Chemical Principles in the Laboratory 12th edition Slowinski Solutions Manual

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Instructor's Manual

for

Chemical Principles in the Laboratory

Twelfth Edition

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INSTRUCTOR'S MANUAL FOR *CHEMICAL PRINCIPLES IN THE LABORATORY, 12TH EDITION*

PREFACE

The adoption of a different laboratory manual always involves a great deal of work, since there will inevitably be some new equipment required and different reagent solutions which must be prepared. The purpose of this manual 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 list in this manual 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 have also listed (in parentheses after each reagent) an estimate of the amount of that reagent normally used by one student. Since reagent solutions and chemicals must be prepared somewhat in advance of the laboratory sessions, these estimates provide a rough guide as to requirements for an entire group of students who will be performing an 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 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 manual.

Since the expense involved in performing a laboratory experiment varies considerably, we have included an estimate of the cost per student, based on 2020 prices, with each experiment. 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 glassblower or by a staff member who is reasonably adept at glassblowing. Making the equipment does take 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 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 also described.

In several experiments we use commercial laboratory instruments. These instruments are now common in most higher education institutions, and within reach for high schools as well. We feel that it is highly desirable to introduce students to such apparatus early in their chemistry careers. 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

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Experiment 33 Preparation of Cu(I)Cl—spectrophotometers
Experiment 41 Preparation of Aspirin—spectrophotometers
Experiment 42 Decomposition of Aspirin—spectrophotometers

Each experiment in the manual includes an "Advance Study Assignment," or "ASA," designed to assist students in preparing for the experiment and particularly (where applicable) in making the calculations required for it. In such cases they offer sample data and (in some detail) step the students through how that data can be used to obtain the desired results. If students work through the ASA for an experiment before coming to lab, they will be well prepared to make the necessary calculations on the basis of the data they obtain in the laboratory, and we encourage you to employ the ASAs as pre-lab exercises.

For each experiment we also include 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.

It is our unfortunate experience that in the modern era some students are sorely tempted to search online for the answers to any pre-lab assignments they are given, and may succumb to that temptation when pressed for time, or in pursuit of higher scores. As in all previous editions, we have changed the Advance Study Assignment questions in this edition; but this time we have done so with a particular eye to making it easier to catch those students availing themselves of the Internet as a tool for cheating. The changes to the questions are fewer and more subtle, such that students finding ASA answers online will have little certainty that what they have found actually applies to this edition of the manual. A consequence of this, however, is that students attempting to cheat and finding answers from the previous edition will not get markedly lower ASA scores: they will instead systematically get the same few questions wrong, and we urge you to be on the lookout for this pattern and actively address the moral failing (as well as the short-sighted intellectual decision) it indicates. You will be doing the student, and society, a favor by including this in your educational mission, and saving yourself many headaches down the road caused by such students arriving in lab without any real pre-lab preparation.

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 and / or this publication. We are eager to help with troubleshooting where we can.

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DISCLAIMER

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2 Resolution of Matter in Pure Substances, I. Paper Chromatography
3 Resolution of Matter into Pure Substances, II. Fractional Crystallization
4 Determination of a Chemical Formula
5 Identification of a Compound by Mass Relationships
6 Properties of Hydrates
7 Analysis of an Unknown Chloride
8 Verifying the Absolute Zero of Temperature—Determination of the Atmospheric Pressure
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General Information on Equipment and Chemical Reagent Requirements

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 chemical requirements 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. Near the end of this manual there is a section dealing with the 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 manual 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 where these can be obtained are listed below, with their addresses and toll-free telephone numbers:

Ace Glass
1430 North West Boulevard
Vineland, NJ 08360-2202
- or -
PO Box 688
Vineland, NJ 08362-0688
1-800-223-4524
aceglass.com
Glassware

Alfa Aesar
(part of Thermo Fisher Scientific)
2 Radcliff Road
Tewksbury, MA 01876-1182
1-800-343-0660
alfa.com
Reagents & Pure Metals

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

Fisher Scientific
(part of Thermo Fisher Scientific)
300 Industry Drive
Pittsburgh, PA 15275-1001
1-800-766-7000
fishersci.com
Reagents & Equipment

