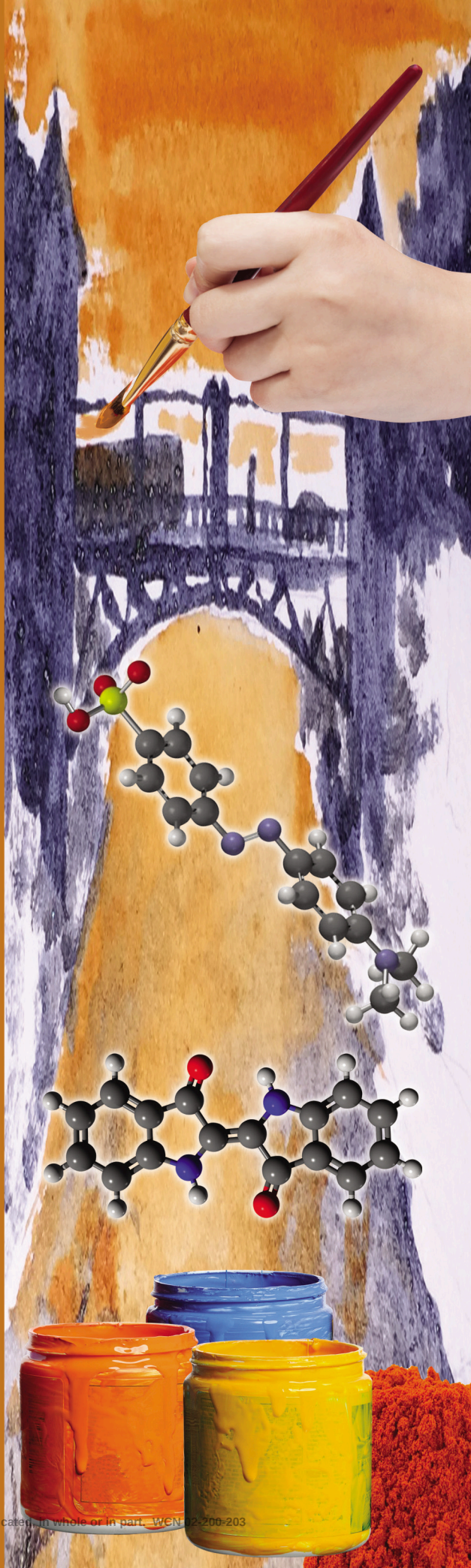
CENGAGE LEARNING LABORATORY SERIES *for*
Organic Chemistry

A Microscale Approach *to* Organic Laboratory Techniques

SIXTH EDITION

Donald L. Pavia
Gary M. Lampman
George S. Kriz
Randall G. Engel



A Microscale Approach *to* Organic Laboratory Techniques

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This book is dedicated to
our organic chemistry laboratory students

Preface

STATEMENT OF MISSION AND PURPOSE IN REVISING THE TEXTBOOK

The purpose of this lab book is to teach students the techniques of organic chemistry. We desire to share our love of the organic chemistry lab and the joy it brings us with our students! In this edition, we have provided many new, up-to-date experiments that will demonstrate how organic chemistry is evolving. We have updated and improved many of the standard experiments from previous editions, and we have added some new ones. For example, we have included some experiments involving dyes and soap. To make the connection of organic chemistry to our everyday world even more real, we have added a project experiment that asks the students to formulate a paint and then use it in an art project. We think that you will be enthusiastic about this new edition. Many of the new experiments will not be found in other laboratory manuals, but we have been careful to retain all of the standard reactions and techniques, such as the Friedel-Crafts reaction, aldol condensation, Grignard synthesis, and basic experiments designed to teach crystallization, chromatography, and distillation.

SCALE IN THE ORGANIC LABORATORY

Experiments in organic chemistry can be conducted at different scales using varying amounts of chemicals and different styles of glassware. We have two versions of our laboratory textbooks that teach organic laboratory techniques. Our microscale book (A *Microscale Approach to Organic Laboratory Techniques, Sixth Edition*) makes use of ₹14/10 standard tapered glassware. Our vesion of a "macroscale" textbook (*A Small Scale Approach to Organic Laboratory Techniques, Fourth Edition*) uses the traditional larger scale ₹19/22 standard tapered glassware. The fourth edition of our small scale book was published in 2016.

Over the years that we have been involved with developing experiments, we have learned that students can easily adjust to working with the small laboratory equipment that is used in this microscale book. As students and faculty learn to appreciate the impact of laboratory classroom experiments on the environment, they become more aware that it is not necessary to consume large quantities of chemicals. Students come to appreciate the importance of reducing waste generated in the organic laboratory. All of us, students and faculty alike, are becoming more "green."

MAJOR FEATURES OF THE TEXTBOOK THAT WILL BENEFIT THE STUDENT

When we published our first organic laboratory textbook in 1976, a major goal was to demonstrate to students how organic chemistry significantly impacts our lives in the real world. This was accomplished by including experiments with a real-world connection and by including many topical essays that related the experiments to everyday world applications. In this edition, we have taken this emphasis to a new level. For example, we have added two new experiments involving the synthesis of two widely used dyes, methyl orange and indigo. These dyes can then be used to formulate a paint in the experiment Formulation of a Paint and Art Project. Not only do students learn about the chemistry involved in the formulation of a paint, but they also paint a picture of their own creation. Many students at North Seattle College and the University of Washington report that this is one of their favorite experiments in the organic laboratory class! We have also added a new essay on Dyes that gives further examples of how these new experiments are related to our everyday lives.

