Chemistry 50 and 51 Laboratory Manual General Information

Mt. San Antonio College
Chemistry Department

2019 - 2020

TABLE OF CONTENTS

PREFACE

The laboratory component of a chemistry course is an important and integral part of the learning experience for all chemistry students. The laboratory provides the students with a hands-on experience in learning chemistry skills and techniques while conducting experiments. The laboratory has its own unique measurable objectives that are introduced, practiced, and assessed through a variety of learning experiences, including inquiry-based and/or open-ended learning. The laboratory manual has been specifically designed with the following objectives:

- To complement and reinforce concepts learned in lecture

- To conduct experiments using "green" chemical principles—less hazardous or toxic materials, smaller sample sizes, reduction of waste, etc.

- To build student confidence in performing safe and proper laboratory skills and techniques

- To encourage a greater appreciation and understanding of the proper use of lab equipment, glassware, and instrumentation

- To develop computer skills in data acquisition and analysis

- To promote critical thinking in the design, execution, and analysis of laboratory experiments

- To introduce the organizational use of the laboratory notebook in preparation for technical writing in lab reports

- To provide a lab manual at the lowest possible cost to students

The objective of the laboratory component of the course is for the chemistry student to gain the necessary skills and confidence to work safely, efficiently, and independently in any future chemistry laboratory.

PREFACE

SAFETY

The chemistry laboratory is potentially a hazardous place and in order to have a safe chemistry laboratory experience for everyone, students must know and adhere to the following guidelines.

Before coming to lab
- Students should be thoroughly familiar with the experiment being performed by reading the entire procedure ahead of time. Be familiar with the hazards posed by chemicals, apparatus, and procedures. Know how to protect yourself and others.

- Students should come to the lab properly attired. This means wearing closed-toe shoes that cover the entire foot, tying long hair back, and removing dangling jewelry. Avoid loose fitting clothing, bulky sweaters, synthetic fabrics (due to flammability concerns), and clothing that expose bare skin such as mid-drifts, short skirts or short shorts.

- Students should know the location of safety items such as the safety shower, eye wash stations, first aid kit, fire extinguishers, material data safety sheet (MSDS), chemical-spill kits, and exits. In addition, students should know proper procedures for using the safety shower and eye wash station, and know what to do in case of an accident.

Working in the lab
- Chemical splash goggles, that provide a complete, snug seal around the eyes, MUST be worn in the laboratory area at all times. Safety glasses or spectacles do not provide adequate protection from chemical splashes and cannot substitute for safety goggles.

- Eating, drinking, chewing gum or tobacco, smoking, or applying cosmetics is not allowed in the lab areas. Avoid skin contact with chemicals. While in lab, avoid touching your face (eyes, mouth and nose). At no time should students taste chemicals and, absolutely no pipetting by mouth. Remember to wash your hands frequently throughout the lab, and especially when you leave the lab.

- Never perform unauthorized experiments or make changes to the quantities or types of chemicals used in the experiment. Any alteration must have prior professor approval. Remember, never leave your work unattended and never work alone in the laboratory.

- Avoid trip hazards. That means keeping your book bags, sweaters or other personal items off the floor and out of the walkways and storing them in the cubbies where provided. Make sure that locker drawers are kept closed when not in use.

- When conducting laboratory activities, be sure to carefully read labels on all reagent bottles, notify your professor of all accidents no matter how small, and report and clean-up all chemical spills. Do not pour excess chemicals back into the reagent bottles. Never remove chemicals from the laboratory without proper authorization.

- When moving about in the laboratory, walk slowly and anticipate other students backing up or changing directions suddenly. If you stumble or fall while carrying glassware or chemicals, try to project the items away from you and others.

SAFETY

- Good housekeeping is part of safety and good laboratory practices. This includes keeping your immediate area clean and clutter free, avoiding drips, drizzles and spills when transferring liquid chemicals, and maintaining a professional attitude. Keep chemicals and glassware away from the edges of the lab bench. Dispose of chemical wastes, broken glass, and paper towels in appropriate containers.

- Take extra precaution when using glassware and heat. Be sure that the glassware used is free from cracks and other defects. When heating test tubes, be careful not to point the test tube at yourself or another student. Protect your hands with paper towels and use lubrication when inserting thermometers and tubing into stoppers.

- Chemicals that give off toxic, flammable, or offensive vapors should be used in the fume hood. Keep your head out of the hood and be sure that the window sash is pulled down as far as possible during use. The sash should be completely pulled down when not in use.

Waste disposal

- Solid and chemical wastes should be disposed of in the labeled chemical waste container found in the fume hood. They should never go down the drain, unless specifically instructed to do so.

- Used paper towels, used matches (be sure to wet them first), etc should be disposed of in the regular trashcan.

- Broken glassware should be placed in the specially marked broken glassware containers. They should never be put into the regular trashcan.

In case of accidents

- Inform your professor of any accidents, no matter how small. Seek immediate medical attention following the accident as instructed.

Chemical spills/splashes:

- For small liquid spills on the skin, wash the affected area immediately with flowing water for 15 minutes

- For spills covering a large part of the body, immediately get underneath the chemical safety shower. Remove any contaminated clothing while under the shower and deluge the affected body area with water for at least 15 minutes.

- For chemical splashes in the face while wearing goggles, immediately go to the eye wash station and flush the area with water for 15 minutes. Do not remove goggles until the chemicals have been washed off. If chemicals got into the eye, pry the eyes open and flush the eyeball with water from the eyewash for at least 15 minutes.

SAFETY

Spill Clean Up:

o   For solid spills, use a dustpan and broom to sweep up the chemicals and dispose of the chemicals in the chemical waste container. Never use your hands to scoop up the chemicals. For spills in the balance, turn off the balance and use the brush to gently sweep the chemicals onto the lab bench first.

o   For liquid spills on the lab bench or floor, use paper towels or a special absorbent (in the chemical spill kit) to absorb the chemicals, following professor's directions. For acids and bases, neutralize the spills. If a flammable material is spilled, immediately extinguish all flames and turn off all electrical appliances. Once the area has been properly treated, wash the area with soap and water and wipe dry. Used paper towel or special absorbent must be disposed of in the chemical waste container.

https://tinyurl.com/MTSACchemlab-acidspill
https://tinyurl.com/MTSACchemlab-basespill

Fires/burns:

o   A small fire in a container can be suffocated by placing a watch glass over the mouth of the container. Remove any possible flammable materials away from the area and turn off any electrical appliances and sources of heat.

o   Be prepared to move away and evacuate the area if the fire is burning over a large area or cannot be smothered quickly. Remember to use stairs and not the elevator. Do not use the fire extinguisher unless you have been specifically trained to do so.

o   If your clothing catches on fire, stop, drop and roll to smother the flames. The fire can also be put out using the safety shower. Once the fire is put out, wrap the person in a blanket or coat to prevent shock and exposure. Place clean, wet, cold cloths on burned areas.

Cuts:

o   For a small cut, wash the area with soap and water. Apply pressure to stop bleeding if needed and cover the wound with a Band-Aid.

o   For larger cuts with lots of bleeding, compress the wound with a cloth or pad to control bleeding. Elevate the injury above the heart if possible and wrap the person in a blanket or jacket to prevent shock.

Understanding Chemical Labels

The chemicals that you will use in this course are labeled according to the Globally Harmonized System (GHS) which is the international standard for chemical labels. The labels use words and pictograms to identify safety issues associated with each chemical. Each chemical label will contain the following information:

- Product Identifier (chemical name)
- Signal Words ("Danger" or "Warning")
- Hazard Statements (type and degree of hazard)
- Precautionary Statements (recommended measures to prevent or minimize adverse effects)
- Supplementary Information
- Pictograms

There are nine different pictograms that represent the various types of hazards that a chemical may pose. The pictograms and their meanings are shown below.

Health Hazard	Flame	Exclamation Mark
• Carcinogen • Mutagenicity • Reproductive Toxicity • Respiratory Sensitizer • Target Organ Toxicity • Aspiration Toxicity	• Flammables • Pyrophorics • Self-Heating • Emits Flammable Gas • Self-Reactives • Organic Peroxides	• Irritant (skin and eye) • Skin Sensitizer • Acute Toxicity (harmful) • Narcotic Effects • Respiratory Tract Irritant • Hazardous to Ozone Layer (Non-Mandatory)
Gas Cylinder	Corrosion	Exploding Bomb
• Gases Under Pressure	• Skin Corrosion/Burns • Eye Damage • Corrosive to Metals	• Explosives • Self-Reactives • Organic Peroxides
Flame Over Circle	Environment (Non-Mandatory)	Skull and Crossbones
• Oxidizers	• Aquatic Toxicity	• Acute Toxicity (fatal or toxic)

The following is an example of a chemical label for 3M HCl.

Whenever you use a chemical in the laboratory, you should carefully read the label to make sure that you are using the correct chemical, that you understand the risks associated with using that chemical, and that you know what to do in case of an accident.

More detailed information about each chemical is available in the Safety Data Sheet (SDS) for that chemical. SDS's for all of the chemicals used in the chemistry laboratory are stored in a binder in the prep room. If you would like to view the SDS for any chemical, just ask your instructor.
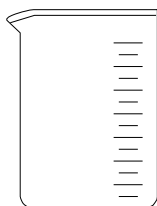
References:
Based on the Mt. San Antonio College Chemistry Department Laboratory Safety Rules
Adapted from *Safety in the Academic Chemistry Laboratories*, Volume 1, 7th Edition, a publication of the American Chemical Society and the Joint Board-Council on Chemical Safety.
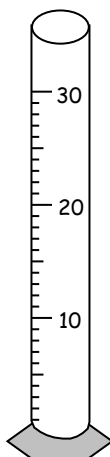
SAFETY

(This page intentionally blank.)

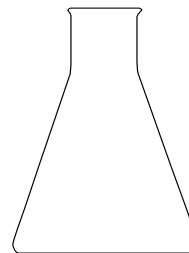(This page intentionally blank.)

EQUIPMENT

Beaker
- come in various sizes
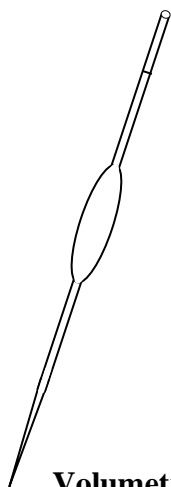- markings allow measurement of different volumes
- not very precise

Graduated Cylinder
- come in various sizes
- calibration markings allow measurement of different volumes
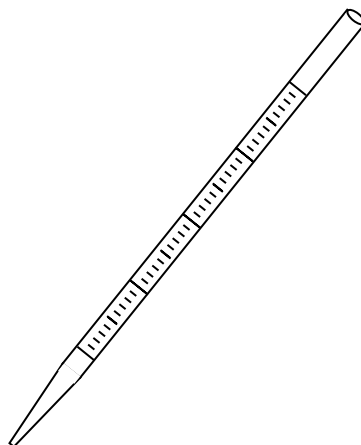- medium precision

Erlenmeyer Flask
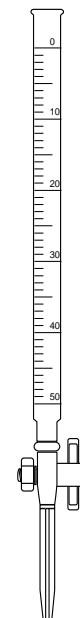- come in various sizes
- not very precise

Volumetric Pipet
- come in various sizes
- single calibration mark allows delivery of only one specified volume
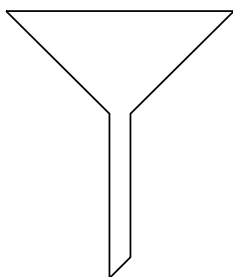- very precise

Graduated Pipet
- come in various sizes
- calibration markings allow measurement of different volumes
- very precise

Buret
- come in various sizes
- calibration markings allow delivery of any desired volume within calibration range
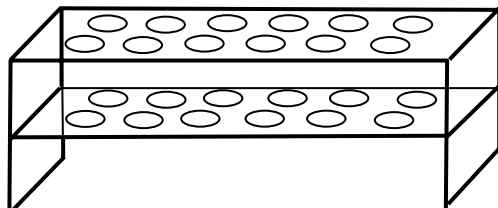
Funnel
- For pouring liquids into flasks

9

EQUIPMENT

Test Tube
– used for holding solutions

Test Tube Rack
– used for holding test tubes
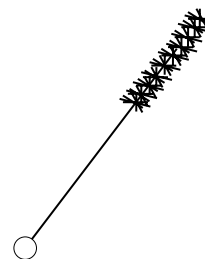
Test Tube Brush
– used for cleaning test tubes
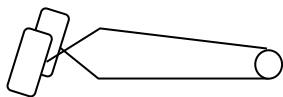
Test Tube Holder
– used for holding a test tube while heating in a Bunsen burner flame
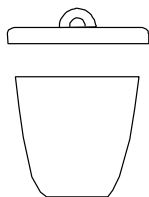
Crucible with Lid
– used for heating solids to high temperatures

Crucible Tongs
– used for handling hot objects
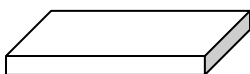
Ceramic Fiber Square
– protects lab bench from hot items

Wire Gauze
– placed on ring stands to support flasks while heating
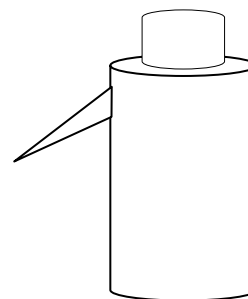
Wash Bottle
– fill with de-ionized water for final rinse of glassware
– for adding water to flasks
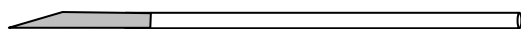
Stirring Rod with Rubber Policeman
– for stirring solutions
– for scraping solids
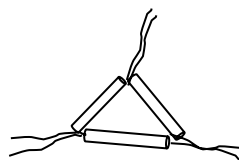
Clay Triangle
– Sits on a ring clamp for holding crucibles or funnels

EQUIPMENT

Thermometer
- measures temperature
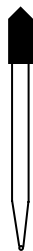
Medicine Dropper
- for dispensing drops of solution
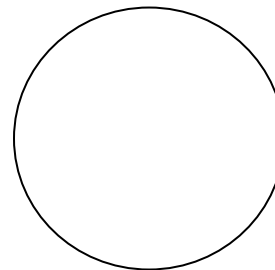
Filter Paper
- separates solids from liquids

Rubber Stopper
- come in various sizes
- used to close test tubes and flasks
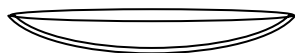
Watch Glass
- used to cover beakers
- used to hold/observe solids
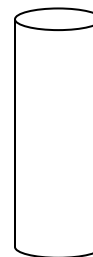
Vials of Litmus Paper
- come in two colors—red and blue
- for determining the acidity/basicity of solutions

Spatula
- used to scoop solids
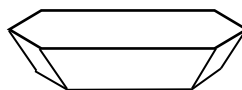
Weigh Boat
- used to weigh solids
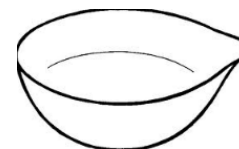
Evaporating Dish
- used to evaporate liquids

EQUIPMENT

(This page intentionally blank.)

TECHNIQUES

The following sections review the proper use of some common laboratory equipment and the theory behind some common laboratory techniques. Links to YouTube videos demonstrating several of these techniques are also provided.

Heating

It is sometimes necessary to heat chemicals in the laboratory. The laboratory has hot plates that can be conveniently used to heat solutions. However, for more precise control of heating, or when heating solid materials, a Bunsen burner is generally used. Bunsen burners burn natural gas (CH_4) distributed to the laboratory benches via marked taps. To use a Bunsen burner, first check to make sure that the tubing is firmly attached to the base of the burner and is free from holes or tears. Attach the tubing to the natural gas valve on the bench top and place the Bunsen burner toward the middle of the bench and away from any combustible materials.

There are two valves on the Bunsen burner, one at the base of the unit for controlling gas flow and one on the shaft for controlling the amount of air (O_2) that mixes with the gas.

tubing

air flow valve
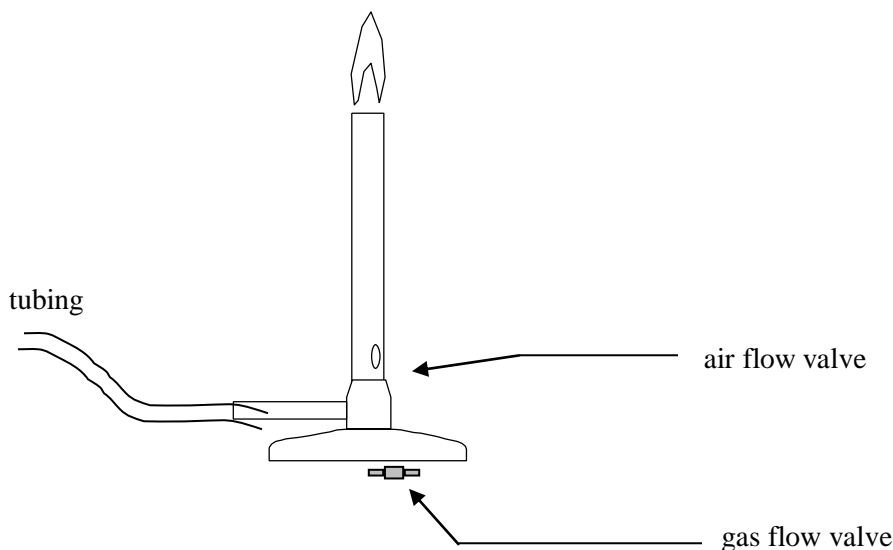
gas flow valve

Figure A

Open the gas flow valve on the Bunsen burner about half way and close the air flow valve. Open the natural gas valve on the bench top until you can hear the hissing of the gas flow through the Bunsen burner. Use a striker to ignite the flame by holding the striker about 2 inches above the top of the Bunsen burner and squeezing the striker handle to generate a spark. If you are unsuccessful in igniting the flame after about 15-20 seconds, turn off the gas and wait for the gas to dissipate before trying again (an excess buildup of gas may result in a flash flame and you losing your eyebrows!). Once the flame is ignited, use the air control valve to increase the amount of oxygen in the flame until you have an almost invisible blue flame with a darker blue inner cone. Never leave a lit Bunsen burner unattended and make sure that all glassware being heated is properly secured using ring-stands and/or clamps. Also, be sure to let the Bunsen burner cool before putting it away.

TECHNIQUES

Cleaning and Labeling Glassware

Glassware should be cleaned before use. A solution of detergent can be used to clean most glassware followed by repeated rinsing with tap water and a final rinse with deionized water. Glassware is clean when it drains without leaving droplets on the walls. If the above procedure does not adequately clean your glassware, ask your instructor for assistance.

