Chemical Principles in the Laboratory

Second Edition

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Chemical Principles in the Laboratory

PREFACE

The laboratory of the medieval alchemist was a formidable and mysterious place. A modern chemistry laboratory, with its walls lined with reagent bottles and its benches arrayed with sensitive instruments, also presents an initial aura of mystery to a beginning student of science. It is the goal of *Chemical Principles in the Laboratory, Second Edition,* to transform the mystery of the scientific unknown into the excitement of scientific discovery.

To accomplish this transformation, we have prepared a large number of experiments, and we have arranged these experiments in two patterns. First, experiments are sequenced to help students acquire laboratory skills in a systematic fashion and to develop the scientific insight they need as they enter more sophisticated areas of chemistry: Elementary experiments introduce fundamental topics such as measurement techniques and stoichiometry; more advanced experiments lead naturally into independent projects and into higher-level laboratory work. Second, experiments are organized into thematic groups so that students can achieve technical and intellectual mastery of several areas of experimental chemistry. The arrangement of experiments is flexible: One experiment may be chosen from a group, or students may develop a series of coordinated experiments into an extended laboratory project on a topic such as water pollution (Experiment 13), consumer products (Experiments 6 and 15), or drugs and vitamins (Experiments 24 and 25).

We have selected the experiments in *Chemical Principles in the Laboratory* to immerse students in challenging, interesting, and contemporary laboratory problems. The experiments reinforce important chemical concepts, teach modern laboratory techniques, and emphasize present-day interpretations of laboratory measurements. All the experiments have been carefully designed to maximize laboratory safety, economy of expendable materials, and freedom from environmental pollution.

We have chosen the sequence of experiments to conform to the sequence of topics in most general chemistry courses, and in particular to the sequence of topics in *Chemical Principles, Second Edition,* by Robert S. Boikess and Edward Edelson. Each group of experiments begins with suggested textbook readings, prelaboratory questions, and objectives for the topic being investigated. The principles necessary for understanding the theory of each experiment are explained. All the experiments have been carefully class-tested, in most cases over a period of four years, by students at Rutgers University.

An *Instructor's Manual and Storeroom Guide* is available as a combined volume with the *Instructor's Manual to accompany Chemical Principles, Second Edition.* It lists all required supplies for each experiment and provides recipes that can be used by the stockroom staff for the preparation of all solutions. It includes suggestions for the instructor to ensure that each experiment is carried out smoothly and efficiently. It also gives answers to all the questions asked of the students and presents sample data for many experiments.

We wish to express our appreciation to our many students and colleagues who helped devise and test these experiments.

Lester R. Morss
Robert S. Boikess

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INTRODUCTION

The essence of any science is that mental constructs (models and theories) must be tested and refined by objective measurements on real systems (experiments). The goals of laboratory work in a general chemistry course are somewhat different from the goals of laboratory work in research. Primarily, we design laboratory "experiments" (which have predictable results) *to emphasize and to reinforce important textbook concepts*. Almost as important, we select laboratory "exercises" *to teach essential experimental skills* which will be useful in your future scientific work. In addition, we also provide extended experimental investigations and problems to encourage you *to apply your knowledge of chemistry* in a creative and independent way.

This laboratory manual is designed to present you with experiments that are *challenging, coordinated investigations* related to your study of chemical principles. These experiments utilize modern techniques and apparatus and emphasize operations performed by practicing scientists. Wherever possible, they are quantitative and related to contemporary problems in pure and applied chemistry. These experiments have also been designed to be efficient, safe, economical, and nonpolluting.

