Objectives

This experiment provides a “hands on” introduction to computer-based scientific measurement. It will also show you how easy-to-use mathematical tools can help us learn a lot about how things work.

After you have completed this experiment, you will be able to:

• Operate the MicroLAB software and calibrate sensors.
• Apply these tools and skills to measure boiling and freezing points and to observe fractional distillation.
• Use graphs and regression lines to analyze and evaluate experimental data.
• Identify an unknown organic compound by measurement of its physical properties.

Background

Science is a search for understanding of the physical, chemical, and biological universe that surrounds us. To build this understanding, we…

• Look for unexplained behavior in nature.
• Design experiments that will yield information concerning this behavior.
• Perform the experiments and collect this information.
• Organize the information to look for generalizations that we call principles or laws of science.

This course is an opportunity for you to participate in this search and, in the process, to learn many of the basic principles of chemistry. These principles govern human and plant metabolism, our generation and use of energy, and the manufacture of materials that house, clothe, and transport us. Measurement is indeed the basic science, since it is by measuring we can build and test models that help us understand the real world.
Measurement and the Role of Instruments and Computers

Many things that we would like to measure are not directly observable. We can tell whether something is hot or cold by feeling it, but we cannot estimate its temperature accurately. If the object is very hot or cold, we could be injured while making the estimate. We can tell whether a substance is acidic or basic by its taste or feel (acids taste sour and bases feel slick), but again we risk injury if the acid or base is strong. We can tell whether light is dim or bright, but we cannot quantify its value. Radioactivity cannot be detected by our ordinary senses, and exposure to radiation becomes evident only in its long-term medical effects. To solve this problem, scientists and engineers have developed instruments. These devices interact with the physical, chemical, or biological quantity of interest and transfer information about the quantity to the observer (Figure 1).

In a way, instruments are like a football team. The team has one objective—to move the ball across the opponent’s goal. To accomplish this task, the team divides up the work among eleven players. Some—guards and tackles—are blockers. They clear the field for a runner or pass receiver and protect the quarterback when he passes. The ends are pass receivers. Members of the backfield kick and run. The quarterback usually passes.

Instruments divide up the work in a similar fashion (Figure 2—next page). Sensors pick up the quantity you wish to measure and convert it to a weak electrical signal. This weak electrical signal is amplified and mathematical functions are performed on it by a data processing circuit. Finally, the resulting number is presented to the observer on a readout device, which might be a meter, a digital display, a computer screen, or a printed page. Just as the football team, all instruments have the same set of players and the same objective—to help you gain information you could not otherwise see.

Instruments are usually designed to perform a specific function. To measure pH, we can use as a sensor, a measurement-specific pH electrode. The electronic data processing circuit is designed to take the weak electrical signal from the pH electrode and produce a number representing pH which is displayed on the readout. The resulting instrument is called a pH meter. To measure temperature, we can use a semiconductor temperature
sensor, (a thermistor, or an integrated circuit temperature probe) and connect it to an electronic data processing circuit that will convert the weak electrical signal from the sensor to a number representing Celsius or Fahrenheit temperature. This number is then displayed on a readout. Digital thermometers used in the home and in hospitals are examples of this type of instrument.

Because each sensor produces a different current or voltage in response to the quantity it measures, a different data-processing formula or electronic circuit is required for each sensor. For this reason, until recently instruments were almost always designed for one purpose: Measurement of pH, or pressure, or radioactivity, or temperature, etc.

![Diagram of instrument system](https://example.com/diagram.png)

**Figure 2.** All instrument systems have the same structural organization. They have a sensor, an electronic data processing circuit, and a readout. They differ only in their measurement-specific sensors and the mathematical tasks performed by their sensor-specific data processing circuits. A common power supply (batteries or a plug-in AC power supply) provides the energy to move electrons in the instrument’s electronic circuits.

Development of inexpensive, high-performance personal computers has changed this situation considerably. A computer can be programmed to perform the data-processing mathematics required by any sensor. Sensors are connected to the computer through a general-purpose interface unit that will take a weak electrical signal from the sensor and produce a digital number to transfer to the computer. The computer also has temporary random access memory and hard disks for long-term storage of programs or data. A system composed of a personal computer and a flexible data acquisition interface (Figure 3) can make almost all of the instrumental measurements required in a biological and physical science laboratory.
Figure 3. A computer-based instrument can be constructed by using a data acquisition interface to digitize the sensor signal and send it to a computer. Mathematical processing of the sensor signal can then be done in software in the computer, using a different data processing equation for each type of sensor. The advantage of this is that one computer/interface combination can serve many different sensors by simply changing software. Once written, the software can be used over and over without cost. The computer can also store and graph data. The result is a very flexible and cost-effective instrument.

The MicroLAB Interface

The MicroLAB Environmental Interface will take the electrical signal from any sensor that produces either a voltage or a current, and will amplify and digitize this signal for presentation to the computer. Flexible and easy-to-use software enables you to tell the MicroLAB which measurements to make, and will help you to organize and analyze the data you collect.

The remaining sections of this experiment will show you how to use the MicroLAB interface system in your laboratory.

You will learn to…
- Select and calibrate sensors.
- Write computer programs that enable you to collect data needed to conduct common chemistry experiments.
- Use a spreadsheet and graph to analyze your data.
- Store your data for future reference.

As you will quickly find out, MicroLAB’s software is straightforward enough that you can use it easily whether or not you have had much experience with computers.
Figure 4. The MicroLAB Environmental Interface will accept signals from almost any environmental sensor, and will translate the sensor signal into information a computer can understand. It will do this with research-grade precision.

Looking Ahead

After completing the material in the next section, you should be able to...

- Identify the functional components of the MicroLAB main software window.
- Use the MicroLAB software to select sensors.
- Understand the importance of accurate sensor calibration, and calibrate common sensors.
- Program the MicroLAB to measure time, temperature, pH, light, and pressure.
- Compute statistical properties of your data, including mean, standard deviation, and distribution.
The MicroLAB software is a group of programs that will help you set up and calibrate your sensors, design and perform experiments, and collect and analyze your data. You can perform graphing and analysis operations to gain an understanding of the principles involved in the experiment.

**Getting Started I: The MicroLAB Software Sensor Calibration and Programming**

This section is an introduction to the use of this software. It begins with choosing sensors and calibrating them. It then demonstrates the use of these sensors with an easy-to-use mouse-driven “drag-and-drop” method of designing experiments. You will also learn to use the MicroLAB spreadsheet and graphing package.

**Starting the MicroLAB Software**

Find the MicroLAB icon on your computer screen and double click on it. This will open the MicroLAB software.

When you start the MicroLAB software, a dialog box will appear (Figure 5). Here you can select the general purpose MicroLAB software, single purpose “instrument” programs, or pre-written programs in tabbed experiment areas.

![Figure 5](image)

Figure 5. When you open the MicroLAB software, the first dialog box will ask you to select which program to run. The MicroLAB icon selects the general purpose MicroLAB software. You can build almost any experiment you wish with a few mouse clicks with the MicroLAB software. The other icons select single purpose “instrument” programs—spectrophotometry, kinetics, and others. The tabs allow you to select MicroLAB programs that have been pre-written for certain experiments.
The MicroLAB Main Screen
The MicroLAB Main Screen consists of six views, as shown in Figure 6. The function of each of these windows is discussed in the following paragraphs.

Figure 6. MicroLAB's main screen has six views that permit the user to select sensors, plot data in a graph, display it in a spreadsheet or as a large digital number, observe (and modify if one chooses) the data acquisition program MicroLAB is running, and finally to start, stop, and repeat an experiment.

Data Sources and Variables View
This view and its control buttons allow one to select sensors or to write formulas or equations that will run “live” in the data acquisition program. The lower part of the window (not visible in Figure 7) lists curve fit formulas that have been generated on the graph.
Figure 7. The Data Sources and Variables view displays the sensors and formulas chosen by the experimenter. Buttons allow one to add, remove, or edit sensors or formulas. When curve fits have been generated, they will appear at the bottom of this view. In this example, Timer 1 has already been selected as a data source. We will learn how to do this in a moment.

Sensor Inputs
MicroLAB will accept sensor inputs into nine connectors on its front panel (Figures 8, 9).

The square MicroLAB “Universal Sensor Ports” (USP) are labeled A, B, and C. They accept MicroLAB sensors with Category-5 (CAT-5) data cable connectors. Other sensor inputs accept pH or dissolved oxygen electrodes, a pressure hose (0–2 atm), a thermocouple, a dual banana plug for voltage measurements, a conductance probe, and a counter.

