APPLICATION NOTE | DDS CALORIMETERS

C1.5 ALTERNATIVE METHOD FOR CALORIFIC MEASUREMENT OF FOOD SAMPLE – DRIED FISH

INTRODUCTION

This Application Note aims to determine the most efficient method to successfully burn a sample, that has previously been difficult to burn successfully.

AIM

- We wanted to confirm that this sample was possible to be successfully burnt in a calorimeter and that a sufficient temperature rise was achieved. We also wanted to confirm, that it was able to burn a sample mass of 0.2g or greater, without damaging the lid assembly or crucible inside the Vessel.
- To confirm that the sample was not dangerous or highly flammable.

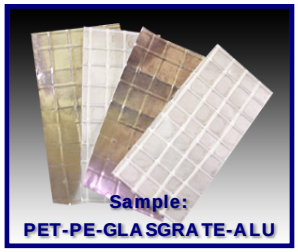
SAMPLE PREPARATION
GENERAL INFORMATION

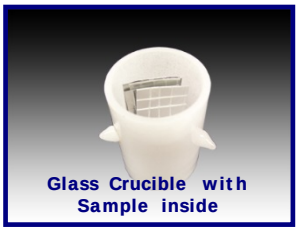
- A calorimeter is used to determine the calorific value of any substance that can be ignited. The substance must be in liquid or solid form.
- The sample to be measured must be a representative sample and homogeneous.
- The sample should either be measured as whole or should be ground into a powder, mixed well and then pressed into a tablet form using a pellet press. Pressing the sample into a tablet prevents splattering when the sample burns. Splattering is when un-burnt sample is thrown out of the crucible during the combustion process, thus causing inaccurate results. However, some samples once they have been ground into a powder will not easily press into tablets using a pellet press, because the fibres will not adhere to each other irrespective of the pressure exerted during the pelleting process. An alternative method to ignite the sample without it splattering during the burning process is to place the powder (ground sample) inside a gelatine capsule. The capsule ignites easily thus causing the sample to ignite while confining the sample during the ignition phase. The calorific value of each batch of gelatine capsules must be determined beforehand. This value along with the mass of the capsule will then need to be used in the spike application of the calorimeter.
- With polymers and composites it is difficult to grind the material into a powder form and to press the sample into a tablet or fit it into a gelatine capsule. Therefore small pieces may need to be cut to fit into the crucible.
- All samples should have no moisture present before analysing. Freeze-drying the sample can remove the moisture. With polymers and composites no moisture is present.

TEST SPECIFIC INFORMATION

- We cut four pieces of 3cm x 1.5cm strips. We noted that the sample had two colour sides – one side was shiny silver and the other side was matt silver.

- We made use of a special glass crucible, instead of the regular stainless steel crucible.
- We used a weight of 0.2669g, but a mass of 0.3g would also be fine.





SPIKING (FOR INFORMATION PURPOSES ONLY)

If a sample does not ignite easily or not at all, then the spiking method of ignition can be used. In this method a benzoic acid tablet is added to the crucible with the sample. The benzoic acid burns easily and ignites the sample; the energy of the benzoic acid is removed from the calculation of the calorific value. Many polymer and composite sample analysis are performed using the spiking method. However, in this specific analysis, no spiking was necessary.

ANALYSIS

GENERAL INFORMATION

Once the sample has been prepared the determination can be carried out in the normal method.

When using firing cotton, ensure that the firing cotton touches the sample. If you are using tablets, lay the cotton on the bottom of the crucible and then move the tablet on top of the cotton. During the filling process do not knock the vessel, to ensure that the tablet does not move off the cotton.

When substances are being analysed for the first time, always check after the determination for any residue on the walls of the vessel and check that the entire sample has been burnt.

After a determination, clean the inside of the vessel and the crucible before starting the next determination.

TEST SPECIFIC INFORMATION

For this particular analysis, we filled the vessel to the standard 3000KPa.

We ran one firing, in order to ensure that the sample would be successfully burned without any damage to the inside of the Vessel.

RESULTS FOR SAMPLE: PET-PE-GLASGRATE-ALU

- The sample burned completely and did not damage the Centre Electrode and Deflector Plate. This indicated that a mild burn took place (in other words, there was no explosion).
- There was a very small amount of some acidic residue, which was easily cleaned out.

Firing Temp.: 29.25°C
Ambient Temp.:21.00°C

The resultant temperature rise was an acceptable 7°C.

CONCLUSION

Using the correct procedure and a glass crucible we were able to confirm that this sample is able to be successfully burned in a calorimeter, without any damage to the Vessel or other parts.