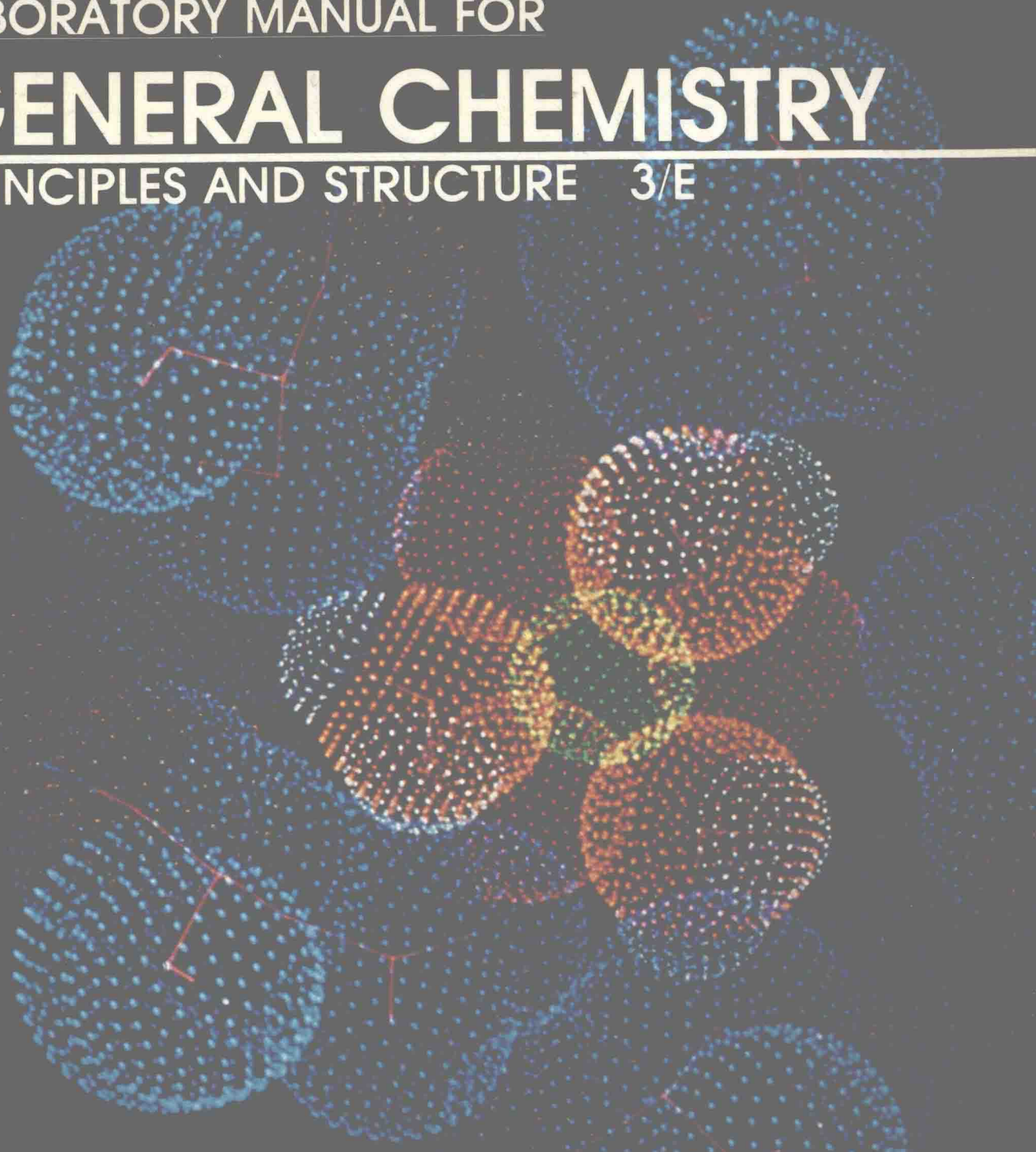
LABORATORY MANUAL FOR

GENERAL CHEMISTRY

PRINCIPLES AND STRUCTURE 3/E



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GENERAL CHEMISTRY

PRINCIPLES AND STRUCTURE 3/E

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Brady/Holum, *FUNDAMENTALS OF CHEMISTRY*, Second Edition, John Wiley & Sons, 1984

