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		2 November 2015	003
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Standard Operation Procedure – Calorimetric Tests

1. Scope and Application

- Heats of combustion as determined in an oxygen bomb calorimeter are 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 or other standardising material whose calorific value is known.
- These measurements are obtained by burning a representative sample in a high pressure oxygen atmosphere within 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 heat combustion of the sample is then calculated by multiplying the temperature rise in the calorimeter by the previously determined energy equivalent or heat capacity determined from a standardising material.

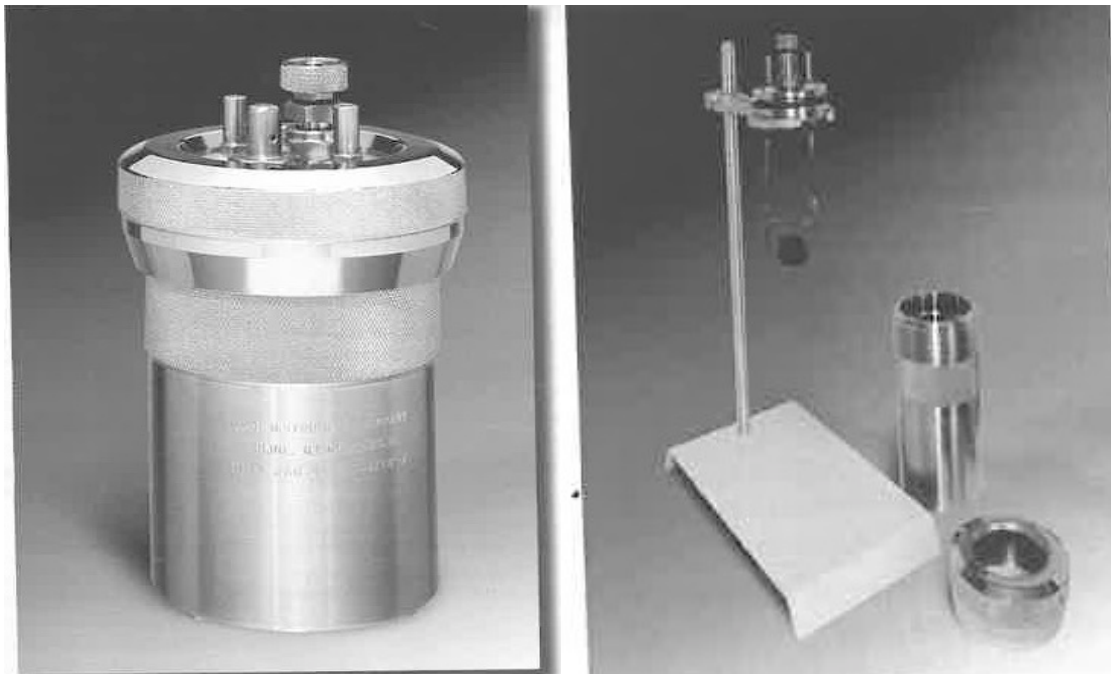
2. Summary

- Calorimetry is the science of measuring quantities of heat, as distinct from temperature. The instruments used for such measurements are known as calorimeters. The oxygen bomb calorimeters, which are the standard instruments for measuring calorific values of liquid and solid combustible samples.
- The calorific value (heat of combustion) 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.

3. Apparatus



Parr 6200 Oxygen Bomb Calorimeter



1180P Oxygen Combustion Bomb

Bomb head support stand

4. Reagents

- Benzoic acid tablets for standardisation (should always be tested in pellet form).
- Ethylene glycol– combustion aid. Heat combustion= 4033cal/g. Mass (0.2160g= 4drops)

5. Sampling

- Dry sample for 24 hours at 105°C in a crucible.
- Grind dried sample to a powder form.
- Weigh out a gram of sample into the sample vial (0.5- 0.7g for faecal matter).

- The bomb should never be charged with a sample, which will release more than 8000 calories when burned in oxygen, and the initial oxygen pressure should never exceed 40 atmospheres (590 psi).
- Never charge the bomb with more than 1g of combustible material.

