Application Note

Instrument: TGA801

Determination of Moisture and Ash in Graphite

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Graphite is a stable form of carbon that exists in nature or can be synthetically produced. Graphite has many applications in our daily lives including its use in pencils, lubricants, electrodes, batteries, and carbon fiber. Synthetic graphite is a man-made substance manufactured by the high temperature processing of amorphous carbon materials. The types of amorphous carbon used as precursors to graphite are many, and can be derived from petroleum, coal, or natural and synthetic organic materials. Synthetic graphite is more pure in terms of carbon content and tends to behave more predictably. Graphite is a very popular material used in electronics and batteries due to its great conductive properties. It is the most commonly used substance to serve as the anode material in lithium-ion batteries due to its relatively low-cost and its energy density. Graphite in the form of carbon fibers is nearly five times stronger than steel and three times lighter. Therefore, carbon fibers and sheets have a wide variety of uses in the manufacturing world, including the automotive, aerospace, military, sporting equipment and construction industries.

The purity or grade of graphite is directly related to its suitability for different applications. The purer the graphite, the lower the ash content. The ash values of graphite samples can be utilized to compare the relative purity of various grades of graphite. Therefore, ash determination in graphite is a useful quality control process in the graphite industry.

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 more accurate mass change measurements in heterogeneous materials.

The LECO TGA801 is a macro thermogravimetric analyzer designed to determine moisture, volatile and ash content 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**

ASTM C561: Standard Test Method for Ash in a Graphite Sample
ASTM C562: Standard Test Method for Moisture in a Graphite Sample

**Modified versions of ASTM C561 and ASTM C562 were utilized for the generation of data included in this application note.*

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Typically, samples should be ground to a fineness of <1.0 mm.

Accessories

621-331 Ceramic Crucibles, 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 typically found in graphite.

Analysis Time

Method 1:	~7.5 h
Method 2:	~4.0 h

Methodology

Two different methods were utilized for the determination of ash when generating data for this application note. The first method closely follows the ash steps detailed in ASTM C561. The second method is an alternative method utilizing fewer ash steps, resulting in a faster analysis time with comparable results.

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	2.3
Minimum Sample Weight	1.8000
Maximum Sample Weight	2.2000

Method Step Parameters

Moisture	*Method 1*	*Method 2*
Step Type	Preset	Preset
Preset Method Step	Moisture	Moisture
Cooling Option	Active	Active
Crucible Lids	No	No
Start Temperature	25.0 °C	25.0 °C
End Temperature	110.0 °C	110.0 °C
Ramp Rate	6.0 °C/min	8.0 °C/min
Hold Time	15 min	15 min
Maximum Time	180 min	180 min
Atmosphere	Air	Air
Flow Rate	10.0 L/min	10.0 L/min
Final Weight	At Constancy	At Constancy
Constancy Window	9 min	9 min
Constancy Level	0.0005 g	0.0005 g

Ash Step-1	*Method 1*	*Method 2*
Step Type	Custom	Custom
Step Name	Ash Step-1	Ash Step-1
Cooling Option	Active	Active
Crucible Lids	No	No
Start Temperature	110.0 °C	110.0 °C
End Temperature	500.0 °C	500.0 °C
Ramp Rate	6.0 °C/min	15.0 °C/min
Hold Time	2 min	2 min
Maximum Time	180 min	180 min
Atmosphere	Oxygen	Oxygen
Flow Rate	8.0 L/min	8.0 L/min
Final Weight	At End Of Step	At End Of Step

Ash Step-2	*Method 1*	*Method 2*
Step Type	Custom	--
Step Name	Ash Step-2	--
Cooling Option	Active	--
Crucible Lids	No	--
Start Temperature	500.0 °C	--
End Temperature	750.0 °C	--
Ramp Rate	4.0 °C/min	--
Hold Time	30 min	--
Maximum Time	300 min	--
Atmosphere	Oxygen	--
Flow Rate	8.0 L/min	--
Final Weight	At Constancy	--
Constancy Window	9 min	--
Constancy Level	0.0005 g	--

Ash	*Method 1*	*Method 2*
Step Type	Preset	Custom
Preset Method Step/Step Name	Ash	Ash
Cooling Option	Active	Active
Crucible Lids	No	No
Start Temperature	750.0 °C	500.0 °C
End Temperature	950.0 °C	950.0 °C
Ramp Rate	5.0 °C/min	4.0 °C/min
Hold Time	60 min	30 min
Maximum Time	420 min	360 min
Atmosphere	Oxygen	Oxygen
Flow Rate	8.0 L/min	8.0 L/min
Final Weight	At Constancy	At Constancy
Constancy Window	9 min	9 min
Constancy Level	0.0005 g	0.0005 g

Method Step Calculations - Moisture

Calculation Type	Preset
Preset Method Step	Moisture
Measurement Type	Mass Ratio
Enable Calibration	Disabled
Moisture Calculation	(((Initial Mass – Moisture Mass)) ÷ Initial Mass)

Method Step Calculations - Ash

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

Method Step Calculations - Ash Dry

Calculation Type	Preset
Preset Method Step	Ash Dry
Measurement Type	Mass Ratio
Enable Calibration	Disabled
Ash Dry Calculation	(Ash x ((1 ÷ ((1 – Moisture)))))

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

	Method 1				Method 2			
Sample	*Initial Mass (g)*	*% Moisture*	*% Ash*	*% Ash Dry*	*Initial Mass (g)*	*% Moisture*	*% Ash*	*% Ash Dry*
Graphite	2.0028	0.07	0.96	0.96	1.9918	0.03	0.93	0.93
Sample #1	2.0051	0.09	0.97	0.97	2.0227	0.04	0.96	0.96
	2.0017	0.10	0.91	0.91	1.9999	0.04	0.94	0.94
	2.0296	0.09	0.95	0.95	2.0015	0.04	0.94	0.94
	1.9970	0.10	0.95	0.96	2.0061	0.04	0.95	0.95
	Avg =	***0.09***	***0.95***	***0.95***	***Avg =***	***0.04***	***0.94***	***0.95***
	s =	***0.01***	***0.02***	***0.02***	***s =***	***<0.01***	***0.01***	***0.01***
Graphite	2.0039	0.12	0.43	0.43	2.0087	0.07	0.41	0.41
Sample #2	1.9925	0.12	0.42	0.42	2.0043	0.07	0.41	0.41
	2.0068	0.12	0.42	0.42	1.9973	0.08	0.41	0.41
	2.0050	0.12	0.42	0.42	2.0024	0.07	0.41	0.41
	2.0117	0.12	0.42	0.42	2.0149	0.06	0.41	0.41
	Avg =	***0.12***	***0.42***	***0.42***	***Avg =***	***0.07***	***0.41***	***0.41***
	s =	***<0.01***	***<0.01***	***<0.01***	***s =***	***0.01***	***<0.01***	***<0.01***
Graphite	2.0167	0.07	0.08	0.08	2.0196	0.02	0.09	0.09
Sample #3	2.0143	0.07	0.09	0.09	2.0048	0.01	0.09	0.09
	2.0115	0.08	0.07	0.07	2.0318	0.01	0.07	0.07
	2.0092	0.07	0.08	0.08	2.0022	0.02	0.07	0.07
	2.0062	0.07	0.08	0.08	2.0010	0.02	0.06	0.06
	Avg =	***0.07***	***0.08***	***0.08***	***Avg =***	***0.02***	***0.08***	***0.08***
	s =	***<0.01***	***0.01***	***0.01***	***s =***	***0.01***	***0.01***	***0.01***

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