UNITIZED EXPERIMENTS IN ORGANIC CHEMISTRY

FOURTH EDITION

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PREFACE

The widespread acceptance and endorsement, by instructors and students alike, of the first three editions of *Unitized Experiments in Organic Chemistry* has been highly gratifying. These editions have filled the long-standing need for a versatile and flexible manual of organic chemistry featuring a rich variety of tested experiments that could be coordinated closely with the lecture work into a single unit.

The proven advantages of the earlier editions stand out even more vividly in the fourth. Interesting new experiments have been added, giving the instructor increased flexibility in synchronizing the laboratory work with the lectures, whether aliphatic and aromatic chemistry are treated together or separately. The new experiments include: a discussion of the value of Vitamin C in nutrition and procedures for the analysis of it in commercial tablets and in fruits and vegetables; a discussion of the physiological effects of ethyl alcohol, and the analysis of it in beer or wine by vapor-phase chromatography; the isolation of caffeine from tea and the detection of the active ingredients of Excedrin; a discussion of pheromones and the synthesis of an alarm pheromone for ants; and a discussion of the pharmacological properties of aspirin, together with procedures for the synthesis of the drug and its quantitative determination in commercial tablets. The major objective of the new experiments is to provide material of interest to students majoring in the life sciences. Several of the previous experiments have been deleted to make room for the new ones, others have been modified, and still others have been reorganized and combined.

In particular, with the invaluable assistance of Professors D. R. Deardorff and D. L. Smith of the University of Arizona, we have made a major revision of the experiment on Qualitative Organic Analysis. Also, numerous additional infrared and nuclear magnetic resonance spectra have been included in many experiments, and a discussion of ultraviolet and visible spectroscopy has been added to Experiment 26, Aldehydes and Ketones.

Unitized Experiments in Organic Chemistry represents an embodiment of the conviction that a tremendous amount of organic chemistry can be taught—and learned—in the laboratory. Not only the techniques, skills, and philosophy involved in organic synthesis, but, if the experiments are well designed, much of the fundamental theory and factual material of organic chemistry, as well, can be mastered by the student during the several hours a week which he spends in the laboratory. This can be achieved for an entire class only if every student, the best as well as the poorest, works to capacity, thoughtfully and productively, and only

if the lectures and laboratory work are coordinated into a single integrated unit. These are the goals which we hope the present manual will aid the instructor in achieving.

Carefully tested time schedules, along with thought-provoking questions, are provided with most experiments to challenge the student to maximum efficiency. Optional experiments are provided for superior students who are able to complete the regular work in less than the allotted time.

Every experiment has been designed not only to teach the basic skills and techniques of organic laboratory work but also to clothe in flesh and blood the skeleton of words to which lectures and textbooks are necessarily restricted. Each day's experiment is preceded by an introduction which places the work of the day in proper context in the scheme of organic chemistry and encourages the student to think about the important principles that are being illustrated. Together, the discussion and experimental work constitute a complete, finished unit which affords the student a real sense of accomplishment and leaves him with a sharply defined picture of what the day's assignment is designed to teach.

Some of the early units cover the theory and practice of the most important fundamental techniques employed in the organic chemistry laboratory. Each discussion is followed immediately by an experimental section designed to drive home the underlying principles involved. The discussions are, however, sharply divided from the experimental sections so that the student may refer to them continuously without being forced to re-read detailed instructions for a specific experiment. The early introduction of these units on techniques, which may be performed in regular order or assigned individually at such times as will enable the teacher to keep the laboratory work completely synchronized with the lectures, provides a high degree of flexibility. Once the student has completed these early experiments, he should be prepared to perform the remaining experiments, both those on aliphatic and those on aromatic compounds, in any order the teacher may wish to follow.

Further flexibility is made possible by the inclusion of more units than can be performed normally in a two-semester course so that the instructor may exercise considerable selection. For a one-semester course, we have found highly successful the plan of assigning different experiments to different groups, with an opportunity provided for comparison of results. In this way, each student is given an insight into the experimental aspects of many more experiments than he can perform personally.