Another real-world experiment that we are especially excited about is Preparation of Soap. This experiment was developed by one of our organic chemistry students, who is a professional soap maker! Students learn about the chemistry of soap making, and they make a bar of soap that can be used at home. We have also included a new essay on Soap.

A number of experiments are linked together to create multistep syntheses. The advantage of this approach is that you will be doing something different from your neighbor in the laboratory. Wouldn't you like to be carrying out an experiment that is not the same as your neighbor's? Maybe you will be synthesizing a new compound that hasn't been reported in the chemical literature! You and your fellow students will not all be doing the same reaction on the same compound: for example, some of you will be carrying out the chalcone reaction, others the "green" epoxidation, and still others the cyclopropanation of the resulting chalcones.

GREEN CHEMISTRY

We have continued an emphasis on Green Chemistry in this edition. The Green Chemistry experiments decrease the need for hazardous waste disposal, leading to reduced contamination of the environment. These experiments use less toxic reactants and solvents. For example, water is used as a solvent in some experiments. Almost all experiments have been reduced in scale compared to the traditional macroscale experiments. Experiments that are particularly good for illustrating the Green Chemistry approach include Biodiesel, Chiral Reduction of Ethyl Acetoacetate, Aqueous-Based Organozinc Reactions, Grubbs-Catalyzed Metathesis of Eugenol with 1,4-Butaanediol, Diels-Alder Reaction with Anthracene-9-methanol, and Green Epoxidation of Chalcones. We have also added a new Green oxidation reaction using Oxone® in an Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol. Oxone® is a more reliable alternative to bleach, which we have used in previous editions of this textbook.

In keeping with the Green Chemistry approach, we have suggested an alternative way of approaching qualitative analysis. This approach makes extensive use of spectroscopy to solve the structure of organic unknowns. In this approach, some of the traditional tests have been retained, but the main emphasis is on using

spectroscopy. In this way, we have attempted to show students how to solve structures in a more modern way, similar to that used in a research laboratory. The added advantage to this approach is that waste is considerably reduced.

NEW TO THIS EDITION

Many of the new experiments in this edition demonstrate the relationship between organic chemistry and our everyday lives. This edition also includes updating of the essays and the chapters on techniques. New experiments added for this edition include:

Experiment 26	Preparation of Soap
Experiment 33	An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol
Experiment 46	Preparation of Methyl Orange
Experiment 47	Preparation of Indigo
Experiment 48	Formulation of a Paint and Art Project

New Essays include:

Soap
Dyes

As in previous editions, the technique chapters include both microscale and macroscale techniques. Many of the references in the technique chapters have been updated. New material on diastereotopic protons has been added to Technique 26, Nuclear Magnetic Resonance Spectroscopy. Technique 29, Guide to the Chemical Literature, has been revised.

SUPPORTING RESOURCES

Please visit http://www.cengage.com/chemistry/pavia/microorglab6e for information about student and instructor resources for this text.

ACKNOWLEDGMENTS

We owe our sincere thanks to the many colleagues who have used our textbooks and who have offered their suggestions for changes and improvements to our laboratory procedures or discussions. Although we cannot mention everyone who has made important contributions, we must make special mention of Albert Burns (North Seattle College), Charles Wandler (Western Washington University), Emily Borda (Western Washington University), Frank Deering (North Seattle College), Jacob Frank (North Seattle College), Gregory O'Neil (Western Washington University), James Vyvyan (Western Washington University), Khushroo Daruwala (University of Washington Bothell), Scott Clary (North Seattle College), and Timothy Clark (University of San Diego).

In preparing this new edition, we have also attempted to incorporate the many improvements and suggestions that have been forwarded to us by the many instructors who have been using our materials over the past several years.

We are especially grateful to James Patterson, faculty member of North Seattle College, who has given us permission to include several of his experiments in our

textbooks. His ideas and enthusiastic support of our textbooks for many years have contributed immensely to the success of our textbooks.

We thank all who contributed, with special thanks to our Senior Product Manager, Lisa Lockwood; Associate Content Developer, Brendan Killion; Content Project Manager, James Zayicek; Associate Marketing Manager, Ana Albinson; and Associate Program Manager, Sharib Asrar at Lumina Datamatics.

We are especially grateful to the students and friends who have volunteered to participate in the development of experiments or who offered their help and criticism. We owe special thanks to Sean Ichiun Choe, organic chemistry student at North Seattle College, who developed and wrote most of Experiment 24 (Preparation of Soap). Sean's expertise as a soap maker in the real world is reflected in this valuable addition to our book. Sean also made valuable contributions to the Soap essay.

We are also grateful to Alish O'Sullivan, student at North Seattle College, who painted the picture of the Montlake Bridge, which appears on the cover of this textbook. This painting was created by Alish while performing the new experiment, Formulation of a Paint and Art Project, which appears in this textbook.

