OPERATIONAL ORGANIC CHEMISTRY: [A PROBLEM-SOLVING APPROACH TO THE LABORATORY COURSE 3RD ED

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Operational Organic Chemistry

A Problem-Solving Approach to the Laboratory Course

THIRD EDITION

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To my wife, Maureen,
our two abys, and the abbess,
Max, Deli, and Hildegard of Bingen

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Preface

This book is based on the first and second editions of *Operational Organic Chemistry: A Laboratory Course*, but every page of the second edition has been revised or rewritten and the focus of the book had been altered, so it is being issued with a new subtitle. Part I contains experiments designed to teach basic laboratory techniques, here called "operations" and identified by numbers preceded by "OP." Part II contains experiments that are correlated to lecture topics covered in most sophomore-level organic chemistry courses. Four experiments were deleted and 12 new ones were added, including a new techniques experiment (12); two spectral analysis experiments (13, 33); analysis of an analgesic drug by TLC (15); a nucleophilic substitution reaction of an ambident nucleophile (20); the synthesis of ethanol by fermentation (27); a multistep synthesis involving a ring contraction (30); a Wittig synthesis (40); a haloform oxidation (42); and two qualitative analysis experiments (39, 50). In addition, the Advanced Projects section (previously Part IV) has been extensively revised and converted to an independent research experiment (58).

The Minilabs have been removed from the Experiments sections (Parts I/II) and put in a section of their own (Part III). Eleven Minilabs were deleted or converted to Experiments, 18 new Minilabs were added, and the rest were extensively revised.

The Qualitative Organic Analysis section (Part IV) was completely reorganized to consolidate the former "Methodology" and "Procedures" sections, and the material relating to IR and NMR spectral analysis was moved to the corresponding Operations (OP-34, OP-35). The Operations (Part V) have been updated to incorporate advances in instrumentation and techniques. A separate HPLC operation (OP-33) has been added, and new or expanded sections on flash chromatography, reversed-phase column chromatography, capillary gas chromatography, Fourier-transform IR and NMR spectrometry, ^{13}C NMR spectrometry, GC-MS, and modern mass spectrometers are included.

Appendix V, Planning an Experiment, now includes directions for writing flow diagrams, and Appendix VII, The Chemical Literature, has been revised and updated to include a more detailed description of literature sources and information about searching the printed and online versions of *Beilstein* and *Chemical Abstracts*. The Bibliography has been updated to include new works and revisions since 1988, most of the pre-1980 material has been deleted, and a new section on software has been added.

Many people were involved in the preparation of this edition and have earned my sincere gratitude for their contributions. I am grateful to David Todd of Pomona College, whose correspondence about the "Evelyn effect" inspired my revision of Experiment 21; William Haag of Lake Superior State University, who reviewed the new HPLC operation; Mike D'Agostino, who tested some of the the new procedures; and my wife Maureen, for her exemplary patience and moral support. I am indebted to Matthew Hart, John Challice, and Lisa Kinne of Prentice Hall, for their assistance and encouragement throughout the project; and Pat McCutcheon, Julie Hollist, and the staff of WestWords, for their skillful pro-

duction of the book. Most of the spectra in this book and the *Instructor's Manual* are reproduced from the spectral libraries of the Aldrich Chemical Company, whose generosity is gratefully acknowledged. I would also like to thank the following reviewers, whose suggestions and comments were invaluable: Philip J. Chenier (University of Wisconsin, Eau Claire), Gary W. Earl (Augustana College), Barbara L. Gaffney (Rutgers University), John C. Gilbert (University of Texas at Austin), Philip D. Hampton (University of New Mexico), Ulrich Hollstein (University of New Mexico), Floyd Kelly (Casper College), Jhong K. Kim (University of California, Irvine), Claire Olander (Appalachian State University), John P. Richard (State University of New York at Buffalo), Kerri Scott, (University of Mississippi), Jason Stenzel (University of Idaho), James M. Takacs (University of Nebraska at Lincoln), and Darrell J. Woodman (University of Washington).

