually and carefully examine the subject matter content of their courses with judicious revision in view.
2. If the high school chemistry course is to be kept up to date it seems obvious that the first requirement is that the teacher be up to date in his knowledge of chemistry and its applications.
3. Quality in teaching is more important than quantity; it is not a crime not to cover the entire textbook.

It is reasonable to assume that acquiring ability to use the methods of science can best be accomplished through allowing, or better still, requiring, students to practice the use of them.[21] It follows that the best place for such practice is in the laboratory phase of science teaching. The first step in learning the method of science is learning to distinguish carefully between what one observes and what one concludes from his observations.

Nothing can take the place of laboratory work in a beginning course in chemistry. No other science presents more attractive or more vivid phenomena than can be observed at first hand and from which the elementary theory of the subject can be built up by processes of simple

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[20] "High School Chemistry--Keeping the Course Up to Date," The Science Teacher, 23:407-8, December, 1956.

[21] Clarence H. Boeck, "Teaching Chemistry for Scientific Method and Attitude Development," Science Education, 37:81, March, 1953.

reasoning. The first experiment the student carries out should direct his attention to steps and procedures in scientific investigation and observation. Succeeding experiments should make these steps and procedures habitual and help the student understand and remember facts and principles.

Through experimental work students make simple chemical discoveries and applications. Through experimental verification they are convinced of the truth of the laws and theories of chemistry. The student repeats many of the classic experiments and thereby discovers the truths which the great pioneers established. The work must be done carefully, systematically and honestly, as it was done by the pioneers.

Authors of laboratory manuals endeavor to write directions for experiments in such a way as to emphasize the importance of correct procedures and good techniques. However, nothing can replace a good example set by teacher demonstrations or the personal direction of the teacher in developing laboratory skills. The teacher must constantly stress correct laboratory techniques.

There are certain experiments which for various reasons are generally carried out as demonstrations by the teacher or students. Some of these involve an element of danger, some involve difficult techniques, and some require skilled presentation to bring out the implications of the activity. The demonstration is one of the effective tools of science teaching. In general, a well-planned demonstration is superior to one which which the teacher has had little or no experience.

There are many recognized functions which classroom demonstrations in science can and do serve.[22]

a. To illustrate a principle or a fact. (most common use)
b. To visualize processes.
c. To show materials and specimens.
d. To portray methods and techniques.
e. To stimulate interest.
f. To create a problem situation.
g. To find information.
h. To evaluate student achievement.

Fewer laboratory exercises, experiments and demonstrations of better design will pay bigger dividends than a larger number done in cookbook style.[23]

Demonstrations, individual experiments, textbook study and library reference work must all be integrated. Otherwise much of the value of each is lost.

It would be well for the teacher of high school chemistry to keep in mind that the truly effective teacher seldom, if ever, was born that way.[24] Neither is the most successful chemistry teacher necessarily the one who knows the most chemistry.[25] The most effective teaching is that which touches the spirit.

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[22] Robert Stallberg, "Science Demonstrations for Improved Learning," The Science Teacher, 22:277, November, 1955.

[23] "High School Chemistry--Keeping the Course Up to Date," The Science Teacher, 23:408, December, 1956.

[24] Frederick B. Dutton, "Editors Outlook," Journal of Chemical Education, 34:365, August, 1957.

[25] Williams, p. 261.

# CHAPTER II

# CHEMISTRY DEMONSTRATIONS

This portion of the project was to determine how certain chemistry experiments can be made more successful and/or effective. The experiments chosen were those indicated as "Teacher Demonstration" in all or nearly all of the laboratory manuals examined. They are designated as demonstrations for various reasons such as a certain amount of danger or unpleasantness (in case of halogens) or because strict attention must be given to details. In many cases the entire purpose of an experiment is defeated if it is not related, by explanation, to a practical application. A great deal of work was done in setting up for each demonstration a concrete and clearly stated purpose based upon its applications.

Six popular laboratory manuals and accompanying textbooks were carefully examined before each experiment was carried out in the laboratory. These were found on the National Science Foundation reading shelf in the library. The manuals are listed at the end of this chapter. Reference to the manuals will be made by number.

## Reduction

Purpose: To demonstrate on a small scale one of the important reactions used in metallurgy, the science that deals with obtaining metals from their ores. Three-fourths of all natural elements are metallic, and oxides are one of the most important types of ores. The demonstration therefore has two purposes. The immediate purpose is to obtain metallic copper from copper oxide and the fundamental long-range purpose is to help students gain an understanding of the reduction process.

Procedure and Result: There are two experiments commonly used to illustrate reduction. One is the reduction of steam by hot iron (1:31).[26] The second is the reduction of copper oxide by hydrogen (3:31), (5:27), (6:24).

To reduce copper oxide a hydrogen generator was set up. This consisted of a generating bottle in which mossy zinc was placed. The bottle was fitted with a two-hole stopper, with a thistle tube for adding sulfuric acid and a delivery tube for escape of hydrogen. The delivery tube was attached to a drying tube containing calcium chloride. Weaver (6:61) suggests that anhydrous calcium sulfate could be used as the drying agent. From the opposite side of the drying tube there was extended a glass tube to deliver the dry hydrogen into the test tube containing the copper oxide to be reduced. When the apparatus was fully assembled and properly supported so that the open end of the test tube was slightly lower than the closed end, dilute sulfuric acid was added to the generator so that the end of the thistle tube was covered by the liquid. After two minutes the copper oxide was heated in the test tube by applying a Bunsen burner. As the copper oxide was reduced it changed in color from black to pink. When the color was completely changed the burner was removed but hydrogen was passed over the reduced

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[26] Refer to manual number one, page 31, as listed at end of chapter.

residue while it cooled. The cooled residue was removed and a small area polished by rubbing on a smooth glass surface.

To reduce steam by iron an apparatus was set up consisting of a flask half filled with water supported over a Bunsen burner, an ignition tube in which powdered iron was placed to be heated by a second Bunsen burner with fishtail and a trough for collecting gas by water displacement. When the apparatus was assembled the two burners were lighted so that steam was passed by the delivery tube into the ignition tube, over the heated iron and out through another delivery tube into the trough. After about two minutes, samples of the escaping gas were collected and tested with a burning splint.

Both procedures were carried out successfully when certain precautions were taken. Hydrogen from the generator gradually reduced the copper oxide to copper. Weaver (6:24) called for stick form of the oxide but result was not different from powdered form. The copper could be identified by its properties.

Steam was reduced by the heated iron. The gas when tested with a burning splint gave an explosion which is characteristic of hydrogen. Students would have previously prepared hydrogen in the laboratory. The product in the ignition tube can be tested with wet red litmus paper which turns to blue, indicating that it was a basic anhydride ($Fe_2O_3$).

Conclusion: Both reactions should be used to demonstrate reduction so that students will know reduction is not applied only to reduction of metallic oxides. It would be well to mention that other applications of the term involve no oxygen but only a change in valence (studied later in course). To insure success rather than a mishap, precautions should include wrapping the hydrogen generator with a towel, waiting until apparatus is cleared of air before heating the copper oxide, keeping flame away from open end of test tube, and waiting until residue is cooled before removing from hydrogen.