Finally, we wish to thank our families and special friends, especially Neva-Jean Pavia, Marian Lampman, and Karin Granstrom, for their encouragement, support, and patience.

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How To Use This Book

OVERALL STRUCTURE OF THE BOOK

This textbook is divided into two major sections (see Table of Contents). The first section, which includes Part One through Part Five, contains all of the experiments in this book. The second major section includes only Part Six, which contains all of the important techniques you will use in performing the experiments in this book. Interspersed among the experiments in Part One through Part Three is a series of essays. The essays provide a context for many of the experiments and often relate the experiment to real world applications. When your instructor assigns an experiment, he or she will often assign an essay and/or several techniques chapters along with the experiment. Before you come to lab, you should read all of these. In addition, it is likely that you will need to prepare some sections in your laboratory notebook (see Technique 2) before you come to the lab.

STRUCTURE OF THE EXPERIMENTS

In this section we discuss how each experiment is organized in the textbook. To follow this discussion, you may want to refer to a specific experiment, such as Experiment 13.

Multiple Parts Experiments

Some experiments, such as Experiment 13, are divided into two or more individual parts that are designated by the experiment number and the letters A, B, etc. In some experiments, like Experiment 13, each part is a separate but related experiment, and you will most likely perform only one part. In Experiment 13, you would do Experiment 13A (Isolation of Caffeine from Tea Leaves) or Experiment 13B (Isolation of Caffeine from a Tea Bag). In other experiments, for example Experiment 32, the various parts can be linked together to form a multistep synthesis. In a few experiments, such as Experiment 22, the last part describes how you should analyze your final product.

Featured Topics and Techniques Lists

Directly under the title of each experiment (see Experiment 13), there will be a list of topics. These topics may explain what kind of experiment it is, such as isolation of a natural product or Green Chemistry. The topics may also include major techniques that are required to perform the experiment, such as crystallization or extraction.

Required Reading

In the introduction to each experiment, there will be a section labeled Required Reading. Within this section, some of the required readings are labeled Review and some are labeled New. You should always read the chapters listed in the New section. Sometimes it will also be helpful to do the readings in the Review section.

Special Instructions

You should always read this section since it may include instructions that are essential to the success of the experiment.

Suggested Waste Disposal

This very important section gives instructions on how to dispose of the waste generated in an experiment. Often your instructor will provide you with additional instructions on how to handle the waste.

Notes to Instructor

It will usually not be necessary to read this section. This section provides special advice for the instructor that will help to make the experiment successful.

Procedure

This section provides detailed instructions on how to carry out the experiments. Within the procedure, there will be many references to the techniques chapters, which you may need to consult in order to perform an experiment.

Report

In some experiments, specific suggestions for what should be included in the laboratory report will be given. Your instructor may refer to these recommendations or may have other directions for you to follow.

Questions

At the end of most experiments will be a list of questions related to the experiment. It is likely that your instructor will assign at least some of these questions along with the laboratory report.

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PART 1

Introduction to Basic Laboratory Techniques

Introduction to Microscale Laboratory

This textbook discusses the important laboratory techniques of organic chemistry and illustrates many important reactions and concepts. In the traditional approach to teaching this subject, the quantities of chemicals used were on the order of 5–100 grams, and glassware was designed to contain up to 500 mL of liquid. This scale of experiment we might call a **macroscale** experiment. The approach used here, a **microscale** approach, differs from the traditional laboratory course in that nearly all the experiments use small amounts of chemicals. Quantities of chemicals used range from about 50 to 1000 *milli*grams (0.050–1.000 g), and glassware is designed to contain less than 25 mL of liquid. The advantages include improved safety in the laboratory, reduced risk of fire and explosion, and reduced exposure to hazardous vapors. This approach decreases the need for hazardous waste disposal, leading to reduced contamination of the environment. You will learn to work with the same level of care and neatness that has previously been confined to courses in analytical chemistry.

This experiment introduces the equipment and shows how to construct some of the apparatus needed to carry out further experiments. Detailed discussion of how to assemble apparatus and how to practice the techniques is found in Part Six ("The Techniques") of this textbook. This experiment provides only a brief introduction, sufficient to allow you to begin working. You will need to read the techniques chapters for more complete discussions.

Microscale organic experiments require you to develop careful laboratory techniques and to become familiar with apparatus that is somewhat unusual, compared with traditional glassware. We strongly recommend that each student do Laboratory Exercises 1 and 2. These exercises will acquaint you with the most basic microscale techniques. To provide a strong foundation, we further recommend that each student complete most of Experiments 2 through 18 in Part One of this textbook before attempting any other experiments in the textbook.

READ Technique 1 "Laboratory Safety."

