SECOND EDITION

Introduction to Laboratory Chemistry: Organic and Biochemistry

Williams · Richardson · DeBey · Kelley · Lien

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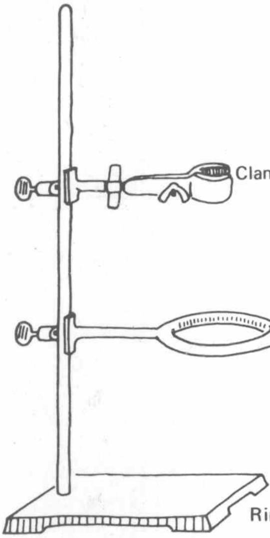

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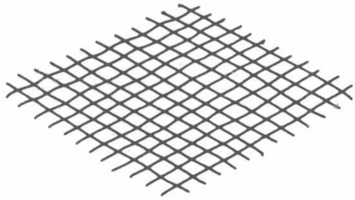
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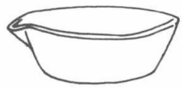
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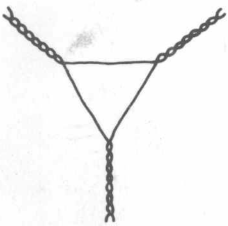
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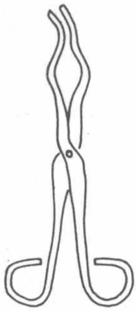
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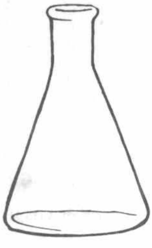
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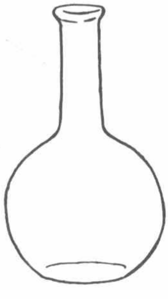
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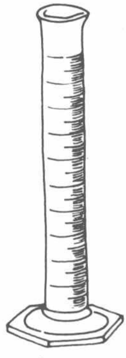
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Preface

When the publishers began to talk about the possibility of a second edition of this manual, it brought mixed feelings. We were anxious to make some changes that we saw as necessary after having used the first edition but we were concerned about just how much should be changed. We solicited the opinions of many of the users of the manual, thought about their suggestions, thought about comments our students had made, and finally produced this revised edition.

We have not changed the basic level of presentation. The book is still intended for students with little or no previous preparation in chemistry, most of whom are not chemistry majors. We have retained most of the experiments and added only a few, and most of the changes we made were in organization.

We have begun each experiment with a list of objectives and have included longer introductions to most experiments. In those places in which our students had found the directions hard to follow, we have expanded or rewritten them. Some of the experiments were too long for a normal laboratory period and we have made some changes that should decrease the time required. In other cases, we have separated an experiment into two parts. These can be done in two different laboratory sessions, or one section may be done primarily by demonstration.

We have also reorganized by moving the questions and conclusions to the end of an experiment in many instances. We have provided space for observations in the experimental sections and moved the summaries of data and conclusions to a separate section. We believe this should make the observations and conclusions more meaningful, and also make it easier for the laboratory instructor to check conclusions.

The experimental reports have been expanded so that they reflect better the material covered in the experiment while still providing an opportunity for the student to draw conclusions and make generalizations that go beyond the experiments performed. In the first edition the experimental report frequently had other material on the reverse side, material that the students wanted to retain in their manuals, so we have also placed all experimental report sheets at the back of the book. We have also compiled an instructor's manual to accompany the laboratory manual. It has suggestions for further work, answers to some of the work exercises, and other materials that should be helpful, particularly to the instructor who is using the manual for the first time. Copies of the instructor's manual are available from the publisher.

We have made some changes for purely practical reasons. In a time when the prices of chemicals and other supplies are increasing, we have specified decreased amounts of some materials and decreased concentrations of some solutions. We have also listed the equipment and supplies needed for each experiment in addition to those normally contained in a laboratory locker. Minor changes in the order of experiments and occasionally of sections of experiments were made so that they would conform more closely to the order of topics in the text that this manual is designed to accompany—*Introduction to Chemistry, Second Edition* by A. L. Williams, H. D. Embree, and H. J. DeBey (Reading, Mass.: Addison-Wesley, 1973).

We have resisted the temptation to make this a larger book but have added a few sections. The new sections include discussions of conversion of temperatures from one scale to another; expanded discussions of molarity, normality, and percentage as a method for expressing concentration of solutions; a section on dimensional analysis; and additional material on net ionic equations.

