

## TABLE OF CONTENTS

### Microscale Approach to Organic Laboratory Techniques (Sixth Edition)

Preface	4
Equipping the Organic Chemistry Laboratory	6
Waste Management Guidelines	12
Laboratory Equipment and Supplies	17
Organic Laboratory Techniques Practical Exams	
Organic lab practical exam advice for instructors	25
Organic lab practical exam instructions for students	26
Organic lab practical exam instructions and report sheet for crystallization	28
Organic lab practical exam instructions and report sheet for extraction	29

### Chemicals and Supplies for Each Experiment Answers to Questions

Experiment 1	Introduction to Microscale Laboratory	30
Experiment 2	Solubility	30
Experiment 3	Crystallization	34
Experiment 4	Extraction	38
Experiment 5	A Separation and Purification Scheme	42
Experiment 6	Chromatography	46
Experiment 7	Infrared Spectroscopy and Boiling-Point Determination	52
Experiment 8	Simple and Fractional Distillation	54
Experiment 9	Acetylsalicylic acid	57
Experiment 10	Isolation of the Active Ingredient in an Analgesic Drug	59
Experiment 11	Acetaminophen	61
Experiment 12	TLC Analysis of Analgesic Drugs	63
Experiment 13	Isolation of Caffeine from Tea or Coffee	68
Experiment 14	Isopentyl Acetate (Banana Oil)	72
Experiment 15	Essential Oils: Extraction of Oil of Cloves	76
Experiment 16	Spearmint and Caraway Oil: (+)- and (-)-Carvones	78
Experiment 17	Isolation of Chlorophyll and Carotenoid Pigments from Spinach	83
Experiment 18	Ethanol from Sucrose	86
Experiment 19	An Introduction to Molecular Modeling	89
Experiment 20	Computational Chemistry	89
Experiment 21	Reactivity of Some Alkyl Halides	90
Experiment 22	Nucleophilic Substitution Reactions: Competing Nucleophiles	94
Experiment 23	Synthesis of <i>n</i> -Butyl Bromide and <i>t</i> -pentyl chloride	99
Experiment 24	4-Methylcyclohexene	103
Experiment 25	Methyl Stearate from Methyl Oleate	106
Experiment 26	Preparation of Soap	110
Experiment 27	Gas Chromatographic Analysis of Gasolines	115
Experiment 28	Biodiesel	118
Experiment 29	Chiral Reduction of Ethyl Acetoacetate; Optical Purity Determination	121
Experiment 30	Nitration of Aromatic Compounds Using a Recyclable Catalyst	124