Figure 8. MicroLAB’s front panel has three “Universal Sensor Ports” that will accept rectangular MicroLAB sensor plugs. Red lights above each sensor input indicate that the port has been selected in the program. Lights on the upper part of the panel indicate which of the three internal timers are operating and whether the spectrophotometer is scanning a blank or a sample. Up to eight inputs can be measured simultaneously. (The pressure input counts for three because of its long-term drift correction circuit.) The green light in the MicroLAB logo indicates that the power is on.
Chapter 1

The Choose Sensor Screen
Soon we will set up MicroLAB to make pressure measurements. Let’s learn how MicroLAB helps you to select and connect sensors.

Click the Add Sensor button (see Figure 7). This allows one to select the sensors MicroLAB will observe during an experiment.

This button brings up a new window showing the front panel of the MicroLAB interface (Figure 9). By clicking the down arrow in the sensor menu, the various sensor options are displayed. You can move the highlight with the down arrow or your mouse. When the desired sensor is highlighted and selected by double-clicking with your mouse, MicroLAB will then highlight the possible sensor input jacks on its front panel in red for your selection. For example, if a pressure sensor is selected, the pressure input will be highlighted in red.

Clicking on the lighted input will select that input port, label the input button window with the name of the port, and the whole port will become highlighted in red. If you wish, you can label the sensor with a descriptive name. Clicking Next will take you to a calibration routine if your selected sensor needs to be calibrated. Clicking the Finish button will then add the sensor information to the Data Variable section. Other sensors may be selected in the same fashion. When an input is selected, a red light lights up beside it on the MicroLAB panel to assist in connecting the sensor.

Click Cancel to close the sensor selection screen. We will return to this screen in a few moments.
Control Buttons
MicroLAB’s control buttons permit one to start, stop, or repeat an experiment.

![MicroLAB's control buttons](image)

*Figure 10. MicroLAB’s control buttons permit one to start, stop, or repeat an experiment. When activated by Level II program steps, Switches A and B permit branching of the data acquisition program.*

Switches A and B permit branching of special purpose programs when they are included in programs written in MicroLAB’s Level II programming language.

The First Experiment: Making a Pressure Sensor
To see how easy it is to write a MicroLAB program, let’s make a simple pressure sensor. MicroLAB’s Level I programming is accomplished by simply dragging a data source from the Data Source Formulas view to a display. Let’s begin by selecting Pressure with the “Choose Sensor” screen (Figure 9). Click on the red-outlined pressure input. It will turn all red. A pressure sensor calibration window will appear. You can select factory calibration in four different unit systems, or you can calibrate the pressure sensor yourself. For this experiment, let’s measure gas pressure in Torr. After you have made this selection, click Finish to close this window.

![Pressure sensor calibration window](image)

*Figure 11. The pressure sensor can report data in four different factory-calibrated measurement systems. Pick “Torr” for this experiment.*
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Figure 12. Use your mouse to drag and drop the “Pressure” data source to the digital display. You can also drag data sources to the graph axes and spreadsheet, and you can drag from one display to another. Try dragging the pressure from the digital display to Column A of the spreadsheet.

Take your gas pressure syringe, open it to about 40 mL, and connect it to the pressure input. Try moving the syringe plunger and watch the pressure change.

Figure 13. Connect your gas pressure syringe to the MicroLAB pressure input.
The Second Experiment: Add a Timer to the Pressure Experiment
Adding a timer to your sensor list will allow you to watch the pressure change over time.

Use the “Choose Sensor” process to add a timer. Select “Timer 1” from the three red blocks on the MicroLAB panel image. Run the timer in seconds (Figure 14).

![Timer Options](image1)

*Figure 14. Set your timer to run automatically, when you click the “Start” button. Select “seconds” as the time unit for this experiment.*

Now drag the timer to the x-axis of the graph and Column A of the data table. Drag the pressure to the y-axis of the graph and to Column B of the data table (Figure 15).

![Graph setup](image2)

*Figure 15. Set up your displays by dragging the pressure and time to the appropriate places in the graph and data table.*
Start your experiment and watch the graph move. Place your hand on the syringe to heat the gas inside slightly. Watch the pressure change.

Now stop the experiment. The graph will autoscale to make the largest display of the change in your data. Often you can’t see much of a change during an experiment, but when the graph autoscales, you can see small changes very well.

The Third Experiment: Add Temperature to the Sensor List
Add a temperature sensor to your experiment. Select a thermistor, and when the request to calibrate the sensor comes up, select Model 103 Thermistor.

Figure 16. Your syringe may have a three-way Luer stopcock. The rotating handle allows you to close any of the three inlets to the valve. In this picture the external connection (lower left) is closed. The inlet covered by the handle is closed. If you want to open the syringe to the air, turn the valve to put the handle over the connection to the hose to the MicroLAB. To open the MicroLAB pressure sensor to the air, turn the handle toward the syringe.

Figure 17. We will use an existing calibration file for the thermistor. It is Model 103 Thermistor.
When you have selected a pre-recorded calibration file, the calibration graph will pop up to be viewed.

![Thermistor Calibration Graph](image1.png)

**Figure 18.** The thermistor calibration graph is a logarithmic curve. Regular curves can provide useful calibrations if a mathematical equation can be fitted to the curve.

Drag the temperature sensor to the right-hand y-axis, start the program, and watch the temperature graph develop. Try touching the tip of the thermistor. You can see the temperature change. Stop the experiment and look at the expanded graph (Figure 19).

![Temperature Graph](image2.png)

**Figure 19.** You can see change in temperature in this graph. Note that the pressure reading looks very erratic. But then look at the actual change in pressure. It is almost zero, but the autoscaled graph showed very tiny changes caused by electrical noise.

Click Repeat Experiment, and we will try a gas pressure/temperature experiment.
A Fourth Experiment: How Fast Does the Thermistor Respond to Changes in Temperature?
Thermistors do not respond to changes in temperature instantaneously. For this experiment, we will start the thermistor in ice water, waiting long enough for its temperature to equilibrate, and then put it in a Styrofoam cup or beaker of hot water. You can leave your pressure reading in the experiment—it will produce extraneous data, but you can ignore it. Or you can right-click on the graph axis with the pressure reading, and remove that from the display.

Try this experiment. Note that it takes about the same amount of time for the thermistor to go half of the way from a temperature $T_1$ to the final temperature, regardless of where you pick $T_1$. The result is that the thermistor responds quickly at first, and then more slowly as it approaches equilibrium with the final solution temperature.

It is important to wait long enough for a sensor to reach equilibrium with its new surroundings before recording the measurement.

A Fifth Experiment: Does Slightly Heating an Enclosed Sample of Gas Cause the Pressure to Change?
To explore the effect of slight heating on the pressure of an enclosed gas sample, we will place the thermistor against the gas syringe (the open 40 mL volume), and then after starting the experiment, heat the gas by holding the syringe and thermistor tightly in the palm of your hand.

Before you do this experiment, drag the thermistor from the right-hand $y$-axis to the $x$-axis in place of time. Now the MicroLab will plot pressure on the $y$-axis and temperature of the gas sample on the $x$-axis.

Start the experiment running, wait a few seconds to get a baseline temperature and pressure, and then hold the syringe and thermistor in your palm. Watch the graph. Can you explain what is happening and propose an explanation for it?

Changing Data Presentation
The graph axes and the digital displays default to displaying two decimal points. You can change this as well as the color of the display or graph point and line by right-clicking on the chosen display and then changing the dialog box that pops up. Figure 20 shows how to change appearance of the digital display, and Figure 21 shows how to change the appearance of the graph display.
Figure 20. This dialog box allows you to change the color and number of decimal places displayed for the selected digital display. Try changing the color of the display and the number of decimal places displayed.

Figure 21. This dialog box allows you to change the properties of the graphic display. Try changing the color of a graph line, the type of point displayed, and the number of decimal places displayed at the axes tick marks.
Statistics

MicroLAB will make statistical calculations on your data. It can compute minimum and maximum values for your data, the standard deviation (the range within which 2/3 of your data falls) and it will present a frequency distribution graph of your data.

Set up a temperature-time program, displaying data on the graph, as digital numbers, and in spreadsheet columns. Start this program running, wait about ten seconds to develop a temperature baseline, and then place your thumb on the temperature sensor until it reaches about 30 degrees Celsius. Then take your thumb off and wait for about 30 seconds for the sensor to cool. Stop the program. The graph will rescale, and you can see how the temperature changed (Figure 22).

Now put your mouse in the temperature column of the spreadsheet and right-click. A dialog box comes up asking if you would like to change column properties (title, the number of digits displayed to the right of the decimal point, or scientific or fixed point notation). You can also clear the column, or call up the statistical information on this data. Call up the statistical screen (Figures 23 and 24).

Figure 22. This data shows heating and cooling of a temperature sensor. This will be the source of our data for demonstration of the statistics screens.
Figure 23. A statistical summary of the data presented in Figure 22. The “bin” size for this display is one standard deviation.

