Determination of Molar Mass via the Dumas Method

1. Purpose

In this experiment you will determine the molar mass of an unknown liquid using a simplified variation of the Dumas method.

2. Background

In the early 19th century, Jean-Baptiste Dumas, a distinguished French chemist, created a relatively simple method for determination of the molecular weight of a substance. With this method, molecular weight is calculated by measuring the mass of a known volume of a vaporized liquid. Because the concept of the mole had not been developed in Dumas' era, he computed relative molecular weights based on relative gas densities. Though Dumas got mixed results based on erroneous assumptions concerning elements in the gas phase, he is credited with establishing values for the molecular weights of thirty elements.

In the modern version of the Dumas procedure, an Erlenmeyer flask is used rather than the glass bulb of Dumas' day. The temperature, pressure and volume of the vapor are determined and the molar mass is found utilizing the Ideal Gas Law.

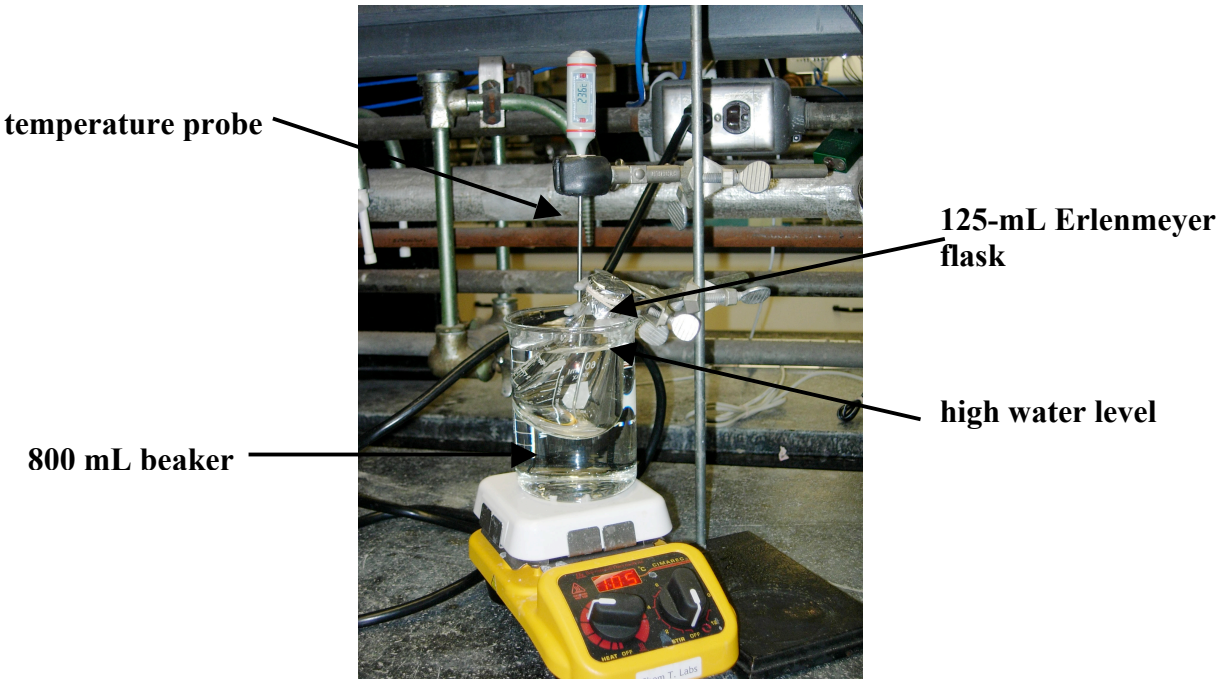
3. Procedure

Overview: A sample of a volatile liquid is added to a pre-weighed flask. The flask is submerged in a boiling water bath to vaporize the liquid. Because an excess amount of liquid is used, the volume of vapor produced is greater than the volume of the flask. Upon heating, the vapor that is created initially pushes the air out of the flask and then the vapor begins exiting the flask until the pressure inside the flask is equal to the atmospheric pressure. The mass of the vapor remaining in the flask is obtained by reweighing the flask. Additional measurements are made to determine the pressure, temperature and volume of the sample. Three trials will be completed to determine the average molar mass of the unknown liquid.

NOTE: All data should be tabulated. Data tables are to be prepared before attending lab.