Experiment 31	Reduction of Ketones using Carrots as Biological Reducing Agent	126
Experiment 32	Resolution of $\alpha$ -Phenylethylamine and Determination of Optical Purity	127
Experiment 33	An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol	133
Experiment 34	Multistep Reaction Sequence: The Conversion of Benzaldehyde to Benzoic Acid	137
Experiment 35	Triphenylmethanol and Benzoic Acid	144
Experiment 36	Aqueous-based Organozinc Reactions	151
Experiment 37	Sonogashira Coupling of Iodosubstituted Aromatic Compounds with Alkynes Using a Palladium Catalyst	154
Experiment 38	Grubbs-Catalyzed Metathesis of Eugenol with 1,4-Butenediol to Prepare a Natural Product	157
Experiment 39	The Aldol Condensation Reaction: Preparation of Benzalacetophenones (Chalcones)	160
Experiment 40	A Green Enantioselective Aldol Condensation Reaction	163
Experiment 41	Preparation of an $\alpha,\beta$ -Unsaturated Ketone via Michael and Aldol Condensation Reactions	164
Experiment 42	Preparation of Triphenylpyridine	166
Experiment 43	Wittig Reaction: Preparation of 1,4-Diphenyl-1,3-Butadiene	167
Experiment 44	Relative Reactivities of Several Aromatic Compounds	170
Experiment 45	Nitration of Methyl Benzoate	173
Experiment 46	Preparation of Methyl Orange	174
Experiment 47	Preparation of Indigo	177
Experiment 48	Formulation of a Paint and Art Project	181
Experiment 49	Benzocaine	183
Experiment 50	<i>N,N</i> -Diethyl- <i>m</i> -Toluamide: The Insect Repellent "OFF"	188
Experiment 51	Sulfa Drugs: Preparation of Sulfanilamide	190
Experiment 52	Preparation and Properties of Polymers: Polyester, Nylon, and Polystyrene	199
Experiment 53	The Diels-Alder Reaction of Cyclopentadiene with Maleic Anhydride	203
Experiment 54	Diels-Alder Reaction with Anthracene-9-methanol	205
Experiment 55	Photoreduction of Benzophenone and Rearrangement of Benzpinacol to Benzopinacolone	206
Experiment 56	Luminol	210
Experiment 57	Identification of Unknowns	211
Experiment 58	Preparation of a C-4 or C-5 Acetate Ester	227
Experiment 59	Competing Nucleophiles in $S_N1$ and $S_N2$ Reactions: Investigations using 2-Pentanol and 3-Pentanol	229
Experiment 60	Friedel-Crafts Acylation	233
Experiment 61	The Analysis of Antihistamine Drugs by Gas Chromatography-Mass Spectrometry	247
Experiment 62	The Use of Organozinc Reagents in Synthesis	248
Experiment 63	Synthesis of Naproxen by Palladium Catalysis	249
Experiment 64	The Aldehyde Enigma	253
Experiment 65	Synthesis of Substituted Chalcones: A Guided-Inquiry Experience	255
Experiment 66	Green Epoxidation of Chalcones	259
Experiment 67	Cyclopropanation of Chalcones	261

Experiment 68	Michael and Aldol Condensation Reactions	263
Experiment 69	Esterification Reactions of Vanillin: The Use of NMR to Solve a Structure Proof Problem	264

### Answers to Problems in the Techniques Section

Technique 1	Laboratory Safety	266
Technique 2	The Laboratory Notebook, Calculations, and Laboratory Records	266
Technique 3	Laboratory Glassware: Care and Cleaning	266
Technique 4	How to Find Data for Compounds: Handbooks and Catalogs	267
Technique 5	Measurement of Volume and Weight	268
Technique 6	Heating and Cooling Methods	269
Technique 7	Reaction Methods	271
Technique 8	Filtration	272
Technique 9	Physical Constants of Solids: The Melting Point	274
Technique 10	Solubility	274
Technique 11	Crystallization: Purification of Solids	275
Technique 12	Extractions, Separations, and Drying Agents	279
Technique 13	Physical Constants of Liquids: The Boiling Point and Density	283
Technique 14	Simple Distillation	284
Technique 15	Fractional Distillation, Azeotropes	285
Technique 16	Vacuum Distillation, Manometers	288
Technique 17	Sublimation	289
Technique 18	Steam Distillation	290
Technique 19	Column Chromatography	291
Technique 20	Thin-Layer Chromatography	293
Technique 21	High-Performance Liquid Chromatography (HPLC)	294
Technique 22	Gas Chromatography	295
Technique 23	Polarimetry	296
Technique 24	Refractometry	298
Technique 25	Infrared Spectroscopy	299
Technique 26	Nuclear Magnetic Resonance Spectroscopy	300
Technique 27	Carbon-13 Nuclear Magnetic Resonance Spectroscopy	301
Technique 28	Mass Spectrometry	301
Technique 29	Guide to the Chemical Literature	301
Correlation of Experiments with Lecture Topics		303

## PREFACE

*Introduction to Organic Laboratory Techniques: A Microscale Approach (Sixth Edition)* continues our dedication to the microscale approach to the teaching of the organic laboratory. In this edition we have devoted considerable effort toward improving the safety of all of the experiments. Technique Chapter 1, “Laboratory Safety,” places strong emphasis on the safe use and disposal of hazardous chemicals. We have included information on Material Safety Data Sheets (MSDS) and Right-to-Know laws. We have continued to update and improve instructions for the handling of waste products that are produced in the experiments. We recommend that virtually all waste, including aqueous solutions, be placed into appropriate waste containers.

