Introduction to Small Scale Chemistry

Goals

- Introduce small-scale techniques
- Record both qualitative and quantitative observations
- Draw conclusions from results

Introduction

Small scale chemistry techniques involving a scaling down of reaction size and doing chemistry *in plastico* rather than using traditional glassware. Due to the decreased quantities of chemical reagents, the hazards, chemical waste and costs of experiments are also decreased. The obvious physical differences in small-scale chemistry leads to a very useful chemical difference. A distinct advantage to small-scale chemistry is the opportunity to work with reactions that would be difficult or dangerous on a larger scale.

"The drop is the container"

The surface tension of water allows us to replace our traditional test tube with drop size quantities, the drop being its own container. The volume of the drop can become reproducible with practice and consistent approaches.
Smaller amounts of reagents may require visual optimization using a hand lens (magnifying glass). This is usually kept at hand during all small-scale experiments.

"Good practice at any scale"

Consistent results that lead to meaningful conclusions require reproducibility. The most important technique in providing meaningful results in small-scale chemistry is avoiding contamination. The tip of any small scale pipet must be kept clean by avoiding contact with anything else, e.g., another drop, the reaction surface, another pipet. *Any contaminated pipet should be given to your instructor*. Reproducible results require *precision*. Whenever more than one trial or more than one sample is being tested for comparison, all of the trials/samples must be treated as similarly as possible. Many times students cannot control the *accuracy* of a balance but they can make sure that they use the same one throughout an experiment in an appropriate and precise manner.

Principles of Observation

Observations in the laboratory can be either quantitative or qualitative. Quantitative observations are called **measurements** and usually require the recording of a number and the unit of measure. Qualitative observations describe what is seen in the laboratory. Words that are more than superficially descriptive must be used when qualitative observations are made, e.g., the word "blue" does not convey anything specifically meaningful to someone who was not present. Adjectives describing color, size, vigor of reaction, or any other observable are a critical tool in the recording of experimental observations.

When reacting two substances together it is imperative that you monitor the reaction immediately upon mixing and continue to observe until no further change occurs. Some chemical reactions are slow and some are fast. Sometimes more than one reaction can occur as laboratory conditions may influence results (air, light, fumes, etc.).

"But what does it all mean?"

The interpretation of observations is what occurs in the formulation of **conclusions**. To simply state what you see is important. To extrapolate some meaningful conclusion from your data or sets of data requires more than observation.

Safety

Act in accordance with the laboratory safety rules of Cabrillo College.
Wear safety glasses at all times.
Avoid contact with all chemical reagents and dispose of reactions using appropriate waste container.

Materials

10-mL graduated cylinder, top-loading centigram balances, deionized water, transfer pipets, calculator, green food dye solution

Experimental Procedure

Techniques:

***Microburet "Pipet" Techniques I* – The Basics**

The microburet, often referred to as the pipet, is a versatile tool.

1. Fill 2 wells of a 24-well tray with water that has been colored with green food dye. The green dye solution is in a plastic bottle obtained from Reagent Central. Be sure to promptly return the bottle containing the green dye solution to Reagent Central.

2. Fill 2 wells of the 24-well tray with distilled water. The distilled water is available in plastic squeeze bottles located in a cabinet at the side of the room.

3. To make a microburet select a long stem pipet and cut the stem with your scissors at a point about 2 cm from the bulb. NOTE: *make the cut at right angles!*

4. Begin by cleaning the microburet. Draw a little distilled water up into the bulb, shake it so that all internal surfaces have been wetted.

