

CARBON BLACK DETERMINATION IN PLASTICS VIA OXYGEN-FREE MICROWAVE PYROLYSIS



An alternative and rapid method for determination of carbon black in olefin plastics using Milestone's PYRO microwave muffle furnace and self-closing quartz crucibles

I INTRODUCTION

The addition of carbon black to an elastomer can have a positive effect on its mechanical, electrical and physical properties. However, since carbon black is far more expensive than the elastomer production batch, a balance is usually sought between product performance and profitability. Therefore, a test to rapidly determine the carbon black content of elastomer formulations is desirable. Furthermore, the shorter the analysis time, the sooner the data can be used to adjust raw material components to the blender.

Reference standard test methods (i.e. ASTM D1603) provide gravimetric determination of carbon black content after pyrolysis of the sample under nitrogen.

The use of Milestone's self-closing quartz crucibles allows for work under inert conditions without the use of nitrogen gas, thus providing a valid alternative method which is rapid, reliable and easy to use.

In this study, different types of plastics with varying carbon black content were processed to evaluate the reliability and accuracy provided by the Milestone PYRO microwave muffle furnace and self-closing quartz crucibles.



I EXPERIMENTAL

In this technical note, a comparison study between the results obtained using self-closing crucibles in the PYRO and a conventional muffle under nitrogen (ASTM D1603) was performed. Two different plastic types (polyethylene and polypropylene) with different carbon black content were processed.

INSTRUMENT

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

It is equipped with a full stainless steel door and a cavity volume in excess of 70 liters, thus allowing the use of a large muffle resulting in enhanced sample throughput (up to 25 crucibles).



Figure 1 – Milestone's PYRO

The system additionally employs a rotating diffuser which evenly distributes the microwaves throughout the cavity. This not only assures even heating throughout, but also improves reproducibility.

A unique ceramic muffle allows microwave radiation to pass through and rapidly raise the temperature of a silicon carbide plate. The temperature is directly controlled in the muffle, providing constant feedback to the software which regulates the power in accordance with the customer defined program.

SELF-CLOSING CRUCIBLES

Milestone's new self-closing quartz crucibles allows for work in an oxygen-free environment without the use of inert gas.



Figure 2 – Self-closing quartz crucibles

Thanks to the special design of Milestone's self-closing quartz crucibles, the vacuum generated during the ashing process helps to perfectly seal them under inert atmosphere (without O_2).

The working principle is showed in Figure 3.

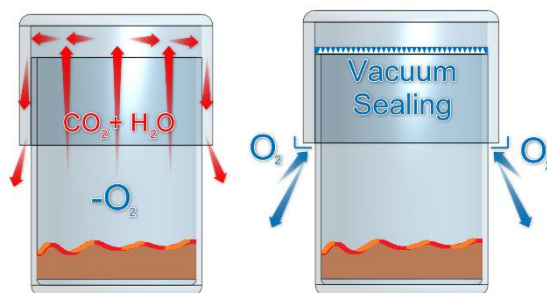


Figure 3 – Self-closing principle.

APPLICATION REPORT

PYRO | POLYMER



Advantages of self-closing crucibles:

- No N₂ consumption
- Shortest analysis time
- Ease-of-use

ANALYTICAL PROCEDURE

Crucibles with lids were initially preconditioned at constant weight. Samples were then weighed into quartz crucibles and placed inside the PYRO muffle. The ashing program is described in Table 2.

Sample	Sample amount	Carbon black (CB) content (expected)
Polyethylene A	3 g	40.0 %
Polyethylene B	3 g	50.0 %
Polypropylene A	3 g	18.5 %
Polypropylene B	3 g	30.0 %

Table 1 - Sample amount and expected values.

STEP	TIME	T2	POWER
1	00:20:00	600 °C	1800 W
2	00:10:00	600 °C	1800 W

Table 2 – PYRO microwave ashing program

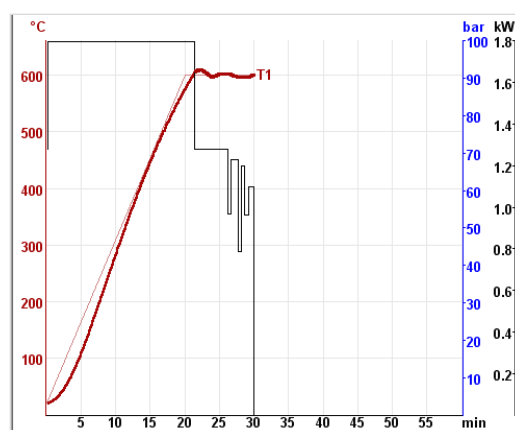


Figure 4 – Ashing profile



| RESULTS AND DISCUSSION

The performance of the Milestone PYRO (High sample throughput muffle) in combination with self-closing quartz crucibles was evaluated through a recovery study on polyethylene and polypropylene samples at different carbon black (CB) content levels.

The evaluation was made in comparison

to the results obtained following ASTM method D1603 under nitrogen with a conventional muffle. The analytical results are shown in Table 3, with recoveries in line with the expected values and RSDs below 2%. This demonstrates the robustness and reproducibility of an oxygen-free ashing procedure with self-closing crucibles in the Milestone PYRO.

	Polyethylene A		Polyethylene B		Polypropylene A		Polypropylene B	
	CB (%) (n=5)	RSD (%)	CB (%) (n=5)	RSD (%)	CB (%) (n=5)	RSD (%)	CB (%) (n=5)	RSD (%)
<i>PYRO (self-closing crucibles)</i>	40.1	1.18	49.9	1.37	18.3	2.03	30.2	1.61
<i>ASTM D1603</i>	40.1	1.06	49.8	1.68	18.4	1.85	30.1	0.92

Table 3- Carbon black content and RSD% on different types of plastics (S1,S2 – Polyethylene; S3,S4 – Polypropylene)

| CONCLUSION

The data shown in this technical note demonstrates that the results obtained with the Milestone PYRO and self-closing crucibles are fully comparable with the ones obtained with ASTM D1603, both in terms of accuracy and repeatability.

In addition, the PYRO's oxygen-free ashing technology provides great ease-of-use (no inert gas supply), superb time savings (10 min @ 600°C) and high productivity (up to 25 samples/cycle).

| ABOUT MILESTONE

With over 50 patents and more than 20,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 30 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers' worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.

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