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MF 14.2, page 178
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Brady/Humiston, *PRINCIPLES AND STRUCTURE GENERAL CHEMISTRY*, Fourth Edition, John Wiley & Sons, 1986

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Amend, *INTRODUCTORY CHEMISTRY: MODELS AND BASIC CONCEPTS*, John Wiley & Sons, 1977

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Segal, *CHEMISTRY, EXPERIMENT AND THEORY*, John Wiley & Sons, 1985

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OVERVIEW

In our writing of the third edition to this laboratory manual we have incorporated many of the suggestions and comments offered by the users and reviewers of previous editions. While change is inevitable for a new edition, we have attempted to maintain the strengths of past editions while improving upon the quality of other portions of the manual.

The sections on Laboratory Safety and Laboratory Techniques and the Appendices have been retained from previous editions. Reviewers and students have praised the quality of these sections. As a result of the trend toward descriptive chemistry in the general chemistry program, two new experiments have been written and class-tested with an enthusiastic response from students: Experiment 4, Copper Chemistry and Percent Recovery, cycles copper through a number of oxidation states; the metal is recovered and a percent recovery is determined; Experiment 43, A Study of Transition Metal Ions, studies the effect of different ligands on the color and stability of complex ions. The multitude of colors that appear during the three-hour laboratory period is truly fascinating to the students.

Many experiments assigned at the early part of a one-year course can oftentimes only be qualitative in nature. Because of a thirst for students wanting to record and analyze data, several additional quantitative experiments (16, 17, and 18) appear early, without a loss of any appreciation for the chemical principles or analysis. This convenience allows more flexibility in designing a more interesting and varied laboratory program for students.

Several experiments have been expanded: the spectrophotometric determination of K_{sp} (Experiment 33) now allows some flexibility in assigning a particular CrO_4^{2-} salt; the aspirin synthesis includes a titrimetric analysis (Experiment 32); and the molecular weight determination of a gas (Dumas method) includes a consideration of the van der Waal equation (Experiment 19).

A concerted effort was made to improve the clarity of instruction and presentation of information. This was accomplished by using additional figures and tables, more detailed line drawings, footnotes, more explicit equations, and less verbiage.

The Teacher's Manual to this manual continues to be most explicit in presenting the details of each experiment. Sections of each experiment include an Overview, Instructor's Lecture Outline, Teaching Hints, Chemicals Required (the amounts and instructions for their preparation), Special Equipment, Suggested Unknowns, answers to the Prelaboratory and Post-Laboratory Assignments, and a Laboratory Quiz. Users of the laboratory manual have made mention of this addition to the overall laboratory package.

PREFACE

The chemistry laboratory is one of the most interesting and exciting learning centers on campus. In a well-organized, meaningful, hands-on laboratory experience, students have an opportunity to support and develop chemical theories using their own observations. collection of data, and interpretation of results. In developing the logical thought processes that are required for understanding the complex experimental-theoretical relationships in chemistry, students must not only acquire the problem-solving and interpretive skills in lecture, but also the manual techniques and safety awareness in the laboratory. The first hand experience and visual aids of the laboratory that complement lecture theory provide students with a long-lasting appreciation of chemistry—something they will *not* soon forget.

The purpose of writing this laboratory manual was two-fold: to provide students with a hands-on learning experience for relating lecture theory to laboratory practice (and the inherent difficulties that result) and to develop good, safe, manual laboratory skills. The collection of reproducible, reliable data is more likely when meticulous, recurring laboratory techniques are practiced.

This manual covers two semesters (or three quarters) of general chemistry. A student may expect to spend three hours per experiment in the laboratory; limited, advanced preparation and/or extensive calculations may lengthen this time. Although the manual parallels Brady and Humiston's, *General Chemistry, Principles and Structure, 4th Edition*, the experiments are chosen and written so that they may accompany any general chemistry text.

