

EXPERIMENTS IN GENERAL CHEMISTRY

Whitten and Gailey

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PREFACE

Chemistry is an experimental science. We know what we know about chemistry because we (literally thousands of scientists) have observed it to be true. We do not suggest that theory is less important than experiment, only that theories must be subjected to testing in the laboratory.

We have attempted to provide an opportunity for students to learn to make measurements and observations, and then to interpret their measurements and observations. In the first part of this manual we provide an introduction to most of the laboratory techniques that students will use. Written descriptions are illustrated by art work that is large and clearly drawn so that students can "see for themselves."

The appropriate chapters, sections, and topics in the texts by the same authors are listed at the beginning of each experiment. Since topics are listed, students can usually find the corresponding sections in other general chemistry books with little effort.

A rather detailed review of the relevant theory and principles is given in the Introduction to each experiment. The Procedure section of each experiment begins with a listing of the relevant sections in Laboratory Techniques. Procedures are described in considerable detail so that students can set up apparatus correctly and quickly. Safety precautions are emphasized at appropriate points throughout the manual.

Each experiment has a set of Prelab questions--usually both qualitative and quantitative questions are included. We use these prelab questions as admission tickets to the laboratory.

We have provided report forms that have spaces for observations and interpretations. Since most experiments also have an "unknown", we have provided a space for the unknown number as close to the result as possible to facilitate grading.

The laboratory directions (only) for the exercises in qualitative analysis are included in this manual so that students do not have to bring large (expensive) textbooks into the laboratory. These directions are copied verbatim from GENERAL CHEMISTRY WITH QUALITATIVE ANALYSIS by the same authors.

We have included a reasonable number of quantitative and descriptive experiments so that each professor has considerable latitude in choosing experiments.

The experiments may be divided into classes as shown below.

Experiments

1, 2, 3

4, 5, 6

7

8

9, 10, 11, 12, 13

14, 15, 16, 17, 18

Classifications

Basic laboratory techniques
(simple measurements and observations)

Gravimetric techniques

Introduction to inorganic synthesis

Structure and bonding

Introduction to reaction chemistry
(organization and interpretation of data)

Introduction to physical measurements

Experiments

19, 20

21, 22

23, 24, 25, 26

27, 28, 29, 30

31, 32, 33, 34, 35

36, 37, 38

39, 40, 41, 42, 43, 44

Classifications

Acid-base chemistry
(titrations)

Oxidation-reduction reactions
(titrations)

More sophisticated physical
measurements

"Descriptive" experiments and
more inorganic synthesis

Introductory organic experiments

Qualitative analysis

The Instructor's Manual provides detailed lists of chemicals, equipment, and the amount of time required for each experiment. Copies are available to all adopters of this laboratory manual.

TO THE STUDENT

These experiments were written to provide a meaningful laboratory experience. They were designed to assist you in learning to make measurements and observations and in learning to interpret them. The laboratory exercises provide an opportunity for you to participate in chemistry and draw reasoned conclusions based on your work.

You should study the assignments for the day carefully before you come to the laboratory. At the beginning of each experiment there are references to the appropriate sections in the texts by the same authors, 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 each exercise 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 questions and hand them in as you enter the laboratory. The prelab questions serve as an "admission ticket". Arrange your work neatly so that your instructor can read it easily.

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 calculation. No credit will be given for simply "writing down" answers to questions that require calculations.

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. Report forms should be turned in before you leave the laboratory.

A word about safety is necessary. Study the section on Safety and Laboratory Rules before the first laboratory period. Safety rules are designed to provide a safe environment in which all may work. Many experiments contain comments about safety. Be alert---read instructions carefully and then follow them. No food or drink is allowed in the laboratory. You must wear safety goggles. You must wear shoes. No unauthorized experiments or horseplay are permitted. Only students who are assigned to a particular laboratory at a particular time may work at that time. Those who abuse equipment, waste chemicals, or disregard instructions will be expelled from the laboratory.

