STANDARD AND MICROSCALE EXPERIMENTS IN

General Chemistry

FOURTH EDITION

BISHOP • BISHOP • WHITTEN

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Cover Credit: A ribon of magnesium metal reacts with aqueous HCl to give H_2 gas and aqueous $MgCl_2$ (Photograph by Charles D. Winters)

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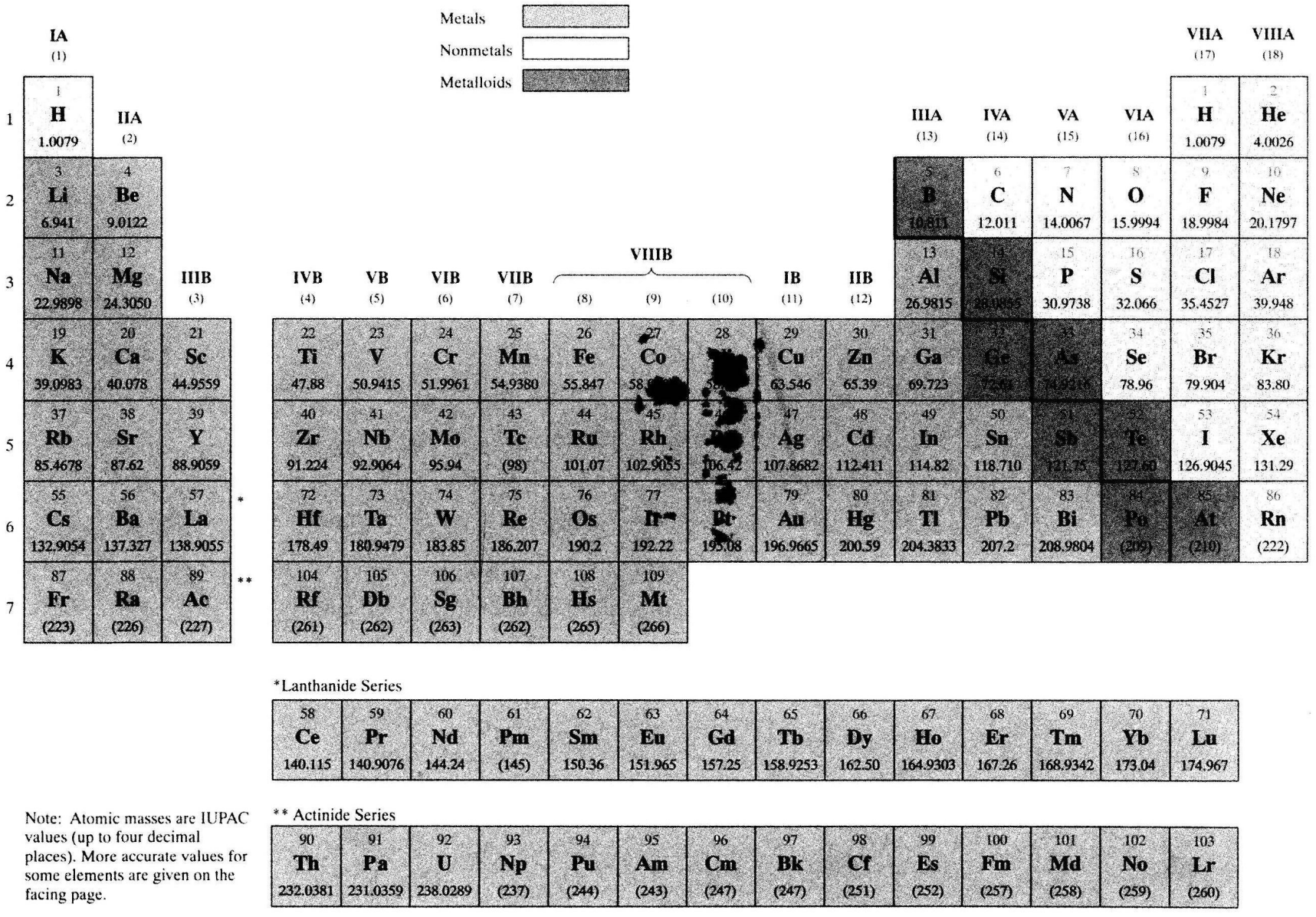
PERIODIC TABLE OF THE ELEMENTS



Note: Atomic masses are IUPAC values (up to four decimal places). More accurate values for some elements are given on the facing page.

PREFACE

Chemistry is an experimental science. What we know about chemical systems comes from experimental observations made by thousands of scientists. Theories provides a framework for understanding and explaining experimental observations and a basis for designing new experiments.

