

**STANDARD OPERATION PROCEDURE**  
**Faculty of Biosciences, NMBU**

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**Method name: Calorimetry (energy determination)**

BIOVIT-nr: Arb1015

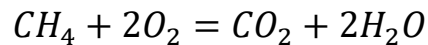
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**1. Introduction**

Calorimetry is defined as the measurement of released or absorbed heat and involves the experimental measurement of the released or absorbed energy from a reactive system, such as a combustion or chemical reaction.

At BIOIYT, calorimetry is mainly used to determine negotiable energy in feed, intestine and faeces samples, but energy in other materials and liquids can also be determined.

The samples are completely incinerated in a closed system with excess of oxygen (bomb calorimeter). During combustion, the chemical bonds between the atoms change and when all the material has been burned, all the organic molecules will be converted to mainly CO<sub>2</sub> and H<sub>2</sub>O, cf. combustion of methane in excess oxygen.



There will also be an inorganic residue (ash) that is not incinerated.

**2. Reagents**

- Oxygen gas, O<sub>2</sub>- most affordable quality
- Nitrogen gas, N<sub>2</sub> - most affordable quality

**3. Risk assessment**

The bomb calorimeter must not be opened while there is pressure in the combustion chamber.

**4. Equipment**

- PARR 6400 Bomb Calorimeter
- Cotton thread (DMC, F75579-Paris Cedex 12, 50g / 520m)
- Weight (0.0001 g)
- Pellet press
- Metal wire (PARR 840DD2)
- Sample holders (small metal containers (crucibles))

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## 5. Sample material

The sample amount should not normally exceed a dry weight of 1 g. For fat-rich samples, the amount can be reduced to approximately 0.5 grams.

The material must be dry and preferably compressed into a pellet (does NOT apply to fat-rich samples).

## 6. Special remarks

When analyzing fat-rich samples, the sample should not be pelleted. When pelleting such samples, the fat will be forced out of the sample and one will experience too low analysis values. Fat-rich samples are placed directly in the crucible. Press down lightly with a glass rod before weighing.

## 7. Work procedure

### Calibration

The calibration is performed by burning 5-10 benzoic acid tablets and checking that the released energy is 26,453 MJ/kg (between 26,32-26,64 MJ/kg). Calibration of the instrument should be done at least twice per. year.

The calibration result is shown on the display.

The control: 1 benzoic acid and one control sample of chicken feed.

### Before starting

There are two tanks, one in plastic and one in steel, under the instrument. The plastic tank contains used water from the instrument and is emptied when it is approx. half full. The steel tank contains pure RO water. Check that there is enough washing water before starting the analysis. Before the steel tank, which is under pressure, can be opened, a valve must be opened.

1. Insert the plugs, two pcs.
2. Turn on the power, the switch is located on the back of the instrument. Wait about 1 min for update. NOTE! Pressure: Calorimeter operation, Press: HEATER and PUMP should be ON.
  - It takes about 15 minutes before the instrument is ready to analyze samples.
3. On the display you can see the temperatures to be achieved
  - The JACKET temperature should be 29-30 °C

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- BUCKET temperature 27-28 °C (initial analysis can start at 24-25 °C).
- 4. Open the valves for the oxygen and nitrogen gas (the gases give the correct pressure automatically).
- 5. Pellet the sample (DO NOT apply to high-fat samples) using the pellet press (blue).
- 6. Tare a metal crucible.
- 7. Place the sample in the crucible and note the weight.
- 8. Remove the suspension device from the bomb itself.
- 9. Make sure that the “hanging” device is dry (use paper for drying).
- 10. Insert the hanger into the stand next to the instrument.
- 11. Place the crucible, with the sample, in the hanger and cut to 10 cm of the cotton thread.
- 12. Tie the cotton thread around the metal thread in the device and make sure that the thread comes into good contact with the sample. Preferably place the thread under the pellet.
- 13. Moisten the O-ring in the bomb container: Place the device in the bomb container and turn it fixed to the LEFT.
- 14. Close the lid.
- 15. Press "START" and "ENTER".
- 16. Run data file space warning - Press Yes.
- 17. Rinse tank level warning - Press Yes.
- 18. Accept auto sample ID - Press Yes.
- 19. Enter bomb Id - Press enter.
- 20. Enter sample - weight - enter weight.
- 21. Press "ENTER" and the analysis is in progress.
- 22. When the analysis is completed, the instrument is washed automatically.
- 23. The lid is opened and the suspension device can be unscrewed from the bomb container (to the right).
- 24. Carefully remove the suspension device from the instrument to avoid as much spillage as possible (the crucible is full of water).

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25. Place the used crucible in the plastic box to the right of the instrument and dry the hanging device well with paper.
26. Repeat steps 5 to 25 when analyzing several samples.

**After use**

Turn off the gas, turn off the power switch and finally; pull out the plug.

**8. Calculation**

The result is calculated in MJ/kg.

**9. Troubleshooting**

"MISSFIRE" - appears when the thread is not properly in contact with the sample. Try again.

"LOW PRESSURE" - the bottom valve is in the wrong position or the oxygen pressure is too low.

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