This edition of the Microscale book continues the tradition of including stand-alone technique experiments: Solubility, Crystallization, Extraction, Separation and Purification Scheme, Chromatography, Simple and Fractional Distillation, and Infrared Spectroscopy and Boiling Point Determination (Experiments 2-8). These seven experiments emphasize understanding of and proficiency in performing the techniques.

The new experiments are listed in the Preface of the Textbook. These include several new “green” chemistry experiments and some project-based experiments. In the latter experiments, students must either solve a significant problem or they must generate all or part of the experimental procedure. The Green Chemistry essay has been updated and some of the experiments have been modified to make them more “green.” We also offer an alternative way of solving unknowns using mainly spectroscopy.

We have included Chemical Abstract Services (CAS) registry numbers for each of the chemicals. In this way, you should find it easier to locate chemicals when alternative names may be used in catalogs. We hope that this instructor's manual will assist you in preparing solutions, chemical reagents, supplies, and equipment necessary for each experiment that you choose to do. The lists of chemicals and equipment required for each experiment are based on the amount required for ten students. For chemicals, the amounts indicated include some excess. At the end of the manual we have included a section that correlates the experiments with topics presented in standard organic lecture courses.

The time required for each experiment is given in laboratory periods. It is assumed that a laboratory period is about three hours in length. For laboratory

periods that are either shorter or longer, appropriate adjustments must be made.

The technique chapters of the textbook are designed to stand independently from the experiments. You may have a favorite experiment that you like to do in your course. If this is the case, you can freely add your experiment and still take advantage of the technique chapters in the textbook. Since both standard-scale and microscale techniques are described in the technique chapters, you may even add some small-scale experiments and still be able to refer your students to the appropriate sections in these chapters for information on each technique.

A new feature of the Instructor's Manual is the inclusion of some laboratory practical exams that test students on two basic organic laboratory techniques: crystallization and extraction. You may find these exams to be a useful way of evaluating student technique. The idea is to have students perform techniques without the textbook and without looking over another student's shoulder for help!

If you encounter problems with any of the experiments in the Textbook or if you need help in setting up your laboratory, please contact us. We would also like to hear from you if you have any suggestions for improvements in techniques or in any of the experiments.

Donald L. Pavia

Phone: (360)-734-9301  
E-Mail: pavia@comcast.net

Gary M. Lampman

Phone: (360)-733-9054  
E-Mail: lampman@chem.wvu.edu

George S. Kriz

Phone: (360)-650-3126  
E-Mail: George.Kriz@wwu.edu

Department of Chemistry, MS 9150 FAX: (360)-650-2826  
Western Washington University  
Bellingham, Washington 98225

Randall G. Engel E-Mail: Randall.Engel@seattlecolleges.edu  
North Seattle College  
9600 College Way N  
Seattle, WA 98103

## Experiment 1

### INTRODUCTION TO MICROSCALE LABORATORY

TIME ESTIMATE: 1 hour

CHEMICALS AND SUPPLIES PER 10 STUDENTS:

#### Laboratory Exercise 1

Hexane	6 mL
Automatic pipet (100 to 1000 $\mu$ L range) (Option A)	
Dispensing pump, 1-mL size, adjusted to deliver 0.500 mL (Option B)	
Graduated pipets, 1.0 mL (Option C)	10
Pipet pumps (Option C)	10
Waste disposal container for hexane	

#### Laboratory Exercise 2

Pasteur (disposable pipettes)	10
Rubber bulbs	10

**CAS Registry number:**

Hexanes 110-54-3

---

## Experiment 2

### SOLUBILITY

TIME ESTIMATE: Parts A-D (3 hours); Part E (1 hour)