To the Instructor

My thirty-some years of teaching organic chemistry have convinced me that many science students don't really know how science *works*. They attend class, absorb some knowledge, and learn some problem-solving techniques, but all too often they see little relationship between what they have learned in class and what they do in the laboratory. They approach the organic chemistry lab as if its sole purpose were to *make* something and not to *learn* something. That is not necessarily their fault. If we, as lab instructors and lecture professors, fail to convey to students that the essence of science is careful, dispassionate observation coupled with the critical thinking skills needed to discover the meaning of what is observed, it is not surprising that they fail to gain much of value from their laboratory experiences.

I agree with those chemists who have championed the idea that organic lab experiments should be designed not merely to synthesize a product but to solve a scientific problem (see, for example *J. Chem. Educ.* 1991, *68*, 232). This book uses problem-based experiments to help students develop the observational and critical thinking skills that are essential prerequisites for a successful career in science—or in virtually any professional field. The expressed purpose of each major experiment in this book is the solution of a problem through the application of scientific methodology, with the student as the problem solver. Before each experiment the student must first define the problem based on information provided in a hypothetical scenario. After a preliminary reading of the experiment, the student is expected to develop a working hypothesis regarding its outcome. During the experiment, the student gathers and evaluates evidence bearing on the working hypothesis and, as necessary, re-evaluates and revises the hypothesis based on experimental observations and data. Finally, the student tests the hypothesis by obtaining a melting point, a spectrum, a gas chromatogram, or by some other means, and arrives at a conclusion. In reporting the experiment, the student is expected to describe how he or she applied scientific methodology in solving the problem, as illustrated by the sample report in Appendix III.

To stimulate interest and provide a frame of reference for the problems, students are asked to view themselves as "consulting chemists" working for an institute operated by their college or university. Various individuals and

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organizations come to the institute with their scientific problems, and the problems are relayed to the "project group" comprising each lab section, to be solved individually or by collaboration. Although some of the scenarios may appear a bit contrived, most are designed to suggest the kinds of tasks a practicing chemist might be called upon to perform.

To implement this problem-solving approach in the organic chemistry lab, I have rewritten and reorganized every experiment in Parts I and II. Each experiment now includes "Applying Scientific Methodology," a new section intended to help the student understand the problem, formulate a meaningful hypothesis, and solve the problem. Experiment 1 describes in some detail how the student can apply scientific methodology to the solution of a problem, so students should be asked to read the relevant parts of that experiment even if you choose not to assign it. The section "Understanding the Experiment" replaces the old "Methodology" section of each experiment, but has been revised to clarify the purpose of the experiment. Students now follow "Directions" rather than carry out a "Procedure"; while this is a minor change, it is intended to help students see the experiment as describing a course of action rather than providing a recipe to be followed mechanically. The Directions also include hazard warnings, directions for disposal of chemicals, questions to test the student's understanding of the experiment, and occasional reminders to observe and describe a phenomenon. Many of the old collateral projects and library topics have been combined in a new section, "Other Things You Can Do," which may also refer to one or more Minilabs that are related to the experiment.

Because each experiment in this book is formulated as a problem for the student to solve, the outcome of an experiment is not explicitly stated in the experiment. In some experiments such as Experiments 32 and 38, the identity of an organic reactant is not specified. Therefore it is imperative that the instructor or laboratory coordinator obtain a copy of the *Instructor's Manual* (ISBN: 0-13-919267-0), which is provided free of charge by Prentice Hall to adopters of this book.

John W. Lehman

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Introduction

Problem Solving in the Organic Chemistry Laboratory

Organic chemistry is not most people's idea of a "fun" course, but that is no reason for failing to enjoy your organic chemistry lab experience. Many kinds of work can be enjoyable if you have the opportunity to use your imagination and to test your mental and manual skills. During this lab course you will play the role of a consultant in a Consulting Chemists Institute operated by your college or university. When D. K. Little wants to know what happens to his company's food preservative in stomach acid, when Rusty Tappet accidentally pours diesel fuel into a barrel of racing fuel, when Gilda Lilly wants to know the color of a triphenylmethane dye, or when the Olfactory Factory needs a way to convert an oversupply of anisole to a perfume ingredient, you and the other members of your project group will be called upon to solve their problems.