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

Frey Scientific
80 Northwest Boulevard
Nashua, NH 03061-3000
1-800-258-1302
freyscientific.com
Equipment, K–12 focus

Reagent World
2048 East Francis Street
Ontario, CA 91761-7722
1-800-573-9280
reagentworld.com
Reagents & Equipment

Sargent Welch Science Education
(part of VWR Scientific Products)
PO Box 92912
Rochester, NY 14692-9012
1-800-727-4368
sargentwelch.com
Reagents & Equipment

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

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

Thomas Scientific
PO Box 99
Swedesboro, NJ 08085-6099
1-800-345-2100
thomassci.com
Reagents & Equipment

VWR Scientific Products
(Part of Avantor)
Radnor Corporate Center
Building One, Suite 300
100 West Matsonford Road
Radnor, PA 19087-4569
1-800-932-5000
vwr.com
Reagents & Equipment

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GENERAL INFORMATION FOR LABORATORY SUPERVISORS

The main purpose of this manual is to serve as a source of information to those who have some role in the operation of a general chemistry laboratory in which our manual is used. For each of the experiments, we first attempt to spell out in detail the chemical and equipment needs, such that chemical and equipment preparations may be more readily made for each week of laboratory work. After this comes information directed toward those working with the students in the laboratories. For each experiment we include some general comments; these may involve a discussion of likely sources of experimental or calculation difficulties, expected experimental accuracy, an estimate of how long it will take for a class to complete the experiment and calculations, and properties of the unknowns which were used. Also included for each experiment is a realistic set of sample data and calculations, and solutions to all of the Advance Study Assignment questions. (Please see the Preface for an important note about the approach used in updating the ASAs for this edition.)

How this material will be used obviously depends on the laboratory supervisor. We have found it very helpful to have weekly meetings with all those involved in teaching a laboratory course, at which we discuss the sort of material included here. This will help ensure that all are sufficiently familiar with the theory behind each experiment, the procedures to be employed, difficulties to be avoided, and approaches to the calculations.

Advance Study Assignments are included in the manual as one approach that can be used to encourage students to prepare for lab. If the laboratory session is to be effective, students must arrive ready to work, not just ready to start finding out what they will be doing that day. It is rare for a student to truly prepare in advance unless given some sort of assessment based on such preparations, though that may take the form of an absolute deadline (the end of lab period) for completing the experiment. We do not have the perfect solution to ensuring students prepare for the laboratory, but offer the ASAs for your consideration. We have, however, based our time estimates on classes in which students were reasonably well informed about the experiment by the time they got to lab. [Please see the preface for an important note about the ASAs, if you avail yourself of them. Note also that they may be administered (both assigned and assessed) online using OWL.]

Some of the experiments include "Take it Further" optional applications, outlined right before the report page. These are intended to give students a role in planning experiments that have relatively practical applications; they are based on the procedures described, but require student input with regard to sample preparation and size as well as some modifications of the procedure and the calculations. We simply ask the student to solve a particular problem, with no suggestions as to how to proceed. You may decide to ignore these optional extensions of the experiments, or have students select them rather than do the experiment with the regular procedure. In general, these require more thought from the student than he or she may be used to, and give results that, at least initially, will not be as good as they would get if they were to simply follow the directions in the manual. You should talk with the students before they actually do their experiment to make sure that it will not involve any potentially dangerous steps.

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

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 (and avoid) toxicity and carcinogenicity.

C. Density of a solid:

200 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. We suggest choosing metals from this list:

A.	aluminum shot	F.	nickel shot
B.	bismuth shot	G.	tin shot
C.	chromium pieces	H.	zinc shot
D.	cobalt shot	I.	titanium wire segments
E.	copper shot	J.	zirconium wire segments

Cost per student, excluding the recoverable portion of metal and liquid samples: 38¢

Metal samples, approximate cost per 50 g (purchased in ~500-g quantities):

aluminum	$5.20	cobalt	$24.20	tin	$8.60	bismuth	$11.90
copper	$6.54	titanium*	$37.73	chromium	$12.70	nickel	$9.90
zinc	$6.27	zirconium*	$70.33	(Half that if you use plasma torch wire, but it is exhausting to cut!)			

