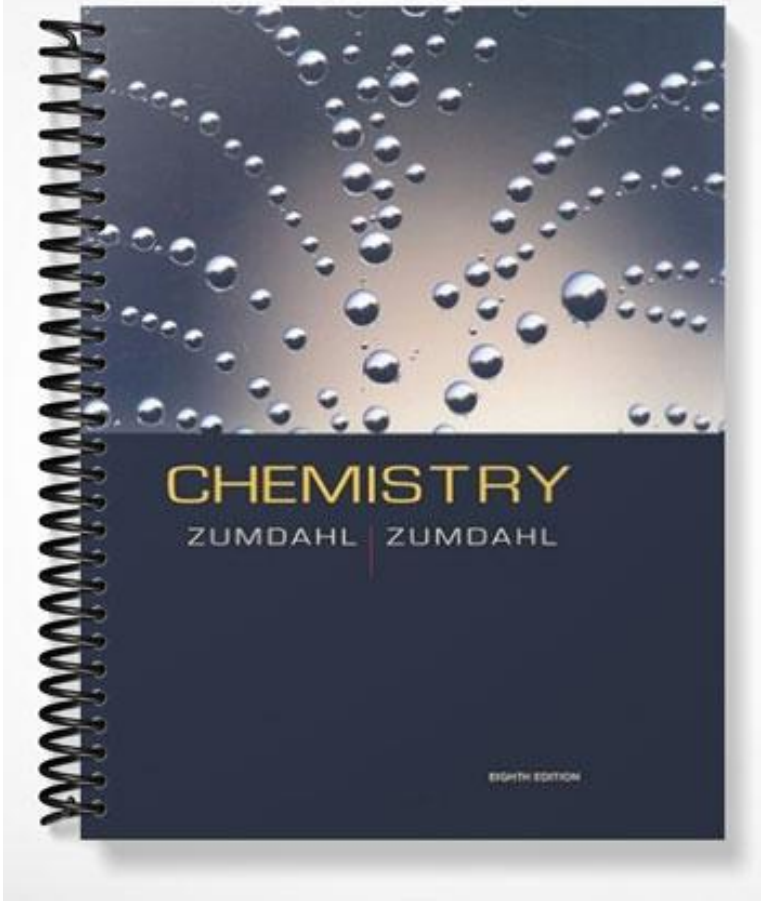
SOLUTIONS MANUAL



Instructor's Resource Manual

Experimental Chemistry

EIGHTH EDITION

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Preface

The person responsible for the administration of a general chemistry laboratory program is often the hardest working member of his or her department. There is an endless number of details associated with this job: scheduling laboratory experiments, scheduling lab instructors, ordering supplies, preparing reagents, grading lab reports, and so on, *ad infinitum.* The reader perhaps can tell that this is the author's own occupation!

This *Instructor's Resource Guide* is intended to make the job as painless and as rewarding as possible. The notes for each experiment are divided into four parts, described below. In particular, detailed recipes are provided for the preparation of the many reagents required, which in itself will save the instructor much time and effort.

Part A of each set of notes is a general introduction to each experiment from the instructor's point of view, illustrating what students are expected to learn from the experiment, and oftentimes pointing out pitfalls in the experiment. Specific suggestions for specialized equipment, or possible modification of the experiment, are given for many experiments.

The materials required for each experiment are given as **Part B** of the notes. In particular, instructions are given for the preparation of the many solutions required for the laboratory experiments. Since a school may be running several sections of laboratory in different rooms at the same time, instructions are most commonly given in terms of preparing one liter of a particular solution, even though the experiment may call for more or less than this as actually performed. One liter was chosen as a standard amount, since it should be relatively easy from this to calculate the actual amounts of solute/solvent needed for your particular situation. For some special reagents, requiring more complicated preparation, instructions are given for the specific amount required for the experiment as performed. The instructor should also take into account that in some instances a given substance may be available in both a hydrated and an anhydrous form: It is always specified in the notes which form the amounts given refer to.

Part C of these notes contains answers to the Pre-Laboratory Questions, whereas **Part D** has the answers to the Post-Laboratory Questions. Questions requiring students to look up a definition or explanation are keyed as far as possible to sections of the Zumdahl text. Questions involving the looking up of data are generally keyed to the *CRC Handbook of Chemistry and Physics.*

Comments, criticism, and suggestions are always welcome from users of *Experimental Chemistry* and this *Instructor's Resource Guide.* Over the years I have heard from users as close as the next town, and from as far away as the other side of the world. I always appreciate your help in improving these materials for your students.

