



ASTM D5630 STANDARD TEST METHOD FOR ASH CONTENT IN THERMOPLASTICS



An alternative and rapid method for measurement of inorganic residues in polymers compound samples.

| INTRODUCTION

Polymers represent a broad class of compounds with a tremendous range of physical properties.

Most of quality control labs of polymers manufacturer, rely on the ASTM D5630 for the measurement of inorganic residues in their polymer samples.

Inorganic residues from plastics may be antiblock, fillers, reinforcements, catalyst residues, colorants, etc. The quantitative amounts of each are important variables of the manufacturing process.

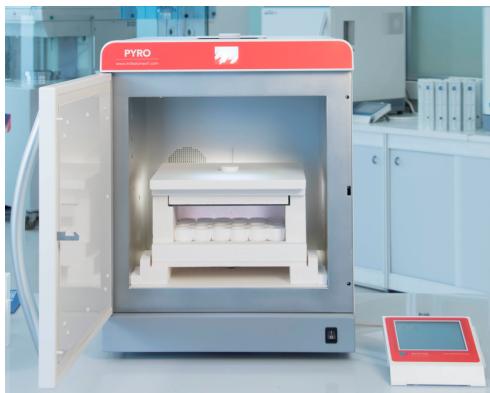
The ASTM D5630 describes a specific ashing method for the quantitative evaluation of inorganic content of a wide range of different thermoplastic materials. Traditional tools like electrical muffle furnaces, have their own set of limitations – long ashing time, long cooling, high power consumption and 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.



I | EXPERIMENTAL

INSTRUMENT



Picture 1 - Milestone PYRO Microwave ashing system

The new Milestone PYRO is an advanced microwave muffle furnace, suitable for 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

ASTM D5630 describes two different procedures (Procedure A and B) with details on sample amount and ashing temperature.

We have weighed accurately 10 g of Polypropylene sample in four UltraFAST crucibles (see details in Table 1).

INDUSTRY REPORT

PYRO | POLYMERS



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

Crucibles were then 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	Polypropylene	10.0254	UltraFAST
2	Polypropylene	10.0341	UltraFAST
3	Polypropylene	10.0506	UltraFAST
4	Polypropylene	10.0054	UltraFAST

Table 1 – Details about sample type, amount and crucibles. The sample amount must be in compliance with the Table X1.1 or X1.2 of ASTM D5630.

Step	Time	T1	Power
1	00:40:00	800°C	1800 W
2	00:05:00	800°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 %
Polypropylene (1)	1.5983	2.0113	4.12
Polypropylene (2)	1.9627	2.3761	4.12
Polypropylene (3)	1.9863	2.4024	4.14
Polypropylene (4)	1.7746	2.1868	4.12

Table 3- Obtained results, % of residues

CONCLUSION

Milestone's PYRO microwave ashing offers the big advantage to run all samples in only one step guaranteeing a complete safety environment for the users.

Due to its higher muffle capacity, faster heating and UltraFAST crucibles, the sample processing throughput is higher than conventional electrical muffle system, making PYRO the perfect solution for all laboratories with a high working flow.

The results shown in this report are very reproducible thanks to the great temperature homogeneity across all the muffle.

In addition, the ability to reach high temperatures in short time makes Milestone PYRO the suitable system for the ASTM method D5630.

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.



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