A Laboratory Investigation of CONCEPTS IN CHEMISTRY

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A LABORATORY INVESTIGATION OF CONCEPTS IN CHEMISTRY

PREFACE

Although various laboratory manuals have been published recently that emphasize an open-ended "problem" approach, they are suitable only for small laboratory classes. We strongly felt the need for a laboratory manual that could be used in classes as large as thirty students, and yet that would be stimulating, provocative, and that would give insight into the meaning of ideas in chemistry.

The goals of our manual, and method of carrying out these goals are as follows:

1. We have attempted to prepare the student for an understanding of the concepts involved in a particular experiment by prefacing each experiment with a series of leading questions that he must answer prior to carrying out the experiment. These questions are assigned the week before the experiment is to be done. They are handed in at the beginning of the first laboratory period for which the experiment has been assigned. Although the student is therefore made aware of the idea involved, the preliminary questions are carefully formulated so that they avoid giving the student too much information on the problem he is preparing to investigate.

2. Each experiment is preceded by a brief historical discussion of the evolution of the ideas relating to the experiment. This analysis of how the concepts developed, and what individuals contributed to their development, emphasizes chemistry as an evolving, dynamic, changing field of knowledge which owes its growth to the efforts of many people from many countries. The usual authoritarian approach of teaching chemistry as a "crystallized, authoritative, anonymous" body of knowledge, as Bentley Glass wrote about science in general, is so demolished. Chemistry in the laboratory becomes a study of ideas and their genesis in the physical world. This structure of ideas is seen as the product of the minds of individuals, rather than as an area of knowledge completely cut off from its relationship to human beings.

3. The experiments are chosen so that they illustrate the significant concepts in chemistry. They include experiments in physical chemistry (e.g., solid-state problems, chemical equilibria, and reaction rates), quantitative analysis, semimicro qualitative analysis, and inorganic preparations which emphasize some specialized techniques. To stress the "problem" approach, most experiments include the use of "unknowns" and quantitative determinations. To encourage the student into becoming an active participant in the laboratory experience, only the minimum information and instructions are supplied. The order of experiments is mostly sequential because we feel that this illustrates the cumulative nature of science (although some experiments may be omitted without losing the trend of the developing ideas). This approach has the additional advantage of stimulating the student to use ideas, concepts, information, and techniques of previous experiments, instead of routinely following instructions which have little relation to previous laboratory experiences.

4. The experiments are followed by a series of concluding questions the student answers after he has completed each experiment. Their purpose is to further aid the student in extracting from the apparent chaos of the laboratory experience the most

significant ideas that can be inferred by carrying out a particular experiment. The questions emphasize these points: What inference is one justified in making based on the results of the experiment? Can a valid hypothesis be formulated on the basis of the experiment? What are the limitations of the particular experiment? How precise are the measurements? What is the relationship between this experiment and various concepts accepted in science?

5. Quadrilled pages have been inserted in this manual to enable the student to keep a complete and permanent record of the data he accumulates as he carries out his experiments. The student is expected to insert a carbon and blank copy sheet between each page, and to give the copy of his work to his instructor. The use of the manual according to this method serves several purposes: It introduces to the student the professional method of keeping a permanent record of his laboratory data; it discourages "dry-labbing," for the instructor has a copy of each student's record; duplicate page numbering makes available to the instructor a complete record; the copy may possibly serve as a substitute for a laboratory report for the instructor who feels the writing of a more formal report is unnecessary; the use of quadrilled paper encourages the recording of data in a tabular form and enables the student to include graphs as part of his permanent record.

The preliminary questions, the historical discussion, the experiment itself, and the questions at the end of each experiment point up that fruitful concepts and theories in chemistry are based upon a solid foundation of study of the physical world. Too often, chemistry courses introduce concepts that are never justified. In the laboratory there exists a superb opportunity to illustrate the interplay between theory and experimentation. The aim of this manual is to emphasize this relationship.

RULES FOR THE LABORATORY

1. Work in the laboratory <u>only with supervision</u>.
2. Perform unassigned or unlisted experiments <u>only with written approval</u> of your instructor.
3. Never remove chemicals or supplies from the laboratory.
4. Wear safety or prescription glasses at all times.