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1

The Laboratory Balance: Mass Determinations

A. GENERAL NOTES

The correct measurement of mass is essential to students' future performance in the general chemistry laboratory, and this experiment will give the instructor a chance to determine what students may have difficulties in the future. In particular, students need much help in learning to read *measuring scale*s correctly. In everyday life, altogether too many devices have digital displays, and students may have learned to trust such devices absolutely and may not be able easily to read an analog scale correctly.

It is especially difficult for students to appreciate the concept of *estimating* between the smallest scale divisions of an analog device to obtain the last figure of the measurement. This experiment is a good place to discuss with them the concept that some numbers in a measurement are "certain" and some are "uncertain". On a triple beam balance, for example, the students see how having to estimate the position of the pointer between scale divisions makes the last significant figure "uncertain".

As this experiment progresses, it is wise for the instructor to examine student data *as they are being recorded* by circulating in the lab. In this manner, if it is discovered that a student is reading or recording data incorrectly, the student can be corrected *before* he or she mentally reinforces the incorrect technique.

This experiment is written without specific reference to a particular type of balance, since so many types of balances are available. The instructor should present a short discussion of the particular type of balance present in the laboratory and should demonstrate the use of the balance to small groups of students. It is helpful to post instructions for the balance on the wall above them for student reference after the demonstration.

If more than one type of balance is available for student use, discuss the rationale for which balance is to be used in which circumstances (e.g., why an analytical balance capable of mass determinations to 0.0001 g would not be used when an experiment calls for only an approximate amount). Since modern balances are so expensive, the instructor should explain and enforce rules for balance use (especially *cleanliness*).

Estimated time to complete: 60–75 minutes

B. MATERIALS REQUIRED (GIVEN PER 25 STUDENTS)

2–3 unknown objects per student for mass determination. These may be such things as rubber or glass stoppers of different sizes, or items sold commercially for such purposes. The items should be coded or marked somehow for identification (very small items may be dispensed in coded vials). The correct masses of the items should be measured by the instructor or stockroom staff before the laboratory so that students may check their results. Students must be encouraged to turn in these items at the end of the lab period for use by later sections.

C. ANSWERS TO PRE-LABORATORY QUESTIONS

1. Mass is a measure of the *amount of matter* in a sample, whereas weight is a measure of the *force of gravity* acting on a sample. In the laboratory, we determine the *mass* of a sample by comparison (on a balance) to reference masses.

2. This statement means that the overall mass of the sample should be in the neighborhood of 5 grams but that the precise mass of the sample must be known to the third decimal place.

3. The mass (amount of matter in the object) would not change; however, since the force of gravity is less on the moon, the object would have a lower weight.

4. Most balances contain metallic parts. Having liquids spilled could cause corrosion of the metal parts. In addition, if the balance is electronically powered, liquids can destroy the electronic circuits of the balance, and there would be a danger of electrical shock to the user.

5. The "difference" method typically means that the object has its mass determined when contained in a beaker, and then the mass of the empty beaker is subtracted from the combined weight. This is primarily to keep corrosive chemicals away from the pan of the balance. In the case of balances without an electronic tare feature (e.g., triple-beam balances), using the difference method also avoids having to ensure that the instrument balances when there is no load on the pan each time the balance is used (this assumes that all mass determinations are done by the difference method, and within a relatively short time frame).

D. ANSWERS TO POST-LABORATORY QUESTIONS

1. Most mass measurements are made by *difference* to allow for discrepancies in the tare weight exhibited by the balance. Using the same balance allows many such discrepancies to cancel out.

2. If an object is warm, it heats the air around it. Since warm air rises, an upward-flowing air current is created around the balance pan, which results in an apparent mass that is lower than the true mass.

3. The typical electronic balance costs in excess of $1000. If a reagent were spilled into the mechanism of the balance, it would almost certainly destroy the balance. Even very careful weighings of reagents on the pan of the balance are likely to leave a residue which would invalidate future determinations. Such reagents might also corrode the pan of the balance which would also affect the masses determined on the balance.