There is a container of grease pencils on each laboratory bench. Use these to label your glassware when doing experiments. The grease marks are most conveniently removed using a *dry* paper towel.

Reading Analog Scales

Many measurements that you make in the laboratory require you to read a value from an analog scale. It is important to report all measurements to the proper decimal place to convey the level of precision of the measurement. Whenever you read a marked scale, the last digit you report as significant is the one you estimate. (Record all the values associated with the marked scale + go out one more decimal place for the estimated digit.) Follow the steps below to properly record measurements read from an analog scale.

1. determine the value associated with marked divisions on the scale
2. decide to what level of precision you can estimate values between the marks and mentally divide the space between markings
   a. a general rule of thumb is to estimate to 1/10th of the marked divisions
   b. sometimes it makes more sense to estimate to a half, fourths or fifths depending on how close the markings are together and what the scale is
3. read the measurement by reading the scale and the estimated digit based on your imagined scale
4. record the measurement and its associated precision (the last decimal place of the measurement should be the same decimal place as the ± precision)

10 mL

Level of meniscus
= 9.34 mL
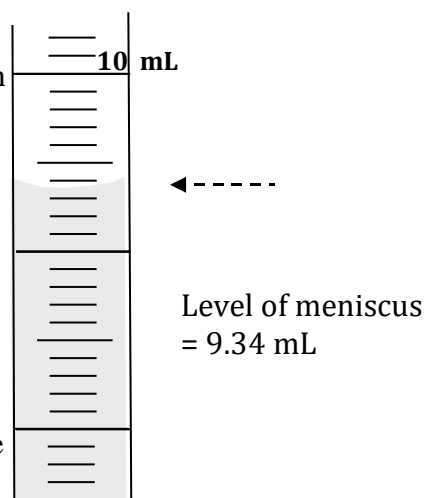
Figure B

When reading marked scales on volumetric glassware, always read the bottom of the meniscus (the curved surface that liquids form in glass tubes) at eye level.

TECHNIQUES

Volumetric Flasks

Volumetric flasks **(Figure C)** are designed "to contain" (TC) a specific volume of liquid. Volumetric flasks have just one calibration mark in the neck of the flask. Volumes using volumetric flasks should generally be reported to ± 0.01 mL. The following list details the proper procedures for using a volumetric flask to make a solution of known molarity from a solid solute.

- Clean flask and rinse with DI (or other solvent)
- Quantitatively transfer pre-weighed solid solute to volumetric flask using a funnel. Rinse the weigh boat and funnel with solvent into the flask to ensure that all solid is transferred to the flask.
- Dissolve the solid by adding a small portion of solvent and swirling the flask; continue swirling and adding solvent until the solid is dissolved before the level reaches the neck of the flask
- Add solvent from squirt bottle until the level approaches the calibration mark
- Add final drops of solvent with a dropper until the bottom of the meniscus is at the calibration mark when read at eye level.
- After the desired level is achieved, cap the bottle and invert repeatedly while holding the cap in place to mix completely.
- Never heat solutions in volumetric flasks because heat can cause the glassware to warp and affect the volume
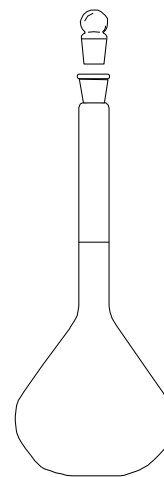
Figure C

Graduated Cylinders

Graduated cylinders (**Figure D**) are designed either "to contain" (TC) or "to deliver" (TD) variable volumes of liquid. They have many calibration marks. You must look carefully at the markings to determine the range and level of precision for that particular graduated cylinder. Remember to estimate between marked divisions and record the volume to the correct number of significant figures (the known plus the estimated digit).
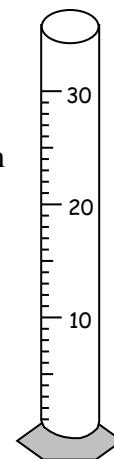
Figure D

<center># TECHNIQUES</center>

Volumetric Pipets

Volumetric pipets (**Figure E**) are designed to deliver (TD) a specific volume of liquid. Volumetric pipets have just one calibration mark. Volumes using volumetric pipets should generally be recorded to ± 0.01 mL. The following list details the proper procedures for using a volumetric pipet to transfer a specific volume of solution.

- o Check for cleanliness by filling with DI water and letting drain. If any drops are left inside, the pipet must first be cleaned. See your instructor for cleaning instructions.
- o In all of the following, be careful not to get solution inside the pipet pump or bulb. If this should happen, inform your instructor so that the proper steps can be taken to clean the pump and minimize damage. Pumps or bulbs should not be left on the pipets when they are not in use. Also be careful not to let your pipet roll off the benchtop.
- o To condition the pipet, pour a small amount of reagent into a separate flask. Use a pipet pump or suction bulb to draw some reagent into the pipet, remove the bulb and tilt and swirl the pipet so that the inner walls of the pipet are rinsed with the solution past the calibration mark. Discard rinse solution to waste and repeat two more times.
- o To load the pipet, pour the required amount of stock reagent into a new clean, dry beaker. Draw the reagent from the beaker into the pipet **past** the calibration line using the suction bulb or pump. Hold the pipet tip against the bottom of the flask and then quickly replace the bulb with your index finger for better control.
- o Wipe the outside of the tip with a Kimwipe.
- o Touch the tip of the pipet to the edge of the reagent beaker (above the solution level) and then let drain to the calibration line so that bottom of the meniscus is at the calibration marking when viewed at level.
- o Drain the liquid into the desired receiving flask while holding the tip against the wall of the flask [do not blow out]
- o Rinse the pipet with DI water after use (do not let solutions dry inside)

Figure E

https://tinyurl.com/MTSACchemlab-volpipet
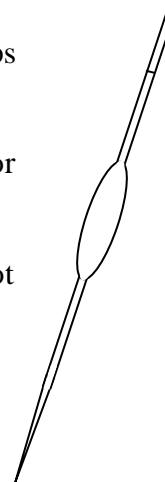
Graduated Pipets (Mohr)

Graduated pipets (**Figure F**) are designed to deliver (TD) variable volumes of liquid. Graduated pipets have many calibrations markings. You must look carefully at the markings on the pipet to determine the range and level of precision for that particular pipet. Remember to estimate between marked divisions and record the volume to the correct number of significant figures (the known plus estimated digit). The following procedures describe two ways to use a graduated pipet to deliver a specific volume of liquid.

https://tinyurl.com/MTSACchemlab-Mohrpipet

Figure F

<center>16</center>
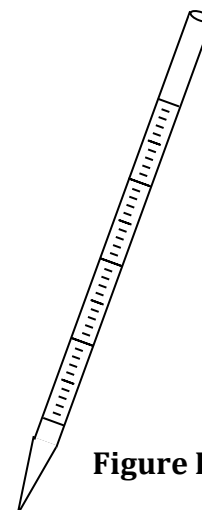
Graduated Serological Pipet (Blow-out)

- o This type of pipet has a **double blue line** at the top.
- o Examine the calibration markings and determine the correct loading to achieve the desired delivery. For example, if you are using a 10-mL graduated pipet and want to deliver 2.00 mL, load the pipet to the 8.00 calibration line (10.00mL-2.00mL = 8.00 mL) **(Figure G)**
- o Check for cleanliness by filling with DI water and letting drain. If any drops are left inside, the pipet must first be cleaned. See your instructor for cleaning instructions.

10-mL graduated pipet
set to deliver 2.00 mL
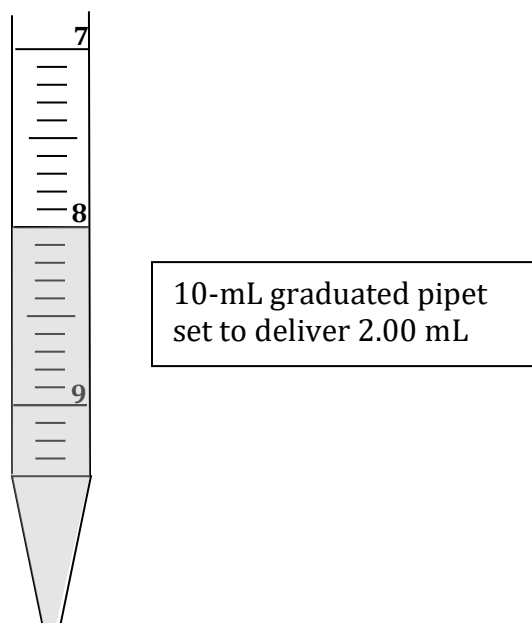
Figure G

- o To condition the pipet, pour a small amount of reagent into a separate beaker. Use a pipet pump or suction bulb to draw some reagent into the pipet, remove the bulb and tilt and swirl the pipet so that the inner walls of the pipet are rinsed with the solution past the desired calibration mark. Discard rinsings to waste and repeat two more times.
- o To load the pipet, pour the required amount of stock reagent into a new clean, dry beaker. Draw the reagent from the beaker into the pipet **past** the desired calibration line using the pump or suction bulb. Hold the pipet tip against the bottom of the flask and then quickly replace the pump or bulb with your index finger for better control.
- o Wipe the outside of the tip with a Kimwipe.
- o Touch the tip of the pipet to the edge of the reagent beaker and then let drain to the desired calibration line so that bottom of the meniscus is at the calibration marking when viewed at level.
- o Drain the liquid into the desired receiving flask while holding the tip against the wall of the flask. Use the pipet pump or bulb to force the last drop of liquid out of the pipet.
- o Rinse the pipet with DI water after use (do not let solutions dry inside)

https://tinyurl.com/MTSACchemlab-serologicalpipet

Volume by Difference (less common)
- o Follow the same procedures described above for conditioning the pipet.
- o To load the pipet, pour the required amount of stock reagent into a new clean, dry beaker. Draw the reagent from the beaker into the pipet **past** the desired level using the suction bulb or pump. Hold the pipet tip against the bottom of the flask and then quickly replace the bulb or pump with your index finger for better control.
- o Wipe the outside of the tip with a Kimwipe.
- o Touch the tip of the pipet to the edge of the reagent flask and then let drain to the desired level. Read and record the volume to the proper place value.
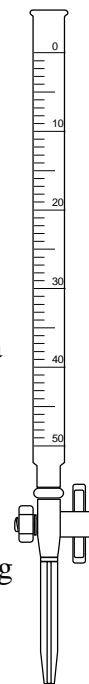- o Drain the desired amount of liquid into the receiving flask while holding the tip against the wall of the flask. Read and record the final volume in the pipet. The volume of liquid delivered is the difference between the final reading and the initial reading.
- o Rinse the pipet with DI water after use (do not let solutions dry inside)

Burets

Burets (**Figure H**) are designed to deliver (TD) a variable volume of liquid. The stopcock allows the chemist to control the flow of the solution and deliver a range of volumes precisely. Our burets have calibration markings every 0.1 mL from 0-25 mL and measurements using burets should be recorded to ± 0.01 mL. Note that the zero volume marking is at the top of the buret. Follow the procedures below to correctly use a buret to transfer solution.

- o Check the buret for cleanliness by filling with DI water and letting drain. If a drops are left in the buret, the buret needs to be cleaned.
- o Condition the buret with solution [3x] by pouring a small amount (about 2-3 mL) of solution into the buret using a funnel (stopcock closed). Rotate the bu horizontally and swirl the solution to coat the inner walls of the buret past the mark. Drain the rinse solution to waste through the stopcock.
- o Fill the buret with fresh solution to above the 0 mark using a funnel (being careful not to overflow). Eliminate air bubbles in the tip before use by allowi solution to drain through the tip, or by opening the stopcock and giving the buret a careful shake downward to dislodge the bubbles (be careful not to brea the tip—ask your instructor for help if needed).
- o Check the buret for leaks by reading and recording the initial volume, waiting minutes, and reading volume again to make sure level is the same
- o Always start the experiment **below** the 0.00 mL mark (initial readings of 0.00 mL are not recommended and may not be accepted by some professors). Record the initial volume by reading the bottom of the concave meniscus using a dark card to help you see the level. Avoid parallax by keeping your eye on t same level as the solution. **(Figure I)**
- o Drain the liquid slowly
- o For titrations, deliver a fraction of a drop near the end point
  - ▪ Let a partial drop form on the tip and then squirt off with DI or
  - ▪ Rapidly rotate the stopcock 360°° to deliver partial drop
- o Record the new volume reading. The volume of solution delivered is equal to the final volume reading minus the initial volume reading.
- o Rinse the buret with DI water and store upright and filled with DI water.

Figure H

Level of meniscus = 9.68 mL

Figure I

https://tinyurl.com/MTSACchemlab-buret1
https://tinyurl.com/MTSACchemlab-buret2
https://tinyurl.com/MTSACchemlab-buret3
https://tinyurl.com/MTSACchemlab-buret4
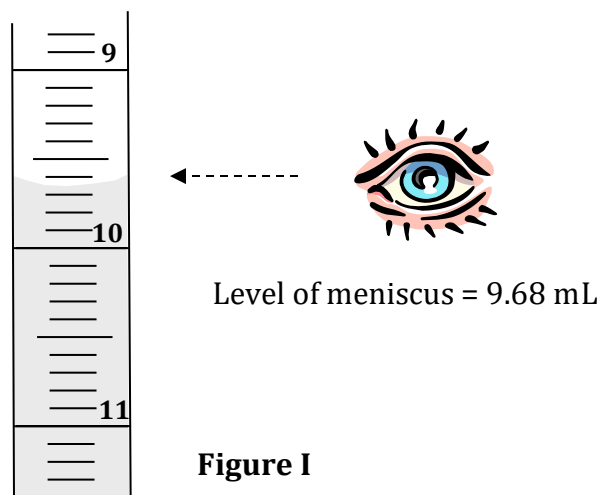
Analytical Balances

Analytical balances are designed to accurately and precisely weigh samples to a tenth of a milligram.

General guidelines:
- o Weigh only materials at room temperature and close the balance case during weighing for the most precise measurements.
- o Keep the balance pan and balance room clean. Clean up and/or report any spills immediately for proper cleanup by instructor.
- o Use weigh boats or weighing papers to hold solid materials.
- o Add solids to the weigh boat or weighing paper outside the balance case to avoid spilling inside the balance case.
- o Use the same balance for all measurements and close the sliding glass doors for best precision

Weighing by Taring:
- o Place the weigh boat on the balance pan and press the "tare" button to zero the balance.
- o Remove the weigh boat and add the approximate amount of solid to the boat outside the balance case.
- o Return the boat to the pan to weigh the solid. Add or remove solid as necessary (outside the case) until the desired mass is achieved. (Excess solid must be disposed of as waste; never returned to the stock bottle.)

Weighing by Difference:
- o Tare the balance with nothing on the balance pan.
- o Determine the mass of the empty weigh boat.
- o Remove the weigh boat and add the approximate amount of solid to the boat outside the balance case.
- o Return the boat to the pan to weigh the solid (the mass of the solid will be the mass of the boat with the solid minus the mass of the empty boat). Add or remove solid as necessary (outside the case) until the desired mass is achieved. (Excess solid must be disposed of as waste; never returned to the stock bottle.)

https://tinyurl.com/MTSACchemlab-balances
https://tinyurl.com/MTSACchemlab-toploading
https://tinyurl.com/MTSACchemlab-analytical
https://tinyurl.com/MTSACchemlab-solidtransfer

Solution Preparation

Many experiments require you to make solutions of a given concentration from either a pure solid or liquid reagent, or from a concentrated stock solution containing the desired reagent. The concentration of a solution tells you how much *solute* is dissolved in a given amount of *solution* (or *solvent*). There are several common ways to express the concentration of a solution—percentages, molarity, and molality. The definitions of each of these are reviewed here, as well as the theory and calculations involved in making solutions from pure reagents or by diluting concentrated stock solutions.

https://tinyurl.com/MTSACchemlab-solutiontransfer

Percent Concentration

Percentages always express the ratio of the "part" to the "whole" in terms of 100 units. For solutions, this means the amount of solute divided by the amount of solution and multiplied by 100%. Percent solution compositions are commonly done on a mass to mass basis, a volume to volume basis, or a mass to volume basis as shown below. When solving for a percent concentration, use the equation form. However, if you're given a percent value and asked to calculate another quantity from it, it is more convenient to use the relationship as a conversion factor.

Table 1: Percent Concentration Equations

Relationship	Equation:	Conversion Factors:
mass % (%m/m)	$= \dfrac{\text{mass solute}}{\text{mass solution}} \times 100\%$	X mass % A = $\dfrac{\text{x grams A}}{100\text{ g solution}}$ -or- $\dfrac{100\text{ g solution}}{\text{x grams A}}$
volume % (%v/v) (esp. when solute is a liquid or gas)	$= \dfrac{\text{volume solute}}{\text{volume solution}} \times 100\%$	X volume % A = $\dfrac{\text{x mL A}}{100\text{ mL solution}}$ -or- $\dfrac{100\text{ mL solution}}{\text{x mL A}}$
mass/volume % (%m/v) or (%w/v)	$= \dfrac{\text{mass solute}}{\text{volume solution}} \times 100\%$	X %m/v A = $\dfrac{\text{x grams A}}{100\text{ mL solution}}$ -or- $\dfrac{100\text{ mL solution}}{\text{x grams A}}$

Making a solution to a given mass % involves weighing out a known mass of the solute and adding solvent (usually water) until the total mass of the solution is the desired mass. The mixture is then mixed to dissolve the solute. For example, to make 100 grams of a 15.00% by mass sodium chloride solution, you would weigh out 15.00 grams of sodium chloride in a 250-mL beaker or Erlenmeyer flask and add water until the total mass of the solution (sodium chloride + water) was 100.00 grams.

To prepare a solution of a given volume %, you need to use **volumetric glassware** for measuring the solute and for containing the final solution. One hundred mL of a 15.00% by volume solution of ethanol would be prepared by using a **graduated pipet** to transfer 15.00 mL of ethanol to a 100.00 mL **volumetric flask** and filling to the mark with water (total solution volume = 100.00 mL) and then thoroughly mixing.

Preparing a solution of a given mass/volume % involves weighing out the desired mass of solute and dissolving it in a **volumetric flask**. For example, 50.00 mL of a 15.00% (m/V) solution of sodium chloride would be prepared by weighing out 7.50 grams of sodium chloride and transferring it to a 50.00 mL volumetric flask. Next a small amount of water is added to dissolve the solid. More water is added with swirling until the solution level is at the calibration mark.

Molarity
The molarity of a solution tells you how many moles of the solute are dissolved per liter of solution. This is the relationship most commonly used by chemists as moles are the unit of choice for stoichiometry calculations.