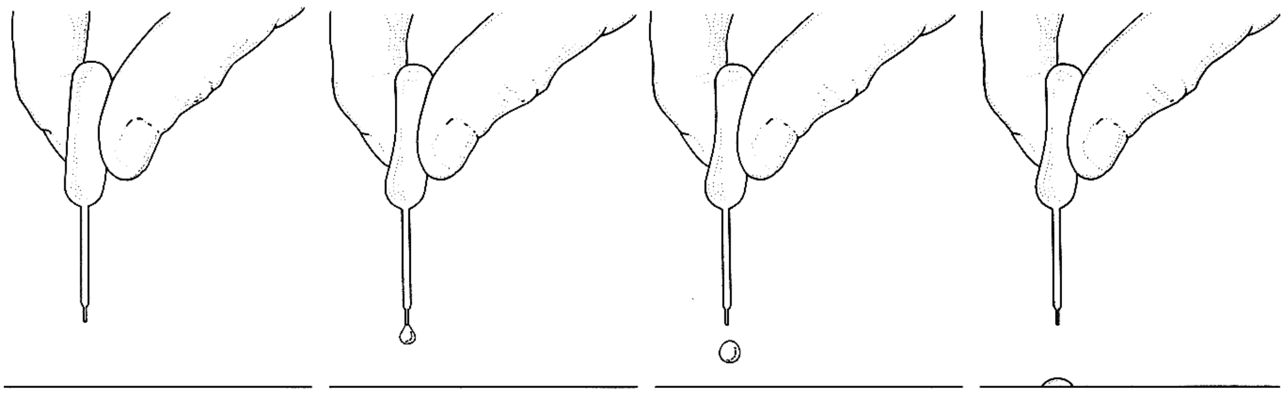
5. Holding the microburet vertically, expel to the waste cup. Press firmly to get the last drops out.

6. Now draw a little green food dye into the bulb, shake to rinse the bulb, and expel to the waste cup.

7. Finally, squeeze the bulb and draw green food dye into the microburet.

   Note: This sequence (steps 4, 5, 6, and 7) is known as *good wash, rinse, and transfer technique*.

8. Practice producing pools of various size on the labtop surface using the single drop *standard delivery technique*. To do so, hold the microburet *vertically* over the reaction surface as in the figure below, so its tip is about 1 to 2 centimeters above the reaction surface. Never touch the tip of the pipet to the reaction surface. "Touching" drops onto the reaction surface causes contamination of the contents within the microburet. Always drop the drops, never touch the drops onto the reaction surface. This technique, with the microburet held *vertically*, is called the *standard delivery technique*.



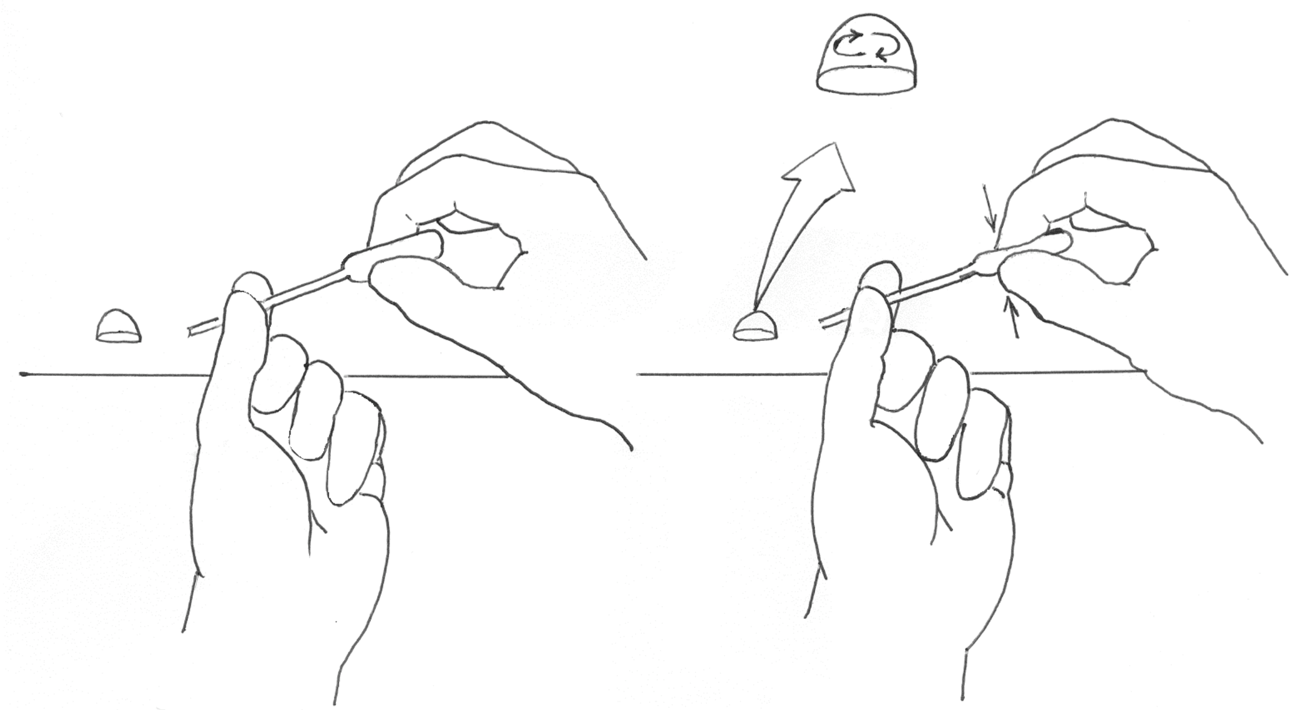
9. Make another microburet.