Special emphasis has been placed on clear, accurate, and reliable experimental directions, given in sufficient detail to lead to successful and satisfying results in the hands of even the most inexperienced beginner; at the same time special skill and technique will be rewarded with sufficiently superior results to stimulate and challenge the best of students. Hazards and pitfalls have been either eliminated or properly recognized

and pointed out. Often alternative procedures are described to allow for differences in equipment available to students at different institutions.

Students in the beginning courses in organic chemistry lack the experience to organize their work efficiently on their own. The present manual is based on the philosophy that at this stage the very best training for the future is that which shows the student how his work can be organized for maximum productivity.

Over the years, the authors have been led to the conviction that by far the most successful experiments in organic chemistry are those whose results the student can submit as an actual preparation or report as a numerical answer or an unknown. The interest and enthusiasm with which our own students have received the present manual have confirmed that conviction.

Unitized Experiments in Organic Chemistry is dedicated to the proposition that laboratory work in organic chemistry should be a stimulating and rewarding experience.

We are deeply grateful to many who have made suggestions for the improvement of some of the experiments. Our thanks are extended particularly to Dr. Harold L. Jackson, of the E. I. DuPont de Nemours Company, and to the faculty members of the Division of Organic Chemistry of the University of Massachusetts.

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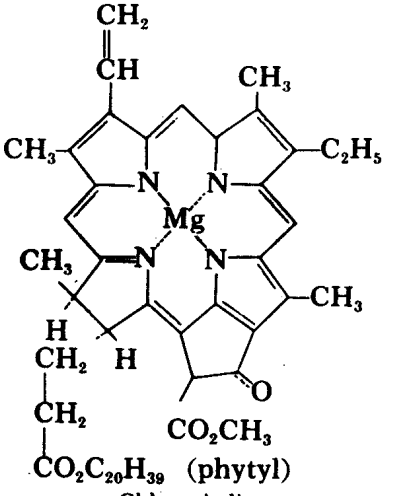
FOREWORD TO THE STUDENT

The practice of organic chemistry is both an art and a science. The elucidation of the structure[1] and the total synthesis[2] of such a complex compound as, for example, chlorophyll-a, is sheer artistry. But underlying such brilliant work, and indispensable to it, is a thorough mastery of the fundamental scientific principles upon which the separation, purification, identification, and reactions of organic compounds are based.



Chlorophyll-a

It is the purpose of your laboratory work in organic chemistry both to train you in the art and to school you in the scientific principles upon which the art is founded. It may perhaps be possible for you to muddle through the laboratory phase of your course in organic chemistry and achieve results of a sort without a clear understanding of what you are doing and why you are doing it. But truly distinguished achievement, characterized by rapid, efficient performance of each experiment with isolation of a high purity product in maximum yield, will require on your part a real understanding of the principles which underlie all laboratory techniques and procedures. Such achievement does not just happen. It will be possible only if you plan your work carefully in advance and work purposefully during each laboratory period.

Above all, it is important that you study the experiment of the day care-

[1] Richard Willstatter (1872–1942) was the successor to Baeyer at Munich. The investigation of the structure of chlorophyll was one of his most important accomplishments in research.

[2] Robert B. Woodward and his associates at Harvard University succeeded in attaining a total synthesis of chlorophyll-a. *J. Amer. Chem. Soc.*, 82, 3800 (1960).

fully *before* coming to the laboratory. ***In advance*** (1) master the theoretical principles involved, (2) in your notebook list in condensed form the equipment and chemicals (with amounts) required, so that a single trip to the stockroom or balance will suffice, and (3) write your own outline of the procedure in a form you can follow rapidly and write the balanced equations for the reactions involved.

In the laboratory, work with an open, inquiring mind, recording at once, not what you think is supposed to happen, but what you actually observe. A good scientist is, first of all, a careful observer. As you work, ask yourself questions; if, after a determined effort you cannot answer them, do not hesitate to ask your instructor. Many of the most provocative and interesting questions in organic chemistry are raised by beginning students.

Your experiments will be graded on the quality and quantity of your product, your technique, your notebook report, and your understanding of the practice and principles involved in the experiment as revealed by oral or written quizzes which your instructor will give from time to time. The quality of your product is judged from physical appearance and from such physical constants as melting point, boiling point, density, and refractive index. The quantity is expressed as the "percentage yield."