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Lloyd E. Logan, Watkinsville, Georgia, drew most of the illustrations. We appreciate his understanding and skill.

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SAFETY AND LABORATORY RULES

The laboratory is a safe place in which to work if appropriate precautions are taken and sound judgment is exercised. The laboratory can be a dangerous place if one is careless. Therefore, the first rule of the laboratory is:

EXERCISE CAUTION AND GOOD JUDGMENT

The second rule is:

KNOW THE LOCATIONS OF ALL SAFETY EQUIPMENT AND THE FIRST AID KIT. The first time you go into the laboratory, you should learn the location of:

- a. the fire extinguisher
- b. the safety shower
- c. the "eye-wash" fountain
- d. the first aid kit

Your instructor will provide specific instructions for the use of each at the first laboratory meeting.

The third rule is:

ALWAYS FOLLOW DIRECTIONS EXACTLY UNLESS YOUR INSTRUCTOR PROVIDES MODIFICATIONS.

The fourth is:

ALWAYS READ LABELS CAREFULLY. Often two or more bottles that contain the same reagent are located side-by-side on the reagent shelf. The addition of concentrated nitric acid, HNO_3 , to a reaction mixture when the directions call for the addition of dilute nitric acid may result in a reaction so vigorous that concentrated nitric acid spatters over a large area. Concentrated nitric acid is very corrosive! It destroys skin almost immediately! Many other examples can be cited. Therefore, you should always read labels carefully.

The fifth is:

ALWAYS CALL FOR HELP LOUDLY WHEN AN ACCIDENT OCCURS. This alerts your instructor and others who are working nearby. Even when an accident appears quite minor, you should notify your instructor immediately.

Commonly accepted safety precautions include the following:

1. Wear safety glasses in the laboratory at all times. Contact lenses should not be worn in the laboratory at any time.
2. Wear shoes that provide protection to your feet, i.e., shoes that "shed" liquids, at all times. Sandals and "open top" shoes are not permitted in the laboratory.

3. Long hair must always be confined. Long hair is a serious fire hazard.
4. Laboratory aprons or coats provide protection for your clothing--never wear expensive clothing into the laboratory.
5. Never eat, drink, or smoke in the laboratory. These activities are strictly prohibited.
6. Consider all chemicals to be dangerous (hazardous) unless you are specifically instructed otherwise.
7. Never taste chemicals unless specifically instructed to do so.
8. Never smell gases or vapors directly. When you are specifically instructed to report the odor of a chemical, gently waft the vapors toward your nose cautiously.
9. If chemicals come into contact with your eyes or skin, wash immediately with large amounts of water. Yell for help while you are washing. Contaminated clothing should be removed immediately. Your modesty will suffer only slightly and you may save considerable portions of your skin!
10. Never rub your eyes unless you are absolutely sure that there are no chemicals on your hands.
11. Volatile chemicals that are poisonous, irritating to the skin, or that have unpleasant odors should always be used under the fume hood.
12. Never point a test tube that is being heated toward yourself or anyone else. It may "spatter" chemicals over a large area.
13. Most organic liquids are flammable, as are some gases such as hydrogen. Never use flammable chemicals near a flame or a "hot" hot plate.
14. Clean up all broken glass immediately. Similarly, spilled chemicals should be cleaned up immediately--consult your instructor for details.
15. Always pour concentrated acids into water, never water into acid. Some concentrated acids (sulfuric acid, for example) release enough heat to cause severe spattering. Hot, concentrated acids are very corrosive.
16. Observe carefully the safety precautions that are included in the experiments. The section on Laboratory Techniques includes many safety precautions.
17. Always be alert to the possibility of an accident by your neighbors. You could be a victim of their mistakes. Always advise them of any unsafe practices you observe. If necessary, inform your instructor immediately.
18. Always maintain an orderly, business-like attitude and a clean, orderly working space.
19. Working alone in the laboratory is strictly prohibited. An instructor must always be present.
20. Neither unauthorized experiments nor unauthorized modifications of assigned experiments are permitted.
21. If you have any doubt about what to do in any set of circumstances, consult your instructor.
22. Always keep doors to lab desks and laboratory drawers closed except when you are placing something into or removing something from your drawer or desk. Open doors and drawers obstruct the aisle--such obstruction may cause serious accidents.