10. Follow the *good wash, rinse, and transfer technique* (steps 4-7) to fill the pipet with deionized water from one of the wells in the 24-well tray.

11. Drop a single drop of deionized water onto the reaction surface.

12. Now drop a drop of the green food dye solution on top of the drop of water, being careful not to touch the tip of the microburet to the deionized water drop.

13. Throughout this semester we will observe many chemical reactions between two substances conducted in this manor. While some visible change may accompany the initial mixing of two substances, often times thorough mixing is required to allow the reaction to go to completion. You can stir a hemispherical droplet on a reaction surface by gently blowing air from an empty long-stem pipet as shown below. Use one hand to steady the tip of the pipet and aim it at the side of the droplet. With the other hand gently squeeze the bulb repeatedly. The moving air will cause the droplet to swirl, mixing the contents without causing contamination.



14. Practice mixing drops of green food dye solution and deionized water until you are comfortable with the technique.

15. When finished, clean the reaction surface by first absorbing the liquid onto a small amount of microtowel. Dispose of the contaminated microtowel into the solid waste bins provided. Next wipe the surface with a paper towel and deionized water. Finally, dry the surface with a paper towel.

Microburet "Pipet" Techniques II – Affects of Drop Angle

The microburet is a simple constant-drop-volume delivery device when properly used.

1. Refill the microburet containing the green food dye solution.

2. Locate the 1 × 12 well strip in your chemistry kit. The well holds a volume of 0.40 mL when filled to the rim so that there extends a slight convex bulge above the rim of the well.

3. Count how many drops it takes to fill a single 0.40-mL well using the *standard delivery technique*. To do so, hold the microburet *vertically* (90º) over the well so its tip is about 1 to 2 centimeters above the well strip. Remember, never touch the tip of the pipet to the reaction surface. "Touching" drops onto the reaction surface causes contamination of the contents within the microburet. Always drop the drops, never touch the drops onto the reaction surface. Record your result.

4. Repeat the process to verify your result. Record your result.

5. Now tilt the microburet at 45º angle. Count how many drops it takes to fill the well with the microburet at 45º. Record your result.

6. Repeat the process to verify your result. Record your result.

7. Now tilt the microburet so that it is horizontal (0º) over the well and see how many drops are required in this position. Record your result.

8. Repeat the process to verify your result. Record your result.

The point here is that in order to obtain consistent results, you must be consistent in your delivery technique!

Using a balance

- ❑ Never place reagents directly on the pan of a balance.
- ❑ Use the same balance throughout an experiment.
- ❑ Record all of the numbers given in the digital output.
- ❑ Take into account the mass of the weighing container by:

"Weighing by difference" where you will weigh the empty container and record its mass. The mass of the object is obtained by weighing the container with the object and then subtracting the mass of the empty container, or

"Taring the balance" by placing the empty container on the pan of the balance and then pressing the "tare" button to zero the balance. You may now place the object into the container and the balance will read the mass of the object only.

Design an experiment to find the mass of one drop of water, delivered by the *standard delivery technique*, in units of milligrams. Repeat your experiment to verify your results. Be sure to record your procedure and results.

Introduction to Small Scale Chemistry

Name_____________________________

Section____________ **Date**_______

Experimental Data: Record all your observations.

1. Affects of Drop Angle.

Number of drops to fill 0.40-mL well

	Trial 1	Trial 2	Trial 3
Standard Delivery Technique Vertical (90º)			
Halfway (45º)			
Horizontal (0º)			

2. Mass of a drop of water delivered by the *Standard Delivery Technique*.

Procedure:

Data:

Mass of a single drop __________________________