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INTRODUCTION TO

Organic Laboratory Techniques

A Microscale
Approach

Second Edition

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**A MICROSCALE
APPROACH**

SECOND EDITION

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P R E F A C E



We have been pleased by the favorable response that the first edition of our textbook on microscale techniques received. We recognize the features that made the original edition successful, and we have attempted to maintain these strengths in our revisions. Nevertheless, we felt that some improvements were necessary, and these improvements should enhance this second edition.

In preparing the new edition, we focused on two important areas. First, we devoted considerable effort toward improving the safety of the experiments. The introductory chapter, “Laboratory Safety,” has been extensively rewritten to place greater emphasis on the safe use and disposal of hazardous chemicals. Information describing Material Safety Data Sheets and Right-to-Know laws has been added. Second, we have devoted substantial effort to improving the reliability of our experiments. In some cases, we found it advisable to increase the scale of the experiments in order to improve student success. Although we have adjusted the scale of the experiments, we have nevertheless tried to be faithful to the original concept of the microscale laboratory by keeping the overall scale of experiments at a level at which they can still be accomplished in microscale glassware.

Besides modifying virtually all the experiments from the original edition, we have added some new experiments. These include:

Experiment 15 4-Methylcyclohexene

Experiment 18 Markovnikov and Anti-Markovnikov Hydration of 1-Methylcyclohexene

Experiment 37 Reactions of Diazonium Salts

Experiment 51 Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem

Experiment 55 Analysis of a Diet Soft Drink by HPLC

One of the criticisms of microscale organic laboratory courses is that students do not receive adequate training in the use of conventional-scale equipment, nor do they gain experience in such traditional techniques as extractions using separatory funnels or distillations using distillation heads and condensers. We are sensitive to this criticism and have endeavored to include a selection of conventional-scale experiments to give students an opportunity to master these techniques. In this edition, we have included some new conventional-scale experiments, including:

Experiment 56 Preparation of a C-4 or C-5 Acetate Ester

Experiment 60 Isolation of Essential Oils from Allspice, Cloves, Cumin, Caraway, Cinnamon, or Fennel

In addition, we have added a new Techniques chapter, Technique 13, “High-Performance Liquid Chromatography (HPLC).” Each of the new experiments and modifications has been class-tested. We believe that these additions and modifications will greatly enhance the book.

We should also like to point out that an instructor's manual accompanies our textbook and is available from Saunders College Publishing. The Instructor's Manual contains complete instructions for the preparation of reagents and equipment for each experiment, as well as answers to each of the questions. Other comments that should prove helpful to the instructor are also included in the instructor's manual. We strongly recommend that instructors obtain a copy of this manual.

We owe our sincere thanks to the many colleagues who have used our textbooks and who have offered their suggestions for changes and improvements in our laboratory procedures. Although we cannot mention everyone who has made valuable contributions, we must make special mention of Cathy Lyle (Bellevue Community College), Rosemary Fowler (Cottey College), Siegfried Lodwig (Centralia Community College), James Patterson (University of Washington), and John Rodriguez (Ace Glass Company).

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Donald L. Pavia
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EXPERIMENTS WHICH CAN BE PERFORMED WITH MINIMAL EQUIPMENT

A frequently-expressed criticism of microscale methods in the organic chemistry laboratory is that the initial expense of purchasing specialized glassware kits and other equipment is excessive. We believe, however, that this is not necessarily the case and that a large variety of experiments can be accomplished with only minimal equipment.

The experiments listed below can be executed satisfactorily using equipment which includes:

- Erlenmeyer flasks, 20–50 mL
- Beakers, 30 mL
- Filter flasks, 50 mL
- Hirsch funnels
- Screw-capped centrifuge tubes, 15 mL
- Pasteur pipets
- Microscale spatulas
- Watch glasses

Using these pieces of equipment, which are likely to be found in most laboratories, the instructor can select an interesting variety of experiments. Note that very few of these experiments involve the isolation of liquids (see the following list). The isolation of most liquids requires specialized pieces of glassware that are not included in the above list.