To solve such a problem you must *think* before you *act*. In other words, you will need to read the experiment, understand the problem, and try to predict a likely outcome of the experiment before you actually carry out the experiment in the laboratory. Your prediction, stated clearly in writing, will become your *working hypothesis.* In many cases the most likely outcome will become apparent after you read the experiment, especially if you apply the concepts you have learned in the organic chemistry lecture. In other cases there may be several reasonable outcomes and you will have to make an "educated guess" as to the most likely outcome. During the experiment you will need to gather evidence that may support your hypothesis—or prove it wrong. That means making careful observations and gathering data that relate to the problem. As you evaluate the evidence, you may decide that your original hypothesis was wrong—or at least incomplete—and needs to be revised. By the time you finish the experiment you will have the opportunity to test your original or revised hypothesis and arrive at a conclusion. In this way each experiment will help you apply your observational and critical thinking skills, as well as your lab skills, to the solution of each problem. The next section will tell you, in more detail, how to approach and solve a scientific problem.

Scientific Methodology

Anyone expecting to start a career in some field of science or technology, or a field that is based on scientific knowledge and principles (such as medicine), must learn and apply sound scientific methodology. Although there is no universal "scientific method" that all scientists adhere to rigorously, most of them follow at least some of the following steps when dealing with a scientific problem:

1 Define the problem.
2 Plan a course of action.
3 Gather evidence.
4 Evaluate the evidence.
5 Develop a hypothesis.
6 Test the hypothesis.
7 Reach a conclusion.
8 Report the results.

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Defining the Problem. To a scientist, a "problem" is not so much a perceived difficulty as an *opportunity* for exploring and learning more about some aspect of the physical world. A problem may be inherent in an assigned task, or it may arise from anything that the scientist is curious about, such as an unexplained phenomenon or an unexpected observation. A problem is often defined in the form of a question: What is the identity of the liquid my instructor gave me? What is the mercury concentration in a Lake Superior salmon? How do fireflies generate light? In this laboratory course the problem associated with each experiment will be described in the *Scenario* that leads off the experiment. While performing an experiment, you may be able to formulate additional problems worth investigating.

Planning a Course of Action. A scientist must plan his or her own course of action for solving a scientific problem. This often requires that the scientist carry out a literature search to glean information and data relating to the problem, decide which experimental methods and instruments to use, and develop a detailed procedure to be followed. In most lab courses the procedure is "in the book" and the student simply follows the procedure as if it were a recipe for baking a cake. That is true of a few basic experiments in this textbook, but for most of them you will have to perform some calculations and develop your own experimental plan based on the information and directions given in the experiment. In a few cases you will be required to develop a procedure of your own.

Gathering Evidence. Scientists will gather as much evidence as they feel is needed to solve a problem and convice other scientists that their solution is correct. Evidence is gathered by making careful *observations* and *measurements*. Making accurate measurements, such as measuring the mass or melting point of the product of a chemical synthesis, requires a certain amount of skill and know-how. You can obtain such skills by, for example, watching your instructor demonstrate the operation of an instrument, then practicing on the instrument until you obtain consistent and accurate results *before* you use it to make a measurement you intend to report. If you are not sure how to use an instrument properly, ask the instructor to show you.

To make valid observations you must be *objective*, reporting only what you actually saw and not what you expected to see. A wildlife biologist who expects wild chimpanzees to behave just like chimpanzees at the zoo is not likely to make any important discoveries about chimpanzees! Keep the following in mind when you make observations.

1 Don't confuse an *observation* with an *inference*. An observation is whatever you perceive with your senses (sight, smell, touch, taste, or hearing) during an event. An inference is a guess about the *cause* of the event. Writing "The solution turned brown upon addition of 0.1 M $KMnO_4$" records an observation. Writing "The solution must have contained an alkene because it turned brown upon addition of 0.1 M $KMnO_4$" is an inference.

2 Be prepared to be surprised. If you observe something you didn't expect, don't simply disregard the observation or report what you thought you should have seen. Regard an unexpected observation as an opportunity to learn something you didn't know before—something that might lead to a new discovery.