RULES TO BE OBSERVED IN THE LABORATORY

1. Report all accidents and injuries, however minor, to the instructor.

2. If any liquid reagents are spilled, flood the area with water immediately.

   a. For acid burns: Saturated sodium hydrogen carbonate ($NaHCO_3$) solution can also be used and is at the main sink.

   b. For alkali burns: Saturated boric acid (H_3BO_3) solution can also be used and is also at the main sink.

3. When inserting glass tubing into a rubber stopper:

   a. The glass tubing must be fire-polished.

   b. Wrap your hands in a towel, and hold the glass near the stopper.

   c. Do not force the glass into the hole. Use a gentle twisting motion, and a lubricant: e.g., water or glycerine.

4. Do not taste any chemicals unless so directed. Should you accidentally imbibe any reagent, rinse your mouth with water and report to the instructor immediately.

5. When examining the odor of a reagent or reaction, do not put your head directly over the container. Use your hand to fan the odors in the direction of your nose and inhale carefully.

6. If poisonous or noxious gases are going to be evolved from your experiment, perform the experiment in the hood.

7. When diluting concentrated acids, be sure to add the acid to water, while stirring the mixture.

8. Wear safety glasses or prescription glasses at all times while you are in the laboratory. A lab coat or apron will protect your clothing.

9. For disposal of waste material

   a. Put all paper, matches, and broken glass into the wastebasket.

   b. Put soluble solids in the sink, dissolve them with a large quantity of water from the tap, and wash them down the drain.

   c. Pour acid bases and other liquids down the drain with much water.

d. Put insoluble solids in specially assigned jars.

e. For special waste problems, your instructor will give directions.

10. For reagents:

a. Do not remove reagents from the shelf at any time.

b. To obtain chemicals, take a watch glass or beaker to the reagent shelf with you.

c. Take only what you need.

d. Do not place corks or stoppers down on the reagent bench. Hold in your hand as shown by the instructor.

e. Do not return reagents to the stock bottles at any time.

f. Do not insert pipets or droppers into reagents.

g. Read all labels twice, once before and once after taking the chemical.

h. Be sure the bottle of reagent has the cork or cover replaced when you finish using it. Reagents must not be left standing uncovered.

11. For glassware:

a. All glass tubing must be fire-polished before use.

b. Do not heat graduated cylinders or glass bottles at any time.

