Application Note

Instrument: CHN828

Determination of Carbon, Hydrogen and Nitrogen in Coke

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Carbon, hydrogen, and nitrogen determination is part of the ultimate analysis of coke fuel material, aiding in characterizing the material and providing information that can be utilized in calculating material/energy balances and efficiencies, as well as emissions potentials for the coke fuel.

The LECO CHN828 is a combustion carbon, hydrogen, and nitrogen determinator that utilizes a pure oxygen environment in a vertical quartz furnace, ensuring complete combustion and superior analyte recovery. Combustion gases are swept from the furnace through an afterburner containing a reagent to scrub sulfur compounds from the gas stream prior to collection in the ballast volume. The combustion gases are collected in a ballast where they equilibrate and mix before a representative aliquot (3 cm^3 or 10 cm^3 volume) of the gas is extracted and introduced into a flowing stream of inert gas (helium or argon) for analysis. The aliquot gas is carried to an infrared cell (IR) for the detection of carbon (CO_2) and a thermal conductivity cell (TC) for the detection of nitrogen (N_2). A separate portion of the ballast gas is transferred to an IR cell for the determination of hydrogen (H_2O).

Method Reference

ASTM D5373: Standard Test Methods for Determination of Carbon, Hydrogen and Nitrogen in Analysis Samples of Coal, and Carbon in Analysis Samples of Coal and Coke.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Samples should be prepared in accordance to ASTM D346/D346M. Reference materials should be prepared as directed by the certificate prior to analysis. In accordance with ASTM D5373, coke samples should be analyzed "as received" and analytical values should be corrected for moisture following analysis. Moisture values should be determined within the same day the coke samples are analyzed.

Accessories

502-186 Small Tin Foil Cups, and 501-614 Spatula

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

Note: ASTM D5373 requires the use of pure compounds for calibration.

*Method Parameters**

Gas Type	Helium
Furnace Temperature	950 °C
Afterburner Temperature	850 °C
Nominal Mass	1.0000 g
Purge Cycles	3

*Ballast Parameters**

Ballast Equilibrate Time	10 s
Ballast Not Filled Timeout	300 s
Aliquot Loop Fill Pressure Drop	200 mm Hg
Aliquot Loop Equilibrate Time	6 s
Dose Loop Size	Large (10 cm^3)
Interleave Analysis	Yes
Sample Drop Detection	Disabled

*Element Parameters**

	Carbon	*Hydrogen*	*Nitrogen*
Integration Delay	4 s	**	4 s
Starting Baseline	15 s	**	15 s
Post Baseline Delay	0 s	**	14 s
Use Comparator	No	**	No
Integration Time	13 s	**	35 s
Use Endline	Yes	**	Yes
Endline Delay	19 s	**	25 s
Ending Baseline	15 s	**	15 s
Use Hydrogen Correction	—	Yes	—

**Refer to 828 Series Operator's Instruction Manual for Parameter definitions.*

***Hydrogen determination is performed using a "stop-flow" analysis technique; therefore, the element parameters for hydrogen are not adjustable.*

Burn Profile

Burn Step	*Furnace Flow*	*Time*
1	4.00 L/min	15 s
2	1.00 L/min	End

Procedure

1. Prepare the instrument for operation as outlined in the operator's instruction manual.
2. Calibration.
   a. Condition the System.
      i. Select two to five replicates in the Login screen.
      ii. Weigh ~0.1 g of a coke reference or sample material into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize trapped atmosphere by twisting the top edges of the foil together.
      iii. Enter conditioning sample mass and identification into the Login screen.
      iv. Transfer the tin foil cup containing the conditioning sample to the appropriate position in the sample carousel.
      v. Perform steps 2a.ii through 2a.iv for each conditioning sample to be analyzed.
      vi. Initiate the analysis sequence.
   b. Determine Blank.
      i. Select five or more Blank replicates in the Login screen.
      ii. Initiate the analysis sequence.
      iii. Set the blank using at least five blank results following the procedure outlined in the operator's instruction manual.

   Note: The standard deviation of the last five blanks should be less than or equal to 0.001% (10 ppm) for all three elements when utilizing helium as a carrier gas. Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.

   c. Calibrate.†
      i. Select the desired number of reference material replicates in the Login screen (minimum of five).
      ii. Weigh an appropriate mass of a suitable reference material (pure compound) into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize entrapped atmosphere by twisting the top edges of the foil together.
      iii. Enter reference material mass and identification into the Login screen.
      iv. Transfer the tin foil cup containing the reference material to the appropriate position in the sample carousel.
      v. Perform steps 2c.ii through 2c.iv for each reference material sample to be analyzed.
      vi. Initiate the analysis sequence.
      vii. Calibrate the instrument following the procedure outlined in the operator's instruction manual.
      viii. Verify the calibration by analyzing an appropriate mass of another suitable reference material and confirm that the results are within the uncertainty provided on the certificate.