The percentage yield for any reaction represents the extent, expressed in per cent, to which the reactants were converted into the isolated product. It is calculated as follows:

$$\text{Percentage yield} = \left(\frac{\text{actual yield}}{\text{theoretical yield}}\right)(100)$$

where the actual yield = the weight or volume (for a gas) of product actually isolated

and the theoretical yield = the weight or volume (for a gas) of product that would be formed from the starting materials used if the reaction proceeded 100 per cent as indicated by the balanced equation.

For example, let us calculate the percentage yield of the ester, ethyl acetate, if 33.6 g. were isolated from the reaction of 35.0 g. of acetic acid with 75.0 g. of ethyl alcohol. According to the balanced equation (see Experiment 31),

$$\underset{\substack{60.1\text{ g.}\\(1\text{ mole})}}{CH_3{-}\overset{\overset{O}{\|}}{C}{-}OH} + \underset{\substack{46.1\text{ g.}\\(1\text{ mole})}}{C_2H_5OH} \rightleftharpoons \underset{\substack{88.1\text{ g.}\\(1\text{ mole})}}{CH_3{-}\overset{\overset{O}{\|}}{C}{-}OC_2H_5} + HOH$$

theoretically 60.1 g. (1 mole) of acetic acid reacts with 46.1 g. (1 mole) of ethyl alcohol to yield 88.1 g. (1 mole) of ethyl acetate.

Hence the theoretical yield of ethyl acetate from 35.0 g. of acetic acid is

$$\left(\frac{35.0\text{ g.}}{60.1\text{ g.}}\right)(88.1\text{ g.}) = 51.3\text{ g.}$$

and from 75.0 g. of ethyl alcohol,

$$\left(\frac{75.0\text{ g.}}{46.1\text{ g.}}\right)(88.1\text{ g.}) = 143.3\text{ g.}$$

In other words, the ethyl alcohol is present in theoretical excess, and the acetic acid is the limiting reagent in determining the theoretical yield. No matter how great an excess of ethyl alcohol is added, the maximum yield of ethyl acetate that can be obtained from 35.0 g. of acetic acid is 51.3 g. This, then, is the theoretical yield of ethyl acetate. The percentage yield is equal to

$$\left(\frac{33.6\text{ g.}}{51.3\text{ g.}}\right)(100\%) = 65.5\%$$

Esterification is an equilibrium reaction and addition of an excess of the cheaper reagent, ethyl alcohol, through the mass action effect, increases the weight of ethyl acetate produced per unit weight of the more costly reactant, acetic acid.

You may prefer to solve problems of this type by calculating the number of moles of the limiting reactant used and of the product isolated. Thus, in the synthesis of ethyl acetate, we see from the equation that, for every 1 mole of acetic acid which reacts, 1 mole of ethyl acetate is formed. But 35.0 g. of acetic acid is only

$$\frac{35.0\text{ g.}}{60.1\text{ g./mole}} = 0.582\text{ mole of acetic acid}$$

and the theoretical yield of ethyl acetate is therefore 0.582 mole of ethyl acetate.

The actual yield of ethyl acetate, 33.6 g., is

$$\frac{33.6\text{ g.}}{88.1\text{ g./mole}} = 0.381\text{ mole of ethyl acetate}$$

The percentage yield is therefore

$$\left(\frac{0.381\text{ mole}}{0.582\text{ mole}}\right)(100\%) = 65.5\%$$

If, in the synthesis of ethyl iodide (Experiment 20), 3.5 g. of phosphorus, 25.0 g. of iodine, and 19.7 g. of ethyl alcohol are used, it can be calculated from the balanced equation for the over-all reaction

$$\underset{\substack{61.96\text{ g.}\\(2\text{ moles})}}{2P} + \underset{\substack{761.5\text{ g.}\\(3\text{ moles})}}{3I_2} + \underset{\substack{276.4\text{ g.}\\(6\text{ moles})}}{6CH_3{-}CH_2OH} \rightarrow \underset{\substack{935.9\text{ g.}\\(6\text{ moles})}}{6C_2H_5I} + 2H_3PO_3$$

that the iodine, by far the most expensive of the reagents, is the limiting reagent. From 25.0 g. of iodine, the theoretical yield of ethyl iodide is

$$\left(\frac{25.0\text{ g.}}{761.5\text{ g.}}\right)(935.9\text{ g.}) = 30.7\text{ g.}$$

