Measuring the Energy Content of Food using the Bomb Calorimeter

Aim: The aim of this laboratory exercise is to apply the First Law of Thermodynamics to determine the energy content (i.e. food energy) of some simple foods.

Theory:

Calorimetry is the science of measuring quantities of heat, as distinct from "temperature". The instruments used for such measurements are known as **calorimeters**. The most common type of calorimeter is the oxygen bomb calorimeter.

The **Calorific Value** of a sample may be broadly defined as the number of heat units liberated by a unit mass of a sample when burned with oxygen in an enclosure of constant volume.

The calorific value as determined in an oxygen bomb calorimeter is measured by a substitution procedure in which the heat obtained from the sample is compared with the heat obtained from combustion of a similar amount of benzoic acid whose calorific value is known. These measurements are obtained by burning a representative sample in a high-pressure oxygen atmosphere within a metal pressure vessel – called a bomb. The energy released by this combustion is absorbed within the calorimeter and the resulting temperature change within the absorbing medium is noted. The calorific value of the sample is then calculated by multiplying the temperature rise in the calorimeter by a previously determined energy equivalent determined from previous tests with a standardizing material.

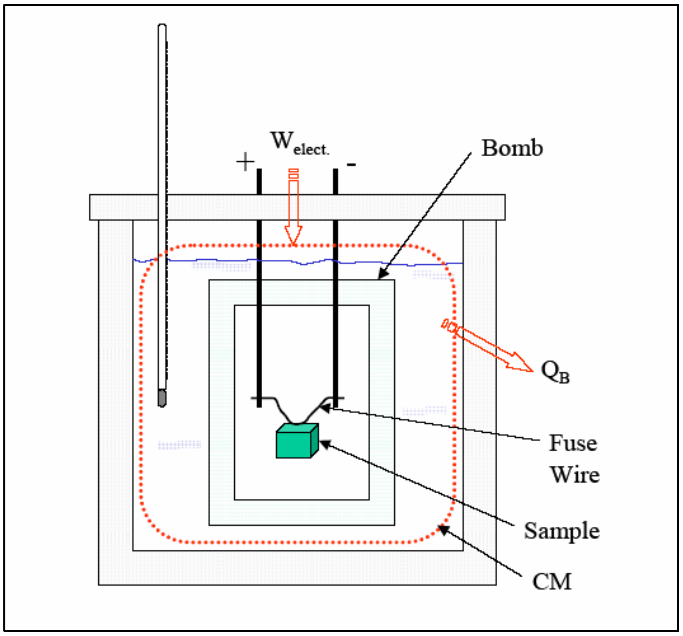

Fig. 1- Bomb Calorimeter

Apparatus:

- Food sample (weighed);
- Oxygen bomb calorimeter;
- Fuse wire;
- Thermometer;
- 1.2 litres of water;
- Supply of oxygen;
- Power source;
- Stop watch.

Procedure:

The bomb calorimeter is set up as shown in Fig.1 above.

1. **Prepare the sample in the bomb**. The food sample is put in the crucible together with a piece of cotton wool. A known length of fuse wire is passed through the sample with both ends of the wire being attached to the 2 electrodes of the bomb.
2. **Closing the bomb**. Care must be taken not to disturb the sample when moving the bomb head to the bomb cylinder. Check the sealing ring to be sure that it is in good condition and moisten it with a bit of water so that it will slide freely into the cylinder; then slide the head into the cylinder and push it down as far as it will go. For easy insertion, push the head straight down without twisting and leave the gas release valve open during this operation. Set the screw cap on the cylinder and turn it down firmly by hand to a solid stop. When properly closed, no threads on the cylinder should be exposed.
3. **Filling the bomb**. Connect the oxygen supply to the bomb and close the gas release valve. Open the filling connection control valve slowly and watch the gauge as the bomb pressure rises to the desired filling pressure (usually 30 atm., but never more than 40 atm.); then close the control valve.
4. **Fill the calorimeter bucket**. Put the bomb inside the calorimeter bucket by attaching the lifting handle to the screw cap and lowering the bomb with its feet spanning the circular boss in the bottom of the bucket. Handle the bomb carefully during this operation so that the sample will not be disturbed. Remove the lifting handle and add 1.2 litres of water inside the bucket.
5. **Final settings**. Connect the power source to the bomb and put the thermometer inside the bucket. Set the cover on the jacket and start the stirrer.
6. **Read and record temperatures**. Let the stirrer run for 5 minutes to reach equilibrium before starting a measured run. At the end of this period start a timer and read the temperature. Read and record temperatures at one-minute intervals for 5 minutes. Then, at the start of the 5th minute...
7. **Stand back from the calorimeter and fire the bomb.**
8. **Recording change in temperature**. The bucket temperature will start to rise within 30 seconds after firing. This rise will be rapid during the first few minutes;

then it will become slower as the temperature approaches a stable maximum as shown by the typical temperature rise curve of Fig. 2. Record temperatures at one-minute intervals until the difference between successive readings have been constant for 3 minutes. Usually the temperature will reach a maximum; then drop very slowly. But this is not always true since a low starting temperature may result in a slow continuous rise without reaching a maximum. As stated above, the difference between successive readings must be noted and the readings continued at one-minute intervals until the rate of the temperature change becomes constant over a period of 3 minutes.