HEATING METHODS

Aluminum Block

The most convenient means of heating chemical reactions on a small scale is to use an **aluminum block.** An aluminum block consists of a square of aluminum that has holes drilled into it. The holes are sized to correspond to the diameters of the most common vials and flasks that are likely to be heated. Often there is also

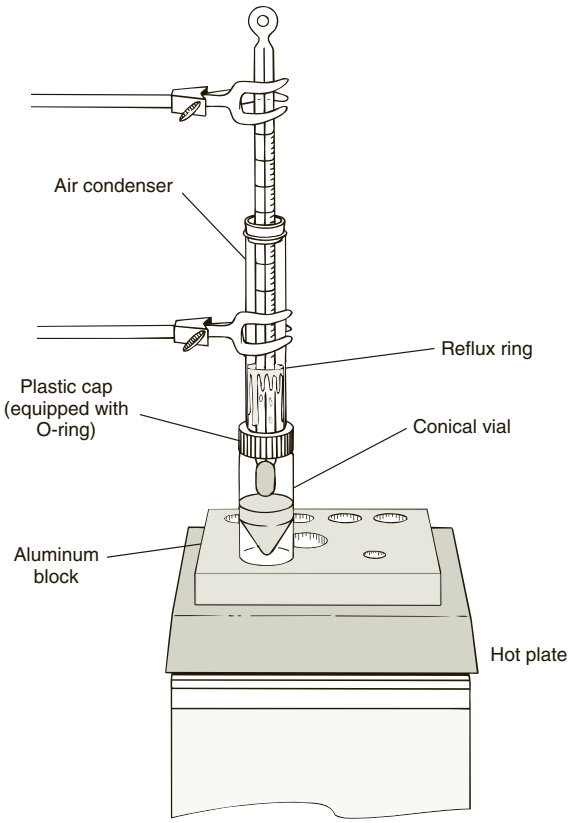


Figure 1
Aluminum block with hot plate and reflux apparatus.

a hole intended to accept the bulb of a thermometer, so that the temperature of the block can be monitored. However, this practice is not recommended. The aluminum block is heated by placing it on a hot plate. An aluminum block is shown in Figure 1. Note that the thermometer in this figure is not used to monitor the temperature of the block.

CAUTION

You should not use a *mercury* thermometer in direct contact with an aluminum block. If it breaks, the mercury will vaporize on the hot surface. Instead, use a nonmercury thermometer, a metal dial thermometer, or a digital electronic temperature-measuring device. See Technique 6, Section 6.1.

It is recommended that an equipment kit contain two aluminum blocks, one drilled with small holes and able to accept the conical vials found in the glassware kit and another drilled with larger holes and able to accept small round-bottom flasks. The aluminum blocks can be made from inexpensive materials in a small mechanical shop, or they can be purchased from a glassware supplier.

Sand Baths

Another commonly used means of heating chemical reactions on a small scale is to use a **sand bath.** The sand bath consists of a Petri dish or a small crystallizing dish that has been filled to a depth of about 1 cm with sand. The sand bath is also heated by placing it on a hot plate. The temperature of the sand bath may be monitored by clamping a thermometer in position so that the bulb of the thermometer is buried in the sand. A sand bath, with thermometer, is shown in Figure 2.

We recommend that an aluminum block, rather than a sand bath, be used as a heating source whenever possible. The aluminum block can be heated and cooled quickly, it is indestructible, and there are no problems with spillage of sand.

Water Bath

When precise control at lower temperatures (below about 80°C) is desired, a suitable alternative is to prepare a **water bath.** The water bath consists of a beaker filled to the required depth with water. The hot plate is used to heat the water bath to the desired temperature. The water in the water bath can evaporate during heating. It is useful to cover the top of the beaker with aluminum foil to diminish this problem.

CONICAL REACTION VIALS

One of the most versatile pieces of glassware contained in the microscale organic glassware kit is the **conical reaction vial.** This vial is used as a vessel in which organic reactions are performed. It may serve as a storage container. It is also used for extractions (see Technique 12). A reaction vial is shown in Figure 3.

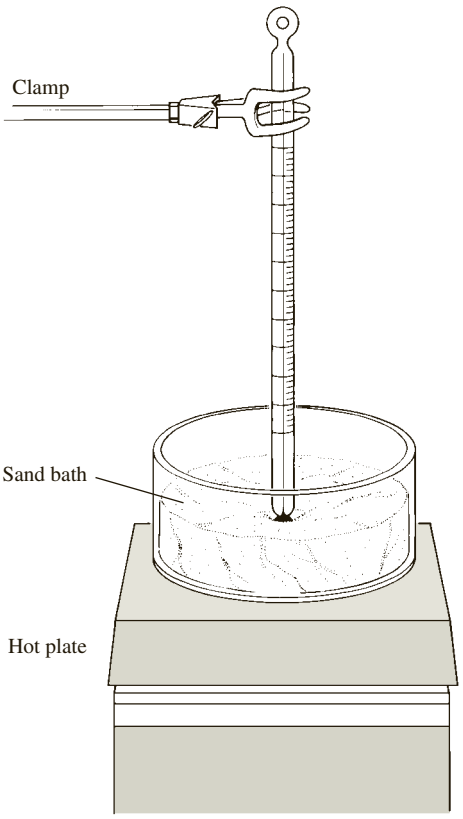


Figure 2
Sand bath with hot plate and thermometer.