We have attempted to provide an opportunity for students to learn to make measurements and observations, and to interpret the data from these measurements and observations. Some of the earlier experiments in this manual provide an introduction to basic laboratory techniques used in all chemistry laboratories. These techniques will also be used in later experiments. The equipment which professional chemists use may be more sophisticated, but the principles in simple equipment used in introductory laboratories are the same.

Written descriptions are illustrated by art work that is large and clearly drawn so that students can "see for themselves."

A. **References** list all materials which are an *essential* part of the experiment:
   1. Appropriate chapters, sections, and topics in the textbook
   2. Specific Laboratory Techniques and Appendices needed for each experiment
   3. Safety material to review before laboratory
   4. Materials in **BACKGROUND** that precede each group of experiments helps students associate theory with the experiment, and helps laboratory instructors prepare for preliminary lectures

Students can find text references in other general chemistry books with little effort by referring to the topic names. However, the Laboratory manual materials must be included in preparation for the experiments.

B. Groups of experiments exploring common theories and principles have a rather detailed review of the relevant theories and principles given in **BACKGROUND material** before each group of experiments.A brief **Introduction** to each experiment goes more into detail for theories and principles dealing with that specific experiment.

C. **Procedures** in each experiment describe in detail the experimental steps so that experience is gained without waste of time and chemicals. The student can set up the necessary apparatus correctly and quickly. Students who have limited experience in the laboratory can still perform the experiment and will be guided in collecting data. Yet students who will take advanced laboratories will have established good lab practices. Students are warned of hazards or dangerous materials by **CAUTIONS**. Safety precautions are emphasized at appropriate points throughout the manual. We find it useful to require the student to perform the safety exercise (pp. 29–31) during the check-in laboratory, and to take the safety quiz (p. 33). Frequent reference to these safety measures directs attention to the appropriate hazards in a given laboratory and the safety measures required. Special NOTES are included to remind the student of why certain things should be done.

D. Each experiment has a set of *Prelab Questions* designed encourage the student to develop an understanding of the principles and procedures involved in the experiment

before laboratory. The answers to the *Prelab Questions* are found for the most part in the **BACKGROUND** and **Introduction**. The laboratory instructor may choose to use these *Prelab Questions* as 'admission tickets' to the laboratory to encourage this study. A set of *Post Lab Questions* follows the Report Form. These questions check for understanding of factors that affect the experimental results. These are designed to be answered during the experiment and can be removed for grading.

E. We have provided **Report Forms** with space for observations and interpretations. Since most experiments also have an "unknown," we have provided a space for the unknown number close to the result to facilitate grading.

E. For the experiments in qualitative analysis, the *laboratory directions* and a brief description are included in this manual so that students do not have to bring large (expensive) textbooks into the laboratory. The steps are those found in the qualitative analysis chapters in *GENERAL CHEMISTRY WITH QUALITATIVE ANALYSIS*, 6th edition, by Whitten, K. W., Davis, R. E., and Peck, L., published by Saunders College Publishing (2000). A minimum amount of discussion is included in each experiment. However, the student must prepare for the laboratory by reading the chapters in the text in order to fully grasp the chemistry involved in each step.

We have included a reasonable number of quantitative and descriptive experiments so that each laboratory director has considerable flexibility in the choice of experiments . A list of experiments grouped by common concepts and theories is shown below.

Experiments	**Classifications**
1	Review of simple mathematics
2,3, 4	Basic laboratory techniques (simple measurements and observations)
5, 6, **7**	Solutions, solubility and separation tech niques
8, **9**, **10**, **11**, 2, 13	Chemical reactions in aqueous solutions and synthesis
14, **15**, **16**, 17	Periodic table and periodicity
18, **19**, 20, 21	Mole, molecular weight and gas laws
22	Colligative Properties
23, 24, 25	Acid-base chemistry (titrations)
26, **27**	Oxidation-reduction reactions
28, 29	Energy
30, 31	Kinetics
32, 33, 34, 35, 36	Equilibrium
37, 38, **39**, 40	Descriptive chemistry
41, 42, **43**, 44	Introduction to organic chemistry
45, 46, 47, 48, 49	Introduction to Qualitative Analysis

Some **microscale experiments (boldfaced)** that require small amounts of chemicals and small-size equipment are listed below:

5, 7, 8, 9, 10, 11, 15, 16, 23, 26, 27, 30, 31, 32, 39, 41 and the qualitative experiments (45–49).