9. **Opening the bomb**. After the last temperature reading, stop the motor and lift the cover from the calorimeter. Lift the bomb out of the bucket and open the gas release valve on the bomb head to release the gas pressure before attempting to remove the cap. After all pressure has been released, unscrew the cap and lift the head out of the cylinder. Examine the interior of the bomb for soot or other evidence of incomplete combustion. If such evidence is found, the test will have to be discarded.
10. **Fuse wire burnt**. Remove all unburned pieces of fuse wire from the bomb electrodes; straighten them and measure their combined length in centimeters. Subtract this length from the initial length of fuse wire used and enter this quantity on the data sheet as the net amount of wire burned - L_f.

Calculations:

The raw data should be plotted, as shown below (Fig. 2), and inspected for anomalies. There are a few features worth noting. First, the data should have a very slight positive slope during the Pre-period (if the initial water temperature is about 1.5 °C below room temperature to start). During the Post-period, there should be a very slight negative slope. Calculations are based on identifying the Pre and Post-periods, and on identifying the time to achieve 60% of the total temperature rise.

The following data should be available at the completion of a test:

a = time of firing
b = time when the temperature reaches 60 percent of the total rise
c = time at beginning of period in which the rate of temperature change has become constant
T_a = temperature at time of firing
T_c = temperature at time c
r_1 = rate (temperature units per time, °C/sec) at which temperature was rising during the 5-min. period before firing
r_2 = rate (temperature units per time, °C/sec) at which the temperature was rising during the 5-min. period after time c. If the temperature was falling instead of rising after time c, r_2 is negative and the quantity $-r_2$ (c-b) becomes positive and must be added when computing the corrected temperature rise

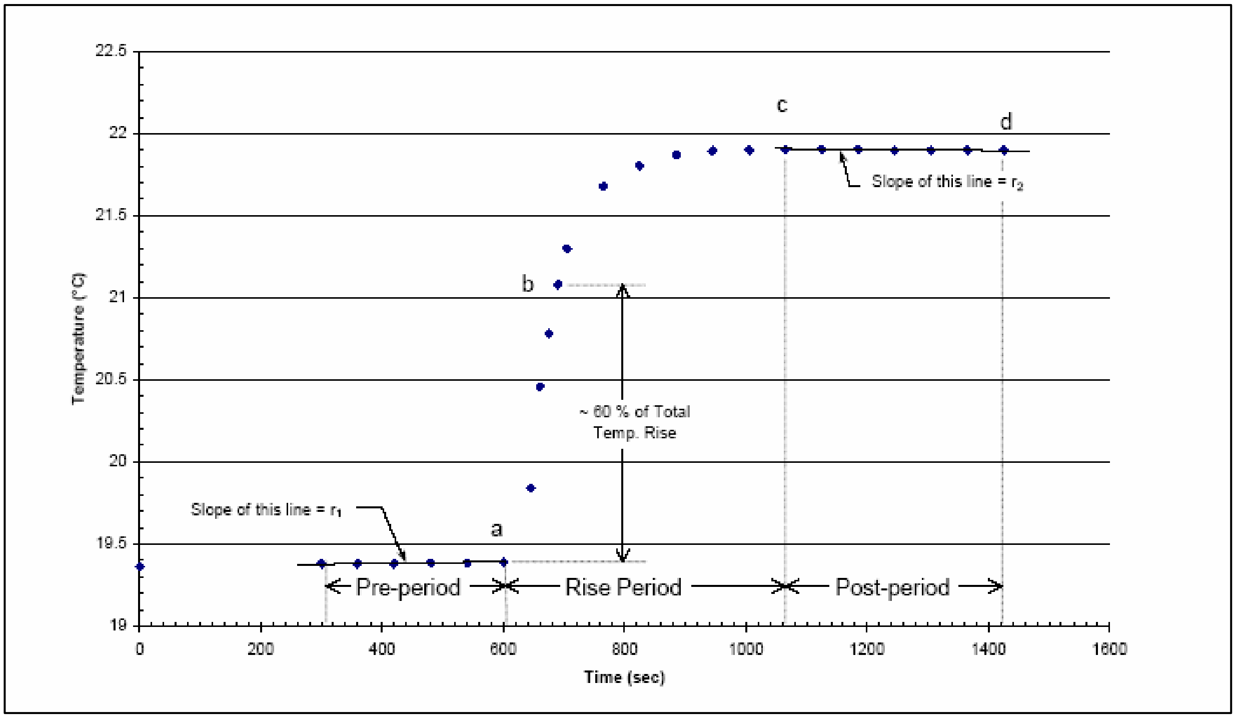

Fig. 2 – Typical Temperature Rise Curve

L_f = centimeters of fuse wire <u>consumed</u> in firing
Cth_{Cal} = thermal capacitance of the calorimeter
M_s = mass of test sample in grams

Temperature Rise:
Compute the net corrected temperature rise, ΔT, by substituting in the following equation:

$$\Delta T = (Tc\text{-}Ta) - r_1(b\text{-}a) - r_2(c\text{-}b)$$

Calibrating the Oxygen Bomb Calorimeter

The constant volume combustion bomb has been calibrated using a standard sample of benzoic acid according to the procedure below. The calibration is based on a simple First Law analysis of the bomb. Once the bomb has been calibrated, a similar analysis may be used to obtain the energy value of any test sample. Please refer to Fig. 1 above.