Table 2: Molarity Equations

Relationship	Equation	Conversion Factors
molarity	$M = \dfrac{\text{moles solute}}{\text{L solution}} = \dfrac{n}{V}$	x M A = $\dfrac{\text{x moles A}}{1\text{ L solution}}$ -or- $\dfrac{1\text{ L solution}}{\text{x moles A}}$
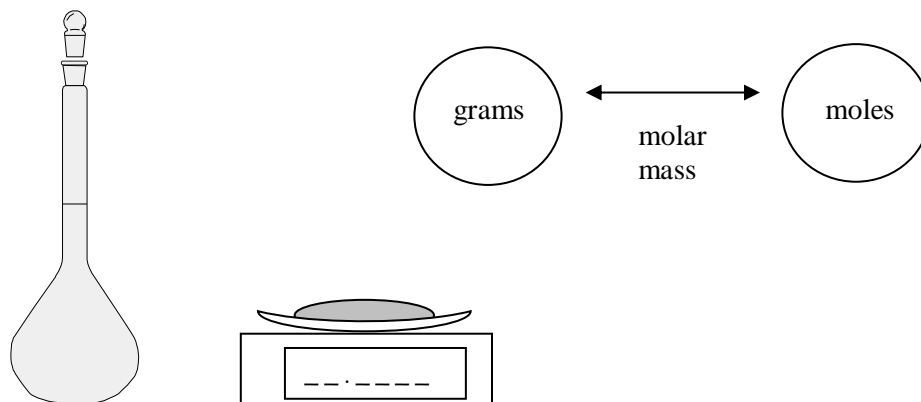
Figure J

Volumetric flasks are used for making solutions of known molarity because they are designed **to contain** a certain volume of **solution**. **Analytical balances** are also used to weigh out the required mass of **solute** to achieve the desired number of moles of solute. The weighed solute is added to the volumetric flask and dissolved in a small amount of solvent. More solvent is added with swirling until the solution level is at the base of the neck of the flask. The final drops of solvent are added with a dropper until the bottom of the meniscus is at the calibration line, then the flask is capped and inverted to mix thoroughly.

Dilution

Dilution is the process of taking a more concentrated solution and adding solvent to it to reduce the overall concentration. In a dilution, solute is *transferred* from the stock solution to the dilute solution, so the moles of solute in the diluted solution (n) is equal to the moles of solute transferred.

$$M = \frac{n_{transferred}}{V_{flask}}$$

$$n_{transferred} = M_{stock} \cdot V_{pipet}$$

concentrated stock solution
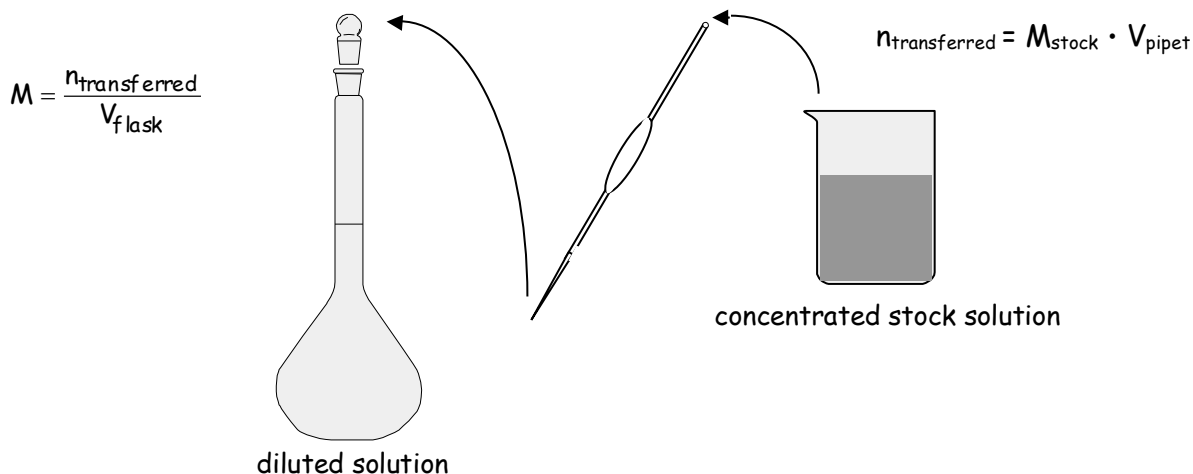
diluted solution

Figure K

TECHNIQUES

Volumetric flasks and **volumetric pipets** are generally used for diluting solutions. A precise volume of stock or concentrated solution is transferred via graduated or volumetric pipet to a volumetric flask and then the volumetric flask is filled to the mark with solvent. The volume of the final solution is the volume of the volumetric flask, and the moles of solute in the diluted solution is obtained from the concentration and volume of stock solution transferred. (Experimental note: The transfer pipet or buret should be conditioned with the concentrated stock solution before use. The volumetric flask should NOT be conditioned because this would add an unknown extra amount of solute. It is OK if the volumetric flask is wet with solvent, however, as we will add solvent anyway to the mark.)

In some cases, dilutions are made by assuming that the volumes of stock solution and solvent are additive (not strictly true, but often a good enough assumption given the other errors involved in the experiment, especially if the solutions are already somewhat dilute). In other words, a volumetric flask is not used. In these cases, remember that the volume of the dilute solution is equal to the sum of the volumes of the transferred stock solution and the added solvent.

The equation $M_1V_1=M_2V_2$ is often used in solving dilution problems. This equation works because, as stated above, the moles of solute taken from the stock solution equals the moles of solute that end up in the dilute solution ($n_1 = n_2$ and $n=MV$). Be careful, however, to correctly assign each variable and **never use this equation for solving stoichiometry problems** because for chemical reactions, n_1 rarely equals n_2!

Titration

Titration is a method of volumetric analysis and has at its heart a chemical reaction for which there can be some indication of completion (see equation below). In **Figure L** below, the reactant A is dissolved in the *titran*t solution in the buret. The reactant B is dissolved in the solution in the receiving flask. The buret delivers a variable and precisely known volume of titrant. If the molarity of the titrant is known, then the moles of reactant A delivered can be determined from $n = MV$.

$$xA(aq) + yB(aq) \rightarrow products$$
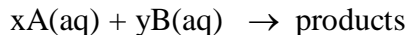
The equivalence point of the titration occurs when stoichiometrically equivalent moles of A have been added to B. For example, if x = 2 and y = 1, then the equivalence point is when twice as many moles of A have been added as the original moles of B in the flask. This relationship allows the calculation of the moles of B originally in the flask from the molarity and volume of titrant added.

A key aspect of titrations is knowing when to stop (i.e. when the equivalence point has been reached). Sometimes the reaction itself provides a visual clue to the equivalence point, but more often, chemical dyes known as indicators are be used for this purpose. In the case of

titrant :
solution A
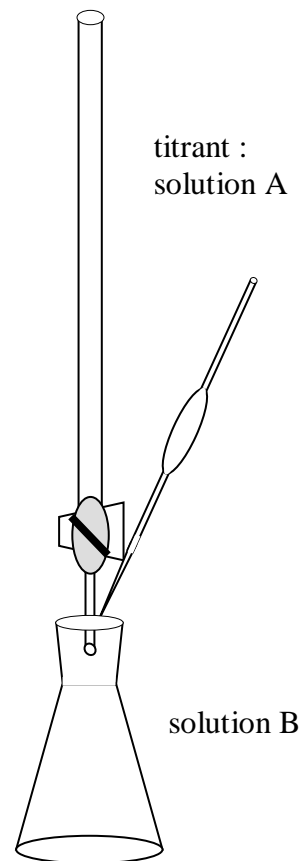
solution B

Figure L

acid-base titrations, indicators are molecules that undergo a color change depending on the pH of the solution. Table 3 shows some common indicators for acid-base titrations. The goal is to stop the titration as close as possible to the equivalence point, so indicators are chosen that change color as close as possible to the pH at the equivalence point. However, there is always some error (indicator error) or difference between the endpoint of the titration (when the titration is stopped due to the indicator) and the true equivalence point.

Table 3: pH Ranges of Some Common Acid-Base Indicators

Indicator	pH range	color change
Crystal Violet	0-1.8	yellow to blue
Thymol Blue	1.2-2.8	red to yellow
Bromphenol Blue	3.0-4.6	yellow to blue
Methyl Red	4.8-6.0	red to yellow
Bromthymol Blue	6.0-7.6	yellow to blue
Phenolphthalein	8.2-10	colorless to pink
Alizarin Yellow R	10.1-12.0	yellow to red

https://tinyurl.com/MTSACchemlab-titration1
https://tinyurl.com/MTSACchemlab-titration2

Vacuum Filtration

Filtration is a method of separating solid particles from a mixture by passing the mixture through a filter (either paper or sintered glass) in a funnel. The solid particles are retained on the filter and the rest of the solution passes through the filter and is usually collected in a flask. In gravity filtration, the solution is poured into the funnel and allowed to drip through by gravity alone. This can be a very slow process if the filter is very fine and/or there are a lot of solid particles that clog the filter. When this happens, vacuum filtration can be used. In vacuum filtration, the funnel is inserted into a filter flask through a rubber stopper or adapter. A filter flask looks like an Erlenmeyer flask except that it has a side arm for connecting tubing. The other end of the tubing is connected to the vacuum line on the bench top. When the vacuum line is opened, the vacuum pulls on the solution in the funnel causing it to go through the filter faster. Ideally, you should also set up a "trap" between the filter flask and the vacuum line as shown in **Figure M** to collect any solution that might get pulled into the vacuum line.

Tubing

Tubing to vacuum line

Filter flask
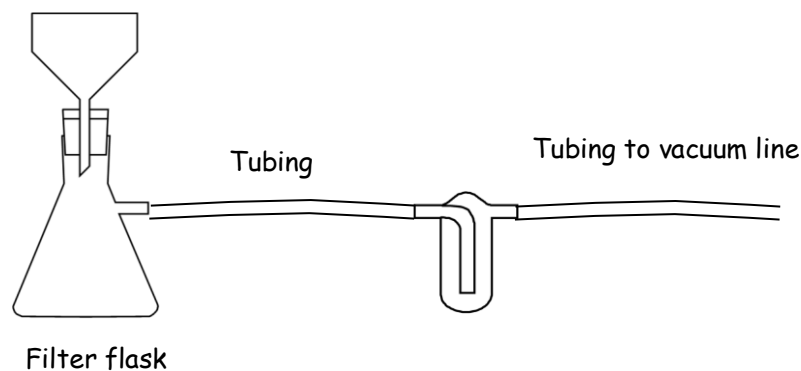
Figure M

Spectrophotometry and Beer's Law
Spectrophotometry is an easy and quick way to determine the concentration of solute in a solution provided the solute absorbs light. Lab instruments for spectrophotometry are available that measure absorbance of visible and/or ultraviolet light. Solutions that are colored and transparent, absorb light in the visible region of the electromagnetic spectrum (a solution that appears green, absorbs the complement of green which is red). The Mt. SAC chemistry department has two instruments for doing visible spectrophotometry—Spec 20's and Pasco Spectrometers. The instrument components and the theory behind the measurements of the two are very similar.

Spectrophotometric instruments commonly contain a light source, a wavelength selection component (prism or grating), a sample holder and a light/photon detecting device. The light detecting device measures the amount of light passing through the sample, I, and compares it to the amount of light passing through a "blank", I_o. The percent transmission value is converted to a quantity called absorbance, A, as shown in the equations below.

$$\%T = \left(\frac{I}{I_o}\right) \cdot 100\%$$

$$A = -log(T) = -log\left(\frac{I}{I_o}\right)$$

According to Beer's Law, the absorbance of a solution, A, is directly proportional to the concentration, c, of light absorbing solute in the solution. (One caveat is that the absorbance measured must be for a single wavelength or color of light.) The proportionality factor is known as the Beer's constant and is given the symbol *"b"*.

$$A = bc$$

Although b can be determined from a single measurement of the absorbance of a solution of known concentration, it is more common to make a series of measurements and construct a

TECHNIQUES

"Beer's Law Plot." In this method, a number of solutions are prepared of varying concentrations of solute and their absorbance at a particular wavelength is measured. A graph of absorbance (y-axis) vs. concentration (x-axis) is constructed. Solutions that follow "Beer's Law" will yield a graph with a linear relationship between absorbance and concentration. The slope of the best-fit line gives the constant **b**. This value of b is then used to determine the concentrations of solutions of unknown concentration from their measured absorbance according to:

$$b = slope\ of\ Beer's\ plot$$

$$c_{unknown} = A_{measured} / b_{from\ slope}$$

Beer's Law Plot

$$y = 3.74951x$$
$$R^2 = 0.99994$$

X-axis: **[X] (M)**
Y-axis: **Absorbance**
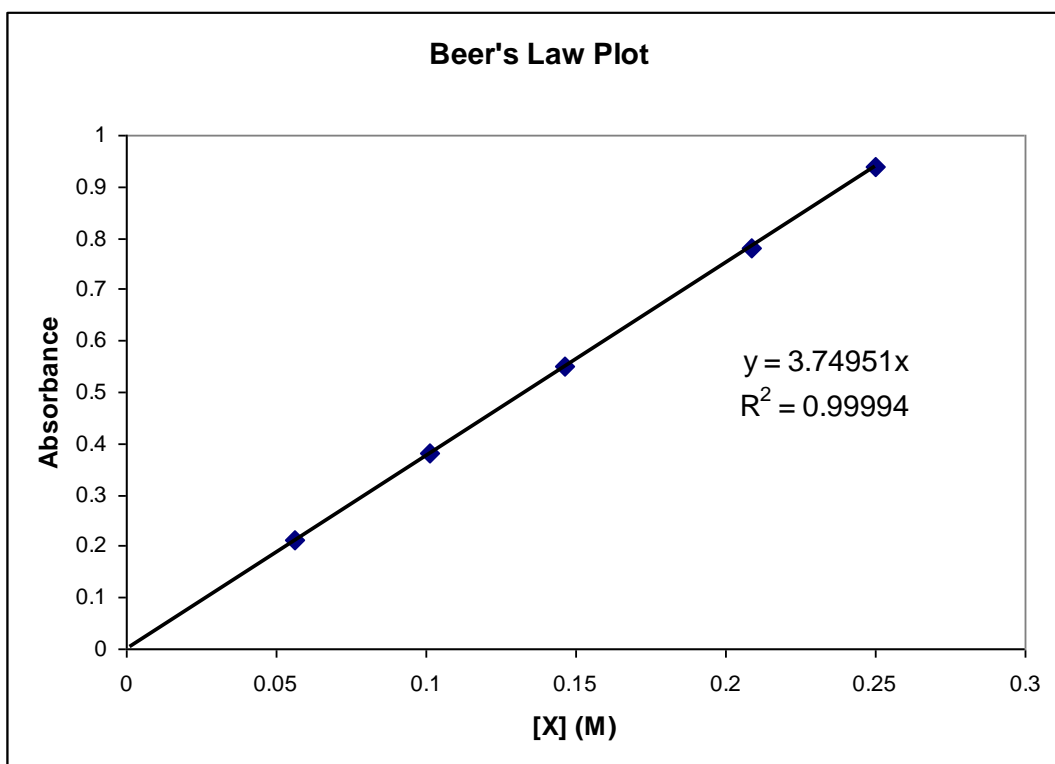
Figure N

Note that spectrometers and colorimeters actually measure a voltage from the light sensing device and convert that to a % transmission and/or absorbance. Therefore, when using a spectrometer or colorimeter, you must first calibrate the instrument using the uncolored solvent as a blank so that the instrument knows what voltage corresponds to 0% transmission (no light getting through) and 100% transmission (no light being absorbed by the analyte). Specific instructions for calibrating and using the Spec 20's and/or the MeasureNet colorimeters will be provided separately.

Measurement of pH

It is often useful to know the acidity or "pH" of a solution. The pH of a solution is related to the molar concentration of H^+ ions by

$$pH = -log\ [H^+]$$

The approximate pH of a solution can be determined by using various indicators (see titration section) and/or pH strips that have indicators in them. Recall that indicators are dye molecules that change color in different pH environments. The red and blue litmus strips in your chemistry lockers can indicate if the pH of a solution is less than or greater than 7 (an acidic solution will turn blue litmus paper red, while a basic solution will turn red litmus paper blue). Other types of strips with multiple different indicators can determine the pH of a solution to ± 1 pH unit. When a higher level of precision/accuracy is required, a pH electrode/meter are generally used.

A pH electrode contains a glass bulb that is sensitive to the concentration of H^+ ions—it adsorbs varying amounts of H^+ ions depending on the concentration of H^+ in solution. The electrodes we use also incorporate Ag/AgCl reference electrodes for measuring the voltage across the glass bulb due to the adsorbed charges. The inner filling solution in the electrode has a constant $[H^+]$ and therefore the voltage measured across the glass membrane by the reference electrodes is related to the $[H^+]$ in the solution being measured (outer solution).

Special care must be taken with the pH electrodes as the glass bulb can be easily damaged. It is important to soak the bulb in an appropriate solution (some electrodes are soaked in pH 7 buffer, while others require a KCl solution) between measurements to keep it from drying out. Rinse the electrode well with deionized water before and after measurements, but NEVER dry the electrode with a Kimwipe or paper towel as this can scratch the glass surface.

The pH electrode is connected to a pH meter that reads the voltage and converts it to the pH scale. pH meters must first be calibrated using buffers of known pH value (buffers are solutions that resist changes in pH). These buffers are commercially available at a variety of pH values. We have buffer solutions of pH = 4.0, pH = 7.0, and pH = 10.0. Generally a "two-point" calibration is performed. If you know your solution is acidic, you would calibrate your pH meter using the pH 4.0 and pH 7.0 buffers. On the other hand, if you know your solution is basic, you would calibrate the pH meter using the pH 7.0 and pH 10.0 buffer solutions. Specific instructions for calibrating the pH meters will be provided with the meters.

Pasco Spectrometers

The Chemistry Department has purchased Pasco Spectrometers for use in the Chemistry 50 and 51 Laboratory curriculum. These spectrometers can connect wirelessly through Bluetooth to computers, tablets or mobile devices, or connect directly to a laptop or computer via USB cables.

The software to run these devices is free and can be downloaded to your personal computer from the following website, or to your mobile device from your app store.

www.pasco.com

Spectrometry

Version: 2.2.1

Released 1/17/2019

Mac/Windows

Free Download

Free Apps

Download on the App Store   GET IT ON Google Play

Using Chromebooks?

The website shown above also offers video tutorials to help you learn how to use the spectrometer and the software. The following videos are recommended.