Suitable amounts of reactants are two grams of copper oxide to be reduced and twenty grams of mossy zinc to be used in the hydrogen generator.

If accurate balances are available, the test tube should be weighed before (with its CuO) and after (with its Cu residue).

Equations: $H_2SO_4 + Zn \longrightarrow ZnSO_4 + H_2$

$CuO + H_2 \longrightarrow Cu + H_2O$

$4H_2O + 3Fe \longrightarrow Fe_3O_4 + 4H_2$

$Fe_2O_3 + 3H_2O \longrightarrow 2Fe(OH)_3$

## Chlorine—Its Preparation and Properties

Purpose: To illustrate that an element which never occurs free in nature can be prepared from its compounds in large amounts for commercial use. While most free chlorine is obtained from sodium chloride by electrolysis, its liberation from hydrochloric acid is a convenient method for use in the laboratory. Much chlorine is used for bleaching and for making bleaching compounds. This action is used as a part of the experiment. Additional uses are in purification of drinking water and in manufacture of various compounds.

The purpose can be said to be threefold: to prepare an element from one of its compounds, to illustrate combustion without oxygen, and to illustrate oxidation without oxygen.

Procedure and Result: This experiment was included in all manuals used: (1:61), (2:87), (3:237), (4:45), (5:181), and (6:95). All manuals called for perparation of chlorine gas by oxidation of concentrated hydrochloric acid by manganese dioxide. Four manuals (1, 2, 3, and 5) called for the collecting bottles to be arranged in series. Reacting materials were to be placed in a flask. One (No. 4) called for filling the bottles one at a time and putting them aside, right side up, with glass cover plates over the mouths of the bottles. One manual (no. 6) called for the reaction to be carried out in a test tube lying almost horizontally, with a delivery tube to carry the chlorine into a trough of water, to be collected in four test tubes by displacement of water. Both procedures were satisfactory but the first is difficult to manipulate when bottles are removed from the series.

Various substances, including sodium, hydrogen, antimony, iron (steel wool), copper, and magnesium were made to combine with chlorine by heating or burning. The bleaching action of chlorine was tested on colored cotton cloth, litmus paper, ink writing on paper, and newspaper print. All except the printer's ink were bleached to some extent.

Conclusion: A steady flow of chlorine gas from the generator can be had by placing the flask holding the reactants in a container of water. (A coffee can was used.) The generator is thus kept at a constant temperature, the boiling point of water. The collection of one bottle of gas at a time does not allow any more chlorine to escape than the series method if the collecting bottle is set on some support such as an overturned coffee can or beaker. The support is moved from under the bottle and the filled bottle removed and replaced. The support is again placed under the bottle in which gas is being collected. A circle of cardboard through which the delivery tube passes remains in place and covers each bottle as it is being filled with gas. Each filled bottle should be quickly covered with a glass plate. (A little chlorine in the room and hallways advertises the chemistry department.)

Since there are several reactions that can be carried out to show the properties of chlorine, it would be well to prepare several bottles of the gas ahead of time and seal them with waxed corks. Then the generator need be run only long enough for the class to see the method of preparation. Bottles of pure chlorine can be prepared if the gas is collected by displacement of water. Contrary to popular belief, chlorine is not very soluble in water.

There is always a certain amount of danger when a burning jet of hydrogen from a hydrogen generator is used. A jet of natural gas can be arranged by removing a Bunsen burner from its rubber tube and inserting the larger end of a blow pipe into the tubing. Gas issuing from the smaller end can be lighted and inserted into a bottle of chlorine.

To bleach most materials they must be left in the chlorine water several hours or overnight.

A small amount of chlorine can be made by putting a little bleaching solution (sodium hypochlorite) in a bottle and adding a little concentrated hydrochloric acid. Cover with a glass plate, leaving a small opening for air to escape.

Equations: $MnO + 4HCl \longrightarrow 2H_2O + MnCl_2 + Cl_2$

$2Na + Cl_2 \longrightarrow 2NaCl$

$H_2 + Cl_2 \longrightarrow 2HCl$

$$2Sb + 3Cl_2 \longrightarrow 2SbCl_3$$

$$2Fe + 3Cl_2 \longrightarrow 2FeCl_3$$

$$Cu + Cl_2 \longrightarrow CuCl_2$$

$$Mu + Cl_2 \longrightarrow MgCl_2$$

$$CH_4 + 2Cl_2 \longrightarrow 4HCl + C$$

$$H_2O + Cl_2 \longrightarrow HCl + HClO$$

$$NaClO + 2HCl \longrightarrow NaCl + H_2O + Cl_2$$

## Bromine—Its Preparation and Properties

Purpose: To acquaint students with the only nonmetallic element which normally exists as a liquid. Also to show students the similar and dissimilar properties of two members of the halogen family. In addition the displacement of bromine from its compounds by chlorine is shown as a part of the experiment. This same method on a larger scale is used to produce large quantities of bromine for industrial use, especially in the manufacture of antiknock gasoline, organic dyes, sedatives, and silver bromide which is used in photography.

Procedure and Result: There are two methods used for preparation of bromine, one using a test tube generator (1:65), (2:93), (5:189) and (6:103), and one using a retort to hold the reactants (4:49), (5:189) and (6:103). The reaction may be carried out in a generating flask (3:249).

All methods call for reacting a mixture of manganese dioxide and sodium bromide or potassium bromide with sulfuric acid.

The bromine produced is collected in a test tube filled with cold water supported in a beaker of cold water.

The quantities of the dry materials called for varied from one gram of each to five grams of each, and the amounts of acid from just enough concentrated acid to moisten the dry chemicals to ten milliliters of concentrated acid. Consequently the different procedures gave varying amounts of bromine.

Tests made with the bromine included the following:

Two drops liquid bromine were placed in a beaker. A small amount of powdered antimony (warmed) sprinkled into the beaker reacted with the bromine vapor vigorously, producing a light (sparks) and heat.

One drop of liquid bromine was placed in three milliliters of carbon tetrachloride. This reddish-brown solution was then added to an equal volume of bromine water (pale yellowish-brown) and shaken. The water became clear because bromine dissolves preferentially in the carbon tetrachloride. This was repeated using chloroform and carbon disulfide. Similar results were obtained.

To five milliliters of bromine water a solution of sodium hydroxide was added drop by drop. The color of the mixture gradually faded as the free bromine changed to bromide and bromate ions which are colorless.

A small crystal of potassium bromide was added to three milliliters of water forming a colorless solution. Three milliliters of carbon tetrachloride were added. The mixture remained colorless. Three milliliters of chlorine water were added and the new mixture was shaken. The carbon tetrachloride formed a layer in the bottom of the tube and was reddish brown due to the bromine displaced by the chlorine.

Two milliliters of bromine water were added to two milliliters of potassium iodide solution. The mixture was clear, indicating that the free bromine had gone into some compound. Addition of small amount of carbon tetr chloride indicated presence of displaced iodine by its purple color.

A small piece of rubber tubing was placed in bromine water in a test tube which was then closed with a stopper. After two days the rubber was found to be darkened and very brittle, indicating it had been oxidized by the bromine.