A. Experimental apparatus

Assemble the apparatus as shown in figure 1. Be sure there is sufficient water in the 800 mL beaker to surround the bottom of the flask, but not so much that it overflows upon immersion of the flask. You do not need the foil and wire (or band) attached at this time. NOTE: To help assist observation of the unknown in part B, it is a good idea to mount the flask at a slight angle instead of perfectly vertically.



When you have the basic set-up accomplished, remove the flask. Place a boiling chip or two in the bath water and begin heating the bath. Dry the flask thoroughly. Cover the flask mouth with a small piece of foil and secure with wire or rubber band. Using a push-pin, make a pinhole in the foil cap. Measure (to the nearest 0.0001 g) and record the mass of the empty flask (w/ foil cap and wire).

B. Vaporizing the Unknown

Remove the cap and add approximately 2 mL of unknown to the flask. Secure the cap to the flask, and immerse the flask in the heating bath. Secure the flask with the clamp. IT IS EXTREMELY IMPORTANT THAT NO WATER GETS INSIDE THE FLASK. Mount the temperature probe in the bath, so it is as close to the unknown as possible. As the bath heats the liquid and the liquid begins to boil, follow the vapor up through the flask with your eye. Soon vapor will start to escape through the pin hole. The vapor is easiest to see if you are at eye level with the top of the flask and look across the top of the flask. The vapor will look like a colorless swirling cloud or jet. (It also helps to look toward a light source.) When all of the liquid has vaporized, including any droplets that

may have formed on the neck, no more vapor will be observed escaping through the pin hole (this should take no more than one to two minutes). NOTE: It can be difficult to observe the unknown vaporizing and streaming due to bubbling on the flask and water vapor generated by the bath. If seeing the vaporization is too difficult, simply allow the liquid to vaporize for approximately one minute after the bath has reached a steady boil.

As soon as the vaporization is complete (no more liquid in the flask, no more visible vapor escaping through the pinhole), record the temperature of the bath and remove the flask from the bath. CAREFULLY, THOROUGHLY AND QUICKLY, dry the outside of the flask. IT IS EXTREMELY IMPORTANT THAT THE EXTERIOR OF THE FLASK BE COMPLETELY DRY. Allow the flask to cool to room temperature, weigh and record the mass. (Do not remove the cap before weighing.) Repeat section **B** two more times. It is not necessary to empty or clean the flask between trials. If necessary, add more water to the water bath for trials 2 and 3. When the heating bath is no longer needed, turn off the hot plate.

C. Obtaining the Volume and Pressure of the Vapor

After the final weighing, remove the cap and wire. Fill the flask completely full with water. Measure the amount of water the flask can hold using a graduated cylinder

Record the barometric pressure. This is the same as the pressure in the flask.

D. Clean Up and Handling of Excess Unknown

Perform clean up and handling of excess unknown after the three trials in accordance with general laboratory guidelines and/or specific TA/Staff instructions.

4. Calculations

Before leaving lab,

a. Calculate the mass of unknown vapor in the chamber for each trial.

b. For each trial, calculate the number of moles of unknown vapor in the flask just after vaporization using the ideal gas law.

c. For each trial, calculate the molar mass of the unknown using n = mass/MM.

d. Calculate the mean value and standard deviation for the molar mass of the unknown.

e. Show your work to your instructor. If the calculation is correct, he/she will initial it and give you the name of your unknown. From its formula, calculate the molecular weight of the unknown.

f. Calculate the percent error of your measured molecular weight.

$$\%\ error = \frac{|\ accepted\ value - \exp erimental\ value\ |}{accepted\ value} \times 100$$

5. Discussion/Conclusion

Report the mean value of the unknown molar mass and the % error. Compare your value to the accepted value of the molar mass. Consider the following when discussing your results: What assumptions are made in this procedure, how do they contribute to the error and how do the different sources of error impact the results? For example, here are some questions to get you started:

From the time the mass of the flask is first measured until the final measurement, it is handled a number of times with oily fingers. How does this technique affect the reported mass of the vapor in the flask, and the final calculated molar mass? Is this error significant relative to other sources of error?

What is the measurement error of the graduated cylinder? Is this significant relative to other sources of error?

The barometric pressure is measured at the Eugene airport. If the pressure is different in the laboratory, how will it affect the results?

The flask is completely filled with vapor *only* when it is removed from the hot water bath. However, when the flask cools some of the vapor condenses in the flask. As a result of this observation, will the reported molar mass of the liquid be too high, too low, or unaffected?