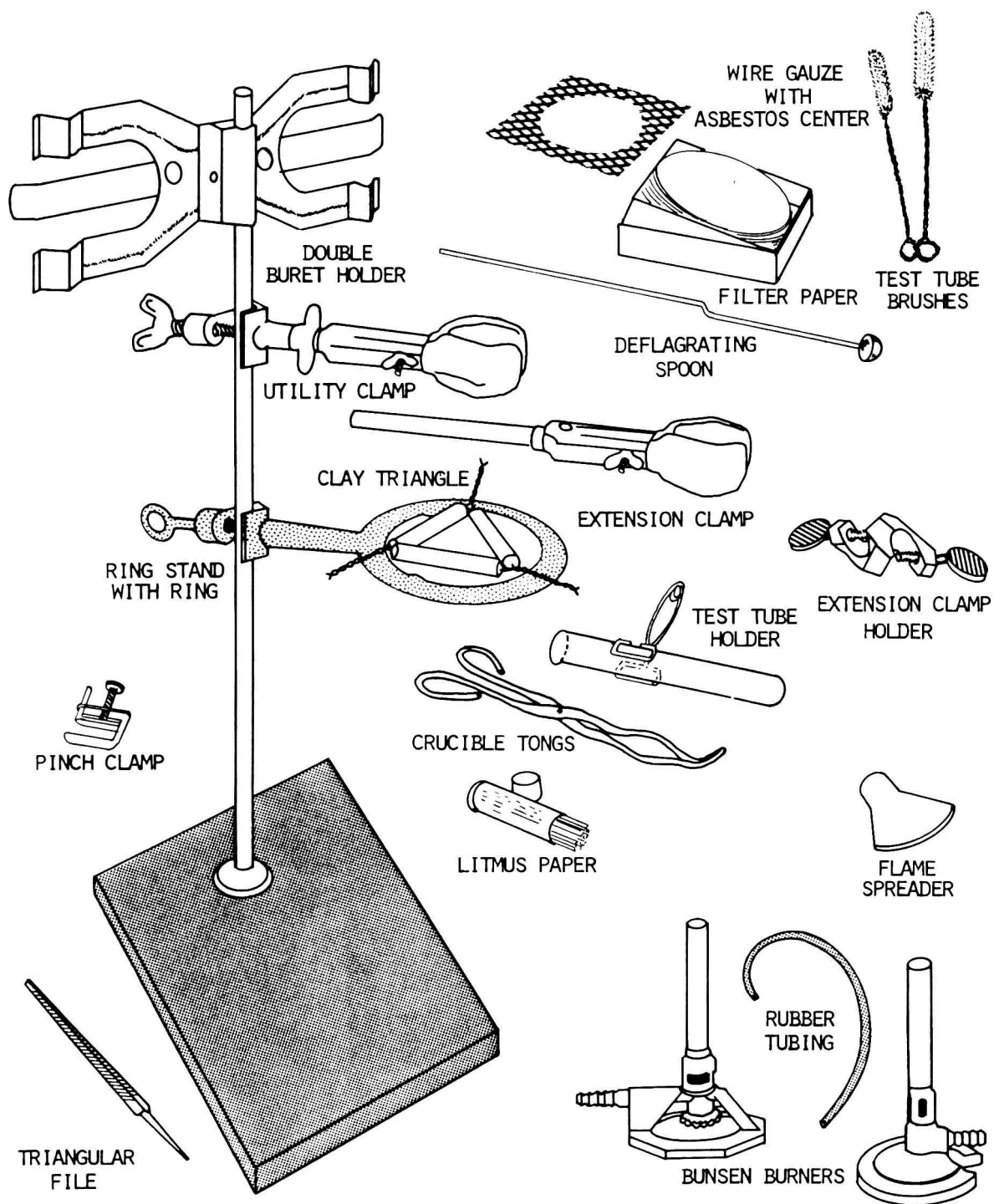


FIGURE I-1 COMMON LABORATORY EQUIPMENT