The top of the “Column Statistics” display shows the minimum and maximum values for this data set, and the mean, median, and standard deviation. The data is divided into “bins” for the frequency distribution display. In this experiment, the standard deviation was 2.3805 degrees Celsius. You can set the “bin” width to be from one standard deviation to 1/8 standard deviation. Look first at the one standard deviation bin display (Figure 23). This says that, although the average temperature during the experiment was 27.4471 degrees Celsius, the sensor spent most of its time within one standard deviation of 29 degrees Celsius.

If you switch to smaller bins, such as displayed in Figure 24, you can get a finer presentation of where the data is clustered.
Figure 24. This is the same data, with a bin size of 1/8 standard deviation. You can see better where the sensor spent its time during the experiment.

Using the Statistics Function to Evaluate Sensors
The standard deviation is also a good way to check your sensor for noise. Try putting your temperature sensor in an Erlenmeyer flask of tap water, let it stabilize, and then collect about 100 data points. Put this data in a spreadsheet column, and then run the statistics program. How much variation can you see? What percentage is the standard deviation of the average (mean) signal? This is the error introduced by electrical noise in the sensor signal. Try the same experiment with your pH electrode in a pH 4 buffer solution, or with your pressure sensor, open to the air.

The Experiment Steps View
MicroLAB Level I programs build a data acquisition program in the Experiment Steps view as you drag sensors to display views. This program is written in modern object-oriented computer programming symbolism. The program illustrated in Figure 25 (next page) is a “repeat until” loop. It tells the MicroLAB to repeat everything between this initial command and the “until” statement. In this case, the loop will repeat until the stop button is pressed, either on the control panel or on the Main Screen.

By left-double-clicking your mouse on the “Repeat every 0.5 seconds” line, you can call up a dialog box that will allow you to change the time between measurements. Try this, changing to two seconds between measurements. Run this program to see how it works.
Click a mouse right button in the experiment steps box. You can display programming buttons that allow you to add and modify MicroLAB’s program steps. We will learn to run these buttons later in the course.

![Experiment Steps Diagram]

**Figure 25.** MicroLAB’s “Experiment Steps” window shows you the computer program that is being run to collect your data. The Level I program is written by the computer as you drag and drop data variables into display windows.

![Flowchart Diagram]

**Figure 26.** The basic MicroLAB program runs a simple loop in which it reads all of the sensors, performs some signal averaging, and sends the data to the computer. It then waits a selected time interval (0.5 seconds is default) and repeats this process until the stop button is pressed.

You can check the “Program Steps” window to make sure the MicroLAB is making its measurements in the way you intend. Instruments can be “black boxes” to their users. MicroLAB’s goal is to have the user in control.
Calibrating a pH Electrode

Our choice of sensors so far has been determined by the need to calibrate them.

- The pressure sensor was factory calibrated, and required only choice of the display units.
- The temperature sensor had a factory-provided calibration chart, but you had to select the calibration file. (You also could have hand-calibrated it.)
- pH sensors vary from one unit to another, and always have to be field calibrated.

This pH calibration is quite quick and easy. You will need the following materials:

- The MicroLab interface
- A pH electrode
- pH buffer solutions: pH 4, 7, and 10

Calibration Procedure

Use the Select Sensor screen to select the pH electrode. Click on the red-circled BNC input to let the software know that this is where the pH electrode will be connected, and select pH from the pH/Dissolved Oxygen choice.

Now choose “Perform New Calibration.”

Figure 27. To calibrate the pH electrode, choose “Perform New Calibration.”
The following calibration screen (Figure 28) will then come up. This screen does several tasks for us:

- It allows you to add a calibration point.
- After you have some calibration points, you can select a curve fit.

Figure 28. Use this screen to add a calibration point. The program will move to the sensor equilibrium screen (Figure 29) and then will revert back to this screen after each calibration point is entered. You then use this screen to select the curve fit you wish to use.
Figure 29. This screen allows the operator to watch the sensor come to equilibrium with its standard solution. The rate-of-change meter will center and the sensor history display will flatten out at equilibrium. If you check the “Show Advanced Meter Controls” box, you can change the rate of change meter sensitivity. Some sensors need more or less sensitivity on this meter than others.

The sensor equilibrium screen does several tasks for us:
1. It displays the current output (a voltage or a current) from the sensor.
2. It provides the operator a chance to enter the true value of the standard.
3. It demonstrates when the sensor comes to equilibrium with the standard solution. The display with the green bar shows the rate of change. As the sensor comes to equilibrium, the rate of change will fall to zero and the red line will center on the green. The second display presents a “history” of the sensor output. You can see the sensor stabilize on this display.

It takes two calibration points to make a straight line and to define a calibration equation. However, there is no room for error. A wise choice is to use pH standards at opposite ends of the range—pH 4 (acid) and pH 10 (base), and then draw the calibration line. Then use an intermediate pH standard to “prove” that your line is correct.

For each of your pH buffer standards:
- Rinse the pH electrode well in distilled water.
- Place it in a small (10 mL) amount of new buffer solution.
- Enter the buffer pH in the calibration screen.
- Watch your equilibrium indicators, and stir carefully with the pH electrode until the sensor reaches equilibrium.
- Press “OK” when the sensor is at equilibrium. Your calibration point will be recorded on the graph, and you can start another calibration point or do the curve fit and accept the calibration.
When you have enough calibration points, choose a curve fit (see Figure 28). If the line is straight you have a good calibration. You may wish to use different curve fits if linear does not work. If one of the points looks “bad,” you can remove it from the calculation by right-clicking on it. The point turns into an “X” and is not counted, but it is not lost, either. You can return it to the data set with another right click.

![Calibration graph](image)

Figure 30. Here is a good pH calibration graph. The correlation coefficient is 0.99989—very close to the perfect straight line (1.000).

When you click “Finish,” you will be given an opportunity to name the calibration file, which will then be saved.
Looking Ahead

In the next section, we will use the measurement skills we just learned to determine the boiling point of pentane, the freezing point of glacial acetic acid, and to find out if evaporation causes cooling. This will be followed by using the software to organize and analyze data, and learning how to modify experiments to use different sensors.

After completing the material in the following section, you should be able to…
• Measure the boiling point of a liquid.
• Measure the freezing point of a liquid.
• Measure the degree of cooling of an evaporating liquid.
• Organize and analyze your data.
Getting Started II: Setting Up Experiments and Collecting Data

There are several ways you can use MicroLAB to collect data for your experiments. In the previous section, we learned to calibrate sensors and to use the mouse “drag-and-drop” method to set up your own experiments. Sometimes, however, it is useful and quick to use an experiment program that is already set up for you. This section will show you how to do this. It will also show how you can create “template” experiments that you can save and call up later if you want to repeat that particular experiment.

This section will introduce three short experiments in which temperature varies with time. One of these involves measurement of the boiling point of a low-boiling hydrocarbon, pentane. This compound boils at about body temperature. The second involves measurement of the freezing point of glacial acetic acid, which freezes only a little below room temperature. The third experiment involves evaporation of shaving lotion. By watching the temperature change as a small sample of shaving lotion evaporates, you can get a good idea of how many different components are in the shaving lotion.

We will ask you to do the first small experiment two different ways. First, by using the drag-and-drop experiment design method, and then by selecting and using a data collection program that has been pre-written for you. For the next two experiments, you can use whichever method you prefer.

Identifying the Problem
Defining the problem is the first step of an experiment. The first problem we will choose is a simple one. Pentane (C\textsubscript{5}H\textsubscript{12}) is a light hydrocarbon molecule. Because it is light and is “nonpolar,” it is not attracted to other molecules and will boil almost at body temperature. Thus, our problem for this experiment: What is the boiling point of pentane?

```
        H   H   H   H   H
        |   |   |   |   |
H - C - C - C - C - C - H
        |   |   |   |   |
        H   H   H   H   H
```

*Figure 31. Pentane is a 5-carbon hydrocarbon molecule (it is made only of hydrogen and carbon). It boils more easily than water.*

Designing the Experiment
Experiment design is one of the most important and most difficult aspects of science. Here you define what you will measure and how well your variables are controlled.

Design of an experiment is a two-fold task. First we have to ask ourselves what information is required to draw a reasonable conclusion concerning the experimental problem. Then we have to decide how best to acquire and display or record this information.
Problem: Determine the boiling point of pentane.

What data must we collect to solve this problem?
We need to watch the temperature change with time.
• Temperature
• Time

How do we want to present this data?
• A graph of temperature vs time will show when the temperature stabilizes as the substance boils.

Digital displays of temperature and time will provide a useful visual monitor of the experiment.

Will we use sensors that need to be calibrated?
• The temperature sensor will have to be calibrated.

Question One: What information will it take to draw a conclusion about this problem?
We will need to collect temperature data as the liquid is heated and finally boils. Because it takes energy for the molecules to move from the liquid to gaseous state, the temperature will stabilize as boiling takes place. How many measurements will we need? We will have to have a series of temperature readings, probably at least one per second, over the period of time required for the liquid to be heated to boiling.