For Chem 50:
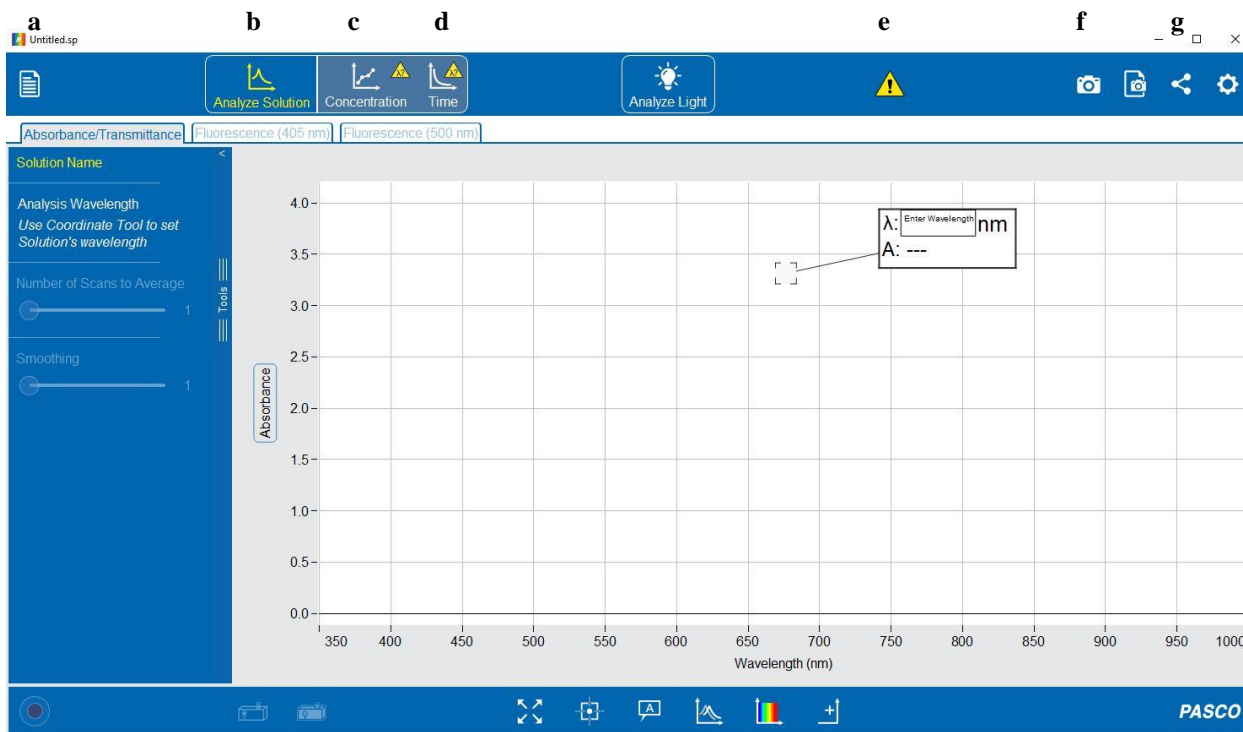– PASCO Spectrometer: Beer's Law

For Chem 51:
– PASCO Spectrometer: Beer's Law
– PASCO Spectrometer: Kinetics

The following labeled screen shots show the tools and options available for the experiments you'll encounter in this course. Specific directions can also be found in the experiments themselves.

TECHNIQUES

Pasco Spectrometer Analyze Solution Screen and Icon Labels

a

EXPERIMENT OPTIONS

b

ANALYZE SOLUTION

c

CONCENTRATION

d

TIME

e   CONNECTION STATUS

f

JOURNAL SNAPSHOT

g

SHARE

h

START RECORDING   STOP RECORDING

i

CALIBRATE DARK

j

CALIBRATE REFERENCE

k   SCALE TO FIT

l
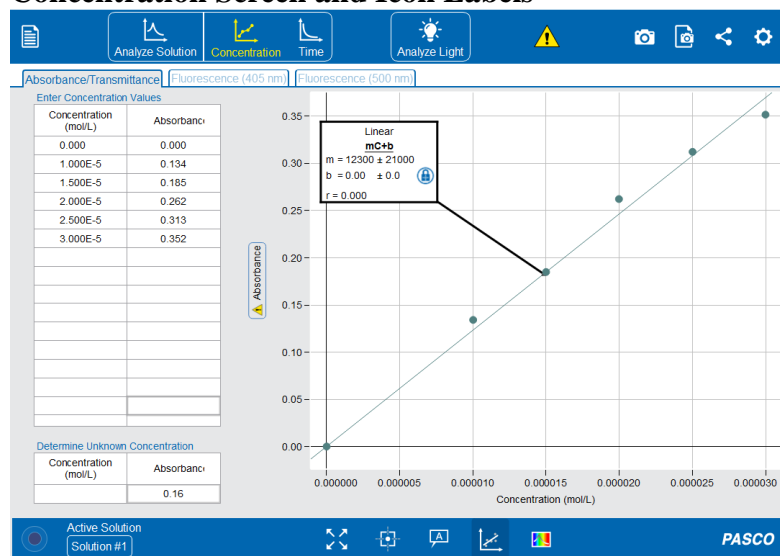

ADD COORDINATE

m   SHOW COLOR SPECTRUM

TECHNIQUES

Concentration Screen and Icon Labels

n

SHOW LINEAR FIT

o

SHOW LIVE SCAN DISPLAY

p

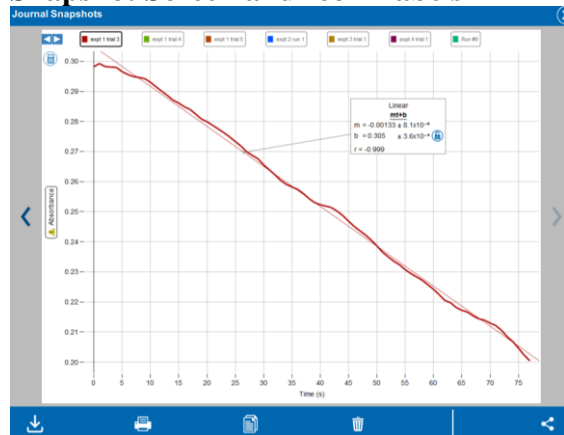SHOW SELECTION TOOL

q

COPY TO CLIPBOARD

Time Screen and Icon Labels

p
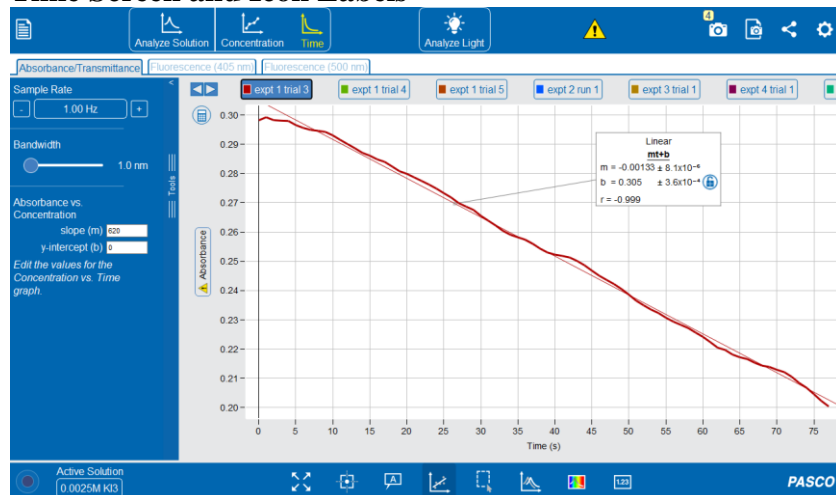
Snapshot Screen and Icon Labels

q

Vernier Go Direct Sensors

The Chemistry Department has purchased a variety of Vernier Go Direct Sensors for use in the Chemistry 50 and 51 Laboratory curriculum. These devices can connect wirelessly through Bluetooth to computers, tablets or mobile devices, or connect directly to a laptop or computer via USB cables.

The Graphical Analysis 4 software to run these devices is free and can be downloaded to your personal computer from the following website, or to your mobile device from your app store.

www.vernier.com

The website also offers video tutorials to help you learn how to use the devices and the software. The following videos are recommended.
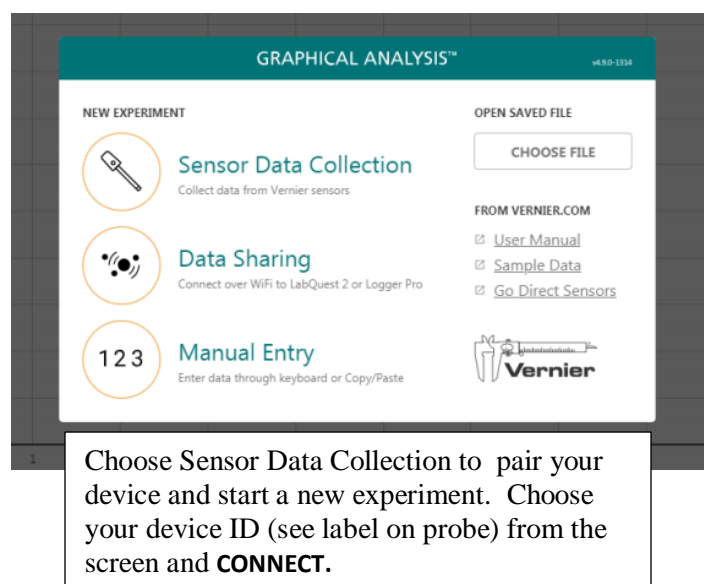
For Chem 50:
– Intermolecular Forces with Evaporation of Alcohols Using Go Direct Temperature
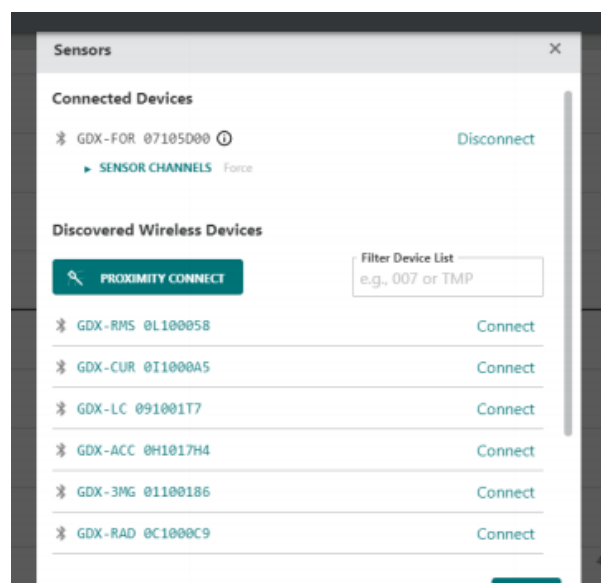
For Chem 51:
– Common Household Acids and Bases Using Go Direct pH
– Strong Acid and Base Titration Using Go Direct pH

The following labeled screen shots show the tools and options available for the experiments you'll encounter in this course. Specific directions can also be found in the experiments themselves.

Choose Sensor Data Collection to pair your device and start a new experiment. Choose your device ID (see label on probe) from the screen and **CONNECT.**

Vernier Graphical analysis icons and screens

TOOLBAR BUTTONS – TOP BAR

File Menu

Collect Button

View Options

Other Options

Untitled

COLLECT

Untitled

New Experiment

Open...

Save

Save As...

Export

1 Graph

2 Graphs

3 Graphs

Table

Meter

Graph and Table
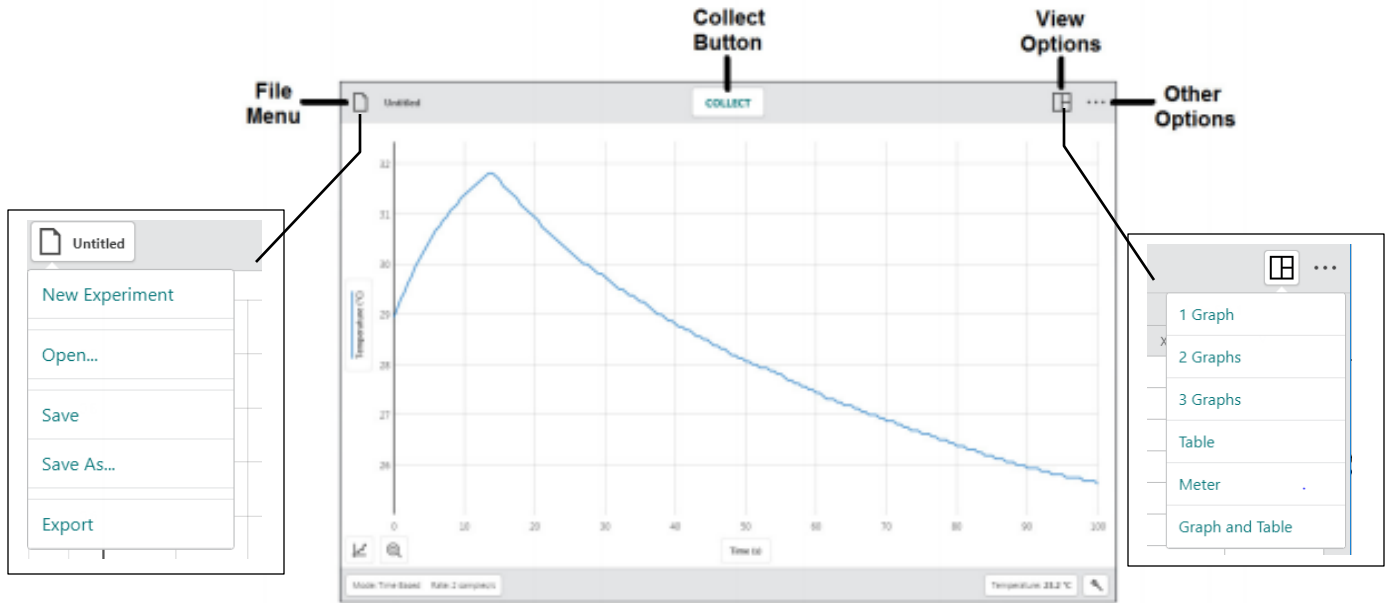
TOOLBAR BUTTONS – BOTTOM BAR

Sensor Setup

Data Collection Settings

Sensor Meter
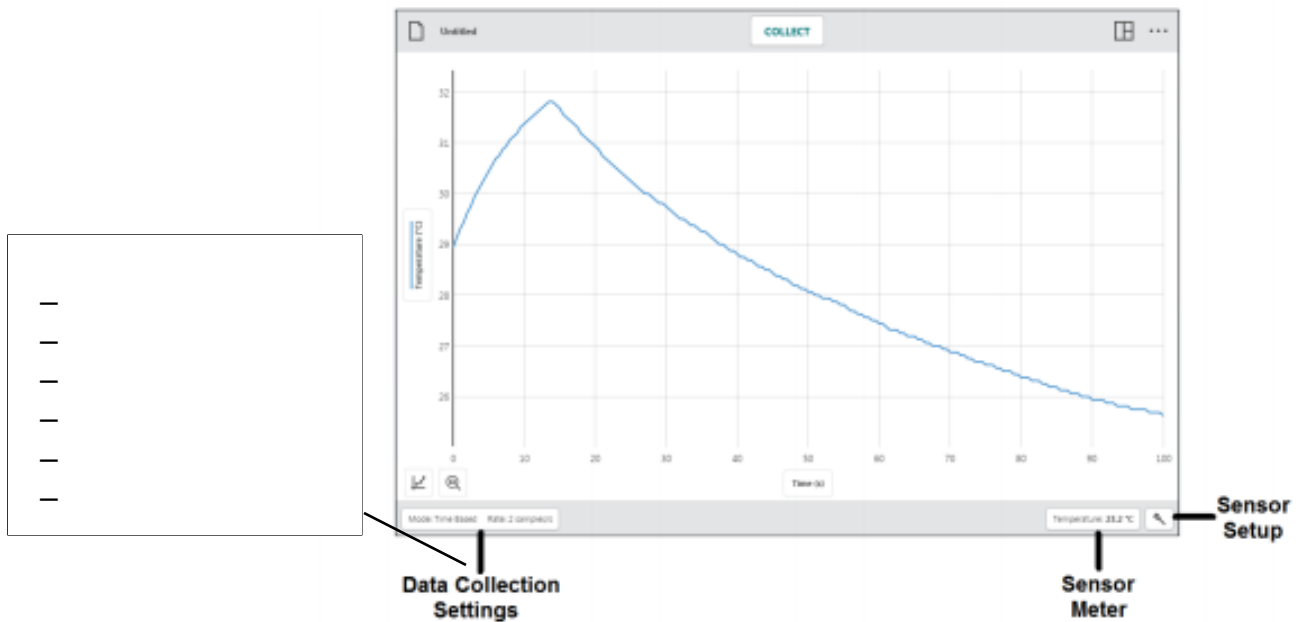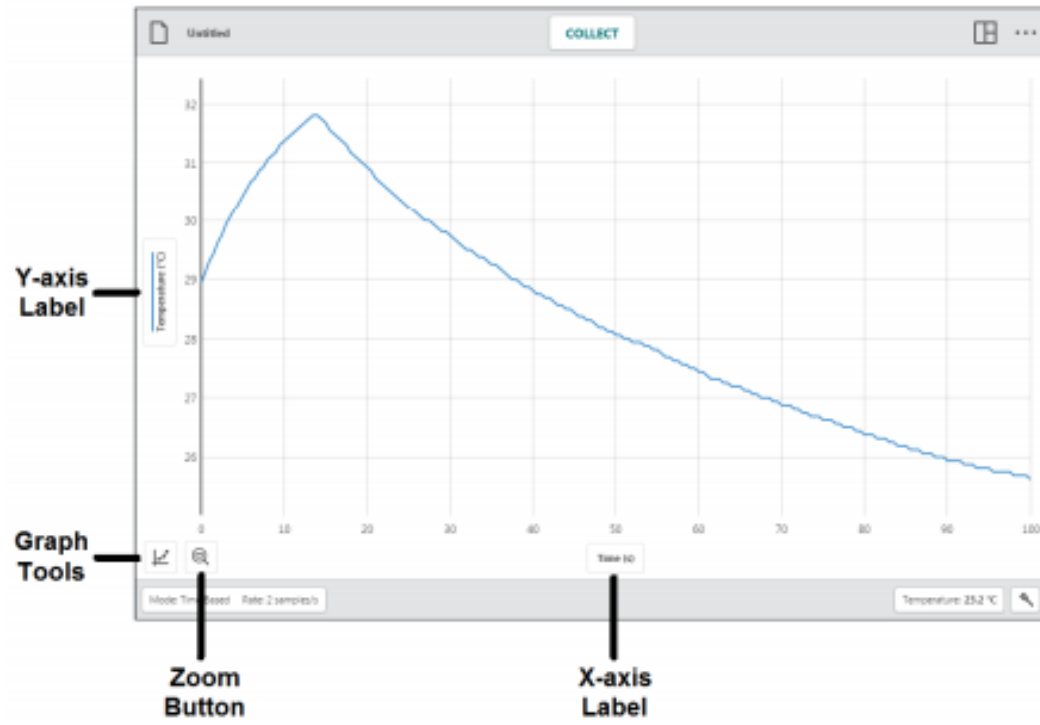
TECHNIQUES

NOTEBOOK

The notebook is an important tool for a laboratory chemist. **The notebook is the appropriate place to record all lab-related information – answers to pre-lab questions, notes from pre-lab lectures by your professor, experimental procedures, all experimental data (measurements or observations), calculations, results, conclusions, rubrics for lab reports, and optionally, answers to post lab questions.** Suggestions for appropriate use of the laboratory notebook are:

- All information entered into the lab notebook must be written in ball point pen. Pencils or markers are not acceptable methods of archiving important experimental information.