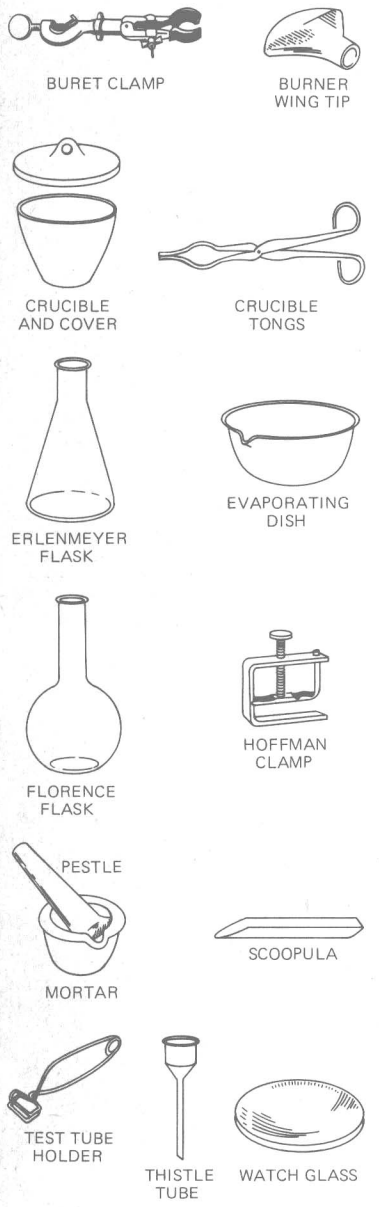
A. LABORATORY POLICIES

Please read and follow these policies before beginning laboratory work.

1. During the first week of classes, locker and stockroom arrangements will be explained. Since the administration of a laboratory involves coordination of the activities of many students, be sure to note and to follow requirements that may require careful attention (locker assignments, payment of fees, lock combinations, laboratory hours, etc.).
2. You will be required to wear *safety glasses with side shields* at all times in the laboratory. In most laboratories, prescription eyeglasses may be worn if they are fitted with plastic side shields. Shoes are also a necessity in the laboratory. You should also wear old clothes or invest in a laboratory coat or apron.
3. Your instructor will point out the location and proper use of the safety shower, eye wash, fire extinguisher, and fire blanket. Remember their locations and the locations of emergency exits from the building. Read the chapter on Laboratory Safety *now*, and again during the middle of the semester.
4. Laboratory housekeeping is the responsibility of *everyone* in the laboratory. Carry out the following responsibilities:
   a. Pour out only the amounts of chemicals you need. Do not hoard or waste chemicals; yields are seldom graded. In general, do *not* pour anything back into a reagent bottle.
   b. Clean up any spills (especially in or near a balance) at once. Report any unusual spills or breakages to your laboratory instructor. The stockroom staff will assist in emergencies.
   c. Dispense malodorous reagents (such as concentrated HCl and NH_3 solutions) in a hood if practicable. Set up and conduct all experiments that release fumes directly in a hood, so that all gaseous products enter the hood intake.
   d. *Never* insert an unclean spatula or pipet into a reagent bottle. Your instructor will show you how to dispense reagents so as to minimize spilling—especially inside a balance chamber.
   e. Many reagents (such as metal samples that do not undergo chemical reactions in certain experiments) can be recycled; many solid

reagents cannot be safely disposed of in trash receptacles. When recycling or waste containers are provided, use them. Do not pour insoluble solids in sinks; solutions that may be discarded in sinks should be flushed thoroughly with water.

5. Organize your laboratory time so that you will have *cleaned up* your apparatus and be *out of the laboratory* at the end of the laboratory period. Your instructor must remain in the laboratory until all students have left; be courteous by completing your work promptly.



B. LABORATORY LOCKERS

A typical laboratory equipment list is shown below.

1 beaker, 30 cm^3
1 beaker, 100 cm^3
1 beaker, 250 cm^3
1 beaker, 400 cm^3
1 beaker, 600 cm^3
1 burner, Bunsen
1 burner wing tip
1 clamp, buret
1 clamp, Hoffman
1 crucible, porcelain with cover
6 dropping bottles
1 evaporating dish
1 flask, Erlenmeyer, 25 cm^3
1 flask, Erlenmeyer, 125 cm^3
1 flask, Erlenmeyer, 250 cm^3
1 flask, Florence, 500 cm^3
1 funnel
1 graduated cylinder, 10 cm^3
1 graduated cylinder, 100 cm^3
6 medicine droppers
1 mortar, porcelain
1 Nichrome wire
1 pestle, porcelain
60 cm of rubber tubing, small
60 cm of rubber tubing, burner
1 scoopula
2 stirring rods, glass
6 test tubes, 7.5 cm
10 test tubes, 10 cm
6 test tubes, 15 cm
1 test tube, ignition, 15 cm
1 test tube, ignition, 20 cm
1 test tube holder
1 test tube rack
1 thermometer, −10 to 150°C
1 thistle tube, plastic
1 tongs, crucible
1 triangle, clay sides
1 watch glass, 8 cm
1 wire gauze
1 wash bottle, plastic

Your instructor will help you identify unfamiliar items during the check-in laboratory period. Maintain your laboratory ware in clean condition all year.