Note the uncertainty indicated by the word “probably.” You can hardly ever guess the conditions of an experiment the first time you run it. For this reason, experienced scientists always run an experiment quickly at first, knowing that they will immediately learn some things that will cause them to change the experimental conditions before they can collect useful data. Controlling important experimental conditions is as important as choosing which measurements to make and how often to make them. As a general rule, realize that you will always run an experiment at least twice.

Question Two: If we collect data with a computer and laboratory interface, how will we want to present this data? Will you wish to present data as a graph, on a digital display, or as a list of numbers in a spreadsheet? Or all three?

Building an Experiment
After you have decided what sensors you need, whether or not you need to calibrate them, and how to display your data, set up your MicroLAB screen to collect data. A sample screen setup is illustrated in Figure 32.

Testing the Program
Test the program using your thumb as a heat source. Make sure the sensor and data collection program are working properly before you start the experiment.
Figure 32. A sample data display for the boiling point experiment consists of a time/temperature graph and digital displays of instantaneous time and temperature. This general display setup will handle almost any experiment where you want to monitor temperature as an experiment proceeds.

Running Experiments

Problem 1: Boiling Point of Pentane

In a small test tube, place a small boiling chip and about 1 mL of pentane. The MicroLAB sensor is sensitive enough that you do not need much sample, and 1 mL of pentane boils at the same temperature as 100 mL of pentane.

Figure 33. Your temperature sensor should not touch the bottom of the test tube when you are making boiling point or freezing point measurements. It needs to measure the temperature of the liquid, not the glass.
Obtain a Styrofoam cup, and place about 50 mL of relatively hot water (45–50 degrees Celsius) in the cup. Pentane boils about body temperature, so the water doesn’t need to be extremely hot. Drop one or two boiling chips in the test tube. This will promote smooth boiling and the pentane is less likely to boil out of the test tube. Place your temperature sensor in the pentane, but do not let it touch the bottom of the test tube (Figure 33). If it touches the bottom, you will measure the temperature of the test tube instead of the temperature of the pentane. Start the program running, and place the test tube in the water. Observe the graph, and determine the point where the pentane is boiling and the temperature stops rising. Note that the temperature will remain constant until all the pentane evaporates. What is the boiling point of pentane?

**Saving and Exporting the Data**

Once you left-click on Stop, the data collection is complete. Now you will need to decide if you want to save the data that has been collected. The quickest way of saving your data is by pressing the “floppy disc” icon at the top of the screen on the windows tool bar. This will save your data as a “.exp” file with the title that you initially selected at the onset of the experiment. You will have access to your USB drive via “Save As” option under the “File” heading on the tool bar at the top of the MicroLAB Main Window. Before you repeat an experiment, you should save your data by this method.

*Figure 34. You can choose a pre-written experiment from the tabbed experiment area choices. Choose “Time and temperature,” and then select “Freezing Point.” This will set up an experiment to monitor time and temperature while the acetic acid is cooled and freezes.*
Running Experiments

Problem 2: The Freezing Point of Acetic Acid

The next experiment involves measurement of the freezing point of concentrated (glacial) acetic acid. The measurements you will make are the same—time and temperature. However, this time let’s try using a pre-written experiment.

Start the MicroLAB software, and from the “Choose an Experiment” screen, select the tab “time and temperature” (Figure 34). When this experiment has loaded, check the data displays for the graph and the digital display. It should be the same as what you built in the last experiment, except this was stored, ready to use.

Note that you have to have a calibration file for your temperature sensor. You can highlight the temperature sensor in the sensor block, click on edit (button below) and check to see what calibration file is being used. You can recalibrate the sensor at this point if you need to.

Discard the hot water in your Styrofoam cup and fill it with crushed ice. Add some cold water so that you have about a 50–50 ice-water slush mixture. What should be the temperature of this liquid? Now obtain about 1 mL of concentrated (glacial) acetic acid in a clean, dry test tube. This should occupy about 1 cm in the test tube. Acetic acid is the active ingredient in vinegar, but vinegar is only 5 percent acetic acid. Be careful not to spill any of the concentrated acid on your clothes or fingers. Don’t directly “sniff” the acetic acid, it could burn your sinuses!

Place your temperature sensor in the acetic acid, again holding the thermistor so that it does not touch the bottom of the test tube as in Figure 35. Start your program, and then place the test tube into the ice-water mixture. Hold it still. Liquids cool until they reach their freezing point. The temperature then remains constant while the liquid freezes. Some liquids “supercool”; that is, their temperature will fall below the freezing point.
point and then pop back up to the freezing point when they freeze. At this point, freezing is extremely rapid. Can you see any evidence of supercooling with your acetic acid sample? If it will not freeze after getting close to the temperature of the ice-water bath, try tapping the temperature sensor on the bottom of the test tube.

After it has frozen, melt the sample by placing your test tube in warm water, and then try freezing it again, this time stirring with the thermistor. Does this work any differently? What is the freezing point of acetic acid?

Your laboratory instructor will tell you where to discard your acetic acid sample.

Running Experiments

**Problem 3: Evaporation of a Liquid**

When you climb out of a swimming pool on a warm day, you immediately feel cold until the water evaporates and you are dry. When a nurse swabs your arm with an alcohol pad to disinfect it before a shot, your arm feels cold until the alcohol evaporates. Does liquid evaporating from your skin really make you cold, or is it just in your head?

We could check this by placing a drop of water (or alcohol) on the tip of a temperature sensor and observing the temperature change, if any, as the liquid evaporates.

**Question:** Does temperature really drop as a liquid evaporates?

To explore this question, you will need to take the same kind of data as in the last two experiments. You need to watch how the temperature changes with time as the experiment proceeds. This time you can either build your own experiment or use a pre-written experiment. It is your choice.

Dry your temperature sensor tip with a paper towel. Obtain a small sample of tap water that is near room temperature. Wrap a small piece of filter paper around the tip of the temperature sensor and secure it with a small rubber band. This will give more area for the liquid to evaporate from. Start your program, dip the temperature sensor into the water and remove it, shake it briefly once, and hold it vertically. Watch the graph. Does the temperature really drop when water evaporates, or is it just something you think you feel?

Dry the temperature sensor and repeat this experiment, this time with a new filter paper and a sample of ethyl alcohol. Does this behave differently than water?

Now try a sample of a shaving lotion and compare it to your previous samples. What can you draw from this information?
Drawing Information from Graphs

Figure 36. This data shows the change in temperature that occurred as a drop of shaving lotion evaporated off of the tip of a temperature sensor. It takes heat to change the shaving lotion from a liquid to a gas, and this heat comes from the temperature sensor. If you put shaving lotion on your face, you provide the heat and your face feels cold. We will take a closer look at this data in the next section. The “glitch” was caused by moving the thermistor. Motion of the air briefly increased the rate of evaporation.

Graphs such as the evaporation of a liquid, illustrated in Figure 36, can tell us a lot about the behavior of the system we are investigating. In this case, it is apparent that something is taking a lot of energy away from the temperature probe as the alcohol evaporates. If the alcohol is on your arm, you provide the energy and feel cold when that energy leaves. The energy required to change a material from a liquid to a vapor is called the heat of vaporization.

For alcohol, this value is about 1000 Joules per gram. For water, about 2260 Joules is required to evaporate one gram. This energy is removed from the material that is in contact with the liquid. This is why hypothermia is such a danger if you become wet when out hiking. The water evaporating from your clothes removes heat from your body. Evaporation of less than a quart of water from your clothes can dangerously lower your body temperature.
Looking Ahead
This section has presented a hands-on overview of the MicroLAB’s Level I programming, and introduced pre-written experiments as an alternative to get an experiment started rapidly. However, whether you set up your own data display or recall one from the list of pre-written experiment programs, you still need to make sure you understand what the data acquisition program will do for you.

The next section will deal with ways to draw mathematical relationships from experimental data and we will use these tools to see how many different compounds can be detected in shaving lotion.

After completing the material in the next section, you should be able to…

- Use the **Spreadsheet** to graph and organize experimental data.
- Define the term **Slope** and use a **linear regression** to calculate the slope of the best straight line through your data set.
- Use the correlation coefficient to estimate the quality of your data.
- Use the slope-intercept equation produced by the linear regression to predict values within and outside the range of your measured data points.
Getting Started III: Drawing Information from Graphs

Science is a search for understanding of the physical, chemical, and biological universe that surrounds us. To build this understanding, we...

- Look for unexplained behavior in nature.
- Design experiments that will yield information concerning this behavior.
- Perform the experiments and collect this information.
- Organize the information to look for generalizations that we call principles or laws of science.

Using Graphs

In this section, we will learn to use graphs to draw information from experimental data. Part of this chapter involves fitting mathematical relationships to experimental data that is plotted on a graph. Graphs of experimental data can provide a historical record of the behavior of the variables in an experiment. Graphs can also be used to show relationships between two variables, one usually controllable and the other not.

