Welcome to the Chemistry Lab

Introduction

The experiments in this lab manual have been chosen to introduce you to some important laboratory equipment and techniques. In addition, some of the experiments provide an opportunity for you to test your laboratory skills by analyzing “unknown” samples. Each experiment is designed to be completed within a two-hour period. You should be able to complete the work in the time allotted. This time allotment does assume, however, that before coming to the laboratory you have read the introduction to the experiment, have answered the pre-laboratory problems (which introduce needed concepts), and have carefully read the experimental procedure.

Laboratory Notebook and Reports

A lab report must be submitted for each experiment that you perform. For most experiments, the report will be in three parts: the pre-lab due at the beginning of the lab period, the data and observations collected while in the lab, and the calculations and post-lab questions to be completed after the procedure. The majority of the labs in this manual have a report sheet created for you. If no report sheet is provided, you must create your own.

All submitted reports MUST BE TYPED, and should contain the following elements, when applicable, in the order given:

Pre Lab (do NOT staple or otherwise attach to any other document):

1. Your name, section number, lab partner(s), and date.
2. The title of the experiment and the date on which it is performed.
3. Answers to any pre-lab problems (on separate paper, unless ample room is obviously provided).
Data and Observations:

4. Data and observations are to be entered directly into your laboratory data sheet(s). Neatness is important, but honesty and accuracy are equally as important.

   Numerical data must be identified by a tag (e.g. “mass of Erlenmeyer flask – empty”) and include units (g, mL, etc.). Use the correct number of significant figures to report your data.

   For example:
   
   Mass of empty Erlenmeyer flask 52.650 g
   Mass of flask + benzoic acid 53.492 g

   Note that you record both masses, not just the difference between the two. Similarly, for measurements with a buret, you should record both the initial and final buret readings, not just the difference between them.

   If a mistake is made while recording data, draw one line through the incorrect entry and write the correct value nearby. It may turn out that the first value will be needed after all.

Final Report:

5. If there is a printed report sheet in this manual, use it to summarize your data. Also include your name, section number, lab partner(s), and date.

6. Calculations: There should be a separate section devoted to the calculations. They should be completely separate from your data and results. You must show every mathematical calculation that you perform, so that your lab assistant may easily follow your work (or mistakes – in order to give partial credit).

7. Answers to any Post Lab questions belong on separate paper unless ample room is obviously provided.

Pre and Post Lab calculations are designed to be performed individually, with the answers given to the correct number of significant figures and with the correct units.

Pre and Post Lab questions are designed to be answered individually. Answers should be concise, legible, and in your own words (do not plagiarize or otherwise copy from texts, websites, or your classmates).
Laboratory Safety Rules

Laboratory accidents can result in loss of time, damage to clothing and other property, and personal injury. By following suitable precautions, you can anticipate and prevent situations that could lead to accidents.

You must make yourself completely familiar with the following safety rules. You will be required to sign a Laboratory Safety Contract stating that you have read these rules and agree to abide by them. At the beginning of the second laboratory period you will take a short multiple-choice quiz on the rules; if you miss more than one question, you must pass a retest before you will be permitted in the laboratory.

Rules

1. Do not work in the lab unless the lab assistant or instructor is present. No unauthorized experimentation is allowed.

2. Work carefully with full awareness of what you are doing, so as to avoid dropping or breaking equipment or spilling chemicals. Keep reagents and equipment well back from the edge of the lab bench. Never run in the lab.

3. You must provide your own pair of safety goggles and wear them in the lab at all times. Safety glasses MUST meet or exceed ANSI Z87.1 (this should be indicated on the packaging). A lab apron or lab coat should also be worn at all times while working in the lab.

4. You must wear shoes without open spaces; sandals and open-toe shoes are not acceptable.

5. Confine long hair and neckties; they may catch fire, get into chemicals, or get caught in apparatus. Loose jewelry or rings can also be a hazard. Frilly or flared clothing, especially synthetics, are not safe around flames unless covered with a lab apron or coat.

6. Do not bring food or drink into the lab. No eating or smoking is permitted in the lab.

7. Never taste a chemical. Smell a chemical by fanning the air over the container to waft the vapor to your nose; never smell directly. Do not touch chemicals with your hands unless specifically directed to do so; if contact occurs, immediately flush the area with cool water.

8. Mercury vapor is invisible but toxic over time. A broken thermometer should be reported immediately to the instructor.
10. Never look directly into an open vessel in which a reaction is occurring that could cause spattering. When heating materials in any container, be sure that the open end does not point in the direction of other persons or yourself.

10. If any chemical gets into your eyes, flush with water for at least 15 minutes.

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11. In case of fire, turn off the burner first. If there is a fire in a beaker, try to smother it with a watch glass or wet paper towel placed over the beaker. For an open flame, use the fire extinguisher pointed at the base of the flame. If the fire is uncontrollable, close all windows and evacuate the room and pull the fire alarm located near the emergency exit or in the hallway. After evacuating to a safe place, call 911, and campus security @ 365-3544.

