Experiment 12

Bomb Calorimetry

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

A bomb calorimeter will be calibrated using a sample of known benzoic acid and then used to determine the calorie content of an unknown corn chip. This value will be compared to the tabulated nutritional facts.

Background

Combustion is one of the most important types of reaction studied by means of calorimetry. We can measure the amount of heat produced from a combustion reaction by measuring the temperature change effected on the water-filled container surrounding it. A bomb calorimeter is a device specifically designed to contain combustion reactions and the heat they produce. It is an insulated container of water in which a metal "bomb" (reaction chamber) is placed with a stirrer to circulate the water and a thermometer to measure the temperature (Fig. 1). The "bomb" is a strong metal container that can withstand the high pressures associated with the rapid combustion of a material and transmit the heat to its surroundings very quickly. Inside the bomb is a sample holder, an electrical ignition system to start the reaction, and enough oxygen gas to achieve complete and rapid combustion (Fig. 2).

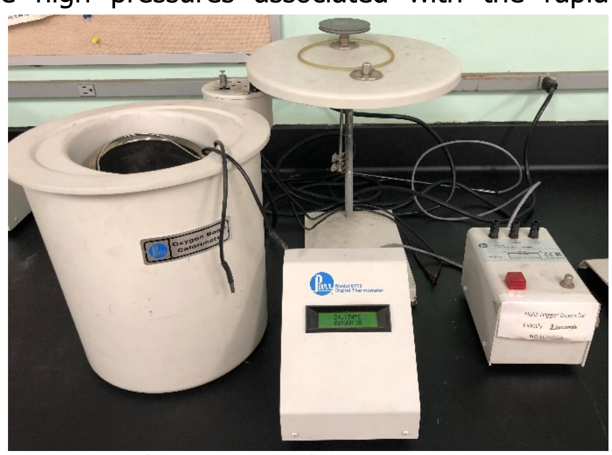


Figure 1: Bomb Calorimeter Set-Up

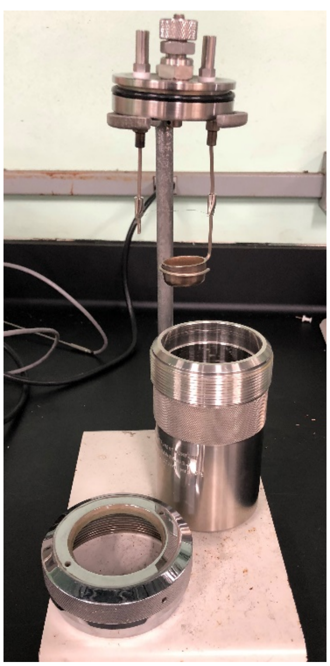


Figure 2: Bomb Set-Up

The sample is placed in the sample holder with sufficient oxygen pumped into the bomb to allow complete combustion. The reaction chamber is connected to an electrical circuit to allow external ignition and then immersed in the water and the initial temperature measured over a length of time to ensure that it is stable. Ignition sends a spark to begin combustion. The heat produced is trapped by the water, causing an increase in temperature, and the final temperature reach is recorded over a length of time. This increase in temperature corresponds to the amount of heat generated (evolved) by the combustion reaction.

To calculate the heat of combustion for an unknown, the relationship between the temperature change of the water inside the calorimeter and the heat produced from a known reaction must be measured first in a process called calibration. The relationship is called the heat capacity of the calorimeter (C_{cal}), which includes the bomb, water, stirrer, thermometer, and entire container. The heat absorbed by the calorimeter is dependent on this constant and the change in temperature, as seen in Eqn. 1.

$$q_{cal} = C_{cal} \times \Delta T_{cal} \qquad \text{Eqn. 1}$$

Inside the calorimeter, a small piece of string is used to hold the sample in place. This string also fully combusts, adding heat, with a known heat of combustion of 3.59 cal./cm. Thus the heat the calorimeter absorbs due to both the reaction and the string releasing heat during combustion is shown in Eqn. 2.

$$q_{cal} = -(q_{rxn} + q_{string}) \qquad \text{Eqn. 2}$$

Calibration is performed by using a sample of a substance with a known heat of combustion, such as benzoic acid, and observing the resulting temperature change of the calorimeter when a known amount of the substance is combusted. As each calorimeter is different, calibration must be performed for each instrument individually.

The initial and final temperatures will be determined graphically. The temperature inside the calorimeter will be recorded over time and linear trendlines will be used to find an accurate average for both.