- Any mistakes or deletions should be lined (~~strikethrough~~) or X'd out, not covered with "white out", obliterated or erased. No pages should be torn out of the notebook.

- Data (measurements and observations), calculations, and results of calculations should be labeled clearly and systematically, and entered directly into the notebook while the experiment is conducted (not on scraps of paper to be copied later into the notebook). Measurement numbers should never appear in the lab notebook without units or labels! Your professor may suggest additional steps for highlighting of experimental results.

- Large data sets should be compiled into data tables of like measurements, and labeled accordingly. Likewise, large sets of results should be compiled into labeled results tables.

- Information relating to a particular experimental "trial" should be labeled clearly and be kept together with all other information regarding that trial.

- An up-to-date table of contents should be maintained, with experiment titles and page numbers.

- Somewhere in the notebook, be sure that the student's name and the professor's name is written legibly, in the event that the notebook is lost, so it can be returned to its owner.

Your professor may suggest additional guidelines for proper recording of information in the notebook. Below are some examples of different formats of entering information into the notebook. The first example shows labeled descriptions of data on the left side of the page's middle line, and recorded measurements and calculations on the right of the middle line:

Part 2	
Trial 1:	
Mass of the beaker:	2.9360 g
Mass of the beaker + water:	38.8147 g
Calculation of mass of water:	(Mass of beaker + water) – (mass of beaker) = mass of water
	38.8147 g – 2.9360 g = 35.8787 g

TECHNIQUES

When multiple measurements of the same type are collected or several trials will be conducted, it is helpful to organize the recorded data into data tables, like the following two examples:

Measurement Description	Trial 1	Trial 2	Trial 3	Trial 4
Mass of beaker	2.9360 g	2.9363 g	2.9358 g	2.9364 g
Mass of beaker and water	30.2365 g	32.8465 g	31.2005 g	30.1115 g

Trial #	Mass of beaker (grams)	Mass of beaker and water (grams)
1	2.9360	30.2365
2	2.9363	32.8465
3	2.9358	31.2005
4	2.9364	30.1115

Before an experiment, it is extremely important to completely read the experimental procedures to determine how much data will be collected, and how many experiment trials will be conducted. Then, decide which organized format will be the best to record your data and calculations. Prepare your lab notebook with a data table if possible before collecting and recording that data. Remember that information that is gathered from a published resource is also considered data. Be sure to label the source for all published data. When all calculations have been completed, try to compile all of the results into one results table if possible.

Clearly written and organized data in the lab notebook requires thoughtful preparation before each lab session. When data is written into the lab notebook in an organized manner, it becomes easier to understand the written information at a later date, especially when writing the lab report. If the data and results are clearly labeled and organized, a written lab report becomes much easier to create. Also, if experimental results are suspect, then review of clear, organized data and results by your professor becomes much easier to do.

ACCURACY AND PRECISION

An important aspect of reporting experimental data and calculated results from experimental data is including an assessment of their precision and accuracy. Sometimes it is possible and/or necessary to achieve extremely precise and accurate results (say less than 1% error), but other times it may not be necessary or even possible to be so precise or accurate. You need to understand the limits of your results and be able to convey them to your readers. There are many ways to do this, but the following discussion summarizes the conventions we will use in this course.

Accuracy and Precision in Measured Quantities

When we measure something in the laboratory, the precision and accuracy of our measurement is determined by the measuring device we use and the skill of the user.

precision = how close two or more measurements of the same quantity agree
How precise we can be is often determined by the gradations on the measuring device (example, use of two rulers with cm vs. mm markings and resulting differences in confidence in length measurements).

accuracy = how close a measurement is to the "true" value of the quantity measured
The accuracy of a measurement is often determined by whether the measuring device is properly calibrated and used correctly.

Errors

There are different kinds of "errors" that affect precision and accuracy.

systematic error = an error that is always about the same amount and in the same direction, usually caused by a faulty measuring device or an error in the method. Systematic errors can usually be found and corrected.

random errors = small deviations in repeated results that can be + or – from the "true value". Random errors cannot be eliminated—only improved by using more precise measuring devices.

sample variance = random error strictly applies to the repeated measurement of the same object or quantity. If you are trying to determine the average mass of a jelly bean and weigh 10 different jelly beans separately, the different masses you obtain are actually representative of sample variance rather than random error—each different jelly bean actually has a different mass than the other; they are not the same object!

Whenever we communicate the results of measurements, we need to tell the audience how confident we are in those measurements—in other words, what the precision and accuracy of the results are.

ACCURACY AND PRECISION

There are a number of ways to report the <u>precision</u> of a result depending on how many measurements are made:
- – for single measurements use the appropriate number of significant figures
- – for multiple measurements of the same quantity use
  - the range
  - % deviation
  - standard deviation

The <u>accuracy</u> of the result is usually reported as the % error from the true value (if known).

Significant figures and rounding:
When reporting the results of a measurement, you should report all the digits that you know for sure and the first one that you have to estimate.

RULES FOR COUNTING SIGNIFICANT FIGURES

1. *Nonzero integers*. Nonzero integers always count as significant figures.

2. *Zeros*. There are three classes of zeros:

   - – *Leading zeros* are zeros that precede all nonzero digits. They are never significant.
   - – *Captive zeros* are zeros that fall between nonzero digits. They are always significant.
   - – *Trailing zeros* are zeros that follow all nonzero digits. They are significant only if the number is written with a decimal point.

        Example: 200 has one significant figure

        200. has 3 significant figures

        200.05300 has 8 significant figures

3. *Exact numbers*. Counting numbers (8 apples) and definitions (1 kg = 1000g) are exact and are assumed to have an unlimited number of significant figures. In other words, they do not limit the number of significant figures reported in a calculated result. Metric to metric conversion factors are exact and English to English conversion factors are exact, **however** English to metric conversion factors are not exact (except for 1 in = 2.54 cm exactly).

4. *Addition and Subtraction*. In addition and subtraction, the rightmost significant figure in the result is determined by the rightmost significant figure of the least precise value (the least precise value is the one with its last significant figure in the highest place value).

5. *Multiplication and Division*. In multiplication and division, the number of significant figures in the result is equal to the number of significant figures in the original number with the fewest significant figures.

ACCURACY AND PRECISION

In calculations, keep a few additional figures during intermediate steps and only round at the end. Two common ways of denoting "extra" figures are to either underline the last significant figure in a result or subscript any unsignificant figures in a result. For example, if the result of a calculation was 10.05498746 on a calculator, but should only have 4 significant figures, write

$$10.0\underline{5}499 \quad \text{or} \quad 10.05_{499}$$

Basic statistics:

Often, we will make multiple measurements of a given quantity and report the average

$$average = \frac{\sum x_i}{n}$$

where x_i are all the individual measurements of a given quantity, Σ means to add up all the x_i's, and n is the number of individual measurements. Why do we do this? All measurements are subject to (hopefully small) random errors—some of which are likely to cause the measured value to be too high and some that will cause it to be too low. By taking multiple measurements that average out those small errors, we hopefully get a better idea of what the "true" value is. For example, if the density of a sample was measured three times with the following results: 1.056 g/mL, 1.078 g/mL, and 1.065 g/mL, the average result would be 1.066 g/mL.

One way to report the precision of a series of measurements of the same quantity is to report the range. The range is defined by giving the low and high values associated with a measurement. In the above example, you would report 1.066 g/mL (1.056-1.078 g/mL).

Another option for reporting precision is to report the percent deviation for each measurement.

% deviation = (Experimental value – average value) x 100 %
Average value

Note that the % deviation will be positive if the experimental value is greater than the average value, but negative if the experimental value is less than the average value. In the density example from above, the results would be reported as 1.056 g/mL (-0.9%), 1.078 g/mL (+1%), 1.065 g/mL (-0.09%)

The range and % deviation are useful when a small number of measurements are made (usually 3-5). When more measurements are made, a statistical analysis using standard deviation is usually performed.

When many measurements are made of the same quantity, theory shows that the measurement values should form a bell-shaped curve called a "Gaussian" curve. The x-axis of the Gaussian curve represents the measured values and the y-axis represents the probability of making that particular measurement. It is most probable to make a measurement very close to the average value (peak of curve) and less and less likely to make measurements with large errors.

ACCURACY AND PRECISION

95% of the area of the curve lies
between these lines

68% of the area of the curve
lies between these lines

Meaning of Standard Deviation: If you
were to make one more measurement of
this quantity, 68% of the time that
measurement would be expected to fall
within +/- s of the average.

$\bar{x} - s$ $\bar{x}$ $\bar{x} + s$
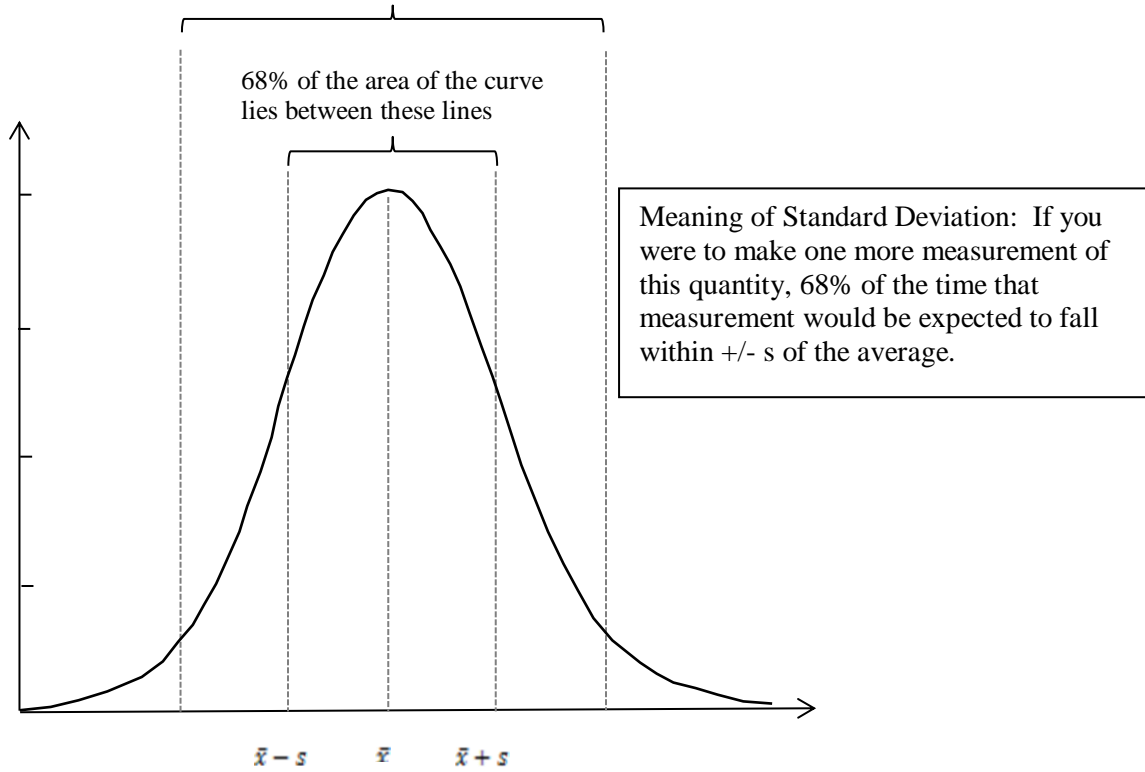
The Gaussian or Normal Distribution Curve

In a Gaussian curve, the average represents the best estimate of the true value and the width of
the curve estimates the precision of the measurement. From this curve, one can show that s,
which is called the **standard deviation**, is

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}}$$

where x_i is each measurement, $\bar{x}$ is the average, and n is the number of measurements made. The
smaller the value of s, the more precise the measurements are. The figure below shows two
Gaussian curves with the same average value. The dashed curve has a smaller standard deviation
because the measured values are closer to the average (more precise). The standard deviation
should be reported to 2 significant figures.
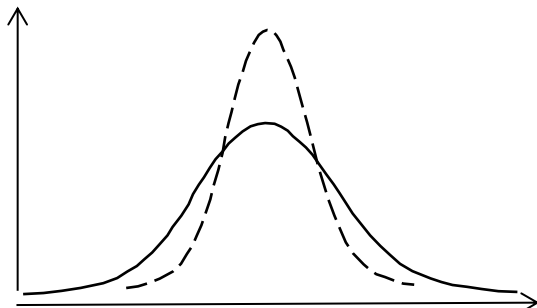
ACCURACY AND PRECISION

In the density example used above, the standard deviation would be 0.011 g/mL. Therefore, the result would be reported as 1.066 ±0.011 g/mL. (Note that the standard deviation actually overestimates the precision when only a few data points are taken. Correction factors can be applied, but we will not make use of these in this course.)

You can also calculate the % standard deviation or relative standard deviation:

$$\text{\% standard deviation} = \frac{\text{std. dev.}}{\text{average}} \times 100\%$$

Again from our density example, the % standard deviation would be 1.0%.

What about accuracy? Accuracy compares some measured value or average value to a hypothetical "true" value known by some other means. We report the accuracy of a measurement only when we have information about what the "true" value is. Accuracy is usually reported as error or percent error:

error = (measured value or average) – (true value)

$$\text{\% error} = \frac{\text{error}}{\text{true value}} \times 100\%$$

If the measured value is larger than the true value, the % error will be positive, but if the measured value is smaller than the true value, the % error will be negative. If the true value in our density example were 1.059 g/mL, then the % error would be -0.7%.

Evaluation of Experimental Precision and Accuracy

This is one of the most difficult jobs of a chemist and one that requires experience and wisdom to achieve. In this course, you will be asked to critically evaluate the results of your experiments including possible errors. The following discussion offers some guidelines, but each situation is different, so you will have to use your chemical intuition and critical thinking skills!

Once you have determined the precision and accuracy of your results for a given experiment, how do you assess whether or not your results are reasonable? One way is to compare your precision and accuracy to the precision and accuracy of the glassware and equipment you used in the experiment—you can't do better than the equipment you have! If your precision and accuracy agree with the precision and accuracy of the equipment, then you've done the best job possible with the equipment you have and your "errors" represent the random errors associated with the measurements you made. If your precision and accuracy are less than expected based on the equipment, then and only then do you need to start looking for other sources of error. Errors can creep in from many places, and may be inherent in the chemistry or method of analysis. **Don't automatically assume that you used the equipment incorrectly.** Think also about the chemistry of the experiment *e.g.* did the reaction go to completion? are there any interferences? etc.

ACCURACY AND PRECISION

Comparison of Experimental and Expected Precision: Significant Figures

One way to evaluate the *expected* precision of your experiment is to use the rules of significant figures.

Example:

Let's say you were measuring the density of a certain rock. You weighed the rock on an analytical balance and obtained 5.0387g ($\pm$ 0.0001g). You then measured the rock's volume by the method of displacement and obtained 52.13 mL for the initial volume and 54.58 for the final volume ($\pm$ 0.01mL). The density, reported to 3 significant figures based on the volume measurements, would be:

$$d = \frac{mass}{volume} = \frac{5.0387 \text{ g}}{(54.58 \text{ mL} - 52.13 \text{ mL})} = \frac{5.0387 \text{ g}}{2.45 \text{ mL}} = 2.06 \text{ g/mL}$$

Writing the result this way implies that your result is precise to $\pm$ 0.01 g/mL. If you measured the density of the same rock 5 times this way and your results all fell within 2.05-2.07 g/mL, then you would say your results were consistent with the precision of the equipment used.

This simple method is useful, but it can overestimate the expected precision of a result, especially when several operations were performed in obtaining the result and/or when measurements are not $\pm$ 1 in the last digit. More rigorous statistical methods are available, but for our purposes, significant figures will give a useful guide.

Evaluation of Accuracy

Remember that an evaluation of accuracy depends on knowing the true value. If the true value lies within the range of your results, then your result agrees with the true value. If the true value is outside your range, then there may be a systematic error.

Case Studies

Let's look at the following two case studies to illustrate these points. Using density again, let's assume the following densities were calculated from experimental data and for each case, 3 significant figures are allowed for the results.

Case 1	Case 2
21.2_7 g/mL	1.07_5 g/mL
21.3_5 g/mL	1.09_5 g/mL
21.3_9 g/mL	1.05_4 g/mL

The "true value" for Case 1 is 21.7 g/mL and the "true" value for Case 2 is 1.06 g/mL. Evaluate the precision and accuracy of each case.

Answer:

For Case 1, the results could be reported using the standard deviation as 21.3 ± 0.061 g/mL. The relative standard deviation is 0.29% and the % error is -1.8%. Are the results

precise? Are the results accurate? Using the rules of significant figures, we would expect all of our values to be within ± 0.1 g/mL. Since all values did lie within 21.3 ± 0.1 g/mL, we would say that the results are precise. The percent error is only -1.8%, which seems pretty accurate, but notice that all of the measured values are lower than the true value and that the precision is "better" than the accuracy. This points to a systematic error—some error is making all of our measurements too low.

For Case 2, the results could be reported as 1.07 ± 0.021 g/mL. The relative standard deviation is 1.9%. The % error is +0.9%. Are these results precise? Are they accurate? In this case, using the rules of significant figures, we expect all of the measured values to lie within 1.07 ± 0.01 g/mL, however our values range from 1.05 to 1.10 g/mL, and are therefore not as precise as our individual measurements seem to indicate is possible. Something about our technique or measurements are not as precise as the equipment we're using. On the other hand, the "true value" of 1.06 g/mL lies within the range of our results (1.05-1.10 g/mL) and therefore we would say that these results are accurate.