12. Skin burns should immediately be placed under cold running tap water for 5-10 minutes in order to remove the heat.

13. If clothing catches on fire, use a fire blanket or safety shower.

14. Do not force a glass tube or thermometer into a stopper; the glass can break and gash or stab you. Instead, lubricate the end of the glass with glycerin; hold both the glass and the stopper with a cloth towel to protect your hands; grasp the glass close to the stopper; insert with a slow twisting motion (see figure below).

15. Report all accidents and injuries to the instructor as soon as emergency action has been initiated.

16. Never place chemicals directly on the balance pan. Instead, use a beaker, flask, watch glass, or piece of weighing paper.

17. Read the label on a bottle twice before using the contents. Never contaminate the contents of a bottle by putting reagents back into it. Do NOT waste reagents; if you take too much, share it with others who still need your chemical.

18. Label any sample or mixture that you prepare.
19. Never mix any reagents unless specifically directed to do so.

20. When mixing water and acid, always add acid to water otherwise violent spattering may occur. Remember the word “acid” alphabetically comes before “water,” so acid-into-water.

21. Dispose of waste chemicals as directed. If a waste container becomes full, tell the instructor or lab assistant so they can get an empty replacement. Don’t just ignore the situation.

22. Do not use cracked glassware, as it may break when stress is put on it. Place broken glassware in specifically designated containers in order to prevent injury to the cleaning personnel.

23. If you discover that bottles of chemicals need to be refilled, tell the instructor or lab assistant.

24. Keep the lab bench and tables clean. Wipe up all spills. Acid spills should be neutralized with sodium bicarbonate (which can be obtained from your instructor or lab assistant).
Quantitative observations in chemistry laboratory work routinely include measurements of such quantities as masses, volumes, and temperatures. Despite the apparent diversity of these measurements, they all share one general feature that’s important to notice. They all involve the reading of a scale— the markings on a graduated cylinder, a meter stick, a beam balance, an optical scale, etc. Many instruments in the modern laboratory have digital readouts. You should not be lulled, however, into thinking that they are absolutely accurate, since somewhere, at some time, the digital scale was calibrated by a human being reading a scale.

The thermometer at the right will serve for our present discussion.

Read this thermometer and write your reading down here: __________°C. (Do it NOW!)

Without being able to peek over your shoulder, it’s hard to discuss your actual measurement, but a safe bet would be that your answer is “24-point-something.” Assuming that the marks on the thermometer were correctly placed, we can say that the “2” and the “4” are known with total certainty. However, more can be said about the temperature. You would probably agree that the reading is not as high as 24.9 °C, and it is certainly higher than 24.0 °C, or something in this neighborhood. Maybe the best answer would be 24.3 ± 0.1 °C, indicating the apparent doubt we have about how far the mercury is between 24 °C and 25 °C. Our best guess for the number is 24.3, and the uncertainty is about ± 0.1.

Before continuing, an important definition: We will say that the Celsius temperature is known to three significant figures. The number of significant figures is found by looking at the position of the first digit in the uncertainty (the tenths position in this example) and then counting all the digits in the number (from the left) up to and including this position.

Sometimes significant figures are explained by saying that we count all the digits whose certainty is known (“2” and “4”) plus the first uncertain digit (the “3”). This can be misleading, however, because if the reading is 24.9 ± 0.1, then the “ones” digit is not certain (it is “4” in 24.8 and 24.9, but it is “5” in 25.0), but the “best guess,” 24.9, still has three significant figures.
Chances are that you know your weight to three significant figures also. “I weigh about 152 pounds” suggests that your actual weight might be 150-154 pounds or so. The uncertainty is about ± 2, in the ones position. The number 152 has 3 digits up to and including the ones position, or 3 significant figures.

If this is where things ended with significant figures, all that we would have is a definition to play with, but there is more –

General Assumptions Regarding Significant Figures

1. Notice that the number of significant figures is determined by the person doing the measuring. You need to decide the uncertainty in your scale readings. Naturally, however, if the instrument is out of adjustment, there will be “built-in” error or uncertainty that you might not be aware of. In some cases, this might reduce the number of significant figures you can claim to have, just as a badly-sighted rifle would reduce target practice scores. In Chemistry, we assume “well-sighted rifles,” so the uncertainty is presumed to be something between you and the scale you are staring at. This assumption is not always justified – in some cases, you may want to blame the instrument or procedure itself for errors you find. Fine! Just supply evidence and reasons!