Example Problem: Calibration of C_{Cal}

The combustion of 1.000 g of benzoic acid ($C_7H_6O_2$) will produce -26.38 kJ of heat. A 2.500 g sample of benzoic acid and 10.0 cm of string with a heat of combustion of -3.59 cal/cm was combusted in a bomb calorimeter. The trendlines for before ignition was y = 0.0038x + 20.122 and after ignition was y = 0.0132x + 24.906 with an intercept of x = 5.2 min., calculate the heat capacity of the calorimeter (C_{cal}) in kJ/°C.

Step 1: Solving for C_{Cal} from Eqn. 1

Since $q_{cal} = C_{Cal}\,\Delta T$, rearranging for C_{cal} gives:

$$C_{cal} = \frac{q_{cal}}{\Delta T}$$

Step 2: Find the heat released by the reaction, q_{rxn}

$$2.500 \text{ g benzoic acid} \times \frac{-26.38 \text{ kJ}}{1.000 \text{ g benzoic acid}} \times \frac{1000 \text{ J}}{1 \text{ kJ}} \times \frac{1 \text{ cal.}}{4.184 \text{ J}} = -15762 \text{ cal. evolved}$$

Step 3: Find the heat released by the string, q_{string}

$$10.0 \text{ cm} \times \frac{-3.59 \text{ cal}}{1 \text{ cm}} = -35.9 \text{ cal. evolved}$$

Step 4: Find the heat absorbed by the calorimeter, q_{cal}

$$q_{cal} = -(q_{rxn} + q_{string}) = -(-15762 \text{ cal.} + -35.9 \text{ cal.}) = 15798 \text{ cal.}$$

Step 5: Find the change in temperature, ΔT

Before ignition: y = 0.0038(5.2) + 20.122 = 20.142°C

After ignition: y = (0.0132)(5.2) + 24.906 = 24.975°C

$\Delta T = (T_{final} - T_{initial})$ = 24.975°C – 20.1˚C = 4.875°C

Step 5: Find C_{cal}

$$C_{cal} = \frac{15798 \text{ cal.}}{4.833^{\circ}\text{C}} = 3269 \text{ cal./}^{o}\text{C}$$

This relationship means that for the apparatus in the example, a 1°C temperature increase corresponds to the reaction and string producing 1260 cal. of heat. A 2°C increase would indicate that the reaction produced 2520 cal. of heat (2°C x 1260 cal./°C), etc.

In the first half of this experiment, you will calibrate your bomb calorimeter with a known quantity of benzoic acid to find C_{cal}. In the second half of this experiment, you will use your calculated C_{cal} to find the heat of combustion (calorie content) for a food item provided by your instructor and compare it to the tabulated values on the nutrition label. The example shown is of a corn chip.

Example Problem: Finding the Caloric Content for a Food Item

Using the same calorimeter as in the previous example, a 2.8555 g corn chip and 10.0 cm of string are combusted. The trendlines before ignition was y = 0.0105x + 19.812 and after ignition was y = 0.0087x + 27.623 with an intercept of x = 5.3 min. Calculate the caloric content for the chip in Cal./serving.

Step 1: Find the change in temperature, ΔT

Before ignition: y = 0.0105(5.3) + 19.812 = 19.868°C

After ignition: y = 0.0087(5.3) + 27.623 = 27.669°C

ΔT = 27.669°C – 19.868°C = 7.801°C

Step 2: Find the heat absorbed by the calorimeter, q_{cal}

$q_{cal} = (3269 \text{ cal./°C})(7.801\text{°C}) = 2.550 \times 10^4 \text{ cal.}$

Step 3: Find the heat released by the string, q_{string}

$$10.0 \text{ cm} \times \frac{-3.59 \text{ cal.}}{1 \text{ cm}} = -35.9 \text{ cal}$$

Step 4: Find the heat released by the reaction, q_{rxn}

$q_{cal} = -(q_{string} + q_{rxn})$ so $q_{rxn} = -q_{cal} - q_{string} = -2.550 \times 10^4 \text{ cal.} -(-35.9 \text{ cal.}) = -25460 \text{ cal.}$

Step 5: Converting q_{rxn}

$$-25460 \text{ cal.} \times \frac{1 \text{ Calorie}}{1000 \text{ calories}} = -25.460 \text{ Calories}$$

Step 4: Find heat of combustion. ΔH_{rxn}

$$\frac{-25.460 \text{ Calories}}{2.8555 \text{ g}} = -8.9161 \text{ Cal./gram}$$

Step 5: Converting heat of combustion

$$\frac{-8.9161 \text{ Cal.}}{1 \text{ gram}} \times \frac{28 \text{ grams}}{1 \text{ serving}} = -250 \text{ Cal./serving or } 250 \text{ Cal./serving evolved}$$

Procedure

Part I: Calibration of the Calorimeter with Benzoic Acid

1. Fill the calorimeter water bucket with 2.00 L of distilled water using a volumetric flask. Place the water bucket inside the calorimeter.