Systematic Error
The concept of systematic error relates to a reproducible "problem" with an experiment that always gives results that are "off" in one direction. Systematic error is therefore different from *random error* which almost always comes from difficulties reading devices (precision) and gives results that are "off" sometimes higher and sometimes lower, but never predictably one way or the other. We often report these as ± values, such as saying the volume measured was 10.1±0.1 mL. We think the value is 10.1, but because we are interpreting that last digit, we have an uncertainty of 0.1 mL that could be higher or could be lower.

Since systematic error always happens in one direction, we can often determine what effect a systematic error had on our **results**. For example, if we are transferring a solid from a weigh boat to a flask to make a solution, and we see some crystals that did not come off of the weigh boat, we know we have a systematic error that reduces the amount of solid that transferred to the flask. The effect of this systematic error is always reported related to the **results** of the experiments, not just the data or calculation that has the error. In this case we state the effect of the systematic error on the solution concentration. The *actual solution concentration will be less than we calculate*.

We can't usually say the exact effect of our systematic error (if we could, then we should just correct our results!), but we can often estimate it. In this example, I might estimate (by looking at it) that 1% of the solid was left in the weigh boat. Estimates are most often represented as a percentage and are generally rounded whole numbers (1%, 2%, 5%, 10%, etc., but not 3.5% or 17%.) The 1% of solid left in the weigh boat would result in a *calculated* solution concentration that was 1% too high. When writing about the effect of systematic error to begin with, you can use language that looks something like the following (once you get better, use your own words!)

> *As a result of _____ (describe the systematic error), the actual _____ (a result, such as concentration) is about _____ (a number with units or a %) [higher/lower] than the [recorded, measured, calculated] _____ (same result name as before)*

ACCURACY AND PRECISION

For the example here: As a result of all the solid not being transferred from the weigh boat, the *actual* solution concentration is about *1% lower* than the *calculated* solution concentration.

When you try to identify systematic errors in your experiments, the questions to ask are:
1. What part of the procedure or calculation has the systematic error? (transferring the solid)
2. What is a numerical estimate of the error? (1% of the solid was not transferred)
3. What is the numerical effect on the **result** (not just the effect on your *data!*)? (The *calculated* solution concentration will be approximately 1% higher than the *actual* solution concentration.)
   a. The phrasing of this can be difficult! You have to consider what the effect of your error is on your calculated result, and the language can sometimes get mixed up. Be sure that whatever sentence you put together makes sense!

An example of systematic error

Beth is making some 2.50M solutions by measuring out 40.0 mL of water using a graduated cylinder. She pours this into a flask where she has already measured out the correct amount of solid. Later in the day, someone tells her that she should have been reading the meniscus from the bottom, not from the top where she was actually reading it.

a) *What part of the procedure has the error?*

The reading of the graduated cylinder.

b) *What is a numerical estimate of the error?*

Since the top of the meniscus might have been around 0.3-0.5 mL higher than the bottom of the meniscus, we estimate that the volume read was about 0.4 mL too high, or as a percentage, about 0.4 mL / 40.0 mL * 100% = 1%. Note how we compared our error to our data. A 0.4 mL error in a 40.0 mL measurement (1% error) is much less significant than a 0.4 mL error in a 4.0 mL measurement (10% error).

c) *What is the numerical effect on the result?*

The measured V was higher than the actual V. Beth thought she was measuring 40.0 mL but was actually measuring about 39.6 mL. However, in discussing systematic error we are not concerned primarily with the effect on the data, but the effect on the result: the concentration of the solution. We remember

$$Molarity = \frac{moles\ solute}{L\ solution}$$

Since Beth actually had less volume than she thought, her actual molarity is larger than her calculated molarity. For example, if she had measured 0.100 moles of solute, we convert our volumes from mL to L and we can determine the *actual* molarity compared to Beth's *calculated* molarity.

ACCURACY AND PRECISION

$$Calculated\ molarity = \frac{0.100\ mol}{0.0400\ L} = 2.50M$$

$$Estimated\ actual\ molarity = \frac{0.100\ mol}{0.0396\ L} = 2.53M$$

Since we estimated the error it is not possible to say that her actual molarity is 2.53M, but performing the actual calculation with the measured data and the "corrected" data makes it very clear how our systematic error affects our result.

Based on this discussion, we conclude

As a result of reading the meniscus in the graduated cylinder from the top instead of from the bottom, the actual solution concentration is about 1% higher than the calculated solution concentration.

SPREADSHEET AND GRAPHING WITH EXCEL

In the general chemistry curriculum at Mt. SAC, the Excel program is used to record and manipulate data, perform error analysis, and construct graphs. Although many students may be quite familiar with Excel already, the following tutorial is written to provide a basic introduction to Excel and to provide a review of commonly used functions in the course. The following screenshots and directions are for Excel 2016 and may vary depending on the version of Excel and if your computer is a Mac or PC.

<u>Layout of the Spreadsheet</u>

Once you have opened the Excel program, choose **Normal** under **View** in the main menu bar located at the top of the screen. You will be presented with a basic format of a spreadsheet.

**Main Menu Bar** for Excel (varies depending on the version of Excel and Mac or PC)
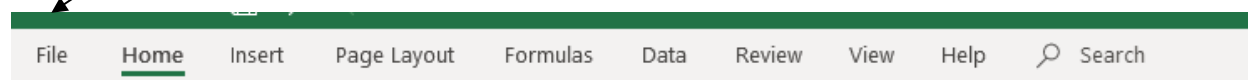
The spreadsheet is made up of a series of columns (labeled A, B, C, etc.) and rows (labeled 1, 2, 3, etc.). When you click on any box in the spreadsheet, the column and row will be highlighted (darker shade as compared to the other rows or columns). In addition, the cell that is selected will be highlighted around the edges to alert you that this is the **active cell** (the cell you selected) and its location is referred to as **B6**.
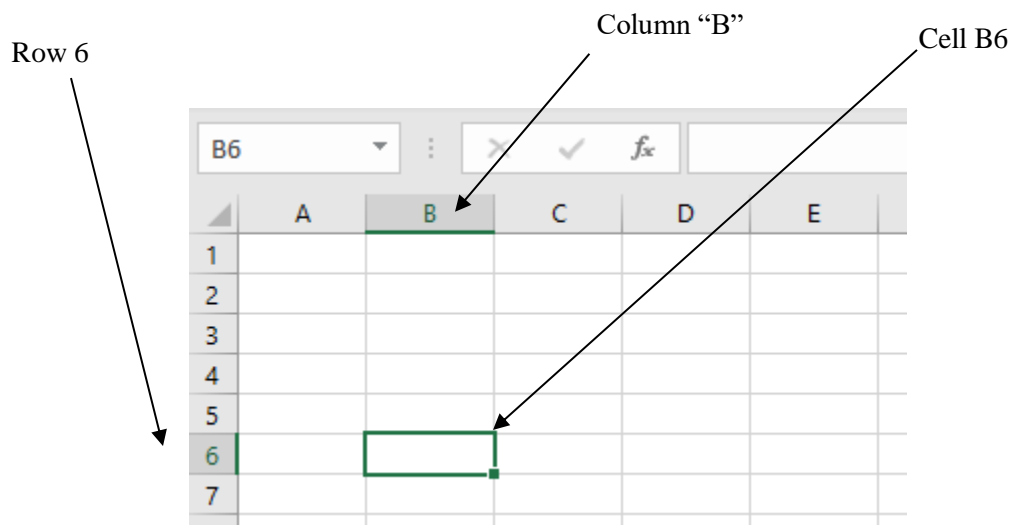

<u>Selecting a Group of Cells</u>

To select cells **A3** through **D14**, click on cell **A3** and hold and drag the cursor to cell **D14**. The group of cells will be highlighted as shown in the figure below.
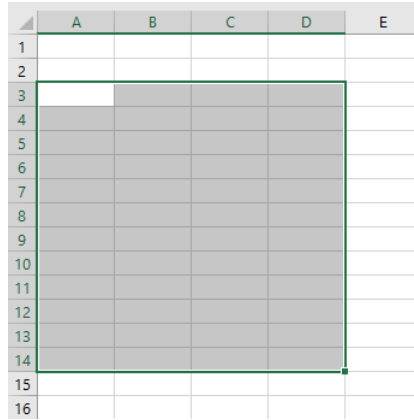
## Formatting of Cells and Cell Contents

The **Format** option in the **Cells** section of the **main menu bar** allows you to do many things with the look of the cells as well as the contents of the cells. Click on **Format**, then **Format Cells…**, and you should see the following:
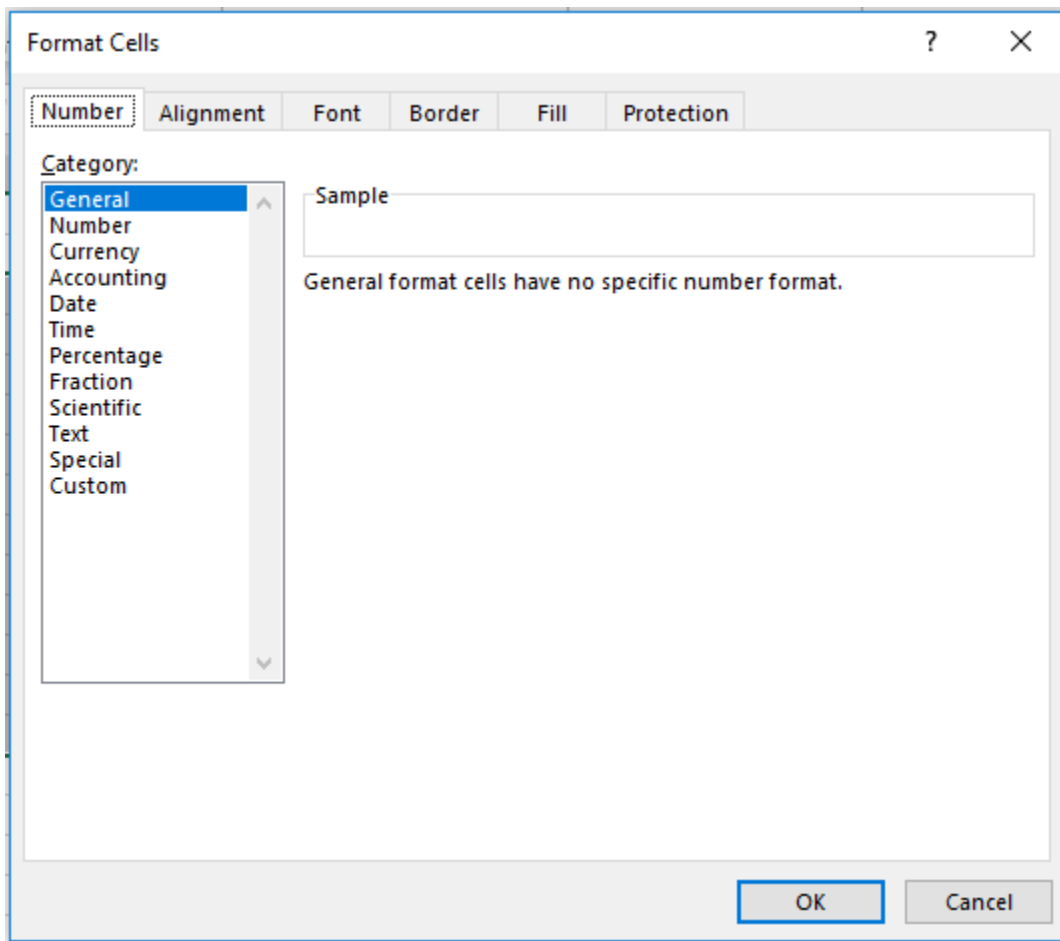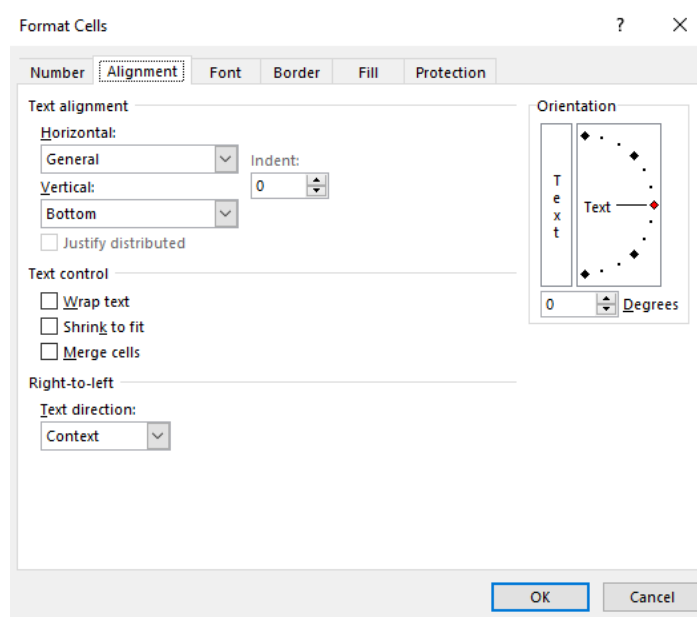
- **Number Format**
  - Changes the way values are written ($, scientific notation, etc.) and the number of decimal places desired (click on Number and it gives an option to identify the number of decimal places)
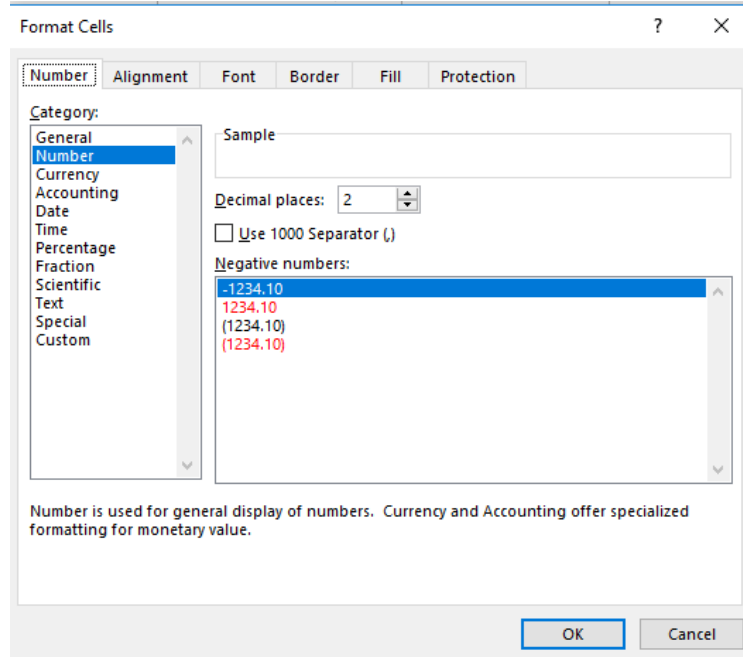
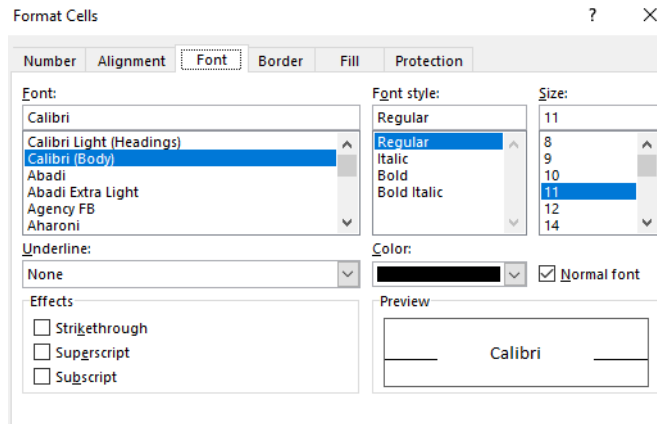
- **Alignment Format**

  - Changes the way the contents are aligned in the cells
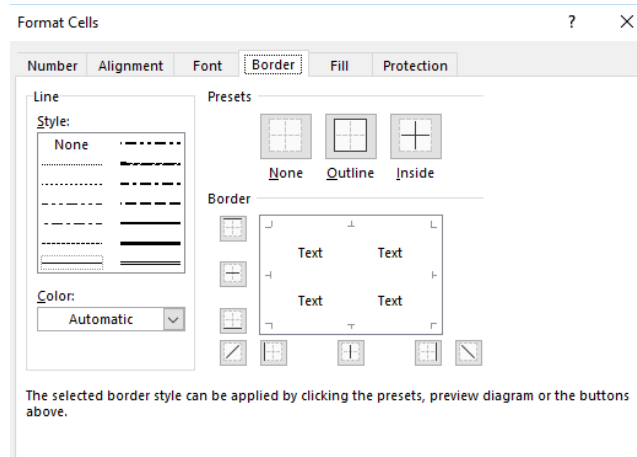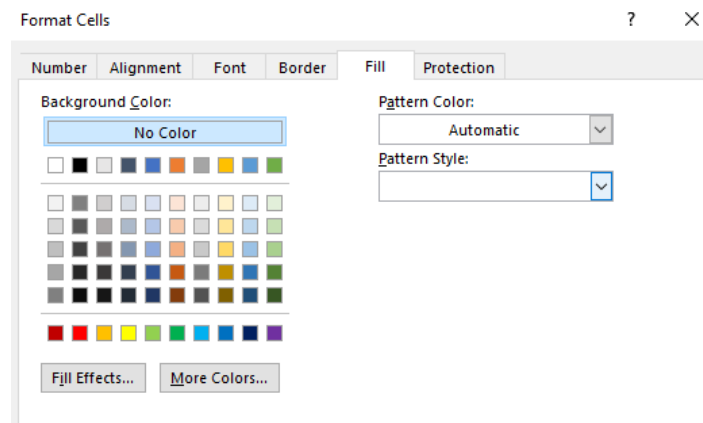
- **Font**
    - Allows you to change the type, size and style (superscript, etc.) of font.

- **Border**
    - Allows you to change the border of the cells or a group of cells and the style of border.

- **Fill**
    - Allows you to change the color of the cell and its contents

SPREADSHEET AND GRAPHING WITH EXCEL

Types of Data Entry
- **Numbers (e.g. 352)**
  - Highlight the cell and type in the number and hit the **Enter** or **Return** key.
  - Changing Formatting (scientific notation, decimals, number of digits or placeholders, etc.)
    - Select the cell(s) to be formatted
    - Select **Format** and then **Format Cells** on the **main menu bar**
    - From here, the most common selection under the **Number** menu item in the category section is **Number** or **Scientific**. You may choose the number of decimal places here too. This is useful when ensuring that you report numbers to the correct number of significant figures.
    - In addition to number formatting, you may also choose to change the alignment, font, size, etc.

- **Text or labels (e.g. concentration)**
  - Changing Formatting (font, size, style, etc.)
    - Process is the same as for formatting numbers. Select **Format** and then **Format Cells** under the **main menu bar**, and then choose the type of formatting you want (font, size, etc.).