Three drops of bromine were placed in a gas bottle and warmed gently. A burning splint was inserted in the vapor formed. The splint continued to burn.

Conclusion: The experiment is worthwhile and can be carried out as a demonstration in a ventilated classroom with safety. The reactants should be heated until the brown fumes have cleared out of the retort or generating test tube. Small amounts of bromine, bromine water, and chlorine water should be prepared ahead of time so that more time can be given to the tests for properties of bromine.

The most satisfactory amounts of reactants are three grams of sodium bromide, three grams of manganese dioxide and ten milliliters of dilute sulfuric acid.

A small amount of fresh chlorine water can be prepared by bubbling chlorine gas through water, or by adding a few drops of concentrated hydrochloric acid to three or four crystals of potassium chlorate. The test tube is allowed to fill with chlorine gas and water is then added.

It should be pointed out to the class that more than one reaction may occur in the preparation of bromine and that a series of equations can be written for these possible reactions.

Some authors indicate that the potassium sulfate formed in the preparation of bromine is the bisulfate of potassium.

Equations: $2KBr + H_2SO_4 \longrightarrow K_2SO_4 + 2HBr$

$4HBr + MnO_2 \longrightarrow 2H_2O + MnBr_4$

$MnBr_4 \longrightarrow Br_2 + MnBr_2$

$MnBr_2 + H_2SO_4 \longrightarrow MnSO_4 + 2HBr$

or the summary equation:

$2KBr + MnO_2 + 2H_2SO_4 \longrightarrow Br_2 + K_2SO_4 + MnSO_4 + 2H_2O$

$2Sb + 3Br_2 \longrightarrow 2SbBr_3$

$Br_2 + 2NcOH \longrightarrow NaBr + NaBrO + H_2O$

$2KBr + Cl_2 \longrightarrow 2KCl + Br_2$

$2Kl + Br_2 \longrightarrow 2KBr + I_2$

## Iodine--Preparation and Properties

Purpose: To show a third member of the halogen family which, unlike chlorine (a gas) or bromine (a liquid), is a solid at room temperature. To show further the three elements have similar chemical properties and are prepared from their compounds by identical procedures except for the methods of collection of the free elements. To point out to students who do not already know that the liquid commonly called iodine is really a solution of iodine in alcohol called a tincture. The displacement of iodine by chlorine and by bromine is also shown.

Procedure and Result: In all procedures a mixture of potassium iodide (or sodium iodide), manganese dioxide and sulfuric acid were heated in a container with the liberated iodine allowed to condense on some cool object or a cool portion of the test tube.

Two manuals (1:69) and (2:95) called for the mixture to be heated in a test tube in an almost horizontal position. With heat applied only at the closed end of the test tube, iodine vapors condensed on the cooler sides of the upper open end. Three manuals called for the mixture to be heated in a beaker covered by an evaporating dish in which there was cold water. Two manuals asked that the mixture be heated in an evaporating dish covered by a flask partially filled with cold water (6:105) or by an inverted funnel with a cotton plug closing the upper end.

Amounts of dry reactants varied from one-half gram of each to five grams of each, and amounts of acid from two milliliters dilute to ten milliliters of concentrated acid.

Tests used to study the properties of iodine included the following:

The starch-iodine test in which a drop of iodine solution (in potassium iodide and water solution) was added to a thin solution of starch, producing a dark blue color. This is used as a test for either starch or free iodine.

Solubility of iodine was checked in various liquids with results as indicated:

In $H_2O$, very slightly soluble, very pale brown.

In KI solution, soluble, light brown.

In alcohol, soluble, dark brown.

In $CS_2$, soluble, purple.

In $CCl_4$, soluble, purple.

Sublimation was shown by heating some of iodine and allowing the vapor to sublime on the sides of the test tube.

A bit of iodine in sodium hydroxide solution formed a colorless mixture indicating formation of colorless ions.

A piece of white cloth stained with tincture of iodine was placed in a hypo solution. The color faded but did not disappear.

A small bit of iodine was heated in a beaker. A bit of powdered antimony sprinkled into the vapor combined vigorously with the iodine.

Carbon tetrachloride was added to a colorless solution of potassium iodide. The mixture remained colorless. When chlorine water was added, the mixture took on a purple color, indicating presence of displaced iodine. The test was repeated using bromine water to displace iodine from potassium iodide.

The test was repeated twice more using alcohol in place of the carbon tetrachloride. The displaced iodine produced a brown mixture.

Conclusion: The important uses of iodine should be emphasized in connection with this experiment. One of these is in the physiology of man and is the reason for the preparation and use of iodized salt. Iodine and its compounds are important in the manufacture of many substances including drugs, organic dyes, photographic compounds and Polaroid glass.

This experiment, as well as the previous one (bromine), shows the difference between atoms and ions in the displacement tests.

On the basis of the displacement relationship of the three halogens, their relative activity can be established. In turn the heats of formation of related compounds as sodium chloride, sodium bromide and sodium iodide can be compared.

The heating of the reactants in a beaker covered with a cooled evaporating dish is a convenient and satisfactory method of preparation. A suitable ratio of reactants is one gram of the iodide and dioxide with two milliliters of dilute acid. This produces a sufficient amount of solid iodine and yet avoids an excess of vapor.

In making the solubility and displacement tests, carbon tetrachloride should be used rather than carbon disulfide because of the flammable nature of the latter.

Equations: $2KI + MnO_2 + 2H_2SO_4 \longrightarrow MnSO_4 + K_2SO_4 + 2H_2O + I_2$

$2NaOh + I_2 \longrightarrow NaI + NaIO + H_2O$

$3I_2 + 2Sb \longrightarrow 2SbI_3$

$2KI + Cl_2 \longrightarrow 2KCl + I_2$

$2KI + Br_2 \longrightarrow 2KBr + I_2$

### Electrolysis

Purpose: To acquaint students with the important process of electrolysis in which a compound is separated into component parts. An important application is the electrolysis of copper sulfate with copper electrodes to obtain free copper. Many other elements and compounds are produced by the tons by this method. Since students would have previously prepared and studied both oxygen and hydrogen, they will readily recognize these products from their properties.

Procedure and Result: Two methods were given in manuals (1:35), (2:19) and (5:45). These made use of the Hoffman apparatus or a battery jar set up. All three favored the use of the Hoffman apparatus. One manual (4:21) gave instructions only for the battery jar apparatus. One manual (6) did not include an experiment for electrolysis but described the Hoffman procedure in the accompanying text book.[27]

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[27] Elbert C. Weaver and Lawrence S. Foster, Chemistry for Our Times (New York, 1954), p. 69.

The Hoffman apparatus was supported on a ringstand with two clamps. The apparatus was filled with distilled water to the bottom of the stopcocks on the side tubes. These were then closed and a little more water added to the center tube to check for leaks around the stopcocks. The electrodes of this apparatus were of platinum foil. These were connected by copper wire to a battery charger, which in turn was plugged into a wall socket. When the current was allowed to flow through the pure water, only a very slow reaction was detected. The water was drained from the apparatus and dilute sulfuric acid was added in ratio of one part acid to twenty-five parts water. More acid was added later to observe whether ratio of reaction changed as concentration was increased.