Note that Pre-lab question 5 and Post-lab question 3 are similar: this is intentional. Balances cost too much to take a chance on students ruining them by weighing chemicals directly.

2

The Use of Volumetric Glassware

A. GENERAL NOTES

The correct measurement of volume is essential to students' future performance in the general chemistry laboratory, and this experiment will give the instructor a chance to determine what students may have difficulties in the future. In particular, students will need a great deal of help in learning to read measuring scales correctly. It is especially difficult for students to appreciate the concept of estimating between the smallest scale divisions of a device to obtain the last figure of the measurement.

As this experiment progresses, it is wise for the instructor to examine student data *as they are being recorded* by circulating in the lab. In this manner, if it is discovered that a student is reading or recording data incorrectly, the student can be corrected *before* he or she mentally reinforces the incorrect technique. For example, if the instructor notices that a student is recording buret volumes to only *one* decimal place, it is possible to explain to the student on the spot, while the buret is in front of him or her, how and why we record such volume readings to a second, estimated decimal place.

This experiment introduces students to volumetric measuring glassware of both low and high precision. In particular, the use of a volumetric transfer pipet is covered since a number of the later experiments make use of such pipets. Because several types of pipet safety bulbs may be available, the instructor should demonstrate the type of bulb the students will actually use.

This experiment provides a good place to discuss the difference between *precision* and *accuracy*. For example, students will realize that a buret measures a contained volume to a higher level of *precision* than do the rough marks on a beaker. However, if through a manufacturing error, the entire scale of the buret is displaced from its correct location, the volume reading for the liquid contained may not be *accurate.*

Estimated time to complete: 75–90 minutes

Since students may not have yet covered the concept of density in their lecture course, it is suggested that the calculations of pipet volumes/mass of water transferred be covered at the chalkboard.

B. MATERIALS REQUIRED (GIVEN PER 25 STUDENTS)

Set-up of several different-sized graduated cylinders containing different amounts of colored water. The cylinders should be marked with a code letter for students to distinguish them. Either the cylinders should be sealed with a rubber stopper to prevent evaporation, or the instructor should read and record the volume of liquid in the cylinders at the start of the lab period.

25 25-mL pipets

25 pipet safety bulbs

25 50-mL burets and buret stands/clamps

5–6 buret brushes

C. ANSWERS TO PRE-LABORATORY QUESTIONS

1. For the 1-mL pipet: [(5)(0.05)/(1.00)](100) = 25% error

   For the 5-mL pipet: [(5)(0.05)/(5.00)](100) = 5% error

   For the 10-mL pipet: [(5)(0.05)/(10.00)](100) = 2.5% error

2. For the 10-mL sample: [(0.5)/(10)](100) = 5% error

   For the 20-mL sample: [(0.5)/(20)](100) = 2.5% error

   For the 40-mL sample: [(0.5)/(40)](100) = 1.25% error

3. A rubber safety bulb is always used to prevent aspiration of toxic or corrosive substances into the mouth.

4. "TC" indicates that the device is intended "to contain" the marked volume; "TD" means the device is intended "to deliver" the indicated volume. The temperature is usually specified since the volume changes with temperature. So, for example, a 250-mL volumetric flask marked "TC-20" would contain 250.0 mL at 20°C. A 5-mL pipet marked "TD-20" would deliver 5.00 mL at 20°C.

5. Volumetric glassware must be scrupulously clean in order that the volumes indicated by the manufacturer will be correct. Washing such glassware after use will make it easier to clean for the next use, and also will prevent any corrosive materials from reacting with the glass of the container and changing the volume of the container. For example, sodium hydroxide reacts slowly with glass: if a pipet or buret was used to dispense a sodium hydroxide solution, not rinsing the pipet or buret to remove the sodium hydroxide would gradually change the volume of the pipet or buret.

D. ANSWERS TO POST-LABORATORY QUESTIONS

1. It is likely that students will conclude that the buret and the pipet permit similar precision, whereas the graduated cylinder permits less precision. Typically, a graduated cylinder is used when only an approximate volume is needed. A volumetric transfer pipet is used when a precise, specific volume is needed, and a buret may be used when a precise, nonspecific volume is required.

