Introduction 1

Instrumentation in Lab

Purpose

A variety of commonly-used equipment in the laboratory will be introduced with instructions for their proper use and maintenance for review during experimentation throughout the lab course.

Background

A chemistry laboratory is full of a great deal of equipment, much of which you will be introduced to and use to perform the experiments in this lab manual. Knowing how to use and care for your equipment safely as well as how to record data appropriately in your data sheets, lab notebooks, and lab reports will help you complete your experiments more efficiently and yield better, more accurate results.

This list is composed of the most commonly used equipment in General Chemistry I lab and is by far not a comprehensive list of all that you will see in this class and your future chemistry courses. It is divided into the following categories:

Measuring Mass

Measuring Volume

Measuring Temperature

Measuring Pressure

Measuring Absorbance

Using LabQuest 2

Using Graphical Analysis

Measuring Mass

Electronic Balances

Most commonly mass is measured by using the electronic balances in the laboratory classroom. The balances are accurate to 0.001 g or 0.0001 g.

1. With all sliding plexiglass doors closed, tare the balance. It should read 0.0000 g with nothing on it.
2. If measuring a chemical, use a clean, dry weigh boat or other glassware. Do not ever put a chemical directly on the balance. The mass of the weigh boat or glassware can either be recorded and subtracted from the total mass or tared out.
3. Close all doors. Allow the mass to stabilize and then record **every** digit shown on the screen.
4. Do not add or pour chemicals on the balance. Remove your weigh boat or glassware, add the chemical, and then reweigh.
5. Always use the **same** balance throughout one experiment. This minimizes potential error.
6. Clean up any spills **immediately.** If you are unsure how, ask your instructor.
7. Do not pour any excess reagent back into the reagent containers. Dispose of it in the appropriate waste container instead to avoid contamination.
8. Glassware should always be room temperature when weighed—not too hot and not too cold.

Measuring Volume

Liquids should always be measured to the bottom (if concave) or the top (if convex) of the meniscus.

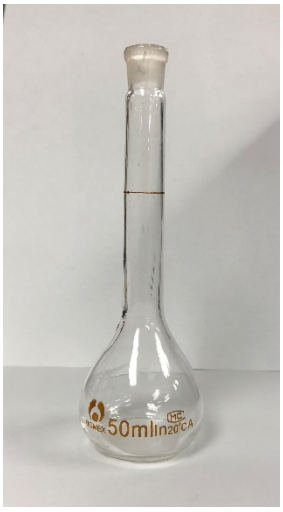
Graduated Cylinders

Your assigned lab drawer should have a large (100 or 50 mL) and small (10 mL) graduated cylinders. These are marked in increments of 1 mL or 0.1 mL and are best for measuring liquids.

1. Make sure the graduated cylinder is clean and dry.
2. Add the chemical carefully until the lowest part of the meniscus touches the line for the desired volume.
3. Record the actual volume measured. You should include one estimated digit past the markings.
4. Avoid cross-contaminating solutions by washing your graduated cylinder thoroughly with deionized water between chemicals.
5. Do **not** pour excess reagent back into the bottles. Dispose of it in the appropriate waste container instead to avoid contamination.

Volumetric Flasks

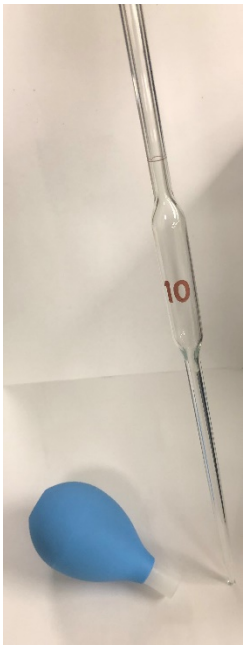
Volumetric flasks are calibrated to measure accurately and hold a single volume of liquid which is indicated on the glassware itself.



1. Make sure the volumetric flask is clean and dry.
2. Add the chemical carefully until the lowest part of the meniscus touches the calibration mark.
3. Dry any liquid that is in the neck of the flask above the calibration line with a paper towel to remove it.
4. Record the volume marked on the glassware. The decimal places to be included will also be marked. As a rule, volumetric glassware is considered accurate to the 0.01 mL (e.g., a 50 mL volumetric flask should be recorded as 50.00 mL).
5. Do **not** heat or chill volumetric glassware. The expansion of the glass will change the volume.