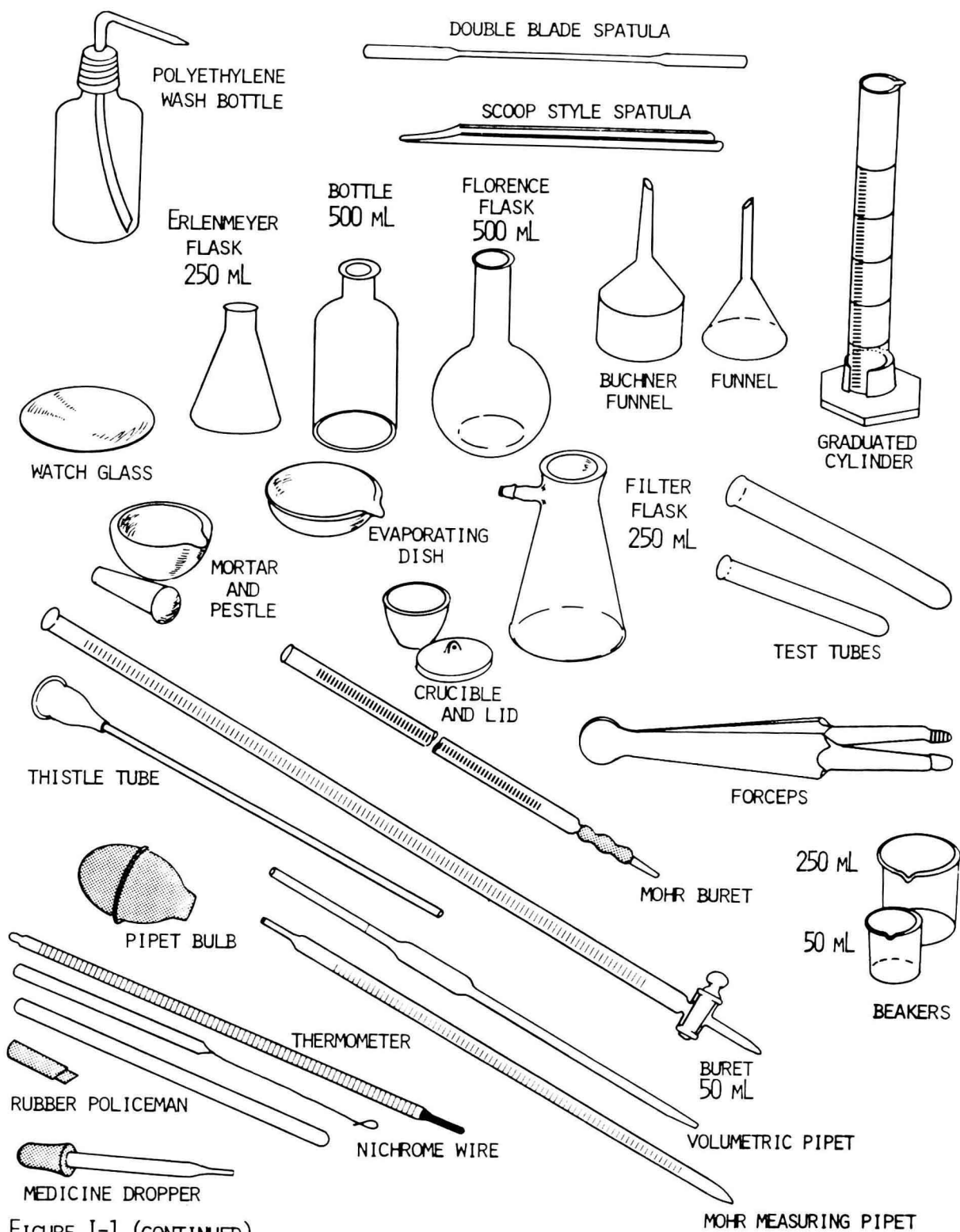


FIGURE I-1 (CONTINUED)

