Instructor's Manual

Chemical Principles in the Laboratory

Eleventh Edition

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INSTRUCTORS' GUIDE FOR
CHEMICAL PRINCIPLES IN THE LABORATORY, 11TH EDITION

PREFACE

The adoption of a different laboratory manual always involves a certain amount of work on the part of the stockroom personnel and the laboratory supervisor at the institution, since there will inevitably be some new equipment which is required and different reagent solutions which must be prepared. The purpose of this guide is to make the transition to "Chemical Principles in the Laboratory" as easy as possible, and to assist in the matter of having the laboratory session work smoothly and productively.

To this end we have divided the guide into two rather distinct parts. In the first part we have listed the equipment and chemical needs for each of the experiments. The equipment specified is that not already included in the recommended student equipment listed in Appendix VI of the lab manual. The amounts given are those we have found necessary, or convenient, for handling one laboratory section of 20 students. Since most courses in general chemistry will be larger than this, some much larger, we also have listed in parentheses after each reagent an estimate of the amount of that reagent that would be used by one student. Since reagent solutions and chemicals must be prepared somewhat in advance of the laboratory sessions, these estimates should serve at least as an initial rough guide as to requirements for the entire group of students who will be performing the experiment. Amounts actually used will obviously vary, depending on conditions, and your experience with a given experiment will give you a better estimate. In some cases the cost of the chemicals required may be very significant, and we have kept the amounts used in such instances to a practical minimum. Directions for the preparation of all reagent solutions used in the experiments are given at the end of this guide.

Since the expense involved in performing a laboratory experiment varies considerably, we have included an estimate of the cost per student, based on 2007 prices, with each experiment. Most experiments cost about 50¢ per student, for a total of roughly $8 per student per semester, exclusive of the cost of recoverable metals and chemicals. In some of the experiments we call for equipment that may not be immediately available. For the most part, this equipment is easy to make, and can be constructed by a departmental glass blower or by a staff member who is reasonably adept in glassblowing. Making the equipment does take a little time and plans must be made in advance for its construction. The experiments in which non-commercially available glassware is desirable are the following:

Experiment 8 Measurement of the Atmospheric Pressure – U-tubes
Experiment 9 Molar Mass of a Volatile Liquid – Vapor flasks
Experiment 15 Vapor Pressure of a Volatile Liquid – Modified pipets

In all cases, we have described in the equipment section how the apparatus can be made. Where it is not feasible to make the equipment, it is usually possible to use alternate experiments on similar topics; a vapor flask for Experiment 9 made from commercially available components is described.

In several experiments we use commercial laboratory instruments. These instruments are now extremely common in all graduate schools and industrial laboratories and in many undergraduate institutions. We feel that it is highly desirable to introduce students to such apparatus in their first course. In addition to analytical and top-loading balances, which we use in many of the experiments, the following experiments include procedures involving the indicated instruments:

Experiment 23 Determination of an Equilibrium Constant - spectrophotometers
Experiment 25 pH and Buffer Properties - pH meters
Experiment 26 Determination of a Solubility Product - spectrophotometers
Experiment 29 Synthesis of a Coordination Compound - spectrophotometers
Experiment 32 Voltaic Cell Measurements - pH meters or high resistance voltmeters
Experiment 33 Preparation of Cu(I)Cl - spectrophotometers
Experiment 41 Preparation of Aspirin - spectrophotometers
Experiment 42 Decomposition of Aspirin - spectrophotometers

The second half of this guide is directed to the laboratory supervisor. For each experiment, we have included some general comments, an estimate of the time required to complete the experiment and calculations, complete answers to all Advance Study Assignment questions, and a sample set of experimental data and calculations.

Although most of the experiments we have included have been tested and found to work quite well, there may well be problems which arise, in any of the many areas that are involved in the operation of a laboratory program, which we have not anticipated. The authors would sincerely appreciate any comments and suggestions from users of the lab manual or this guide. We are eager to help with troubleshooting where we can.

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List of Experiments

Experiment

EQUIPMENT AND CHEMICAL REAGENT REQUIREMENTS FOR EXPERIMENTS

Note to stockroom personnel:

In stating equipment and chemical reagent needs, we have adhered to the following norms:

I. Laboratory equipment requirements are for a class of 20 students.

II. Suggested reagent container sizes are those we have found convenient for use with lab sections of 20 students. Typically we use four bottles of each reagent, one per lab bench. Amounts are usually more than one section of students will use, so that we do not need to refill the bottles during a lab session, or even (in most cases) during a day of sessions. Following the statement of the number and kind of container and the reagent, we list in parentheses the amount of that reagent which we think an average student will use. This should give you a rough guide as to requirements of chemicals for ordering purposes, as well as for preparation of solutions in advance.