3

Density Determinations

A. GENERAL NOTES

Density is a useful property of matter and one that is often misunderstood by students. It might be helpful to have set out in the laboratory, for students to compare, several samples that have the same mass, but have very different densities (e.g., a pound of plastic foam packing "peanuts" and a pound of lead pellets). The age-old question, "Which weighs more, a pound of feathers or a pound of lead?", can be a real problem for students.

It may be necessary to review the *geometry* involved in the calculation of the volume of the regularly shaped solid.

Estimated time to perform: 120–150 minutes

B. MATERIALS REQUIRED (GIVEN PER 25 STUDENTS)

25 regularly shaped solids, coded with identification numbers. The densities of these solids should be measured by the techniques discussed in Part A of the experiment for grading purposes. Such solids are sold commercially from science education vendors.

2–3 500-g bottles of metal pellets or chunks. A container should be provided for collecting these pellets/chunks. The metal may be spread out on a large evaporating dish or tray and dried in a 110°C oven for 1–2 hours for reuse. It is recommended that the pellets/chunks be of irregular shape to prevent them from scattering if spilled.

unknown liquid samples (25 mL per student)

If desired, individual samples may be dispensed in coded test tubes or vials. Otherwise, the instructor should be provided with several coded 8-ounce bottles of the different liquids for dispensing to students. Possible unknown liquids include

95% ethanol

cottonseed oil

corn syrup

5–30% sodium chloride solutions

1–2 kg sodium chloride

Salt sold at the supermarket for pickling or as Kosher salt is more than adequate, and much less expensive

C. ANSWERS TO PRE-LABORATORY QUESTIONS

1. A characteristic property of a pure substance is a property that is the same, regardless of the source of the substance.

2. Volume of metal = 26.8 – 21.7 = 5.1 mL

   Density of metal = 18.45 g/5.1 mL = 3.6 g/mL

3. Mass of liquid = 40.1825 – 32.4257 = 7.7568 g

   Density of liquid = 7.7568 g/10.00 mL = 0.7757 g/mL

4. Density of alcohol = 15.71 g/20.00 mL = 0.7855 g/mL

   [35.25 mL × (0.7855 g/1 mL)] = 27.69 g

   [125.4 g × (1 mL/0.7855 g)] = 159.6 mL

5. Specific gravity is the *ratio* of the density of a sample to the density of some reference substance (usually water) at the same temperature. Since specific gravity is a ratio of two densities, it has no units.

D. ANSWERS TO POST-LABORATORY QUESTIONS

1. If the solid were hollow, the volume measured would be larger than the actual volume of the matter in the solid. If the volume measured is too large, the density determined will be too low.
2. If air bubbles adhered to the metal pellets, the volume determined would be larger than the actual volume of the metal pellets. If the volume determined were too large, the density determined from it would be too low.
3. Assuming that a linear relationship between density and concentration holds true, one could measure the density of a solution of unknown concentration and then use the graph to read off the concentration corresponding to that density.
4. The volume of a liquid varies with temperature. Since the calculation of density involves the volume of the liquid, the density of the liquid would also vary with temperature.

4

The Determination of Boiling Point

A. GENERAL NOTES

Temperature measurements are an important part of this and several later experiments. Characteristic temperatures (such as the boiling and melting points of pure substances) are an important part of many experiments, whereas in other experiments, a change in temperature is used as an index of reaction.

The experiment is written in terms of the use of "red-liquid" thermometers. Although such thermometers tend to be somewhat less accurate, the avoidance of the problems associated with the traditional mercury thermometer makes them desirable. If mercury thermometers are still in use at your institution, some means should be provided for the collection and legal disposal of mercury if any thermometers are broken. If possible, you should consider switching immediately to red-liquid thermometers to avoid the hazard of mercury vapor being released into the laboratory.

Since water is being used for the heating bath for boiling point determination, unknown liquids must be chosen so as to have boiling points less than 100°C.

