Application Note

Instrument: TGA801

Moisture, Volatile Matter, Ash, and Fixed Carbon Determination in Coke

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Moisture, volatile matter, and ash are all important solid fuel characterization measurements for coke materials. The moisture, volatile matter, and ash results are typically among the primary parameters used for assessing the quality of a solid fuel material. The moisture result is utilized for calculating the dry basis results of other analytical results. The ash result is utilized in the ultimate analysis calculation of oxygen by difference (ASTM D3176) and for calculating material balance and ash load purposes in industrial boiler systems. The volatile matter result indicates the coke yield on the carbonization process, providing additional information on combustion characteristics of the materials, and establishes a basis for purchasing and selling the solid fuel materials. Fixed carbon is a calculated value of the difference between 100 and the sum of the moisture, ash, and volatile matter where all values are on the same moisture reference base. The LECO TGA801 is a macro thermogravimetric analyzer that allows up to 19 samples to be analyzed simultaneously.

Sample Preparation

Samples should be prepared in accordance with ASTM Practice D346. Coke reference materials such as those offered by LECO and NIST comply with this preparation methodology.

Accessories

621-331 Large Ceramic Crucibles, 529-048 Large Ceramic Covers, 621-011-507 Double Ended Scoop

Calibration Samples

Calibration samples are used to calibrate volatile matter content of samples and are normally not required for moisture or ash determination. Select a minimum of three coke reference materials with known dry basis volatile matter content. The reference materials must cover the full range of expected volatile matter content of the coke and samples that will routinely be analyzed. NIST, LECO, or other suitable reference materials may be used.

Sample Mass ~1.0 g

Method Reference

ASTM D7582 Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis

Analysis Time ~4 h

Method General Parameters

Crucible Type	Ceramic
Minimum Crucible Weight	20.0
Maximum Crucible Weight	30.0
Crucible Density	3.0
Lid Density	3.0
Sample Type	Other
Sample Density	1.5
Minimum Sample Weight	0.40
Maximum Sample Weight	1.10

Method Step Parameters

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 L/min
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

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 L/min
Final Weight	At End Of Step

Step Type	Preset
Preset Method Step	Ash
Cooling Option	Active
Crucible Lids	No
Start Temperature	600.0 °C
End Temperature	950.0 °C
Ramp Rate	6.0 °C/min
Hold Time	0 min
Maximum Time	180 min
Atmosphere	Oxygen*
Flow Rate	3.5 L/min
Final Weight	At Constancy
Constancy Window	9 min
Constancy Level	0.0005 g

**Air can be substituted for oxygen, flow rate should be set to 10 L/min.*

Method Equation Parameters

Equation Name	Equation Text
Moisture	(((Initial Mass - Moisture Mass)) ÷ Initial Mass)
Volatile	(((Moisture Mass - Volatile Mass)) ÷ Initial Mass)
Ash	(Ash Mass ÷ Initial Mass)
Fixed Carbon	(1 - (((Moisture + Volatile) + Ash)))
Volatile Dry	(Volatile x ((1 ÷ ((1 - Moisture)))))
Ash Dry	(Ash x ((1 ÷ ((1 - Moisture)))))
Fixed Carbon Dry	(1 - ((Volatile Dry + Ash Dry)))

Procedure

1. Create and/or select a method using the parameters described above following the procedure in the TGA801 Instruction Manual.
2. Login and load samples following the procedure outlined in the TGA801 Instruction Manual.
3. Calibrate for volatile matter by using a minimum of three coal reference materials by following the procedure outlined in the TGA801 instruction manual.
4. After calibration for volatile matter is complete, analyze unknown samples following the procedure outlined in the TGA801 instruction manual.

Typical Results (Dry Basis)

Sample	Mass (g)	Moisture %	Volatile %	Ash* %	Fixed Carbon %	Batch
Calcined	1.0235	0.15	0.39	0.79	98.83	1
Petroleum Coke	1.0247	0.16	0.44	0.82	98.75	1
n=10	1.0278	0.14	0.25	0.76	98.99	1
	1.0376	0.18	0.20	0.81	99.00	1
	1.0500	0.16	0.48	0.81	98.71	1
	1.0203	0.09	0.27	0.81	98.92	2
	1.0037	0.12	0.33	0.81	98.86	2
	1.0097	0.11	0.31	0.74	98.95	2
	1.0646	0.12	0.52	0.80	98.68	2
	1.0230	0.14	0.29	0.80	98.91	2
	Avg =	***0.14***	***0.35***	***0.79***	***98.86***	
	s =	***0.03***	***0.11***	***0.02***	***0.12***	

Sample	Mass (g)	Moisture %	Volatile %	Ash* %	Fixed Carbon %	Batch
Petroleum	1.0049	0.67	13.30	0.30	86.41	1
Coke	0.9984	0.68	13.08	0.28	86.64	1
n=10	1.0034	0.69	12.98	0.26	86.76	1
	0.9996	0.66	13.15	0.28	86.58	1
	1.0075	0.67	13.15	0.26	86.60	1
	1.0069	0.57	13.22	0.29	86.50	2
	0.9988	0.59	13.22	0.37	86.41	2
	1.0248	0.64	13.16	0.36	86.48	2
	1.0093	0.65	13.17	0.32	86.51	2
	1.0439	0.65	13.17	0.32	86.51	2
	Avg =	***0.65***	***13.16***	***0.30***	***86.54***	
	s =	***0.04***	***0.09***	***0.04***	***0.11***	

Metallurgical	1.0081	1.15	2.39	11.62	85.99	1
Coke	1.0062	1.20	2.18	11.63	86.19	1
n=10	1.0353	1.19	2.39	11.66	85.94	1
	1.0215	1.21	2.26	11.62	86.12	1
	1.0113	1.16	2.31	11.65	86.04	1
	1.0062	1.07	2.33	11.65	86.02	2
	1.0249	1.09	2.22	11.67	86.10	2
	1.0085	1.06	2.26	11.65	86.09	2
	1.0148	1.07	2.36	11.70	85.95	2
	1.0139	1.07	2.42	11.67	85.91	2
	Avg =	***1.13***	***2.31***	***11.65***	***86.03***	
	s =	***0.06***	***0.08***	***0.03***	***0.09***	

**Performed in an oxygen environment.*

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2015 Q-994 | *LECO is a registered trademark of LECO Corporation.*

Form No. 203-821-571 6/19—REV1