Experiment	Title
1	Isolation of the Active Ingredient in an Analgesic Drug
2	Acetylsalicylic Acid
3	Acetaminophen (this must be scaled up to avoid use of a Craig tube)
4	TLC Analysis of Analgesic Drugs
5	Isolation of Caffeine from Tea (without the final sublimation step)
9	Isolation of Chlorophyll and Carotenoid Pigments from Spinach
10	Reactivities of Some Alkyl Halides
26	Coenzyme Synthesis of Benzoin
27	Benzil
28	Benzilic Acid
29	Tetraphenylcyclopentadienone
30	The Aldol Condensation: Preparation of Benzalacetones and Benzalacetophenones (use a Hirsch funnel instead of a Craig tube)

33	1,4-Diphenyl-1,3-Butadiene
34	Relative Reactivities of Several Aromatic Compounds
35	Nitration of Methyl Benzoate
37	Reactions of Diazonium Salts
38	Chromatography of Some Dye Mixtures
41	Preparation of a Detergent
44	Preparation and Properties of Polymers: Polyester, Nylon, and Polystyrene
45	The Diels-Alder Reaction of Cyclopentadiene with Maleic Anhydride (scale up to allow the use of a Hirsch funnel instead of the Craig tube)
46	Benzyne Formation and the Diels-Alder Reaction: Preparation of 1,2,3,4-Tetraphenylnaphthalene
47	Photoreduction of Benzophenone
48	Luminol
53	Identification of Unknowns
54	Carbohydrates
55	Analysis of a Diet Soft Drink by HPLC

NOTE: The experiments cited in this list avoid the preparation of liquid samples. It is an easy matter, however, to add experiments which utilize *conventional-scale* equipment for the preparation of liquid samples. In this way, you can begin performing microscale with very little specialized equipment and then work your way up to a fully-developed microscale laboratory by purchasing individual pieces to augment your microscale equipment over time.

In addition, there are several *conventional-scale* experiments that appear in our book. These experiments utilize glassware that is already present in the conventional organic laboratory.

23	Resolution of (\pm)- α -Phenylethylamine and Determination of Optical Purity
56	Preparation of a C-4 or C-5 Acetate Ester
57	Synthesis of <i>t</i> -Pentyl Chloride
58	Nitration of Methyl Benzoate
59	Hydrolysis of Methyl Salicylate
60	Isolation of Essential Oils from Allspice, Cloves, Cumin, Caraway, Cinnamon, or Fennel

Once an introductory selection of experiments has been established, it is less expensive to add particular pieces to the glassware that is already available, in order to add new experiments to the schedule. This alternative approach is likely to be much less expensive than purchasing a set of complete glassware kits in one large purchase.

TABLE OF CONTENTS



<i>Welcome to Organic Chemistry!</i>	2
<i>Laboratory Safety</i>	5
<i>Advance Preparation and Laboratory Records</i>	22
<i>Laboratory Glassware</i>	30

Part One INTRODUCTION TO MICROSCALE METHODS

Essay	Introduction to Microscale Laboratory	42
Experiment 1	Isolation of the Active Ingredient in an Analgesic Drug	59
Essay	Aspirin	63
Experiment 2	Acetylsalicylic Acid	66
Essay	Analgesics	70
Experiment 3	Acetaminophen	74
Essay	Identification of Drugs	78
Experiment 4	TLC Analysis of Analgesic Drugs	79
Essay	Caffeine	83
Experiment 5	Isolation of Caffeine from Tea	87
Essay	Esters—Flavors and Fragrances	92
Experiment 6	Isopentyl Acetate (Banana Oil)	95
Essay	Terpenes and Phenylpropanoids	99
Experiment 7	Essential Oils from Spices: Oil of Cloves	103
Essay	Stereochemical Theory of Odor	107
Experiment 8	Spearmint and Caraway Oil: (+)- and (–)-Carvones	110
Essay	The Chemistry of Vision	119
Experiment 9	Isolation of Chlorophyll and Carotenoid Pigments from Spinach	124

