

## Ovens, Furnaces & Heating Equipment

### Factors to consider when choosing the correct furnace for your ashing requirements

Robert Prior, Carbolite Gero Ltd, robert.prior@carbolite-gero.com

#### Overview of the ashing process

A knowledge of the characteristics of various ashing procedures and types of equipment available is extremely useful for any practitioner to obtain accurate results.

The two most common types of ashing are dry ashing and wet ashing. In recent years microwave technology has been introduced in both wet and dry ashing methodologies to reduce the length of time the process takes.

Wet ashing is a processing which organic substances are oxidised with an oxidising agent(s) and / or the use of a combination of acids. This process is often selected in preference to dry ashing if the individual is looking to run specific elemental analysis on the sample.

This article will focus primarily on Dry Ashing.

#### Dry Ashing

This is best defined as the process of removing all the organic material from a sample through heating in air. Many dry food samples such as dry vegetables and grains need no preparation before dry ashing occurs. However, some materials (such as fresh vegetables) will need to be dried prior to ashing and high-fat products such as meat and dairy may need to have their fat content removed and then dried before the ashing process can take place.

The proportional mass of residual ash may be the objective of the test. Alternatively, it may be an intermediate step in the preparation of a sample for other purposes such as characterisation of the inorganic components through X-Ray Fluorescence, mass spectroscopy or for tests such as ash fusibility.

The ashing process is frequently used in the testing of foods, plastics, and hydrocarbon materials such as coal. Ashing tests may be defined within standard test methods, typical examples of which are ISO BS ISO 2171:2010 (Cereal), BS ISO 247:2006 (Rubber) and BS ISO 1171:2010 (Coal & Coke).

When ashing, achieving complete combustion of the sample is of vital importance. This can best be accomplished with a purpose-built ashing furnace. Of course, ashing some materials can and will create a large amount of smoke so in certain cases specialised furnaces (which are designed to provide more airflow or process the smoke further an afterburner or catalytic oxidiser) may be required or advisable.

In addition, where alumina or silica dust could contaminate the results or where the ashing process will result in the production of corrosive vapours, a fused quartz chamber ashing furnace may be recommended as the correct solution.

#### Choosing the correct furnace for your requirements

The most important decision for anyone looking to use a furnace to ash material is whether the furnace should be dedicated to the task for materials analysis to a standard methodology or whether the furnace will also be used for general heat treatment applications in addition to ashing.

Other questions will include whether the process must conform to a given test method; whether the burn-off will generate aggressive fumes that could cause furnace damage or be hazardous; whether the airflow will be sufficient; how large the samples will be; how intensive the workload might be and finally, whether contamination of the ash with silica or alumina would be detrimental.

There is no single solution that meets all requirements and therefore the questions above need to be answered to ensure you select the correct ashing furnace solution within your available budget and normal workflow.

#### Ashing food in a laboratory setting

A good ashing example in a laboratory setting might be for those wishing to determine the mineral nutrient content (Ca, Mg, Ne, K, Fe, Zn, Cu, and Mn)

from composite food groups such as dairy, meats, cereals & rice and vegetables. The complete procedure requires the technician to prepare an ash by using heat and nitric acid to decompose the organic matter and subsequently dissolving the inorganic residue in an appropriate volume of dilute hydrochloric acid.

The apparatus used will typically include a drying oven, muffle/chamber furnace, hot plate and silica crucibles or quartz beakers holding between 45 and 80ml and a watch glass of appropriate diameter. The reagents used will typically be deionised water (H<sub>2</sub>O), reagent grade nitric acid (HNO<sub>3</sub>), reagent grade concentrated hydrochloric acid and diluted HCl.

Usually, the process begins with weighing an amount of the sample (typically around 5g of material) into a selected crucible. Crucibles can be made of various materials including steel, porcelain or quartz each with their own advantages and disadvantages depending on the temperature required for ashing or any potential chemical reactions or contamination possibilities. The sample(s) may then be dried in an oven at around 100°C for between 12 and 16 hours. Once a sample is dry the crucible and sample will be placed in an ashing furnace for a further period that could reach 16 hours reaching a temperature of 450°C or higher.

The crucible can then be removed from the furnace and allowed to cool before the technician moistens the ash with H<sub>2</sub>O and then adds between 2 to 4ml of HNO<sub>3</sub> which should be enough to cover it. The crucible is then covered with a watch glass and refluxed on a hot plate for around 60 minutes before the watch glass is removed. If necessary, the technician may return the crucible to the hot plate to gently evaporate and remaining acid as failure to do this can lead to the ignition of the organic residue.

The sample will then be returned to the ashing furnace for a further hour at a temperature of around 375°C (as at higher temperatures potassium nitrate decomposes violently). This step can be elongated if necessary until a white ash remains. Around 2 - 5ml of diluted HCl can then be added to the resulting white ash which is then dissolved by gently boiling the solution before it is cooled and H<sub>2</sub>O added and finally the elements are determined using atomic absorption spectrophotometry (AAS).