- **Formulas (e.g. =C1+D1)**
  - Excel makes performing mathematical operations on a series of data very easy. You can add columns of numbers or do any other type of operation.
  - Formulas always start with an "=" sign
  - Mathematical symbols in order of operation include
    - Parentheses ( ) or brackets [ ] to group expressions
    - Exponents (a caret symbol, ^)
    - Division (a forward slash, /)
    - Multiplication (an asterisk, *)
    - Addition (a plus symbol, +)
    - Subtraction (a minus symbol, -)

For example, make a spreadsheet that contains the following information:

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm3)
6					
7	1	2.32	4.23	8.42	
8	2	2.31	4.26	8.32	
9	3	2.34	4.21	8.39	
10					
11					

Next, change the 3 in cm3 to a superscript. Highlight the 3 and then go to **Format Cells…**, and check the **Superscript** box (see below). Then hit **OK** to make the change.
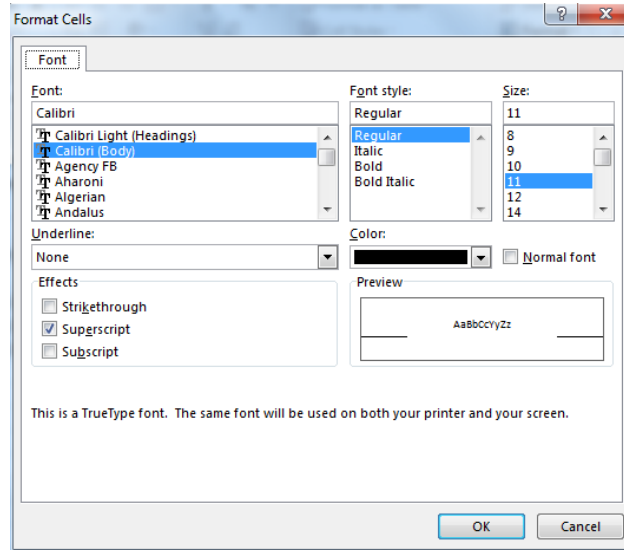
Now, let's change the format of the cells so that the contents of all cells in the table are aligned in the center. First, click and drag to highlight the table, then go to **Format**, **Cells…**, and **Alignment**.
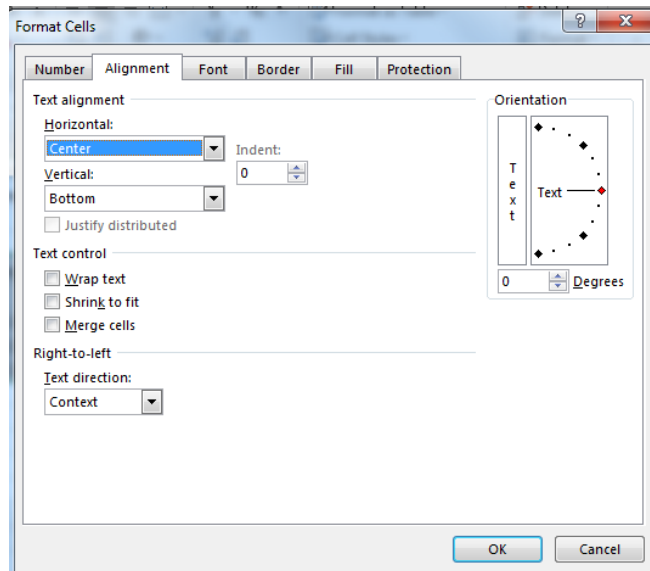
Next, choose **Center**, and then hit **OK**. Your spreadsheet should look as follows:

SPREADSHEET AND GRAPHING WITH EXCEL

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	
8	2	2.31	4.26	8.32	
9	3	2.34	4.21	8.39	
10					
11					

To calculate the volume of the block (L x W x H) using the formula function in Excel for trial 1, first click on cell **E7**. Then type in the equal sign, "=" (all formulas begin with an equal sign), and click on each of the three cells that contain the desired data with the multiplication sign in between: **B7 * C7 * D7**.

SUM   ✕ ✓ f_x =B7*C7*D7

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	=B7*C7*D7
8	2	2.31	4.26	8.32	
9	3	2.34	4.21	8.39	
10					
11					

After pushing the **Return** key, the screen looks like the following:

E8   ✕ ✓ f_x

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	82.630512
8	2	2.31	4.26	8.32	
9	3	2.34	4.21	8.39	
10					
11					

You have now calculated the volume by multiplying the length times the width times the height. Instead of calculating the volume by hand, we did it by using Excel by entering a formula. If you double click again on cell **E7**, it will show you the formula that was used. Next, let's copy the formula in cell **E7** and paste it in the remaining two cells in order to

52

calculate the volume for trials 2 and 3. First copy the formula in cell **E7** by clicking on the cell, then by pushing the **Control** and **c** button at the same time. You can do this another way by going under **Edit** in the **main menu bar** and clicking on **Copy** (cell E7 should now have a dashed highlighting box around it). Next click on the lower right corner of the cell (cursor becomes a thin plus sign) and drag to highlight cells **E8** and **E9**. When you let go, the spreadsheet should look as follows:
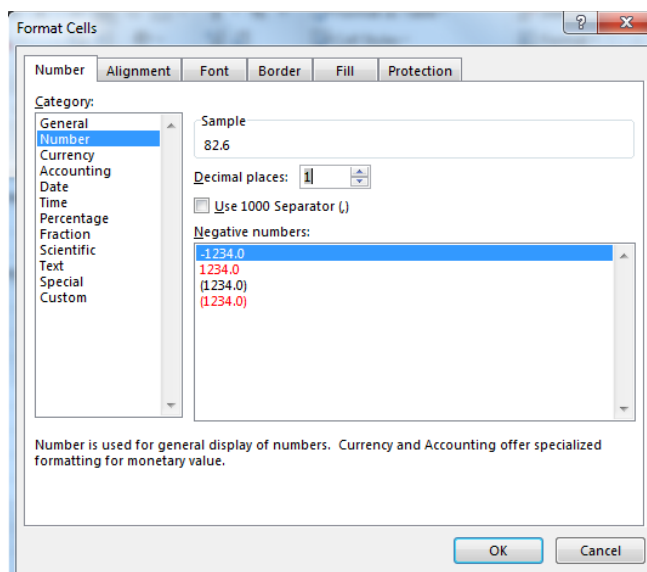
	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm^3)
6					
7	1	2.32	4.23	8.42	82.630512
8	2	2.31	4.26	8.32	81.873792
9	3	2.34	4.21	8.39	82.653246
10					

The volumes for Trials 2 and 3 have been calculated. Double click on cells E8 and E9 to see the formula for that cell. Notice that the cell references have been updated. By copying the cell in **E7** we essentially copied the "relative" cell. In other words by pasting the formula in the next two cells below this, the relative location is kept (column B) but the row number changes. This allows you to copy and paste formulas for a large amount of data which makes your life MUCH easier!

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm^3)
6					
7	1	2.32	4.23	8.42	82.630512
8	2	2.31	4.26	8.32	81.873792
9	3	2.34	4.21	8.39	=B9*C9*D9
10					

The number of significant figures for volume should be 3 so the format for the cells **E7-E9** needs to be changed to reflect this. Make sure that these cells are highlighted. Then go to **Format** in the **main menu bar,** then **Cells…** then **Number**.

Click on **1** decimal place and hit **OK** and now the results are shown with three significant figures.

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm^3)
6					
7	1	2.32	4.23	8.42	82.6
8	2	2.31	4.26	8.32	81.9
9	3	2.34	4.21	8.39	82.7
10					

You can further customize your table by making borders, colors, etc. For example, let's place a border around the entire table and around each cell. Click and drag to select cells of the table and then click **Format**, **Cells…**, and then **Border**. From here, choose the
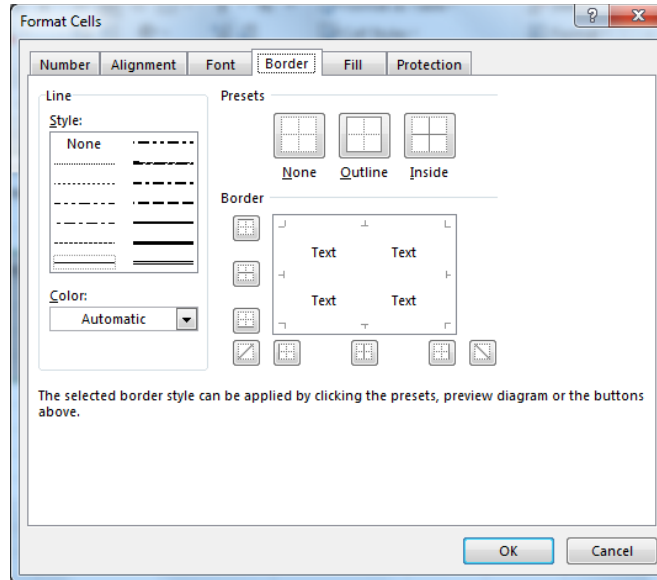
Outside, **Inside** borders and increase the **Line** thickness for the border. Play around with the options to get more familiar with these features.

The result is as follows:

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	82.6
8	2	2.31	4.26	8.32	81.9
9	3	2.34	4.21	8.39	82.7
10					

Statistical Analysis and Other Functions

Functions can be used to calculate the average, standard deviation, etc. on a large set of data. Below is a list of some common functions:
- Average: **=average(cell range)**
- Standard deviation: **=stdev.s(cell range)**
- Other functions: (see your **Excel help** section)

If we wanted to calculate the average volume and standard deviation (an indication to the level of precision of the measurements), then it is easiest to use functions. Type "**avg volume:**" and "**std dev**" below the "Height" column.

	A	B	C	D	E	F
1	Measurements to determine the volume of a rectangular piece of wood.					
2	Three trials were performed.					
3						
4						
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)	
6						
7	1	2.32	4.23	8.42	82.6	
8	2	2.31	4.26	8.32	81.9	
9	3	2.34	4.21	8.39	82.7	
10						
11				avg volume:		
12				std dev:		
13						
14						

Then click on cell **E11**. Enter "**=average(**and then click on cell E7 and drag to cell E9 and enter a closed parenthesis**)"**. Select **Return**.

	A	B	C	D	E	F
1	Measurements to determine the volume of a rectangular piece of wood.					
2	Three trials were performed.					
3						
4						
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)	
6						
7	1	2.32	4.23	8.42	82.6	
8	2	2.31	4.26	8.32	81.9	
9	3	2.34	4.21	8.39	82.7	
10						
11				avg volume:	=average(E7:E9)	
12				std dev:		
13						

The average value of volume is calculated for you and is shown in cell **E11**.

	A	B	C	D	E	F
1	Measurements to determine the volume of a rectangular piece of wood.					
2	Three trials were performed.					
3						
4						
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)	
6						
7	1	2.32	4.23	8.42	82.6	
8	2	2.31	4.26	8.32	81.9	
9	3	2.34	4.21	8.39	82.7	
10						
11				avg volume:	82.4	
12				std dev:		
13						
14						

You can also calculate the standard deviation of the volume by selecting first the cell where the data is to be located, E12, and then by entering "**=stdev.s** (and choose the cell range by clicking and dragging on cells E7..E9)".

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	82.6
8	2	2.31	4.26	8.32	81.9
9	3	2.34	4.21	8.39	82.7
10					
11				avg volume:	82.4
12				std dev:	=stdev.s(E7:E9)
13					

Select **Return** to show the value of the standard deviation.

	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	82.6
8	2	2.31	4.26	8.32	81.9
9	3	2.34	4.21	8.39	82.7
10					
11				avg volume:	82.4
12				std dev:	0.443600896
13					

At this point, you should change the number of significant figures in the standard deviation by changing the format.

|	A	B	C	D	E
1	Measurements to determine the volume of a rectangular piece of wood.				
2	Three trials were performed.				
3					
4					
5	Trials	Length (cm)	Width (cm)	Height (cm)	Volume (cm³)
6					
7	1	2.32	4.23	8.42	82.6
8	2	2.31	4.26	8.32	81.9
9	3	2.34	4.21	8.39	82.7
10					
11				avg volume:	82.4
12				std dev:	0.44
13					

SPREADSHEET AND GRAPHING WITH EXCEL

Graphing in Excel

Make a spreadsheet with the following information (be sure to also do all necessary formatting changes).

	A	B	C
1			
2	Calibration Curve for Fe(SCN)$^{2+}$ solutions		
3			
4			
5		Concentration (M)	Absorbance
6		1.00E-03	0.103
7		2.00E-03	0.211
8		3.00E-03	0.301
9		4.00E-03	0.409
10			
11			

You may have to change the column width so that all information can be seen. You can do this two ways. The first way is to move your cursor over the vertical line between the **B** and **C** column heading. Your cursor should change from a thick white plus sign to a thin black plus sign . Then click on this spot and move your cursor to expand (or shrink) the column width.

You may also do this by going to **Format**, **Column Width** to adjust the column width. You can change the column width on a single column or a block of columns.
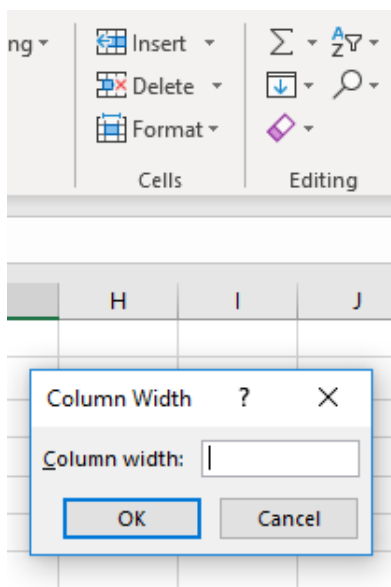
Next, in order to graph the data to make a plot of Concentration versus Absorbance data (this is sometimes called a Beer's plot or a calibration curve), you need to **highlight the data section of your table** and then click on **Insert**, and then in the **Charts** section, choose the type of chart you'd like to make.

58

# SPREADSHEET AND GRAPHING WITH EXCEL

The most common chart to select is **XY (Scatter)** as shown in the circle above. You generally do not want lines to go from each point on your plot (like a dot-to-dot picture). Therefore, select the chart with only points by clicking on the correct picture icon under **Chart sub-type**. A plot will appear on your spreadsheet screen.
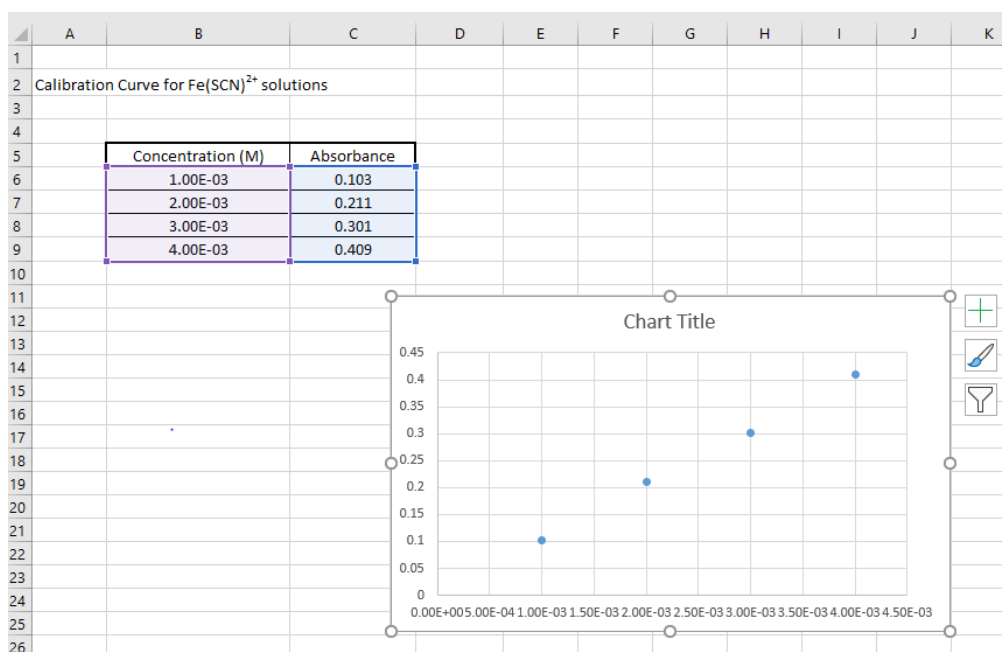
SPREADSHEET AND GRAPHING WITH EXCEL

You will need to verify that the x and y values have been chosen correctly and you will also want to edit the appearance of the plot to show axis titles, etc. When the chart is highlighted, the chart tools will be visible in the top menu bar. First, choose **Select Data**. The "Chart data range:" will show the highlighted cell range chosen for your plot (**B6:C9**--the two dots between the cell addresses means to use all cells including and in between these cells).
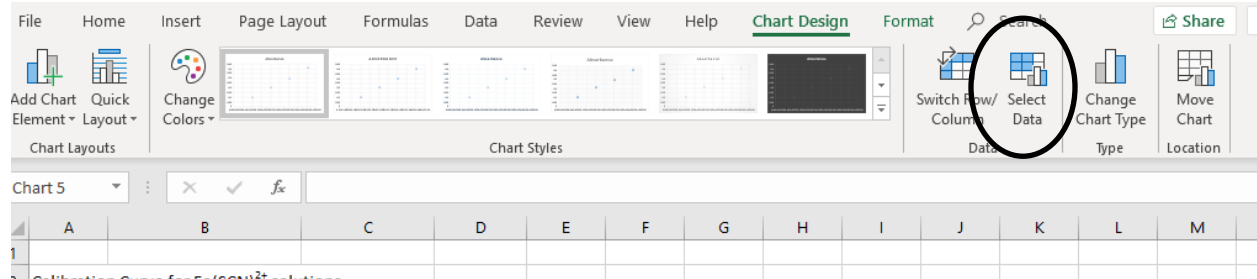
The left column will show the series of data plotted. Highlight Series 1 and then click on the **Edit** series icon.
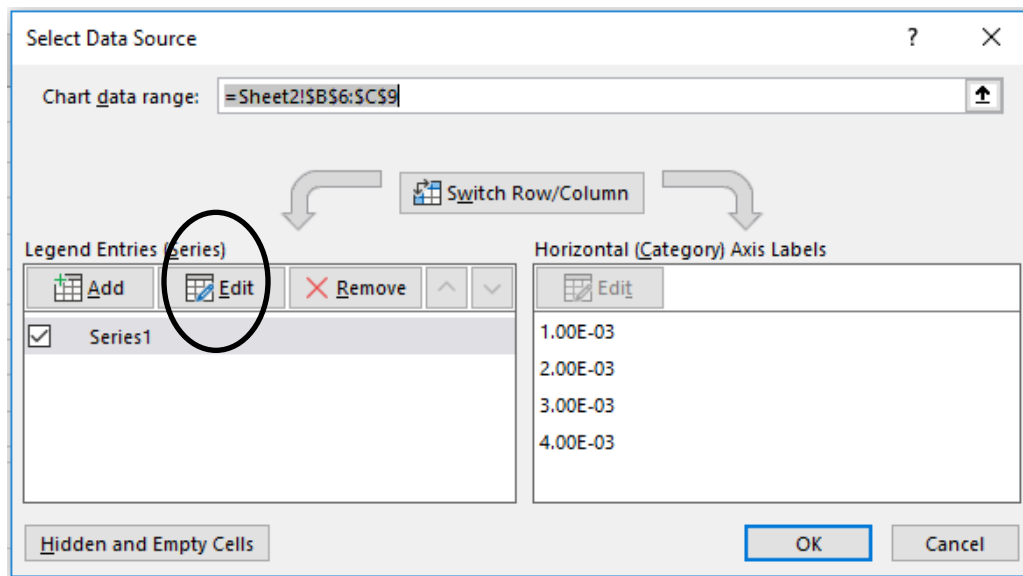
You can type in a name for your series—for example, "Absorbance". The Series X values show that cells B6 to B9 have been chosen as the x values. Similarly, cells C6 to C9 are the y values. This is correct for our plot (concentration on the x-axis and absorbance on the y-axis). However, if necessary, these ranges can be changed by clicking on the up arrow and highlighting a different range of cells. Choose **OK** to finish.