The manual has a sufficient number of experiments so that the laboratory director can vary the experiments from one year to the next. On the other hand, a suggested menu of experiments for predominantly non-chemistry, science majors is as follows:

First semester: Exps. 1, 2, 3, 5, 6, 7, 8, 9, 11, 15, 16, 19

Second semester: Exps. 23, 26, 27, 28,30, 32, 37, 38, 41, 42, 43, 44

TO THE LABORATORY DIRECTOR

A training program on safety in the chemistry laboratory is an absolutely essential part of preparing laboratory instructors for supervision in undergraduate chemistry laboratories. Laboratory safety training materials are available from the American Chemical Society. We suggest that materials such as "Starting with Safety", the 35 minute videtape developed by Lois Wickstrom, be used the first laboratory period. A formal section on safety must be presented at each orientation meeting of the lab director with laboratory instructors, and at the pre-lab meeting of laboratory instructors with students, in order to promote an awareness of lab safety.

This manual has used updated references, but the laboratory director is responsible for updating safety standards for the conditions in their own laboratories. It is the responsibility of the laboratory instructor to point out hazards to students. Laboratory instructors and students should have access in the laboratory to reference documents on safety such as the following:

(1) Safety in Academic Chemistry Laboratories, Department of Educational Activities, ACS, 1155 16th St. N.W., Washington, D.C. 20036.

(2) NIOSH Publications No. 78-104A, 104B and 77-205 on Toxic Effects of Substances and Carcinogens. Superintendent of Documents U. S. Government Printing Office, Washington D. C. 20402.

(3) Material Safety Data Sheets (MSDS) for hazardous chemicals should be available from the vendor and should be posted in the laboratory.

(4) Safe Handling and Disposal of Chemicals, J. T. Baker Inc., 222 Red School Lane, Phillipsburg, NJ 08865

The *Instructor's Manual* provides detailed lists of chemicals, equipment, the amount of time required for each experiment and suggested answers to the pre- and post- lab questions. Copies are available to all adopters of this laboratory manual.

About 40 percent of the experiments in this manual are micro-scale. Experiments in which learning the use of standard, "real", quantitative equipment or techniques is a major objective, remain standard. However, chemicals that create a serious disposal problem have been decreased in quantity. If the chemicals are non-hazardous, can be dumped down the sink or are relatively cheap, then standard amounts have been suggested.

We have included a reasonable number of quantitative and descriptive experiments so that each laboratory director has considerable flexibility in the choice of experiments . A list of experiments grouped by common concepts and theories is shown below.

Experiments	**Classifications**
1	Review of simple mathematics
2,3, 4	Basic laboratory techniques (simple measurements and observations)
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First semester: Exps. 1, 2, 3, 5, 6, 7, 8, 9, 11, 15, 16, 19

Second semester: Exps. 23, 26, 27, 28,30, 32, 37, 38, 41, 42, 43, 44

TO THE STUDENT

The best way to acquire a deep, clear understanding of the nature of chemistry is in "hands-on", laboratory experiments with the real chemicals and real equipment which chemists use. The experiments in this manual emphasize the good, solid laboratory techniques of planning, carrying out, and reporting the results of actual investigations of chemical systems without wasting your time and chemicals . In higher level courses you will be able to use what you have learned in designing your own experiments. In many of the experiments you will use the same types of equipment that the professional chemist uses. In others you will use microscale equipment and microscale quantities for safety and to decrease wastes. In either case you will do real chemistry. In some of the beginning experiments you will learn to collect data from measurements, to observe changes, and to interpret your data and observations. The laboratory exercises provide an opportunity for you to participate in chemistry and draw reasoned conclusions based on your work.

BEFORE THE LABORATORY

The manual emphasizes safe handling of chemicals and the use of laboratory techniques which provide a safe laboratory. Study the section on *Safety and Laboratory Rules* before each laboratory period so that you will know the appropriate safety measures for the experiment. Safety rules are designed to provide a safe environment in which all may work. Many experiments contain comments about safety. You are required to perform the safety exercise (pp.29-31) and take the safety quiz (pp. 33-34). Your laboratory instructor will have access to Materials Safety Data Sheets provided by the vendors for those chemicals that have hazards associated with them. These should be posted in the lab and appropriate waste containers should be provided for those chemicals which are not to be placed in waste baskets or poured down the sink.

You should carefully study the assignment for the day before you come to the laboratory. At the beginning of each experiment there are references to the appropriate sections in the textbook and to the laboratory manual (including safety), followed by a brief discussion of the important principles of the experiment. These discussions contain information that will be helpful as you perform the experiments.