A mathematical tool called the curve fit enables us to produce the “best” smooth line through a set of data points. The curve fit also tells us how closely the data approximates the line and provides an equation relating the y and x variables.

In the next section, we will use the graphical analysis techniques developed in this section to identify an unknown organic compound.

Slope and the Linear (First Order) Curve Fit

Let’s start out by examining the data from the shaving lotion evaporation experiment we performed at the end of the previous section. Shaving lotion is comprised of several different components—water, an alcohol, and other compounds. Each of these boils at a different temperature. Can we use this graph to find out how many components are easily identifiable?

When the shaving lotion evaporates, most of the lowest boiling compound evaporates first, cooling the sensor. As this compound is depleted, the next lowest boiling compound begins to evaporate, and so on. One can usually see the change from one compound to another as a change in the slope of the temperature–time graph.

A sample graph for shaving lotion is presented in Figure 37. Using this figure as a model, click on the “Select Domain” button on your graph, and use your mouse (left button down) to draw a box that encloses the first relatively straight section of your graph.
Figure 37. Use the “Select Domain” button and your mouse to select the range of the graph you plan to curve fit.

Now click “Analysis.” A smaller box will pop up asking you if you want to do a curve fit, or plot a derivative or an integral. Pick “Curve Fit” (Figure 38).

Figure 38. The “Analysis” button pulls up a dialog box you can use to choose which type of graphical analysis you wish to perform.

Following this, another box will pop up asking you to choose a graph series for analysis. If you have only one line on your graph, you will only have one choice (Figure 39).
Figure 39. The “Choose Graph” dialog box tells you the bounds of the data domain you have chosen, and which variables are involved. If you have two lines on the graph, you will have to select which y-axis variable will be used.

When you click “OK” on the “Choose Graph Series” box, the “Curve Fit” screen will appear (Figure 40). On the left side of this screen are a series of mathematical relationships one can use to fit the data. These go from the simplest (linear) to exponential and logarithmic relationships.

Figure 40. The “Add Curve Fit” screen permits you to fit a set of data to one of several different mathematical relationships. The simplest of these is linear. The resulting equation and correlation coefficient are presented at the top of this screen. If you press the “Accept and Save” button, the curve fit equation is stored at the bottom of the sensors/formulas window.

Click on linear or first order, and observe your curve fit. This relationship gives a straight line. The equation for the line and its correlation coefficient are presented at the top of the graph. If the correlation coefficient is 1.000, the mathematical relationship you have chosen is a perfect fit to your experimental data. Smaller correlation coefficients indicate a poorer fit. Try relationships other than linear, and look at the correlation coefficient. Is linear the best fit for the graph segment you chose?
If you have what appears to be a bad data point, place the mouse on it, right-click, and you can delete this data point from your regression calculation, although it will stay on your graph. After performing this function, you will observe that the excluded data point will change from a green point to a red point. Notice in the graph that if some of the points are removed, the linear curve fit will calculate a slightly different equation and correlation coefficient. To bring the data point back into the calculated curve fit, simply right-click on it. Even if all of your data looks good, try removing a point to learn this technique.

Return to linear or first order fit, and look at the curve fit equation. It is in the form $y = mx + b$, where $y$ is the temperature, $m$ the slope of the line, $x$ the time, and $b$ the y-axis intercept when $x = 0$. Label your curve fit with a specific name that signifies the area of interest on the graph to which you have zoomed (“First Segment,” for example). This equation will be saved in the Data Sources/Formula View with your specific label when you press the “Accept and Save this Curve Fit” button. In a few moments we will compare this equation with equations for other, later segments of the temperature–time graph produced by the evaporating shaving lotion.

The slope of a graph (m) often yields important information in an experiment. In this case, it shows how rapidly the sensor is cooling and thus how rapidly that component of the shaving lotion is evaporating.

Slope is defined as the amount of change in the y-axis value for a unit change in the x-axis value, ($\Delta y/\Delta x$, where $\Delta$ refers to a “change” in a variable). The units of slope in this experiment will be degrees Celsius change per second. The faster a compound evaporates, the more rapidly the sensor temperature will change.

Repeat the domain choice and linear regression for each of the straight line segments you can locate in your data. Store these equations in your Data Sources/Formulas View and record the slope of each segment of your collected data in Data Table 1.

<table>
<thead>
<tr>
<th>Component</th>
<th>Slope (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td></td>
</tr>
</tbody>
</table>

Now compare the slope values for each of these graph segments. Do these slope values tell you anything about the volatility of each component?
Using Graphs to Determine Density—Hand-Entered Data

Density is defined as mass of a sample for a unit volume. In the System Internationale (SI) system of units, density is usually expressed as grams per milliliter.

To experimentally determine the density of a sample, we have to determine the mass of a known volume of the liquid and then divide mass by volume:

\[
\text{Density} = \frac{\text{mass}}{\text{volume}} = \frac{\text{g}}{\text{mL}}
\]

However, if we measure only one mass and one volume, there is a reasonable chance of experimental error. How could multiple measurements be combined to produce a more accurate value?

Suppose that we began with an unknown liquid and graduated cylinder, and measured the mass and volume of five different volumes of liquid. Data from such an experiment is reproduced in the accompanying Table 2. The mass reported is the total mass of the graduated cylinder and liquid. Let’s hand-enter this data into a spreadsheet and graph it for analysis.

Table 2. Experimental Data for Density Determination.

<table>
<thead>
<tr>
<th>Volume (mL)</th>
<th>0</th>
<th>7.1</th>
<th>12.0</th>
<th>16.5</th>
<th>24.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass (g)</td>
<td>46.1</td>
<td>55.0</td>
<td>61.1</td>
<td>66.7</td>
<td>76.1</td>
</tr>
</tbody>
</table>

Setup for Hand-Entered Data

Close your MicroLAB program using the “X” in the upper right corner of the screen. Now click the MicroLAB icon, and select “Hand-Entered Data,” and “OK” (Figure 41). Note that the sensor selection and start/stop buttons grey out, and that the spreadsheet and graph will become active for hand-entered data only.

Figure 41. Select MicroLAB, Hand Enter, and OK to start hand entry and analysis of data.
Press the “Add Column Labels” button in the lower right window. Use your mouse to select Column A, and label it “Volume” (mL). Do the same for Column B, labeling it “Mass” (g).

Now use your mouse and keyboard to enter the data from Table 2, pressing the down arrow each time to enter the data into the spreadsheet and to move down to the next cell. Set up your graph by dragging the “Mass (grams)” column label to the y1-axis of the graph, and the “Volume (mL)” column label to the x-axis of the graph.

When all five data sets have been entered into the spreadsheet, press the “Accept Data” button in the lower right-hand window. The data in the spreadsheet is now sent to the graph and plotted. The graph will auto-scale to use all of the window space to display this data (Figure 42).

Adding a First Order Curve Fit Line
Use the “Set Domain” button and your mouse to draw a box selecting all of your data. (You can just click “Analysis” and MicroLAB will use all of your data.) Now click the “Curve Fit” button, and run a linear (first order) curve fit line through your data. Is the correlation good? Can you improve it by removing a data point? You only have five data points, and if you remove more than two, you will be down to a three point graph and you can’t identify bad data points after that. Hopefully most of your data will be good.

Now let’s consider the significance of the slope-intercept equation of this mass/volume data. This equation is presented in a slope-intercept form:

$$ y = mx + b $$

below the graph and in the box above the graph. For this set of data, the y units are mass and the x units are volume. The slope (m) is defined as the change in y for a given change in x, or

$$ m = \Delta y / \Delta x = \text{grams/mL} $$
The linear regression function may be used to determine the density of a substance. When mass \( y \) is plotted against volume \( x \), the slope of the line is mass/volume or density. To save this curve fit you must give it a name (circled in the figure).

The units of the slope (m) are grams per milliliter. This is a density unit. The slope of the graph in fact is the best averaged value for the density of the liquid that takes into account all of the experimental data.

The variable \( b \) is the y-intercept or value of y when \( x = 0 \) (units of mass). In this case, it is the mass of the empty graduated cylinder.

What is the density of the liquid used in this demonstration experiment?

**Significant Figures on Spreadsheets and Graphs**

The density is reported in the linear regression equation as 1.2485 g/mL. Now, is this realistic? Note that none of the data that went into the calculation had more than three significant figures.

This number has five reported figures. *It must be rounded to be no better than the worst data that went into making it.* In this case, the density should be reported as 1.25 g/mL and the intercept as 46.1 g. Therefore the linear curve fit equation corresponds to:

\[
\text{Mass (g)} = (1.25 \text{ g/mL})(\text{volume mL}) + 46.1 \text{ g}
\]

You can set the number of decimal places displayed in MicroLAB’s spreadsheet by right-clicking on a column of data and then selecting the number of decimal places from the resulting dialog box. Try changing the number of decimal places displayed in one of your spreadsheet columns.