Current was again passed through the solution. When 30 ml. of hydrogen had been liberated, the current was shut off and the volumes of the two gases were compared and found to be almost exactly two to one (30 ml. of hydrogen and 14.9 ml. of oxygen). The hydrogen was allowed to flow slowly into an inverted test tube. When the test tube was brought to a flame there was an explosion. The current was again passed through the solution until about 30 ml. of oxygen had been liberated. This was allowed to pass into a test tube and tested with a glowing splint which burst into flame. A comparison of volumes again showed a two to one ratio, 30 + 30.1 ml. of hydrogen and 30 ml. of oxygen.

Because of difficulty in locating material for electrodes, in sealing copper wire into glass tubing, and in setting up a suitable apparatus, the battery jar procedure was not carried out.

Conclusion: The importance of the electrolysis process would justify the purchase of the Hoffman apparatus for carrying out this experiment. While it would not be used many times each year, it takes up little space and is quickly and easily assembled. The difficulty of setting up the battery jar apparatus makes it impractical.

In using the Hoffman apparatus the liberated oxygen should be passed from the apparatus through a rubber elbow and short piece of rubber tubing into a test tube. Because oxygen is slightly heavier than air it cannot be forced upward into a test tube as is the hydrogen.

Equations: $2H_2O \longrightarrow 2H_2 + O_2$

$2H_2 + O_2 \longrightarrow 2H_2O$ (test for $H_2$)

$C + O_2 \longrightarrow CO_2$ (test for $O_2$)

## Thermit Process

Purpose: To demonstrate an important oxidation-reduction reaction used to reduce oxides of certain metals which are otherwise difficult to reduce. These include oxides of magnesium, chromium, titanium, silicon, boron, manganese, and uranium. The thermit process, sometimes called the Goldschmidt Process, is a common way of manufacturing or producing these metals.

To show further how the same method, using iron oxide, can be used as a welding process in which the resulting molten iron is allowed to flow into a break in some heavy metal object. The break is surrounded by a clay mold. The process is used in the repair of rails, heavy machinery, ship hulls, propeller shafts, rudder frames, and locomotive parts. Such objects cannot be taken into a shop, and repairs must be made on the spot.

In war time this process has been the basic principle of incendiary bombs.

Procedure and Result: Following the only manual (1:179) which gave satisfactory instructions for the process, the thermit mixture was prepared by mixing eleven grams of ferric oxide with eight and one-half grams of aluminum oxide. Two sheets of filter paper were folded as for use in a funnel and wet with water. The thermit mixture was poured from the paper on which it was mixed into the filter paper which was then placed inside a small ring supported on a ring stand. A coffee can of clean sand was placed beneath the ring. A three-inch piece of magnesium ribbon was stuck into the center of the thermit mixture. Around the bottom of this fuse was placed an ignition mixture of potassium chlorate and magnesium powder. Amounts of these last two chemicals was not specified so about one-fourth teaspoonful of each was used. This was packed down into the thermit mixture so that it would not ignite from the burner flame used to ignite the upper end of the magnesium fuse.

The magnesium ignited the ignition mixture which ignited the thermit mixture. There was a dense cloud of white smoke and some bits of material were thrown out into the air but fell harmlessly onto the table top and concrete floor within a small radius.

After all material had cooled an acorn-shaped mass of metal was found in the sand. This was broken open as directed in another manual (4:97) and tested with a magnet. The inner part was attracted to the magnet, but the glasslike outer shell was not. Only the inner portion was the reduced iron.

One other manual mentioned above gave directions for "Reduction of an Oxide with Aluminum," but failed to name or give amount of reactants to be used in the thermit mixture. In this procedure the filter paper cone with its contents was to be placed down in the sand.

Using the same procedure described above, the experiment was repeated with the material placed in the coffee can of sand. There was no noticeable difference in the results.

No other manual of the six included in this survey gave instructions for carrying out the experiment. In both of the two which did give it, the experiment was designated as a teacher demonstration. However, all six of the associated text books had some material on the process.

One text[28] explains the process in terms of the great attraction of aluminum for oxygen and mentions its use in thermit welding and in the incendiary bomb. Another text[29] emphasizes the great heat of the reaction which results in molten iron and describes and illustrates the welding process. All other texts had an illustration of either the welding process or the incendiary bomb.[30]

Conclusion: This experiment should be carried out as a demonstration with full explanation and not as a "quickie" used only as a spectacular. If it is used as a quickie it should be repeated following, or with, proper explanation. This is warranted by the important

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[28] Jaffe, p. 394.

[29] Oscar E. Lanford, Using Chemistry, (New York, 1955), p. 616.

[30] Weaver and Foster, p. 426.

commercial applications of the process.

It should be explained, or better yet demonstrated, that the flying bits of material will char or even set fire to paper they fall upon. In the incendiary bomb an enclosing case is made of magnesium which is blown into thousands of extremely hot fragments which set fire to any flammable material they fall upon.

Overly adventurous students can be discouraged from trying the experiment on a larger scale or from trying to make a bomb by explaining that several cases have been reported in which sight or limbs have been lost by amateur bomb makers. But the best insurance is to keep all chemicals which may be dangerous under lock and key.

Equation: $2Al + Fe_2O_3 \longrightarrow Al_2O_3 + 2Fe$

## Chemical Combinations

Purpose: To show that the substance(s) resulting from a chemical reaction has (have) properties unlike those of the substance(s) entering into the reaction. To do this, iron and sulfur were combined to form iron sulfide and zinc and sulfur were combined to form zinc sulfide.

Procedure and Result: Sulfur (powdered) and iron (filings or powder) were heated together as instructed in reference manuals (1:15), (2:107), (3:17), and (5:28). Sulfur (powdered) and zinc powder (dust) were mixed and ignited (on asbestos) as directed in manuals (1:140), (2:107), (4:9), (5:205), and (6:39). No iron sulfide was formed using iron filings as directed in manuals (2) and (3). No iron sulfide was found in the product formed using one-half test tube of sulfur and one-fourth test tube of iron powder as directed in manual (1). Manual (5) called for seven grams iron powder and four grams powdered sulfur. When heated these quantities gave an initial glow which spread throughout the mass and produced one large lump of iron sulfide and only a small amount of sulfur fumes.

When equal bulk of sulfur and zinc were mixed and ignited as instructed in manuals (1) and (2) there was a steady blue flame which spread over the surface of the mixture. The sulfur burned away, leaving molten zinc which quickly hardened. When a mixture of sulfur and zinc in a 2/1 ratio by weight was ignited as instructed in manuals (4), (5), and (6) there was a vigorous reaction which produced a mushroom cloud of white vapor. The powdery white residue was zinc sulfide.

Products of both reactions, iron sulfide and zinc sulfide, can be verified (as sulfides) by checking with hydrochloric acid for formation of hydrogen sulfide which is readily detected by its odor.