Estimated time to perform: 150–180 minutes

B. MATERIALS REQUIRED (GIVEN PER 25 STUDENTS)

10 × 75 mm test tubes

thermometers (–20°C to 110°C)

ice

2–3 vials (100 count) melting-point capillaries

rubber bands (size 8) or short lengths of rubber tubing (scissors will be needed to cut the tubing)

barometer

triangular files or glass scorers

boiling chips

boiling-point samples

If desired, individual small samples may be prepared and dispensed in coded 2-dram polyseal vials. Otherwise, 2–3 250-mL coded polyseal bottles of liquid may be provided to the instructor, who will dispense small amounts of a liquid to the students. Suitable unknowns include

acetone	b.p. 56°C	methanol	b.p. 65°C
95% ethanol	b.p. 78.2°C	2-propanol	b.p. 82.4°C

C. PRE-LABORATORY QUESTIONS

1. Thermometers are very fragile and must be checked for accuracy before use. The thermometer will be calibrated by checking its readings in two equilibrium systems of known temperature: a mixture of ice and liquid water at 0°C, and a boiling water bath (whose exact temperature depends on the atmospheric pressure).
2. A characteristic property of a substance is one that does not change under given conditions, regardless of the source of the substance. For example, both water on earth and water from Mars would boil at 100°C at 1 atm.
3. The broken capillary serves two purposes. First, the rough edge provides an uneven surface at which nucleation of bubbling can occur. Second, the capillary will contain a sample of the vapor of the liquid being determined: when heating is stopped, the collapse of this vapor into the liquid state can be seen easily by the experimenter and the temperature at which this happens can be taken as the boiling point of the liquid.
4. Boiling represents the point at which the pressure of vapor escaping from the surface of a liquid is equal to the magnitude of the pressure above the liquid. In a container open to the atmosphere, the pressure above the liquid is the day's barometric pressure. At lower barometric pressures, the temperature required to reach the point where the pressure of the liquid equals the barometric pressure will be lower.
5. acetone, 56°C; methyl ethyl ketone, 79.6°C; methanol, 65°C; 2-propanol, 82.4°C; 1-propanol, 97°C (*CRC Handbook*)

D. POST-LABORATORY QUESTIONS

1. In high-altitude areas, the barometric pressure is lower than at sea level. A food will take longer to cook to the point of "doneness" at high altitude, particularly if heated in water or if the food contains a high percentage of water, because the water is boiling at a lower temperature.
2. As long as both ice and water are present, the mixture is assumed to represent the equilibrium between solid and liquid phases and the temperature remains constant.
3. Solids and liquids are virtually incompressible and are not nearly as affected by changes in pressure as would be the gaseous state (boiling).

5

The Determination of Melting Point

A. GENERAL NOTES

Temperature measurements are an important part of this and several later experiments. Characteristic temperatures (such as the melting points of pure substances) are an important part of many experiments, whereas in other experiments, a *change* in temperature is used as an index of reaction.

The experiment is written in terms of the use of "red-liquid" thermometers. Although such thermometers tend to be somewhat less accurate, the avoidance of the problems associated with the traditional mercury thermometer makes them desirable. If mercury thermometers are still in use at your institution, some means should be provided for the collection and legal disposal of mercury if any thermometers are broken. If possible, you should consider switching immediately to red-liquid thermometers to avoid the hazard of mercury vapor being released into the laboratory.

The experiment calls for Thiele tube oil baths for the heating of melting- and boiling-point samples. Although Thiele tubes are relatively expensive, their use makes it unnecessary for students to stir the oil baths and avoids possible burns. If it is not possible to obtain Thiele tubes, 250-mL beakers half full of oil may be provided, and students should be advised to stir such oil baths vigorously during heating. If beakers of oil are used, the beakers must be supported safely to keep them from any chance of tipping over. If you have access to the type of melting point apparatus commonly found in Organic Chemistry labs (Mel-Temp, Thomas Hoover, etc.), you certainly should consider using that.

A common hazard exists whenever oil is used in such heating baths. If any *moisture* is introduced into the oil, such moisture will superheat when the oil bath is subsequently used by students, and the heated oil will splatter badly. ***The instructor should examine the oil baths before the beginning of each laboratory period*** and should ***replace*** any oil that appears cloudy due to the presence of moisture. It is best to use plain paraffin oil in the heating baths, since it does not begin to smoke at as low a temperature as other common oils. It is assumed that students' thermometers have been checked for accuracy (see Experiment 4). If not, such a check would be appropriate before this experiment.

Estimated time to perform: 150–180 minutes

B. MATERIALS REQUIRED (GIVEN PER 25 STUDENTS)

thermometers having a range from 0°C to 200°C

25 Thiele tubes (or other) paraffin oil-filled baths

2–3 vials (100 count) melting-point capillaries

rubber bands (size 8) or short lengths of rubber tubing (scissors will be needed to cut tubing)