Part Two PREPARATIONS AND REACTIONS OF ORGANIC COMPOUNDS

Experiment 10	Reactivities of Some Alkyl Halides	134
Experiment 11	Nucleophilic Substitution Reactions: Competing Nucleophiles	137
Experiment 11A	Competitive Nucleophiles with 1-Butanol or 2-Butanol	140

Experiment 11B Competitive Nucleophiles with 2-Methyl-2-Propanol	143
Experiment 11C Analysis	144
Experiment 12 Hydrolysis of Some Alkyl Chlorides	147
Experiment 13 Synthesis of <i>n</i> -Butyl Bromide and <i>t</i> -Pentyl Chloride	153
Experiment 13A <i>n</i> -Butyl Bromide	156
Experiment 13B <i>t</i> -Pentyl Chloride	158
Experiment 14 Elimination Reactions: Dehydration and Dehydrohalogenation	160
Experiment 14A Dehydration of 1-Butanol and 2-Butanol	164
Experiment 14B Dehydrobromination of 1-Bromobutane and 2-Bromobutane	165
Experiment 15 4-Methylcyclohexene	168
Essay Petroleum and Fossil Fuels	173
Experiment 16 Gas Chromatographic Analysis of Gasolines	181
Essay Fats and Oils	186
Experiment 17 Methyl Stearate from Methyl Oleate	191
Experiment 18 Markovnikov and Anti-Markovnikov Hydration of 1-Methylcyclohexene	196
Experiment 18A Hydroboration–Oxidation of 1-Methylcyclohexene	200
Experiment 18B Oxymercuration of 1-Methylcyclohexene	205
Experiment 18C Analysis Procedures	207
Experiment 19 Phase-Transfer Catalysis: Addition of Dichlorocarbene to Cyclohexene	210
Essay Ethanol and Fermentation Chemistry	219
Experiment 20 Ethanol from Sucrose	222
Experiment 21 An Oxidation–Reduction Scheme: Borneol, Camphor, Isoborneol	227
Experiment 22 Chiral Reduction of Ethyl Acetoacetate: Optical Purity Determination Using a Chiral Shift Reagent	238
Experiment 22A Chiral Reduction of Ethyl Acetoacetate	239
Experiment 22B NMR Determination of the Optical Purity of (S)-(+)-Ethyl 3-Hydroxybutanoate	242
Experiment 23 Resolution of (±)- α -Phenylethylamine and Determination of Optical Purity	247
Experiment 23A Resolution of (±)- α -Phenylethylamine	248