Conclusion: Heating iron and sulfur together gives the desired result, formation of iron sulfide, when the two elements are used in a 2/1 ratio by weight. The slight excess of sulfur, as compared to the 56/32 ratio of their atomic weights, is necessary because some sulfur vaporizes before the chemical combination begins. Also it is necessary to use iron in the form of iron powder in order to obtain any appreciable amount of iron sulfide. If iron filings are used, all of the sulfur vaporizes before any chemical reaction takes place.

Zinc dust and powdered sulfur will react vigorously if used in a 2/1 ratio by weight. An excess of zinc slows the reaction.

Equations: $Fe + S \longrightarrow FeS$

$$FeS + 2HCl \longrightarrow H_2S + FeCl_2$$

$$Zn + S \longrightarrow ZnS$$

$$ZnS + 2HCl \longrightarrow H_2S + Zn\,Cl_2$$

## Titration

Purpose: To show how the strength expressed as normality or molarity can be determined. Also to emphasize that a normal solution of any acid contains one gram of replaceable hydrogen and a normal solution of any base contains seventeen grams of hydroxyl per liter. Thus solutions of all acids and bases of equal normality have the same neutralizing ability per milliliter. If an acid or base of known concentration is available (or it can be prepared) it can be used to determine the concentration of a basic solution which in turn can be used to determine the concentration of another acid of unknown concentration.

Procedure and Result: Titration of an acid and a base was carried out as instructed in manuals (1:93), (4:69), (5:111), and (6:79). All four manuals specified sodium hydroxide (NaOH) and hydrochloric acid (HCl) except manual (6) which did not name any specific acid but called for an unknown acid of unknown normality. Normality or molarity was satisfactorily determined by each of the methods which were basically the same.

Conclusion: The lower the concentration of a solution, the greater will be the error introduced by inaccuracy in weighing out the chemical (acid or base) used in the standard solution. On the other hand, the lower the concentration, the greater the accuracy attained by titration, assuming that the end point is correctly determined. With an accurately standardized solution the concentration of an unknown solution can be determined. In turn, the amount of a chemical contained in a solution can be found.

Using this experiment in high school, students should be instructed to calculate molarity (or normality) at least twice. One titration should be made of acid with base to a pink end point and one of base with acid to a clear end point. The mean of the two (or more) trials should be considered the correct value.

Equations: $NaOH + HCl \longrightarrow NaCl + H_2O$

$$N\ (\text{base}) = \frac{V\ (\text{acid})}{V\ (\text{base})} \times N^2\ (\text{acid})$$

$$N\ (\text{acid}) = \frac{V\ (\text{base})}{V\ (\text{acid})} \times N\ (\text{base})$$

## Ammonia Fountain

Purpose: To show the extreme solubility of ammonia (gas) and to show one use of an indicator (phenolphthalein).

Procedure and Result: Fountains were made by two methods. To prepare ammonia, nine grams of ammonium chloride ($NH_4Cl$) and seven grams of calcium hydroxide ($Ca(OH)_2$) were mixed and heated in a test tube. Ammonia ($NH_3$) was collected in an inverted flask. In the second method, a small amount of ammonium hydroxide ($NH_4OH$) solution was heated in a flask to liberate ammonia which filled the flask. Each flask was closed with a one-hole stopper and glass tube, then inverted in a beaker of water to which a few drops of phenolphthalein had been added.

No instructions for this demonstration were given in the manuals but reference to the ammonia fountain was made in textbooks which generally accompany the manuals.

A very weak (slow) fountain resulted from the first method when the flask was cooled with paper towels wet in ice water. The second method gave a beautiful result, a forceful pink fountain.

Conclusion: Neither method can be considered very successful in showing the great solubility of ammonia, which is its purpose. By the first method the pressure of ammonia inside the flask cannot be made greater than that of the atmosphere. Results should be much better if ammonia were obtained from a cylinder and used in the fountain before the pressure is lost. The fountain produced by the second method is probably due more to the partial vacuum in the flask resulting from heating the flask and then allowing it to cool than to solubility of ammonia.

Equations: $2NH_4Cl + Ca(OH)_2 \longrightarrow CaCl_2 + 2NH_3 + 2H_2O$

$NH_3 + H_2O \longrightarrow NH_4OH$

$NH_4OH \longrightarrow NH_3 + H_2O$

$NH_3 + H_2O \longrightarrow NH_4OH$

## REFERENCE MANUALS USED IN CHAPTER II

1. Ames, Maurice U., and Bernard Jaffe. Laboratory and Workbook Units. New York: Silver Burdett Company, 1955.
2. Brownlee, Raymond B., Robert W. Fuller, and Jesse E. Whitsit. Laboratory Experiments in Chemistry. Boston: Allyn and Bacon, 1955.
3. Carpenter, Floyd F., and Robert H. Carleton. Comprehensive Units in Chemistry. Chicago: J. B. Lippincott Company, 1950.
4. Ferris, Everett M., and Oscar E. Lanford. Laboratory Manual for Using Chemistry. New York: McGraw-Hill Book Company, Inc., 1956.
5. Rawlins, George M., and Alden H. Struble. Chemistry in Action in the Laboratory. Boston: D. C. Heath and Company, 1956.
6. Weaver, Elbert C. Laboratory Introduction to Chemistry. New York: McGraw-Hill Book Company, Inc., 1952.

# CHAPTER III

## ANALYTIC PROCEDURES

One of the most important and one of the oldest applications of chemistry is in analysis. By analytic methods the composition of naturally occurring substances was first determined. Dozens of elements were first discovered through use of such techniques by the outstanding analytical and mineralogical chemists of the eighteenth and nineteenth centuries.[31]

As time goes by and more analytic procedures are worked out, the authors of text books and teachers are forced to make decisions as to what should and can be included in the high school chemistry course. Some authors publish a huge laboratory manual which includes every experiment known to high school chemistry. The teacher then must make the selections alone. This can be an almost impossible task, especially for the beginning teacher. Other authors conscientiously attempt to select a maximum number of experiments that would be completed and to include those which will contribute most to the students.

Many larger high schools are meeting the demand for more chemistry, and especially more analytical work, by offering a second year of chemistry which is devoted largely to qualitative and quantitative analysis. Some colleges are permitting graduates of such courses to

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[31] L. H. Cragg and R. P. Graham, An Introduction to the Principles of Chemistry (New York: 1955), p. 25-26.

enroll with advanced standing upon passing specially prepared tests.

In order to avoid repetition as much as possible, the findings of the writer regarding analytic procedures will be presented in survey form.

Eight laboratory manuals were carefully examined to determine what tests are most frequently included. Laboratory execution of these tests revealed some advantages and some disadvantages of the various procedures. In addition to the six manuals used in connection with the previous chapter two more manuals are included in this survey. For the reader's convenience all eight are again listed in alphabetical order at the end of this chapter. Any reference to them will again be made by number and page.

Two tests were included in every one of the eight manuals. These were the flame test for certain metals and the borax bead test for certain other metals. Metals detected by flame tests include barium, calcium, lithium, potassium, sodium, and strontium. Detected by borax bead tests are chromium, cobalt, ferric iron, manganese, and nickel. The identifying colors for these tests will be given in a later portion of this chapter.

