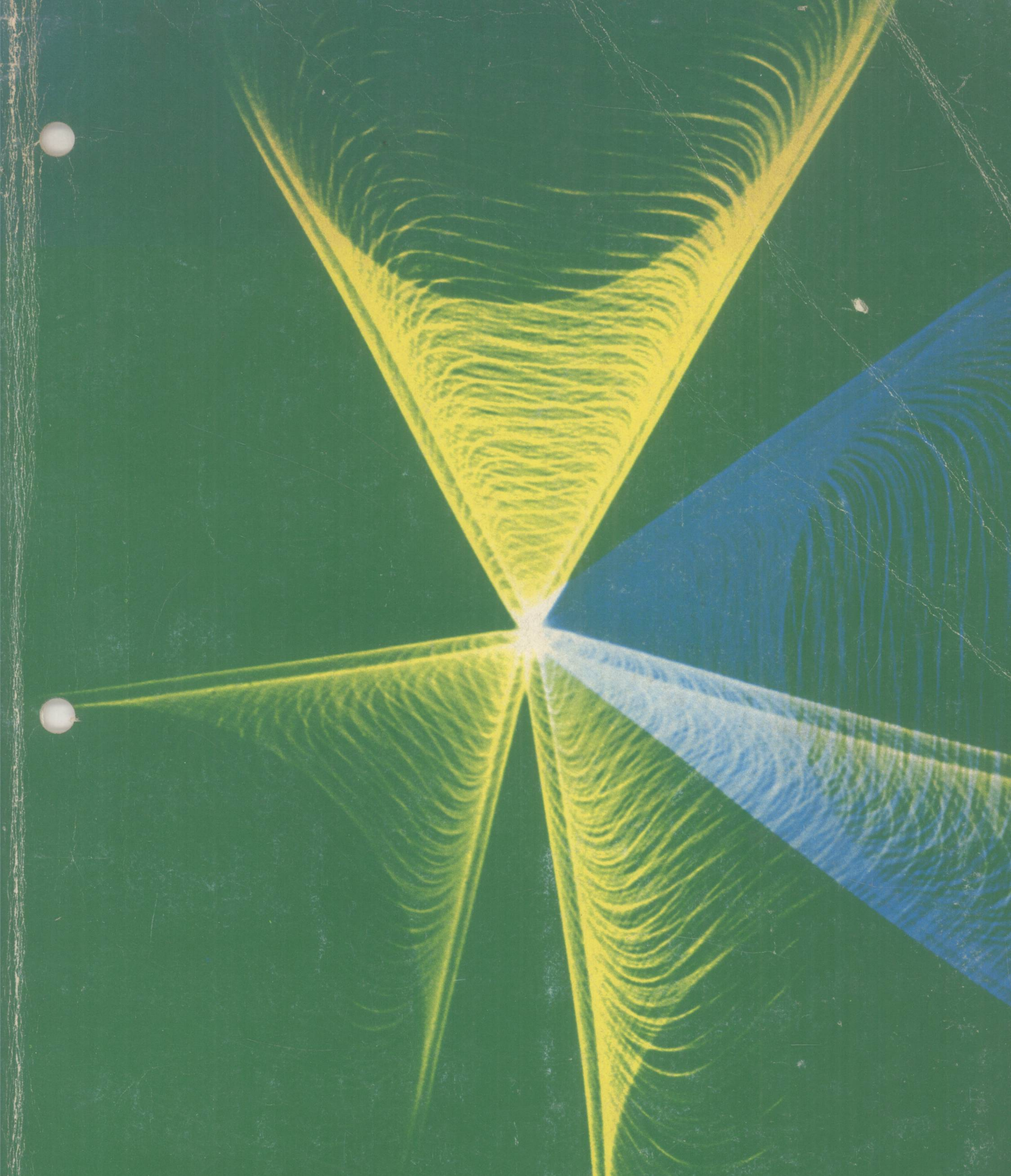

OURTH EDITION

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XPERIMENTS IN GENERAL CHEMISTRY

Experiments in GENERAL CHEMISTRY

FOURTH EDITION

[redacted]

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To The Instructor

An instructor's manual has been prepared as an aid for more effective use of *Experiments in General Chemistry*, Fourth Edition. It contains suggestions for the conduct of the experiment and a list of chemicals and equipment needed for each student. Directions are also provided for the preparation of solutions.

