

## Instrument: TGA801

### Determination of Ash in Various Plastics and Plastic Blends

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Note: The methodology outlined in this application note allows for the simultaneous analysis of various plastic types.

#### 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 to remove the presence of 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 analysis 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 are 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 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 allow 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 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 the analysis. The TGA801 then performs all the required analysis steps. There are a wide range of plastics with different compositions and varying decomposition temperatures. Therefore, multiple steps are required when analyzing plastics of unknown composition to ensure that different materials effectively decompose prior to the final ash step. Another precaution taken to prevent the sample material from spontaneously igniting during analysis and potentially resulting in splatter and sample loss is performing the first four steps 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 four steps, the subsequent ash step switches to an air atmosphere and ramps the furnace temperature up to the ASTM D5630 recommended temperature to obtain the ash value. 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  $\text{CaCO}_3$ , 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.

Note: Please reference the appropriate Safety Data Sheets (SDS) for safe handling of all materials and samples.

## Accessories

621-331 Ceramic Crucibles, 529-048 Ceramic Crucible Covers, 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 plastics.

## Analysis Time ~6–7 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	1.8000
Maximum Sample Weight	2.2500

### 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	360.0 °C
Ramp Rate	25.0 °C/min
Hold Time	30 min
Maximum Time	120 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	360.0 °C
End Temperature	400.0 °C
Ramp Rate	1.0 °C/min
Hold Time	30 min
Maximum Time	120 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At End Of Step

#### Step-3

Step Type	Custom
Step Name	Step-3
Cooling Option	Passive
Crucible Lids	Yes
Start Temperature	400.0 °C
End Temperature	435.0 °C
Ramp Rate	1.0 °C/min
Hold Time	30 min
Maximum Time	120 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At End Of Step

#### Step-4

Step Type	Custom
Step Name	Step-4
Cooling Option	Passive
Crucible Lids	Yes
Start Temperature	435.0 °C
End Temperature	485.0 °C
Ramp Rate	1.0 °C/min
Hold Time	10 min
Maximum Time	120 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	485.0 °C
End Temperature	900.0 °C
Ramp Rate	15.0 °C/min
Hold Time	15 min
Maximum Time	180 min
Atmosphere	Air
Flow Rate	10.0 LPM
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

### Method Step Calculations

#### Ash

Calculation Type	Custom
Measurement Type	Mass Ratio
Calculation Name	Ash
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
<b>Storage Container</b>	2.0659	0.585
Polypropylene, High Density	2.0543	0.487
Polyethylene, Polystyrene	2.0211	0.530
Blend	2.0621	0.484
	2.0416	0.435
	$\bar{x}$ =	<b>0.504</b>
	$s$ =	<b>0.056</b>
<b>Utensil</b>	2.0304	0.153
Polystyrene	2.0379	0.161
	2.0975	0.153
	2.0855	0.146
	2.0770	0.142
	$\bar{x}$ =	<b>0.151</b>
	$s$ =	<b>0.007</b>
<b>Food Storage Bag</b>	2.0203	0.134
<b>(entire bag including zipper)</b>	2.0055	0.151
Low Density Polyethylene	2.0037	0.122
	2.0472	0.149
	2.0334	0.135
	$\bar{x}$ =	<b>0.139</b>
	$s$ =	<b>0.012</b>
<b>Food Storage Bag</b>	2.3081	0.233
<b>(zipper portion)</b>	2.3294	0.244
Low Density Polyethylene	2.3017	0.243
	2.3184	0.219
	2.3154	0.225
	$\bar{x}$ =	<b>0.233</b>
	$s$ =	<b>0.011</b>

Sample	Initial Mass (g)	% Ash
<b>Food Storage Bag</b>	2.0184	0.691
<b>(without zipper portion)</b>	2.0416	0.676
Low Density Polyethylene	2.0087	0.681
	2.0169	0.668
	2.0885	0.670
	$\bar{x}$ =	<b>0.677</b>
	$s$ =	<b>0.009</b>
<b>Styrofoam Bowl</b>	2.0640	1.050
Polystyrene	2.0529	1.059
	2.0559	1.039
	2.0343	1.036
	2.0385	1.031
	$\bar{x}$ =	<b>1.043</b>
	$s$ =	<b>0.011</b>
<b>Food Container</b>	2.0302	1.206
Polypropylene	2.1067	1.154
	2.0349	1.171
	2.0322	1.170
	2.0621	1.153
	$\bar{x}$ =	<b>1.171</b>
	$s$ =	<b>0.022</b>
<b>Food Container Lid</b>	2.0215	0.291
Polypropylene	1.9810	0.267
	1.9945	0.212
	2.0677	0.241
	2.0270	0.222
	$\bar{x}$ =	<b>0.247</b>
	$s$ =	<b>0.033</b>

$\bar{x}$  = Sample Mean;  $s$  = Sample Standard Deviation