2. Obtain one pellet of benzoic acid. Weigh it accurately and record its weight on your data sheet.

3. Cut a 10.0 cm length of string. Tie one end of the string around the benzoic acid tablet and the other to the ignition wire between the electrodes in the bomb. The tablet may either rest in the sample holder or hang in the air above it. The string must not come untied during any part of the experiment. A loose string will not carry the ignition spark and no combustion will occur.

4. Close the bomb by carefully lowering the lid with sample vertically into the bottom portion. Place the large O-ring cover over the top and hand-tighten it fully.

5. Ensure that the pressure valve is closed by screwing it down by hand. Pressurize the bomb to 30 atm of oxygen from the provided tank (Caution! Do not exceed 35 atm!).

6. Using the provided clamps, carefully place the bomb into the water bucked into the calorimeter, ensuring no water is spilled or removed. Connect the electrodes to the wire connectors in the bomb's lid (it does not matter which). If any continuous gas bubbles are observed, then the bomb is leaking and will need to be removed and reassembled before continuing.

7. Place the calorimeter's lid over the top, being careful to insert the stirrer in the water. Connect the two wheels by pulley and start the motor to begin stirring. Connect the outer wires to the ignition trigger around the "ground" and "10 cm" knobs. Lower the steel thermometer into its slot on the calorimeter's lid.

8. Turn on the temperature reader and begin recording the temperature every 30 seconds in the data table. The reader is both a timer and a thermometer.

9. At four minutes, ignite the bomb by pressing the ignition button. The indicator light should be on while the ignition trigger is pressed. Hold the trigger for **exactly three seconds** and no longer, then release.

10. Continue monitoring the temperature. By the next reading at 4.5 minutes, the temperature should have increased by a degree Celsius or more.

11. Continue to record the temperature every 30 seconds until 12 minutes total.

12. Stop the stirrer. Carefully remove the pulley, the thermometer, and then the calorimeter's lid. Remove the bomb and the water.

13. Release the pressure in the bomb by opening the pressure valve. Open the bomb and clean any residue that may remain. If any sample remains behind, weigh it. Dry the bomb thoroughly inside and out.

14. Pour out the water and dry the bucket thoroughly inside and out.

Part II: Finding the Heat of Combustion for a Food Item

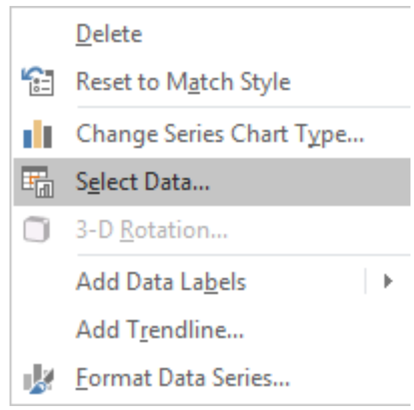
1. Repeat Steps 1-14 in Part I. For Step 2, use the food item assigned by your instructor instead of the benzoic acid pellet.

Part III: Finding ΔT for Both Trials

1. Prepare two graphs of time (min.) versus temperature (°C), one for Part I and one for Part II.

2. For each graph, find the change in temperature by adding two trendlines: before ignition and after ignition.

On Excel:



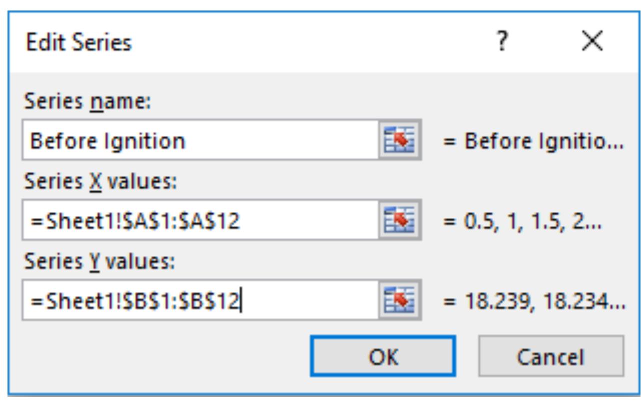
a. Right click on any data point on the graph. From the pop-up menu, click “Select Data”.

b. Under “Legend Entries (Series)”, click “Add”.

c. In “Series name”, type “Before Ignition”.

d. In “Series X values”, click the button at the right end of the box and then drag and select all x-values that correspond to the times before ignition. Click the button on the right of the box to go back.

e. In “Series Y values”, delete what is typed there, click the button on the right end of the box, and then drag and select all y-values that correspond to the temperatures before ignition. These should be ordered pairs with what you selected in Step D. Click the button on the right of the box to go back. Click “OK”.



f. Under “Legend Entries (Series)”, click “Add”.

g. In “Series name”, type “After Ignition”.

h. Repeat Steps D-E to select the times and temperatures that correspond to your data after ignition.

