Application Note

Instrument: TGA801

EMPOWERING RESULTS
Elemental Analysis | GC Mass Spectrometry | Metallography

Determination of Ash in Polypropylene

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Fillers are often added to polymers during the manufacturing process to allow for the development of specific properties within the material. For example, Titanium Dioxide is commonly added to polymers for its UV stabilization properties, while glass fibers are commonly added for enhanced strength. Some fillers even make the plastic easier to mold and shape while ensuring the stability of the polymer.

Variations in the amount of filler can cause the product to fail; therefore, determining the filler content of plastic is an important aspect of quality control monitoring. The various types of fillers that are added to polymers are typically inorganic materials. Ash is the inorganic remainder that is left after heating a polymer at high temperatures in the presence of Oxygen to remove water and organic matter. Therefore, the ash content of a plastic is reflective of the relative filler content in that plastic and can be used as a quality control parameter. The standard test method for ash content determination is thermogravimetric analysis (TGA).

Instrument Model and Configuration

Thermogravimetric analysis (TGA) is an analytical technique in which changes in sample mass due to changes in the physical and chemical properties of materials is measured as a function of temperature and/or time. TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain, due to decomposition, oxidation, or loss of volatile materials such as moisture. Macro TGA systems typically use a nominal one-gram sample mass to allow for more accurate mass change measurements in heterogeneous materials.

The LECO TGA801 is a macro thermogravimetric analyzer designed to determine mass loss of materials by measuring the change in mass of the sample as a function of the oven temperature while controlling the atmosphere and ventilation rate. The TGA801 allows up to 19 samples to be analyzed simultaneously.

Method Reference

ISO 3451-1, Method D: Plastics–Determination of Ash–Automated Instrument Method

ASTM D5630: Standard Test Method for Ash Content in Plastics

Note: A modified version of ASTM D5630 was utilized for the generation of data included in this application note.

Method Summary

The traditional manual method for determining ash in plastic materials using a muffle furnace includes multiple steps, including: a pre-ash step, an ash step, and a cooling step. The pre-ash step involves placing a sample over a burner to ignite the sample and allowing it to burn until all combustible material has been removed prior to placing the sample into a muffle furnace for the ash step. Following the ash step, the sample is removed and stored in a desiccator until cooled before the final weight is taken. Each of these steps requires that the analyst records the crucible/sample mass and physically moves each sample to the various locations.
The TGA801 simplifies this process by allowing the analyst to utilize an automated method that allows for the analysis of up to 19 samples simultaneously during the same batch analysis.

Using the TGA801 automated method, the analyst loads samples into crucibles, then the system prompts the user to add crucible lids prior to beginning analysis. The TGA801 then performs all the required analysis steps. To prevent the sample material from spontaneously igniting during analysis and potentially resulting in splatter and sample loss, the first two steps are performed in a Nitrogen atmosphere with the addition of crucible lids. These steps also incorporate hold times and a slower furnace ramp rate to allow the sample to adequately decompose. These steps take the place of the pre-ash step that is employed in the traditional manual method. Once the decomposition products have been removed during the first two steps, the subsequent ash step switches to an air atmosphere and ramps the furnace temperature up to the ASTM D5630 recommended temperature. Crucible lids are also used during the ash step to prevent a potential bias due to residue on the underside of the crucible lids.

To prevent decomposition byproducts from entering the lab environment, it is important that the instrument be properly vented as outlined in the TGA801 Instruction Manual.

This methodology may not provide the absolute filler content in all cases. This includes polymers filled with $CaCO_3$, nano clays, and Carbon black.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Typically, samples should be in powder or pellet form for optimal results.

Accessories

621-331 Ceramic Crucibles, 529-048 Ceramic Crucible Covers (Lids), 621-011-507 Double Ended Scoop.

Sample Mass ~2.0 g

Note: A sample mass of ~2.0 g is recommended for this application due to the low ash levels present in polypropylene.