* 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.

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This experiment is ordinarily done on the same day that the students check into the lab. We would suggest keeping the locker equipment used previously, since the items required for use with this manual are very typical of what is commonly stocked in student lockers, and any additions can be made as the need arises. See Appendix VI of the lab manual for a suggested list of locker equipment.

In the first part of this experiment the students should be introduced to the analytical balances in the lab. In most schools these balances are electronic, with a digital readout. They measure mass to the nearest ± 0.0001 g and have easy taring. Whatever type of balances you have, demonstrate their operation and warn students of common pitfalls: they are not error-proof! Students should weigh a few coins, to practice, and they should find that the mass of any two objects is equal to the sum of their masses when measured separately. (This provides a means for them to check their technique, and the equipment.)

Essentially, any reasonably pure liquids can be used as unknowns. Some instructors have used aqueous salt solutions. The density values obtained will vary somewhat from literature values, depending on the temperature and purity of the liquid, but they should be consistent. The precision possible is very high, with 0.2% a reasonable allowable error. Metal densities will also vary from literature values, and will be subject to errors of up to about 1%. Students get better precision if more metal is used: 50 g will usually fit in the pycnometer. The precision possible in this experiment, as written, is at least ten times that possible if graduated cylinders are used. There exists a lower limit on error, in that the mass of the air in the "empty" pycnometer is assumed to be zero, while it is actually (a negligible but non-zero) approximately thirty-two milligrams. Metal samples should be recovered, dried, and reused, since they are expensive. Recovery and reuse of the liquids is reasonable. The risk of contamination exists, but is not likely to cause problems in our experience.

Calculations made by students should generally be done on a hand calculator. Students should bring calculators to lab when calculations are required. The experiment will take about 1.5 hours. It can be sped up and the use of acetone avoided if the students make all dry measurements first; however, many students find this logically challenging when it comes to the calculations, hence the instructions as written. Some schools combine this experiment with Experiment 2, determining just the density of a metal. Another option is to have the students go through the Introduction to Excel in Appendix VII.

Densities in g/cm^3 at 20 °C

A	aluminum	2.70	I	ethanol, 95%	0.810
B	bismuth	9.81	II	2-propanol	0.786
C	chromium	7.15	III	*n*-heptane	0.684
D	cobalt	8.86	IV	cyclohexane	0.779
E	copper	8.96	V	toluene	0.867
F	nickel	8.91			
G	tin	7.29			
H	zinc	7.13			
I	titanium	4.51			
J	zirconium	6.49			

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Sample data and calculations:

The mass of a group of objects is equal to the sum of the masses of the objects when weighed separately.

Unknown liquid: ethanol Unknown solid: copper Ambient temperature: 22 °C

Determination of the density of an unknown liquid

mass of empty flask plus stopper	36.765 g
mass of stoppered flask plus water	65.264 g
mass of stoppered flask plus liquid	59.930 g
mass of water	28.499 g
density of water at 22 °C = ρ_{H_2O} (22 °C) [From Appendix I]	*0.997_7 g/cm^3
volume of flask = volume of water = mass of water / ρ_{H_2O} = V	28.5_6 cm^3
mass of liquid	23.165 g
density of liquid (ρ) = mass of liquid / V	0.811 g/cm^3

**We are allowed only three significant figures in the density of liquid water, and thus the volume of the flask. We are limited by the precision with which the ambient temperature is known [in this case, ± 1 °C, which implies an uncertainty in the density of* $\pm \frac{1}{2}(\rho_{21°C} - \rho_{23°C}) = \pm 0.0002$ g/cm^3 *and thus that the fourth significant figure in the density is not significant, being uncertain by more than one].*

Determination of the density of an unknown metal

mass of stoppered flask plus metal	55.415 g
mass of stoppered flask plus metal plus water	81.823 g
mass of metal	18.650 g
mass of water	26.408 g
volume of water = $\dfrac{26.408\ \text{g}}{0.998\ \text{g/cm}^3}$	26.5 cm^3
volume of metal = 28.6 cm^3 – 26.5 cm^3	2.1 cm^{3}L
density of metal = $\dfrac{18.650\ \text{g}}{2.1\ \text{cm}^3}$	8.9 g/cm^3

The error in the density of the metal will be larger than that in the liquid. The reason is that the volume of the metal is small and is the least accurately determined quantity in this experiment.

