Gross Heat in Petroleum Coke

LECO Corporation; Saint Joseph, Michigan USA

Instrument: AC600

Introduction

Fuel grade petroleum coke is a high carbon material produced from the petroleum oil refining process. Gross calorific value determination in petroleum coke is most often used in calculating the total calorific value for a quantity of petroleum coke for fuel value purposes. The gross calorific value and sulfur value of the petroleum coke can also be used to calculate if the fuel meets regulatory requirements for industrial fuel use.

Method Reference

ASTM D5865, ASTM D346

Sample Preparation

Sample must be pulverized to pass 250 µm (No. 60) sieve, prepared in accordance with ASTM D346.

Accessories

774-204 SST Crucibles, 776-978 Glass Scoop, Tweezers

Calibration Sample

Benzoic acid pellets made from NIST 39j, or LECO 774-208 Benzoic Acid Pellets.

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

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 Benzoic Acid Pellet.
4. Calibration
   a. Weigh ~1.0 g Benzoic Acid Pellet into a 774-204 Crucible.
   b. Place the crucible containing the sample on to the crucible holder of the combustion vessel.
   c. Tie a single cotton thread fuse to the electrode wire and place one end of the fuse under the sample.
   d. Carefully close the combustion vessel and secure the closure ring.
   e. Pressurize the vessel with oxygen.
   f. Submerge the vessel in a container of distilled water to check for leaks.
   g. Place the combustion vessel on the electrode connectors of the loading mechanism.
   h. Press the green analyze button to initiate analysis.
   i. When the analysis has completed, the loading mechanism will raise. Remove the vessel and depressurize.
   j. Follow steps 5a through 5d for all samples.
   k. Repeat steps 4a through 4j 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 to 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 applicable standard.
6. Analyze Samples
   a. Weigh ~0.75 g of a petroleum coke sample.
   b. Place the crucible containing the sample on to the crucible holder of the combustion vessel.
   c. Tie a single cotton thread fuse to the electrode wire and place one end of the fuse on top of the sample.
   d. Follow steps 4d through 4i for the sample analysis and 5a through 5d for nitrogen correction.

Organic Application Note

e. Repeat steps 6a through 6d for all petroleum coke samples.

Note: For the best results, petroleum 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

Sample	*Mass (g)*	*BTU/lb*
502-684 Lot: 12237	0.7567	15247
Petroleum Coke RM	0.7502	15238
	0.7572	15217
	0.7562	15224
	0.7502	15234
	X=	***15232***
	s=	***12***
Petroleum Coke Sample	0.7526	14072
	0.7585	14062
	0.7522	14060
	0.7541	14061
	0.7570	14039
	X=	***14059***
	s=	***12***

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