The flat base of the vial allows it to stand upright on the laboratory bench. The interior of the vial tapers to a narrow bottom. This shape makes it possible to withdraw liquids completely from the vial, using a disposable Pasteur pipette. The vial has a screw cap, which tightens by means of threads cast into the top of the vial. The top also has a ground-glass inner surface. This ground-glass joint allows you to assemble components of glassware tightly.

The plastic cap that fits the top of the conical vial has a hole in the top. This hole is large enough to permit the cap to fit over the inner joints of other components of the glassware kit (see Figure 4). A Teflon insert, or liner, fits inside the cap to cover the hole when the cap is used to seal a vial tightly. Notice that only one side of the liner is coated with Teflon; the other side is coated with a silicone rubber. The Teflon side generally is the harder side of the insert, and it will feel more slippery. The Teflon side should always face toward the inside of the vial. An O-ring fits inside the cap when the cap is used to fasten pieces of glassware together. The cap and its Teflon insert are shown in the expanded view in Figure 3.

NOTE: Do not use the O-ring when the cap is used to seal the vial.

You can assemble the components of the glassware kit into one unit that holds together firmly and clamps easily to a ring stand. Slip the cap from the conical vial over the inner (male) joint of the upper piece of glassware and fit a rubber O-ring over the inner joint. Then assemble the apparatus by fitting the inner ground-glass joint into the outer (female) joint of the reaction vial and tighten the screw cap to attach the entire apparatus firmly together. The assembly is illustrated in Figure 4.

The walls of the conical vials are made of thick glass. Heat does not transfer through these walls very quickly. This means that if the vial is subjected to rapid changes in temperature, strain building up within the glass walls of the vial may cause the glass to crack. For this reason, do not attempt to cool these vials quickly by running cold water on them. It is safer to allow them to cool naturally by allowing them to stand.

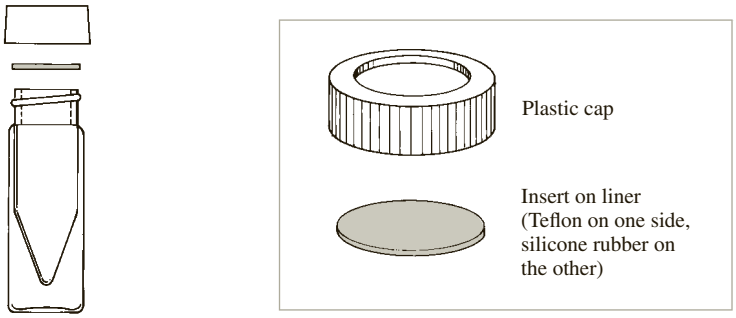


Figure 3
A conical reaction vial. (The inset shows an expanded view of the cap with its Teflon insert.)

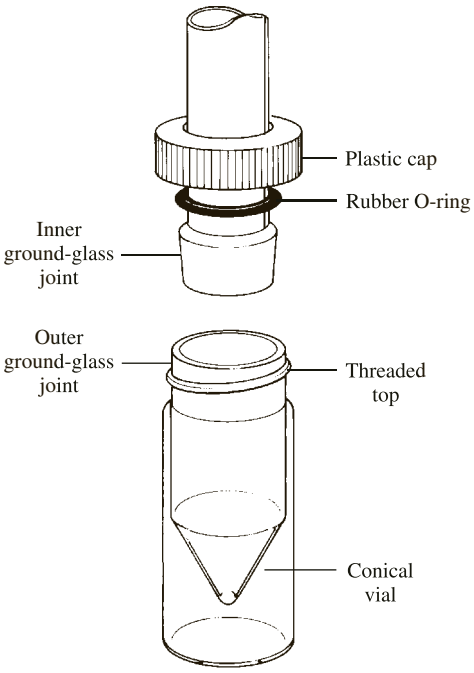


Figure 4
Assembling glassware components.

Although the conical vials have flat bottoms intended to allow them to stand up on the laboratory bench, this does not prevent them from falling over.

NOTE: It is good practice to store the vials standing upright inside small beakers.

The vials are somewhat top-heavy, and it is easy to upset them. The beaker will prevent the vial from falling over onto its side.

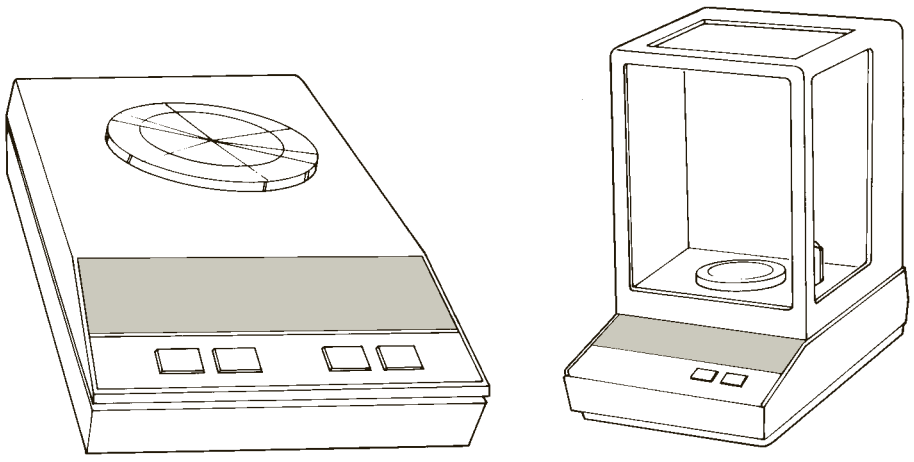
MEASUREMENT OF SOLIDS

Weighing substances to the nearest milligram requires that the weighings be done on a sensitive **top-loading balance** or an **analytical balance.**

NOTE: You must not weigh chemicals directly on balance pans.