Included in six of the manuals (3 and 7 excepted) was a procedure for the identification of an unknown salt. Five manuals (1, 6, and 8 excepted) had an experiment for the separation of lead, silver and mercurous mercury. Food tests were included in all manuals except 2, 4, and 5. Cobalt nitrate tests were included in four manuals (1, 2, 5, and 6). These tests are used to detect the presence of aluminum, magnesium, and zinc.

Three manuals included tests for identification of fibers (1, 2, and 7), tests for ferric and ferrous ions (5, 6, and 7), and tests for percentage of water in crystalline salts (1, 7, and 8). Tests for the acid ions were given in manuals (2) and (4), and tests of soils and fertilizers were given in (1) and (3).

One manual (2) gave separate tests for chloride and sulfate ions and separate tests for nitrate carbonate, sulfite, and sulfide ions. One (7) gave an analytical procedure for the determination of the percentage of oxygen in potassium chlorate. Another (1) included consumer tests of certain commercial products and tests of properties of some commercial plastics.

The manuals (except 2) included tests for chlorides, sulfates, nitrates and sulfides as part of the experiments dealing with preparation and properties of hydrochloric acid, sulfuric acid, nitric acid and hydrogen sulfide, respectively.

After careful evaluation of all tests found in the eight manuals, an attempt was made to select a minimum number of varied tests which should be included in a high school first year chemistry course. Nine testing procedures were chosen and each will be briefly summarized.

The order in which these experiments are given will coincide with the order in which related material will be presented in most texts.

## Determination of Water of Crystallization

Procedure: A porcelain crucible and its cover are cleaned and dried by heating over a blue flame. After cooling the crucible and cover are weighed (ask for accuracy to 0.01 gram). This weight with others to follow are recorded in a table as given below. About three grams of barium chloride is placed in the crucible and covered and a second weighing is recorded.

The covered crucible is supported on a pipestem triangle so that its bottom is a short distance above the tip of the inner cone of the Bunsen burner flame. The crucible is heated gently at first and then strongly for ten to twelve minutes.

After cooling, the covered crucible is weighed. The heating, cooling and weighing should be repeated until successive weights are the same. After the final weight is taken the table is completed and determination of the percentage of water of crystallization (or water of hydration) is made by simple calculations.

Recommendations: Barium chloride crystals will give best results because they are neither deliquescent nor efflorescent and are easily obtained in the pure state.

Initial heating of crucible must be gentle or water of crystallization may be driven off explosively carrying off some of the salt.

Heating to constant weight is most desirable but if time does not permit repeated heating, cooling, and weighing, the crucible and contents should be strongly heated 25 to 30 minutes before a weight is taken and recorded.

## TABLE I

## DATA FOR WATER OF CRYSTALLIZATION DETERMINATION

(1) Weight of covered crucible and barium chloride crystals ________ g

(2) Weight of empty covered crucible . . . . . . . . . . . . ________ g

(3) Weight of crystallized barium chloride . . . . . . . . . ________ g

(4) Weight of covered crucible and contents--1st heating . ________ g

(5) Weight of covered crucible and contents--last heating. ________ g

(6) Weight of anhydrous barium chloride. . . . . . . . . . . ________ g

(7) Weight of water lost by barium chloride. . . . . . . . . ________ g

(8) % water = $\frac{\ldots\ldots g(\text{wt. of water})}{\ldots\ldots g(\text{wt. of salt})}$ x 100 = . . . . ________ %

Optional Calculations of Number of Molecules of Water in One Molecule of Barium Chloride Crystal ($BaCl_2 \cdot x\ H_2O$)

$$\frac{g(\text{wt. of Crystallized } BaCl_2)}{g(\text{wt. of anhydrous } BaCl_2)} = \frac{X\ \ (\text{mol. wt. of Crystallized } BaCl_2)}{(\text{mol. wt. of anhydrous } BaCl_2)}$$

(9) Calculated molecular weight of crystallized $BaCl_2$. . . ________

(10) Molecular weight of anhydrous $BaCl_2$ . . . . . . . . . . ________

(11) Molecular weight due to water . . . . . . . . . . . . ________

(12) $\frac{\ldots\ldots(\text{mol. wt. due to water})}{18\ \ (\text{mol. wt. of water})}$ = . . . . .molecules of water in the crystal

# Detection of Impurities in Water

Procedure: Each reagent or combination of reagents is added to a prepared sample of impure water and to a small amount of tap water. Any reaction of either sample is recorded in Table II, page 41.

To each sample a few drops of Nessler's reagent is added. The liberation of a gas indicates presence of ammonia which in turn usually indicates contamination by organic matter.

To each sample is added one or two drops of potassium permanganate (to produce a distinct violet color) and about 2 ml. of concentrated sulfuric acid. A fading of the violet color indicates presence of organic matter.

To each sample is added 1 ml. of concentrated nitric acid and a few drops of silver nitrate solution. A cloudy appearance indicates presence of chlorides which in large quantities indicate contamination by surface drainage. A confirmatory test is the addition of ammonium hydroxide which will clear the cloudy solution.

To each sample is added 1 ml. of acetic acid and 3 ml. of ammonium oxalate solution. A cloudy appearance indicates presence of calcium which always indicates hardness in water.

To each sample is added 1 ml. of concentrated nitric acid and 5 ml. of barium chloride solution (added slowly). Formation of a white precipitate indicates presence of sulfates. (See Table II, page 41)

Recommendations: The prepared impure water should contain each of the five impurities being tested for. One-third to one-half test tube of water should be used for each sample. If the ammonia reaction is faint, it may be increased by gentle heating.

## TABLE II

## RESULTS OF TESTS FOR DETECTION OF IMPURITIES IN WATER

| Chemical Reagent | Reaction with Impure Water | Impurity Indicated | Reaction with Tap Water | Impurity Indicated |
|---|---|---|---|---|
| Nessler's Reagent | Gas is liberated | Ammonia | None | None |
| Potassium Permanganate and Sulfuric Acid | Color fades | Organic matter | Color unchanged | None |
| Nitric Acid and Silver Nitrate | Becomes milky | Chlorides (Salts) | Remains clear | None |
| Acetic Acid and Ammonium Oxalate | Becomes cloudy | Calcium (hardness) | Remains clear | None |
| Nitric Acid and Barium Chloride | White precipitate | Sulfates | No precipitate | None |

## Percent of Oxygen in Potassium Chlorate

Procedure: About three grams of finely powdered manganese dioxide is strongly heated in a porcelain crucible for about five minutes to drive off any volatile impurities which may be present. The powder is then allowed to cool.

A thoroughly clean test tube is weighed accurately to 0.01 gram. This weight and other data are recorded in Table III, page 44. About seven grams of potassium chlorate is placed by means of a spatula or folded paper near the bottom of the test tube which is again accurately weighed. The previously heated and cooled manganese dioxide is now added and the test tube accurately weighed. The two compounds are mixed in the test tube by rotating and tapping the tube.

Fit the test tube with a stopper and delivery tube and support it with a clamp and ring stand. A large graduated cylinder (2 liter capacity) used as the collecting bottle is filled with water and inverted in a trough over the end of the delivery tube.

The mixture in the test tube is now heated slowly at first and then more strongly but always so that the bubbles of gas can be counted. The heating is continued until no more oxygen is coming off. The stopper and delivery tube are then removed from the test tube which is carefully set aside to cool.