3 Write down your observations as you make them or shortly afterward. If you wait too long you are likely to leave out important details.

4 Record your observations clearly, completely, and systematically. For example, if you are carrying out the same test on a series of samples, you should record your observations in a table.

Evaluating the Evidence. Evaluating the evidence involves assessing the reliability of your experimental results and looking for clues among your results that may point to a solution for the problem. For example, searching the infrared spectrum of an unknown liquid for evidence of a specific functional group and comparing its boiling point with the boiling points of known compounds may help you solve the problem "What is the identity of the liquid my instructor gave me?" Solving such problems often requires clear and logical thinking; in other cases a more creative, intuitive approach can be valuable. In either case, all of your reasoning and intuition may be fruitless if your experimental results are unreliable. The validation of experimental results requires first asking yourself whether the results make sense physically; in some kinds of experiments (but none in this book) it may also require a statistical analysis of experimental data. If you obtain a melting point that is much lower than the literature value, a product mass that is higher than the theoretical yield for a synthesis, or any other result that seems suspect, then you need to find out whether the result is, in fact, erroneous. You should review everything you did that led to that result, using notes from your lab notebook to jog your memory as necessary. Perhaps you only need to repeat a melting point or dry a product longer, but in any case you should find out what you did wrong and correct it.

Developing and Testing Hypotheses. A hypothesis can be regarded as an "educated guess" made to explain the results of one or more experiments. Hypotheses help us see the significance of an object or event that would otherwise mean little. For example, the movements of the planets seemed erratic and mysterious before Copernicus developed his hypothesis that the earth revolves around the sun. A hypothesis must be *testable* to have validity; that is, it must be formulated in such a way that experiments can be devised whose outcome might prove the hypothesis *wrong*. John Dalton's hypothesis that atoms are indivisible was proved wrong after it was shown that bombarding uranium atoms with neutrons caused them to split into smaller atoms. But Dalton's more fundamental hypothesis that all matter is made of atoms has been tested repeatedly over the years and never proved wrong, so it is generally accepted as true.

Most of the experiments in this book require you to formulate and test a hypothesis based on a problem outlined in the Scenario. A hypothesis can be proposed and revised at any time during the course of an experiment. Many scientists develop a working hypothesis as soon as a problem has been defined, since they often have some idea how the experiment may turn out—or how they hope it will turn out. An appropriate working hypothesis and the method of testing it may become apparent upon reading the experiment. For example, after reading Experiment 9 you should be able to develop a working hypothesis such as "The red pigment in Brand X tomato paste was (or was not) isomerized during processing" and then test your hypothesis by recording the ultraviolet-visible spectrum of the pigment in solution, as directed. A working hypothesis often turns out to be wrong, in which case the scientist must be willing to discard it and formulate a new hypothesis. One drawback of a working hypothesis is that the scientist may become so attached to it that he or she will overlook or ignore evidence that contradicts it. For this reason some scientists prefer to explore a problem without any preconcieved ideas about the outcome—which is not always easy to do. In most of the experiments in this book you can, after reading the experiment, formulate a working hypothesis, but you should be ready to revise or abandon it without regret if the evidence does not support it.

Reaching Conclusions. If a hypothesis passes all the tests you carry out, then you are ready to state a conclusion, such as "The liquid my instructor gave me is benzaldehyde." This does not necessarily mean that your conclusion is correct. You may have not carried out enough tests or carried them out accurately enough to justify your conclusion. But if you have performed an experiment carefully and reasoned logically, you will probably arrive at a valid conclusion.

Reporting Results. You should report all results of an experiment as clearly, completely, and unambiguously as possible. Write up your results in correct English using complete sentences and correct spelling. Be as specific as you can, avoiding such generalities as "My yield was lower than expected due to human error." Label any tables clearly, giving names or standard abbreviations for all physical properties and the units in which they are measured. Construct graphs using accurately ruled graph paper (never notebook paper or paper you have ruled yourself), with the dependent variable plotted on the y axis and the independent variable on the x axis. Label both axes with the physical quantity being graphed and its units, if any. Select appropriate uniform scale intervals so that your data points extend most of the way up and across the graph paper. If the relationship you are graphing is linear, draw the straight line that best fits the data points.