Note: not ALL data on your graph should be included in these trendlines.

i. Click “OK”.

j. On the graph, add a trendline to the data highlighted as “Before Ignition” and the data “After Ignition”. Be sure to show equations for both.

3. Print out each graph.

4. Using a ruler and a pencil, extrapolate both trendlines to the edges of the graph. Then turn the ruler vertically and draw a line that intersects both extrapolated trendlines in such a way that the area of the upper and lower triangles created by the line, the trendline, and the curve itself have approximately the same area (Fig. 4). Continue this line down to the x-axis and estimate the value.

5. Plug in your x-value from Step 4 to **both** trendline equations to solve for y. These y-values correspond to your initial and final temperatures for each trial.

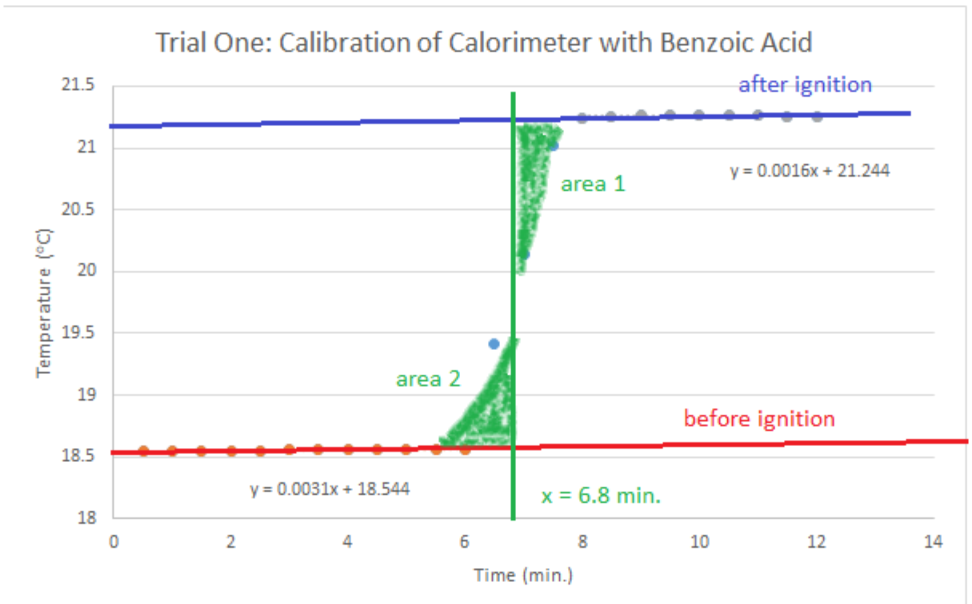


Figure 4: Finding initial and final temperature graphically

Part IV: Finding Heat of Combustion for a Food Item

1. Using your initial and final temperatures from your calibration with benzoic acid graph, find C_{cal} in cal./°C.

Note: remember that the string burned inside the bomb with the sample. You will need to subtract the heat evolved from the burning string (the heat of combustion for the string is -3.59 cal./cm).

2. Using your C_{cal} calculated in Step 1 and your initial and final temperatures from your food item graph, find the heat of combustion for the food item in cal./gram, then convert to Calories/gram and Calories/serving.

3. Compare your calculated value in Step 2 to the tabulated value listed on the nutrition label.

Experiment 12—Data Sheet

Name: ______________________________________ **Calorimeter:** _________________

Partners: ____

Part I: Calibration of the Calorimeter with Benzoic Acid

1. Mass of benzoic acid (g) ____________________

2. Heat released by the reaction (q_{rxn}, cal) ____________________
show calculation:

3. Initial length of string (cm) ____________________

4. Final length of string (cm) ____________________

5. Length of string burned (cm) ____________________
show calculation:

6. Heat released by string burning (q_{string}, cal) ____________________
show calculation:

7. Heat absorbed by the calorimeter (q_{cal}, cal) ____________________
show calculation:

8. Initial temperature of the calorimeter ($T_{initial}$, °C) ____________________
show calculation (use graph equation):