12. Always use a glass or porcelain container for weighing any substance on the balance.

13. Keep books and papers away from working areas to avoid damage.

14. Anticipate danger. Know the location of the fire extinguisher and the emergency shower.

15. Before leaving the laboratory, wash your desk and wipe it dry.

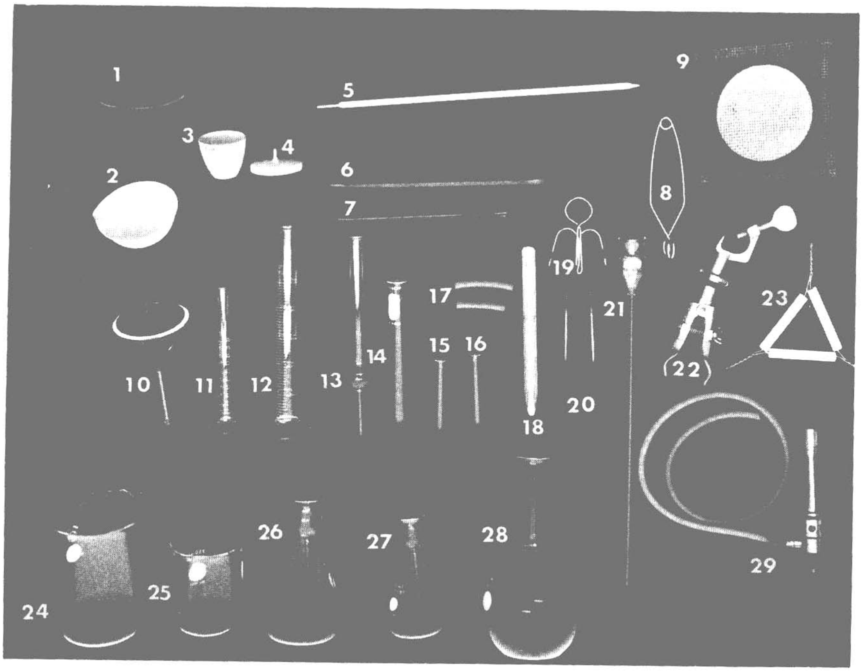
APPARATUS

All apparatus in assigned drawer and cabinet must be inspected by the instructor and student at the beginning and at the end of the semester. Any apparatus not acceptable must be replaced from the stockroom. No apparatus will be accepted by the stockroom unless it is clean and dry.

Figure A Number	Item	Quantity
24,25	beaker, 150 ml	1
24,25	beaker, 250 ml	1
24,25	beaker, 400 ml	1
29	Bunsen burner	1
19	clamp, pinch type	1
3, 4	crucible and cover (small)	1
13	drying tube ($CaCl_2$)	1
2	evaporating dish	1
27	flask, Erlenmeyer, 125 ml	1
26	flask, Erlenmeyer, 250 ml	1
28	flask Florence, 500-ml	1
10	funnel, short stem	1
11	graduated cylinder, 10 ml	1
12	graduated cylinder, 50 ml	1
18	scoopula	1
15,16	test tubes, 8 cm	6
14	test tubes, 15 cm	2
8	test tube holder	1
23	triangle, clay	1
5	thermometer, 110°C	1
22	utility clamp	1
1	watch glass	1
	wing top or flame spreader for burner	1

Nonreturnable (unless in good condition)

9	asbestos centered wire gauze	1
6	file, triangular	1
20	medicine dropper	2
17	rubber connectors	2
	towel	1

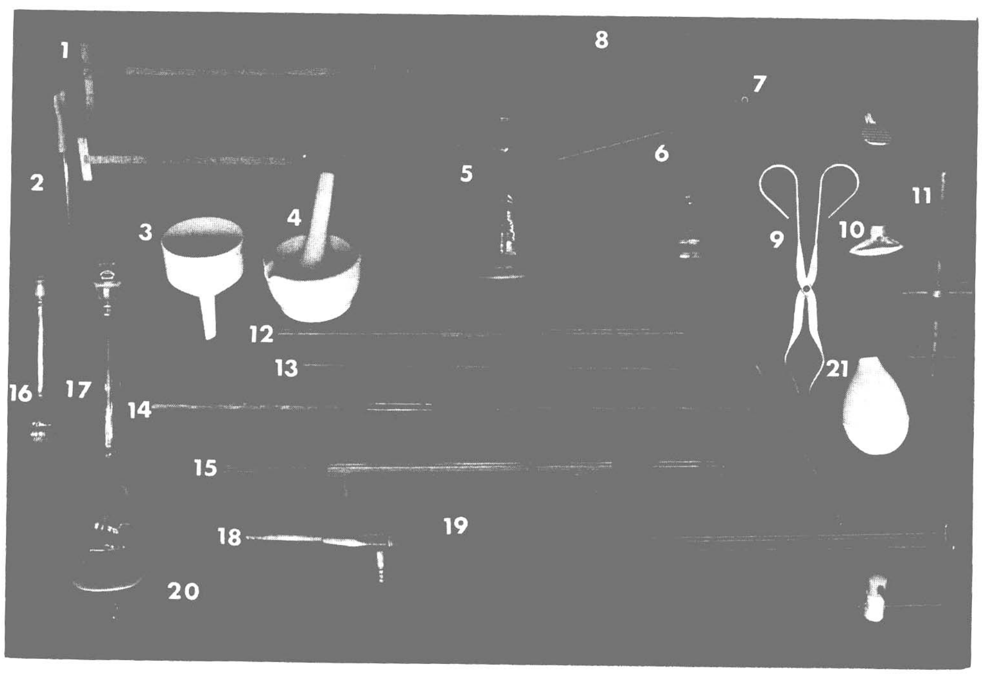


Apparatus in Drawer (Figure A)