> Note that the y data points are considered series data. If you have more than one set of y data (several series of data), you can add more to the existing plot by selecting **Add** and choosing appropriate cell ranges for the x and y values. You can also delete a series of data by first clicking on the series of interest in the **Series box** and clicking on **Remove**.
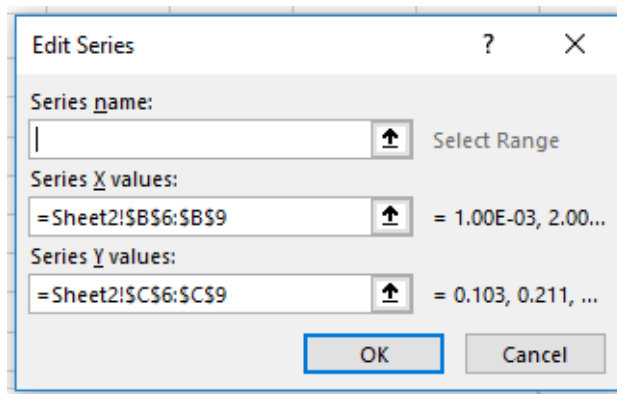
Click on the Chart Title in the chart window and type "Calibration Curve for $Fe(SCN)^{2+}$ solutions." (The superscripted 2+ charge can be done by highlighting the 2+ in the textbox and then choosing the Font option from the Home menu.)
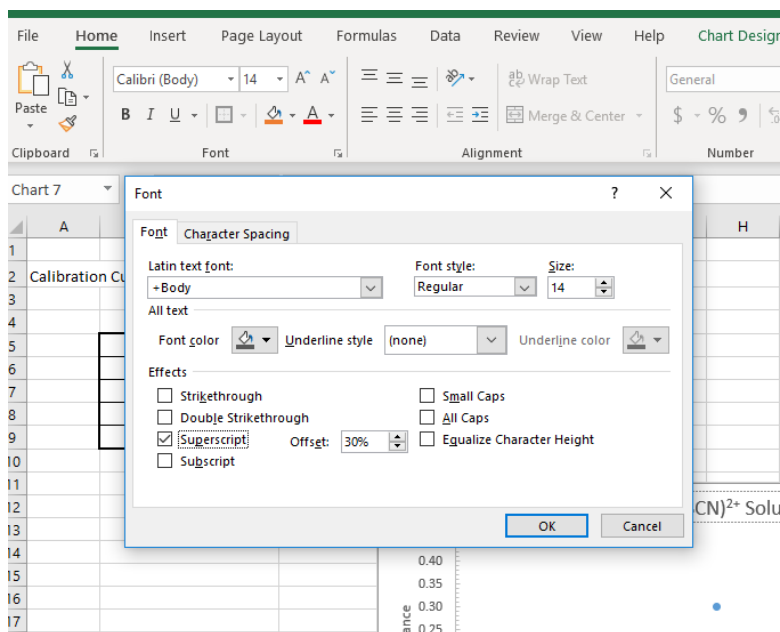
Next, Click on the + sign next to the chart to add Axis Titles to the chart and remove gridlines. Change the x-axis title to "Concentration (M)" and the y-axis title to "Absorbance" by clicking in the axis title box and typing in the correct titles.
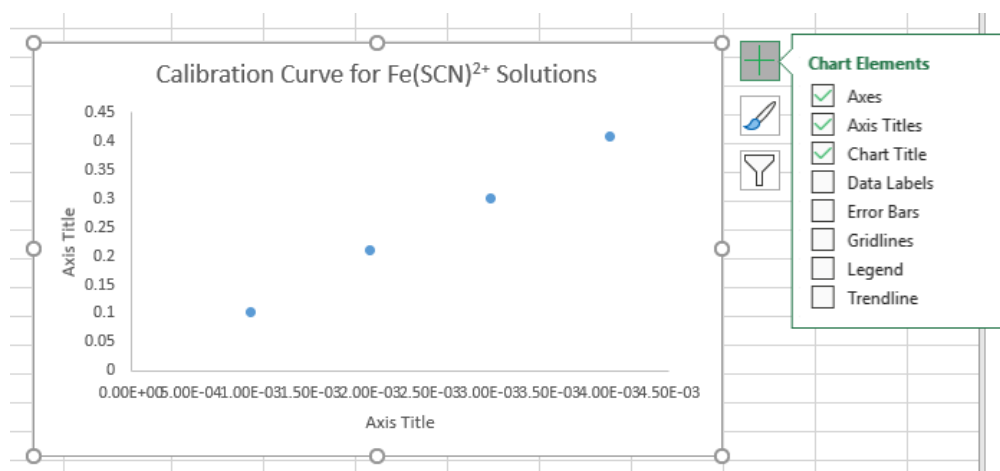
The legend option in the Chart Elements shows what series were plotted. The legend option is most useful if more than one series is plotted (*i.e.* more than one set of y values are plotted on the same graph which then requires a legend to distinguish between the data sets). Since there is only one set of y values plotted, deselect the **Legend** box so that no legend appears on the graph. The **Data labels** option in this menu allows you to show the x and y values for each data point. This is not commonly used on graphs, so deselect.

Double click on the x-axis values in the chart to bring up the Format Axis menu shown to the right (similarly, double clicking on the y-axis values will allow you to format the y axis).

From this menu, you can change the minimum and maximum x-axis values and major and minor unit divisions. You can also add tick marks and change how the axis labels are formatted. Play with these options to format your chart as shown on the next page.
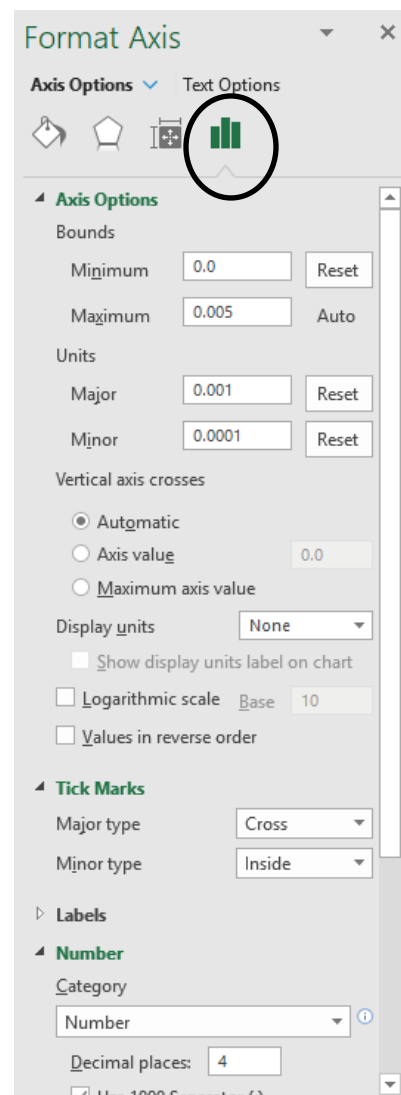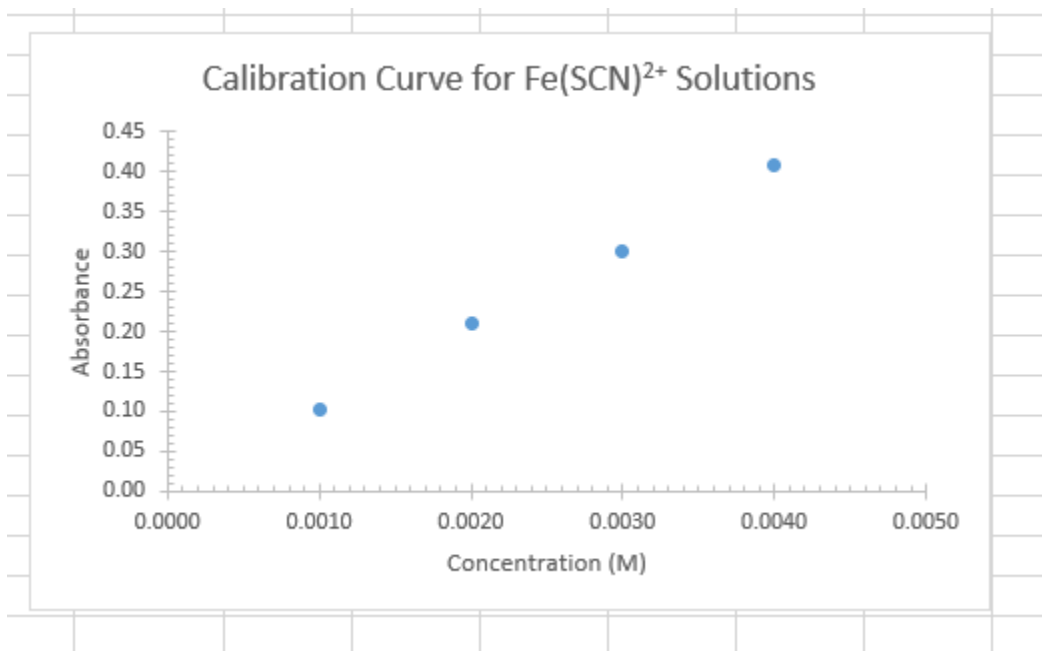
Here, the axis labels and major and minor tick marks have been chosen to reflect the level of precision of the measured values (*e.g.* the absorbance data has 3 significant figures, so using 2 decimal places for the axis labels represents the decimal places known for certain).

If you want to manipulate anything on the graph to further modify it, click once anywhere on the graph. You will notice that the graph is outlined now and that there is a new menu that appears at the top for graphing. The spreadsheet now has two parts, a data part (where the cells are located) and a graph part. If you want to alter anything on the data part of your spreadsheet, just click any cell and the main menu bar changes so to reflect that you are working on the data part of the spreadsheet (gives you menu options for changing the data part of the spreadsheet). If you click anywhere on the graph part, the main menu bar changes to reflect that you are working on the graph (gives you menu options for changing the graph).

Next we want to add a linear regression **trendline** to the plot. This will add the best-fit line to the data. The slope of this line would give the Beer's constant. The Beer's constant is useful so that when you measure the absorbance of an unknown $Fe(SCN)^{2+}$ solution, you can determine its concentration by using Beer's Law: $A = bC$ where A is absorbance, C is concentration and b is Beer's constant. This equation works if the y-intercept is equal to zero, *i.e.* the best-fit line passes through (0, 0) on your graph. To do the linear regression, return to the **Chart Elements** menu and click on **Trendline**.
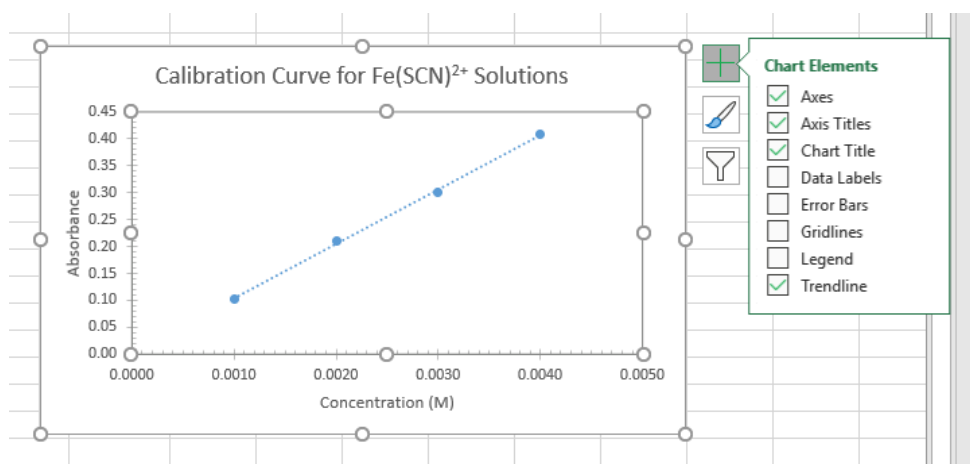
By holding the cursor to the right of the Trendline label, you can access an additional menu to format the trendline. Choose Linear, and set the options as shown below to force a 0,0 intercept and display the equation and R^2 value on the chart. (Backward forecasting extends the line drawn to the origin.)
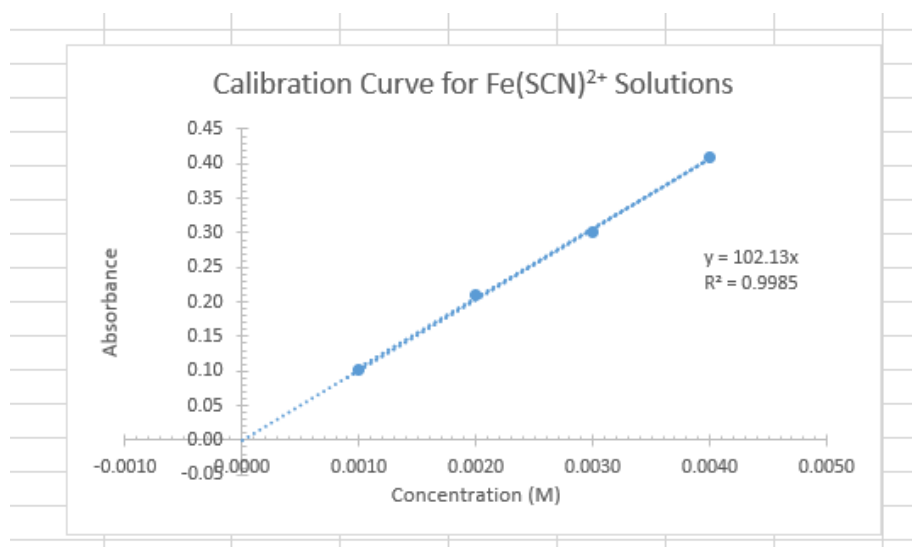
The equation of the line (y = mx + b) gives two pieces of important information, namely the slope (m) and the y-intercept (b). The R^2 value gives you an indication as to the degree that the data best fits a line (generally the closer the data fit a straight line, the higher the R^2 value. The maximum value for R^2 is 1).

The displayed equation and the R^2 value can be moved around on the chart if needed so that it is not on the line by clicking on the text box and dragging it to another spot on the chart.

There may be a time when you need to increase the number of significant figures on the slope and/or y-intercept in your regression output (some programs round it to 1 significant figure and this introduces a lot of rounding error in any subsequent calculations using this data). You can estimate the number of significant figures that should be in your value of the slope by doing a quick calculation by hand of the slope by choosing two data points on the line and estimating these points to the proper number of significant figures.   Use the correct rules for significant figures when doing the calculation to determine the number of significant figures that should be in your final slope value. Then, adjust the number of decimal places in the output of the regression data to have a few more digits than required in order to minimize rounding errors in subsequent calculations that require the use of the slope value. Click on the trendline label and use the Format Trendline Label menu that appears to adjust the number of significant figures shown.
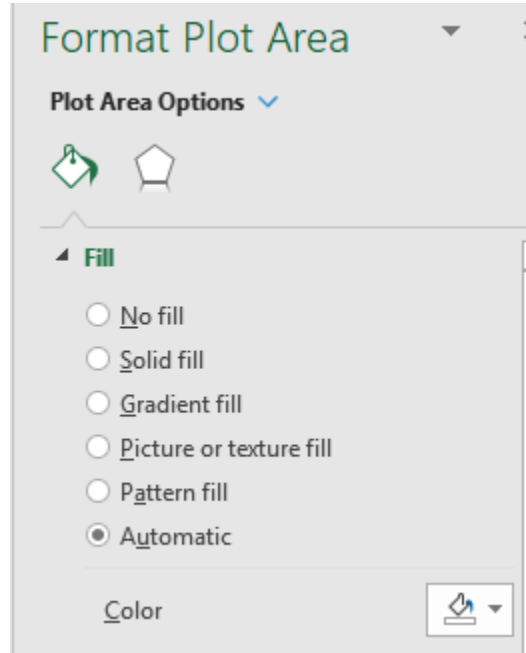
SPREADSHEET AND GRAPHING WITH EXCEL

Printing charts and graphs

Unless color is needed, it is best to keep the color of the plot area of the chart white. Click on the plot so that it is selected and the **Format Plot Area** menu will appear**.** Choose **Automatic** or **color white.**

If both the data table and chart are to be printed on the same page, select both by clicking and dragging the mouse over the entire area. Notice that selected area has a gray background.   Under **Page Layout**, select **Print Area**, then **Set Print Area**. The graph or the data table and graph can be printed in one of two styles: Portrait (vertical) or Landscape (horizontal). Choose the appropriate style under **Page Layout**, **Orientation**. Then choose **Print** under **File**. You may also need to adjust the scaling so that everything fits on one page. It is always a good idea to do a Print Preview so that any mistakes can be fixed before the actual printing. Doing so will save you time and paper.

If only the graph is to be printed, click on the chart so that it is highlighted and then click **File** and then **Print**.

There are a variety of short cuts and icons that can be used to make working in Excel easier that are not covered in this tutorial. Although the format of the spreadsheet might change depending on the version of Excel or Mac/PC, the basic guidelines will remain fairly the same. For additional help with items not covered in this tutorial or on more advanced Excel features, see your professor.