Many chemicals can react with the metal surface of the balance pan and thus ruin it. All weighings must be made into a container that has been weighed previously (**tared**). This tare weight is subtracted from the total weight of container plus sample to give the weight of the sample. Some balances have a built-in compensating feature, the tare button, that allows you to subtract the tare weight of the container automatically, thus giving the weight of the sample directly. A top-loading and an analytical balance are shown in Figure 5.

Balances of this type are quite sensitive and expensive. Take care not to spill chemicals on the balance. It is also important to make certain that any spilled materials are cleaned up immediately.



A. Top-loading balance B. Analytical balance

Figure 5
Laboratory balances.

MEASUREMENT OF LIQUIDS

In microscale experiments, liquid samples are measured using a pipette. When small quantities are used, graduated cylinders do not provide the accuracy needed to give good results. There are two common methods of delivering known amounts of liquid samples, **automatic pipettes** and **graduated pipettes.** When accurate quantities of liquid reagents are required, the best technique is to deliver the desired amount of liquid reagent from the pipette into a container whose tare weight has been determined previously. The container, with sample, is then weighed a second time in order to obtain a precise value of the amount of reagent.

Automatic Pipettes

Automatic pipettes may vary in design, according to the manufacturer. The following description, however, should apply to most models. The automatic pipette consists of a handle that contains a spring-loaded plunger and a micrometer dial. The dial controls the travel of the plunger and is the means used to select the amount of liquid that the pipette is intended to dispense. Automatic pipettes are designed to deliver liquids within a particular range of volumes. For example, a pipette may be designed to cover the range from 10 to 100 μL (0.010 to 0.100 mL) or from 100 to 1000 μL (0.100 to 1.000 mL).

Automatic pipettes must never be dipped directly into the liquid sample without a plastic tip. The pipette is designed so that the liquid is drawn only into the tip. The liquids are never allowed to come in contact with the internal parts of the pipette. The plunger has two **detent,** or "stop," positions used to control the filling and dispensing steps. Most automatic pipettes have a stiffer spring that controls the movement of the plunger from the first to the second detent position. You will find a greater resistance as you press the plunger past the first detent.

To use the automatic pipette, follow the steps as outlined here. These steps are also illustrated in Figure 6.

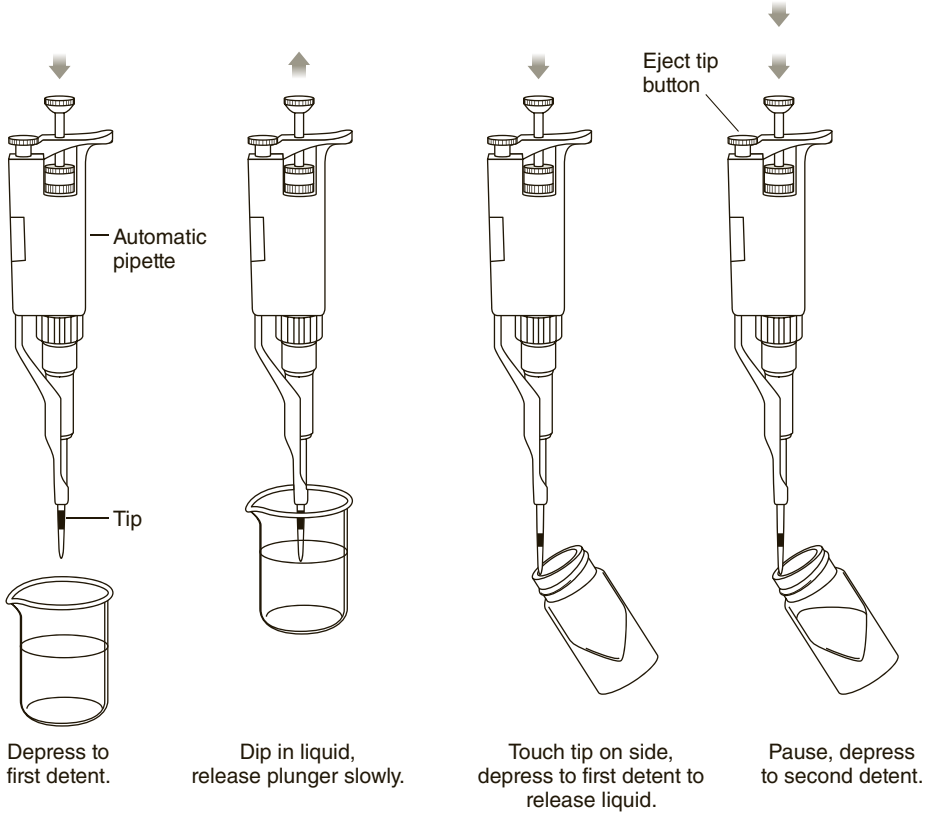


Figure 6
Use of an automatic pipette.