To enhance the development of laboratory technique, the measurement of some parameter of an unknown sample is included in experimental procedures where appropriate; several trials are generally recommended. Occasionally students are required to present data graphically and to express the precision of their results using average and standard deviations. Some unknowns are commercial products such as aluminum cans, oven cleaners, muriatic acid, vinegar, antacids, bleach, alum, and aspirin.

Simple laboratory glassware and equipment, shown at the front of the manual, are necessary for completing most experiments. Where appropriate, the apparatus or technique is shown with a line drawing or photograph. In addition to analytical balances, a slide projector (Experiment 10), spectrophotometers

(Experiments 27 and 33), and pH meters (Experiment 31) are suggested; however, if this instrumentation is unavailable, these experiments can be omitted without penalizing students. A special note on Experiment 10: adopters of the manual can obtain the necessary set of slides from Wiley.

The manual has four major sections:

Laboratory Safety. Information on self-protection, general laboratory rules, and handling chemicals and glassware are listed.

Laboratory Techniques. Nineteen techniques are thoroughly described and illustrated with photographs. Each experiment refers to this section for details in properly performing the experiment.

Experiments. Forty-seven experiments are sub-divided into twelve principles—they are:

A. **Introduction.** Four experiments introduce students to the basic tools for scientific measurements and techniques for identifying and separating substances in mixtures.

B. **Mole Concept.** Five experiments stress chemical formulas, stoichiometry, and periodic relationships.

C. **Atomic and Molecular Structure.** Three experiments provide insight into the structure of individual atoms and molecules and the nomenclature of inorganic compounds.

D. **Chemical Reactions and Analysis.** Five experiments review the reactions and preparations of salts, metals, acids and bases, oxidizing and reducing agents, and the analyses of a number of corresponding compounds.

E. **Gases.** Three experiments use various gas law relationships in investigating gaseous systems.

F. **Solutions.** Two experiments: one investigates a solution's colligative properties, the other looks at heat exchange processes.

G. **Kinetics.** Two experiments give a qualitative and quantitative viewpoint of reaction rates.

H. **Chemical Equilibria and Analysis.** Nine experiments provide a conceptual study of equilibrium as well as a quantitative measure of equilibrium constants. The acid-base ionic equilibria systems include pH, hydrolysis, buffer relationships, and several volumetric analysis procedures.

I. **Electrochemistry.** Two experiments cover the concepts of galvanic and electrolytic cells.

J. **Qualitative Analysis.** A preface and six experiments outline the analysis of eight common anions and sixteen common cations.

K. **Transition Metal Chemistry.** Three experiments cover the formation and stability of transition metal complex ions and the synthesis of four compounds: an alum and three nickel(II) coordination compounds.

L. **Organic Chemistry.** Two experiments present a qualitative overview of the classes of organic compounds by testing for functional groups.

Appendices. Nine appendices include glassworking, the treatment of data, graphing data, test reactions for common substances, vapor pressures of water, concentrations of acids and bases, water solubility of common salts, conversion factors, and common names of chemicals. Reference to these appendices are made in the experiments where appropriate.

Each experiment has six sections.

Objectives. One or more statements establish the purposes and goals of the experiment.

Introduction. Several paragraphs present an overview of the chemical principles studied in the experiment and the methodology for using and interpreting them.

Techniques. Reference is made to the Technique section and Appendices as guides for improving the quality of gathering data and presenting results.

Experimental Procedure. Detailed, stepwise directions are presented for collecting data. Careful attention to safety is stressed in the experimental procedure. While all potentially hazardous chemicals cannot be eliminated, those that do present a danger are flagged with a CAUTION, followed by a brief statement of the danger.

Prelaboratory Assignment. Students are required to answer questions about the experiment prior to entering the laboratory. The questions are easily answered after studying the introduction and experimental procedure; sample calculations for the day's experiment are often included.

Report Sheet. The Report Sheet guides the student through the observations and the collection of data. Steps for completing the calculations are also presented. A thorough understanding of the experiment is necessary to complete the in-depth Post-Laboratory Assignment.