If the actual yield of ethyl iodide is 21.0 g., then the

$$\text{percentage yield} = \left(\frac{21.0 \text{ g.}}{30.7 \text{ g.}}\right)(100\%) = 68.4\%$$

Working the same problem on a mole basis, we can see from the equation that 3 moles of iodine yield theoretically 6 moles of ethyl iodide, or 1 mole of iodine yields 2 moles of ethyl iodide. But 25.0 g. of iodine is only

$$\frac{25.0 \text{ g.}}{253.84 \text{ g./mole}} = 0.0985 \text{ mole of iodine}$$

and yields theoretically (2) (0.0985) = 0.197 mole of ethyl iodide.

The actual yield of ethyl iodide, 21.0 g., is

$$\frac{21.0 \text{ g.}}{155.98 \text{ g./mole}} = 0.135 \text{ mole of ethyl iodide}$$

The percentage yield is then

$$\left(\frac{0.135 \text{ mole}}{0.197 \text{ mole}}\right)(100\%) = 68.4\%$$

The percentage yield obtained in a given reaction is an indication both of the suitability of the reaction for synthetic purposes and of the skill and technique of the investigator. Many organic reactions are reversible, most are accompanied by competing side reactions which lead to the formation of by-products, and almost all are relatively slow as compared, for example, to the familiar reaction of sodium hydroxide solution with hydrochloric acid or of silver nitrate with sodium chloride in solution. For these reasons, careful control of such factors as time of reaction, temperature, solvent, concentration, pH, product removal, and judicious use of catalysts are of the utmost importance in organic reactions. But even under optimum experimental conditions, very few organic reactions afford the desired product in 100 per cent yield.

For every reaction there is an inherent limitation on possible yield imposed by the nature of the reaction itself and competition from side reactions. Other factors being equal, a reaction in which a maximum yield of 95 per cent may be achieved is certainly to be preferred above one in which the maximum attainable yield is 25 per cent. But the percentage of this limiting yield that is actually realized in a given case depends upon the personal factor; it is at this point that the skill, technique, and ingenuity of the experimenter come into play. Nothing will be more personally rewarding or gratifying to you in the organic laboratory than the

type of intelligent and skilled workmanship which exploits each reaction to its fullest extent.

Writing Your Laboratory Notebook

We have stated in the Foreword and repeat here for emphasis that before coming to the laboratory you should:

(a) Read carefully the discussion and directions for the experiment in this manual.

(b) In your textbook, turn to the subject of the experiment and study the presentation given there.

(c) Write up the experiment in your notebook using the outline on p. xxi, omitting only the observations and answers to questions that inherently cannot be supplied until the work has been done.

Careful observance of this plan for your laboratory work is the very essence of meaningful experimentation. By following such a prearranged plan you will have no trouble in completing the laboratory work according to the time schedule given with the experiment. On the contrary, you will wonder why the schedule is so slow.

Temperatures are in degrees centigrade.

A time schedule, in minutes, is given for completing almost every assignment within the three-hour laboratory period. This schedule is given in bold-face numbers in the margins.

It should be kept in mind that this time schedule is valid only if all necessary apparatus is available to the student when he enters the laboratory. In Experiment 2, for example, the student will probably not be able to keep up with the time schedule if he first has to learn how to prepare melting-point tubes and then make all that he needs. If ready-made melting-point tubes are available, however, he will have no difficulty in maintaining the schedule. Similar considerations apply to many of the experiments.

Safe Working Procedures and Avoidance of Accidents

All too often chemical laboratories are the scene of accidents—mostly minor ones but some of serious nature. These so-called accidents do not merely *happen;* they *are caused* by improper or careless procedures. Close observance of the precautions, given in the following list, will prevent directly most such mishaps and indirectly will aid the student in acquiring those habits of safety which will be of inestimable value to him not only in the laboratory but elsewhere as well.

