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Experiment 1: Orientation to Laboratory Safety and Equipment. Calibration of a Thermometer and Pipette.

Before We Begin

Materials

- ≥ 150 ºC thermometer
- 100-250-mL beaker
- hot plate
- 1.0-mL plastic pipette
- jack

Chemicals

- deionized water
- ice

Hazards, Precautions and Waste Disposal

- To prevent spillage, glassware should always be clamped to a ring stand with Bunsen clamps, especially when they will contain boiling liquids.
- Hot plates should be placed on jacks so that the heat source can be safely lowered from the liquid when finished.

Reading and Review

- For an overview of material safety data sheet (MSDS) information as well MSDS information for specific chemicals, go to:
    a. http://msds.chem.ox.ac.uk/interpretingmsds.html
    b. http://www.chemexper.com
    c. http://www.chemfinder.com
- Watch the video, *Program 1: Indentification, Assembly & Care of Specialty Glassware*.

Experimental Summary

- Watch the safety video and lab tour. Discuss lab policies.
- Determine your lab partner(s). Your instructor may assign partners.
- Calibrate a thermometer and construct a calibration curve.
- Calibrate a pipette and construct a calibration curve.
- Follow your instructor's directions for recording data and observations. You may be instructed to use the *Data & Analysis* page at the end of the experiment or a laboratory notebook.

Special Instructions

The barometer at Des Plaines is located at the front of the 2232 between the lab and the hallway.

The Organic Chemistry Laboratory Environment

Did you know?
The National Fire Protection Agency (NFPA) diamond divides chemical hazards into four categories: Health, Fire, Reactivity and Specific. Except Specific, all are rated on a scale from 0-4.

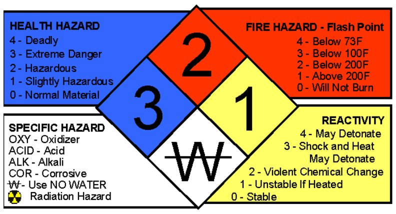

Figure 1.1: National Fire Protection Agency Diamond.

Working in an organic chemistry laboratory is quite different than working in a general chemistry laboratory. One major difference is the nature and hazards associated with the chemicals. Many organic materials are quite flammable. The best way to deal with the flammability problem is to evaporate flammable solvents in the hood with a glass cover nearby (to smother a potential fire). Never use a Bunsen burner in the lab unless specifically directed by your instructor. Be sure to properly assemble any reaction apparatus without leaks that might expose flammable vapors to a temperature beyond their flash point. Another difference is that organic chemicals can also be quite toxic and readily adsorbed through the skin. The best way to deal with this problem is to immediately wash any skin exposed to chemicals thoroughly with hot soapy water. If working with particularly toxic materials, it is good to protect your hands with nitrile gloves and to wear a lab coat.

Working in the organic chemistry laboratory is also very different because of the size and scale of the laboratory equipment and chemicals. All of Oakton’s organic experiments are done at the micro or semi-micro scale level. You will generally be working with less than one milliliter (mL) of a liquid and less than one gram (g) of a solid. The smaller scale requires specialty equipment, which is also small – this equipment is in a kit in your drawers. Because of the smaller quantities, micro scale labs provide a safer environment – it is much less likely that exposure will involve significant quantities or that fires cannot be readily smothered with a watch glass. It is also more cost effective in terms of chemicals used and more environmentally friendly in terms of reduced amounts of wastes. The smaller scale work will also demand careful techniques in order to obtain a measurable product.

For Experiment 1 you will be calibrating two frequently used tools – the thermometer in your drawer and the plastic disposable pipettes (the preferred device for small volumes).

Procedure A: Calibration of a Thermometer

Double Check!
The thermometer in your drawer should have an upper temperature range of 150ºC. If you have a thermometer with an upper range of only 100 ºC or 110 ºC, exchange it in the stockroom for a thermometer with the correct upper range.

Did You Know?
Boiling point is defined as the temperature at which the atmospheric pressure (P_{atm}) is equal to the equilibrium vapor pressure (P_{eq}) of a liquid. Since the pressure and temperature of gases are directly related by the ideal gas law (Equation A1-1), we have to make sure that both the measured temperature and the standard temperature correspond to the same atmospheric pressure.

You may be surprised to learn that the thermometer in your drawer may not accurately measure temperature. This does not make it useless, however. A calibration curve can be constructed by plotting known temperatures of water against measured temperatures. In our case we’ll use the boiling point (bp) and freezing point/melting point (mp) of water since these values are known.

Freezing Point Measurement

Determine the freezing point of water (or melting point of ice) with your thermometer. Prepare a slurry of deionized water and ice in a 100 mL beaker. Allow 5-10 min for equilibrium to be established. Insert your thermometer; wait for a few minutes; then, check the temperature. Wait a few more minutes and recheck the temperature. Continue until there is no further change in the temperature showing on your thermometer. The temperature of the slurry should be close to 0 ºC (the standard melting point of water).

Boiling Point Measurement

Determine the boiling point of water (or condensation point of steam). Fill a 150-250-mL beaker halfway with deionized water and heat it to boiling on a hot

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$$

Equation A1-1: Combined Ideal Gas Law.