Appendixes II and III suggest ways of recording and reporting experimental results. Your instructor will let you know what kind of report he or she prefers.

Organization of this Book

Operational Organic Chemistry is divided into five parts. Part I contains 12 experiments whose purpose is to help you learn basic laboratory techniques (here called "operations") by applying them to the solution of a problem. For example, you will measure a melting point not just to learn how to measure melting points, but also to establish the identity of a substance you have isolated or synthesized. Once you have mastered the basic operations, you will apply them in the experiments of Part II, which are correlated with topics from your lecture course. These experiments will help you apply what you have learned in the lecture course and increase your proficiency in the laboratory.

Each experiment in Parts I and II contains a list of lecture-course topics and information under most or all of the following headings.

Operations. This heading is followed by a list of the operations to be used in the experiment, each preceded by an operation number such as OP-5. The description for each operation can be located quickly by using the large operation numbers in the right-hand corners of the odd-numbered pages in

Part V. Operations being used for the first time are in boldface type; you should read their descriptions thoroughly before you come to the laboratory. Once you have used an operation, you should not have to reread the entire description the next time you use it, but you should at least read the Summary and review the General Directions (if provided) to refresh your memory. Eventually you should have to refer to the operation descriptions only if you encounter experimental difficulties or are applying an operation in a new situation.

Before You Begin. Under this heading you will find a list of things to do before you come to the laboratory, which always includes reading the experiment and reading or reviewing the operations. Starting with Experiment 5 you will be expected to write an experimental plan for each experiment, as described in Appendix V, and you will usually need to carry out some calculations. Your instructor may require that you have your experimental plan and calculations approved before you begin an experiment.

Scenario. The Scenario presents a hypothetical situation involving the scientific problem you are to solve. A typical Scenario will describe a chemistry-related problem being experienced by a company or individual, and tell what role you will play in its solution.

Applying Scientific Methodology. This section is intended to help you formulate and solve the problem posed in the Scenario by using the methods described under *Scientific Methodology* on page 2.

Background Essay. Each background essay appears under a different descriptive heading. The essay will often show the relation between the lab work and related concepts from the lecture course. It may also relate historical sidelights or interesting facts that show the "real-world" relevance of the experiment.

Understanding the Experiment. This section describes the main purpose of the experiment, explains the theoretical basis of the experiment as necessary, and helps you understand the experimental methodology. It may also provide information that will help you interpret your results or cope with unexpected complications as they arise.

Reactions and Properties. For most experiments, this section gives balanced equations for synthetic reactions and tabulates the relevant physical properties of reactants, products, and other chemicals. The Properties Table contains the data needed for most of the pre-lab calculations.

Directions. This section describes the course of action you will follow to carry out the experiment. You should not follow it mechanically, as you would a recipe, but try to understand the purpose of each operation you are performing. The "Understanding the Experiment" section will help you do that. The directions for most Part I experiments are more detailed than those for Part II. By the time you get to Part II, you will be expected to know how to perform most laboratory operations proficiently without the aid of frequent reminders.

Safety Notes. Characteristics of some hazardous chemicals and precautions for their use are described in the Directions under this heading. See *Laboratory Safety* (p. 9) for general information about laboratory hazards.

Report. This section describes specific calculations, analytical data, or other information that should be included in your laboratory report. Your instructor will describe the format you should follow and indicate any additional material that should be included.

Exercises. Your instructor will assign exercises to be completed and turned in with your laboratory report.

Other Things You Can Do. The "other things" may include additional experiments or Minilabs you can perform, or library research projects you can do. You must have your instructor's permission to start any project marked by an asterisk. You should read the relevant sections of Appendix VII, *The Chemical Literature*, and scan the Bibliography for possible sources before you begin a library research project.

Part III of the text contains 46 short experiments called Minilabs. Minilabs take only part of a lab period and do not require extensive write-ups. They are designed to add flexibility to the lab course and "fill in the gaps" when, for example, a two-week experiment can be finished in one-and-a-half lab periods.