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Advance Study Assignment:

1. a. mass of water = mass of filled flask – mass of empty flask
= 68.090 g – 34.166 g = 33.924 g

b. volume of flask = mass of water / density of water = $\dfrac{33.924\ \text{g}}{0.997\ \text{g/cm}^3} = 34.0\ \text{cm}^3$

2. a. mass of liquid = mass of flask filled with liquid – mass of empty flask
= 57.418 g – 34.166 g = 23.252 g

b. density of liquid = mass of liquid / volume of flask = $\dfrac{23.252\ \text{g}}{34.0\ \text{cm}^3} = 0.683\ \text{g/cm}^3$

3. a. mass of metal = (mass of flask + metal) – mass of empty flask
= 306.150 g – 34.166 g = 271.984 g

b. mass of water = (mass of flask + metal + water) – (mass of flask + metal)
= 309.827 g – 116.150 g = 3.677 g

vol. of water = mass of water / density of water
= $\dfrac{3.677\ \text{g}}{0.997\ \text{g/cm}^3} = 3.69\ \text{cm}^3$

c. volume of metal = volume of flask – volume of water
= 34.0 cm^3 – 3.69 cm^3 = 30.3 cm^3

density of metal = mass of metal / volume of metal = $\dfrac{271.984\ \text{g}}{30.3\ \text{cm}^3} = 8.97\ \text{g/cm}^3$

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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 of 6 M HCl with 400 mL of ethanol and 400 mL of *n*-butanol (15 mL per 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 per student):
  - 0.10 M $AgNO_3$
  - 0.10 M $Co(NO_3)_2$
  - 0.10 M $Cu(NO_3)_2$
  - 0.10 M $Fe(NO_3)_3$
  - 0.10 M $Bi(NO_3)_3$

Preparation of unknowns:

Use two or three drops of the solutions listed below, in micro (10 × 75 mm) 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: 31¢

This experiment is quite short, taking about 2 hours, and involves more time for the students to wait for the liquid to move up the paper than is desirable. This experiment could readily be combined with another.

To reduce toxicity, we have substituted Bi^{3+} for Hg^{2+} as one of the metal ions to be studied. We have chosen an eluting solvent which we find gives good resolution. Since an aqueous staining solution is used to develop the spots, hood usage is not essential; however, wearing gloves is recommended, and the staining solution mist should not be inhaled.

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Sample data and calculations:

Ion present	Ag^+	Co^{2+}	Cu^{2+}	Fe^{3+}	Bi^{3+}
Color dry (no stain)	faint grey	aqua	yellow-green	yellow	colorless
Color after staining	light yellow	grey	reddish brown	deep blue	yellow-orange
Distance solvent moved (L) {mm}	79	80	79.5	81.5	82
Distance cation moved (D) {mm}	1	47	49	61	76
$R_f \equiv D / L$	0.01	0.59	0.62	0.75	0.93

The unknown will produce spots only for those components that are present.

Advance Study Assignment:

1. $R_f = \dfrac{\text{distance sample moves}}{\text{distance solvent moves}}$ The solvent front is located (pretty consistently, in this case) 80. mm from the point of application, so the denominator in this equation will consistently be 80. mm. The distance the sample moves is determined by measuring the distance between the point of application line and the center of each spot. For spots from left to right:

$$R_f = \frac{12.5\text{ mm}}{80.\text{ mm}} = 0.16 \qquad R_f = \frac{24\text{ mm}}{80.\text{ mm}} = 0.30 \qquad R_f = \frac{33\text{ mm}}{80.\text{ mm}} = 0.41$$

$$R_f = \frac{48.5\text{ mm}}{80.\text{ mm}} = 0.61 \qquad R_f = \frac{68.5\text{ mm}}{80.\text{ mm}} = 0.86$$

2. Obviously, each student's answer here should differ; but a good answer will mention both the mobile and the stationary phases, and something about components interacting with them differently. One example: "Different components partition their time in the mobile phase (dissolved in the solvent) and the stationary phase (in this experiment, attached to the paper) differently, so as the mobile phase moves the components move with it to differing extents."

