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

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TGA701

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 (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 TGA701 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 Method D2013 or ASTM Practice D346. Coke reference materials such as those offered by LECO and NIST are properly prepared.

Accessories

621-331 Ceramic Crucibles, 529-048 Ceramic Cover

Calibration Samples

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

Sample Mass

~1 gram

Method Reference

ASTM D7582

Analysis Time

~5 hours

Method General Parameters

Crucible Type	Ceramic
Crucible Density	3.00
Cover Density	3.00
Sample Type	Coke
Sample Density	1.50
System	
Significant Digits	F6
Barometric Pressure	760
Cover Open Half	800
Cover Open Full	450
Furnace Over Temp.	1200
Increment Sample Names	Disable
Crucible Locations	Auto Find
Temperature Check	False
Alarm On Errors	True
Cover Option	Half Open
Carousel Option	Weigh Position
Batch Mode	False
Enable Lid Dispenser	False
Balance	
Readability	4
Balance Environment	Stable
Balance Constancy	Absolute Weight (grams)
Minimum Weight	0.9000
Maximum Weight	1.1000
Zero Deviation	0.0005
Retake Initial Weights	False

Method Step Parameters

Parameter	*Moisture*	*Volatile*	*Ash*
Covers	No	Yes	No
Start Temp. °C	25	107	600
End Temp. °C	107	950	950
Ramp Rate	6	50	6
Ramp Time (h:m)	00:13	00:16	00:58
Hold Time (h:m)	00:15	00:07	00:00
Total Time (h:m)	00:28	00:23	00:58
Max Time (h:m)	00:00	00:00	00:00
Atmosphere	Nitrogen	Nitrogen	Oxygen*
Flow Rate	High	High	Low*
Window	3	0	3
Comparator	0.0005**	100.0000	0.0005**
Final Weight	At Constancy	At End of Step	At Constancy

**Air can be substituted for oxygen, flow rate should be set to high.*
***Balance constancy set for Absolute Weight (grams).*

Organic Application Note

Delivering the Right Results

Method Equation Parameters

Equation Name	*Equation Text*
Moisture	(([Initial Mass]-[Moisture Mass])/[Initial Mass])*100
Volatile	(([Moisture Mass]-[Volatile Mass])/[Initial Mass])*100
Ash	([Ash Mass]/[Initial Mass])*100
Fixed Carbon	100-([Moisture]+[Volatile]+[Ash])
Volatile Dry	[Volatile]*(100/(100-[Moisture]))
Ash Dry	[Ash]*(100/(100-[Moisture]))
Fixed Carbon Dry	100-([Volatile Dry]+[Ash Dry])

Procedure

1. Create and/or select a method using the parameters described above following the procedure outlined in the TGA701 Instruction Manual.
2. Calibrate for dry volatile matter by using a minimum of three coke reference materials by following the procedure outlined in the TGA701 Instruction Manual.
3. After calibration for volatile matter is complete, analyze unknown samples following the procedure outlined in the TGA701 Instruction Manual.

Typical Results (Dry Basis)

Sample	*Mass g*	*% Moisture*	*% Volatile*	*% Ash*	*% Fixed C*
NIST SRM 2718	0.9845	0.40	10.55	0.28	89.16
Green Petroleum	0.9811	0.39	10.59	0.24	89.17
Coke	1.0552	0.39	10.62	0.17	89.21
10.6% Vol*	1.0366	0.39	10.66	0.18	89.16
0.18% Ash*	0.9940	0.41	10.58	0.22	89.20
	X =	***0.40***	***10.60***	***0.22***	***89.18***
	s =	***0.01***	***0.04***	***0.05***	***0.02***
NIST SRM 2775	1.1633	0.32	1.18	5.72	93.10
Foundry Coke	1.0946	0.31	1.32	5.75	92.94
1.31% Vol**	1.1858	0.32	1.25	5.72	93.02
5.77% Ash**	1.1718	0.31	1.35	5.74	92.91
	1.0475	0.32	1.46	5.73	92.81
	X =	***0.32***	***1.31***	***5.73***	***92.96***
	s =	***0.01***	***0.11***	***0.01***	***0.11***
NIST SRM 2776	1.0162	0.27	0.97	7.97	91.06
Furnace Coke	1.1827	0.26	0.91	7.92	91.17
0.98% Vol**	1.1829	0.29	0.92	7.90	91.18
8.06% Ash**	1.1674	0.26	1.02	7.97	91.01
	1.1483	0.27	1.09	7.95	90.96
	X =	***0.27***	***0.98***	***7.94***	***91.08***
	s =	***0.01***	***0.07***	***0.03***	***0.10***

**Information values **Reference values*
Refer to NIST certificate for definition.

LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
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