Oxygen Bomb Calorimetry

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Oxygen bomb calorimetry is used to determine the caloric value of organic materials. This information is relevant in ecological research for several purposes. For example, oxygen bomb calorimetry can be used to determine the efficiency of an organism's digestive tract in extracting energy or to assess forage quality and the amount of forage an organism must consume to survive. For this lab, we will employ oxygen bomb calorimetry to investigate resource allocation in plants (vegetation vs. fruits) in addition to the transfer of energy from plant biomass to animal biomass. Resource allocation refers to the differential growth of tissues or organs within an individual organism and of course this growth depends upon available energy. Organisms have only a limited amount of energy to allocate to their tissues and this available energy may change through the life span of the individual. Often early in the life of a plant, energy is directed toward establishment and growth. Later in life, energy may be directed towards flowering and reproduction. The timing of changes in resource allocation is termed a life history strategy and differs from species to species.

If oxygen is not limiting, energy is rapidly released when a material is combusted. An oxygen bomb calorimeter efficiently captures this energy as heat in a liquid of known specific heat. By knowing the initial and final temperature of the liquid, the weight of the liquid, plus the weight of the combusted sample, the caloric value of the sample may be determined. Water is the most common liquid used in the measurements because its specific heat is 1.0, i.e. it takes 1 calorie of heat to raise 1 g of water 1°C (or 4.18 Joules for the same gram of water 1°C).

Calorimeter equipment consists of an air-tight combustion chamber, the oxygen bomb, which is completely immersed in a metal bucket filled with water. Once the sample in the oxygen bomb has been ignited electrically, a delicate and accurate alcohol thermometer measures the change in water temperature. The complete capture of the energy released by combustion in the bomb, and its transfer to the water in the bucket is obviously crucial to accurate measurement of the sample's caloric value. Consequently, there must be some means to ensure that none of the heat from combustion is lost. The most accurate calorimeters, adiabatic calorimeters, use a circulating jacket of water that surrounds the water bucket and automatically matches any temperature change in the bomb. We will use a slightly less accurate calorimeter that relies on the dead air space created between two molded fiberglass jackets to prevent heat transfer from the water bucket.

Procedure

Tissue types:

Benzoic acid standard Plant matter (e.g., lentils)
Animal matter (e.g., beef stick)
"Wild card" – your choice of tissue, e.g. brine shrimp, algae, corn flakes

Preparing and loading the sample:

1. Choose one of the sample materials provided, starting with Benzoic acid, and grind it to a fine powder with a mortar and pestle.
2. Place the ground sample into the pellet press and form a firm pellet.
3. Weight the pellet on the balance. If the pellet weighs less than 0.5g, pulverize it, add more sample, and form a new pellet. If the pellet weighs more than 1.2g, carefully shave off material until the pellet weighs less than 1.2g.
4. Place the head of the bomb on its support stand. Attach 10 cm of nichrome wire to the ignition leads in the head of the bomb. Wrap each end of the wire through the "eyes" in the leads. Pull the caps on the leads down until they touch the wire. Form a "U" shaped loop in the nichrome wire (see above).