We have rewritten much of the material to avoid giving the answer to a question before, or soon after, it was asked. We found that too often our introductions contained conclusions and we were depriving students of the most important aspect of science, that of drawing conclusions from experiments.

We hope our revisions have made this a better laboratory manual than the first edition. We will continue to be interested in the comments of instructors and students who use this manual. Although contemplation of a third edition is something we would rather avoid after the labor of producing the second, it will probably come. We would be glad to have your suggestions incorporated in it.

San Jose, California
January 1978

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L.A.K.
O.G.L.

To the Student

The chemistry laboratory is a strange and slightly frightening place to a newcomer. Some of this strangeness is due to the fact that you're not sure exactly what is supposed to happen, and in many cases the instructors are mysterious, because they want you to find some things for yourself. We hope that this manual will help you understand chemistry and will make the laboratory an interesting place.

We have tried to make your laboratory experiences meaningful by beginning each experiment with a statement of the objectives of that experiment. This section is followed by an introduction that includes the things you need to know in order to understand the experiments. Occasionally we have also included practical applications of the experiment or something about the significance of the experiment to your life. We are concerned that you see the ways in which each experiment contributes to your understanding of chemistry and its wider applications.

Another cause for fear in students is that of being surrounded by unfamiliar things, some of which may be inflammable or poisonous, or substances that may produce burns if spilled on your skin or clothing. There is also a general apprehension that things will explode or react violently. Actually, very few common things in the laboratory are likely to explode, although many will burn if exposed to an open flame. While many chemical reagents are toxic (poisonous) or caustic (capable of producing burns), we have written this manual to include primarily those experiments that are safe and we have included cautions when there are dangers. You will find a list of safety instructions at the beginning of the manual. If you follow them, the laboratory is a safe place; if you disregard them, the laboratory will truly be a frightening place for both you, your fellow students, and the instructor!

To many students, chemistry seems to be a series of rules and laws to be memorized. They do not realize that all of these rules and laws are generalizations based on experimentation. It would be too time-consuming to have each of you personally discover each generalization (somewhat like each of us having to rediscover the wheel). However, we have provided experiments from which you can draw some of the conclusions and make the generalizations that make up significant parts of chemistry. In other cases we can let you test or verify some of the basic laws. If you see this aspect of chemistry, it will help to make your time spent in the laboratory a rewarding learning experience.

A few notes on how you may use this book may be helpful. We believe you should read each experiment before coming to the laboratory. This will give you some sense of direction and should keep you from making simple errors because you don't understand the instructions. Once you are in the laboratory you should use all of your senses to observe the things that occur and should be certain to describe as carefully as possible what you have observed. These observations should be written down immediately, and we have provided space for that in the manual. You should wait until you have actually made the laboratory observation before completing these, however. You may be good at guessing, but this is not the way of science. It is sometimes known as "drylabbing." This is the cause of poor understanding and/or poor grades. Such practices are as old as the ancient Greeks who thought the answers were to be found in books and logic and not by performing experiments. Because of such attitudes, modern science did not develop for 2000 years.

Sometimes we have suggested that the instructor perform an experiment (demonstration) and that the students be observers. This is desirable when materials being handled are hazardous or expensive or when only limited equipment is available. Demonstrations are also necessary at times so that you will see a precise change – one you might miss if you were doing the experiment yourself for the first time. In other experiments we have suggested that you work with another student, or that groups of students perform different aspects of an experiment and share results. The difficulty with this, of course, is that you may only understand the part of the experiment that you do. Always be certain that you observe as many parts of an experiment as possible and ask questions about those things you didn't see or don't understand.

At the end of a laboratory experience it is a good practice to look again at the objectives of the experiment. Ask yourself if you have learned the things you should. Have you acquired the skills and made the generalizations? You may test your understanding by doing the work exercises. Your instructor will probably want you to complete the Experimental Report and submit it before you leave the laboratory. This report serves as the final summary of the experiment. If you can complete it with ease, it probably means that you understand the experiment.

We hope this manual will help to make your experiences in the chemistry laboratory interesting and rewarding; we further hope that these experiences will help you both in those specific fields you may choose for specialization and in your general progress toward becoming a more educated person.

The authors

Safety Precautions and Suggestions

Safety precautions are the most important instructions that you will receive in this course. Failure to observe them may cause serious injury to you and (or) laboratory neighbors. The preceding statement is not made with the intention of leading you to believe the laboratory is a dangerous place; it is only when those working in laboratories become careless that accidents happen. If the following precautions are observed, the laboratory will be a safer place for you than the street you cross on your way to class.