Experiment 23B Determination of Optical Purity Using NMR and a Chiral Resolving Agent	252
Essay Detection of Alcohol: The Breathalyzer	254
Experiment 24 Chromic Acid Oxidation of Alcohols	258
Experiment 25 Triphenylmethanol and Benzoic Acid	266
Experiment 25A Triphenylmethanol	272
Experiment 25B Benzoic Acid	274
Essay Thiamine as a Coenzyme	277
Experiment 26 Coenzyme Synthesis of Benzoin	282
Experiment 27 Benzil	286
Experiment 28 Benzilic Acid	289
Experiment 29 Tetraphenylcyclopentadienone	293
Experiment 30 The Aldol Condensation Reaction: Preparation of Benzalacetones and Benzalacetophenones	296
Experiment 31 Enamine Reactions: 2-Acetylcyclohexanone	301
Experiment 32 5,5-Dimethyl-1,3-cyclohexanedione (Dimedone)	312
Experiment 33 1,4-Diphenyl-1,3-butadiene	319
Experiment 34 Relative Reactivities of Several Aromatic Compounds	325
Experiment 35 Nitration of Methyl Benzoate	330
Experiment 36 Friedel-Crafts Acylation	334
Essay Synthetic Dyes	341
Experiment 37 Reactions of Diazonium Salts	346
Experiment 37A Preparation of Methyl Salicylate	348
Experiment 37B Preparation of Methyl Red	350
Essay Food Colors	351
Experiment 38 Chromatography of Some Dye Mixtures	355
Essay Local Anesthetics	360
Experiment 39 Benzocaine	364
Experiment 40 Methyl Salicylate (Oil of Wintergreen)	367
Essay Soaps and Detergents	371
Experiment 41 Preparation of a Detergent	377
Essay Pheromones: Insect Attractants and Repellants	381
Experiment 42 <i>N,N</i> -Diethyl- <i>m</i> -toluamide: The Insect Repellent "OFF"	387
Essay Sulfa Drugs	392
Experiment 43 Sulfa Drugs: Preparation of Sulfanilamide	396
Essay Polymers and Plastics	401
Experiment 44 Preparation and Properties of Polymers: Polyester, Nylon, and Polystyrene	411

Experiment 44A Polyesters	412
Experiment 44B Polyamide (Nylon)	414
Experiment 44C Polystyrene	416
Experiment 44D Infrared Spectra of Polymer Samples	417
Essay Diels–Alder Reaction and Insecticides	419
Experiment 45 The Diels–Alder Reaction of Cyclopentadiene with Maleic Anhydride	425
Experiment 46 Benzyne Formation and the Diels–Alder Reaction: Preparation of 1,2,3,4-Tetraphenylnaphthalene	429
Experiment 47 Photoreduction of Benzophenone	434
Essay Fireflies and Photochemistry	441
Experiment 48 Luminol	445
Essay Chemistry of Milk	450
Experiment 49 Isolation of Casein and Lactose from Milk	456
Experiment 49A Isolation of Casein from Milk	458
Experiment 49B Isolation of Lactose from Milk	459
Experiment 50 Mutarotation of Lactose	461
Experiment 51 Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem	465
Experiment 52 “Pet Molecule” Project	467

Part Three IDENTIFICATION OF ORGANIC SUBSTANCES

Experiment 53 Identification of Unknowns	472
Experiment 53A Solubility Tests	478
Experiment 53B Tests of the Elements (N, S, X)	483
Experiment 53C Tests for Unsaturation	490
Experiment 53D Aldehydes and Ketones	495
Experiment 53E Carboxylic Acids	501
Experiment 53F Phenols	504
Experiment 53G Amines	507
Experiment 53H Alcohols	511
Experiment 53I Esters	516
Experiment 54 Carbohydrates	520
Experiment 55 Analysis of a Diet Soft Drink by HPLC	530

Part Four CONVENTIONAL-SCALE EXPERIMENTS

Essay How to Scale Up a Reaction: Macroscale Methods	534
---	------------

Experiment 56 Preparation of a C-4 or C-5 Acetate Ester	536
Experiment 57 Synthesis of <i>t</i> -Pentyl Chloride	539
Experiment 58 Nitration of Methyl Benzoate	541
Experiment 59 Hydrolysis of Methyl Salicylate	542
Experiment 60 Isolation of Essential Oils from Allspice, Cloves, Cumin, Caraway, Cinnamon, or Fennel	545