C. MAKEUP LABORATORIES

Special reagents for a given experiment are normally kept in the laboratory for a 2- or 3-week interval. Therefore, if you are forced to miss a laboratory period, you must arrange a makeup laboratory period promptly. It is usually inadvisable to attend another instructor's laboratory without prior approval, because space may not be available. If possible, arrange for a makeup laboratory using one of the makeup laboratory approval forms at the end of this chapter.

D. LABORATORY REPORTS

Each week, *before coming to the laboratory,* read the experimental principles and procedures for the experiment that you will do. Compare the procedures with the data sheet, so that you will be sure to record all

necessary information. Answer all prelaboratory questions and write down any questions you wish to have answered during the laboratory period. (The time expended in advance preparation will be more than repaid in an efficient laboratory period and in a worthwhile laboratory report.

Take data in the laboratory only on *data sheets and in ink*. If you make an erroneous entry, strike it out with a single line; do not erase or write over incorrect data. Before leaving the laboratory, be sure that you have

1. Completed all required work and taken all data.
2. Asked your instructor to sign your data sheet.
3. Been told what to include in your laboratory report, including details of data interpretation, error analysis, and assigned problems.

Complete your laboratory reports as directed by your instructor. Work *independently* in performing calculations and in completing your laboratory reports, even if you collect data with a partner. A typical laboratory report includes these four sections (see sample laboratory report on page 4):

1. A cover sheet with your name, section, course number, and instructor's name. Write the title of the experiment and a concise statement of the experimental objectives on the cover sheet; do not copy or paraphrase the text of the laboratory manual, but add any deviations from the procedures in the laboratory manual.
2. Data sheets, signed by your instructor.
3. Data interpretation: calculations, results, graphs, and so on. Include the conclusions you have drawn from the experiment. If interpretations of your results are called for (such as identification or explanation of a particular result or the error or uncertainty of a result), state and justify your interpretations. (See Evaluation of Experimental Data, page 9). If appropriate, criticize the experimental arrangement and recommend improvements.
4. Assigned problems or questions.

Staple your report at the upper left; if it is a makeup laboratory, attach the makeup approval form to the front. Submit your report at the next laboratory session; late reports will be penalized.

Laboratory reports are graded on the following bases:

1. Clarity of statements of objectives, conclusions, interpretations, and criticism. Use concise, declarative sentences in the active voice: Say "I observed . . ." instead of "It was observed that. . . ."
2. Neatness, completeness, and accuracy of data recording and calculations.
3. Error treatment: significant figures, error estimations, uncertainty limits, quantitative discussion of uncertainties. *Note*: Misreading a balance, spilling a sample, and errors in calculation are not experimental errors but blunders. (Experimental error is the error present when careful techniques have been followed.) (See Evaluation of Experimental Data.)
4. Complete references to any books or written procedures you have followed.

SAMPLE LABORATORY REPORT

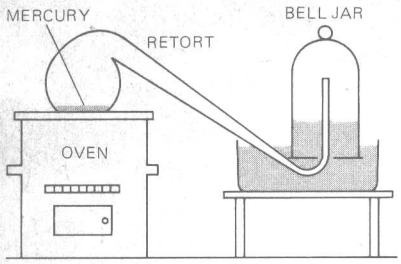


FIGURE 1
Apparatus used by Lavoisier to demonstrate the formation of mercury calx (HgO) and dephlogisticated air (air from which oxygen has been removed) from mercury and air.