1. Goggles or other protective eye equipment approved by OSHA (Occupational Safety and Health Administration) must be worn at all times when in the laboratory unless otherwise specified by the instructor. Contact lenses or regular glasses cannot be substituted for approved eye protective equipment.

2. Shoes or similar adequate foot covering must be worn while in the laboratory. Avoid wearing easily combustible or especially loose fitting clothing. Long hair may catch fire if passed near a laboratory burner, and hair nets or a change in hair style should be used to minimize the danger presented by very long hair.

3. Protective laboratory aprons or coats are desirable but are not generally required in introductory-chemistry laboratories.

4. Note the location of fire extinguishers and other protective equipment such as eye-washing fountains, showers, and fire blankets.

   a) In the case of a minor fire, wet paper towels are efficient for smothering the fire. Use other measures as appropriate and inform the instructor any time that you have a fire.

   b) If your clothing catches fire, the fire should be smothered with a blanket or a heavy coat or you should immediately use a laboratory shower. If you are some distance from a shower, it is better to fall to the floor to prevent flames from reaching your head. Rolling over and over may also help extinguish flames if a blanket or shower are not immediately available.

c) If chemicals of any kind get in your eyes, wash with water for several minutes, preferably at an eye-wash fountain. Always report such incidents to the instructor.

d) If you are burned or injured in any way, be certain to inform the instructor. You may be directed to consult a student health service or your own physician in some instances.

e) If chemicals are spilled on your hands or any other part of your body, wash at once with water. Inform your instructor whenever such chemicals produce an irritation.

f) If chemicals are spilled on your clothing, advise your instructor at once. It may be possible to use sodium hydrogen carbonate or other substances to prevent damage to your clothing.

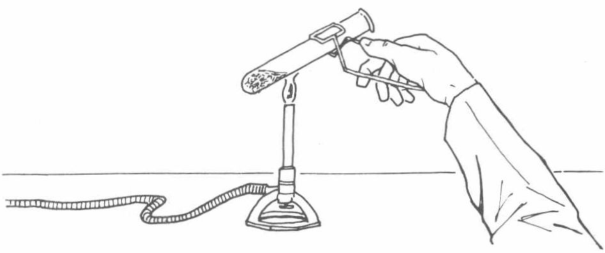
5. Do not taste anything in the laboratory. Do not eat or drink anything in the laboratory.

6. Always read instructions carefully. If you are in doubt, ask your instructor. Be especially careful in the following instances.

a) When heating any material in a glass container be certain that it is borosilicate glass (e.g., Pyrex® or Kimax®).

b) When heating materials in a test tube, point the open end of the tube away from yourself and others. Tilt the test tube and heat near the top of the liquid first. Never heat the bottom of a test tube that contains liquids. (See the illustration below.)

c) Be especially careful when inserting glass tubes or thermometers through rubber or cork stoppers. Lubricate the tube with water, glycerine, or other lubricant, grasp the item to be inserted securely near the stopper, and protect your hands with a towel to avoid cutting your hands. NEVER TRY TO FORCE GLASS TUBING OR THERMOMETERS THROUGH STOPPERS.



d) Do not use mouth suction in filling pipets; use a pipet bulb for this purpose. Ask your instructor to demonstrate the correct technique if you are uncertain.

e) Exercise great care in noting the odor of gases or liquids. Use a fume hood whenever noxious fumes are being produced.

7. Do not work in the laboratory alone. In classes with experienced students, your instructor may permit a group of students to work unsupervised, but you should never work alone. In general, no persons other than class members are allowed in a chemistry laboratory without explicit permission of an instructor.

8. Never perform unauthorized experiments.

9. Keep table tops clean; what looks like a drop of water may be a strong acid or base solution that could damage clothing or books, or injure students.

10. If you find it necessary to obtain liquid chemicals from a stockroom, a plastic bucket should be used to carry such containers of liquids between the stockroom and the laboratory.

11. Unless specifically directed to do so, never return chemicals to reagent bottles or other containers. If you have an excess of a reagent, discard it.

12. Many of the reagents used in organic chemistry and biochemistry are flammable. Care should be taken with such substances, particularly if they are liquid. Do not bring such liquids near flames. Do not pour flammable liquids down a sink since they tend to accumulate and can be ignited in the pipes and cause an explosion. Special instructions will be given in specific experiments for disposal of organic liquids.

