

Student Lab Guide

Physical Properties: Identification of a Pure Liquid



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To identify an unknown liquid by comparing its measured physical properties (such as Boiling Point and Density) with known values.

OBJECTIVE

STUDENT STATIONS

Thermometer	Micropycnometer (1 mL vial)
Disposable pipette	100-150 mL Beaker
Pyrex Test Tube	Small 3-Fingered clamp
Ring stand	Iron Ring
Wire Mesh Screen	Bunsen Burner
Striker	250-400 mL Beaker

APPARATUS AND CHEMICALS

LABORATORY APPARATUS

Ice Water Bath	Hot Water Bath
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CHEMICALS

Labelled Vials of Unknown Liquid

Pure compounds can be obtained after a chemical reaction using a purification process. This purified product can be evaluated by comparing its physical properties with those of the same substance *in pure form*. *Physical properties* are those characteristics of a substance that can be observed or measured without *chemically* changing the substance. Taste, odor, and color are examples of physical properties that are more observable than measurable. Measurable physical properties include **melting point**, **boiling point**, **refractive index**, **density**, **solubility**, and

Student Lab Guide

viscosity. Because **impurities** will alter a compound's physical properties, measurements close to the known values indicate that the substance is of high purity. In addition, careful determination of only a few of these physical properties can discriminate between two possible compounds. Many scientists devoted their lives to collecting and tabulating the physical properties of the elements and compounds. Two books have emerged as references in this area: N. A. Lange's *Handbook of Chemistry* and The Chemical Rubber Company's *Handbook of Chemistry and Physics*. The "handbook" for this lab has been condensed to **Table 1** – eight organic liquids and their densities, melting points and boiling points. With **meticulous laboratory care**, your lab data and Table 1 will allow you to identify your particular unknown.

Physical Properties:

- The **boiling point** of a liquid is the **temperature** at which the vapor pressure of the liquid equals the pressure of the atmosphere above the liquid. At the boiling point, bubbles of vapor form within the liquid and rise unimpeded to the top of the liquid, breaking the surface tension and releasing the vapor into the atmosphere above. If the pressure of the atmosphere above the liquid is exactly 1 atmosphere, then the temperature at the boiling point is called the **normal boiling point**. Because boiling points are pressure dependent, certain corrections to the boiling point measurement must be made if the pressure is significantly different from 1 atmosphere. Fortunately for this lab, as long as the atmospheric pressure is 760 mmHg +/- 10 mm Hg, then the correction will be less than **0.3° C** (well within the error range of reading a thermometer).
- The **melting point** of a substance is the temperature, approached from the solid phase, at which the solid and liquid are in equilibrium. Unlike boiling points, melting points are not affected significantly by pressure changes (pressure becomes important when gases are involved).
- The **density** of a substance relates two common measurements, **mass and volume**, as a ratio that is unique (thus meaningful) to the substance being studied. Whether one has only a speck of a pure substance or a warehouse, the density will be the same.

$$D = \frac{M}{V}$$

Equation 1

The mass of an object is determined with a laboratory balance. Balances have a **TARE** button which allows you to place a sheet of weighing paper (or beaker / watchglass) on the balance and then **tare** the mass to 0.000 g. Once "tared", the balance will report the mass of the sample that is placed on the weighing paper (or in the beaker / watchglass). It is **IMPERATIVE** that you use the **SAME BALANCE** when making measurements during the **SAME LAB!!** We are generally interested in the differences in mass and

Student Lab Guide

this measurement is accurate only if the **SAME INSTRUMENT** is used for both measurements.

Typically, liquid volumes are determined using calibrated glassware such as graduated cylinders, volumetric flasks, burettes, and pipettes. In today's lab, a "**green chemistry**" approach to determining the density and boiling point will be employed. Of the 12 principles of **green chemistry** (<http://www.acs.org/>), the first principle is "it is better to prevent waste than to treat or clean up waste after it is formed." Each student will receive ~ 3.5 mL of an unknown liquid at the beginning of the lab. After the density and boiling point of the liquid is determined, there will only be ~ 1 mL of chemical waste that must be placed in the labeled waste container.

During the Post-Lab assignment, you will calculate the density of your unknown. Compare your calculated density to those listed in Table 1 to identify your unknown.