Prelab questions have been provided as a part of the experiments to assist you in preparing for each laboratory assignment. After you have studied the experiment *and the appropriate sections in your text*, answer the indicated prelab questions and hand them in as you enter the laboratory. Your laboratory instructor may use the prelab questions as an "admission ticket" indicating that you are prepared for the laboratory. Arrange your work neatly so that your instructor can read it easily.

DURING THE LABORATORY

No food or drink is allowed in the laboratory. You *must* wear safety eye protection authorized by your college and/or by the state. You *must* wear shoes. No unauthorized experiments or" horseplay" are permitted. Students should work in the laboratory only at those times authorized by your Instructor. Be alert—read instructions carefully and then follow

them. It is recommended that you place a "check" by each step of the **PROCEDURE** to indicate that you have performed the actions in the instructions. If you omit parts of the procedure, you may fail to observe the planned experiment. Proper planning will prevent poor results and performance.

Careful attention should be paid to the proper use of significant figures and units in all calculations. Set up each problem in an orderly way so that your instructor will have evidence that you understand the calculations. Credit is not usually given for simply "writing down" answers to calculations without supporting evidence of how you arrived at your answer.

Post Lab questions follow the Report Form. These questions require that you think about the experiment rather than "cookbook" the experiment. Hand these in along with the Report Form. At the end of the laboratory period you should hand in the report forms on which you have recorded your data and observations, as well as any calculations and conclusions based on these data and observations. The REPORT FORM and *Post Lab* Questions should be turned in *before* you leave the laboratory.

Clean your equipment and work area before leaving the laboratory. Return all special equipment to your Instructor.

You should have access to the following safety materials:

(1) Safety in Academic Chemistry Laboratories, Department of Educational Activities, ACS, 1155 16th St. N.W., Washington, D.C. 20036.

(2) NIOSH Publications No. 78-104A, 104B and 77-205 on Toxic Effects of Substances and Carcinogens. Superintendent of Documents U. S. Government Printing Office, Washington D. C. 20402.

(3) Material Safety Data Sheets (MSDS)

(4) Safe Handling and Disposal of Chemicals, J. T. Baker Inc., 222 Red School Lane, Phillipsburg, NJ 08865

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LABORATORY TECHNIQUES

A. Handling Chemicals

1. SOLIDS

Solid chemicals are usually stored in wide-mouth bottles. **Always check the label carefully before removing any chemical.** Remove the lid or stopper, place the lid or stopper so that it will not be contaminated, tilt the bottle and roll it gently back and forth until the desired amount of the solid falls into the bottle lid. Do not waste material. If a solid reagent is compacted or "caked" ask your instructor for assistance. **Chemicals should never be returned to the bottles** from which they were removed—discard any excess.

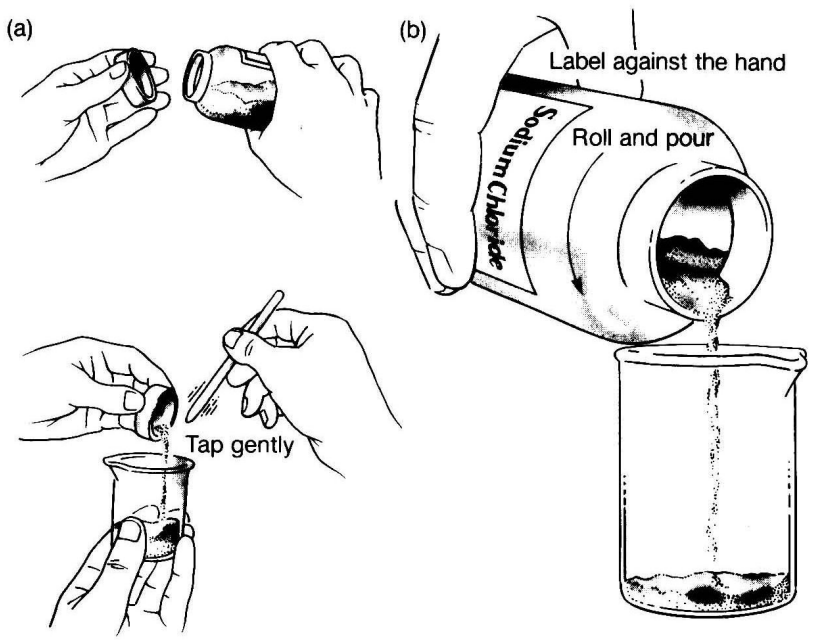

Figure A-1. Transfer of solid from a reagent bottle.

