Parr 1241 Oxygen Bomb Calorimeter

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If you encounter problems while running the instrument, please submit an ***Instrument Incident Report*** *with a description of your problem. Please report urgent issues to Al Fischer, NS 209, dfischer@wcu.edu, x2695!*

Preparing the Bomb

Note: This procedure works for benzoic acid tablets and ethylene glycol. Other samples may require slightly different preparation or bomb conditions. Please consult with the Instrumentation Specialist and/or your faculty advisor before making adjustments.

Remove the bomb

1. Raise the post on the top front of the instrument by pulling the round black knob and lifting. Stop when it clicks into place.
2. Ensure the stirrer has raised with the post and then rotate the lever on the back of the caolorimeter's lid.
3. Turn the lid counter-clockwise to expose the bomb, disconnect the wire leads from the bomb, and remove the bomb from the pail.

Always assume the bomb is pressurized and always wear impact goggles when handling it.

1. *Slowly* crack open the pressure relief valve on the top of the bomb by turning the knurled metal knob counter-clockwise and wait until the pressure has been released.
2. Unscrew the metal ring that holds the lid on and remove the lid from the bomb. Place the lid in the stand holder for sample loading.

Load a sample

1. Inspect the quality of the o-ring on the bomb lid. If it is dirty or dusty you may clean it with a Kimwipe; if it is damaged, please file an Instrument Incident Report and wait for it to be repaired (i.e. do not use the calorimeter).
2. Cut approximately 10 cm of fuse wire and find its mass.
3. Weigh the sample cup and the sample cup + sample to determine the mass of the sample.
4. Place the sample cup (with sample) into the holder on the lid.
5. Attach the fuse wire to the two leads on the lid. Attach the wire so that it is ~ 1mm above your sample *but not touching the sample or any other part of the bomb.*
6. Reassemble the bomb *gently* so as not to disturb your sample. The ring that holds the lid on should be "hand-tight".

Charge the bomb

1. Close the release valve on the bomb.
2. Place the brass connection on the end of the oxygen hose over the inlet valve body. You may use a drop of water as a lubricant if necessary.
3. Open the main valve for the gas tank and ensure there is oxygen in the tank.

4. *Slowly and gently* crack open the knob on the front of the regulator and watch for the pressure in the bomb to increase on the large pressure gauge (you only need to turn a fraction of a turn).
5. Turn off the flow when the pressure reaches 30 atm (or the pressure required by your method).
6. *Slowly and gently* open the release valve on the lid. You should hear hissing and there should be oxygen coming out of the valve. You should *NOT* be able to smell your sample – if you can you are releasing the pressure too quickly. It should take approximately 1 minute for the bomb to depressurize.
7. Recharge the bomb to 30 atm (or the pressure require by your method) as above.

Load the calorimeter

1. Ensure the pail is properly seated on the 3 feet inside the calorimeter.
2. *Gently* place the bomb in the pail so as not to disturb your sample. Make sure it fits over the round boss inside the pail.
3. Attach the electrical connections to the lid.
4. Fill the pail with 2.000 L of de-ionized water. Be sure not to splash any water or get any drops on the sides of the pail.
5. Swing the calorimeter cover shut, lower the alignment rod and stirrer, and rotate the lever to lower the lid.
6. Insert the temperature probe through the port in the lid and begin collecting data with your computer.

Collect data

1. Turn the instrument on by using the **On/Off** switch in the middle of the control panel. Listen to make sure the stirrer is not hitting the pail or the bomb.
2. Let the temperature stabilize for several minutes.
3. Press and hold the **Ignition** button until the light goes off. This usually takes less than a second; if it takes more than 5 seconds release the button and notify the Instrumentation Specialist.
4. Continue to collect data until for at least 5-10 more minutes; you should see a temperature rise within 30 seconds.

Shutdown and cleanup

1. Turn the switch to **Off** to stop the stirrer.
2. Remove the temperature probe, raise the stirrer and alignment rod, move the lid to the side.
3. Disconnect the electrical connections and remove the bomb. Take it to the hood.
4. Dry the bomb completely with a paper towel, and try to wick as much water as possible out of the release valve.
5. Release the pressure in the bomb by turning the knurled knob on the release valve counter-clockwise; this should take approximately 1 minute.
6. Disassemble the bomb; remove and weigh any remaining fuse wire, and look for signs of incomplete combustion of the sample.
7. Rinse and completely dry the inside of the bomb.
8. Empty and completely dry the pail.
9. If dry, re-assemble all components and place them back in the calorimeter.

Precautions

- Always wear safety goggles.

- Do not overcharge the bomb: The sample should never release more than 8000 calories when burned in oxygen (always start with < 0.7 g of sample if unsure, and *never* use more than 1.5 g of combustible material).
- Do not overcharge the bomb with too much oxygen (use no more than 30 atm for most samples and 40 atm is absolute maximum).
- Do not fire the bomb if it is leaking (look for O2 gas bubbles escaping from it).
- Always ensure the bomb is fully submerged in water during firing.

General Notes

- Label ALL samples/containers with:
  1. What the sample **composition** is (e.g. 0.5 M Nitric Acid in Water). Be as descriptive as possible!
  2. The **date** the sample was created.
  3. Your **name**.
- Any unlabelled samples will be disposed of immediately.
- Any samples left in an instrumentation area or shared fume hood will be disposed of. If you need lab or sample storage space please contact Al Fischer or Wes Bintz.