Experiment 1: Use of Common Lab Equipment, and Determining Significant Figures in Equipment[1][2][3]

Measuring the volume of liquids and the mass of matter are two important components of many experiments. In this lab, we will practice using centigram (top loading) balances, and analytical balances. We will distinguish between different types of glassware used in the lab. We will practice using this glassware, and we will use volumetric ware to determine the average volume of a drop. We will determine significant figures for specific tools. All measurements have some degree of uncertainty.[4] Uncertainty represents doubt in the measurement. It is the part of the measurement of which we are not sure.

Common Glassware: Below are pictures of some common glassware.[5]

Chemical Balances: Below are pictures of some common balances[6][7]

Measuring mass:

We use balances to determine the mass of matter. We depend on the balances to give us accurate and precise measurements. Sadly, the balance is often misused and abused. You will need to follow several guidelines to

[1] Adapted from Seager, Spenser; Slabaugh, Michael; Safety Scale Laboratory Experiments for General, Organic, and Biochemistry for Today' 4th ed
[2] General background reading. Good stuff in here. Postma, James, Roberts, Julian L, Hollenberger, J. Leland, Chemistry in the Laboratory, 5th edition pages 15, 18, 21 WH Freeman, NY NY, 2000
[3] This experiment and report sheet could not have been done without the generous support of former students. I would like to especially thank Isabella Germek for her tireless efforts to improve this experiment and especially the report sheet. It is still evolving, Audra Tendzeldam for reviewing the current procedure, and Dr. A. Reyes for his constant support and guidance in the matters of error analysis.
[4] Bell, Stephanie, A beginner's Guide to uncertainty of Measurement, NPL No. 11, Crown Copyright 1999. Issue 2 with amendments March 2001
[5] http://informationsecurity.451research.com/wp-uploads/2013/02/chemistry-glassware.jpg
[6] http://cfnewsads.thomasnet.com/images/large/480/480252.jpg
[7] http://cdn.teachersource.com/images/products/pop/bal160.jpg

Alscher: Note Keep this handy. It has information about equipment that you will use throughout the semester. You should consider referring to this handout when you start labs that use beakers, thermometers, balances, graduated cylinders, and burets

maintain the balances. First, handle the balance with care because they are expensive. Do not drop anything on the balance. You never place chemicals directly on the pan. Use a weighing paper, a watch glass, a beaker, or some other container to measure the mass of chemicals. When you are done using the balance, clean off the pan with a brush. Close the doors of the analytical balances. Put the balance to zero or off.

Mass measurements can be obtained using two methods; we will practice both methods. For both methods, when weighing chemical compounds, we always use a weighing paper or clean container. In the first method, called the "**traditional method**", we measure and record the mass of the container. Then, we measure and record the combined mass of the sample and the container. The mass of the sample is obtained through subtraction. The second method is often called "**the direct method**". In the direct method, we use a container, but we do not record the mass of the container. Instead, we tare the balance, which sets the value of the container at zero by saving the mass of the container. When we add the sample to the container, the balance does the subtraction for us. So instead of us subtracting the mass of the container from the mass of the container and sample, we read only the mass of the sample.

Breezes in the room can affect the mass of the object you wish to measure. A balance reading zero is actually not at zero grams. The balance is measuring a column of air pressing down on the mechanism. When you place an object on the balance, you are displacing some of the air with something that has a different mass for the same volume displaced. When we 'tare' the balance with a weigh boat on the pan, we are essentially subtracting this mass from the mass that we measure next.

Breezes move air around and change the air mass on the balance. This leads to drift in the measurement. Therefore, always keep the doors around the balance area closed and try to be still when you are waiting for the balance to come to a recording point. The analytical balances are much more sensitive than top-loading balances. Glass doors are provided around the balance to prevent such currents. Close the glass doors after you put your sample on the pan. This will help control drift.

Electronic balances have different degrees of sensitivity or precision. You don't always use the most precise balance in the lab for each experiment, but you do use the most appropriate balance for each measurement. The precisions of some common balances are listed below.

Table 1: The precision of common balances

Balance	Standard Uncertainty (g)
Top-loading [Centigram]	±0.01
Analytical	±0.001
Semi-micro	±0.0001

Volumetric Measurements:

We use glassware with two purposes: to deliver a known volume and to contain a needed volume. T**o deliver** glassware (TD)-(burets and pipets) delivers a known quantities of liquids for experiments; to **contain** glassware (TC)-(beakers, graduated cylinders) contains a volume of liquid while experiments are being done, and for other general purposes that need a container of liquid.