1. Watch glass
2. Evaporating dish
3. Crucible
4. Crucible cover
5. Thermometer
6. Triangular file
7. Stirring rod
8. Test tube holder
9. Asbestos wire gauze
10. Funnel, short stem
11. 10-ml graduated cylinder
12. 50-ml graduated cylinder
13. Drying tube
14. 15-cm test tube
15.–16. } 8-cm test tubes
17. Rubber connectors
18. Scoopula
19. Pinch clamp
20. Medicine droppers
21. Thistle tube
22. Utility clamp
23. Clay triangle
24.–25. } Beakers
26.–27. } Erlenmeyer flasks
28. Florence flask
29. Bunsen burner with rubber tubing

Additional Equipment (Figure B)

1. Test tube rack
2. Glass rod with rubber policeman
3. Buchner funnel
4. Mortar and pestle
5. Suction flask
6. Distillation flask
7. Buret clamp
8. Test tube brush
9. Crucible tongs
10. Flame spreader or wing top
11. Cork borers
12.–14. { Different-sized pipets
15. Condenser
16.–17. { Volumetric flasks
18. Aspirator
19. Fractionating column
20. Buret, 50 ml
21. Rubber bulb



THE NOTEBOOK

For most of you experience in the laboratory has been limited. Consequently, detailed directions are given here for keeping your laboratory notebook and writing your reports. The form of these items is similar to that used by professional chemists for recording and reporting research projects. The purpose of the laboratory notebook is to maintain a record of data, observations, information, speculation, and ideas relative to your experiment. These are recorded as you work and consequently may have no special sequence. The purpose of the experiment and its theory, and conclusions that can be drawn from the data, are all entered into the record.

The blank pages at the end of this laboratory manual have been inserted for use as your notebook. This manual, therefore, is a permanent record of both the laboratory instructions and your data. The perforations and duplicate numbered pages allow a copy of your data to be torn from your book to give to your instructor.

Form:

1. Number each page in the upper right-hand corner.

2. Enter your name, date, and your section.

3. Record the title of the project on every page.

4. Use only one side of the page.

5. Record all data in ink while you are performing the experiment. Be sure to make a carbon copy simultaneously. This copy will be handed in to the instructor at the end of each laboratory period.

6. Begin recording observations or data at the top of the page, leave minimal margins, and do not skip lines. Do not remove pages from your notebook.

7. Errors are deleted by drawing a single line through them. Do not blot out errors or erase them. These so-called errors can sometimes provide vital information. When data or observations are crossed out, enter your reasons for questioning their reliability in your notebook.

8. All your data and observations must be entered clearly and legibly so that someone else, who has no specific knowledge of the experiment, can easily understand your work. Include labeled sketches of apparatus if this will help to clarify your work. Precision and accuracy are of great importance in any scientific pursuit. Answer all questions, including those which appear in the preliminary questions and experiment directions.

9. Record all methods of calculation but do not include long arithmetic procedures. Significant figures are to be considered in all your computations involving experimentally measured numbers.

10. The laboratory notebook is also an excellent place to record information from a reference source. In this case you should also make a note of the title and author of the source.

THE REPORT

Should your instructor require a report, the following outline may be used:

Form:

1. Title of the experiment.
2. Dates when the experiment began and when it was completed.
3. Date the report was submitted.
4. Theory.
5. Brief, concise description of the experimental procedure. The apparatus may be described by a labeled diagram.
6. Data and observations.
7. Interpretation of the data and the calculations.
8. Summary and conclusions.
9. Constructive criticism about the experiment.

Reports are evaluated on the basis of the understanding you have shown of the principles involved, the accuracy of your work, and the clarity and brevity of presentation.

Reference: W. J. Gensler and K. D. Gensler, Writing Guide for Chemists, McGraw-Hill Book Company, Inc., New York, 1961.

EXPERIMENTAL LIMITATIONS

Accuracy and Precision

In the study of science it is important to have an exact understanding of the meaning and use of words in that science. The definitions of words in science are specific. This is true of the words "accuracy" and "precision."