2. Notice that in everyday life, you tend to follow an unspoken, intuitive rule: Stop a numerical expression when you reach the position of the first digit of uncertainty. A person says “I weigh about 155 pounds.” He doesn’t say, “I weigh about 155.2386439 pounds.” You followed this intuitive rule with your thermometer reading by stopping at the tenths position. All we ask is that you continue to follow this unspoken guideline in chemistry. Stop a numerical expression when you reach (roughly speaking) the first uncertain digit.

3. Notice, also, in everyday life people assume when they hear a numerical expression that the last digit is the only uncertain one. If you are told that the road you are looking for is 4.6 miles ahead on the right, you would be a little upset to miss the turn, only to discover that it was 3.6 miles instead. If the person had said “about four miles,” you might have started looking for the road earlier. Since the tenths were expressed, you assumed that the “4” was known with certainty – just because that’s the way we intuitively operate. Again, all we ask is for you to make the same assumption. The first uncertain digit is the last one expressed. If you say the magnesium ribbon is 18.46 centimeters long, we will take the “1” and “8” and “4” to be certain, but will assume the “6” is a little doubtful – therefore we declare there are “four significant figures.”
Multiplication and Division

Now, let’s look at what happens when we want to multiply or divide numerical measurements. A student measures a rectangular piece of tile, and reports the measurements: Length: 15.1 cm; Width: 3.2 cm. What, then, is the area of the tile surface?

Here is the way one student worked the problem on a calculator:

\[ \text{Area} = \text{Length} \times \text{Width}. \quad A = (15.1 \, \text{cm}) \times (3.2 \, \text{cm}) = 48.32 \, \text{cm}^2. \]

The student was quite happy with “48.32 square centimeters.” This answer, however, is not only incorrect, but downright dishonest!

Remember that the last digit reported is the uncertain one. The honest length is 15.1 ± 0.1 cm, and the honest width is 3.2 ± 0.1 cm. (The uncertainties might even be larger than this.) The area of the tile could conceivably be \((15.2 \, \text{cm}) \times (3.3 \, \text{cm}) = 50.16 \, \text{cm}^2\). At the other extreme, the area could be \((15.0 \, \text{cm}) \times (3.1 \, \text{cm}) = 46.5 \, \text{cm}^2\).

So what are we certain about in this answer? The area could vary from over 46 to 50 cm², simply due to the doubt that is always built into judging distance between scale markings. The calculated number is 48.32 cm², but the uncertainty is about ± 1.8 cm. Since the first position in the uncertainty is the ones position, that is where the value of the area should “stop,” therefore the correct answer must be “48 cm².” Everything after the doubtful 8 must be chopped off. It is dishonest to report 48.32 cm², simply because that would imply that the first uncertain digit is in the hundredths place. You just don’t know anything approaching this much certainty about the number. If you want higher precision, the only way to get it is to make more precise measurements. Carrying out things to greater extremes on paper when you calculate is simply self-deception and a waste of time.

Another example:

\[ 7 \, \text{SF} \quad \quad 3 \, \text{SF} \quad \quad 3 \, \text{SF} \]
\[ (21.00000) \times (3.00) = 63.0 \quad \text{(NOT 63.00000!!)} \]

The same rules holds true for division:

\[ 21.00000 / 3.00 = 7.00 \]

(Our answer is limited to three significant figures by the 3.00.) If you are multiplying and dividing together, things still work the same way:

\[ [(2.000) \times (4.1) \times (10.000000)] / 3.00000 = 27 \quad \text{(NOT 27.3333333!!)} \]

Here we are limited by the two significant figures in 4.1!
**Zeros as Significant Figures**

We have been implying that the number 10.0 has three significant figures. If the person went to the trouble of putting the zero after the decimal point, there must have been a reason for it; this just happens to be the first uncertain digit, we assume. This is fine. (Of course, many students just add zeros to add zeros, so we have to follow the rules so we know that 10.0 does indeed have three significant figures.)

How many significant figures are there in 10,000 yr? If this represents the estimated time since the last Ice Age, it’s fairly clear that there are five significant figures here! In other words, we are uncertain by more than ± 1 year – in fact, the uncertainty is probably more than hundreds of years, maybe even thousands of years.

How would you express 10,000 yr to indicate that this might be uncertain by, say, at least a few hundred years? You can’t chop the last two zeros – they are needed to represent the size of the number. This is the best way: $1.00 \times 10^4$ yr. In scientific notation we simply leave those zeros that we wish; we are showing three significant figures in $1.00 \times 10^4$; 10,000 to two significant figures would be $1.0 \times 10^4$.

Now work this problem: $(10.0) \times (10.0) = ???$ Well, the answer should have three significant figures, and the answer is obviously “one hundred.” But if you write 100, you are leaving the reader to wonder whether any of the zeros are significant.

Solution: $1.00 \times 10^2$.