9. Final temperature of the calorimeter (T_{final}, °C) ____________________
show calculation (use graph equation):

10. Change in Temperature (ΔT, °C) ____________________
show calculation:

11. Heat Capacity of the calorimeter (C_{cal}, cal/°C) ____________________
show calculation:

Part II: Finding the Heat of Combustion for a Food Item

1. Mass of food item (g) ____________________

2. Initial length of string (cm) ____________________

3. Final length of string (cm) ____________________

4. Length of string burned (cm) ____________________
show calculation:

5. Initial temperature of the calorimeter ($T_{initial}$, °C) ____________________
show calculation (use graph equation):

6. Final temperature of the calorimeter (T_{final}, °C) ____________________
show calculation (use graph equation):

7. Change in Temperature (ΔT, °C) ____________________
show calculation:

8. Heat absorbed by the calorimeter (q_{cal}, cal) ____________________
show calculation:

9. Heat released by string burning (q_{string}, cal) ____________________
show calculation:

10. Heat released by the reaction (q_{rxn}, cal) ____________________
show calculation:

11. Heat of Combustion of food item (ΔH_{rxn}, Cal/g) ____________________
show calculation:

12. Serving Size (from label) ____________________

13. Heat of Combustion per serving (ΔH_{rxn}, Cal/serving) ____________________
show calculation:

13. Percent Error of Heat of Combustion (%) ____________________
show calculation:

Temperature Readings for Combustion of Benzoic Acid

Time (min)	Temperature (°C)
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
Ignition 4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	
9.5	
10.0	
10.5	
11.0	
11.5	
12.0	

Time (min)	Temperature (°C)
12.5	
13.0	
13.5	
14.0	
14.5	
15.0	
15.5	
16.0	
16.5	
17.0	
17.5	
18.0	
18.5	
19.0	
19.5	
20.0	
20.5	
21.0	
21.5	
22.0	
22.5	
23.0	
23.5	
24.0	
24.5	

Temperature Readings for Combustion of Corn Chip

Time (min)	Temperature (°C)
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
Ignition 4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	
9.5	
10.0	
10.5	
11.0	
11.5	
12.0	

Time (min)	Temperature (°C)
12.5	
13.0	
13.5	
14.0	
14.5	
15.0	
15.5	
16.0	
16.5	
17.0	
17.5	
18.0	
18.5	
19.0	
19.5	
20.0	
20.5	
21.0	
21.5	
22.0	
22.5	
23.0	
23.5	
24.0	
24.5	

Experiment 12—Post-Lab Assignment

1. Rank by *increasing* size, starting with the smallest unit: Joule, kilojoule, calorie, Calorie (kcal).

2. Using your data from Part II, calculate the heat of combustion of the food item in Cal./serving while ignoring the heat produced by the combustion of the string (q_{string}). Find the percent error.

3. From the food item's packaging, determine the percentage of Calories/serving that come from carbohydrates, fats, and proteins (hint: you will need to look up the values for the Calories per gram of each produced in the human body).

4. Bomb calorimeters are also common calibrated by combusting naphthalene instead of benzoic acid. If a 2.000 g sample of naphthalene (heat of combustion: -43.6 kJ/g) causes the temperature of a bomb calorimeter to rise by 7.81°C, calculate the C_{cal} of the calorimeter in cal./°C. You may ignore the heat from the combustion of the string (q_{string}).

5. Was your percent error for the heat of combustion of the food item high or low? Does this indicate *accurate* or *precise* results? Give three experimental reasons to account for the difference between your value and the label's.

Experiment 12—Pre-Lab Assignment

Name: ____________________________________

For all calculations, show all work and draw a box around the final answers.

1. A 0.700 g sample of benzoic acid and 10.0 cm of string are combusted in a bomb calorimeter and the temperature of the system increases from 25.00°C to 29.62°C. What is the heat capacity of the bomb calorimeter in (a) kJ/°C and (b) cal./°C?

2. Calculate the moles of $O_2(g)$ needed to completely burn 1.00 grams of sucrose ($C_{12}H_{22}O_{22}$). (hint: you will need the balanced combustion equation for sucrose).

3. Given that a bomb calorimeter has a volume of 1.5 L and an initial temperature of 25.0°C, what pressure of $O_2(g)$ in atm is required to completely combust 1.00 g sucrose?

4. Based on your answer in (3), why is the bomb pressure set to 30 atm in this experiment?

5. A 1.00 g sample of sucrose is combusted in the same calorimeter described in (1) and its temperature increased from 25.00°C to 31.62°C. Calculate the heat of combustion for sucrose in (a) kJ/mol and (b) Cal./mol