The Teacher's Manual (available to adopters from Wiley) presents, for *each* experiment, a general overview, an instructor's lecture outline, teaching hints that an instructor uses for one-on-one discussions (including CAUTIONS), representative or expected data and results, answers to the Post-Laboratory and Prelaboratory Assignments, a Laboratory Quiz, chemicals and special equipment lists, and procedures for preparing all solutions.

The valuable suggestions provided by the following reviewers of the first edition were greatly appreciated:

Sara Steck Melford, *DePaul University*
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Finally we wish to acknowledge the support of our wives, Judi and June, for their encouragement and understanding for the duration of the project and our children, Kyle and Greg, Mark and Karen, who were willing to give up occasional family outings.

The authors invite corrections and suggestions from colleagues and students.

Jo A. Beran
James E. Brady

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CONTENTS

EXPERIMENTS

A. INTRODUCTION

B. MOLE CONCEPT

C. ATOMIC AND MOLECULAR STRUCTURE

D. CHEMICAL REACTIONS AND ANALYSIS

Laboratory Safety

Be sure that you and your partners practice laboratory safety and follow basic laboratory rules. It is your responsibility, not the instructor's, to play it safe. On the inside front cover of this book, there is space to list telephone numbers, the location of important safety equipment, and reference information useful in the laboratory.

This section has guidelines for making laboratory work a safe and meaningful venture. Study each carefully before answering the questions on the Report Sheet of Experiment 1.

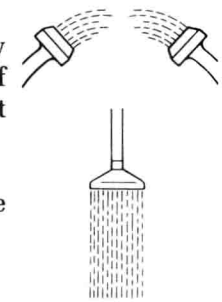
A. SELF-PROTECTION

Always Wear Safety Glasses

1. Safety glasses, goggles, or eye shields must be worn at all times to guard against the laboratory accidents of others as well as your own. Contact lenses should not be worn; if they are, additional eye protection is still necessary.
2. Laboratory aprons or coats (with snap fasteners only) should be worn to protect clothing.
3. Sandals or canvas shoes are not permitted. Wear only shoes that shed liquids.
4. Wear old, non-synthetic clothing which is not torn or frayed. Shirts and blouses should not be frilled or flaired and the sleeves should be close-fit. Synthetic clothes tend to melt to the skin.
5. When working in the laboratory, secure long hair, and remove neckties and scarves.
6. Always wash your face, hands, and arms before leaving the laboratory. Toxic chemicals may be transferred to the mouth.

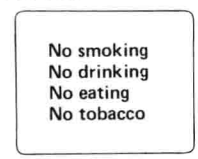
Wash hands before leaving the laboratory

7. Whenever your skin (hands, arms, face, etc.) comes into contact with chemicals, wash it quickly and thoroughly with soap and water. Use the eye-wash fountain to flush chemicals from the eyes and face. *Get help immediately*. Do not rub the affected area, especially the face or eyes, with your hands before washing.
8. Chemicals spilled over a large part of the body require immediate action. Using the safety shower, flood the affected area for at least 5 minutes. Remove all contaminated clothing if necessary. Use a mild detergent and water only (no salves, creams, lotions, etc.). Get medical attention.
9. In case of accident or injury, even if it is minor, notify your instructor at once.
10. Discharge a fire extinguisher at the base of the flames and move it from one side to the other. Small flames can be smothered with a watchglass (towels catch on fire).