Use the box in the upper left corner of the curve fit window to label your curve fit formula “Density.” This name will appear at the top of the graph. Now press the “Accept and Save” button. The curve fit formula will be stored at the bottom of the “Data Sources/Formulas” screen. Use the scroll bar to find it (Figure 44). We will use this in a moment.
Predicting y-Values from the Slope-Intercept Equation

One can use the slope-intercept equation and a pocket calculator to compute the mass (y variable) associated with any volume (x variable) by placing the volume into the equation and solving for mass. Thus, for example, if the volume were 35.0 mL, the mass of the cylinder and liquid should be

\[
\text{Mass (g)} = (1.25 \text{ g/mL})(35.0 \text{ mL}) + 46.1 \text{ g}
\]

\[
\text{Mass (g)} = 89.9 \text{ g}
\]

However, MicroLAB’s graphical analysis package will do this for you as well.

Double right-click on the “Density Plot” line in the Sensors/Formulas block. A “Predict” box will come up. Enter an “x” (volume) value into the box and press “Predict.”

The corresponding y value will appear. Note that you have to make the call on the number of significant figures for the result, based on the precision of your experimental data.
If you push the “Plot Point” button, the predicted point will be displayed on the graph.

Note that you can also go to the “Predict” function by pressing the “Analysis” button on the bottom of the graph. Try this.

The Print Function
You can print the graph in three different ways:
1. Click the print button in the graph window. This is the easiest way to print.
2. Click on the graph in order to make it the “active” window. Then click the printer icon on the top tool bar. You can print other views in the MicroLAB screen in this manner.
3. Click on the graph in order to make it the active window. Then click on the File drop down menu on the top menu bar, and select Print.

An Experiment for You—Determination of Density
Given the data in Table 3, construct a graph and use the linear curve fit to determine the density of the liquid and the mass of the empty graduated cylinder.

<table>
<thead>
<tr>
<th>Volume (mL)</th>
<th>13.0</th>
<th>17.5</th>
<th>24.0</th>
<th>32.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass (g)</td>
<td>50.8</td>
<td>54.9</td>
<td>60.7</td>
<td>68.4</td>
</tr>
</tbody>
</table>

Density = \frac{\text{Mass}}{\text{Volume}} \text{ g/mL}

Predicting Sea Level Boiling Points from Experimental Data
Measurement of the boiling point of a liquid is not as straightforward as it might seem. Boiling occurs when the energy of motion of the molecules in the liquid slightly exceeds the atmospheric pressure pressing down on the surface of the liquid. At this point, liquid molecules can move up into the air above the surface. If the atmospheric pressure is lower, boiling will occur at a lower temperature.

Atmospheric pressure, of course, varies with both altitude and weather. In Bozeman, Montana, and Denver, Colorado, (5000 feet elevation) the normal atmospheric pressure is about 640 torr. At sea level the normal atmospheric pressure is about 760 torr. The pressure is about 84 percent of that at sea level. As a result, liquids boil at a lower temperature at high elevations. Boiling point data, however, is recorded in handbooks at sea level pressures. At sea level the boiling point of water is 100°C, but in Denver it boils at about 94°C.

Boiling point is a useful physical property of materials that is often part of the information we use to identify a compound. To make this identification, however, requires that we know the sea level boiling point of the substance. We measure the boiling point in our laboratory at the altitude and weather conditions that exist at that time. How can an observed boiling point be converted to a sea level value?
An easy way to handle this involves simply obtaining local boiling points for several known compounds and then preparing a graph of observed boiling point and the handbook value for the sea level boiling point of the material. Data from such an experiment is presented in Table 4.

**Table 4. Sample Boiling Point Data for Several Compounds.**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Acetone</th>
<th>Chloroform</th>
<th>Ethanol</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sea Level B.P. (°C)</td>
<td>56.5</td>
<td>61.2</td>
<td>78.5</td>
<td>100</td>
</tr>
<tr>
<td>Observed B.P. (°C)</td>
<td>50.0</td>
<td>58.5</td>
<td>74.0</td>
<td>94.5</td>
</tr>
</tbody>
</table>

When this data is plotted and a first order curve fit is calculated, the equation for the line is:

\[
\text{Sea level B.P.} \, ^\circ\text{C} = (1.01) (\text{observed B.P.}) \, ^\circ\text{C} + 4.43 \, ^\circ\text{C}
\]

Try entering this data and graphing it to see if you get the same result.

Consider now a compound whose observed boiling point is 82°C. To find the sea level boiling point simply requires that we substitute the observed boiling point into the slope-intercept equation for the line:

\[
\text{Sea level B.P.} \, ^\circ\text{C} = (1.01)(82) \, ^\circ\text{C} + 4.43^\circ\text{C}
\]

\[
\text{Sea level B.P.} = 87.3^\circ\text{C}
\]

Use your predict function to enter an “x” value of 82.0 degrees Celsius. Do you come up with this result?

**Looking Ahead**
The next experiment involves identification of an unknown organic compound by measurement of its physical properties—among them density and boiling point. We will use the techniques developed in this experiment to help identify this compound.

After completing the material in the next section, you should be able to…

- Accurately determine mass by use of a chemical balance.
- Accurately determine volume by use of volumetric glassware.
- Calculate density, given experimental values for mass and volume.
- Use graphs to analyze experimental data and to identify “good” and “bad” data points.
- Use a temperature sensor to measure boiling and freezing points.
- Determine solubility properties of an unknown substance.
- Use boiling points, freezing points, and density indices.
- Identify unknown compounds by determination of their physical properties.
Report

Density → Mass/volume graph, Density
Freezing Point → Freezing point graph, Unknown freezing point
Boiling Point → Boiling point correction graph, Unknown boiling point
Solubility → Observations
Unknown Identity → Narrative: Identification of unknown compound

Physical Properties of Matter: Identification of an Unknown Organic Compound

Physical properties of matter are those properties that we can measure without changing the chemical identity of the substance. Color, freezing point, boiling point, density, and solubility are examples of physical properties.

While a given physical property—color, for example, or density, or boiling point—may be the same or very similar for a large number of compounds, the combination of a number of physical properties quite rapidly makes the sample unique.

Consider the students in a 600-student class. If we picked the physical property age 22, or weight 150 pounds, or brown hair, or blue eyes, or shoe size 11, or gender = male, each property by itself would describe a relatively large number of people. However, the combination of these physical properties would probably uniquely identify only one individual in the class.

The tables at the end of the experiment list the physical properties of a number of relatively common organic (carbon-based) compounds. While these tables all contain the same information, they are arranged differently—alphabetically by name (Table 6), by density (Table 7), and by boiling point (Table 8). Your instructor will provide a sample of one of these compounds for you. By using the measurement and data analysis techniques developed in this manual, decide the identity of your compound from those compounds listed in the tables.
Safety Precautions
As you make your measurements, recall that 1/2 mL of a compound has the same boiling and freezing points as 100 mL. Use small samples; they are just as effective, safer, and less expensive to purchase and to dispose.

In addition to the normal laboratory precautions, be aware that many organic compounds are flammable and most organic vapors should be considered toxic, at least to some degree. Be especially aware that others in the lab are also working with organic compounds. Do not use an open flame in the lab.

Materials
Reagents: Several of the compounds listed in the table at the end of this experiment are available to you to test your procedures.

Equipment: Balances, graduated cylinders, test tubes, temperature sensors, beakers, boiling chips.

Unknowns: For an unknown, you will be given one of the compounds in the table found at the end of this experiment.

Experimental Procedure
Using the techniques developed earlier in this section, determine...
1. The density of your sample.
2. The freezing point of your sample.
3. The boiling point of your sample, corrected to its sea level value.
4. The solubility of your sample in water, isopropyl alcohol, and hexane.

Hints
Density and Experimental Error
You can improve your accuracy in reading volumetric glassware by taking a piece of white paper and marking a black rectangle on it with a pencil. Hold this behind the graduated cylinder and move it up until the rectangle is about 1 mm below the meniscus. The meniscus now looks black, and the lowest point can be easily read against the white background. You can read tenths of a milliliter and estimate to hundredths.

Figure 46. Using a black reflector to highlight the meniscus.
Determine the volume and mass of a liquid by determining at least four different samples, each time adding a little more unknown and repeating the determination of volume and mass. By the fourth addition of unknown, the cylinder should be practically full.

Construct a graph relating mass to volume, and determine the density of the substance.

**Balances**

A variety of different balances are used in introductory chemistry laboratories, ranging from simple but accurate triple-beam balances to top-loading automatic balances with digital readout. Your laboratory instructor will review with you the proper operating procedures for your balance.