In this new edition of our laboratory manual, we have attempted to reflect the changes that have occurred in the teaching of general chemistry during the past few years. We have continued the emphasis on the more quantitative aspects of chemistry. Some of the more elementary work, present in the earlier editions, has been deleted to make room for more interesting material. At the same time, we hope that we have adhered to the aims of the first edition of our manual by providing experiments that are both relevant and interesting. Because of the wide diversity in facilities available in various institutions, we have attempted to incorporate as much flexibility as possible in the form of alternate procedures, which may require more or less sophisticated apparatus.

The experiments are designed to encourage and require the students to prepare for the laboratory period. "Prelaboratory Assignments" have been added to encourage such preparation. We have included a concise but complete discussion of the principles involved in each experiment and the objectives of that experiment. References to *Principles of Chemistry with Practical Perspectives* Second Edition, by R. S. Drago have been provided; key words have also been given to permit the students to consult other texts for the principles involved in the experiment. The prelaboratory assignments enable the instructor to evaluate both the students' understanding and their preparedness. We do not expect all of the experiments in this manual to be done in a year. With the exception of 30, 31, and 32, each experiment is independent of any other experiment.

Most of the questions at the end of the experiments require the students to *think* about what they have done in performing the work. Some of the less obvious, but nonetheless important, aspects of the results and procedures are emphasized. We hope to have made a worthwhile contribution in this respect.

We have introduced unknowns wherever it seemed feasible and have also written in the requirement that the students submit products to the instructor. These practices provide the instructor with a more positive check on the students' performance and with some quantitative basis for an assignment of grades.

The scheme of qualitative analysis contained in earlier editions has been shortened. The scheme still demonstrates the essential principles of chemical equilibrium, but the emphasis is no longer upon the detailed separations of individual ions. We have designed the qualitative analysis scheme and the selection of unknowns accordingly. The experiments immediately preceding the qualitative analysis work emphasize the essential principles; the students must then make use of these in *devising their own procedures of analysis.* With such a plan there is little opportunity or temptation for the students to "cookbook." The main point of the scheme is to teach logical deduction under conditions where careful attention must be paid and technique must be applied to the collection of the facts upon which these deductions are based. It is significant that second-semester chemistry students using this scheme have consistently rated this as one of the most interesting and enjoyable of the laboratory experiments.

We sincerely believe that we have written a good laboratory manual, but there is undoubtedly much room for improvement. We shall therefore be very appreciative of any suggestions for future improvements and of notice of any errors or omissions.

Russell S. Drago

Theodore L. Brown

To The Student

A. Safety and Laboratory Rules

1. Anyone attempting unauthorized experiments in the laboratory will be subject to severe disciplinary action.
2. When heating or carrying out a reaction in a test tube, never point this test tube at your neighbor or yourself.
3. Never taste a chemical unless directed to do so. When you are instructed to smell a chemical, do so by gently fanning the vapors toward your face.
4. When cutting glass tubing or inserting the tubing into a stopper, always protect your hand by using a towel. Failure to do this is the cause of most of the accidents that occur in introductory chemistry laboratories.
5. Never pour water into concentrated acid. Always pour the acid slowly into the water while stirring constantly.
6. If you receive a chemical burn by acid, alkali, bromine, or phosphorus, immediately flood the burned area with water. Then have another student summon the laboratory instructor. For all other accidents, immediately get help from the laboratory instructor.
7. If objectionable gases are given off during an experiment, the operations must be performed under a hood.
8. The chemical reagents employed are contained in labeled bottles. Read the label carefully before removing the material, for serious accidents can occur in a few instances if the wrong chemical is used. Reagents should be obtained from the reagent bottles in a clean beaker or test tube. Do not take the reagent bottle to your desk. Do not take any more material than is required, for many of the chemicals used in the laboratory are costly. Never return any unused material to the reagent bottle, and when you have finished with the bottle, return it to its proper place on the shelf.
9. Safety glasses or regular eyeglasses should be worn at all times in the laboratory.