Volumetric Pipettes

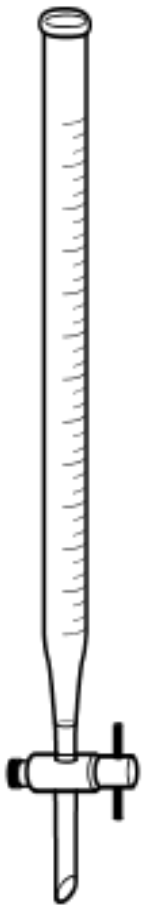
A 5.00 mL volumetric pipette and rubber bulb is provided in your assigned lab drawer. Other volumes will be available for particular experiments. These pieces of glassware are calibrated to measure and deliver a single volume of liquid which is indicated on the glassware itself.



1. Clean the pipette with deionized water by using the rubber bulb to pull enough water up to fill the pipette, then allow it to drain. Only one drop should remain at the very tip; no drops should be stuck inside.
2. If there are drops, the pipette needs to be cleaned. Heat approximately 30 mL of deionized water to boiling. Add a few drops of dish soap. Use the rubber bulb to pull up enough soapy liquid to fill the pipette, then allow it to drain. Repeat with pure tap water and again with pure deionized water. The last wash should leave only a single drop at the tip. If it does not, repeat the process again from the beginning.
3. Using the rubber bulb, pull the liquid up into the pipette until the bottom of the meniscus just touches the calibration mark. This may take repeated tries. Holding your finger over the top of the pipette will keep the liquid in place. It may be easier to pull up more liquid than needed and then let it drip out. To slowly form drops, hold your index finger lightly over the top of the pipette and with your other hand spin the pipette gently. A small drop should begin to form at the very tip.
4. Pull off the rubber bulb and keep your finger on the top of the pipette. Move the pipette over to the desired glassware and release your finger. The liquid should drain out by itself and leave a drop or two at the very tip. Do **not** blow this out; the pipette is calibrated to allow this.
5. Do **not** pipette directly from reagent bottles. Instead, pour a little more of the reagent than needed into a beaker and bring it back to your desk. Use your pipette in your beaker.

Burets

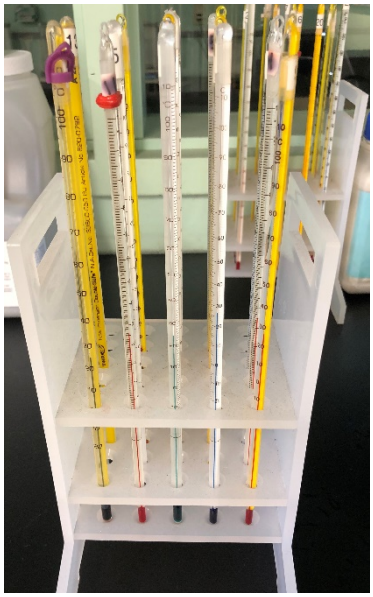
Burets are marked much like graduated cylinders, showing incremental volumes representing 0.1 mL. The top line represents 0.00 mL and the bottom 50.00 mL. When the bottom valve is vertical as shown in the drawing below, the buret is open. When it is horizontal, the buret is closed.



1. Clean the buret with a small amount of the chemical to be used. While the buret is closed, pour some inside and, turning it sideways, roll it carefully around, up, and down the interior of the buret. Try to cover all areas where the chemical will touch. Drain and then dispose of the wash before using.
2. Clamp the buret vertically. Place a beaker underneath it and open the valve all the way, allowing the wash to drain through. The flow should be consistent and there should be no bubbles in the tip. If the flow drips or is unexpectedly slow, check for blockages and clear any out that are found. If bubbles form in the tip, gently tap on the side of the glass to release them.
3. Discard the wash in the appropriate waste container.
4. Fill the buret with your desired chemical somewhere close to but below the 0.00 mL line. It is not necessary to get it right on the line. Hold a white piece of paper behind the buret to read and record the exact measurement as the initial volume. It may be helpful to use a funnel to pour the liquid in with, but be sure to remove it before measuring the initial volume and using the buret.
5. Dispense the liquid from the buret by slowly turning the valve horizontally.
6. When the titration is completed, close the valve by turning it vertically. Allow any drops formed on the tip to fall. Hold a white piece of paper behind the buret to read and record the exact measurement as the final volume.
7. Calculate the volume dispensed by subtracting final – initial.
8. Never fill the buret directly with the reagent container. Instead, pour some chemical into a 50 or 100-mL beaker and then pour it into the buret.
9. When finished with your experiment, wash the buret out with deionized water before returning.

