Gross Calorific Value in Metallurgical Coke

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AC600

Introduction

Metallurgical coke is a porous, high-carbon material produced from a carbonization process of coals with specific rank. Metallurgical coke is primarily used in the process of iron production within a blast furnace where it serves several functions, including a fuel function (heat source). As a fuel, metallurgical coke provides the heat for the chemical reactions within the blast furnace, and the melting of slag and metal. The gross calorific value determination in metallurgical coke is most often used in calculating the total calorific value for a quantity of metallurgical coke to be used in blast furnace operations.

Method Reference

ASTM D5865

Sample Preparation

A representative, uniform sample is required.

Accessories

774-204 Nickel Crucibles, 776-978 Glass Scoop, Tweezers, 502-450-100 Particulate Filter, 502-461 Cotton Thread Fuse (10 cm, 375 pack).

Calibration Sample

LECO 774-208-150 Benzoic Acid Pellets or pellets made from NIST Benzoic Acid Powder.

Method Parameters

Method	TruSpeed™
Standard Mode	ASTM D5865

Thermochemical Corrections

Titrant Energy Value	0.0039683 BTU/ml
Sulfur Correction	23.861 BTU/lb
Calculation Mode	TruSpeed
Analysis Time	5.0 Minutes
Equilibrate Time	1.5 Minutes
Main Time	2.8 Minutes
Stir Speed	13.0

System Parameters – Database

Fuse Type	Cotton
Fuse Length	10 cm
Fuse Combustion Heat	0.006255562 BTU/cm
Significant Digits	5
Result Units	BTU/lb
Sleep Timeout	90 minutes
Water Temperature	25°C
Auto Increment Sample Name	Disable
Alarm	Yes

Procedure

1. Prepare the instrument as outlined in the operator's instruction manual.
2. Choose TruSpeed Method for analysis.
3. Condition the system by analyzing LECO 774-208-150 Benzoic Acid Pellet.
4. Calibration
   a. Place a 502-450-100 Heated Particulate Filter in the 774-204 Crucible as shown in Figure 1.
   b. Weigh ~1.0 g Benzoic Acid Pellet into the crucible.
   c. Place the crucible containing the sample on to the crucible holder of the combustion vessel.
   d. Tie a single 502-461 Cotton Thread Fuse to the electrode wire and place one end of the fuse under the sample.
   e. Carefully close the combustion vessel and secure the closure ring.
   f. Pressurize the vessel with oxygen.
   g. Submerge the vessel in a container of distilled water to check for leaks.
   h. Place the combustion vessel on the electrode connectors of the loading mechanism.
   i. Press the green analyze button in the software or on the instrument to initiate analysis
   j. When the analysis has completed, the loading mechanism will raise. Remove the vessel and depressurize.
   k. Follow steps 5a through 5d for all samples.
   l. Repeat steps 4a through 4k a minimum of five times and calibrate the instrument.
5. Nitrogen Correction
   a. After the vessel has depressurized, rinse the inside of the combustion vessel with distilled water and pour the washings into a clean beaker.
   b. Add 1-2 drops of indicator.
   c. Titrate with Sodium Carbonate until the endpoint is reached.
   d. Enter the titrated amount in milliliters into the nitrogen correction column of the AC600 software*.

   **Note: For the recommended reagents and indicators, see the current version of ASTM D5865.*
6. Analyze Samples
   a. Place a 502-450-100 Heated Particulate Filter in the 774-204 Crucible as shown in Figure 1.

Figure 1. Use a rod or other wide, round-ended object to evenly place the filter into the crucible.

Organic Application Note

b. Weigh ~0.75 g of a metallurgical coke sample into the crucible.
c. Place the crucible containing the sample on to the crucible holder of the combustion vessel.
d. Tie a single 502-461 Cotton Thread Fuse to the electrode wire and place one end of the fuse on top of the sample.
e. Follow steps 4e through 4j for the sample analysis and 5a through 5d for nitrogen correction.
f. Repeat steps 6a through 6e for all metallurgical coke samples.

Note: For the best results, coke samples must also have a sulfur and moisture correction performed. Enter the as determined sulfur value and the as determined moisture value into the respective columns in the AC600 software. The sulfur units for the correction can be found in the method screen.

Typical Results

The following results are confirmed through an external Proficiency Testing Program.

Name	*Mass(g)*	*Gross Heat (BTU/lb)**
774-208-150	1.0711	11370
Benzoic Acid Tablets	0.9026	11373
11375 ± 12 BTU/lb	0.9914	11376
	0.9864	11381
	0.9859	11375
	$\bar{\chi}$=	***11375***
	***s*=**	***4***

Name	*Mass(g)*	*Gross Heat (BTU/lb)***
Metallurgical	0.7507	12586
Coke	0.7514	12615
	0.7516	12604
	0.7577	12605
	0.7534	12587
	$\bar{\chi}$=	***12599***
	***s*=**	***13***

**Corrected for nitrogen and used to calibrate the instrument.*
***Corrected for nitrogen, sulfur and moisture.*

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