B. General Procedures

1. The principles section for each experiment must be read prior to the laboratory period in which the experiment is to be performed. The Prelaboratory Assignments must also be completed. Quizzes on the material will occasionally be given in the laboratory during the course of the semester.
2. All data collected in the laboratory must be recorded directly in the manual. If you complete all the experimental work before the end of the laboratory period, you should complete as many necessary calculations as time permits before leaving the laboratory. The calculations should be carried out in the spaces provided.
3. The technique section of this manual contains detailed instructions on certain labo-

ratory manipulations. Reference will be made to this section in the procedure of many of the experiments. You should become familiar with this material and refer to it when necessary.

4. Paper, matches, or insoluble chemicals should be thrown into a laboratory crock or wastebasket provided for these materials. Do not throw them in the sink. Wash all liquids and soluble solids down the sink with plenty of water.
5. Whenever you are required to use water as a reagent in a chemical reaction, be sure to use distilled water.
6. At the end of each laboratory period, wash and wipe off your desk top. Be sure that the gas and water are turned off. *Return all special equipment to the stockroom.*

Contents

Topic and Approximate Time for Each Experiment (hours)

APPENDIXES

EXPERIMENT 1

The First Laboratory Period: Techniques for Accurate Measurement. Density of an Unknown Liquid

PRINCIPLES

In this experiment, our goal is to increase your proficiency in measurement both from the standpoint of technique and expression. You are familiar with measurement in your daily life—you probably know your height, weight, and how much beer you can consume before feeling it. In the chemical laboratory, the differences are that (1) quantities need to be measured more accurately, and (2) the metric system is employed. Accordingly, if you have not yet done so, read the sections in your textbook on conversion from the English to metric systems and on significant figures and exponents. In *Principles of Chemistry with Practical Perspectives*, Second Edition, by R. S. Drago (which will be abbreviated PCPP-2), read pp. 11 to 14. (For other texts, check the index.) Then carry out the following prelaboratory assignment.

Prelaboratory Assignment

1. How many significant figures are there in

   (a) 8.0025 ________ (c) 0.007 ________

   (b) 1.01 ________ (d) 0.0761 ________

2. Given that 1.00 lb = 454 g, 1.00 in = 2.54 cm, and 1.00 qt = 0.946 liters, convert

   (a) 3.00 inches to cm.

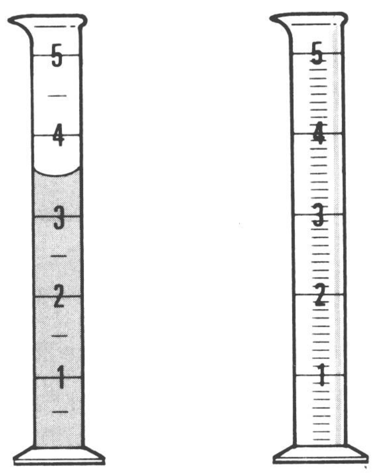
(b) 70 g to lb.

(c) 1.00 liter to quarts.

In the metric system, the prefix micro indicates 10^{-6}, milli 10^{-3}, centi 10^{-2}, deci 10^{-1} and kilo 10^{3}. These prefixes are combined with meters, grams and liters.

Another important point to make regards the accuracy of the measurement. Figure 1-1 shows two graduated cylinders calibrated differently. If you had the graduated cylinder shown in Figure 1-1(a), the liquid level could be estimated to ±0.1 ml. In reading a liquid level, one always reads from the bottom of the meniscus, that is, from the bottom of the curved liquid surface. If you placed exactly the same amount of water into a cylinder six different times, you might very well read this as 3.4, 3.5, 3.4, 3.6, 3.5, 3.6. The figure is recorded to the first decimal point even though this is estimated. Since it is known that all six trials used the same amount of liquid, the average value would be 3.5 and the average *deviation* 0.06. Average deviation is obtained by summing the absolute value (that is, no regard is paid to sign) of the difference between the average and the measured value and dividing by the number of measurements. We would say that our *precision* in carrying out this measurement is 0.06 and the figure is precise to 3.5 ± 0.06. Precision is an indication of the reproducibility of our measurement with particular equipment. It should be emphasized that in the above measurements we do not know the amount of liquid *accurately* to 3.5 ± 0.06 ml. Just because a cylinder is marked 1, 2, 3, 4, 5, . . . , does not mean this is an accurate measure. For example, maybe the tubing used to make the cylinder could have been too narrow or its width may have been uneven over the length of the cylinder. We have stipulated that all six cylinders contain the same amount of water, but the accurate volume can only be ascertained by comparison to an accurate measure.