III. Chemical reagents are listed by both their formula name and their word name. When making labels for containers, it would probably be best, in this course, to include both the formula name and the word name, along with the concentration of reagent, if it is given. For example:

18 M H_2SO_4 or 0.5 M NaOH conc. sulfuric acid sodium hydroxide

We have used to good advantage a commercially available unit, which prints the name of the chemical on 1/2" vinyl tape, which comes in different colors. This makes a neat, chemical-resistant label.

IV. Disposal of used reagents and reaction products must be handled properly. At the end of each experiment we tell the students what they are to do with the products of the reactions they carried out. Following the section directed toward laboratory supervisors there is a section on disposal of the chemical wastes from each experiment. If you follow the procedures we describe, you will minimize the amount of waste that needs to be picked up by a commercial hazardous waste disposal firm.

V. At the end of this guide there is a list of all the reagents used in the experiments, including some of the unknowns, and directions as to how each reagent solution is to be prepared. Chemicals are listed in alphabetical order by their word names, with chemical formulas of the substances given in the preparation directions.

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VI. There are several possible sources of the chemicals, glassware, and apparatus needed in the experiments. Some of the larger supply houses are listed below, with their addresses and toll-free telephone numbers:

Sigma-Aldrich
PO Box 14508
Saint Louis, MO 63178-4508
1-800-325-3010
www.sigmaaldrich.com
Reagents & Equipment

Frey Scientific
PO Box 3000
Nashua, NH 03061-3000
1-800-225-3739 [FREY]
www.freyscientific.com
Equipment, K-12 focus

Flinn Scientific
PO Box 219
Batavia, IL 60510-0219
1-800-452-1261
www.flinnsci.com
Reagents & Equipment

Cole Palmer
625 East Bunker Court
Vernon Hills, IL 60061-1844
1-800-323-4340
www.coleparmer.com
Equipment & Pure Metals

Ace Glass
PO Box 688
1430 North West Boulevard
Vineland, NJ 08362-0688
1-800-223-4524
www.aceglass.com
Glassware

Alfa Aesar
26 Parkridge Road
Ward Hill, MA 01835-8514
1-800-343-0660
www.alfa.com
Reagents & Pure Metals

Fischer Scientific
2000 Park Lane Drive, Suite 2
Pittsburgh, PA 15275-1104 1-
800-766-7000
www.fishersci.com *Reagents*
& Equipment

Strem Chemicals
7 Mulliken Way
Newburyport, MA 01950-4098
1-800-647-8736
www.strem.com
Reagents & Pure Metals

Sargent Welch Science
Education PO Box 4130
Buffalo, NY 14217-0360
1-800-727-4368
www.sargentwelch.com
Reagents & Equipment
(a subsidiary of VWR)

VWR Scientific Products
800 East Fabyan Parkway
Batavia, IL 60510-1406
1-800-932-5000
www.vwr.com
Reagents & Equipment

EXPERIMENT 1

The Densities of Liquids and Solids

Special equipment needed:

20 25 mL Erlenmeyer flasks with standard taper stoppers for use as pycnometers

Reagent available in laboratory:

4 × 250 mL wash bottles of acetone (10 mL/student)

Sample preparations:

A. Mass of a coin:

Students are to furnish their own coins, but you may want to have some pennies available for those who need them.

B. Density of a liquid:

The following liquids are suitable as unknowns. A total of 600 mL of organic liquids are required for 20 students. 30 mL of liquid should be placed in a large, numbered test tube. For the most part, these liquids can be recovered and re-used in the next laboratory section.

I ethanol
II isopropyl alcohol (2-propanol)
III *n*-heptane
IV cyclohexane
V toluene

Note: For large classes, using technical grade solvents will cut costs; the chemical quality of the solvents is not of great importance, provided the actual density is known. Use other solvents if available, but check for toxicity and carcinogenicity.

C. Density of a metal:

250 grams of each metal should be made available as unknowns. Fifty grams should be sufficient to constitute the unknown issued to each student. All solid chunks must be small enough to pass through the neck of the 25 mL Erlenmeyer flask used as the pycnometer. The metal pieces used should not have any entrapped air bubbles, as these will lead to erroneous density values. Metals can be recovered, cleaned easily by rinsing with acetone, and allowed to dry on a paper towel. Prepare one day's unknowns on the previous day, if possible, such that they have time to dry thoroughly. The following metals are suggested:

A. aluminum shot
B. bismuth shot
C. chromium pieces
D. cobalt shot
E. copper shot
F. nickel shot
G. tin shot
H. zinc shot

Cost per student, not counting recoverable metal and liquid samples: 30¢
Metal samples, approximate cost per 50 g (purchased in ~500 g quantities):

aluminum	$4.48	cobalt	$22.60	tin	$7.80	bismuth	$11.00
copper	$6.19	titanium*	$9.60	chromium	$11.50	nickel	$9.10
zinc	$5.74	zirconium*	$59.00				

* For very hard metals, purchase wire and cut it. The wire is hard to cut, but this is the easiest way.
Sources of metals: Alfa, Strem, Fisher, and Flinn, among others; prices vary widely. As with the liquids, the purity is not critical, provided that the metal is nominally pure, rather than an alloy. You will find that high-purity (>99.9%$_{mass}$) metals cost appreciably more than the amounts shown above.