For the process of igniting the fuse and the test sample, and the resulting rise in water temperature, the 1st Law states that:

$$E_2 - E_1 = \text{Energy In} - \text{Energy Out}$$
$$\Delta E = W_{elect.} - Q_B$$

Since Energy is extensive, and changes in kinetic and potential energy are negligible:

$$\Delta E = \Delta U = \Delta U_S + \Delta U_f + \Delta U_W + \Delta U_B$$

Where subscripts:

- s = test sample
- f = fuse wire
- w = water surrounding bomb
- B = bomb
- Δ = (final state – initial state)
  - 1 = initial state
  - 2 = final state

Substituting for ΔE, the 1st Law becomes:

$$\Delta U_S + \Delta U_f + \Delta U_W + \Delta U_B = W_{elect.} - Q_B$$

Combining terms associated with combustion, and separately those associated with resulting temperature rise:

$$\Delta U_W + \Delta U_B + Q_B = -\Delta U_S \; -\Delta U_f + W_{elect.}$$

Reviewing the equation, term by term:

- ΔU_W = energy rise in the water bath $= M_W \, C_{VW} \, (T_2 - T_1)$
- ΔU_B = energy rise in the combustion bomb $= M_B \, C_{VB} \, (T_2 - T_1)$
- Q_B = heat transfer out of the bomb. Q_B is approximately proportional to the temperature rise, and so the quantity $\left(\frac{Q_B}{(T_2 - T_1)}\right)$ is approximately constant.
- $-\Delta U_S = -(U_{Products} - U_{Reactants})_S = (U_{react.} - U_{Prod.})_S$ = total "energy release" from the test sample = $M_S e_S$; where = e_S "Calorific Value" per unit mass of the test sample
- $-\Delta U_f = (U_{react.} - U_{Prod.})_f = L_f \left(\frac{(U_{React.} - U_{Prod.})_f}{L_f}\right) = L_f e_f$ where, e_f is the "calorific value" per unit length of the fuse material, and L_f is the actual length of the fuse consumed.
- $W_{elect.}$ = electrical work necessary to ignite the fuse, and is proportional to the length of fuse consumed = $L_f w_f$

Substituting the above back into the energy equation:

$$\underbrace{\left[\left(MC_V\right)_w + \left(MC_V\right)_B + \frac{Q_B}{\left(T_2 - T_1\right)}\right]}_{Cth_{cal}} \underbrace{\left(T_2 - T_1\right)}_{\Delta T} = M_S e_S + L_f \underbrace{\left[e_f + w_f\right]}_{e_{equiv\,f}}$$

The first term on the left, in brackets, is composed of terms that are approximately constant and together represent the "thermal capacitance" of the calorimeter. The term in brackets on the right represents the "energy equivalent per length of fuse material" (9.627J/cm).

The 1st Law can then be written as:

$$(Cth_{Cal})(\Delta T) = M_S e_S + L_f e_{equiv_f}$$

Solving for the thermal capacitance of the calorimeter:

$$Cth_{Cal} = \frac{M_S e_S + L_f e_{equiv_f}}{\Delta T}$$

Following replicate calibration tests with a reference benzoic acid test sample, it was found that:

Cth_{Cal} = 9.159 KJ/°C.

This value can now be used in conjunction with the temperature rise to account for the energy increase in the water bath and combustion bomb, as well as the heat transfer from the calorimeter when analyzing other test samples.

For other test samples:

$$e_S = \frac{(Cth_{Cal})(\Delta T) - L_f e_{equiv_f}}{M_S}$$

Time (seconds)	Temperature (^{O}C)	Comment
0		Start Timer
60 (+60)		
120 (+60)		
180 (+60)		
240 (+60)		
300 (+60)		← Start Logging Temperature
360 (+60)		Pre – Period
420 (+60)		Pre – Period
480 (+60)		Pre – Period
540 (+60)		Pre – Period
600 (Ignite Time) (+60)		Begin Rise Period
645 (+45)		
660 (+15)		
675 (+15)		
690 (+15)		
705 (+15)		
765 (+60)		
825 (+60)		
885 (+60)		
945 (+60)		
1005 (+60)		
1065 (+60)		
1125 (+60)		
1185 (+60)		
1245 (+60)		
1305 (+60)		
1365 (+60)		
1425 (+60)		
1485 (+60)		
1545 (+60)		

Length of fuse wire inserted (cm): ____

Length of fuse wire left after firing (cm): ______________________________________

Mass of fuel (grams): ___________________________________