6. Safety Precautions

- Do not use too much sample.
- The standard bomb 1108 cannot be expected to withstand the effects of combustible charges which liberate more than 8000 calories frequently.
- This generally limits the total weight of combustible material (sample plus combustion aid) to not more than 1.1gram.
- Do not charge the bomb with more oxygen than is necessary to obtain complete combustion.
- It is best to use the lowest gas pressure that will give complete combustion.
- Lower gas pressure permit higher gas temperatures and greater turbulence, both of which help to secure better combustion. The range is 20- 35 atmospheres. Regulator set at 400psi (3000kpa).
- Keep all parts of the bomb-especially the O-rings, insulated electrode assemblies and valves- in good repair at all times.
- Do not fire the bomb if gas bubbles are leaking from the bomb when it is submerged in water.
- Stand back from the calorimeter for at least 15 seconds after firing and keep clear of the top of the calorimeter.
- If the bomb should explode, it is likely that the force of the explosion will be directly upward.
- SPIKE SETTING
Spike control/ Spiking on/ Spike fixed/ 0.2160g

7. Procedure

1. Open oxygen gas cylinder, flow rate is already set to (400psi-3000kpa), do not alter.
2. Check that distilled water chamber is filled to the mark.
3. Turn on the calorimeter and activate the pump and heater.
4. Allow at least 20 minutes for the calorimeter to warm up and the jacket temperature to stabilise at 29°C.
5. The calorimeter is ready to begin testing.
6. The START key will be available at this time.
7. Fill the calorimeter bucket with 2L of distilled water.
8. Set the bucket in the calorimeter.
9. **SAMPLE PREP:**
 - 0.5g 24 hours at 105°C dried blended sample into capsule.
 - Tie cotton tread which is used as a fuse to ignite the sample onto heat wire in the bomb.
 - Cotton tread should not be immersed in the sample.
 - Slightly above the sample.

- For wet samples (80% moisture content), add 4 drops of ethylene glycol to the sample before combustion.
 - When contact is made through the heating wire, the tread will ignite, drop into the sample cup and ignite the sample.
10. Care must be taken when moving the bomb head from the support stand to the bomb cylinder.
 11. Moisten the base of the bomb with a few drops of water.
 12. Check the sealing ring to be sure it is in good condition and moisten it with a bit of water so it will slide freely into the cylinder and push it as far down as it can go.
 13. Close the bomb and pressurize with oxygen.
 14. The pressure connection to the bomb is made with a slip connector on the oxygen hose.
 15. Slide the connector onto the inlet valve body and push it as far as it can go.
 16. Press the O₂ FILL button and step back until bomb is filled. Takes 1 minute.
 17. Remove the gas connection and attach the lifting handle to the two holes in the side of the screw cap and lower the bomb into the water partially.
 18. Press the banana plugs onto the two ignition wires firmly into the terminal sockets on the bomb head before the head is completely immersed in water.
 19. **Note:** If bubbles continue to rise from the bomb after the air in the screw cap has escaped the test must be stopped.
 20. Do not fire the bomb until the leak has been corrected.
 21. Close the calorimeter cover. This lowers the stirrer and thermistor probe into the bucket.
 22. Make sure that the bucket thermistor does not touch the bucket or the bomb when the lid is lowered.
 23. Select determination under **OPERATING MODE**.
 24. After pressing the START key, the calorimeter will prompt the operator for BOMB ID number, sample ID number, sample weight and spike weight.
 25. The calorimeter will now take over and conduct the test.
 26. During the time it is establishing the initial equilibrium, the status bar will display PREPERIOD.
 27. Just before it fires the bomb, at about 12 minutes, it will sound a series of short beeps to warn the user to move away from the calorimeter.
 28. Once the bomb has been fired the status bar will display POSTPERIOD.
 29. Read of calorific value from screen.
 30. Remove the bomb from the chamber after 3 minutes and depressurise bomb by opening the valve knob slowly. After all the pressure has been released, unscrew the cap and lift the head straight out to avoid sticking.
 31. Remove the chamber containing the ash.
 32. Wipe the inside of the bomb clean and proceed with next sample.
 33. Maintenance must be carried out after every 30 tests. Replace O-ring and wire.
 34. Clean the bomb and soak in citric acid solution overnight or whenever the bomb is dirty.
 35. Aqua regia can also be used to clean the bomb_ remove the wire if it is being used.

8. Calibration

- 1 gram benzoic acid tablets are used for calibration.
- Always use the tablets in a pellet form.
- The heat capacity should be +/- 26 MJ/kg.

9. Results

<u>Sample name</u>	<u>Sample No.</u>	<u>Mass (g)</u>	<u>Gross Heat (MJ/Kg)</u>	<u>EE Value</u>
<u>1-1</u>	1	0.7568	4.1351	2345.4288
	2	0.7532	4.2861	2345.4288
	3	0.7548	4.2236	2345.4288
Average			4.2149	

APPROVAL OF STANDARD OPERATING PROCEDURE

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