Measuring Temperature

Glass Thermometers



A class set of glass thermometers filled with alcohol is available for use in the lab. They are marked to the 1°C and should be recorded to the 0.1°C (estimated digit).

1. Place the thermometer in the substance to be measured. It should be deep enough to cover the lower bulb but not touching the bottom or the sides of the container.

2. Wait at least one minute for the temperature to stabilize.

3. Clean the thermometer between uses to avoid contamination.

4. Use the same thermometer throughout the entire experiment to minimize error.

GoDirect Temperature Probe

For some experiments, the Vernier electronic systems with appropriate sensors will be provided. Refer to the sections of Graphing Analysis 4 and LabQuest 2 for further instructions.

1. The temperature probe can be connected to either a lab computer or a LabQuest 2 interface via USB cord or Bluetooth. Make sure that both are turned on (the sensor should be flashing a red light). If using a lab computer, make sure that Graphing Analysis 4 is open.

2. The software should automatically detect that the temperature probe is connected. The screen should have a red box which shows the current temperature to 0.1°C.

3. If needed, sampling options can be changed to adjust settings. On the LabQuiest 2, press Sensors, then Data Collection. On Graphical Analysis 4, press the Mode button in the bottom left-hand corner.

4. To start recording temperature, press the green triangle at the left-hand corner of the screen. The button should change to a red square. To stop recording, press this same button again.

5. Graphs are recorded automatically. These can be analyzed on the LabQuest 2 interface directly or exported via flash drive to a computer for analysis and printing. Data can also be exported via flash drive.

Measuring Pressure

Atmospheric Barometer

There is one mercury barometer in each laboratory classroom that measures the atmospheric pressure for the day. It is usually kept in the same room as the balances. The units for this barometer are millimeters of mercury (mmHg). It is marked to the 0.1 mmHg.

1. While level with the meniscus of silver mercury inside the upper glass tube, read its height with the ruler attached to the right-hand side (mm). Mercury's meniscus is convex, so read its height at the very top of the curve.
2. If required, also record the temperature of the mercury inside the barometer by using the thermometer attached below it.

GoDirect Pressure Probe

For some experiments, the Vernier electronic systems with appropriate sensors will be provided. Refer to the sections of Graphing Analysis 4 and LabQuest 2 for further instructions.

1. The pressure probe is provided as a kit that includes a syringe and a stopper and tubing for an Erlenmeyer flask. These will be used for particular experiments.
2. The pressure probe can be connected to either a lab computer or a LabQuest 2 interface via USB cord or Bluetooth. Make sure that both are turned on. If using a lab computer, make sure that Graphing Analysis 4 is open.
3. The software should automatically detect that the pressure sensor is connected. The screen should have a red box which shows the current pressure.
4. If needed, sampling options can be changed to adjust settings. Press Sensors, then Data Collection.
5. To start recording pressure, press the green triangle at the left-hand corner of the screen. The button should change to a red square. If the data collection was changed to manual, then a second button will appear that says Keep that will need to be pressed whenever data is ready to be recorded. To stop recording, press the red square button.
6. Graphs are recorded automatically. These can be analyzed on the LabQuest 2 interface directly or exported via flash drive to a computer for analysis and printing. Data can also be exported via flash drive.

Measuring Absorbance

Spectrometer-20

A set of spectrometer-20 instruments may be provided for particular experiments. They can be set to read either percent transmittance (%T) to the 0.1% or absorbance (A) to the 0.001. Since their readings are shown on a digital screen, all digits should be recorded without any estimated place.

