BOILING POINT DETERMINATION

Introduction

This experiment investigates a characteristic physical property, namely the *boiling point*. In future experiments you will study additional physical properties such as *melting point* and *density*. You also will become familiar with the separation technique known as filtering. Physical properties can be used to identify a substance. For example, if you have prepared a white solid, you can determine its physical properties (density, melting point, boiling point, solubility, etc.) and compare these properties to known substances in the Handbook of Chemistry and Physics. If the boiling points, melting points, densities, etc., of the two samples are identical, then the two samples are almost certainly the same substance. The more physical properties measured the more likely the match.

Today's experiment is especially interesting since you will be measuring the boiling point of an unknown liquid using only a very *small amount* of the liquid: This sort of “micro” technique can be very useful in situations where a researcher may only have available a *few drops* of some new substance. It serves as a good introduction to your lab work in the General Chemistry course you are taking.

A second advantage of using this “micro” technique is that the amount of chemical needed and the corresponding waste generated is greatly reduced.

Before coming to the laboratory to perform this experiment, you should read over the sections in your textbook that cover the topics of boiling point and vapor pressure.

The **BOILING POINT** of a substance at a given pressure is a **CHARACTERISTIC** property of the substance, which may be used as a point of **IDENTIFICATION**. The boiling point of a substance is defined as *the temperature at which the pressure of the vapor of the substance becomes equal to the prevailing external pressure above the substance.* In the case of a system **OPEN TO THE ATMOSPHERE** in the lab, the prevailing external pressure is just the lab's **BAROMETRIC PRESSURE (atmospheric pressure)**. The boiling point of a liquid is thus a function of the prevailing pressure above the liquid. Changing the pressure above the liquid changes the boiling point of the liquid. For example, at sea level water boils at 100˚C if the atmospheric pressure is 760 mm Hg (30 in Hg). However, in Denver, where the typical atmospheric pressure is closer to about 660 mm Hg (26 in Hg), the boiling point of water is about 96˚C.

Since a liquid can boil at many temperatures, scientists have defined a reference pressure of 1 atmosphere (760 mm Hg or 30 in Hg) to measure the boiling point of a liquid. The temperature called the **normal boiling point** is defined *as the temperature at which a liquid boils when the external pressure (pressure above a liquid) is exactly 760 mm Hg*. This allows one to compare boiling points of various substances at a given reference point.

In order for a liquid to boil easily, there must be some place where *gas bubbles can form (nucleate)* easily; without this, "*bumping*" or "*superheating*" of the liquid can occur, leading to incorrect results or loss of the liquid being heated. When bulk amounts of liquids are to be boiled in the lab (such as in a water bath), **BOILING STONES** are used for nucleation. (Note: One should put only two or three boiling chips into the liquid being heated, not a handful of twenty to thirty.)

In this experiment, where we are dealing with a very small amount of liquid, a small broken **CAPILLARY TUBE** provides a rough surface upon which gas bubbles can form more easily than on the smooth walls of the container.

The instrument used in the laboratory for temperature measurement is, of course, the **THERMOMETER**. Before a thermometer can be considered *reliable* in its readings, it should be **CALIBRATED AT SOME REFERENCE TEMPERATURES**. That is, several systems of absolutely *known* temperature should be measured with the thermometer. If the thermometer is in error, one can draw a **CALIBRATION CURVE,** which will allow you to make corrections to the observed temperature reading on your thermometer. Applying this correction to the observed temperature reading on your thermometer will allow you to determine the actual temperature.

This curve can be used subsequently to determine the error likely in any given reading. For example, if your thermometer reads one degree too high at the boiling point of water, one would subtract one degree from the reading to get the correct temperature. You will calibrate your thermometer at the freezing point of water (by immersing it in an ice water slushy mixture whose temperature is exactly 0˚C) and at the boiling point of water (whose temperature is near 100˚C, but whose exact temperature depends on the days atmospheric pressure).

We can define the error of the thermometer to be *the observed temperature reading minus the correct temperature*. The correction can be defined as *the number of degrees that must be added or subtracted from the observed value to get the correct value*. For example, on a given day the atmospheric pressure is 770 mm Hg and the boiling point of water at this pressure is 100.4˚C. When we measure the boiling point using the thermometer provided, the temperature read 102.8˚C.

The error would be 102.8˚ - 100.4˚C = 2.4˚C

The correction would be to subtract 2.4˚C from the observed reading or -2.4˚C.

Since the thermometer is reading 2.4˚C too high, it has an error of +2.4˚C and the required correction is -2.4˚C (meaning that we need to subtract 2.4˚C from the observed value.)

ANSWER THE PRE-LAB QUESTIONS BEFORE COMING TO THE LAB EACH WEEK.

CHEMICAL INFORMATION

The unknowns used in this experiment are one of the following substances: methanol, ethanol or isopropyl alcohol (rubbing alcohol).

Methanol – flammable liquid, toxic by ingestion
Ethanol – flammable liquid, toxic by ingestion
Isopropyl alcohol – (rubbing alcohol) flammable liquid, toxic by ingestion

PROCEDURE

WEAR SAFETY GLASSES AT ALL TIMES!!