Before we discuss the proper use of graduated cylinders and other volumetric ware, we need to consider the behavior of liquids in glassware. Water and most aqueous solutions wet the surface of clean glass and, thus, form a curved surface in glass containers. This curved surface, called a **meniscus,** becomes more apparent in narrow container as shown in the picture below. Notice the difference between the wide container and the narrow container. It is much easier to read a volume in a container with a narrow bore.[8]

[8] meniscusprogram.jpg

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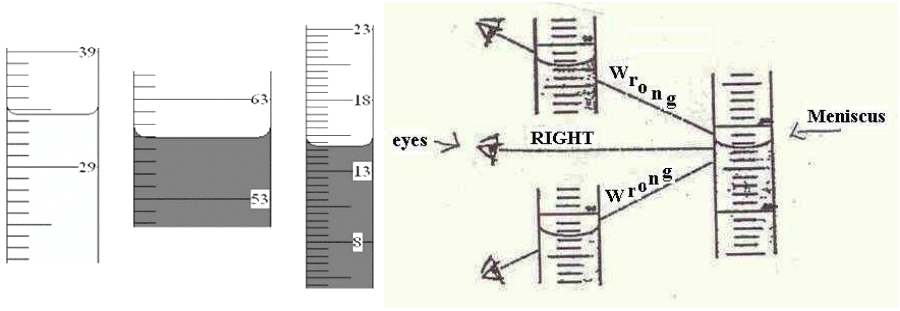
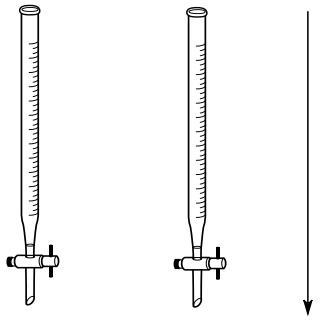


The picture on the right shows the correct orientation of your eye in relation to the meniscus.[9] We read the bottom of the meniscus. The eye is horizontal to the meniscus. When the reader's eye is above the meniscus, the reading will be lower than it should. When the reader's eye is below the meniscus, the reading will read higher than it should be. You will create a meniscus reader for this lab. This is a small card with a black or bright colored rectangle. The colored background reflects off the bottom of the meniscus. This gives a better definition to the meniscus.

When you record the volume of liquid in volumetric glassware, be sure to use all certain digits plus a guess (your first uncertain digit). For example, in the first graduated cylinder, I might read the measurement to be 29. 46 mL. The first three digits are my certain digits. The last number is my guess, or uncertain digit. You will practice determining significant figures on a variety of glassware. Some glassware, however, has the precision pre-determined for you. This uncertainty is listed in the table below. To obtain the correct number of significant figures, use the calibrated marks plus one uncertain digit, which is the best estimate between the calibration marks. This reading provides the correct number of significant figures for the measurement.

Glassware is designed for different uses. Graduated cylinders are designed to measure any liquid volume up to the cylinder capacity. The volume contained in a graduated cylinder is estimated to one decimal place more than the smallest division on the cylinder. Volumetric pipettes are designed to deliver a fixed volume of liquid and therefore have only a single calibration mark that is located on the narrow neck above the bulb. Graduated pipettes and burettes are designed to deliver a volume of liquid up to a maximum of the volume capacity. Scales on these devices read downward.

Note: You read the burette down. The burette shows the volume delivered into the flask not the volume contained in the burette. Watch for a drippy stop-cock. This is a source of error. Make sure that all of the glassware is primed with the solution that you will use for the lab. For this lab, the burettes are primed with water. Your initial volume is always lower than your final volume as a value.



In the first burette, the level of the liquid is 4.00 mL. After delivery of 3.00 mL, the new level is 7.00 mL We read burettes down, starting with a small number and ending with a large

[9] http://www.sciencetoolbox.com/articles/4GraduateCylinder.jpg

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Table 2: Precision of some standard glassware used in lab.[10]

Glassware	Typical Standard Uncertainty
100-mL graduated cylinder	± 0.2 mL
10-mL graduated cylinder	± 0.1 mL
50-mL burette	± 0.02 mL
25-mL pipette	± 0.02 mL
10-mL pipette	± 0.01 mL

Temperature measurements:

During lab, we will be using a liquid expansion thermometer. Liquid expansion thermometers have an expansion liquid, such as Mercury or alcohol in the thermometer. A long piece of glassware (the stem) with a uniformly bored core with a glass bulb at the end is filled with the liquid. Thermometers are calibrated in the factory by immersing the thermometer fully in the solution to be measured. It's not practical for us to use thermometers this way, so this is a source of error that we can account for when using a thermometer because the entire liquid is not exposed to the same temperature. When the thermometer bulb is immersed in the material to be measured (either hot or cold) the liquid will undergo a volume changed by expanding if the material is warm, and contracting if the material is cold. The liquid in the thermometer reaches thermal equilibrium with the material, and one reads the level of the liquid (based on a predetermined scale) in the thermometer. These changes in temperature are linear.