1. Turn on the spectrometer. Make sure it has been running for at least 10 minutes before starting to use.
2. Use small-sized test tubes for your samples. Make sure they are clean and without stains or scratches. Use Kimwipes to clean the outside of the glass for the entire bottom three-quarters of the test tube.
3. Use the knob on the top of the instrument to change to the desired wavelength.
4. With the sample holder empty and its door closed, adjust the reading to 0.0% T using the knob on the front of the instrument labeled "0% T".
5. Create a "blank" by filling a small test tube approximately three-quarters full with pure solvent. Insert this into the sample holder. Close the door. Adjust the reading to 100.0% T using the knob on the front of the instrument labeled "100% T".
6. Remove the blank and close the sample door. Ensure that the instrument still reads 0.0% T. If it does not, readjust the 0% T knob until it does and reinsert the blank again to ensure that it reads 100.0% T. Readjust the 100% T knob if needed. Repeat this as many times as necessary.
7. If desired, change the instrument's reading by pressing the Mode button until it reaches the desired setting.
8. Wipe the outside of the sample test tube with a Kimwipe. Insert it into the sample holder and close the door. Record all digits shown on the screen.

Vernier SpectroVis Spectrophotometer

For some experiments, the Vernier electronic systems with appropriate sensors will be provided. Refer to the sections of Graphing Analysis 4 and LabQuest 2 for further instructions.

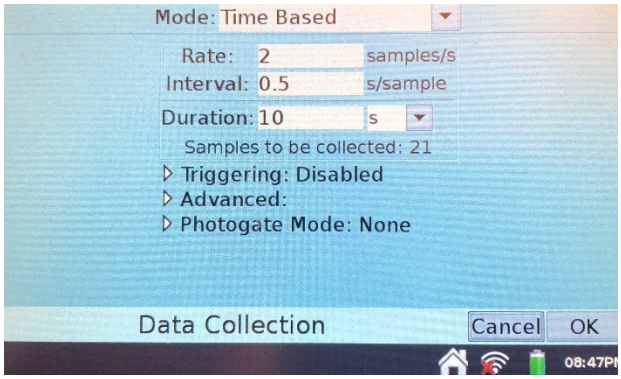
1. The SpectroVis Spectrophotometer will be provided as a kit that includes two small plastic cuvettes and caps. Do not lose this cuvette as it is the only one that will fit into the spectrophotometer.
2. The spectrophotometer can be connected to either a lab computer or a LabQuest 2 interface via USB cord or Bluetooth. If using a lab computer, make sure that LoggerPro 3 is open.
3. To calibrate the spectrophotometer, go to the Experiment menu and select Calibrate, then Spectrometer.
4. Fill one of the plastic cuvettes about three-fourths of the way full of pure solvent as your "blank". Cap it. Clean the smooth sides of the cuvette with a Kimwipe to remove any dirt or fingerprints. Slide the cuvette into the sample holder, making sure that a blank, smooth side is facing the path of the light in the spectrometer indicated by the triangles on opposite sides of the sample holder.
5. Follow the instructions in the dialog box to complete the calibration and then press OK.
6. To find the wavelength of maximum absorbance (λ_{max}), fill a clean cuvette approximately three-fourths full of the solution to be tested. Cap it. Clean the sides with a Kimwipe and then place it in the sample holder of the spectrophotometer.
7. Click Collect. Once a full spectrum has been recorded (all wavelengths included on the x-axis), click Stop to end data collection.
8. To conduct a Beer's Law experiment, click on the Configure Spectrometer Data Collection button. Select Abs vs. Concentration as the collection mode. The wavelength will automatically be selected from the previous graph. To change it, click Clear and select a wavelength on the graph or in the list of wavelengths. Otherwise, click OK.
9. Place your first sample in a clean plastic cuvette. Cap it. Clean the smooth sides of the cuvette with a Kimwipe. Slide the cuvette into the sample holder, making sure that a blank, smooth side is facing the path of the light in the spectrometer indicated by the triangles on opposite sides of the sample holder. Press Keep. When prompted, enter the concentration of the sample and click OK.
10. Repeat Step 9 for as many samples as required. After you have tested the last sample, click Stop to end data collection.
11. If a trendline equation is required, click the Linear Fit button to see the equation displayed on the graph.

Using LabQuest 2

Any electronic Vernier sensor can be connected via USB or Bluetooth to a LabQuest 2 Interface to collect and graph data. This can then be exported to a computer via a USB flash drive for further analysis or printing.

1. Press and release the power button located on the top edge of the LabQuest 2 to turn on or restart the unit. The LabQuest App should launch automatically.