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 back and forth until the desired amount of solid falls out of the bottle into the appropriate container. Replace the lid or stopper and return the bottle to its proper location. Spatulas should not be placed in solid reagent bottles. If a solid reagent is compacted or "caked" ask your instructor for assistance. Neither solid nor liquid chemicals should be returned to the bottles from which they were removed--discard any excess.

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-1.

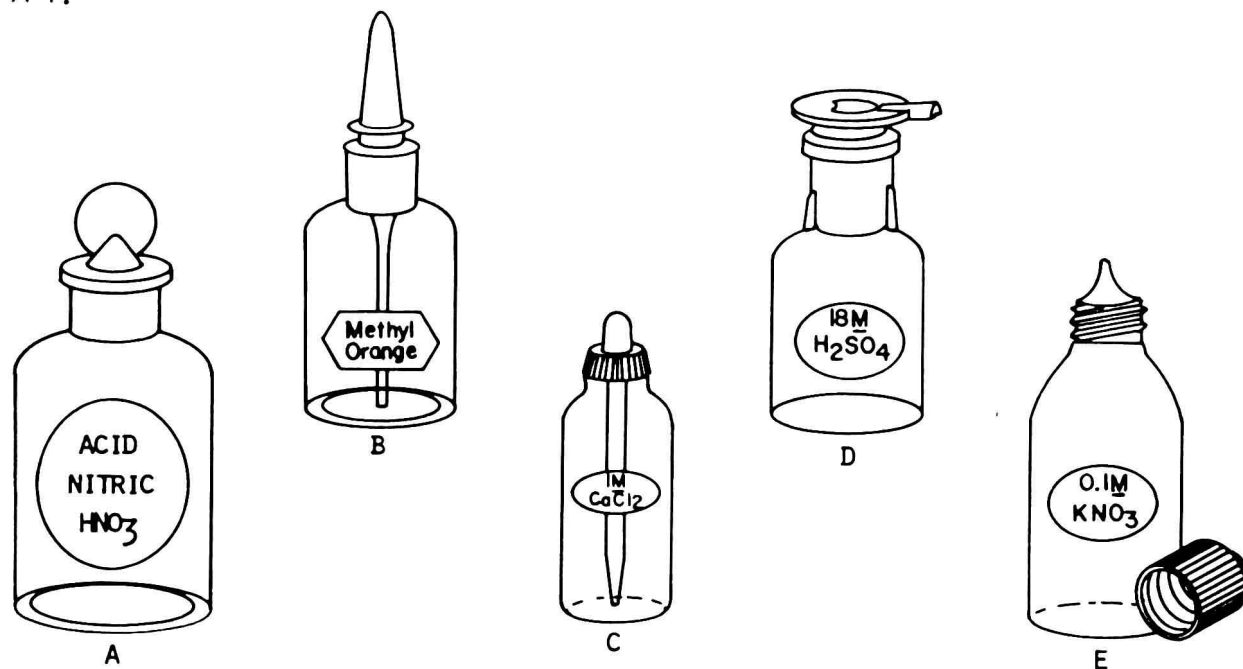


FIGURE A-1 LIQUID REAGENT BOTTLES. (A) STANDARD REAGENT BOTTLE. (B-E) DROPPER BOTTLES.

Dropping bottles are used when very small amounts of the liquids are required in experiments. When larger amounts of liquids are required standard reagent bottles are often used. Figure A-2 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.