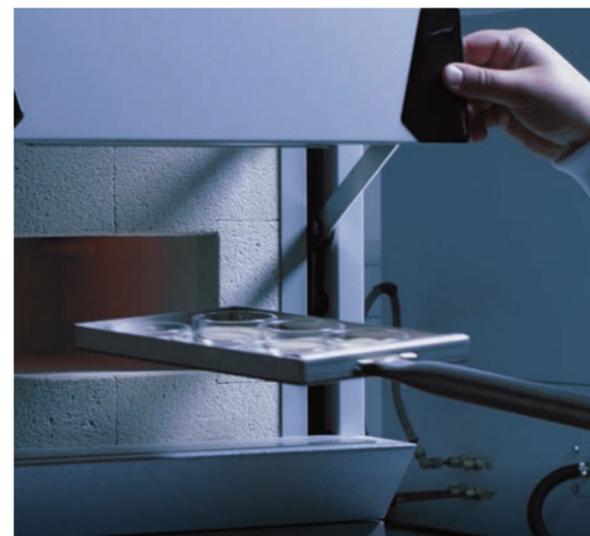


Figure 1: Crucible containing weighed sample.

## Dry ashing with an integrated balance

For many the ability to perform Thermogravimetric Analysis (TGA) is a requirement and furnaces with an integral balance are available.

Weight changes to the sample are monitored during the dry ashing process and any ashing furnace mounted on an integrated balance can be used for 'Loss On Ignition' applications to determine the organic matter content in such materials as sediment, sludge, soil and waste. Inorganic materials such as cement, lime, calcinated bauxite and refractories can also be measured.

The ashing furnace with integrated balance should be capable of reaching up to 1100°C. It would also be extremely beneficial if the heating elements were protected by silicon carbide tiles. A dry ashing furnace with a chamber capacity of between

15 & 20 litres manufactured to hold a standard charge of up to 3Kg (with a high percentage [95%+] of inorganic content) could be perfect especially if the balance had a resolution of 0.01g.



The integrated balance in a dedicated ashing furnace solution should run independently of the furnace control system. The furnace itself can then be supplied with a range of sophisticated digital controllers, programmers and loggers depending on specific user requirements. Of course, specialist furnaces with an integrated balance will inevitably be designed and manufactured to meet exacting standards and to the highest specification to ensure longevity and a substantial return on the initial investment.

Figure 2: Furnace with an integral balance.

## Soil analysis example in ashing furnace with integrated balance

In a recently conducted experiment a UK manufactured ashing furnace fitted with a Eurotherm nanodac controller & integrated balance was used to measure and log small weight differences (mg) during the ashing of a 5.02g organic soil sample.

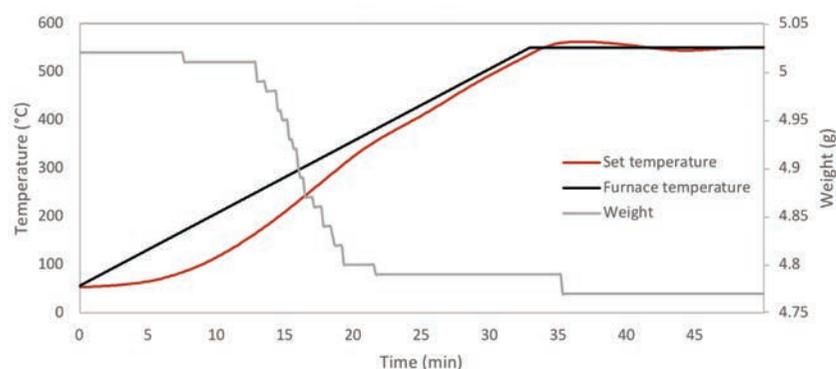


Figure 3: Continuous logging of the set temperature, the measured furnace temperature and the weight of the sample.

The furnace had a target temperature of 550°C with a ramp rate of 15 K/min. Once the target temperature was achieved it was held for 20 minutes and throughout there was a continuous logging of the set temperature, the measured furnace temperature and the weight of the sample. The process data can be seen in Figure 3 where the logger has recorded the elapsed time, the furnace temperature and the weight of the soil sample before, during and after the dry ashing occurred.

## Ashing furnace with afterburner / catalytic converter

Finally, for anyone with a requirement to Ash larger samples or biomass samples which are likely there are ashing furnaces available with a larger combustion chamber that are fitted with a fume exhaust passing through a high temperature afterburner / catalytic converter. This addition removes as much as 40g of carbon-based fumes and noxious gases per load ensuring operator safety at all times.

Table 1

TITLE	BS STANDARD	TEM °C	TIME	SAMPLE SIZE
ANIMAL FEED STUFFS	BS ISO 5984:2002	550°C	3 to 5 hours	5g
MEAT & MEAT PRODUCTS	BS 4401-1:1998	550°C to 600°C	Min 30 mins at 600°C	5g
FATS & FATTY OILS	BS ISO 6884:2008	550°C to 600°C	4 hours	
CONDENSED MILK	BS 1742-4:1991	525 ± 25°C	Several hours	5g
CEREALS & MILLED CEREAL PRODUCTS	BS ISO 2171:2010	900 ± 10°C	2 to 3 hours	5 to 6g
RUBBER: DETERMINATION OF ASH	BS ISO 247:2018	525 ± 25°C 950 ± 25°C	1 hour	1 to 5g
RUBBER COMPOUNDING INGREDIENTS: CARBON BLACK	BS ISO 1125:2015	550 ± 25°C	1 hour	2g
ALUMINIUM MANUFACTURE ELECTRODES	BS ISO 8005:2005	700 ± 10°C	16 hours	20g
DETERMINATION OF ASH OF TEXTILES	BS 6646:1985	700°C	-	2 to 10g
ANALYSIS & TESTING OF COAL & COKE DETERMINATION OF VOLATILE MATTER CONTENT	BS ISO 562:2010	900 ± 5°C	7 mins	1g
ANALYSIS & TESTING OF COAL & COKE DETERMINATION OF ASH	BS ISO 1171:2010	815 ± 10°C	2 to 2.5 hours	1 to 2g