



MICROWAVE ASHING PROCEDURE FOR DETERMINATION OF INORGANIC ASH IN FOOD SAMPLES

An alternative and rapid method for determination of mineral content in food samples

I INTRODUCTION

The ashing test on food samples is commonly used for evaluation of mineral content.

The minerals constituents include potassium, sodium, calcium and magnesium, which are present in larger amounts as well as smaller quantities of aluminum, iron, copper, manganese or zinc, arsenic, iodine, fluorine and other elements present in traces.

Determination of ash content is very important for several reasons. Mineral content in food play an important role from a physicochemical and nutritional point of view.

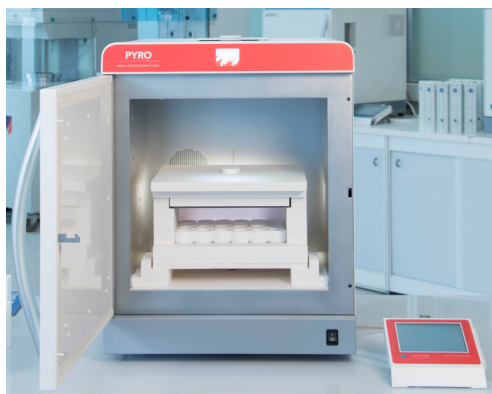
Ash content analysis is also an accepted index of refinement of foods, such as wheat flour or sugar production, since the ash test is a reliable indicator of efficiency of which the separation of bran and germ from the rest of the wheat kernel.

Traditional tools like electrical muffle furnaces, have their own set of limitations – long ashing time, long cooling, high power consumption and low aspiration efficiency.

Milestone PYRO system incorporates all of the benefits of a microwave system working with special fast heating/cooling crucibles while making the ashing application fast, easy, effective, and in complete safety.



| EXPERIMENTAL INSTRUMENT



Picture 1 - Milestone PYRO Microwave ashing system

The new Milestone PYRO is an advanced microwave muffle furnace, suitable for food ashing applications.

It is equipped with a full stainless-steel door and the cavity has a volume in excess of 70 liters, thus allowing the use of a large muffle which in turn enhances the sample throughput.

PYRO system is equipped with two 950 Watt magnetrons for a total of 1900 Watt making it the most powerful microwave muffle furnace system available in the market.

The system additionally employs a rotating diffuser that evenly distributes the microwaves throughout the cavity, assuring a uniform temperature improving the reproducibility.



Picture 2 Milestone PYRO High sample throughput muffle system

PYRO enables the analysts to perform the ashing test of a wide variety of samples.

A unique ceramic muffle allows microwave radiation to pass through and rapidly raise the temperature of a silicon carbide plate. Sample crucibles are placed on a large clean quartz plate and an airflow is induced by a built-in exhaust system.

Any type of crucible (metal, porcelain, quartz, etc.) can be used and for this test we have used the Milestone UltraFAST crucibles (Picture 2).



Picture 2 Milestone UltraFAST crucibles

The UltraFAST crucibles are made of an innovative material that, although chemically and thermally resistant, allows the samples to be surrounded by a constant airflow which accelerates the ashing process. Furthermore, just 30 seconds are enough for the UltraFAST crucibles to go from 1000°C to room temperature.

ANALYTICAL PROCEDURE

We have weighed accurately 4 g of Wheat flour and 1 g of BCR-380R (Milk powder) in UltraFAST crucibles (see details in Table 1).

The crucibles were initially preconditioned at constant weight (preconditioning step time is reduced thanks to the short cooling time of UltraFAST crucibles).



Crucibles were here placed into PYRO microwave ashing unit and ashed with the microwave program described in Table 2 at 800°C.

Crucible	Sample	Weight (g)	Type of Crucible
1	Wheat flour	4.1817	UltraFAST
2	Wheat flour	4.2750	UltraFAST
3	Wheat flour	4.0142	UltraFAST
4	Wheat flour	4.2944	UltraFAST
5	Wheat flour	4.2140	UltraFAST
6	Milk Powder (BCR-380R)	1.1099	UltraFAST
7	Milk Powder (BCR-380R)	0.9580	UltraFAST
8	Milk Powder (BCR-380R)	0.9984	UltraFAST

Table 1 – Details about sample type, amount and crucibles. Milk Powder is a certified standard material (BCR-380R) with an expected ashing residue of $6\pm 0.13\%$.

Step	Time	T1	Power
1	00:25:00	550°C	1800 W
2	00:30:00	550°C	1800 W

Table 2 – Pyro microwave ashing program.

RESULTS AND DISCUSSION

The samples were completely ashed, obtaining a white residue of inorganic material. The crucibles were weighted and percentage of residue was then recalculated.

Sample	Crucible (g)	Crucible+Residue (g)	Residue %
1- Wheat flour	1.7464	1.7616	0.36
2- Wheat flour	1.8052	1.8254	0.47
3- Wheat flour	1.7800	1.7988	0.47
4- Wheat flour	1.6556	1.6753	0.46
5- Wheat flour	1.5836	1.6033	0.47

Table 3- Wheat flour results, % of residues.

Sample	Crucible (g)	Crucible+Residue (g)	Residue %
6- Milk Powder (BCR-380R)	1.8263	1.8920	5.92
7- Milk Powder (BCR-380R)	1.9014	1.9590	6.01
8- Milk Powder (BCR-380R)	1.7526	1.8120	5.95

Table 4- % of ash recoveries of BCR-380R (Milk powder) with an expected ashing residue of $6\pm 0.13\%$.

CONCLUSION

Milestone's PYRO microwave ashing offers the big advantage to run all samples in only one step ensuring full of operations and great performance.

Due to its higher muffle capacity, faster heating and UltraFAST crucibles, the sample processing throughput is higher than conventional electrical muffle system.

The results shown in this report are very reproducible thanks to the great temperature homogeneity across all the muffle while the recoveries on certified standard material are in compliance with the certificate value.

This application report demonstrate how Milestone PYRO is a great tool to perform ashing of food samples.

ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.