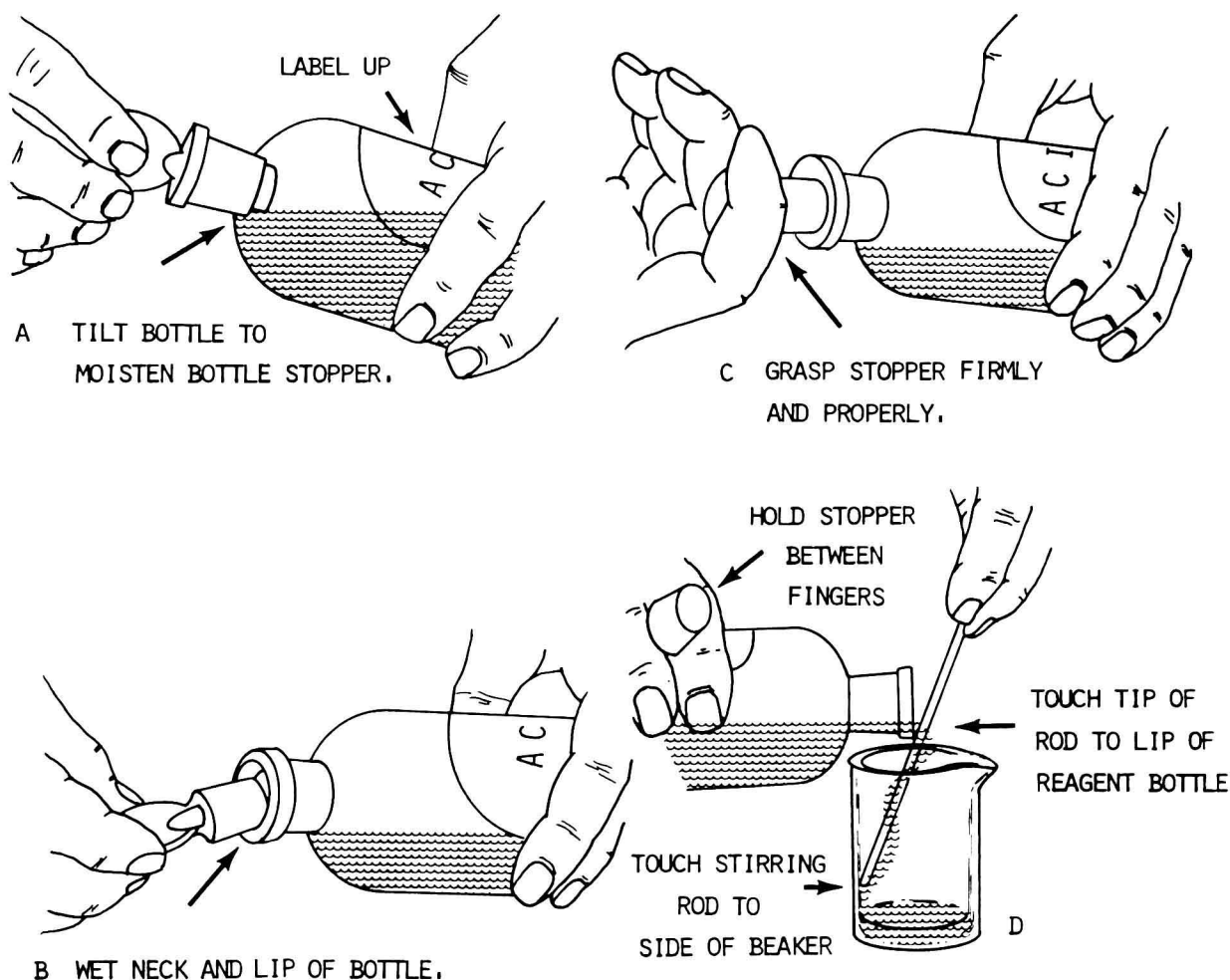


FIGURE A-2 TRANSFER OF A LIQUID FROM A STANDARD REAGENT BOTTLE.