

## **Determination of ash in feedstuffs**

Ash is defined as residues of different elements remaining after the burning of feedstuff sample at (600C□) in muffle furnace.

The planning of feedstuff in the muffle furnace will lead to burning of all organic matter of the feedstuff sample. The ash may contain some foreign elements of non-feed origin such as, stone, gravels, glass pieces, dust, debris, sand (Silica).

In determination process of ash, crucible are used instead of ordinary metal cans. This is due to the tolerance and durability of crucibles to high temperature of muffle furnace and for long time. In concentrates feed sample, ash percentage should not exceed 10% , otherwise the sample is considered as cheat.

Procedure of the experiment:

1. clean and labeled crucible is put in muffle furnace on (500-600C□) for an hour, Then, it is transferred into desiccator for cooling. The aim of this operation is to sterilize the crucible and to exclude any debris mixed with the sample.

2. The cooled crucible is weighed using electrical digital scale and record the weight. This is the empty crucible weight. Half gram (0.5gm) of the feedstuff sample intended for ash determination is weigh by the digital scale and this weigh is considered as ( weight before burning).

3. The crucible containing the sample is placed inside muffle furnace on (500-600C□) for three hours. The temperature of muffle furnace should be gradually elevated because such operation avoid the occurrence of ( spattering of sample) .

spattering of the sample is a process characterized by the ejection and expelling of the sample from the crucible. This case occurs when the temperature of the muffle furnace is abruptly elevated or when the sample is immediately inserted inside the hot muffle furnace.

4. After the termination of burning process, the crucible is taken and should be lift for a while to be cooled partially. The remaining ash is colored usually with white. Later, the sample is put on desiccator until being exactly cooled. It is weighed and recorded as " weight after burning". The crucible containing the ash is kept for determination of silica.

Note book: when ash percentage is elevated or exceed 15%, this sample is regarded as cheat sample.

Calculations:

1. weight of empty crucible.
2. weight of crucible with the sample (the weight before burning).
3. weight of sample =  
weight before burning – weight of empty crucible.
4. weight of the crucible with sample (the weight after burning).
5. Weight of ash = weight after burning – weight of empty crucible.

6. the percentage of ash = 
$$\frac{\text{weight of ash}}{\text{weight of sample}} * 100$$

7. the percentage of organic matter = Percentage of dry matter – percentage of ash. Or ) wt. before burning – wt. after burning

Example: -

Two grams of ground corn sample was put in muffle furnace at (500C□)for three hours. The weight became (0.1gm) after burning. Estimate the percentage of organic matter of the sample, percentage of dry matter of the sample is 95%

Solution:

$$\text{the percentage of ash} = \frac{\text{weight of ash}}{\text{weight of sample}} * 100$$

$$= \frac{0.1}{2} * 100 = 5\%$$

Percentage of organic matter =

Percentage of dry matter – percentage of ash.

$$95 - 5 = 90\%$$

### **Determination of Silica from Ash of feedstuffs**

Silica is defined as a collection of foreign mineral substances of non- feed origin. These substances are added deliberately for commercial cheat. Silica may involve sand, soft stones and dust. Present of silica within feedstuff sample may be due to bad storage. Determination of silica ratio may be carried out because of high percentage of Ash. Also, such determination may be an indicator for cheat of feedstuffs, hence, such indication may determine the possibility and feasibility these feedstuff for feeding farm animals. The height percentage of silica within feedstuffs may cause damage and injury to animal health because of irritation of mucous membranes lining the digestive system. Silica ratio can be determined in the ash of burning feedstuffs can be achieved by addition of hydrochloric acid (Hcl) (concentrated and diluted Hcl). The addition of diluted and concentrated (Hcl) is to separate minerals of feed origin from those present as foreign materials of the ash converting into chlorides of the elements as follows:

Notebook: when the percentage of silica exceeds 10%, the sample is considered as cheat.

Procedures of the experiment:

1. Empty crucible weight is recorded (as mentioned in previous laboratory).

2. Original sample weight is recorded (as mentioned in previous laboratory).
3. Careful addition of (1ml) of conc. Hcl is done to the crucible containing ash. It is better to put a metallic can down of the crucible. The crucible with its contents should be put in oven for twenty minutes until being dried from the acid.
4. Diluted Hcl (previously prepared by addition of three quarters of water to one quarter of acid) to ash sample in small amounts. The mixture should continuously be mixing using steering rod to homogenize the mixture and to break down the formed masses.
5. Washing and filtration processes are carried out using Buchner funnel in which the remaining matter is washed by distil water (preferably lukewarm distil water) to facilitate the operation process. This operation cause a change in pressure leading to draw the salts (chlorides) down wards.
6. The crucible should be put in muffle furnace on (500-600C) for twenty minutes. The crucible is drawn from the oven and weighed.

Calculations:-

1. Weight of empty crucible (as mentioned from previous laboratory).
2. Weight of original sample (as mentioned from previous laboratory).
3. The crucible put out from the muffle furnace and weighed (it is considered as weight of crucible after treatment and burning).
4. Weight of Silica = weight of crucible after treatment and burning- weight of empty crucible.

$$5. \text{ Percentage of Silica} = \frac{\text{weight of silica}}{\text{weight of sample}} * 100$$

Example:-

Weight of feedstuff sample is (1.6gm). after burning of the sample in muffle furnace the weight is (0.4gm). the remaining ash is treated with hydrochloric acid and is burning. The weight after burning is (0.2gm). estimate the percentage of ash and silica in the sample. Does the sample is cheat ?

Solution:

$$\text{Percentage of ash} = \frac{\text{weight of ash}}{\text{weight of sample}} * 100$$

$$= \frac{0.4}{1.6} * 100 = 25\%$$

$$\text{percentage of Silica} = \frac{\text{weight of silica}}{\text{weight of sample}} * 100$$

$$= \frac{0.2}{1.6} * 100 = 12.5 \%$$

**Because silica percentage exceeds 10%, so it is cheat .**

Preparation of ash extract of feedstuff samples:

This operation is carried out after the separation of silica and foreign matters from the ash of the feedstuff. As mentioned earlier, the remaining solution of the receiving flask after filtration process using the acid contain all mineral elements of dietary origin which are important in feeding of farm animals. The solution is diluted up to (100ml) by distil water using measuring flask after this process, the solution is ready for determination of mineral elements of the ash using chemical method. Atomic absorption and emission spectrophotometry are instruments used according the nature of each element intended to be determined.