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Instrument: TGA801

Determination of Carbon Black in Polypropylene

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Introduction

Fillers are frequently incorporated into polymers during the manufacturing process to develop specific properties within the material. For example, Titanium Dioxide is often added to polymers for its ultraviolet (UV) stabilization properties, while glass fibers are used to enhance the strength of the polymer. Certain fillers also facilitate the molding and shaping of plastics while maintaining the stability of the polymer. Furthermore, additives such as Carbon Black can be included to provide enhancements such as color, opacity, UV protection, and tensile strength. Carbon Black is a common additive in plastics intended for food contact applications.

National and regional regulations provide guidance on the approved grade and type of Carbon Black, and some regulations specifically restrict the amount of Carbon Black that may be present in plastics designated for food contact. A common method for determining the content of Carbon Black in polymer materials 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 nineteen 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

**A modified version of ISO 3451-1(Method D) and ASTM D5630 was utilized for the generation of data included in this application note.*

Method Summary

The traditional manual method for determining ash content in plastic materials using a muffle furnace involves several steps: pre-ashing, ashing, and cooling. In the pre-ash step, the sample is ignited over a burner to remove volatile materials before being transferred to the muffle furnace. After ashing, the sample is cooled in a desiccator before final weighing. Each step requires manual handling and recording of crucible/sample weights.

The TGA801 streamlines this process by automating the analysis of up to 19 samples simultaneously. The analyst loads samples into crucibles, adds lids when prompted, and initiates the analysis. The system performs all steps automatically. To prevent spontaneous ignition and sample loss, the first two steps occur in a nitrogen atmosphere with crucible lids, incorporating hold times and a slow furnace ramp rate. These steps replace the manual pre-ash process. Once decomposition is complete, the system switches to an air atmosphere and ramps to the ASTM D5630-specified temperature for the ash step. Lids remain in place during this step to avoid bias from residue on their undersides. Finally, Carbon Black content is calculated by subtracting the ash weight (Step 3) from the weight after decomposition (Step 2).

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.

Note: This methodology may not provide the absolute filler content in all polymers including polymers filled with $CaCO_3$ and nano clays.

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 (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 plastics.

Analysis Time ~6 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	0.2500
Maximum Sample Weight	2.5000

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	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	400.0 °C
End Temperature	500.0 °C
Ramp Rate	1.0 °C/min
Hold Time	30 min
Maximum Time	360 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

Step-3

Step Name	Step-3
Cooling Option	Active
Crucible Lids	Yes
Start Temperature	500.0 °C
End Temperature	800.0 °C
Ramp Rate	30.0 °C/min
Hold Time	30 min
Maximum Time	600 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-2

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

Step-3

Calculation Type	Custom
Measurement Type	Mass Ratio
Calculation Name	Step-3
Enable Calibration	Disabled
Ash Dry Calculation	(Step-3 Mass ÷ Initial Mass)

Carbon Black Residue

Calculation Type	Custom
Measurement Type	Mass Ratio
Calculation Name	% CB Residue
Enable Calibration	Disabled
Ash Dry Calculation	(Step-2 – Step-3)

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)*	*% CB Residue*
Carbon Black	0.2549	100.13
100 % Carbon Black	0.2516	99.86
	0.2513	100.17
	0.2518	100.17
	0.2526	99.87
	$\bar{x}$ =	***100.04***
	s =	***0.16***
Polypropylene	2.0377	<0.02*
Sigma Aldrich PP	2.0148	<0.02*
	2.0152	0.02
	2.0109	<0.02*
	2.0243	<0.02*
	$\bar{x}$ =	**--**
	s =	**--**
Carbon Black Spiked Polypropylene	2.0017	5.05
5.0 % Carbon Black	2.0018	5.06
	2.0037	5.04
	2.0024	5.05
	2.0016	5.03
	$\bar{x}$ =	***5.05***
	s =	***<0.02***
Carbon Black Spiked Polypropylene	1.9999	0.56
0.5 % Carbon Black	1.9995	0.51
	2.0012	0.53
	2.0028	0.50
	2.0011	0.52
	$\bar{x}$ =	***0.53***
	s =	***0.02***

Sample	*Initial Mass (g)*	*% CB Residue*
Polypropylene Sample #1	2.0320	0.13
	2.0496	0.14
	2.0909	0.13
	2.0825	0.14
	2.0310	0.14
	$\bar{x}$ =	***0.14***
	s =	***<0.02***
Polypropylene Sample #2	2.0157	8.11
	2.0007	8.10
	2.0418	8.08
	2.0073	8.09
	2.0604	8.09
	$\bar{x}$ =	***8.10***
	s =	***<0.02***
Polypropylene Sample #3	2.0300	1.86
	2.0090	1.80
	2.0150	1.70
	2.0164	1.66
	2.0228	1.69
	$\bar{x}$ =	***1.74***
	s =	***0.08***

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

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

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