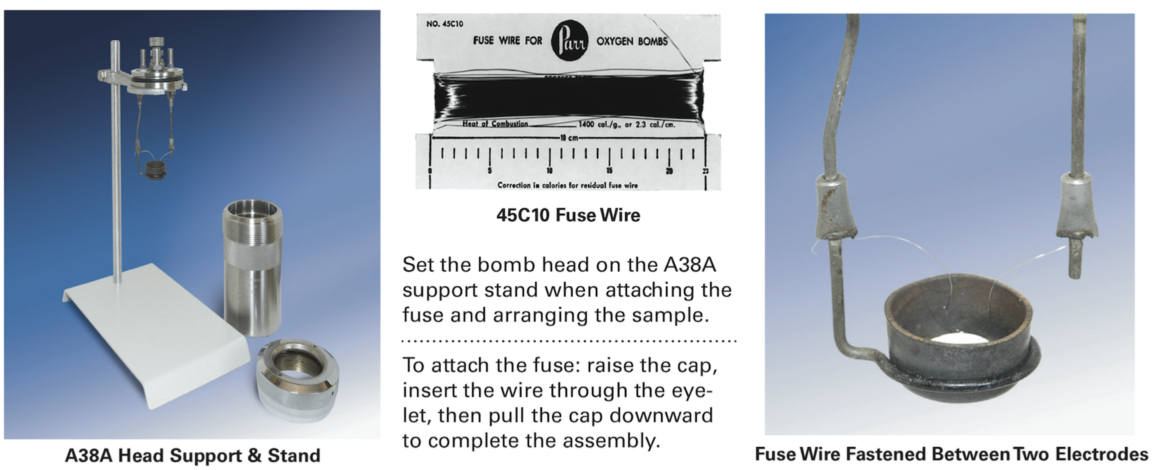

Figure 1: Setting up the nichrome ignition wire in the bomb

5. Place the pellet in the combustion vessel and then place the vessel in the wire loop in the bomb head. Push the nichrome wire down so that it is in firm contact with the sample. **NOTE: The wire must touch the sample in order to ignite it.**

6. Add 1 ml of distilled water to the bomb cylinder. Carefully place the head with the sample into the bomb cylinder; **do not twist it**. Care must be taken that the wire does not become dislodge from the sample. Firmly screw on the cap (hand tighten only).

7. Make sure the gas valve on top of the bomb is closed (hand tighten only).

8. **THIS STEP WILL BE DONE BY THE LAB INSTRUCTOR.** Place the slip connector (at the end of the gas line on the bottled oxygen tank) over the inlet valve (notice there is a small hole in the inlet valve oxygen will pass through this hole into the bomb).

9. **THIS STEP WILL BE DONE BY THE LAB INSTRUCTOR.** Place 25 atm of oxygen into the bomb.

10. The bomb is now ready to be loaded into the calorimetry bucket.

Loading the bomb into the calorimetry bucket:

11. Lower the bomb into the calorimetry bucket with the wire tongs.
12. Fill the calorimeter bucket with 2000 g of distilled water. **Examine the water around the bomb for gas bubbles; if a steady stream of bubbles appear do not proceed further. It may need to be refilled with oxygen after it is tightened.**
13. Using the handle on the calorimeter bucket, lift the bucket and place it into the calorimeter with the stirrer rod nearest the motor – the bottom of the calorimeter is designed so that the bucket will be balanced only in one direction. **NOTE: Never touch the outside of the bucket with your hands!** The reflective surface must not become marred as this will adversely affect the measurements.

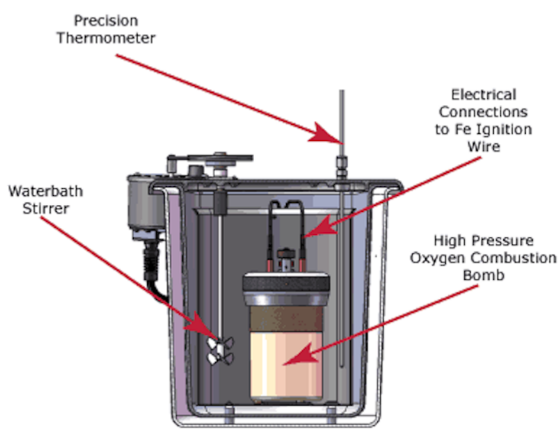

Figure 2: Schematic of bomb calorimeter setup.

14. Attach the two ignition lead wires to the top of the bomb. Be careful not to remove any water from the bucket.
15. Slowly lower the cover of the jacket over the calorimeter bucket, making sure the stirrer is not touching the wall of the bucket or the bomb.
16. ***Slowly*** attach the reading lens to the thermometer and slip the thermometer through its support rod atop the calorimeter jacket. Slowly push the thermometer into the bucket assembly. **NOTE: Do not force the thermometer into the bucket assembly.The thermometer is very expensive.**

17. Connect the rubber belt between the stirrer and its motro. Turn the stirrer motor "ON", and let the stirrer equilibrate the temperature in the water for at least 5 minutes. During this time, record the water temperature about every minute.