Serious and repeated disregard of safety procedures may result in the instructor dismissing a student from a course.

Contents

EXPERIMENT 1

Differences Between Organic and Inorganic Compounds and Tests for Elements Found in Organic Compounds

OBJECTIVES

1. To learn the tests used to identify the elements present in organic compounds.
2. To observe some of the fundamental differences in properties of organic and inorganic compounds.

INTRODUCTION

Early in the development of chemistry as a science, compounds were classified as being either organic or inorganic. Organic compounds came from plants and animals and for a long time it was generally believed that organic compounds could not be made in the laboratory. We now know that this is not true.

Although there are a great number of organic compounds, these compounds contain only a few of the chemical elements. Accurate analysis of the amounts of each element in an organic compound is required to unravel the complexity of its structure. In this experiment we will use some of the methods that have long been used to establish the composition of organic compounds.

Since the elements found in organic compounds are usually incorporated into complex structures, we generally need to use some method of releasing these elements so that we can test for them. We can release hydrogen and carbon by burning the compound. In other cases, we use a strong base to release a substance such as ammonia or to convert an element such as sulfur or phosphorus to a form in which it can be detected.

If we find water being produced when we burn an organic compound, it is evidence that the compound contained hydrogen. Whether the oxygen in the water came from the compound or from the oxygen in the air is more difficult to decide. We generally determine the amount of oxygen in an organic compound by analyzing for all possible other elements and assuming that the remainder is oxygen.

We test for the presence of carbon in a compound by burning the compound to give carbon dioxide and passing the gas formed in the combustion through limewater $Ca(OH)_2$. If a white precipitate (calcium carbonate) forms, we say that the compound we burned contained carbon:

$$CO_2 + Ca(OH)_2 \rightarrow CaCO_3 + H_2O.$$

Since all organic compounds that can be burned give this white precipitate, we can make the statement that all organic compounds contain carbon. There are other methods of detecting carbon in those compounds which cannot be burned.

The simplest test for nitrogen in organic compounds depends on the tendency of the compound to produce ammonia, which we can detect by its odor. Since it may not always be possible to detect the odor of ammonia in an odor-filled laboratory, we can also test for ammonia by exposing a piece of wet red litmus to the fumes coming from a heated solution containing ammonia. If ammonia is present, the litmus paper will turn blue:

$$NH_3 + HOH \leftrightarrows NH_4^+ + OH^-.$$

In more complex analyses for nitrogen, we can volatilize the ammonia and titrate it with an acid.

The test for sulfur depends on producing sulfide ions by heating organic compounds with a strong base. The sulfide ions are then reacted with lead ions to give a dark brown to black precipitate of lead sulfide. Phosphates can be identified by their complex reaction with the molybdate ion to produce a yellow precipitate.

The halogens (chlorine, fluorine, bromine, and iodine) are found in many inorganic compounds but are relatively rare in naturally occurring organic compounds. However, we use organic compounds containing the halogens in many important organic reactions and this experiment will provide the opportunity to study a test for these important chemical elements.

The physical and chemical properties of many of the organic compounds can be used to differentiate them from organic compounds. While some properties are shared by both organic and inorganic compounds, there are several that are distinctly different and we use these for simple tests to tell whether a compound seems to be organic or inorganic. In this experiment you will study three properties: behavior upon heating, conductivity, and ionization (dissociation) of typical inorganic and organic compounds. In the experiments that follow, you will study solubility and other physical as well as chemical properties that can be used to differentiate inorganic from organic substances, as well as to classify organic compounds into different groups.

SPECIAL EQUIPMENT AND SUPPLIES NEEDED FOR THIS EXPERIMENT

Conductivity apparatus

FIG. 1.1
Testing for combustion products of ethanol.

EXPERIMENTAL

I. Tests for Elements Present in Organic Compounds

A. Elements detected by combustion of organic compounds

1. Place approximately 2 ml of the alcohol ethanol (C_2H_5OH) in a crucible and ignite the liquid with a lighted splint. When burning has begun, secure a 250-ml beaker, fill it with cold water, making sure that the bottom and side of the beaker are dry, and hold the beaker above the burning alcohol as shown in Fig. 1.1. Observe any substance that accumulates on the bottom of the beaker.

What is this substance most likely to be?

What is the source of this substance and how was it formed?

Write an equation for this process.

This observation shows the presence of what element(s) in the ethanol?