3. Resolution improves as the distance the solvent front moves increases. Component spots / bands are far more likely to overlap if the solvent front moves only a short distance, and the relative uncertainty in distance measurements is also larger when the distances are small.

4. $10.\text{ microliters} \times \dfrac{1\text{ liter}}{10^6\text{ microliters}} \times \dfrac{10.8\text{ g } Ag^+}{1\text{ liter}} = 10_8 \times 10^{-6}\text{ g } Ag^+ = 110\text{ micrograms } Ag^+$

Note: 10_8 means 108 with the 1 and 0 significant and the subscripted 8 insignificant.

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EXPERIMENT 3

Resolution of Matter into Pure Substances, II. Fractional Crystallization

Special equipment needed:

- 20 Büchner funnels taking 70 mm filter paper, in #6 1-hole stoppers
- 20 250-mL filtering flasks
- 4 boxes 7-cm-diameter Whatman #1 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 pounds

Reagents needed in the laboratory:

- 4×200-mL dropping bottles 6 M HNO_3, nitric acid (2 mL per student)
- 4×200-mL dropping bottles 6 M NH_3, ammonia (6 mL per 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; mix well, then put the resulting standards in (identical) small (13×100 mm) test tubes, labeling these with the concentrations indicated below, and arranging these small tubes in a test tube rack; put 6.0 mL in each small 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	1.0	4	5
0.5	0.5	4.5	5
0.3	0.3	4.7	5
0.1	1.0 mL of 1$\%_{mass}$ sol'n	4	5
0.05	1.0 mL of 0.5$\%_{mass}$ sol'n	4	5
0.03	1.0 mL of 0.3$\%_{mass}$ sol'n	4	5
0.01	1.0 mL of 0.1$\%_{mass}$ 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, of 500 g or so. Mix thoroughly before dispensing, grinding the salts if necessary to keep the mixture homogenous.

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Suggested unknown compositions (for 500-g batches; subscripted zeroes are insignificant):

	SiC (~120 mesh/grit)	KNO_3 (purified grade)	$CuSO_4 \cdot 5\ H_2O$ (technical grade)
I	60. g	40_0 g	40. g
II	80. g	38_0 g	40. g
III	10_0 g	36_0 g	40. g
IV	12_0 g	34_0 g	40. g

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

Cost per student: 37¢

In this experiment we use SiC along with KNO_3 and $CuSO_4 \cdot 5\ H_2O$, with the copper sulfate present essentially as an impurity. The experiment, in this form, is indeed one on purification by fractional crystallization.

Samples should be well mixed and may require a bit of grinding in a mortar to obtain homogeneity. Recovery of the SiC should be essentially quantitative. Students should be able to recover roughly 50% of the KNO_3 after the second crystallization. Keep the purified KNO_3 and SiC for reuse.

The purity of the KNO_3 is assessed by comparing the color intensity of the $[Cu(NH_3)_4]^{2+}$ ion in a test solution against that of a standard. Students should pour (all of) their test solution from the 50-mL beaker in Part C into a small (13 × 100 mm) test tube. Then they should look down the test tube against a white background, comparing the color of their sample against that of a reference tube alongside it. There should be 6.0 mL of solution in each reference tube. There will be on the order of $1\%_{mass}$ $CuSO_4$ impurity after the first crystallization; after the second crystallization the amount of $CuSO_4$ may be undetectable. The purification will be most effective if the slurry to be filtered contains enough water to be stirred easily; the yield will be lower, but the product purity will be better.

This experiment requires about three hours to complete.

