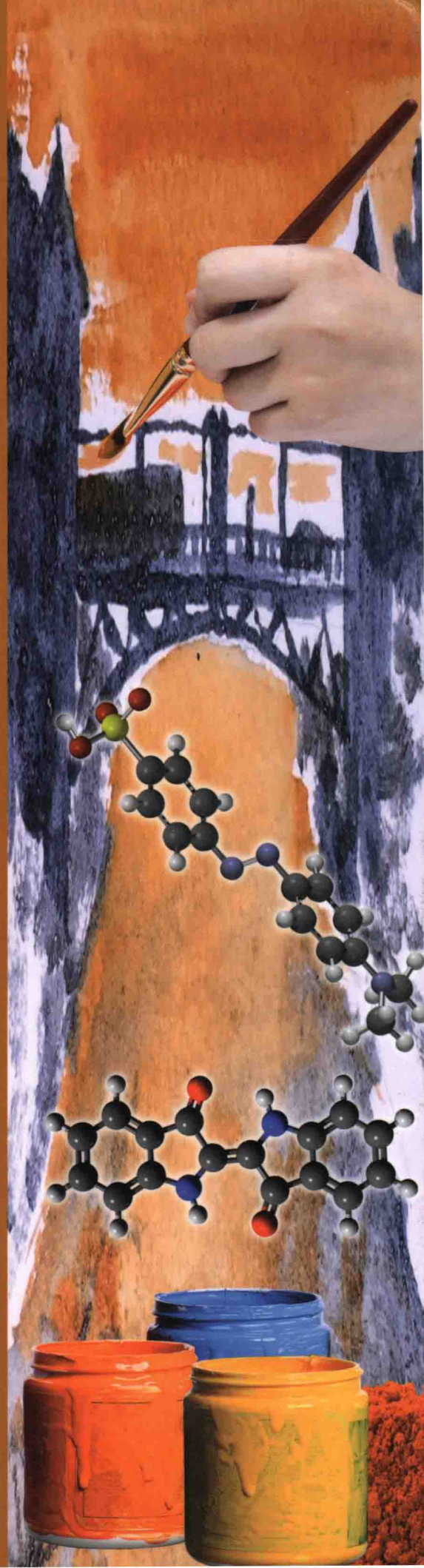
CENGAGE LEARNING LABORATORY SERIES *for*
Organic Chemistry

A Microscale Approach *to* Organic Laboratory Techniques

SIXTH EDITION

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



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A Microscale Approach *to* Organic Laboratory Techniques

SIXTH EDITION

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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