(1) Calibration of the Thermometer

Fill a 150 mL or 250 mL beaker with ice and add water to make a slushy mixture (about 80% ice and 20% water). Too much water will cause your solution to take too long to reach thermal equilibrium. **GENTLY** stir this slushy bath with your thermometer until the temperature reading **NO LONGER CHANGES**. Do **NOT** let the thermometer **TOUCH THE GLASS** of the container (or you will not be reading the temperature of the ice bath). Your thermometer must be read **WHILE IN THE ICE BATH** (it is **NOT** like a body thermometer, whose reading doesn't change unless it is shaken).

Record the thermometer reading to the nearest 0.2^{o}C (i.e., two-tenths of a degree, estimating BETWEEN the marks on the thermometer barrel). Allow the thermometer to warm up in a safe place on the bench.

If a rubber stopper is not already attached to your thermometer, use the following procedure to safely place a rubber stopper onto your thermometer. Using **GLYCERINE** as a **LUBRICANT**, and **PROTECTING YOUR HANDS WITH A TOWEL**, insert the **TOP** of your thermometer about l inch through a rubber stopper in such a way that the thermometer can still be read from 50 to 100^{o}C. (You only need to do this if your thermometer is not already attached to a rubber stopper.)

Obtain a 400 mL or a 600 mL beaker from your lab locker and fill it about 3/4 full with water from the sink.

Obtain a ring stand and a large ring from the area above your work space. Attach the ring to the ring stand. Find the wire gauze in your lab locker and place it on top of the ring. This will give more support to your beaker and also prevent the bottom of the beaker from becoming charred. Place the beaker filled with water on the wire gauze. Attach an adjustable clamp to the ring stand.

Suspend the thermometer in the water, using a clamp to hold the rubber stopper, again taking care that the thermometer does not touch the walls or bottom of the container.

Light your Bunsen burner with your burner lighter (sparker) and allow the water to boil. Wait until your temperature reading no longer changes, and then record the reading to the nearest 0.2°C in your lab notebook.

Your lab instructor will write the day's atmospheric pressure and its corresponding boiling point of water on the blackboard. Record this information in your lab notebook.

If necessary draw a calibration curve (this should be done if your thermometer shows any errors. Your lab instructors will go over during the pre-lab talk how to draw your calibration curve. The calculation section of this experiment will also describe how to draw a calibration curve.

(2) Boiling Point Determination

Obtain a small test tube from the table or hood where lab supplies are located in your lab room.

Obtain a 600 mL beaker from your locker.

Inspect the sample apparatus for assembling your apparatus **CORRECTLY**.

Connect your small test tube to the thermometer by using two rubber bands. (A rubber stopper should be on your thermometer; if not, attach one.)

Obtain a liquid unknown and record the unknown number in your lab notebook. Add sufficient unknown liquid to the test tube to give a liquid column height of about 4 cm (save the rest of the unknown for a repeat determination).

Break a 2 cm length from the **SEALED** end of a small **CAPILLARY TUBE**. Insert the small piece of capillary **SEALED END UP** in the tube of unknown liquid.

Fill your 600 mL beaker with **COLD** water, making sure that there is enough water in the beaker to **COVER** all of the unknown liquid in the sample tube. Be sure NOT to allow water to enter the **TOP** of the test tube (if water mixes with the unknown sample, erroneous results will be obtained).

Immerse the unknown liquid/thermometer apparatus, and then heat the water bath with frequent stirring until bubbles rise in a **STEADY STREAM FROM THE SMALL CAPILLARY**. (The water at this point should **NOT BE BOILING**: If the water is boiling, you have **HEATED TOO MUCH!**) It is likely that you will have boiled away all of your unknown. If this happens, you need to start this process again.

When bubbles have risen steadily from the small capillary for several seconds, REMOVE THE HEAT, stir the water bath **CONSTANTLY**, and record the temperature at the **INSTANT WHEN THE BUBBLES STOP COMING FROM THE CAPILLARY**. (At this point, the pressure of **VAPOR** inside the small capillary is equal to the atmospheric pressure.) **This is your boiling point.** NOTE: When the last bubble comes out of the capillary tube, the unknown liquid will start to fill up the capillary tube. Watch to see if you can observe this happening.

Record the temperature when the bubbling stops to the nearest 0.2^{o}C: This is the boiling point.

To check on your measurements, you will **REPEAT** the determination of boiling point. Refill the beaker with **COLD** water. If necessary, add more unknown liquid to the sample tube to preserve the 4 cm liquid height. The broken capillary used for the first determination will have filled with liquid as the temperature dropped, and cannot be reused (the original capillary does **NOT** have to be removed, however). Add a **SECOND** broken capillary tube to the unknown liquid sample, sealed end up.

Repeat the determination of the boiling point, following the same procedure as above. **YOUR SECOND DETERMINATION OF THE BOILING POINT SHOULD CHECK WITH THE FIRST DETERMINATION WITHIN TWO DEGREES: IF NOT, REPEAT UNTIL YOU GET TWO VALUES THAT AGREE.**

Return your unknown vial to the location from which it was obtained (probably a yellow or blue bin or a plastic tray).