Substance	Chemical Formula	Density (g/mL)	Melting Point (°C)	Boiling Point (°C)
Acetone	C ₃ H ₆ O	0.79	-95.4	56.2
2,3-Butanedione	C ₄ H ₆ O ₂	0.98	-2.4	88
Cyclohexanol	C ₆ H ₁₂ O	0.96	24.0	161
Chloroform	CHCl ₃	1.48	-63.5	61.7
Bromoform	CHBr ₃	2.90	7.5	150
Hexane	C ₆ H ₁₄	0.66	-94.0	69
Isopropyl alcohol	C ₃ H ₈ O	0.79	-89.5	82.4
Methyl alcohol	CH ₄ O	0.79	-98.0	64.7

Table 1

In addition to the determination of the density of an unknown substance, you will also perform a Thermometer calibration as part of the procedure for today's experiment. It will become necessary to generate a calibration curve to be used for your data analysis. Graphing the data obtained from your thermometer calibration will yield a straight line such that the corrected temperature will be equal to the slope (m) of the line multiplied by the experimental temperature plus the y-intercept (b).

$$y = mx + b \quad \text{Equation 2}$$

$$T_{\text{corrected}} = m \cdot T_{\text{experimental}} + b \quad \text{Equation 3}$$

PROCEDURE

A. Thermometer Calibration

Useful measurements result from equipment that is precise and accurate. Precision generally occurs when a great deal of care has been used by a **student** to reproduce a measurement. However, even careful students will obtain "bad" results when their equipment is inaccurate. **Calibration** is a process

Student Lab Guide

where equipment inaccuracies are exposed and corrections are applied. **During any calibration process, it is critical that students exercise the greatest amount of precision possible in order to obtain an accurate instrument.** After the calibration process, careful students can obtain multiple measured values that are both accurate and precise.

At the front of the class are two large beakers of water – one containing ice and water and the other containing boiling water. Each student will measure the temperature of water in the two beakers to calibrate their thermometer.

1. Place your thermometer in the beaker containing ice and water. Make sure the beaker has plenty of ice (at least $\frac{1}{2}$ ice) and **carefully stir the contents with your thermometer to make sure the entire solution is at the same temperature.**
2. After 1 minute, record the temperature (**make sure the thermometer is still in the ice water when you read the temperature – don't lift it out**) to the nearest **0.2 °C**
3. Next, place the **same** thermometer in the beaker containing the boiling water. **Make certain the water is boiling** and do not allow the thermometer to touch the sides of the beaker.
4. After 1 minute record the temperature to the nearest **0.2 °C** - **make sure the thermometer is still in the boiling water when you read the temperature.**

B. Density of a Liquid Unknown

5. Obtain a numbered vial containing a liquid unknown – **Work Individually and Do Not Share This Unknown Vial With Anyone Else In The Class!**
6. Record the **four digit** number of your unknown liquid.
7. Obtain a micropycnometer (a 1-mL vial) and make sure it is dry before proceeding.
8. Place a weighing paper on the balance and TARE the mass to 0.000 g. Then, place the empty micropycnometer on the paper and record its mass.
9. Use a disposable pipette to transfer the unknown liquid into the micropycnometer.
 - Place your unknown vial in a small beaker so it won't overturn.
 - Transfer the unknown liquid into the micropycnometer until it is completely filled with liquid (contains no air bubbles).
 - Add another drop of unknown liquid to the top of the vial so that the liquid level is higher than the top of the micropycnometer.
 - Screw the cap on the micropycnometer and wipe off the outside to remove any liquid that overflowed.
10. TARE a piece of weighing paper at the balance and place the micropycnometer filled with unknown liquid on this paper and record its mass.
11. Remove the unknown liquid with the pipette used previously. **Place This Liquid In A Clean,**

Student Lab Guide

Dry Test Tube (this will be used later to determine the Boiling Point of the unknown)!!

12. Fill the micropycnometer to the top with distilled water using the distilled water bottle.
 - Make sure the micropycnometer is completely filled with water (contains no air bubbles).
 - Add another drop of distilled water to the top of the vial so that the water level is higher than the top of the micropycnometer.
 - Screw the cap on the micropycnometer and wipe off the outside to remove any water that overflowed.
13. TARE a piece of weighing paper and place the micropycnometer filled with water on paper beaker and record its mass.

C. Boiling Point of a Liquid Unknown

14. Secure the test tube from Step 14 with a three-fingered clamp and lower it into a beaker filled with tap water.
15. Place the thermometer in the test tube and let it rest on the bottom of the test tube.
16. Heat the water with a Bunsen burner until the unknown liquid inside the test tube begins to boil – bubbles are forming around the thermometer.
17. Turn off the Bunsen burner.
18. **Hold** your thermometer approximately 1 cm above the boiling unknown liquid for about 20 seconds before recording the temperature to the nearest 0.2 °C – **the liquid should be dripping off the end of your thermometer when you read the temperature.**
19. If the water begins to boil before you see the unknown boil, turn off the gas and proceed with the previous step if you still have liquid in the test tube - *if no liquid remains, contact your lab instructor.*
20. *Lift the test tube out of the hot water immediately after recording the boiling point to prevent further vaporization of your unknown into the lab's atmosphere.*
21. Return the vial containing any “extra” unknown liquid to the Instructor's Desk.

Waste Disposal: Place any liquid remaining from the boiling point determination in the waste beaker in the hood.

Lab Clean-Up: If additional lab sections are scheduled, please wash all supplies with Chemical Soap and tidy the lab station for the next group of students. If your lab section is the last one of the week,

Student Lab Guide

please return all laboratory supplies to the proper storage location. See your instructor to set attendance and for any additional directions prior to leaving lab.