EXPERIMENT 1

EXPERIMENT 2

Resolution of Matter into Pure Substances
I. Paper Chromatography

Special equipment needed:

20 pieces of Whatman #1 filter paper, 19 cm × 11 cm, cut from 57 cm × 46 cm sheets

Whatman #1 circular filter paper, 2" diameter or whatever is convenient, for use in practicing spotting

20 capillary melting point tubes, Kimax #34500-99 or Corning #9530-1

8 12" rulers or meter sticks; put on lab benches

2 tape dispensers

4 spray bottles which produce a fine spray

Reagents needed in the laboratory:

4 × 500 mL Eluting Solution, made by mixing 500 mL 6 M HCl with 400 mL ethanol and 400 mL *n*-butanol (15 mL/student)

Use spray bottles for Staining Reagent (see Directions for Preparing Reagents)

4 × 2 oz dropping bottles of the following solutions (0.5 mL of each/student):

0.1 M $AgNO_3$	0.1 M $Co(NO_3)_2$	0.1 M $Cu(NO_3)_2$
0.1 M $Fe(NO_3)_3$	0.1 M $Bi(NO_3)_3$	

Preparation of unknowns:

Use two or three drops of the solutions listed below, in micro test tubes:

I $AgNO_3$ - $Cu(NO_3)_2$ - $Fe(NO_3)_3$

II $Co(NO_3)_2$ - $Fe(NO_3)_3$ - $Bi(NO_3)_3$

III $Cu(NO_3)_2$ - $Fe(NO_3)_3$ - $Bi(NO_3)_3$

IV $AgNO_3$ - $Co(NO_3)_2$ - $Bi(NO_3)_3$

Cost per student: 25¢

EXPERIMENT 3

Resolution of Matter into Pure Substances
II. Fractional Crystallization

Special equipment needed:

20 Buchner funnels taking 70 mm filter paper in #6 1-hole stoppers
20 250 mL filtering flasks
4 boxes Whatman #1 7 cm diameter filter paper circles
Several aspirators (safety flasks should be installed) or other vacuum source
20 pneumatic troughs or ice cream buckets for ice baths
20 rubber policemen on stirring
rods Ice - crushed, at least 5 lbs.

Reagents needed in the laboratory:

4 × 200 mL dropping bottles 6 M HNO_3, nitric acid (2 mL/student)
4 × 200 mL dropping bottles 6 M NH_3, ammonia (6 mL/student)

Preparation of standard $Cu(NH_3)_4^{2+}$ solutions:

Make a copper stock solution by dissolving 4.2 g $CuSO_4 \cdot 5\ H_2O$ in 500 mL water.
Fill a buret with that solution and another buret with deionized water.
Measure out and combine the volumes (in mL) of the copper (Cu) stock solution, water (H_2O), and 6 M NH_3 (nitric acid) listed below; put the resulting standards in small test tubes, label as to concentration as shown below, and arrange in a test tube rack; put 6 mL in each test tube.

$\%_{mass}$ $CuSO_4 \cdot 5\ H_2O$ in recovered KNO_3	mL Cu stock sol'n	mL H_2O	mL 6 M NH_3
5	5	0	5
3	3	2	5
1	1	4	5
0.5	0.5	4.5	5
0.3	0.3	4.7	5
0.1	1 mL 1% sol'n	4	5
0.05	1 mL 0.5% sol'n	4	5
0.03	1 mL 0.3% sol'n	4	5
0.01	1 mL 0.1% sol'n	4	5

It is recommended that these test tubes be prepared in a hood or well-insulated area, and then stoppered before being made available to the students, so as to minimize ammonia fumes in the laboratory.

It is critical that all test tubes used in this portion of the experiment be of the same diameter.

Preparation of unknowns:

Each student should be given about 20 g of a solid unknown. Prepare in large batches, 500 g or so. Mix thoroughly before dispensing, grinding the salts if necessary to keep the mixture homogenous.

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EXPERIMENT 3

Suggested unknown compositions (for 500 g batches):

	SiC (~120 mesh/grit)	KNO_3 (purified grade)	$CuSO_4 \cdot 5\,H_2O$ (technical grade)
I	60 g	400 g	40 g
II	80 g	380 g	40 g
III	100 g	360 g	40 g
IV	120 g	340 g	40 g

Recover and reuse SiC and purified KNO_3. Keep in separate containers.