

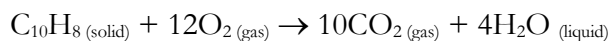
Experiment 2: Heats of Combustion

Adapted from *Experiments in Physical Chemistry* by Garland *et al.*, 8th edition, Experiment 6 (page 152)

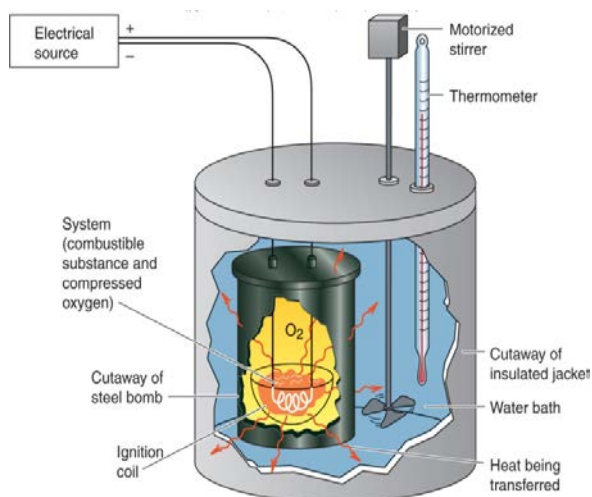
1 Introduction

In this experiment you will be examining the variation in the strengths of the bonds of reactant and product molecules. This will be defined from the heat released on combustion (the enthalpy of combustion or ΔH_{comb}).

You will carry this out using a *bomb Calorimeter* (see right). As the name suggests, this is a device that measures the thermal variations as a material is ignited. You will study the heat of combustion of Naphthalene, C_{10}H_8 , ignited in O_2 , i.e.



Everyday examples of this highly important process are in the oxidation of food (carbohydrates, fats, etc) and fuel (gasoline, etc). Oxidization of fuel is what powers out cars. Likewise, oxidation is how our bodies derive energy from food. Note: Bomb calorimeters are used extensively in the food industry (ever wondered how they define the amount of calories listed on the back of any package containing food?).



2 Theory

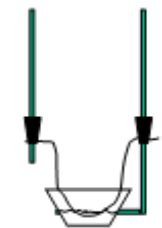
See the theory section of Experiment 6 in the text (p. 152) and the *Principles of Calorimetry* section (pp. 145–151) for a detailed discussion of the theory behind this experiment. You will need to read this section in order to understand how to determine heat of combustions.

3 Experimental procedure

The bomb calorimeter you will be using is very similar to the one described in the text. A schematic of the cross section is shown in Figure 1 on page 153. The procedure you will be carrying out is also essentially the same as described in the text. Additional information on the procedure is given below. You will be carrying out two runs on Naphthalene and one on Benzoic acid. Benzoic acid is required to derive the heat capacity of the bomb calorimeter.

3.1 Loading the sample and fuse wire

- Remove the pan from the bomb (this is supported on one of the electrodes attached to the cap). A cross section of the bomb is shown in Figure 2 on page 154. You may need to unscrew the cap of the bomb to access the pan.
- Weigh and Tare the pan, and then place either - A pellet of Benzoic acid (do this first) or - 0.5g of powdered naphthalene into the pan (you don't need to make a pellet of this) and record their respective weights.
- Place the pan with sample onto the supporting ring attached to one of the electrodes.



- d. Cut a 10 cm length of the supplied fuse wire and weigh this.
- e. Attach the fuse wire to the two electrodes attached to the cap. This should be done in a fashion shown in the figure to the right. The pellet should be over the pan, and the wire should touch only the terminals. The terminals have small holes into which you will thread the wire located under the metal sleeves. Slide the metal sleeve up to access this holes and then slide then down to lock the wire into place. The wire should form a loop, which should come in contact with the sample surface but not the pan.
- h. Set the bomb in the pail. Then set the pail in the calorimeter making sure that it does not touch the inner wall of the jacket.
- i. Make the electrical connection to the top of the bomb (two black wires).
- j. Fill a 2 L volumetric flask with water at 25°C. Pour the water from the flask carefully into the pail. Ensure the bomb is submerged in water, but don't over fill the pale.
- k. Close the system up and ensure the stirrer is able to move freely.
- l. Attach the belt over the stirrer and motor pulleys.
- m. Start the motor (turn small back knob clockwise). You may need to push start the stirrer.

3.2 Preparing the bomb

- a. Once you have prepared the sample and fuse wire, place ~1 mL of water into the bomb, then carefully screw the cap back onto the bomb (hand tight)
- b. Attach the brass fitting at the end of the gas line from the O₂ bottle to the gas inlet on top of the cap of the bomb. Simply press this on.
- c. Ensure the knurled fitting on the top of the cap is turned fully clockwise (this is the bomb gas release valve).
- d. Open the valve on top of the O₂ gas bottle a quarter of a turn (the smaller gauge attached to the O₂ bottle should read ~2000 psi)
- e. Ensure that the pressure release valve on the side of the regulator is closed.
- f. Slowly open the black knob on the front of the regulator. You will hear a hissing sound. Continue opening it until you see a pressure of 25 atm and then close this valve. If you don't see any pressure increase, the pressure relief valve on the side of the regulator or on the top of the bomb is most likely in the open position. Release the pressure to flush most of the atmospheric nitrogen originally present in the bomb. Refill the bomb to 25 atm.
- g. Close the main valve on the oxygen tank and flip the switch on the regulator to release the pressure in the tubing. Remove the brass fitting from the cap and insert the bomb into the pale.

3.3 Making the run

- a. Begin time–temperature readings, reading the thermometer and time once every 30 seconds. The temperature should change at a very slow rate. After this steady rate has persisted for at least 5 minutes ignite the bomb.
- b. Do not interrupt the time temperature readings. Depress the button on the ignition box to combust the sample.
- c. Continue recording the temperature and time every 30 seconds. The temperature will rise and after a few minutes the pail temperature should again show a slow, steady rate of change. Continue readings until the time since ignition has been at least four times the period required for attainment of this steady rate.
- d. When readings are completed turn all switches off.
- e. Disassemble the apparatus, release the bomb pressure, and open the bomb.
- f. Remove and weigh any unburned iron wire. Subtract the weight of unburned iron wire from the initial iron wire weight to obtain the net weight of iron burned. If the inside of the bomb is coated with soot, the amount of oxygen present at the time of ignition was presumably insufficient to give complete combustion and the run should be discarded.
- g. Wipe dry all bomb parts.

4 Calculations and Discussion

- Perform the calculations for experiment *Heats of Combustion* on p. 157 of the text.
- Answer the questions in the *Discussion* section on p. 157 of the text.
- In experiment 9 a *Differential Scanning Calorimetry (DSC)* is used to obtain enthalpy. If you already did experiment 9 discuss how the enthalpy values derived using *bomb calorimetry* and *DSC* differ?