In chemistry, as with other lab sciences, we tend to use the Celsius or the Kelvin scales when working with temperature changes. We prefer mercury thermometers, because they are more accurate, but use alcohol thermometers because of cost and they are less toxic than mercury thermometers when broken.

Alcohol thermometers have a lower accuracy due to alcohol's volatile nature. This means that alcohol in the stem can vaporize when the temperature rises above 78°C. Therefore, the volume change is smaller than it should be when measuring warmer material (since some of it has been converted to gas). Also, alcohol tends to 'wet' the stem bore surface. This is a problem because the liquid sticks to the surface of the glass bore when the temperature drops leading to a lower than expected temperature in each case.

One way to account for these systematic errors is by calibration. Thermometer calibration is performed on material whose results is well known and expected in the specific temperature range that the thermometer is designed to operate. In general, the accuracy of a thermometer is a measure of the ability of the device to measure temperature without error. (We know this is impossible, since all tools have inherent error.) An accurate thermometer should measure temperature changes within +/- 0.5 °C of the actual temperature change.

The thermometer is adjusted such that the readings give the expected results or a correction factor can be determined and used in other experiments and measurements. We can't adjust the markings on our thermometer and we have no guarantee that we will use the same thermometer throughout the semester. We want to calibrate a thermometer to get a 'sense' of the type of systematic error that thermometers add to our data. We will create a calibration correction curve for a thermometer.

In our case, because the markings along the stem cannot be adjusted, we will prepare a Correction Curve for our thermometer so that thermometer readings can be corrected to accurate temperatures. Known temperature baths for our calibration will be generated using the Ice Point and the Boiling Point of pure water.

[10] for more exercises on error, read and do the problems on this web page:
http://chemwiki.ucdavis.edu/Analytical_Chemistry/Quantifying_Nature/Significant_Digits/Uncertainties_in_Measurements
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Error and uncertainty[11]:

This lab is also about uncertainty and error in measurement. Instruments have error; Instruments have uncertainty. No one is perfect when measuring data (even computers). When you measure the mass of water using a balance or the volume of water using volumetric ware, you are exploring uncertainty in measurement. One purpose of this lab introduced you to precision in measurement: using the correct tool for the correct precision (ie. Places after the decimal) We saw Measurements that have a higher degree of precision give the examiner more information about the measurement. Yet, we should not use a tool that is outside of the range of our precision. For example, a student wants to measure 20 mL of water; using the 25-mL graduated cylinder is a reasonable tool, using a 10 mL or 50 mL graduated cylinder is not reasonable. Using the 10 ml graduated cylinder has more precision, but when a tool is used repeatedly for the same measurement, errors are multiplied. The larger graduated cylinder has less precision. Precision also refers to reproducibility.

There are several types of errors that we will encounter in labs. Listed below are some examples of types of errors using a balance to collect experimental data. Significant figures and the calculations that arise from measurement are an important part of this class. Become familiar with these errors as you will discuss them in results and error analysis statements.

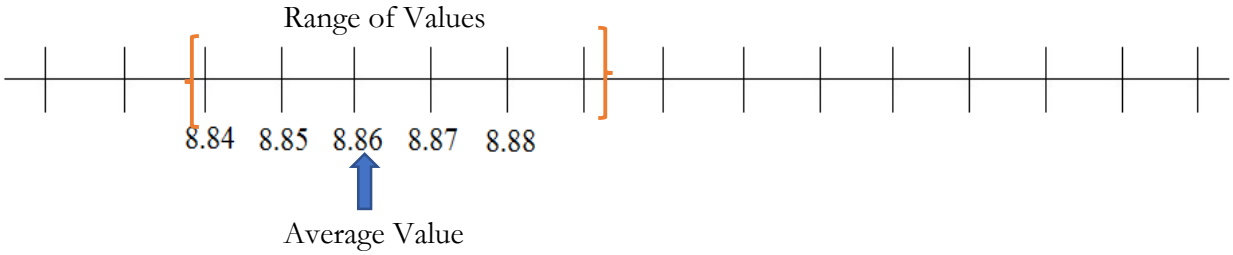
1. **Radom errors.**
2. **Systematic errors**

Accuracy versus Precision:

Accuracy: refers to the proximity of a measurement to the true (or accepted) value of a quantity.

Precision (Reproducibility): refers to the proximity of several measurements (how close they are) to each other.