This can be done easily by using the combined ideal gas law (Equation A1-2) to convert our measured temperature to standard temperature and pressure (STP). Standard pressure (P_{STP}) is 760 torr. Standard temperature is 273.15 K. The atmospheric pressure that day ($P_{measured}$) can be determined by reading the barometer in the lab.

$$\frac{P_{STP}}{T_{STP}} = \frac{P_{measured}}{T_{measured}}$$

Equation A1-2: Combined Ideal Gas Law for Pressure and Temperature.

plate. After the water is boiling vigorously, insert your thermometer, wait a few minutes, then read the temperature registered on your thermometer. The actual temperature of pure, boiling water at 760 mm Hg pressure is 100^{O}C. Read the atmospheric pressure in the lab using the wall-mounted barometer. Use this value to convert your measured temperature to the temperature under standard pressure (see side note).

Calibration Curve

Plot a calibration graph for your thermometer. The x-axis should be actual temperature and the y-axis the temperature as read from your thermometer and corrected to STP. This calibration graph should be done on graph paper, with a title, and the axes clearly identified. It may also be done in Excel. Step 1 above gives you one point and step 2 above gives you a second point – connecting these two points gives you a straight line. Determine the equation of the line you've drawn and write this on your graph. Tape or staple this calibration graph into your laboratory notebook.

Procedure B: Calibration of a Plastic Disposable Pipette

Did You Know?
A graduated cylinder is calibrated to measure the volume at the bottom of the liquid's meniscus. The convex surface of the liquid is caused by capillary action between the liquid and the sides of the graduated cylinder.

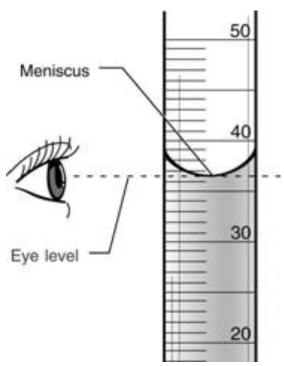

Figure 1.2: Viewing the meniscus in a graduated cylinder.

Calibrate a plastic, disposable pipette by determining the number of drops in 0.5 mL, 1.0 mL, 2.0 mL and 2.5 mL by pipetting into a small graduated cylinder (≤ 10 mL). You may need to fill the pipette bulb or refill the pipette to reach each volume. Each volume should be measured from an empty, clean graduated cylinder. Do not continue counting from the last measurement. Repeat each measurement at least three times. Take the average of all trials and use the averaged values in your calibration curve.

The calibration curve can be constructed by plotting the volume on the x-axis and the number of drops on the y-axis. Draw a best-fit line with a ruler. Then, determine the equation of the line you've drawn and write this on your graph. Alternatively, you may use Excel to plot your data. Use a ***TRENDLINE*** to find the equation of the line. Be sure to set your x and y-intercept to zero.

Each plastic pipette also has markings indicating 0.5 mL and 1.0 mL. Using your calibration curve, you will determine whether these markings are accurate by comparing the recorded number of drops from each pipette-labled quantity and the number of drops calculated from your graph.

1 Data & Analysis

Orientation to Laboratory Safety and Equipment. Calibration of a Thermometer and Pipette.

Name__________________________ Section Day / Time__________ Date___________

Group Members ____

PreLab Questions

None.

Calibration of a Thermometer

Measurements for the bp and mp of Water

standard freezing point of water _________________

standard boiling point of water _________________

current barometric pressure (torr) _________________

measured freezing point of water _________________

measured boiling point of water _________________

corrected measured boiling point of water ___________

Thermometer Calibration Curve

Plot your calibration curve below. Label each axis accurately and mark major divisions.

Questions for Procedure A

1. Show how the corrected boiling point of water was determined using the Ideal Gas Law.

2. What is meant by STP? What are these values.

3. Determine the equation of the line in your graph above. Show your work. Alternatively, attach an Excel graph with a trendline.

4. Using the equation above, determine the true temperature if the measurement on your thermometer read 56 ºC. Show your work.

5. Consider the boiling water in the calibration of the thermometer. Would it make a difference if the water had been boiling for 3 minutes or boiling for 6 minutes (assume there is sufficient water that some remains after 6 min of boiling)? Why or why not?

6. It is essential in this experiment that distilled water is used. Why? What would the effect be on the boiling and freezing temperatures if the water contained non-volatile solutes, such as sodium chloride?

Calibration of a Pipette

Pipette Drops

Record the number of drops for each volume (measured by graduated cylinder). Repeat each experiment three times.

	Drops in 0.5 mL	Drops in 1.0 mL	Drops in 1.5 mL	Drops in 2.0 mL
Trial 1				
Trial 2				
Trial 3				
Average				

Pipette Graduations Accuracy

Each clear plastic pipette has graduated markings of 0.5 mL and 1.0 mL. We want to determine whether these graduations are accurate or not. Draw water into each pipette up to the marked volumes and then determine how many drops are contained in each volume.

	Drops in 0.5 mL (pipette)	Drops in 1.0 mL (pipette)
Trial 1		
Trial 2		
Trial 3		

Pipette Calibration Curve

Using the values in the first table, ***Pipette Drops***, plot your calibration curve below. Label each axis accurately and mark major divisions.

QUESTIONS FOR PROCEDURE B

7. Determine the equation of the line in your graph. Show your work. Alternatively, attach an Excel graph with a trendline.

8. Using the equation above, determine how many drops should be contained in 3.7 mL?

9. Compare the values for 0.5 mL and 1.0 mL from your calibration curve to the measured values from your second table, ***PIPETTE GRADUATION ACCURACY***. What can you conclude about the accuracy of the pipette graduations?

10. Using your data, calculate the volume in milliliters of one drop from your pipette.