Composition of suggested unknowns:

	SiC	KNO_3	$CuSO_4 \cdot 5\ H_2O$
I	$12\%_{mass}$	$80\%_{mass}$	$8\%_{mass}$
II	$16\%_{mass}$	$76\%_{mass}$	$8\%_{mass}$
III	$20\%_{mass}$	$72\%_{mass}$	$8\%_{mass}$
IV	$24\%_{mass}$	$68\%_{mass}$	$8\%_{mass}$

Sample data and calculations:

	Original Sample
mass of 150-mL beaker	65.24 g
mass of sample plus beaker	86.69 g
mass of sample	21.45 g
mass of 50-mL beaker	29.91 g
mass of 50-mL beaker plus KNO_3	30.42 g
mass of KNO_3 used in analysis	0.51 g
$\%_{mass}$ $CuSO_4 \cdot 5\ H_2O$ present in KNO_3	$0.23\%_{mass}$
mass of 150-mL beaker plus KNO_3	79.06 g
mass of KNO_3 recovered	14.32 g

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	Recrystallized Sample
mass of filter paper	0.57 g
mass of filter paper plus purified KNO_3	12.41 g
mass of purified KNO_3 recovered	11.84 g
mass of 50-mL beaker plus purified KNO_3	30.41 g
mass of KNO_3 used in analysis	0.50 g
$\%_{mass}$ $CuSO_4 \cdot 5\ H_2O$ in purified KNO_3	$\approx 0\%_{mass}$

	Original Sample	Recrystallized Sample
mass of SiC plus filter paper	4.28 g	
mass of SiC in sample	3.71 g	
$\%_{mass}$ SiC in sample	$7.3\%_{mass}$	
$\%_{mass}$ sample recovered as KNO_3	$67\%_{mass}$	
percent by mass of sample recovered as pure KNO_3		$55\%_{mass}$

Advance Study Assignment:

1. a. From the graph, 180 grams of $CuSO_4 \cdot 5\ H_20$ will dissolve in 100 grams of water at 100 °C.

   b. 180 g $CuSO_4 \cdot 5\ H_2O$ =^= 100 g H_2O at 100°C (*See note on symbology, below)

   $$3.9\text{ g } CuSO_4 \cdot 5\ H_2O \times \frac{100\text{ g } H_2O}{180\text{ g } CuSO_4 \cdot 5\ H_2O} = 2.2\text{ g } H_2O \text{ required}$$

   c. From the graph, 240 grams of KNO_3 will dissolve in 100 grams of water at 100 °C

   $$37\text{ g } KNO_3 \times \frac{100\text{ g } H_2O}{240\text{ g } KNO_3} = 15\text{ g } H_2O \text{ required}$$

   d. 15 g H_2O would be required. That would dissolve *both* the KNO_3 and the copper sulfate. The KNO_3 in the solution does not appreciably affect the solubility of copper sulfate, or vice versa; the required volumes are *not* additive. (Water saturated with KNO_3 can still dissolve $CuSO_4$.)

2. a. The solution contains 27 g H_2O after the water is added. At 0 °C, the graph indicates that 10. g of KNO_3 will dissolve in 100 g of water. (The trailing decimal point on 10. indicates the trailing zero is significant, and not just a placeholder: 10. has two significant figures, while 10 has only one.)

   $$27\text{ g } H_2O \times \frac{10.\text{ g } KNO_3}{100\text{ g } H_2O} = 2.7\text{ g } KNO_3 \text{ in the solution.}$$

   b. We started with 37 g KNO_3, and about 2.7 g will remain in solution, meaning that about 34 g KNO_3 will crystallize out.

   c. At 0 °C, 20. g $CuSO_4 \cdot 5\ H_2O$ will dissolve in 100 g H_2O. We have 27 mL of water, so we expect up to

   $$27\text{ g } H_2O \times \frac{20.\text{ g } CuSO_4}{100\text{ g } H_2O} = 5.4\text{ g } CuSO_4 \cdot 5\ H_2O \text{ to remain in the solution.}$$

   Since there's a total of 3.9 g copper sulfate pentahydrate present, none of it will crystallize out.

   d. $\%_{mass}$ KNO_3 recovered $= \frac{34\text{ g recovered}}{37\text{ g present initially}} \times 100\% = 92\%_{mass}$ KNO_3 recovered

* We will occasionally use the symbol =^= to indicate an equivalence between one quantity and another. 180 $CuSO_4 \cdot 5\ H_2O$ and 100 g H_2O are equivalent in the sense that you can write a conversion factor from these quantities to find the mass of H_2O required to dissolve a given mass of $CuSO_4 \cdot 5\ H_2O$.