(Page 1)

Antoine Lavoisier

Rutgers College, Chemistry 103, Section 01, October 10, 1777

Instructor: Guillaume Rouelle

Preparation of Dephlogisticated Air

The objective of this experiment was to prepare dephlogisticated air by the method of Priestley and to study its properties. The procedure of Priestley (see Bibliography) was followed except that I modified the procedure by heating mercury in a closed vessel containing a large volume of air (see diagram).

(Page 2)

Initial mass of mercury: 7.350 ± 0.002 g

Final mass of mercury: 7.942 ± 0.002 g

Mass of red mercury calx: 7.942 ± 0.002 g

Initial volume of air: 10.567 l ± 0.020 l

Final volume of air: 10.114 l ± 0.020 l

Temperature: 0°C ± 3°

Barometric pressure: 770 mm Hg

Instructor's signature: G. Rouelle

(Page 3)

CALCULATIONS:
Mass of mercury reacted: 7.350 ± 0.002 g
Mass of mercury calx: 7.942 ± 0.002 g
Mass of dephlogisticated air removed from vessel, assuming all combined to form calx:

7.942 g − 7.350 g = 0.592 ± 0.003 g

Volume of dephlogisticated air: 0.453 ± 0.028 liter
Density of dephlogisticated air: 1.311 ± 0.081 g/liter

CONCLUSIONS:
When red calx of mercury is heated, a gas is evolved which has all the properties attributed to it by Priestley. However, I disagree with Priestley's interpretation that this gas is "dephlogisticated air." My modification of the experiment shows that mercury absorbs the salubrious and respirable part of air. Consequently atmospheric air is composed of two fluids of

different and opposite qualities. I will refer to the eminently respirable air produced on heating mercury calx by the term "oxygine."

ERRORS:
My largest source of error was in measuring volumes. This could be made more accurate by designing a vessel with a narrower cross section which can be more precisely calibrated. A second source of error is the small weight change observed when mercury reacts to its red calx. Since my balance is one of the best available, I cannot diminish this source of error. A source of indeterminate error is the precision with which I can measure temperature: I cannot insert a thermometer into the experimental vessel and the room temperature varies by ±3°C. Using the law of Chanes, the largest error this could introduce into my result for density is 3/298 or 1.0%. Since my precision has a percent error, from volume alone, of (0.028/0.453)100 = 6.2%, I must improve my precision (by replacing the bell jar with a vessel of narrower cross section) before worrying about other sources of error.

(Page 4)

PROBLEMS:
How much water is produced by burning an excess of inflammable air with the oxygine formed from 2 g of red mercury calx?

$$2.00 \text{ g HgO} \left(\frac{0.592 \text{ g O}}{7.942 \text{ g HgO}}\right)\left(\frac{1.12 \text{ g water}}{1.00 \text{ g O}}\right) = 0.167 \text{ g water}$$

BIBLIOGRAPHY:
1. Joseph Priestley, *Experiments and Observations on Different Kinds of Air*, Johnson, London, 1775.
2. Aaron J. Ihde, *The Development of Modern Chemistry*, Harper & Row, New York, 1964 (with apologies).

GENERAL CHEMISTRY
MAKEUP LABORATORY APPROVAL*

Name _____ Regular laboratory date _____

Section _____

Instructor _____

Experiment _____

Reason for makeup _____

Obtain an instructor's approval (by signing below) before coming to the makeup period. Show this approval to the chemistry instructor in your laboratory or in the nearest laboratory and ask his or her permission to work in the laboratory. Complete all work within the regular hours. Ask the instructor to sign your data sheet. You are *never* permitted to work alone. (Attach this approval form to your report to avoid a penalty.)

Signature of laboratory instructor: _____

- -

GENERAL CHEMISTRY
MAKEUP LABORATORY APPROVAL*

Name _____ Regular laboratory date _____

Section _____

Instructor _____

Experiment _____

Reason for makeup _____

Obtain an instructor's approval (by signing below) before coming to the makeup period. Show this approval to the chemistry instructor in your laboratory or in the nearest laboratory and ask his or her permission to work in the laboratory. Complete all work within the regular hours. Ask the instructor to sign your data sheet. You are *never* permitted to work alone. (Attach this approval form to your report to avoid a penalty.)

Signature of laboratory instructor: _____

* If authorized by the instructor in charge of your course.