Analysis Time ~3 hours

General Method Parameters

Crucible Type	Ceramic
Minimum Crucible Weight	19.0000
Maximum Crucible Weight	30.0000
Crucible Density	3.0
Lid Density	3.0
Sample Type	Other
Sample Density	1.0
Minimum Sample Weight	1.2000
Maximum Sample Weight	3.3000

Method Step Parameters

Step-1

Step Type	Custom
Step Name	Step-1
Cooling Option	Passive
Crucible Lids	Yes
Start Temperature	25.0 °C
End Temperature	400.0 °C
Ramp Rate	25.0 °C/min
Hold Time	30 min
Maximum Time	180 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At End Of Step

Step-2

Step Type	Custom
Step Name	Step-2
Cooling Option	Passive
Crucible Lids	Yes
Start Temperature	400.0 °C
End Temperature	450.0 °C
Ramp Rate	1.0 °C/min
Hold Time	5 min
Maximum Time	180 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At End Of Step

Ash

Step Type	Preset
Preset Method Step	Ash
Cooling Option	Active
Crucible Lids	Yes
Start Temperature	450.0 °C
End Temperature	800.0 °C
Ramp Rate	15.0 °C/min
Hold Time	15 min
Maximum Time	240 min
Atmosphere	Air
Flow Rate	10.0 LPM
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

Method Step Calculations

Step-1

Calculation Type	Custom
Measurement Type	Mass Ratio
Calculation Name	Step-1
Enable Calibration	Disabled
Ash Calculation	(Step-1 Mass ÷ Initial Mass)

Step-2

Calculation Type	Custom
Measurement Type	Mass Ratio
Calculation Name	Step-2
Enable Calibration	Disabled
Ash Calculation	(Step-2 Mass ÷ Initial Mass)

Ash

Calculation Type	Preset
Preset Method Step	Ash
Measurement Type	Mass Ratio
Enable Calibration	Disabled
Ash Dry Calculation	(Ash Mass ÷ Initial Mass)

Procedure

1. Create and/or select a method, using the Method Step Parameters listed above, following the procedure outlined in the TGA801 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGA801 Instruction Manual.

Typical Results

Sample	*Initial Mass (g)*	*% Ash*
Polypropylene Sample 1	2.0132	1.409
Yogurt Container	2.0322	1.447
	2.0469	1.467
	2.0378	1.487
	2.0222	1.452
	Avg =	***1.453***
	s =	***0.029***
Polypropylene Sample 2	2.0191	1.255
Food Container	2.0395	1.239
	2.0447	1.247
	2.0184	1.234
	2.0240	1.226
	Avg =	***1.240***
	s =	***0.011***
Polypropylene Sample 3	2.0153	0.280
Container Lid	2.0345	0.281
	2.0632	0.278
	2.0467	0.281
	2.0367	0.299
	Avg =	***0.284***
	s =	***0.008***

Sample	*Initial Mass (g)*	*% Ash*
Polypropylene Sample 4	2.1544	1.069
Yogurt Container	2.0992	1.081
	2.1179	1.046
	2.0760	0.999
	2.1075	1.036
	Avg =	***1.046***
	s =	***0.032***
Polypropylene Sample 5	2.0101	0.029
Food Container	2.0287	0.031
	2.0182	0.037
	2.0100	0.029
	2.0199	0.048
	Avg =	***0.035***
	s =	***0.008***
Polypropylene Sample 6[†]	2.0588	<0.015[††]
Sigma Aldrich	2.0202	<0.015[††]
(Part# 427888)	2.0228	<0.015[††]
	2.0627	<0.015[††]
	2.0139	<0.015[††]
	Avg =	**--**
	s =	**--**

[†]This sample is a high purity material not containing any fillers. As a result, it is expected that this sample will have little to no ash content. Data for this sample demonstrates that the methodology is successful in removing all of the base material, and that the ash values obtained for the other samples are reflective of the filler content in those materials.

[††]Results were below the readability limits of the instrument when analyzing samples at the recommended 2.0 g sample mass.

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