FIGURE 1-1



(a) (b)

It is relatively easy to obtain the mass of an object both precisely and accurately. You can assume the analytical balance or weights in your laboratory are calibrated to produce accuracy to the degree indicated by their scales. Thus, you could weigh the amount of water in the cylinder accurately. Let us assume it weighs 3.91 grams. Table 1-1 lists the density of water at different temperatures. Density is defined as the mass per unit volume, in this case, grams per milliliter and, as can be seen in Table 1-1, this quantity has been very accurately determined for water. If the measurements described above were carried out at 25 °C, we would know our cylinder was capable of accurately delivering

$$3.91 \text{ g} \times \frac{1 \text{ ml}}{0.997 \text{ g}} = 3.92 \text{ ml}$$

The cylinder is still wet, so it contained more than 3.91 g, but the point of interest to us is that it is capable of delivering 3.91 grams or 3.92 ml. Thus, if we fill our cylinder to 3.5 ± 0.06 ml, we can obtain 3.9 ± 0.06 ml. However, if we fill our cylinder to 2.5 ml, we do not know that it will deliver 2.9 ml because, for example, the width of the cylinder could be irregular and our calibration at 3.5 would not pertain at 2.5. For accurate work, we would use a pipet, which delivers the same fixed volume every time. We shall perform this calibration in this experiment. For very accurate work, the pipet would be calibrated. In actual practice, the situation is not as bad as described above. A graduated cylinder for delivering up to five ml would have markings for 0.1 ml as shown in Fig. 1-1(b) and the volume could be estimated

TABLE 1-1
Density of Water (g/ml) as a Function of Temperature

°C	g/ml[a]	°C	g/ml[a]
0	0.999841	15	099
1	900	16	0.998943
2	941	17	774
3	965	18	595
4	973	19	405
5	965	20	203
6	941	21	0.997992
7	902	22	770
8	849	23	538
9	781	24	296
10	700	25	044
11	605	26	0.996783
12	498	27	512
13	377	28	232
14	244	29	0.995944
		30	646

[a]Note that the first four digits in this column are not repeated if they are the same as the entry above.

to ± 0.02 ml. Two figures to the right of the decimal point would be recorded. Manufacturers produce quite reproducible and accurate equipment.

The errors that we have been discussing can be placed in two general categories: (1) *systematic errors* and (2) *random errors*. Systematic errors are constant ones introduced into every measurement. They can arise by having a measuring device with an improperly calibrated scale. An investigator may also introduce a systematic error, for example, by consistently misreading a scale high or low. The conditions under which an experiment is conducted may also lead to systematic errors.

Random errors can be introduced by human errors or a rapid fluctuation of some experimental condition. Random errors have an equal probability of being too high or too low. As a result, if enough measurements are taken, they will average toward zero. We can count on a graduated cylinder being accurate to 4%, and, if this is all that is needed, this equipment suffices.

In view of your past experiences in measuring volumes such as a cup of cooking oil or a shot (1 oz) of whiskey, would you have appreciated how involved an accurate measure of volume could be? This kind of thinking about what you are doing in the course of making measurements is essential to good laboratory technique.