There are some common sense rules that apply to weighing with all types of balances:

- If your laboratory has a separate weighing room, prepare the sample you intend to weigh in the laboratory, not in the balance area. Chemicals must never be taken into the balance area except in a container to be weighed.
- Do not pour chemicals into a container on the balance; spills may damage the balance. **ALWAYS** pour the chemical into the container outside of the balance, then place the container on the balance.
- Do not lean on the balance table while weighing. This usually moves the balance from level and results in an inaccurate weighing.
- Always check the zero of your balance before you begin a weighing.
- Make sure your balance is clean when you finish.

**Melting or Freezing Point**

The melting or freezing point is an important characteristic property of matter. These two terms are synonymous, since they both indicate the temperature at which the change from solid to liquid or liquid to solid takes place. You can quite easily measure the freezing point of a substance by placing a small amount of it (does it matter how much?) in a test tube, which is in turn placed in a beaker containing a 60/40 ice and water mixture. The freezing point is the point at which both solid and liquid are present; this forms a sort of slush that is easiest to work with if the substance is carefully stirred with the temperature sensor while it is cooling. If the liquid fails to freeze after it has reached a temperature of 0°C, record the melting or freezing point as “below 0°C.” (Hint: it may be necessary to add salt to the ice/water mixture to get the temperature of the unknown down to 0°C.)

Write a program to measure the freezing point of your sample. **Note** that you must calibrate your temperature sensor before making any measurements, or use the factory calibration for the Model 103 thermistor.

**Boiling Point**

Boiling point is also a characteristic property that helps us to identify substances as discussed in the previous chapter. The boiling point of a substance is the temperature at which the energy of the molecules is sufficient to allow them to push out of the solution.
into the atmosphere above the liquid surface. Boiling point is therefore dependent on the atmospheric pressure pushing down on the surface of the liquid, as well as the size and “tendency to stick together” of the molecules of the substance.

As discussed, this causes some serious problems at high altitudes, since all boiling point tables are recorded for a standard atmospheric pressure commonly found at sea level. If you are working at a high altitude, you will need a set of standards to develop a boiling point conversion graph. Several compounds and their sea level boiling points are listed in Table 5. Your laboratory instructor may wish to have you use other compounds.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Acetone</th>
<th>Chloroform</th>
<th>Ethanol</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sea Level B.P. (°C)</td>
<td>56.5</td>
<td>61.2</td>
<td>78.5</td>
<td>100</td>
</tr>
<tr>
<td>Observed B.P. (°C)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Since most organic compounds are extremely flammable, certain precautions must be taken to avoid fires during the boiling point determination. You should determine the boiling point by setting up a 100 mL beaker containing about 75 mL of water and heating this water to boiling with a hot plate.

Place about 1 mL of your sample (enough to cover the tip of the temperature sensor) in the bottom of a clean, dry test tube. Place a small chip of broken glass or a **boiling chip** provided by the laboratory instructor into the test tube with the unknown. This will promote smooth boiling rather than bumping, which may result in ejection of a part of your sample from the test tube. Place the test tube in the hot water, stir the sample gently with the temperature sensor, and record the temperature at which boiling takes place.
Construct a graph relating the recorded boiling points for these substances to the handbook value for the boiling points at 760 mm Hg (sea level). When you have determined the boiling point of your unknown, you can use this graph to convert it to the boiling point to be expected at 760 mm pressure. Do you think that barometric pressure will affect the freezing point? Why?

You can also use the slope-intercept equation developed by the linear regression through your data points to compute the corrected boiling point for your compound:

\[ y = mx + b \]

Your compound sea level B.P. = slope \times \text{(your compound B.P.)} + \text{correction factor (b)}

**Solubility**

Solubility tests are usually made by mixing a few drops of solute with the solvent being tested, one drop at a time. Examine the interaction of each drop with the solvent being tested to see if there is complete mixing, i.e., the solute is soluble in the solvent, partial mixing, i.e., only one or two drops mixes with the solvent, or no mixing, i.e., the solute forms a separate phase on the top or the bottom of the solvent being tested. The solvents you will use are water, a highly polar solvent, isopropyl alcohol, a moderately polar solvent, and hexane, (a component of gasoline) a nonpolar solvent. Compounds are classified as infinitely or very soluble (V), slightly soluble (S), or insoluble (I). If the unknown is insoluble, you will see either a cloudy mixture form, or a two-layer system. If the unknown is slightly soluble, you will be able to add several drops of it to the water before you observe the cloudiness. To observe this before you test the unknown, hold a test tube with about 5 mL of water at eye level and add one drop of water to it, holding the tip of the dropper just above the water level. Notice that you see no change in appearance. Now repeat this, with one, two, then three drops of hexane from a dropper in the same manner, and notice that you observe two layers, with the hexane on top of the water. When you mix this by lightly flipping the bottom of the test tube, you will notice a cloudiness appear resulting from the dispersion of the hexane within the water.

**Other Properties**

Color and odor are other characteristic properties of matter. Color is easily observed, but odor (or smell) is more difficult to categorize quantitatively and is often dangerous to determine. As was noted earlier, the fumes from several of the unknowns and solvents are poisonous. Never deliberately inhale the vapors of any volatile chemical without specific instructions to do so.

**Treatment of Data**

To make your job of identification of the unknown a little easier, Table 6, Table 7, and Table 8, which list the properties of some common compounds, are presented at the end of this chapter. These tables should help in the identification of your unknown.
For instance, suppose that you had a substance that boiled at 56 °C corrected to 760 mm pressure. By looking up this temperature in the boiling point index and allowing yourself one degree error in each direction in reading the temperature sensor, you can find code letters for all of the substances listed in the table with boiling points from 55 °C to 57 °C. In this particular example, the number of possibilities has just been cut to three! Turn to the table of physical properties of organic compounds, and you can find the substance and its properties by looking for the code in the left column.

Begin by selecting those compounds that have a boiling point equal to that of the corrected (760 mm Hg) boiling point you determined. Look up each in turn and compare its melting point, density, and solubility with those of your unknown compound. Most of them will be eliminated from consideration very quickly. If all of them seem to be eliminated, try the entries for boiling point slightly lower or higher than your corrected value and see whether you can locate your compound. The density index should be of similar assistance in decreasing the number of possible compounds.

REPORT: This will be the first laboratory report that you will turn in this semester. Your lab instructor has given you a syllabus and guidelines as to the format of this lab write-up. Be sure to include the necessary data, graph printouts, and information that the instructor has required. Your instructor has been issued an answer key as to the identity of the organic unknowns, you should be able to identify it before leaving class today.

Data Summary

PHYSICAL PROPERTIES OF MATTER

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Unknown</th>
<th>Known</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boiling point</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Freezing point</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Density</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solubility in water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solubility in alcohol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solubility in hexane</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Color</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 6. Alphabetical Listing of the Physical Properties of Some Common Organic Compounds.