How many significant figures are there in 0.00033? Before you think too far here, put the number in scientific notation: $3.3 \times 10^{-4}$. Now how many significant figures are there? Both expressions have two significant figures. Apparently the only function of those left-hand zeros in 0.00033 is to keep the decimal point where it belongs. **Zeros to the left of all other numbers are never significant**; they are only place-holders.

To summarize, here are some examples of significant figure determinations:

- $4.00030$ 6 significant figures
- $0.00300$ 3 significant figures
- $1.02050$ 6 significant figures
- $10,000.0$ 6 significant figures
- $10,000$ ? significant figures (ambiguous; could be 1-5)
- $10.000$ 5 significant figures

If you fail to understand any of these examples, check with your instructor.

**Rounding Off**

If we wish to express the number 326.337 to four significant figures, we would write 326.3. To make it have 3 significant figures, since the first nonsignificant figure is less than 5, we simply drop it. How would you express the same number to two significant
figures? 320? 330? 3.2 x 10^2? 3.3 x 10^2? Here, the first nonsignificant figure we are dropping is greater than 5 – the number is closer to 330 than 320. But “330” fails to tell us whether the zero is significant. 3.3 x 10^2 is the clearest answer.

Suppose we wish to express the number 37.52 to two significant figures. Here the first nonsignificant figure being dropped is 5 and it is followed by a non-zero digit, 2. Since the 2 is the second nonsignificant figure, it contributes nothing in helping us make our decision. In fact, we treat 37.52 as though it were 37.50. So, do you round up or round down? Example: Express 37.50 to two significant figures. It is clear there is not really a best way of operating here, since 37.50 is just a close to 37 as it is to 38. Many scientists decide in cases like this to round to the even digit. This rule simply guarantees that over a large number of “round-offs” about half the time we would round up, and half the time round down. Thus 38.5 to two significant figures would also be 38. However, 39.5 would be rounded up to 40, so that the last digit of the number, the zero, is an even number. Using this rule, you will never get an ODD number when you round off a number ending in 5. It is expected that you will follow this rule for any rounding that you do.

Final Comments on Significant Figures

1. Learn the rules – they save time – but remember that the only justification for all of this business is to avoid saying more about a calculated value than you can possibly know. It should be admitted that “significant figures” are only a simple shortcut for indicating the uncertainty of numbers; if you pursue this topic further, you will discover cases where “significant figures” give you incorrect estimates of uncertainty. This should not diminish their routine utility; as long as you “think significant figures” you will avoid gross misstatements of uncertainty (and save yourself a lot of lost points). You should not have to ask “How far do I carry my answers out?” STOP when you reach the first position of uncertainty.

2. Textbook authors and problem writers routinely violate significant figure rules. You will probably notice this more than once in this course. Be assured that we fully intend to consistently follow our own advice, and we will expect you to do the same – particularly on laboratory calculations and on homework and tests.

3. What is the “rule” for adding and subtracting numbers with significant figures? Well, there really isn’t any, if you insist on referring to significant figures. But, there is a simple way of knowing how to round off your addition and subtraction results:

\[
\begin{align*}
2.34000 & = 2.3 \\
0.001 & = 0.0 \\
4.3 & = 4.3 \\
10.82 & = 10.8 \\
+ 3.37 & = 3.4 \\
? & = 20.8
\end{align*}
\]
Round off each term to the first position of uncertainty in any number that contains an uncertain digit. Then add. (Or add the numbers first, and then round back to the least amount of decimal places in any numerical value that was included in the calculation.) In either case, you have avoided extending things beyond the first uncertain digit.

4. Please keep in mind that this entire discussion has been limited to numbers involved with scale-reading type measurements. There are other kinds of numbers – particularly counting numbers or defined numbers – which can lead you astray if you blindly follow rules.

For example, there are exactly 12 in a dozen. The number “12” has what amounts to an infinite number of significant figures: 12.00000000000…… and so on.

There are exactly 1000 milliliters in a liter, because we define it to be that way.

If you count 23 people in a room, that is assumed to be exactly 23.

These kinds of numbers can never limit the number of significant figures in any calculated result from multiplying or dividing. Thus: If I count exactly 144,000 nails, then that is 12,000 dozen nails, exactly, even though you might think that 144,000/12 should be carried out to only two significant figures. Similarly, in converting 1.0612 liters to milliliters I would calculate (1.0612 L)(1000 mL/L) = 1061.2 mL. The number of significant figures (5) in the answer is limited by the number of significant figures in 1.0612 L, NOT by the number 1000, which in this example is an exact number.
Common Laboratory Apparatus

- Crucible
- Crucible tongs
- Test tube rack
- Volumetric flask
- Erlenmeyer flask
- Beaker
- Graduated cylinder
- Büchner funnel
- Pinch clamp
- Test tube holder
- Wire gauze
- Ring stand and iron ring
- Triangle
- Clamp
- Dropper pipet
- Burner