Part Five

THE TECHNIQUES

Technique 1	Measurement of Volume and Weight	550
Technique 2	Heating and Cooling Methods	560
Technique 3	Reaction Methods	569
Technique 4	Filtration	587
Technique 5	Crystallization: Purification of Solids	601
Technique 6	Physical Constants: Melting Points, Boiling Points, Density	620
Technique 7	Extractions, Separations, and Drying Agents	641
Technique 8	Simple Distillation	664
Technique 9	Vacuum Distillation, Manometers	677
Technique 10	Fractional Distillation, Azeotropes	693
Technique 11	Steam Distillation	715
Technique 12	Column Chromatography	723
Technique 13	High-Performance Liquid Chromatography (HPLC)	749
Technique 14	Thin-Layer Chromatography	754
Technique 15	Gas Chromatography	769
Technique 16	Sublimation	785
Technique 17	Polarimetry	791
Technique 18	Refractometry	797
Technique 19	Preparation of Samples for Spectroscopy	803
Technique 20	Guide to the Chemical Literature	822

APPENDICES

Appendix 1	Tables of Unknowns and Derivatives	838
Appendix 2	Procedures for Preparing Derivatives	848
Appendix 3	Infrared Spectroscopy	852
Appendix 4	Nuclear Magnetic Resonance Spectroscopy	871
Appendix 5	Carbon-13 Nuclear Magnetic Resonance Spectroscopy	891
Appendix 6	Index of Spectra	900
Index		903

Introduction

WELCOME TO ORGANIC CHEMISTRY!

Organic chemistry can be fun, and we hope to prove it to you. The organic chemistry laboratory, using microscale experiments, is also a pleasant place to work. The laboratory environment is cleaner and safer than has been true with traditional laboratories, and the level of skills that you will develop will be higher. The personal satisfaction that comes with performing a sophisticated experiment skillfully and successfully will be great.

To get the most out of the laboratory course, you should strive to do several things. First, you need to understand the organization of this laboratory manual and how to use the manual effectively. It is your guide to learning. Second, you must try to understand both the purpose and the principles behind each experiment you do. Third, you must try to organize your time effectively *before* each laboratory period.

ORGANIZATION OF THE TEXTBOOK

Consider briefly how this textbook is organized. There are four introductory chapters, of which this Welcome is the first; a chapter on laboratory safety is second; advance preparation and laboratory records make up the third; and laboratory glassware is the fourth. Beyond these introductory chapters, the textbook is divided into five parts. Part One contains nine experiments, which are intended to introduce you to most of the essential techniques of microscale organic chemistry. Part One also contains an introductory essay that outlines the basic methods of microscale laboratory. This essay contains several laboratory exercises designed to provide practical experience in these methods. We recommend that you perform all or most of the experiments in Part One. Part Two consists of 43 experiments, which may be assigned as part of your laboratory course. Your instructor will choose a set of these experiments. Part Three is devoted to the identification of organic compounds, and it contains three experiments that provide experience in the analytical aspects of organic chemistry. Interspersed within these first three parts of the textbook are numerous covering essays that provide background information related to the experiments. Part Four contains five experiments intended to provide experience with important conventional-scale methods. These include conventional-scale filtration, crystallization, and extraction, plus use of a separatory funnel, simple distillation, and fractional distillation. There is also an essay that describes how to scale up a reaction. Part Five is composed of a series of detailed instructions and explanations dealing with the techniques of organic chemistry, with particular reference to microscale methods.

The techniques are extensively developed and used, and you will become familiar with them in the context of the experiments. Within each experiment, you will find a section, “Required Reading,” that indicates which techniques you should study to do that experiment. Extensive cross-referencing to the techniques chapters in Part Five is included in each experiment. Each experiment also contains a section called “Special Instructions,” which lists special safety precautions and specific instructions to you the student. Finally, each experiment contains a section entitled “Waste Disposal,” which provides

instruction on the correct means of disposing of the reagents and materials used during the experiment.

The Appendices to this textbook contain sections dealing with infrared spectroscopy, nuclear magnetic resonance, and ^{13}C nuclear magnetic resonance. Many of the experiments included in Parts One, Two, Three, and Four utilize these spectroscopic techniques, and your instructor may choose to add them to other experiments.