When cooled to room temperature the test tube with its residue is accurately weighed. This weight completes the data needed to compute the percent of oxygen in potassium chlorate as experimentally determined. This percentage is compared with the percentage calculated from the formula of the chlorate.

The graduated cylinder is now adjusted so that the water levels inside and outside are the same. The temperature of the water is recorded, as is the barometric pressure of the atmosphere. The volume of oxygen in the cylinder is carefully read and recorded. Making a correction for vapor pressure and converting centigrade temperature to absolute, the volume of oxygen can be calculated at standard conditions by the equation:

$$V_2 = V_1 \times \frac{P_1}{P_2} \times \frac{T_2}{T_1} = \text{ml}$$

$V_2$ = volume at standard conditions
$V_1$ = volume recorded from experiment
$P_2$ = 760 mm Hg
$T_2$ = 273° A
$P_1$ = barometric pressure minus vapor pressure
$T_1$ = centigrade temperature plus 273

The weight of the oxygen liberated from the potassium chlorate and the corrected volume of the oxygen can be used to calculate the weight of 22.4 liters of oxygen which is numerically equal to its molecular weight.

Recommendations: This experiment should be performed by each laboratory group or individually. However, it may be done as a demonstration, preferably by two students. Each student in the class should record all data and work out the necessary calculations.

## TABLE III

## DATA FOR DETERMINATION OF PERCENT OF OXYGEN IN POTASSIUM CHLORATE

(1) Wt. of test tube and $KClO_3$. . . . . . . . . . . . . . . . ________g

(2) Wt. of empty test tube. . . . . . . . . . . . . . . . . . . ________g

(3) Wt. of $KClO_3$ used . . . . . . . . . . . . . . . . . . . . ________g

(4) Wt. of test tube, $KClO_3$ and $MnO_2$ . . . . . . . . . . . ________g

(5) Wt. of test tube and residue after heating . . . . . ________g

(6) Wt. of oxygen given off . . . . . . . . . . . . . . . . . ________g

(7) % Oxygen by experiment = $\frac{g(\text{wt. of } O_2}{g(\text{wt. of } KClO_3)}$ x 100 = . ________%

(8) % Oxygen from formula = $\frac{(O_3)}{(KClO_3)}$ x 100 = . . . ________%

(9) % Error = $\frac{(8-7)}{8}$ x 100 = . . . . . . . . . . . . . . ________%

(10) Temperature of water (_____$^{\circ}C$ + 273) . . . . . . . . ________$^{\circ}A$

(11) Vapor pressure of water at this temperature . . . . . ________mm

(12) Barometer reading . . . . . . . . . . . . . . . . . . . . ________mm

(13) Corrected pressure (12-11) . . . . . . . . . . . . . . ________mm

(14) Volume of oxygen under laboratory conditions . . . . ________ml

(15) Calculated volume under standard conditions . . . . . ________ml

(16) Calculated wt. of 22.4 liters of oxygen (gm-mol.wt) . ________g

(17) Accepted gm-mol. wt. of oxygen . . . . . . . . . . . . ________g

## Detection of Ferrous and Ferric Ions

Procedure: Two ml. of freshly prepared ferrous chloride is poured into each of four test tubes. Into each of four more test tubes is poured 2 ml. dilute ferric chloride solution. To the first test tube of each solution is added a few drops of ferricyanide solution. To the second sample of each is added a few drops of ferrocyanide. To the third portion of each is added two drops of potassium thiocyanate solution. To the last test tube of each iron solution is added enough dilute ammonium hydroxide solution to neutralize and then make basic. All observations are recorded in Table IV, page 46.

To show the relation of ferrous and ferric compounds the original solutions should be treated so as to convert each type of ion to the other type. To the remaining ferrous chloride solution is added about 1 ml. of concentrated nitric acid and 3 ml. of concentrated hydrochloric acid, and the mixture is boiled for three minutes. The resulting solution is tested with the four reagents used above. If the tests show the presence of ferrous ions the acid and boiling treatment should be repeated. The remaining original ferric chloride solution is treated with 1 or 2 ml. of iron filings and about 2 ml. of concentrated hydrochloric acid. The solution is boiled for a few minutes until it becomes colorless. Testing will show the presence of ferrous ions and absence of ferric ions.

Equations: $Fe + 2HCl \longrightarrow FeCl_2 \quad H_2$ (Preparation of ferrous chloride)

$FeCl_2 + (Cl) \longrightarrow FeCl_3$ (Oxidation of ferrous chloride)

$FeCl_3 + Fe \longrightarrow 2FeCl_2$ (Reduction of ferric chloride)

Recommendations: To prepare a fresh solution of ferrous chloride for use in this experiment place about 0.5 ml. of iron filings in a small flask and cover with 5 ml. of water and 2 ml. of concentrated hydrochloric acid. Stopper the flask loosely, mix the contents by swirling, and then warm gently. After allowing the reaction in the flask to continue for five minutes, add 50 ml. of water and stopper the flask to prevent oxidation.

TABLE IV

RESULTS OF TESTS FOR DETECTION OF FERROUS AND FERRIC IONS

| Reagent Used | Ferrous Ions | | Ferric Ions | |
|---|---|---|---|---|
| | Color | Name of Precipitate | Color | Name of Precipitate |
| $K_3Fe(CN)_6$ | Deep blue | Turnbull's blue | Brown | No ppt. |
| $K_4Fe(CN)_6$ | White | No ppt. | Deep blue | Prussian blue |
| KCNS | No color | No ppt. | Deep red | No ppt. |
| $NH_4OH$ | White | Ferrous hydroxide | Red brown | Ferric hydroxide |

## Flame Tests for Metallic Ions

Procedure: Concentrated solutions of a soluble salt (preferably the chloride) of lithium, sodium, potassium, calcium, barium, strontium, and copper are prepared. A Bunsen burner is adjusted so that it burns with a colorless flame. A platinum (or nichrome) wire is anchored in a cork or sealed into the end of a piece of glass tubing. To confirm tests a little of each salt may be moistened with hydrochloric acid and tested in the flame.

The prepared wire is dipped into each concentrated solution and brought into the edge of the colorless flame. To clean the wire after each test, dip it into a little hydrochloric acid and bring to the edge of the flame. This is repeated until no color is produced in the flame. If the moistened salts are also tested, the same cleaning procedure must be followed between tests. The flame color is the same for the concentrated solution and the moistened sample of the same salt or of salts of the same metal.

TABLE V

CHARACTERISTIC FLAME COLORS OF METALLIC IONS

| | |
|---|---|
| Lithium. . . . . . . . . | Crimson |
| Sodium . . . . . . . . . . | Yellow |
| Potassium. . . . . . . . | Violet |
| Calcium. . . . . . . . . | Orange-red |
| Barium . . . . . . . . . . | Green |
| Strontium. . . . . . . . | Bright red |
| Copper . . . . . . . . . . | Blue-green |

Recommendations: One or more unknowns should be tested by each student and the results and conclusions checked for accuracy. Some manuals give the flame tests along with the cobalt nitrate and/or borax bead tests. Unless the laboratory period is longer than the regular class period it will not be longer than is needed to complete the flame tests satisfactorily. Those students who finish most quickly may be given extra unknowns.

