

Experimental procedure: A summary of the procedure follows below: 1. Weigh approximately 1 g of sample on a rough balance and make a pellet using a pellet maker. (How to use the pellet maker will be demonstrated to you). 2. Determine masses of clean sample cup and of sample pellet. The pellet mass must be less than 1.10 g, otherwise it might produce too much energy and damage the bomb. (Use the analytical balance for these weighing's). Bomb Calorimeter 3. Tie 10 cm of fuse wire between the electrodes (see diagram). 4. Using the large solution balance, put 2000.0 ± 0.5 g of distilled water and ice in the calorimeter bucket. The ice is used to adjust the water temperature to about 1.5°C below Room Temperature. You can calculate the amount of ice needed by using $\Delta H^\circ_{\text{fus}} = 80 \text{ Cal/g}$ for ice. Use a squeeze bottle to add the final small amount of water to the bucket. Position the bucket inside the insulating shell. 5. Put 1.0 ml of distilled water inside the bomb. Screw on the top cover for the bomb. Connect the Oxygen adapter hose, and slowly add Oxygen gas until the pressure inside the bomb is about 25 atm. Watch the pressure gauge to check for leaks. Keep the oxygen tank shut-off, except when you are pressurizing the bomb. Release the pressure in the line and remove the bomb. Keep the bomb upright, so that the sample does not fall out of the sample cup. Using the special tongs to hold the bomb, insert it into the bucket of water, and connect the two electrical leads. Check to see that there is not a continuous stream of gas bubbling from the bomb, indicating a leak. (DANGER). 6. Put on the lid for the calorimeter shell, including the thermometer, and start the stirrer. Wait about 5 minutes for equilibration, then start to keep your temperature-time data. Using a magnifier, record the water temperature at intervals to the nearest 0.002°C . Elapsed time in seconds; 0, 100, 200, 300, 400 (Firebomb by pushing ignition button) 420, 460, 480, 500, 520, 540, 560, 600, 700, 800, 900. (The temperature rise will be very fast, take the readings without magnifier). CAUTION: (DO NOT STAND OVER BOMB. IF THERE WAS AN EXPLOSION IT WOULD PROBABLY SHOOT UPWARD). 7. Turn-off the stirrer, and remove the cover (CAREFUL, do not break the thermometer). Slowly release excess gas from the bomb. Open the bomb and inspect to see that it was a clean combustion; there should be no black, sooty residue left on the walls. Wash all interior bomb surfaces with distilled water, collecting the washings in a beaker. Titrate the washings with standard Na_2CO_3 solution using methyl orange indicator, to determine how much HNO_3 was formed. Measure the length of fuse wire left unburned. Return to step 1, and repeat the experiment with unknown sample pellet, that is NAPHTHALENE. In careful work, we would do at least three runs with the benzoic acid, and three runs for each subsequent sample, to give us a measurement of our precision. However, this would take too long, so we will only do one run for each