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EXPERIMENT 4

Determination of a Chemical Formula

Special equipment needed:

20 Büchner funnels taking 70 mm filter paper in #6 1-hole stoppers
(It would be preferable to use smaller Büchner funnels, taking a 42.4 mm diameter filter paper circle, #2 Whatman, but if they are not available, the larger funnels will work. The smaller ones are two-piece plastic, and are stocked by Sargent Welch and other supply houses.)

20 250-mL filter flasks

4 boxes Whatman #1 7 cm diameter filter paper circles

Several aspirators (safety flasks should be installed) or other vacuum source

4 Heating lamps (if you don't have four, probably two will be enough)

Reagents needed in the laboratory:

4×200-mL dropping bottles 6 M HCl, hydrochloric acid (1 mL per student)

4×200-mL dropping bottles 95% ethanol (10 mL per student)

20 gauge aluminum wire (20 cm, about 0.25 grams, per student)

4×100-g bottles $CuCl_2 \cdot 2\ H_2O$, copper chloride dihydrate: label as $Cu_xCl_y \cdot z\ H_2O$ (1 g / student)
This compound must be pure. The blue-green crystals should flow freely, and there should be no brown crystals present. The pure compound is stable in the stockroom for years (at least we have found that to be the case at Macalester). If you need to buy some, get A.C.S. purity, which is greater than $99\%_{mass}$, and in 2020 runs about $107 for 500 g.

Cost per student: 50¢

In this experiment the compound whose formula is to be determined is $CuCl_2 \cdot 2\ H_2O$. This compound is stoichiometrically clean, in that it does not tend to lose or absorb water, and can be obtained pure. It loses its water of hydration completely when heated to about 110 °C. The copper it contains is readily reduced to the metal by aluminum. Chlorine content is obtained by difference. The experiment works remarkably well.

The experiment is fairly short, and should take no more than two hours. Students should be able to complete the calculations while in the lab.

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Sample data and calculations:

molar masses: Cu = 63.55 g / mol Cl = 35.45 g / mol H = 1.01 g / mol O = 16.00 g / mol

mass of crucible	11.164 g
mass of crucible and hydrated sample	12.164 g
mass of hydrated sample	1.000 g
mass of crucible and dehydrated sample	11.950 g
mass of dehydrated sample	0.786 g
mass of filter paper	0.117 g
mass of filter paper and copper	0.480 g
mass of copper	0.363 g
moles of copper	5.71×10^{-3} moles
mass of water evolved	0.214 g
moles of water	1.19×10^{-3} moles
mass of chlorine in sample	0.423 g
moles of chlorine	1.19×10^{-3} moles
mole ratio, chlorine:copper in sample	2.08:1.00
mole ratio, water:copper in hydrated sample	2.08:1.00
formula of dehydrated sample	$CuCl_2$
formula of hydrated sample	$CuCl_2 \cdot 2\ H_2O$

Advance Study Assignment:

1. Molar mass values: Cu = 63.55 g / mol Cl = 35.453 g / mol H = 1.01 g / mol
   O = 16.00 g / mol H_2O = 18.02 g / mol CuO = 79.55 g / mol

2. a. moles of Cu = 0.8503 g / 63.55 g = 0.01338 moles
   b. grams of O in sample = 0.9573 g – 0.8503 g = 0.1070 g
   c. moles of O in sample = 0.1070 g / 16.00 g = 0.006688 moles
   d. mole ratio = moles Cu / moles O = 0.01338 mol Cu / 0.006688 mol O = 2.001:1
   e. formula of oxide = Cu_2O
   f. molar mass of the copper oxide = 2 × (63.55 g / mol) + 16.00 g / mol = 143.10 g / mol

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