1. Avoid Cuts and Lacerations

Cuts from broken pieces of glass tubing are among the most frequent accidents in the laboratory. These may be avoided by observing the following rules when inserting a glass tube or thermometer into the hole in a stopper:

a) Bore the hole in the stopper to a suitable size.
b) Lubricate the tube with water, soap solution, or glycerol.
c) Protect your hands with a towel.
d) Rotate the tube slowly and apply pressure gently.
e) Do not use one arm of a bent tube as a lever for application of excessive pressure, but grip the sidearm close to the cork or stopper.
f) Soften a cork stopper in the cork roller before boring a hole in it. The rolling aids in boring a smooth hole which fits the tube securely. The same rules apply to the removal of a tube from a stopper.

2. Guard Against Fire

Remember that many solvents used in the organic chemistry laboratory are flammable. Observe the following precautions:

a) Flammable solvents of boiling point less than 100° should be distilled, heated, or evaporated on the steam bath, not over a Bunsen burner. This includes methanol, ethanol, acetone, benzene, petroleum ether, ligroin, etc.
b) Flammable solvents should be contained in flasks rather than in open beakers.
c) Keep flasks containing flammable solvents away from your own and also from your neighbor's Bunsen burner.
d) Bottles of flammable solvents should not be on your work bench near a lighted burner. Keep them on the side shelf.
e) Do not pour flammable liquids into the waste crocks.

3. Extinguishing Fires

The laboratory is provided with fire extinguishers, a fire blanket, an emergency shower, a bucket of sand, and perhaps other items.

a) Know the location and use of these pieces of equipment.
b) Remember that burning sodium reacts with carbon tetrachloride with explosive vigor. Smother sodium fires with dry sand or sodium carbonate, scrape the material into a pan and carry it out of doors.

4. Protect Your Eyes

a) Wear goggles whenever you are performing an experiment in which there is danger of spattering.
b) Wearing safety glasses in the laboratory at all times is an excellent safety measure.
c) Avoid looking into the open mouth of a test tube or flask in which a reaction is being conducted.

5. Shun Explosive Mixtures

Strong oxidizing agents and easily oxidized material (reducing agents) must be mixed with extreme caution and in small amounts. Never add nitric acid to a flask containing alcohol or other easily oxidized material. The reaction between nitric acid and organic reducing agents can be so violent that a dangerous explosion may result.

6. General Procedures

a) Absorb escaping obnoxious gases in water or other suitable medium or conduct the experiment in a fume hood.
b) Keep the gas and water valves closed except when these utilities are needed.
c) Insoluble waste such as filter papers, match stems, and kindred items must be thrown into the waste jars—not into the sinks.
d) Ordinary rubber stoppers are never used on apparatus where they can be subjected to the action of organic solvents. Most such solvents attack the rubber and cause contamination of the product.
e) Never work in the laboratory alone. A minor mishap that may be of little consequence if someone is at hand to help you, may be serious if you are alone.

f) Do not swing a graduated cylinder or similar piece of glassware in a circular arc to dry it by centrifugal force. Such spattering of your neighbors is inexcusable.
g) Do not put scraps of metallic sodium into the sinks or waste jars. Cover them with kerosene and ask the instructor for the local arrangements for disposing of them.
h) Be neat in your work. If you spill something, clean it up.
i) Never heat an enclosed system and never completely close an assembly of apparatus in which a gas is being evolved; always provide a vent of suitable size.
j) Work with bromine, phosphorus trichloride, acetyl chloride, benzoyl chloride, and other obnoxious materials in the hood. Each laboratory has its own provisions for handling such substances. Ask the instructor for directions.
k) Much of the sloppiness of laboratory tables is caused by a too rapid stream of water flowing through the condenser; a gentle stream is usually sufficient.

Carcinogenic Compounds

The National Institute for Occupational Safety and Health lists the following compounds as potential carcinogens. If any of these compounds are ever used in the laboratory, extreme care should be exercised to avoid inhalation or contact with the skin.