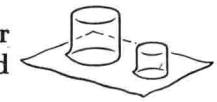


B. LABORATORY RULES

1. Maintain a wholesome, business-like attitude. Horseplay or other careless acts are prohibited. Do not entertain guests in the laboratory.
2. Unauthorized experiments, including variations of those in the laboratory manual, are forbidden.
3. Maintain an orderly, clean laboratory desk and drawer. Immediately clean up all chemical spills, paper scraps, and glassware. Keep drawers or cabinets closed while working and the aisles free of any obstruction. Do *not* place book bags, athletic equipment, etc. on the floor near any lab bench.
4. *No smoking*, *drinking*, *eating*, or *chewing* is permitted at any time because chemicals may possibly enter the mouth or lungs. Your hands may be contaminated with a toxic chemical.
5. Be aware of your neighbors' activities; you may be a victim of their mistakes. Advise them of improper techniques or unsafe practices. If necessary, tell the instructor.
6. Do not work alone. The laboratory instructor must be present.
7. Do not waste time. Prepare for each experiment by completing the Prelaboratory Assignment and by studying the Objectives, Introduction, Techniques, and Experimental Procedure before lab. Always try to understand what you are doing and to *think*, whistle if you like, while working.
8. Believe in your data. A scientist's most priceless possession is integrity. Therefore, be a scientist. An incorrect answer resulting from honest work is infinitely better than a correct one dishonorably obtained.
9. Note beforehand the need for any extra equipment and get it all at the same time from the stockroom.
10. Prepare with care your Report Sheet on each experiment. Record the data *in ink* as you perform the experiment. Data on scraps of paper will be confiscated. Where calculations using data are involved, be orderly for the first set of data. Do not clutter the calculation section with arithmetic details. As you perform the experiment, think through and answer important post-laboratory questions, those intended to give an understanding of the principles on which the procedure is based.
11. Scientists learn much by discussion with one another. Likewise, you may profit by discussion with your laboratory instructor or classmates, not by copying from them. You will also profit by frequent reference to the text. Books are generally more reliable and more complete sources of information than are classmates.
12. For tabular data on the properties of substances, consult the *Handbook of Chemistry and Physics*, Chemical Rubber Publishing Co., Cleveland, Ohio.
13. At the end of the laboratory period, completely clear the lab bench of equipment, clean it with a damp paper towel (and discard), and clean the sinks of all debris. Also clean all glassware used in the experiment.
14. Discard all chemicals as directed by your laboratory instructor. The sink is not the disposal for everything!



C. HANDLING CHEMICALS AND GLASSWARE



1. Clean all glassware with soap or detergent and warm *tap* water. Rinse first with tap water and then once or twice with small amounts of distilled (or de-ionized) water. Distilled water should never be used for washing glassware; it is too expensive.
2. Invert clean glassware on a paper towel to dry; do not wipe or air-blow dry because of possible contamination. Do not dry heavy glassware—graduated cylinders, volumetric flasks, or bottles—over a direct flame.
3. Avoid direct contact with all chemicals. Hands contaminated with potentially harmful chemicals may cause severe eye or skin irritations. Avoid breathing chemical vapors. Use the fume hood as instructed.
4. Read the label on a reagent bottle *twice* before removing any chemicals. The wrong chemical can lead to accidents or "unexplainable" results in your experiments. See Techniques 2, 3, and 4 for instructions in properly transferring chemicals.
5. Avoid using excessive amounts of reagents. *Never* use more than the experiment calls for. *Do not return excess chemicals to the reagent bottle*!
6. Do not insert your own pipet, medicine dropper, or spatula into reagent bottles. Transfer chemicals as shown in Techniques 2, 3, and 4.
7. Discard waste or excess chemicals as follows:

   - Sink: non-flammable, non-toxic, water-soluble liquids followed by large amounts of water.
   - Waste jars (properly labeled): water insoluble liquids, solids, and toxic wastes.
   - Waste basket: paper products only, such as litmus paper, filter paper, and matches.
   - Covered containers (properly labeled): volatile liquids or very reactive chemicals.

   If there is a question about a chemical's disposal, ask your instructor.
8. *Never* taste, smell, or touch a chemical or solution unless specifically directed to do so. Poisonous substances are not always labeled.
9. Always add a reagent slowly; never dump it in. While stirring, slowly pour concentrated solutions into water or less concentrated solutions. This is especially true when diluting concentrated (conc) sulfuric acid.
10. Treat chemical spills as follows:

    - Alert your neighbors and the laboratory instructor.
    - Clean up the spill as directed by the laboratory instructor.
    - If the substance is volatile, flammable, or toxic, warn everyone to shut down the laboratory and evacuate.

Never return excess chemicals to reagent bottle

Always Add The Concentrated To The Diluted Solution Or Water
C → D Or W