Measuring Mass as an Example:



Random Errors (two sided errors) (precision)

They cause a measurement to fluctuate around a mean; sometimes the value obtained is too high, and sometimes the value obtained is too low.
These errors are called random because it is very hard to pinpoint their origin. Examples (on weighing):

- Air currents (lift up or push down).
- Voltage fluctuations in the electrical system.

We cannot measure the actual contributions of these errors…
Nevertheless, we can quantify their overall impact!

[11] From a handout by Dr. Abraham Reyes
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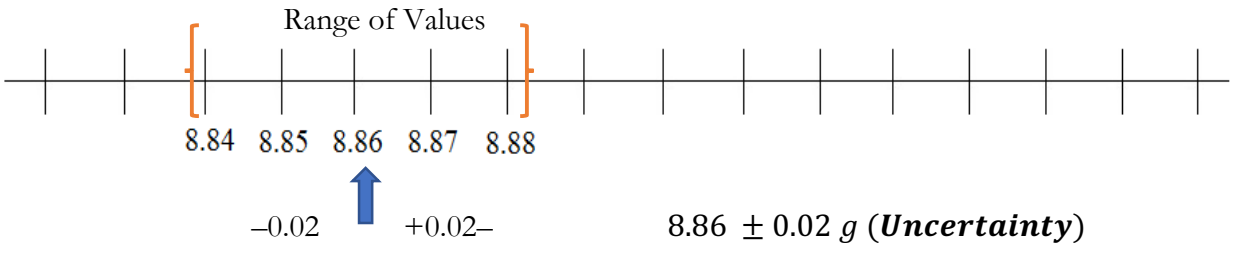
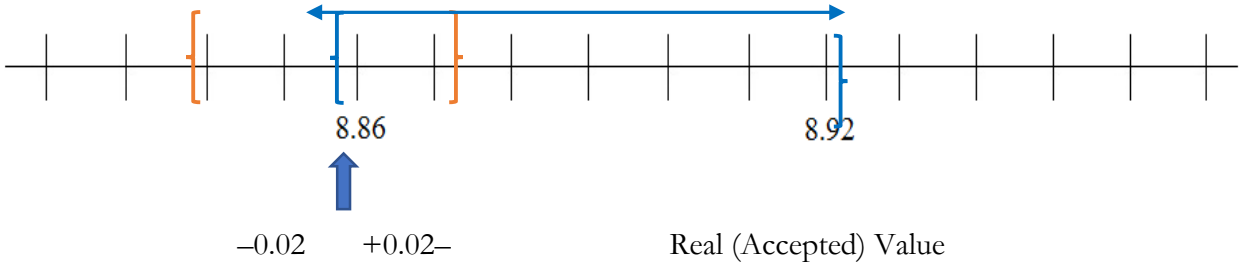


$8.86 \pm 0.02\ g$ (***Uncertainty***)

Precision is given by this range.

Accuracy

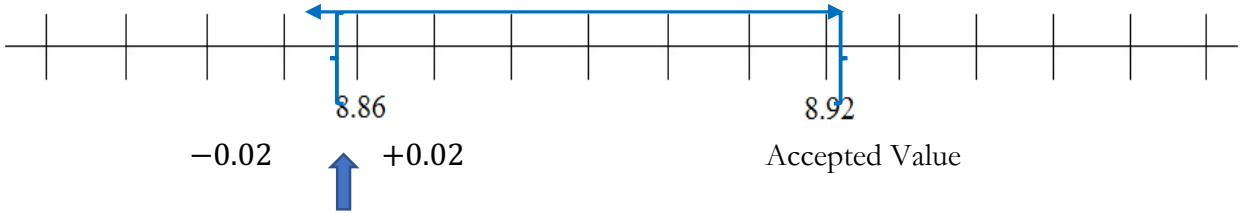


Real (Accepted) Value

There is a marked difference between the measured and the accepted value. This is attributed to Systematic Errors.

Systematic Errors (One sided errors)

Systematic errors cause a measurement to *shift in one direction*. Because of them, the measurement will be *either too high, or too low*.



Accepted Value

Too low due to a systematic error.

These errors have the following sources:

- The instrument.
- The procedure.
- Human error (that's why we need to pay attention!).

Systematic Error (Examples)

Measuring the volume in the graduated cylinder and not being at eye level with the meniscus.

Not tareing the balance properly (*starting at a lower mass, say* $-0.02\ g$)

A thermometer measures $1°C$ *above the actual temperature. (How could you find out if it is well calibrated?)*

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Characterizing Systematic Error

$$\%\ \text{error} = \frac{measured\ value - accepted\ value}{accepted\ value} \times 100$$

Characterizing Random errors (the percent difference) is a good measure of precision.

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