The word accuracy is used to describe the closeness that a value may have to some standard. The standard may have an arbitrary value, as does the standard kilogram in Paris. At the National Bureau of Standards in Washington, D.C., there is an excellent reproduction of the standard kilogram, but it is not an exact copy. If the value of the standard is not arbitrary, it is derived from empirical information, obtained from many experiments, and is an accepted value. It, unlike the value for the standard kilogram, which cannot change unless the definition of the standard changes, may change as more experimental values accumulate.

The word precision is used to evaluate measurement. For example, a notebook page is measured with a meter stick marked off in centimeter units and found to be 28.7 cm long. Since there is no tenths subdivision, the decimal position is an approximation. Repeated measurements of the same page reveal that the decimal position may vary by ±0.1 cm. The precision of these measurements with this ruler are limited by this amount. If the ruler is made of some material which can be distorted, as a metal ruler would be with temperature changes, it might be possible to observe marked deviations of readings in consecutive measurements. This would bring the precision of the instrument into doubt.

Values in very close agreement, however, do not necessarily lead to greater accuracy or more reliable values. The calibration of the ruler may be in error or the ruler may not be a good copy of the accepted standard.

Assuming that the instrument (ruler) is both a good copy of the standard and has been correctly calibrated, would subdividing the centimeter divisions into millimeters increase the accuracy of the values obtained with it; would the values be more precise? Assume that the arbitrary value for the book page has been accepted as 27.7 cm. The readings with this more sophisticated ruler now give values of 27.70, 27.72, and 27.71 cm. Because the accepted value was limited to the nearest tenth of a centimeter, readings to the nearest hundredth of a centimeter do not lead to a more accurate value. As a matter of fact, the value in the hundredth place in this case is misleading, because it implies a known value in the hundredth place and consequently gives it physical meaning. The measurements, however, appear to be more precise.

To sum up, precise measurements depend upon the reliability of the instrument used, the agreement of the values for several careful measurements, and the physical significance of the figures. Accuracy refers to the degree to which measurements approach a most probable or accepted value. It is limited by the sum of the errors inherent in the measurements.

Experimental Error and Percent Error

Error can be defined as the difference between experimentally measured values and the accepted measured value. To refer to the previous example, if the accepted value for the length of the book page is 27.6 cm and the average value of your measurements is 27.7 cm, the error is 0.1 cm. The percent error is calculated as the error, divided by the accepted value, multiplied by 100:

$$\frac{0.1}{27.6} \times 100 = 0.4\% \text{ error}$$

It can be reasoned from the foregoing that the type of instrument used for any particular measurement has its own limiting precision. The choice of instrument to be used is based upon the degree of accuracy required under a given set of circumstances. For example, it is not necessary to know to the pint what the capacity of your car's gasoline tank is because the chances are the fuel pump cannot make use of that last pint. So a device that reads E when you are on that last gallon is a sufficiently accurate measurement. On the other hand, you may want to know to the milligram just how much aspirin you are taking. The following is a list of measuring devices found in your laboratory drawer and the precision of measurement for each one. Study the table carefully so that you will be aware of the degree of precision you can expect when you perform your experiments.

Instrument	Precision of measured value	Range
Platform balance	±0.25 g	0.5 g
Triple beam balance	±0.01 g	0.02 g
Analytical balance	±0.0005 g	0.001 g
50-ml buret	±0.01 ml	0.02 ml
25-ml transfer pipet	±0.01 ml	0.02 ml
5-ml transfer pipet	±0.01 ml	0.02 ml
250-ml volumetric flask	±0.05 ml	0.10 ml
50-ml graduated cylinder	±0.1 ml	0.2 ml
10-ml graduated cylinder	±0.05 ml	0.1 ml
110°C thermometer	±0.1°C	0.2°C
Mercury barometer	±0.25 mm	0.5 mm

You can expect error in measurement from two sources. One comes from the limiting precision of your instruments, that is, the sensitivity of the instruments. The second source of error comes from inaccurately adjusted instruments, which consequently give incorrect values. The second source can give precise but inaccurate results. (Operator errors do not fall into this category because they result from careless or improper use of instruments.)