<table>
<thead>
<tr>
<th>Index Code</th>
<th>Compound</th>
<th>Boiling Point Celsius</th>
<th>Freezing Point Celsius</th>
<th>Density (g/mL)</th>
<th>Solubility</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Water</td>
</tr>
<tr>
<td>A</td>
<td>Acetic acid</td>
<td>118.0</td>
<td>16.6</td>
<td>1.049</td>
<td>V</td>
</tr>
<tr>
<td>B</td>
<td>Acetone</td>
<td>56.5</td>
<td>-95.0</td>
<td>0.792</td>
<td>V</td>
</tr>
<tr>
<td>C</td>
<td>Benzene</td>
<td>80.0</td>
<td>5.5</td>
<td>0.879</td>
<td>I</td>
</tr>
<tr>
<td>D</td>
<td>Carbon tetrachloride</td>
<td>76.8</td>
<td>-22.8</td>
<td>1.595</td>
<td>I</td>
</tr>
<tr>
<td>E</td>
<td>Chloroform</td>
<td>61.2</td>
<td>-64.5</td>
<td>1.498</td>
<td>I</td>
</tr>
<tr>
<td>F</td>
<td>Cyclohexane</td>
<td>81.4</td>
<td>6.5</td>
<td>0.779</td>
<td>I</td>
</tr>
<tr>
<td>G</td>
<td>1,1-Dimethyloxyethane</td>
<td>64.5</td>
<td>-113.0</td>
<td>0.847</td>
<td>S</td>
</tr>
<tr>
<td>H</td>
<td>Ethanol</td>
<td>78.5</td>
<td>-117.0</td>
<td>0.789</td>
<td>V</td>
</tr>
<tr>
<td>I</td>
<td>Ethyl acetate</td>
<td>77.3</td>
<td>-83.6</td>
<td>0.901</td>
<td>S</td>
</tr>
<tr>
<td>J</td>
<td>Ethyl ether</td>
<td>34.0</td>
<td>-116.0</td>
<td>0.714</td>
<td>S</td>
</tr>
<tr>
<td>K</td>
<td>Ethyl propyl ether</td>
<td>64.0</td>
<td>-79.0</td>
<td>0.747</td>
<td>S</td>
</tr>
<tr>
<td>L</td>
<td>Heptane</td>
<td>98.4</td>
<td>-90.5</td>
<td>0.684</td>
<td>I</td>
</tr>
<tr>
<td>M</td>
<td>Isopropanol</td>
<td>82.3</td>
<td>-88.5</td>
<td>0.785</td>
<td>V</td>
</tr>
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<td>N</td>
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<td>0.796</td>
<td>V</td>
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<tr>
<td>O</td>
<td>Methyl acetate</td>
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<td>-98.1</td>
<td>0.927</td>
<td>V</td>
</tr>
<tr>
<td>P</td>
<td>Nitrobenzene</td>
<td>210.0</td>
<td>5.7</td>
<td>1.198</td>
<td>I</td>
</tr>
<tr>
<td>Q</td>
<td>2-Pentyne</td>
<td>56.0</td>
<td>-101.0</td>
<td>0.687</td>
<td>I</td>
</tr>
<tr>
<td>R</td>
<td>2-Propanethiol</td>
<td>60.0</td>
<td>-130.0</td>
<td>0.808</td>
<td>I</td>
</tr>
<tr>
<td>S</td>
<td>n-Propanol</td>
<td>97.2</td>
<td>-127.0</td>
<td>0.804</td>
<td>V</td>
</tr>
<tr>
<td>T</td>
<td>Oleic acid</td>
<td>286.0</td>
<td>4.0</td>
<td>0.895</td>
<td>I</td>
</tr>
<tr>
<td>U</td>
<td>Pentane</td>
<td>36.2</td>
<td>-131.0</td>
<td>0.626</td>
<td>I</td>
</tr>
<tr>
<td>V</td>
<td>Tert-butyl alcohol</td>
<td>82.3</td>
<td>25.5</td>
<td>0.786</td>
<td>V</td>
</tr>
<tr>
<td>W</td>
<td>Water</td>
<td>100.0</td>
<td>0.0</td>
<td>1.000</td>
<td>V</td>
</tr>
</tbody>
</table>

I = insoluble   S = slightly soluble   V = very soluble
Table 7. Physical Properties of Some Common Organic Compounds Listed by Density.

<table>
<thead>
<tr>
<th>Index Code</th>
<th>Compound</th>
<th>Boiling Point Celsius</th>
<th>Freezing Point Celsius</th>
<th>Density (g/mL)</th>
<th>Solubility Water</th>
<th>Solubility Alcohol</th>
<th>Solubility Chloroform</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>Pentane</td>
<td>36.2</td>
<td>−131.0</td>
<td>0.626</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>L</td>
<td>Heptane</td>
<td>98.4</td>
<td>−90.5</td>
<td>0.684</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>Q</td>
<td>2-Pentyne</td>
<td>56.0</td>
<td>−101.0</td>
<td>0.687</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>J</td>
<td>Ethyl ether</td>
<td>34.0</td>
<td>−116.0</td>
<td>0.714</td>
<td>S</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>K</td>
<td>Ethyl propyl ether</td>
<td>64.0</td>
<td>−79.0</td>
<td>0.747</td>
<td>S</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>F</td>
<td>Cyclohexane</td>
<td>81.4</td>
<td>6.5</td>
<td>0.779</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>M</td>
<td>Isopropanol</td>
<td>82.3</td>
<td>−88.5</td>
<td>0.785</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>V</td>
<td>Tert-butyl alcohol</td>
<td>82.3</td>
<td>25.5</td>
<td>0.786</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>H</td>
<td>Ethanol</td>
<td>78.5</td>
<td>−117.0</td>
<td>0.789</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>B</td>
<td>Acetone</td>
<td>56.5</td>
<td>−95.0</td>
<td>0.792</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>N</td>
<td>Methanol</td>
<td>64.6</td>
<td>−97.8</td>
<td>0.796</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>S</td>
<td>n-Propanol</td>
<td>97.2</td>
<td>−127.0</td>
<td>0.804</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>R</td>
<td>2-Propanethiol</td>
<td>60.0</td>
<td>−130.0</td>
<td>0.808</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>G</td>
<td>1,1-Dimethynoxethane</td>
<td>64.5</td>
<td>−113.0</td>
<td>0.847</td>
<td>S</td>
<td>S</td>
<td>V</td>
</tr>
<tr>
<td>C</td>
<td>Benzene</td>
<td>80.0</td>
<td>5.5</td>
<td>0.879</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>T</td>
<td>Oleic acid</td>
<td>286.0</td>
<td>4.0</td>
<td>0.895</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
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<td>Ethyl acetate</td>
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<td>−83.6</td>
<td>0.901</td>
<td>S</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>O</td>
<td>Methyl acetate</td>
<td>57.0</td>
<td>−98.1</td>
<td>0.927</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>W</td>
<td>Water</td>
<td>100.0</td>
<td>0.0</td>
<td>1.000</td>
<td>V</td>
<td>V</td>
<td>I</td>
</tr>
<tr>
<td>A</td>
<td>Acetic acid</td>
<td>118.0</td>
<td>16.6</td>
<td>1.049</td>
<td>V</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>P</td>
<td>Nitrobenzene</td>
<td>210.0</td>
<td>5.7</td>
<td>1.198</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>E</td>
<td>Chloroform</td>
<td>61.2</td>
<td>−64.5</td>
<td>1.498</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
<tr>
<td>D</td>
<td>Carbon tetrachloride</td>
<td>76.8</td>
<td>−22.8</td>
<td>1.595</td>
<td>I</td>
<td>V</td>
<td>V</td>
</tr>
</tbody>
</table>

I = insoluble  S = slightly soluble  V = very soluble
Table 8. Physical Properties of Some Common Organic Compounds Listed by Boiling Point.

<table>
<thead>
<tr>
<th>Index Code</th>
<th>Compound</th>
<th>Boiling Point Celsius</th>
<th>Freezing Point Celsius</th>
<th>Density (g/mL)</th>
<th>Solubility</th>
</tr>
</thead>
<tbody>
<tr>
<td>J</td>
<td>Ethyl ether</td>
<td>34.0</td>
<td>−116.0</td>
<td>0.714</td>
<td>S</td>
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<td>U</td>
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<td>36.2</td>
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<tr>
<td>B</td>
<td>Acetone</td>
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<td>−95.0</td>
<td>0.792</td>
<td>V</td>
</tr>
<tr>
<td>O</td>
<td>Methyl acetate</td>
<td>57.0</td>
<td>−98.1</td>
<td>0.927</td>
<td>V</td>
</tr>
<tr>
<td>R</td>
<td>2-Propanethiol</td>
<td>60.0</td>
<td>−130.0</td>
<td>0.808</td>
<td>I</td>
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<tr>
<td>E</td>
<td>Chloroform</td>
<td>61.2</td>
<td>−64.5</td>
<td>1.498</td>
<td>I</td>
</tr>
<tr>
<td>K</td>
<td>Ethyl propyl ether</td>
<td>64.0</td>
<td>−79.0</td>
<td>0.747</td>
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</tr>
<tr>
<td>G</td>
<td>1,1-Dimethoxyethane</td>
<td>64.5</td>
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<td>0.847</td>
<td>S</td>
</tr>
<tr>
<td>N</td>
<td>Methanol</td>
<td>64.6</td>
<td>−97.8</td>
<td>0.796</td>
<td>V</td>
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<tr>
<td>D</td>
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<td>−117.0</td>
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<td>M</td>
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<td>−88.5</td>
<td>0.785</td>
<td>V</td>
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<tr>
<td>V</td>
<td>Tert-butyl alcohol</td>
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<td>25.5</td>
<td>0.786</td>
<td>V</td>
</tr>
<tr>
<td>S</td>
<td>n-Propanol</td>
<td>97.2</td>
<td>−127.0</td>
<td>0.804</td>
<td>V</td>
</tr>
<tr>
<td>L</td>
<td>Heptane</td>
<td>98.4</td>
<td>−90.5</td>
<td>0.684</td>
<td>I</td>
</tr>
<tr>
<td>W</td>
<td>Water</td>
<td>100.0</td>
<td>0.0</td>
<td>1.000</td>
<td>V</td>
</tr>
<tr>
<td>A</td>
<td>Acetic acid</td>
<td>118.0</td>
<td>16.6</td>
<td>1.049</td>
<td>V</td>
</tr>
<tr>
<td>P</td>
<td>Nitrobenzene</td>
<td>210.0</td>
<td>5.7</td>
<td>1.198</td>
<td>I</td>
</tr>
<tr>
<td>T</td>
<td>Oleic acid</td>
<td>286.0</td>
<td>4.0</td>
<td>0.895</td>
<td>I</td>
</tr>
</tbody>
</table>

I = insoluble  S = slightly soluble  V = very soluble