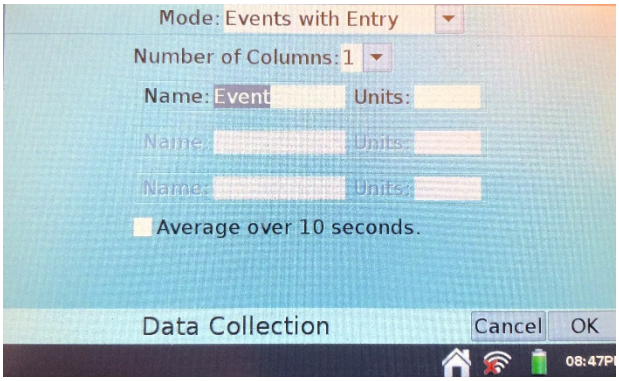
2. To connect via USB, slide the sensor into the correct port. To connect via Bluetooth, turn the sensor on first. Go to Sensors, then Wireless Device Setup, and Go Direct. The interface will automatically scan for sensors. Select yours (it should match the number on the label affixed to the back of the sensor) and click OK.



3. To change settings, go to Sensors then Data Collection. The labs primarily use two modes:

- **Time Based**: The interface will automatically collect data over a set amount of time. The rate changes how many samples are taken per second and the duration changes how long before the interface automatically stops collecting data. Make sure that the duration is much longer than you expect to need; trials can always be stopped early but additional time cannot be added in the middle of a trial.

- **Events with Entry**: The interface will only collect data when instructed. Enter a Name and Units for your independent variable. An additional button will appear at the bottom labeled Keep. No data will be collected unless this button is pressed and a prompt will appear asking for the value of your independent variable.



4. To start data collection, press the green triangle (Start). If doing time-based, the data will be collected automatically and show up as a graph. If doing events with entry, data will only be collected when the Keep button is pressed.

5. To stop data collection, press the red square (Stop).

6. To analyze the graph, go to Graph then Graph Options. Select Autoscale and press OK.

7. To calculate a trendline equation, go to Analyze, then Curve Fit. Select the correct type of line from the drop-down menu under Fit Equation (usually linear). The coefficient (slope, m, and x-

intercept, b) are shown in their corresponding boxes. RMSE stands for Root Mean Square Error and is a way of measuring how close the curve fit is to the actual data. By default the interface will plot a trendline for the entire set of data. If only a particular set is desired, select this on the graph window first before opening Curve Fit.

8. To transfer data, unplug the sensor from the USB port (if using) and insert a flash drive. Go to File, then Export. Select your flash drive as the destination. The file will save as a .txt which can be opened on any computer with Excel or other programs.

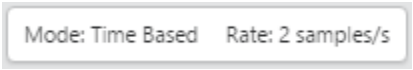
Using Graphical Analysis

Any electronic Vernier sensor can be connected via USB to a lab computer while using Graphical Analysis to collect, graph, and analyze data.

1. Open Graphical Analysis on the lab computer. Select "Sensor Data Collection" on the start screen.

2. Connect your sensor via USB port to the lab computer. The program should recognize the sensor automatically.

3. To change settings, click the Mode button in the bottom left of the screen. The labs primarily use two modes:



- **Time Based**: The interface will automatically collect data over a set amount of time. The rate changes how many samples are taken per second and the duration changes how long before the interface automatically stops collecting data. Make sure that the duration is much longer than you expect to need; trials can always be stopped early but additional time cannot be added in the middle of a trial.
- **Events with Entry**: The interface will only collect data when instructed. Enter a Name and Units for your independent variable. An additional button will appear at the bottom labeled Keep. No data will be collected unless this button is pressed and a prompt will appear asking for the value of your independent variable.

4. To start data collection, press Collect. If doing Time-Based, the data will be collected automatically and show up as a graph with a table on the right hand side. If doing Events with Entry, data will only be collected when the Keep button is pressed.

5. To stop data collection, press the Stop button.

6. To edit the graph, press the button in the bottom left-hand corner. 

- To add a title or change axis values, go to Edit Graph Options.
- To calculate a trendline equation, go to Apply Curve Fit. Select the correct type of line from the drop-down menu under Curve Fit (usually linear). The coefficient (slope, m, and x-intercept, b) are shown in their corresponding boxes. RMSE stands for Root Mean Square Error and is a way of measuring how close the curve fit is to the actual data. By default the interface will plot a trendline for the entire set of data. If only a particular set is desired, select this on the graph window first before opening Curve Fit.