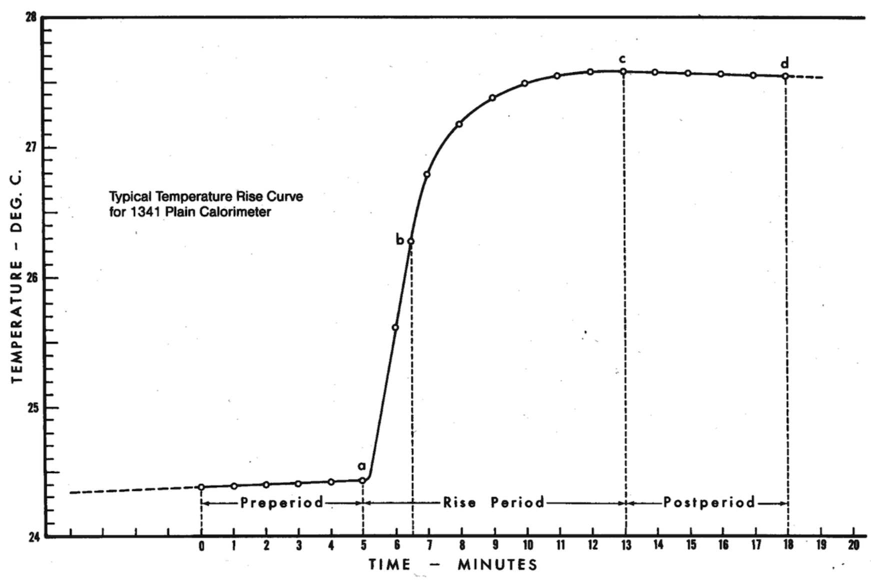

Figure 3: Typical temperature rise after ignition (point "a").

Igniting the sample:

18. No one should be directly over the bomb during the ignition phase of the procedure. After you are sure the temperature has equilibrated the sample can be ignited. Record the time and temperature (initial temperature). Plug in the ignition box. Press the ignition button and hold it down until the red light flashes (about a 0.5 sec flash). Do not hold the ignition button down for longer than 5 seconds.
19. Within 30 seconds after ignition, the temperature in the water should begin to rise. Record the increase in temperature at 1 minute intervals.
20. After about 5 minutes the temperature should reach a constant maximum and remain at that temperature for about 5 minutes. This constant maximum value is the final temperature.
21. Turn "OFF" the stirrer motor, slowly raise the thermometer completely out of the bomb assembly, and remove the top of the calorimetry jacket. Pull the ignition leads out of the bomb and carefully lift the calorimeter bucket out. Rest the bucket on a towel and then remove the bomb from the bucket. Immediately wipe any water drops from the side of the bucket.
22. Slowly open the gas valve on the top of the bomb and allow the gas to release. After the valve is completely open and all gas has been released, unscrew the top of the bomb and slowly remove the head of the bomb and place it on its stand.
23. Scrape any ash from the sample vessel and weigh it. To determine "ash-free dry weight" the ash weight is subtracted from the initial dry weight of the sample.

24. Remove all remaining nichrome wire from the leads and measure this wire with the card provided.
25. After you finish with you combustions:
    a. Empty the water tank, wipe it off with a soft cloth towel only, and turn it upside down to drain on the lab bench.
    b. Rinse the inside of the bomb thoroughly with distilled water and wipe it dry (inside and out) with a soft dry towel.
    c. Wipe the thermometer dry and place it carefully into its paper tube.
    d. Remove any ash or debris from the bomb and the sample vessels.