Discard your unknown liquid from your small test tube into the bottle labeled **WASTE EXPERIMENT 1 BOILING POINT UNKNOWNS**.

Throw all glass to be discarded into the broken glass box located in your lab.

Wash your hands thoroughly before leaving the lab.

REMEMBER TO TURN IN THE YELLOW COPY OF YOUR LAB NOTEBOOK TO YOUR TA.

DRAWING A CALIBRATION CURVE

1. Assign the actual measured temperature to the x-axis.
2. Assign the error (or correction) to the y-axis.
   The error is how much your experimental determined temperature is off from what the actual temperature is. For example, your thermometer reads 2.6˚C (at the freezing point of water, where the actual freezing point of water is 0.0˚C). The error is +2.6˚C. Your temperature is 2.6˚C too high! The correction is what action you must take to get the correct temperature. In this example, one must subtract 2.6˚C from the experimentally determined temperature. The correction would thus be -2.6˚C.
3. Plot the experimentally determined temperature on the x-axis and the error (or correction) on the y-axis. Your graph should now have two dots, one for the boiling point and one for the freezing point.
4. Connect the two points with a straight line.
5. To read your graph, locate your experimentally determined boiling temperature on the x-axis. Draw a straight line up to your calibration curve. Lastly draw a straight line from your calibration curve over to the y-axis. You can now determine the error at that experimentally measured temperature.
6. Apply the error to your experimentally determined boiling point and report it on your report sheet.

EXAMPLE Plot of Actual Temperature vs. Correction

Barometric pressure is 767 mm Hg. The boiling point of water at this pressure is 100.2˚C.

Thermometer reading at 0˚C is 1.3˚C.

Thermometer reading at the boiling point of water is 103.6˚C.

AT THE FREEZING POINT OF WATER

Thermometer error is +1.3˚C and the correction is -1.3˚C.

AT THE BOILING POINT OF WATER

Thermometer error is +3.4˚C and the correction is -3.4˚C.

Plot one point on your graph paper at
X axis = 1.3˚C and Y axis = -1.3˚C
Plot the second point on your graph paper at
X axis is 103.6˚C and Y axis = -3.4˚C
Connect these two points and your graph is now done.

Boiling Point Determination
Report Sheet (37 Points)

Name ___________________________ Date _______________

Lab Instructor ________________ Lab Day ____________

I. Thermometer Calibration

Barometric Pressure ________

Correct boiling point of water ________

Thermometer reading in ice water bath ________

Thermometer reading in boiling water bath ________

Thermometer errors: at 0˚C ________

at 100˚C ________

Thermometer corrections: at 0˚ ________

at 100˚C ________

II Unknown Determination

Code number of unknown

Observed boiling point________ Corrected boiling point ________

________ ________

________ ________

Boiling Point Determination
Post-Lab Questions (33 Points)

Name ___________________________ Date _______________

Lab Instructor ________________ Lab Day ____________

Questions 1 through 8 are worth 3 points each. Questions 9 through 17 are worth 1 point each.

1. Why is it necessary to calibrate your thermometer?

2. Why is it necessary to know the atmospheric pressure in order to calibrate your thermometer?

3. Why do you need to make an ice water slush mixture (instead of only ice) in order to calibrate your thermometer at 0˚C?

4. What function does the inverted capillary tube perform?

5. A student calibrated his/her thermometer and obtained a reading of -2.0˚C in an ice water slush bath. What is the error (including the sign) in the thermometer reading?

6. Rubbing alcohol boils at 82.4˚C at a barometric pressure of 760 mm Hg. If the barometric pressure in a laboratory was 720 mm Hg, would the observed boiling point of rubbing alcohol be higher or lower than 82.4˚C?

7. Describe the proper technique for inserting a thermometer into a rubber stopper.

8. What are the four pieces of safety equipment present in each lab? Give a brief description of how and when to use them.

Answer Questions 9 through 17 either TRUE or FALSE

9. Safety glasses should be worn at all times while in the laboratory regardless of whether you are working on your experiment or not. ______

10. To determine the safety of a chemical read the label. ______

11. Bunsen burners should never be left unattended. ______

12. Never inhale chemical vapors. ______

13. One is allowed to work in the chemistry laboratory before your lab instructor is present. ______

14. Dispose of all chemicals as directed by your lab instructor. ______

15. Do not fool around in the laboratory. ______

16. Broken glass should be put in the broken glass boxes in your lab. ______

17. Clean up solid and liquid spills immediately. ______

Boiling Point Determination
Preliminary Questions (10 Points)

Name ___________________________ Date _______________

Lab Instructor ________________ Lab Day __________

1. Define boiling point.

2. How will an increase in pressure affect the boiling point of a substance?

3. What two reference points will you use to calibrate your thermometer?

4. After finishing your experiment, what is the proper procedure for disposing the remaining unknown in your small test tube?

5. Look up in your textbook or another reference source the boiling points of the three different unknowns used in this experiment.

   Methanol _________ Ethanol ___________ Isopropyl alcohol ________

6. Draw the calibration curve described on page 6.