1. Select the desired volume by adjusting the micrometer control on the pipette handle.
2. Place a plastic tip on the pipette. Be certain that the tip is attached securely.
3. Push the plunger down to the first detent position. Do not press the plunger to the second position. If you press the plunger to the second detent, an incorrect volume of liquid will be delivered.
4. Dip the tip of the pipette into the liquid sample. Do not immerse the entire length of the plastic tip in the liquid. It is best to dip the tip only to a depth of about 1 cm.
5. Release the plunger *slowly*. Do not allow the plunger to snap back, or liquid may splash up into the plunger mechanism and ruin the pipette. Furthermore, rapid release of the plunger may cause air bubbles to be drawn into the pipette. At this point, the pipette has been filled.
6. Move the pipette to the receiving vessel. Touch the tip of the pipette to an interior wall of the container.
7. Slowly push the plunger down to the first detent. This action dispenses the liquid into the container.
8. Pause 1–2 seconds and then depress the plunger to its second detent position to expel the last drop of liquid. The action of the plunger may be stiffer in this range than it was up to the first detent.
9. Withdraw the pipette from the receiver. If the pipette is to be used with a different liquid, remove the pipette tip and discard it.

Automatic pipettes are designed to deliver aqueous solutions with an accuracy of within a few percentage points. The amount of liquid actually dispensed varies, however, depending on the viscosity, surface tension, and vapor pressure of the liquid. The typical automatic pipette is very accurate with aqueous solutions but is not always as accurate with other liquids.

Dispensing Pumps

Some scientific supply catalogs offer a series of dispensing pumps. These pumps are useful in a microscale organic laboratory because they are simple to operate, easy to clean, chemically inert, and quite accurate. The interior parts of dispensing pumps are made of Teflon, which renders them inert to most organic solvents and reagents. A dispensing pump is illustrated in Figure 7.

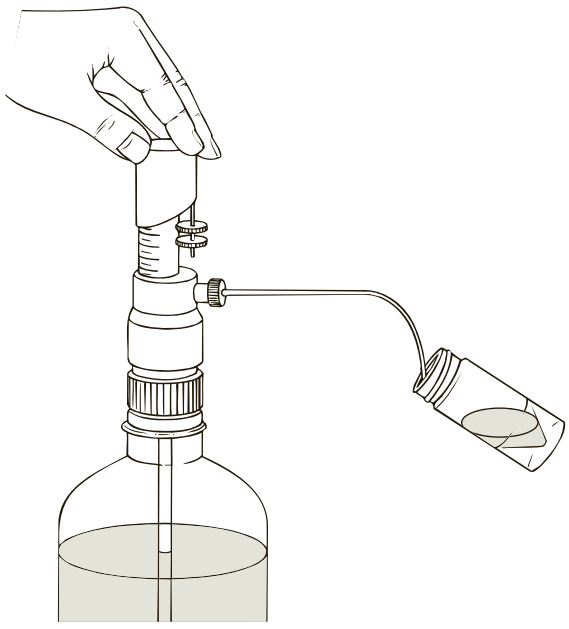


Figure 7
Use of a dispensing pump.

The first step in using a dispensing pump is to adjust the pump so that it dispenses the desired volume of liquid. Normally, the instructor will make this adjustment. Once the pump is adjusted correctly, it is a simple matter to dispense a liquid. Simply lift the head of the pump as far as it will travel. When you release the head, it will fall, and the liquid will issue from the spout. With viscous liquids, the head of the pump may not fall by itself. In such an instance, gently guide the head downward. After the liquid has been dispensed, you should touch the tip of the dispensing tube to an interior wall of the container in order to remove the last drop of liquid.

As with automatic pipettes, dispensing pumps are designed to deliver aqueous solutions with an accuracy of within a few percentage points. The amount of liquid actually dispensed will vary, however, depending on

the viscosity, surface tension, and vapor pressure of the liquid. You should always weigh the liquid to determine the amount accurately.

Graduated Pipettes

A less-expensive means of delivering known quantities of liquid is to use a graduated pipette. Graduated pipettes should be familiar to those of you who have taken general chemistry or quantitative analysis courses. Because they are made of glass, they are inert to most organic solvents and reagents. Disposable serological pipettes may be an attractive alternative to standard graduated pipettes. The 2-mL size of a disposable pipette represents a convenient size for the organic laboratory.

Never draw liquids into the pipettes using mouth suction. A pipette bulb or a pipette pump, not a rubber dropper bulb, must be used to fill pipettes. We recommend the use of a pipette pump. A pipette fits snugly into the pipette pump, and the pump can be controlled to deliver precise volumes of liquids. Control of the pipette pump is accomplished by rotating a knob on the pump. Suction created when the knob is turned draws the liquid into the pipette. Liquid is expelled from the pipette by turning the knob in the opposite direction. The pump works satisfactorily with organic, as well as aqueous, solutions.