ADVANCE PREPARATION

It is essential to plan carefully for each laboratory period so that you will be able to keep abreast of the material you will learn in your organic chemistry laboratory course. You should not treat these experiments as a novice cook would treat *The Good House-keeping Cookbook*. You should come to the laboratory with a plan for the use of your time and some understanding of what you are about to do. A really good cook does not follow the recipe line-by-line with a finger, nor does a good mechanic fix your car with the instruction manual in one hand and a wrench in the other. In addition, it is unlikely that you will learn much if you try to follow the instructions blindly, without understanding them. We can't emphasize strongly enough that you should come to the lab *prepared*.

If there are items or techniques that you do not understand, you should not hesitate to ask questions. You will learn more, however, if you figure things out on your own. Don't rely on others to do your thinking for you.

You should read the chapter entitled "Advance Preparation and Laboratory Records" right away. Although your instructor will undoubtedly have a preferred format for keeping records, much of the material here will help you learn to think constructively about laboratory experiments in advance. It would also save time if, as soon as possible, you read the first six techniques chapters in Part Five. These techniques are basic to all experiments in this textbook. You should also read the essay, "Introduction to Microscale Laboratory," on pp 42-59. The laboratory class will begin with experiments almost immediately, and a thorough familiarity with this particular material will save you much valuable laboratory time.

It is also very important to read the chapter called "Laboratory Safety." It is your responsibility to know how to perform the experiments safely and how to understand and evaluate the risks that are associated with laboratory experiments. Knowing what to do and what not to do in the laboratory is of paramount importance, since the laboratory has many potential hazards associated with it.

BUDGETING TIME

As mentioned in the "Advance Preparation" section of this chapter, you should read several chapters of this book even before your first laboratory class meeting. You should also read the assigned experiment carefully before every class meeting. Having read the experiment will allow you to schedule your time wisely. Often you will be doing more

than one experiment at a time. Experiments like the fermentation of sugar or the chiral reduction of ethyl acetoacetate require a few minutes of advance preparation *one week* ahead of the actual experiment. At other times you will have to catch up on some unfinished details of a previous experiment. For instance, usually it is not possible to determine a yield accurately or a melting point of a product immediately after you first obtain the product. Products must be free of solvent to give an accurate weight or melting point range; they have to be “dried.” Usually, this drying is done by leaving the product in an open container on your desk or in your locker. Then, when you have a pause in your schedule during the subsequent experiment, you can determine these missing data using a sample that is dry. Through careful planning you can set aside the time required to perform these miscellaneous experimental details.

THE PURPOSE

The main purpose of an organic laboratory course is to teach you the techniques necessary for a person dealing with organic chemicals. You will learn how to handle equipment that is becoming increasingly common in many laboratories. You will also learn the techniques needed for separating and purifying organic compounds. If the appropriate experiments are included in your course, you may also learn how to identify unknown compounds. The experiments themselves are only the vehicle for learning these techniques. The technique chapters in Part Five are the heart of this textbook, and you should learn these techniques thoroughly. Your instructor may provide laboratory lectures and demonstrations explaining the techniques, but the burden is on you to master them by familiarizing yourself with these chapters.

Besides good laboratory technique and the methods of carrying out basic laboratory procedures, other things you will learn from this laboratory course are

1. How to take data carefully.
2. How to record relevant observations.
3. How to use your time effectively.
4. How to assess the efficiency of your experimental method.
5. How to plan for the isolation and purification of the substance you prepare.
6. How to work safely.
7. How to solve problems and think like a chemist.

In choosing experiments, we have tried whenever possible to make them relevant, and, more importantly, interesting. To that end, we have tried to make them a learning experience of a different kind. Most experiments are prefaced by a background essay to place things in context and to provide you with some new information. We hope to show you that organic chemistry pervades your lives (drugs, foods, plastics, perfumes, and so on). Furthermore, you should leave your course well trained in organic laboratory techniques. We are enthusiastic about our subject and hope you will receive it with the same spirit.