2. LIQUIDS

Many commonly used laboratory reagents are solutions while others are (pure) liquids such as alcohol or acetone. Liquid reagents are stored in a variety of bottles such as those shown in Figure A–2.

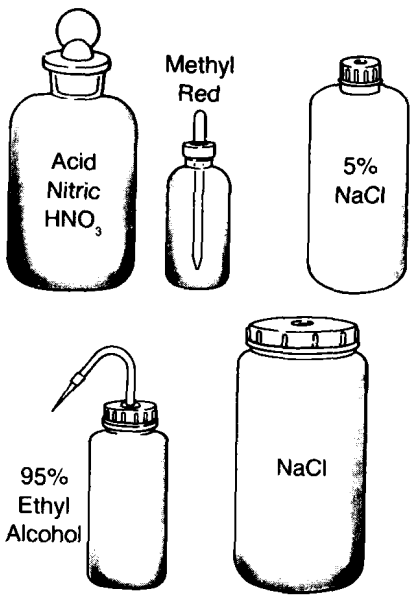

Figure A-2. Common reagent bottles for liquids and solids.

Squeeze dropper bottles or bottles fitted with eye droppers are used when very small amounts of the liquids are required in experiments. When larger amounts of liquid are required standard reagent bottles are often used. Hazardous reagents are stored in special bottles with self–filling dispensers (Brinkman Dispensettes, Akso SMI) Figure A–3 illustrates the transfer of a liquid from a standard reagent bottle. **You should always check the label on a reagent bottle carefully before you remove any liquid from the bottle. Careful checking of labels prevents many unnecessary accidents.**

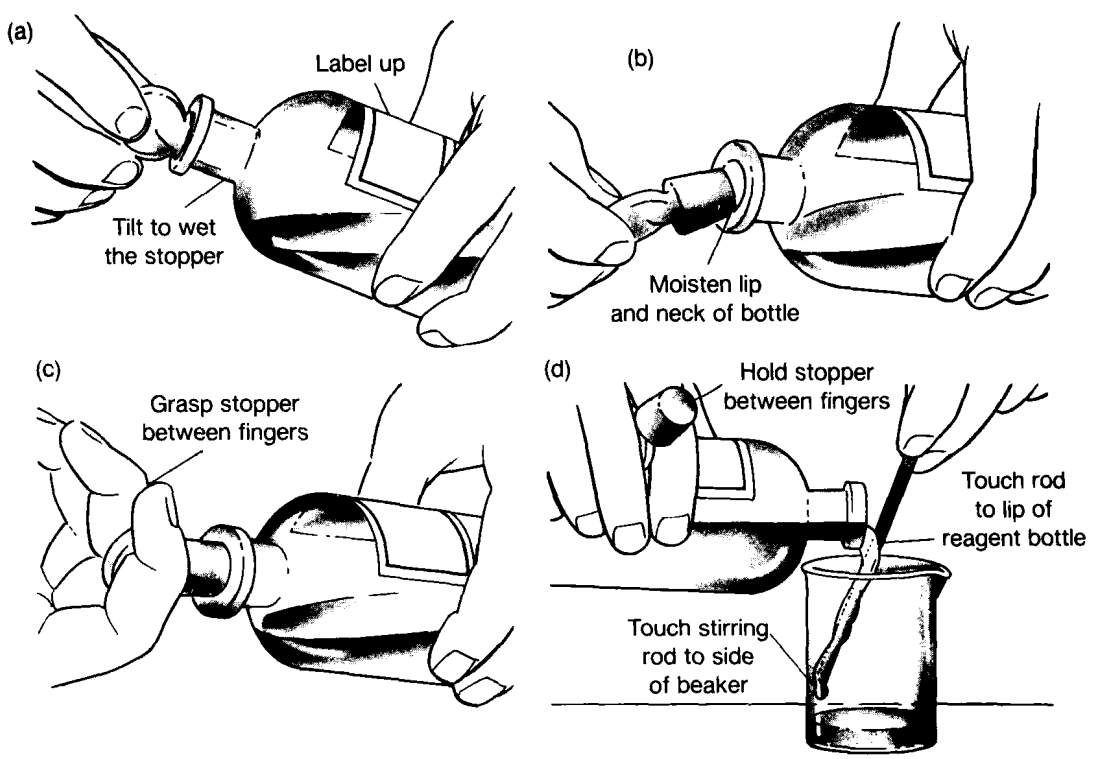

Figure A-3. Transferring liquid from a reagent bottle.