Application Note

Instrument: TGA801

Determination of Moisture, Volatile, and Ash in Biomass/Plant Tissue

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Moisture, volatile matter, and ash are important constituents for the quality characterization of fuel materials. Moisture, as determined by this instrument, is used for calculating analytical results of the fuel to a dry basis. Volatile matter is a primary constituent of solid fuels used in assessing the combustion characteristics. Ash determination is typically used to calculate ash yields for the fuel and other calculations involving the material balance for ash handling and disposal requirements for the fuel utilization.

Thermogravimetric analysis (TGA) is an analytical technique in which changes in sample mass, due to changes in 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.

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.

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, 529-048 Ceramic Covers, 621-011-507 Double Ended Scoop.

Sample Mass ~1.0 g

Analysis Time ~4 h

General Method Parameters

Crucible Type	Ceramic
Minimum Crucible Weight	19.0000
Maximum Crucible Weight	25.0000
Crucible Density	3.0
Lid Density	3.0
Sample Type	Other
Sample Density	1.5
Minimum Sample Weight	0.8000
Maximum Sample Weight	1.2000

Method Step Parameters - Moisture

Step Type	Preset
Preset Method Step	Moisture
Cooling Option	Active
Crucible Lids	No
Start Temperature	25.0 °C
End Temperature	107.0 °C
Ramp Rate	6.0 °C/min
Hold Time	15 min
Maximum Time	180 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0010 g

Method Step Parameters - Volatile

Step Type	Preset
Preset Method Step	Volatile
Cooling Option	Active
Crucible Lids	Yes
Start Temperature	107.0 °C
End Temperature	950.0 °C
Ramp Rate	45.0 °C/min
Hold Time	7 min
Maximum Time	180 min
Atmosphere	Nitrogen
Flow Rate	10.0 LPM
Final Weight	At End of Step

Method Step Parameters - Ash

Step Type	Preset
Preset Method Step	Ash
Cooling Option	Active
Crucible Lids	No
Start Temperature	600.0 °C
End Temperature	750.0 °C
Ramp Rate	3.0 °C/min
Hold Time	15 min
Maximum Time	180 min
Atmosphere	Oxygen
Flow Rate	3.5 LPM
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0010 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 - Volatile

Calculation Type	Preset
Preset Method Step	Volatile
Measurement Type	Mass Ratio
Enable Calibration	Disabled
Volatile Calculation	(((Moisture Mass – Volatile 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 - Volatile Dry

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

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 LECO TGA801 Instruction Manual.
2. Login and load samples following the procedure outlined in the LECO TGA801 Instruction Manual.

Typical Results

Sample	*Batch**	*Initial Mass (g)*	*Moisture %*	*Volatile Dry %*	*Ash Dry %*
Alfalfa	1	1.0042	6.58	74.7	10.4
n = 10	1	1.0057	6.57	74.3	10.5
	1	1.0143	6.54	74.1	10.5
	1	1.0201	6.54	74.3	10.5
	1	1.0434	6.49	74.4	10.5
	2	1.0032	6.54	74.4	10.5
	2	1.0126	6.55	74.3	10.5
	2	1.0033	6.59	74.2	10.5
	2	1.0218	6.50	74.2	10.5
	2	1.0034	6.54	74.3	10.5
		Avg =	***6.54***	***74.3***	***10.5***
		s =	***0.03***	***0.2***	***< 0.1***
Biomass	1	1.0270	5.75	81.0	0.47
n = 10	1	1.0216	5.73	81.0	0.53
	1	1.0137	5.70	80.9	0.55
	1	1.0128	5.76	81.0	0.54
	1	1.0490	5.70	80.8	0.55
	2	1.0112	5.78	80.7	0.47
	2	1.0148	5.75	80.9	0.40
	2	1.0216	5.78	80.6	0.47
	2	1.0276	5.75	80.8	0.46
	2	1.0142	5.77	80.9	0.49
		Avg =	***5.75***	***80.9***	***0.49***
		s =	***0.03***	***0.1***	***0.05***

**Batch 1 and Batch 2 were analyzed separately utilizing a Dual Furnace TGA801.*

Note: LECO recommends that a calibration of y=1x+0 be used for volatile matter determination in biomass.

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