Significant Figures

A necessary corollary to the concept of precise measurement and accurate results is the concept of significant figures. Only those figures should be reported which you know to be reliable values. For example, if a stray dog comes to live at your house, you can estimate from his size and type that he may be 1 year old. But you certainly could not say with any degree of certainty that the dog was 11 months, 3 days old. This statement would be false. If you weigh a sample on a platform balance, the weight can be reported as 25.5 grams, but not as 25.5632 g. Significant figures of any measurement are those known with certainty plus the last number, which is always an estimate. The weight of the sample on the platform balance may vary with succeeding weighings as follows: 25.5 g, 25.7 g, 25.3 g. The last place can vary by as much as 0.5 g and is only approximately known.

Similarly, in performing any arithmetic calculations, only those figures are retained which are significant. The least precise measurement determines the number of figures to be used.

Examples: Addition:

$$\begin{array}{r l} 49.6 & g \\ 3210. & g \\ \underline{0.496} & g \\ 3260.096 & g \end{array} = 3260 \text{ g} = 4 \text{ significant figures}$$

Subtraction:

$$\begin{array}{r l} 32.9 & g \\ \underline{0.0496} & g \\ 32.8504 & g \end{array} = 32.9 \text{ g} = 3 \text{ significant figures}$$

$$\begin{array}{r l} 83.958 & g \\ \underline{83.616} & g \\ 0.342 & g \end{array} = 0.342 \text{ g} = 3 \text{ significant figures}$$

Multiplication and division:

$$\frac{21.2 \text{ g}}{35.457 \text{ g}} = 0.596 \text{ g} = 3 \text{ significant figures}$$

$$\begin{array}{r l} 1.6 & g \\ \times\ \underline{0.46} & g \\ 0.696 & g \end{array} = 0.70 \text{ g} = 2 \text{ significant figures}$$

For multiplication and division problems, one further test is made to help evaluate the significance of the last figure. For example:

$$\frac{1.01\ g}{0.89\ g} = 1.134\ g$$

In this case the number 101 varies by 1 part in 101, and the number 89 varies by 1 part in 89. Therefore, the number of significant figures in the answer will be limited by this. If the answer has only two significant figures, the variation is 1 part in 11; if three significant figures, 1 part in 113; and if four significant figures, 1 part in 1134. It is obvious that three figures give a more accurate picture of the limitations of these particular measurements.

Zeros may be significant or not. One of the answers in the examples above is written as 0.70. The zero to the left of the decimal point, which locates the decimal point, is not significant. The zero to the right of the decimal point is significant. It indicates the reliability of the measurement.

To avoid confusion in reporting zeros and their significance, it is often convenient to express numerical values in exponential form. In this way the above number can be reported as 7.0×10^{-1}. Or, for instance, when very large or very small numbers are reported, the exponential form becomes necessary. Avogadro's number is reported as 6.023×10^{23} (four significant figures). You will find almost all your arithmetic manipulations much simpler if you express numbers in exponential form whenever possible and then perform the computations on a slide rule. If more than three significant figures apply, use logs.

Measurements are expressed by a number and its unit. Never separate the numerical value from its dimension. The units are treated exactly as the number and remain with the number through all algebraic operations. For example, to obtain the volume of a cube 2 cm on a side, write: $2\ cm \times 2\ cm \times 2\ cm = 8\ cm^3$.

Solve the following problems. Use the proper number of significant figures and carry the units of measurement along as you solve the problem.

1. Calculate the volume of a box 1 ft by 6.5 cm by 1 3/8 in. (answer in cm^3).

2. Three different types of volumetric equipment were calibrated using water at 25°C. If the density of water at 25°C is 0.997 g/ml, how would you report the number of grams of water contained in each of the following when filled to the maximum mark?

   a. 500-ml graduated cylinder weighed on a platform balance.

   b. 100-ml graduated cylinder weighed on a triple beam.

   c. 50-ml volumetric flask on an analytical balance.