In this experiment, you will calibrate a pipet and you will then use this calibrated pipet to determine the density of an unknown solution of ethylene glycol ($(HOCH_2)CH_2OH$) in water. This solution is similar to the one in the radiator of your automobile cooling system, if you have one. Since the density depends upon how much ethylene glycol is dissolved and since the freezing point of the solution depends upon how much is dissolved, a service station attendant can tell you at what temperature your radiator will freeze by roughly measuring the density of the radiator contents. The density depends upon the temperature, so the same solution will have a different density at different temperatures. In order to determine how much ethylene glycol is dissolved (remember this determines where it freezes), the temperature must be known. You have to run your car for a while before having the antifreeze checked, because the service station density-freezing point conversion charts are for a hot solution.

Prelaboratory Assignment (cont.)

3. At 30 °C, a sample of water is found to weigh 5.98 g. What is its volume? (See Table 1-1 for its density.)

4. An empty beaker weighs 33.492 g. Water is added at 25 °C and the water plus beaker weigh 38.512 g. The water is poured out and the beaker weighs 33.921 g.

   (a) What is the weight of water added?

(b) What is the weight of water delivered?

(c) What is the volume of water delivered?

A. CALIBRATION OF THE PIPET

PROCEDURE

Add 50 ml of water to a 100-ml beaker and record its temperature. Accurately weigh a clean, dry, empty 50-ml beaker. (Appendix A, which covers laboratory techniques, should be consulted for directions on the use of an analytical balance.) Pipet in 10 ml of water and weigh. (Appendix A should be consulted for directions on the use of a pipet.) Pour out the water, dry the beaker thoroughly, pipet in 10 more ml, and weigh again. Now, pour out the water, dry the beaker, and weigh again.

DATA

Temperature of water __________

Weight of dry beaker __________

Weight of beaker plus water __________

Weight of water I __________

Weight of dry beaker __________

Weight of beaker plus water __________

Weight of water II __________

If the weight of water in the above two experiments does not agree within 0.05% or less, weigh 10 ml more. The percent agreement is obtained by dividing the difference between the two weights by the average weight and multiplying by 100.

Weight of dried beaker __________

Weight of beaker plus water __________

Weight of water III __________

CALCULATIONS

Obtain the density of water from Table 1-1 and calculate the volume of water delivered by your pipet.

B. DENSITY OF AN UNKNOWN LIQUID

PROCEDURE

In this experiment, you will determine the density of an unknown mixture of ethylene glycol and water. You should now be in a position to devise a procedure for determining the density of this liquid. If so, obtain an unknown sample and proceed to do so, using the same 10-ml pipet you used in part A. If you cannot devise a procedure, see your instructor for detailed instruction. If you need help, your top score will be reduced. *Hint*: Before measuring your 10-ml volume of unknown with the pipet, be sure to rinse it with a little bit of the unknown solution. Make two separate determinations and, if they do not agree, make a third. Write your procedure in the space below.

Before leaving the laboratory, obtain the volume of liquid delivered by the pipets of six other students and their standard deviations. Record below.

DATA

CALCULATIONS

Calculate the density of the unknown liquid.

Be sure you have recorded the results for pipet calibration from six other students before leaving.

REFLECTIONS

1. Assume the correct volume of your calibrated pipet was 1.00 ml *less* than the value you found.

   (a) Would the density of your unknown liquid be too high, too low, or not affected?

   (b) Recalculate your unknown density using a volume 1.00 ml larger than your calibrated value. What is the percentage difference of this result compared to your measured value?

2. Suppose in the course of determining the density of your unknown liquid that the inside of the pipet was wet with water from the calibration experiment.

   (a) Would this lead to a density that differs from the true value? If yes, would it be lower or higher than the correct value?

   (b) How would you avoid the problems in (a) without waiting for the pipet to dry?

3. A student came to laboratory late and borrowed a neighbor's calibrated pipet to determine his unknown's density. The punctual student allowed her pipet to drain properly in calibrating it, but the tardy one blew the last little bit out of the end to empty the pipet. Would his unknown result be too low, too high, or not affected by this poor technique?

4. Tabulate the values for the volume of liquid delivered by the pipets of six other students and tabulate the students' standard deviations.

   (a) What is the average value of the volumes delivered?

   (b) Do any of the results differ from the average by more than the precision? ________
   What do you conclude about the company's ability to make pipets (in this price range) that accurately deliver 10.000 ml?