Standardization (From Parr Instrument's Introduction to Bomb Calorimetry)
Before a material with an unknown heat of combustion can be tested in a bomb calorimeter, the energy equivalent or heat capacity of the calorimeter must first be determined. This value represents the sum of the heat capacities of the components in the calorimeter, notably the metal bomb, the bucket and the water in the bucket. Since the exact amount of each of the metals used in the bomb and bucket is difficult to determine and continually changing with use, energy equivalents are determined empirically at regular intervals by burning a sample of a standard material with a known heat of combustion under controlled and reproducible operating conditions. Benzoic acid is used almost exclusively as a reference material for fuel calorimetry because it burns completely in oxygen; it is not hygroscopic and is readily available in very pure form. The amount of heat introduced by the reference sample is determined by multiplying the heat of combustion of the standard material by the weight of the sample burned. Then, by dividing this value by the temperature rise produce in the test, we obtain a resultant energy equivalent for this particular calorimeter. For example:

Consider a standardization test in which 1.651 grams of standard benzoic acid (heat of combustion 6318 cal/g) produced a temperature rise of 3.047°C. The energy equivalent (W) of the calorimeter is then calculated as follows:

$$W = \frac{1.651 \times 6318}{3.047} = 2416 \text{ cal/}^\circ\text{C}$$

For simplicity, the corrections usually applied for heats introduced by the fuse and by acid formation are omitted from the above example.

It is important to note that the energy equivalent for any calorimeter is dependent upon a set of operating conditions, and these conditions must be reproduced when the fuel sample is tested if the energy equivalent is to remain valid. It is readily apparent that a difference of one gram of water in the calorimeter will alter this value by one calorie per degree Celsius. Less obvious but equally important are the changes resulting from different bombs or buckets with unequal masses, different operating temperatures, different thermometers, or even the biases imposed by different operators.

A. Oxygen bomb Calorimetry Data Table

	Sample 1	Sample 2	Sample 3	Sample 4
Sample Wt (g)				
Ash Wt (g)				
Ash-free Dry Wt (g)				
Initial T (°C)				
Max T (°C)				
Δ T (°C)				
Wire correction				
Calories / g AFDW				

Oxygen bomb Calorimetry Assignment

10 pts total:
A. Complete the data table, above (1 pt)
B. Show your calculation for W and of calorific value for 1 sample (3 pt)
C. Complete the related worksheet questions (6 pts)

B. Calculations

Calorific value is the number of calories expended by the ash-free sample in the combustion less the calories generated by the formation of acid in the cylinder and the heat of the combustion of the nichrome wire.

$$\text{Calorific Value} = \frac{W(\text{cal}/^\circ\text{C}) \times \Delta\text{T }(^\circ\text{C}) - \text{ wire correction}}{\text{ash-free dry weight (g)}}$$

W = the amount of energy in cal/°C required to raise the temperature of 2000 g of water, the bucket and bomb by 1°C. This value is unique for each calorimeter. You will need to calculate W for your bomb calorimeter using the Benzoic acid standard.

The wire correction is read directly off the card provided with the nichrome wire.

In the space below, provide your calculations for W:

And here show your calculations for one of the samples in your data table:

C. Questions

Please average the class data and answer the following questions in complete sentences.

1a. Which sample has a higher energy content – consumer or primary producer?

1b. What was the difference in calories per gram? (higher sample – lower sample = difference)

1c. Did you expect more of a difference? Why or why not?

2a. Calculate the Lindeman efficiency from grass to cow trophic transfer using the following equation (show your calculation and circle your answer):

$$\frac{(\text{dry wt of cow}) \times (\text{energy content of beef})}{(\text{days cow raised}) \times (\text{dry wt grass consumed per day}) \times (\text{energy content of grass})} \times 100\%$$

Assume:
This is a free-range cow that eats only grass (no corn or other grains)
Weight of cow = 250kg dry weight
Energy content of beef = average of class data
Days cow raised = 500 days
Amount of grass consumed per day = 10kg/day dry weight
Energy content of grass = average class data

2b. How does your answer compare with the theoretical 10% transfer? Why do you think this is?