## Borax Bead Tests

Procedure: The end of a platinum (or nichrome) wire is bent into a small loop, heated till red hot and dipped into powdered borax. The borax is then heated in the outer portion of a Bunsen burner flame, first swelling and then melting to form a clear glassy bead. The dipping and heating are repeated until a bead the size of a drop of water is formed. The bead while still hot is dipped into a solution of the salt to be tested and again heated in outer flame until the bead melts. The new head is allowed to cool. Color is usually evident if the bead is held in a good source of light. If not, repeat the bead making. Characteristic colors of beads are given in Table VI, page 49.

The bead is removed from the wire by heating it until melted and quickly tapping the wire on the edge of the table top or a waste jar.

Recommendations: One or more unknowns should be tested by each student and the result and conclusion checked for accuracy. This experiment would seem to take only a short time but it takes a bit of practice to make borax beads. A short laboratory period might be combined with the cobalt nitrate tests which follow. If students work in pairs they can run both tests at the same time and take turns so that each runs a part of each test series.

## TABLE VI

## CHARACTERISTIC BORAX BEAD COLORS

| | |
|---|---|
| Nickel | Brown |
| Cobalt | Blue |
| Chromium | Green |
| Manganese | Violet |
| Iron | Yellow |

## Cobalt Nitrate Tests

Procedure: A shallow cavity is scraped into a charcoal stick or a plaster of Paris block. A little of the salt to be used is placed in the cavity and heated strongly by means of a blowpipe. After each test the cavity is cleaned by scraping it or a new cavity is made and used.

To use a blowpipe the Bunsen burner is adjusted to produce a luminous flame about 4 cm high. The tip of the blowpipe is held just inside the flame about 2 cm above the burner so that the flame is blown at an angle on the salt being heated.

After the salt is heated strongly for a short time it is cooled and one or two drops of cobalt nitrate solution are added to the salt.

This mixture is heated strongly until a change in color is noted.

Only three metals are detectable by this test.

Recommendations: The plaster of Paris block is better for this test because the colors are more easily detected. At best they may be difficult to see. Students may be told what color each salt should give and work to that end. One or more unknowns should be used to check the students' accuracy of observation. The sulfates are best for these tests.

TABLE VII

RESIDUE COLORS OF COBALT NITRATE TESTS

| | |
|---|---|
| Aluminum | Blue |
| Magnesium | Pink |
| Zinc | Green |

### Identification of Lead, Silver and Mercurous Salts

Procedure: Two ml. portions of solutions of lead, silver, and mercurous salts are put into separate test tubes. To each solution is added dilute hydrochloric acid until no further reaction occurs. The three test tubes are then heated to boiling in a beaker of water. The precipitate from the lead salt will dissolve. Adding a few drops of potassium chromate solution produces a yellow precipitate.

To the remaining two test tubes a few drops of ammonium hydroxide solution (dilute) is added. The mercurous precipitate turns black and the silver precipitate dissolves.

A mixture containing about 3 ml. of each of the original salt solutions is made in a beaker. Dilute hydrochloric acid is added until no precipitate is formed upon addition. The precipitate is filtered and washed with cold water to remove excess acid. The residue is washed with 10 ml. of boiling water and the filtrate caught in a clean beaker. Adding potassium chromate to the filtrate produces a yellow precipitate indicating the soluble lead chloride has passed through the filter with the boiling water.

The remaining residue on the filter paper is treated with dilute ammonium hydroxide and the filtrate caught. The residue which now remains on the filter paper turns black indicating mercury. The clear filtrate is made acid by addition of dilute nitric acid resulting in reprecipitation of silver chloride.

Recommendations: Each student should be given at least one unknown to check. The unknown may contain one or more or none of the three metals detected by this test procedure.

## Identification of an Unknown Salt

In addition to the tests for metals already summarized, another important test is the use of hydrogen sulfide gas (or thioacetemide). Precipitates of characteristic color are produced in solutions of salts of certain metals as listed in Table VIII, page 52. This test will have been included in the experiment dealing with hydrogen sulfide.

The ammonium radical takes the metallic position in common salts. A test for its presence will have been included in the experiment dealing with ammonia gas. The salt being analyzed is mixed with a little calcium hydroxide and heated. The characteristic odor of ammonia indicates its presence. A piece of moist red litmus held above the heated mixture will turn blue.

TABLE VIII

CHARACTERISTIC COLORS OF METALLIC SULFIDES

| | |
|---|---|
| Antimony | Orange |
| Arsenic | Yellow |
| Bismuth | Brown-black |
| Cadmium | Yellow |
| Copper | Black |
| Lead | Black |
| Manganese | Pink |
| Tin | Chocolate brown |
| Zinc | White |

Tests for nonmetals and nonmetallic radicals which also will have been included in other experiments will be given in brief statements.

Test for a chloride: To the unknown in solution some silver nitrate solution is added. A precipitate which turns purple in the sunlight and is insoluble in nitric acid indicates the presence of a chloride.

Test for a bromide: To the unknown in solution is added some carbon tetrachloride and chlorine water. The mixture is shaken and then set aside. An amber color in the bottom layer shows that free bromine has been liberated from a bromide.

Test for an iodide: Test as for bromide is repeated. A violet color in carbon tetrachloride layer indicates free iodine from an iodide.

Test for a sulfide: To some of the solid unknown (most sulfides are insoluble in water) is added a few drops of dilute hydrochloric acid. The odor of hydrogen sulfide gas shows the presence of a sulfide. The gas turns lead acetate paper a metallic black.

Test for a sulfate: To the unknown in solution is added a little barium chloride solution. A white precipitate which is insoluble in hydrochloric acid shows the presence of a sulfate.

Test for a sulfite: To the solid unknown is added a little hydrochloric acid. The suffocating odor of sulfur dioxide indicates the presence of a sulfite. The gas will decolorize a dilute solution of potassium permanganate.

Test for a carbonate: To some of the solid unknown is added a little hydrochloric acid. The evolution of carbon dioxide gas which, when passed into limewater, causes it to turn milky shows the presence of a carbonate.

Test for a nitrate: To some of the unknown in solution is added some freshly prepared ferrous sulfate solution. A little concentrated sulfuric acid is carefully poured down the side of the tilted test tube. A brown ring formed between the two layers indicates the presence of a nitrate.

Procedure: The student is given a salt in solution or in the solid state and asked to analyze it by using the test procedures for metals (and the ammonium radical) and for nonmetallic elements or radicals of acids. As each salt is correctly reported, the student is given another one to analyze. Most instructors set aside three or four laboratory periods for identification of unknowns and allow students to analyze as many as they have time for.

Recommendations: If the laboratory manual being used does not have a review of all the tests in one place, the student should compile such a review of these tests, based on a list given by the teacher. The tests are then run in systematic order.

## Conclusion

As stated before, this is a recommended minimum of analytical work. Most instructors will want to include food tests and fiber tests in the first year chemistry course. Some will feel that these two tests should be included in the recommended minimum.

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