Part IV is a comprehensive, self-contained introduction to qualitative organic analysis that will help you learn how to identify organic compounds by using chemical and spectral methods.

Part V contains descriptions of all the operations, which are referred to in the Directions by number, in the form [OP-5]. Appendixes I through V contain illustrations of laboratory equipment and information about laboratory notebooks, reports, experimental plans, and calculations. Appendix VI contains tables of properties for qualitative analysis. Appendix VII is a guide to the chemical literature that describes some of the most important works in chemistry and, in some cases, tells you how to use them. The Bibliography lists a large number of useful works in chemistry, from enormous multivolume sets such as *Chemical Abstracts* to short papers from the *Journal of Chemical Education*. References to entries listed in the Bibliography are made throughout the text in the form [Bibliography, F20], where the letter refers to a category and the number to a location within that category; for example, F20 is the 20th book listed under category F, Spectrometry.

Getting Along in the Organic Chemistry Laboratory

Because of wide variations in individual working rates, it is usually not possible to schedule experiments so that everyone can be finished in the time allowed. If all labs were geared to the slowest student, the objectives of the course could not be accomplished in the limited time available. If you get behind in the lab, you will probably be required to put in extra hours outside your scheduled laboratory period in order to complete the course. The following suggestions should help you work more efficiently and finish each experiment on time.

1 *Be prepared to start the experiment the moment you reach your work area.* Don't waste the precious minutes at the start of a laboratory period doing calculations, reading the experiment, washing glassware, or carrying

out other activities that should have been performed at the end of the previous period or during the intervening time. The first half hour of any lab period is the most important—if you can use it to collect your reagents, set up the apparatus, and get the initial operation (reflux, distillation, etc.) under way, you should have no trouble completing the experiment on time.

2 *Organize your time efficiently.* Set up a regular schedule that requires you to read the experiment and operation descriptions and complete the prelab assignments a day or two before the laboratory period—an hour before the lab is too late! Plan ahead so that you know approximately what you will be doing at each stage of the experiment. A written experimental plan, prepared as described in Appendix V, is invaluable for this purpose.

3 *Organize your work area.* Before performing any operation, arrange all of the equipment and supplies you will need during the operation neatly on your bench top, in approximately the order in which they will be used. Place small objects such as spatulas and items that might be contaminated by contact with the bench top on a paper towel, laboratory tissue, or mat. After you use each item, move it to an out-of-the-way location (for example, dirty glassware to a washing trough in the sink) where it can be cleaned and then returned to its proper location when time permits. Keep your locker well organized, placing each item in the same location after use so that you can immediately find the equipment you need. This will also help you notice missing items, so they can be found or reported to the instructor without delay.

You will get along much better in the laboratory if you can maintain peace and harmony with your coworkers—or at least keep from aggravating them—and stay on good terms with your instructor. Following these common-sense rules will help you do that.

1 *Leave all chemicals where you found them.* You will understand the reason for this rule once you experience the frustration of hunting high and low for a reagent, only to find it at another student's station in a far corner of the lab. Containers should be taken to the reagent bottles to be filled; reagent bottles should never be taken to your lab station.

2 *Take only what you need.* Unless they are obtained from calibrated dispensers, liquids and solutions should ordinarily be measured into graduated containers so that you will take no more than you expect to use for a given operation. Solids can usually be weighed directly from their containers or measured from a special solids dispenser.

3 *Prevent contamination of reagents.* Don't use pipets or droppers to remove liquids from reagent bottles, or return unused reagent to a stock bottle. Be sure to close all bottles tightly after use—particularly those containing anhydrous chemicals and drying agents.

4 *If you must use a burner, inform your neighbors*—unless they are already using burners. This will allow them to cover any containers of flammable solvents and take other necessary precautions. In some circumstances you may have to use a different heat source, move your operation to a safe location (for instance, under a fume hood), or find something else to do while flammable solvents are in use.

5 *Return all community equipment to the designated locations.* This may include ring stands, steam baths, lab kits, clamps, condenser tubing, and other items that are not in your locker. Since such items will be needed by

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