4-Nitrobiphenyl
α-Naphthylamine
4,4′-Methylene bis(2-chloroaniline)
Methyl chloromethyl ether
3,3′-Dichlorobenzidine
Bis-(Chloromethyl) ether
β-Naphthylamine
Benzidine
4-Aminodiphenyl
Ethyleneimine
β-Propiolactone
2-Acetylaminofluorene
4-Dimethylaminoazobenzene
N-Nitrosodimethylaniline

Outline Reporting a Laboratory Preparation

Experiment No. __

Title

Materials

Theoretical Yield

Actual Yield

Percentage Yield

Boiling Range (or M.P.)

B.P. or M.P. (from Handbook)

Density (from Handbook)

Equations

Procedure. *State or outline, briefly but clearly, the working methods so* that the experiment may be repeated from your notebook without reference to the Manual. All statements should be impersonal. In many instances one page of your notebook will suffice for the report of the preparation of an organic compound. The description of the chemical properties of a compound, or a series of related compounds can usually be given best by short statements accompanied by chemical equations.

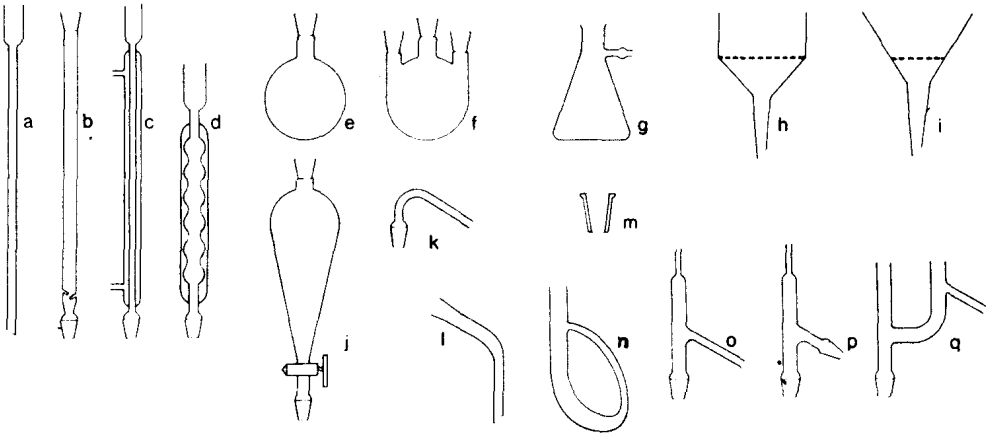
Answers to questions

Suggested List of Desk Equipment

Any suitable type of apparatus may be used for the experiments described in this manual. The authors recommend, however, that standard taper

equipment be employed because it is so easily adaptable to the needs of various laboratory procedures that a few pieces serve many purposes. Furthermore, in addition to its advantage in convenience and in saving much of the student's time, its use introduces the student to modern research techniques.

The recommended list of apparatus is shown below. Unless otherwise indicated, all ground joints are of the 24/40 size.



Types of apparatus used in the organic chemistry laboratory. (a) Air condenser. (b) Fractionating column. (c) Condenser—West type. (d) Condenser—Allihn type. (e) Boiling flask. (f) Boiling flask—3-neck. (g) Filter flask. (h) Büchner funnel. (i) Hirsch funnel. (j) Separatory funnel. (k) Distilling tube. (l) Adapter. (m) Reducing bushing. (n) Thiele melting point tube. (o) and (p) Distilling heads. (q) Claisen distilling head.

Suggested List of Laboratory Equipment

1 Adapter.
7 Beakers, 1 set; 50, 100, 150, 250, 400, 600, 1000 ml.
3 Condensers; air, West drip tip, Allihn, 300-mm jacket.
3 Cork rings to support flasks.
2 Cylinders, graduated; 25, 100 ml.
2 Distilling heads; regular, Claisen.
4 Flasks, boiling; 50, 100, 200, 500 ml.
4 Flasks, Erlenmeyer; 2–125, 2–250 ml.
1 Flask, three-neck; 500 or 1000 ml.
1 Flask, filter; 500 ml.
2 Funnels, separatory; 60, 250 ml.
1 Funnel, Büchner or Hirsch, 5.5 cm.
2 Thermometers; −10° to 110°, −10° to 360°.
Burners, brushes, clamps, test tubes, etc., as required.
Special equipment may be withdrawn from the stockroom.

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