---

**Technique 1**

**LABORATORY SAFETY**

**No problems**

---

**Technique 2**

**THE LABORATORY NOTEBOOK, CALCULATIONS,  
AND LABORATORY RECORDS**

**No problems**

---

**Technique 3**

**LABORATORY GLASSWARE: CARE AND CLEANING**

**No problems**

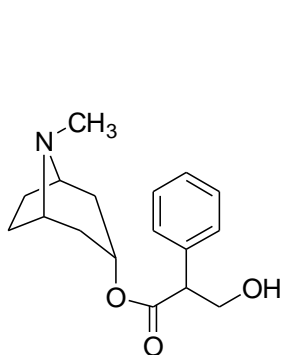
---

## Technique 4

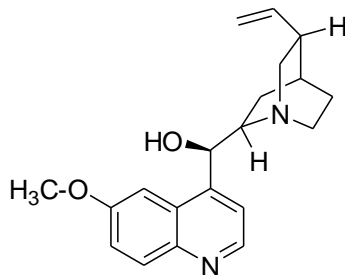
### HOW TO FIND DATA FOR COMPOUNDS: HANDBOOKS AND CATALOGS

#### Answers to Problems

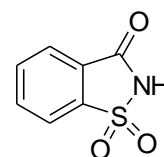
1.



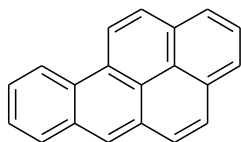
atropine



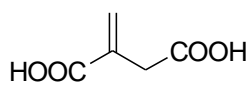
quinine



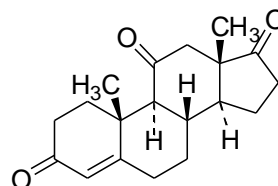
saccharin



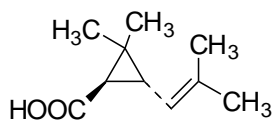
benzo[a]pyrene



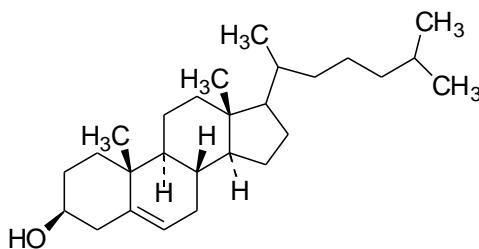
itaconic acid



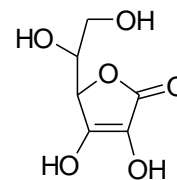
adrenosterone



crysanthemic acid



cholesterol



ascorbic acid



2. Biphenyl: mp 69-72 °C (Aldrich Handbook)  
4-Bromobenzoic acid: mp 252-254 °C (Aldrich Handbook)  
3-Nitrophenol: mp 96-98 °C (Aldrich Handbook)
  3. Octanoic acid: 110-111 °C at 4mm pressure (CRC Handbook)  
Acetophenone, 4-chloro: 273 °C at 760mm and 124-126 at 30mm (CRC)  
2-Heptanol, 2-methyl: 156 °C at 760mm (CRC Handbook)
  4. Octanoic acid: density 0.8615; index of refraction 1.4278  
Acetophenone, 4-chloro: density 1.1922; index of refraction 1.5550  
2-Heptanol, 2-methyl: density 0.8142; index of refraction 1.4238 (CRC)
  5. (*R*)-Camphor: +44.1°  
(*S*)-Camphor: -43°
  6. Poisoning may occur by inhalation, ingestion or skin absorption.  
High concentrations results in depression of the central nervous system.  
Inhalation may cause pulmonary edema.
- 

## Technique 5

### MEASUREMENT OF VOLUME AND WEIGHT

#### Answers to Problems

1. (a) Graduated cylinder  
(b) 1000 µL automatic pipette set at 760 µL. Also can use a dispensing pump.  
(c) Calibrated Pasteur pipette
2. You should preweigh the container and then add 0.76 mL of the liquid using an automatic pipette. The container should then be reweighed. The difference gives the actual weight of the limiting reagent.
3. (a) Diethyl ether,  $d = 0.71 \text{ g/mL}$   
weight =  $(0.71 \text{ g/mL})(0.25 \text{ mL}) = 0.18 \text{ g}$