An alternative, and less expensive, approach is to use a rubber pipette bulb. Use of the pipette bulb is made more convenient by inserting a plastic automatic pipette tip into a rubber pipette bulb.[1] The tapered end of the pipette tip fits snugly into the end of a pipette. Drawing the liquid into the pipette is made easy, and it is also convenient to remove the pipette bulb and place a finger over the pipette opening to control the flow of liquid.

The calibrations printed on graduated pipettes are reasonably accurate, but you should practice using the pipettes in order to achieve this accuracy. When accurate quantities of liquids are required, the best technique is to weigh the reagent that has been delivered from the pipette.

LABORATORY EXERCISE 1

Option A, Automatic Pipette

Accurately weigh a 3-mL conical vial, with screw cap and Teflon insert, on a balance. Determine its weight to the nearest milligram (nearest 0.001 g). Using the automatic pipette, dispense 0.500 mL of water into the vial, replace the cap assembly (with the insert arranged Teflon side down), and weigh the vial a second time. Determine the weight of water dispensed. Calculate the density of water from your results. Repeat the experiment using 0.500 mL of hexane. Dispose of any excess hexane in a designated waste container. Calculate the density of hexane from your data. Record the results in your notebook, along with your comments on any deviations from literature values that you may have noticed. At room temperature, the density of water is 0.997 g/mL, and the density of hexane is 0.660 g/mL.

Option B, Dispensing Pump

Accurately weigh a 3-mL conical vial, with screw cap and Teflon insert, on a balance. Determine its weight to the nearest milligram (nearest 0.001 g). Using a dispensing pump that has been adjusted to deliver 0.500 mL, dispense 0.500 mL of water into the vial, replace the cap assembly, and weigh the vial a second time. Determine the weight of water dispensed. Calculate the density of water from your results. Repeat the experiment using 0.500 mL of hexane. Dispose of any excess hexane in a designated waste container. Calculate the density of hexane from your data.

[1] This technique was described in Deckey, G. A Versatile and Inexpensive Pipet Bulb. *Journal of Chemical Education*, 57 (July 1980): 526.

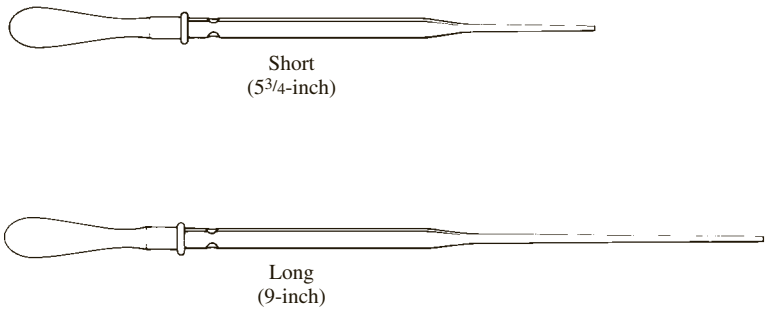


Figure 8
Disposable Pasteur pipettes.

Record the results in your notebook, along with your comments on any deviations from literature values that you may have noticed. See Option A for the density of water and of hexane.

Option C, Graduated Pipette

Accurately weigh a 3-mL conical vial, with screw cap and Teflon insert, on a balance. Determine its weight to the nearest milligram (nearest 0.001 g). Using a 1.0-mL graduated pipette, dispense 0.50 mL of water into the vial, replace the cap assembly, and weigh the vial a second time. Determine the weight of water dispensed. Calculate the density of water from your results. Repeat the experiment using 0.50 mL of hexane. Dispose of any excess hexane in a designated waste container. Calculate the density of hexane from your data. Record the results in your notebook along with your comments on any deviations from literature values that you may have noticed. See Option A for the density of water and of hexane

Disposable (Pasteur) Pipettes

A convenient way of dispensing liquids when a great deal of accuracy is not required is to use a disposable pipette, or Pasteur pipette. Two sizes of Pasteur pipettes are shown in Figure 8. Even though accurate calibration may not be required when these pipettes are used, it is nevertheless handy to have some idea of the volume contained in the pipette. A crude calibration is, therefore, recommended.

LABORATORY EXERCISE 2

Pipette Calibration

On a balance, weigh 0.5 grams (0.5 mL) of water into a 3-mL conical vial. Select a short (5¾-inch) Pasteur pipette and attach a rubber bulb. Squeeze the rubber bulb before inserting the tip of the pipette into the water. Try to control how much you depress the bulb, so that when the pipette is placed into the water and the bulb is completely released, only the desired amount of liquid is drawn into the pipette. (This skill may take some time to acquire, but it will facilitate your use of a Pasteur pipette.) When the water has been drawn up, place a mark with an indelible marking pen at the position of the meniscus. A more durable mark can be made by scoring the pipette with a file. Repeat this procedure with 1.0 gram of water, and make a 1-mL mark on the same pipette.

Additional Pasteur pipettes can be calibrated easily by holding them next to the pipette calibrated in Laboratory Exercise 2 and scoring a new mark on each pipette at the same level as the mark placed on the calibrated pipette. We recommend that several Pasteur pipettes be calibrated at one time for use in future experiments.

Extraction

A technique frequently applied in purifying organic reaction products is **extraction.** In this method, a solution is mixed thoroughly with a second solvent. The second