   †If following ASTM Method D5373, refer to the calibration procedures outlined in the official ASTM Method for guidance. Refer to the 828 Series operator's instruction manual for additional details regarding calibrating the instrument if using fractional masses.

   Note: Once the calibrations have been established, drift correction may be performed on a regular basis in lieu of calibration.

3. Drift Correction.
   a. Condition the System.
      i. Select two to five replicates in the Login screen.
      ii. Weigh ~0.1 g of a coke reference or sample material into a 502-186 Tin Foil Cup and seal the cup in a manner to eliminate entrapped atmosphere by twisting the top edges of the foil together.
      iii. Enter conditioning sample mass and identification into the Login screen.
      iv. Transfer the tin foil cup containing the conditioning sample to the appropriate position in the sample carousel.
      v. Perform steps 3a.ii through 3a.iv for each conditioning sample to be analyzed.
      vi. Initiate the analysis sequence.
   b. Determine Blank.
      i. Select five or more Blank replicates in the Login screen.
      ii. Initiate the analysis sequence.
      iii. Set the blank using at least five blank results following the procedure outlined in the operator's instruction manual.

   Note: The standard deviation of the last five blanks should be less than or equal to 0.001% (10 ppm) for all three elements when utilizing Helium as a carrier gas. Additional blanks beyond the recommended ten may be required in order to achieve the recommended precision.

   c. Drift Correct.
      i. Select the desired number of drift replicates in the Login screen (minimum of three).
      ii. Weigh an appropriate mass of the same reference material (pure compound) that was used to calibrate the instrument into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize trapped atmosphere by twisting the top edges of the foil together.
      iii. Enter reference material mass and identification into the Login screen.
      iv. Transfer the tin foil cup containing the reference material to the appropriate position in the sample carousel.
      v. Perform steps 3c.ii through 3c.iv a minimum of three times.
      vi. Initiate the analysis sequence.
      vii. Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
      viii. Verify the drift correction by analyzing an appropriate mass of another suitable reference material and confirm that the results are within the uncertainty provided on the certificate.
4. Sample Analysis.
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh 0.08 – 0.1g of the coke sample into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize entrapped atmosphere by twisting the top edges of the foil together.
   c. Enter sample mass and identification information into the Login screen.
   d. Transfer the tin foil cup containing the sample to the appropriate position in the sample carousel.
   e. Perform steps 4b through 4d for each sample to be analyzed.
   f. Initiate the analysis sequence.

TYPICAL RESULTS

Data was generated utilizing linear, full regression calibrations for carbon and hydrogen determination, using fractional masses of LECO 502-642 (Lot 1018) Phenylalanine LCRM (65.46% C, 6.77% H). A linear, force through origin calibration was utilized for nitrogen determination using LECO 502-642 (Lot 1018) Phenylalanine LCRM (8.47% N). Drift Correction was performed utilizing ~0.1 g of LECO 502-642 (Lot 1018) Phenylalanine LCRM. The calibrations and drift corrections were verified using LECO 502-896 (Lot 1002) EDTA LCRM (41.00% C, 5.52% H, 9.57% N). Coke samples were analyzed as received and corrected for moisture in accordance with ASTM D5373.

	Mass (g)	*Carbon (%)*	*Hydrogen (%)*	*Nitrogen (%)*
LECO 502-683	0.0859	88.3	0.16	0.98
Lot# 19269	0.0865	88.2	0.14	0.98
Metallurgical Coke	0.0829	88.0	0.13	0.97
88.1% ±1.7% C	0.0825	88.4	0.13	0.98
0.21% H (informational only)	0.0896	88.4	0.12	0.98
0.95% ±0.06% N	***Avg =***	***88.3***	***0.13***	***0.98***
	s =	***0.2***	***0.02***	***<0.01***
LECO 502-684	0.0874	88.8	3.89	1.43
Lot# 18321	0.0865	88.7	3.88	1.43
Petroleum Coke	0.0841	88.6	3.87	1.42
88.5% ±1.3% C	0.0847	88.5	3.86	1.42
3.91% ±0.29% H	0.0822	88.9	3.88	1.42
1.40% ±0.17% N	***Avg =***	***88.7***	***3.88***	***1.42***
	s =	***0.2***	***0.01***	***0.01***
NIST SRM® 2776	0.0815	89.2	0.26	1.21
Furnace Coke	0.0845	89.2	0.25	1.21
89.15% ±1.65% C	0.0828	89.2	0.25	1.22
0.26% ±0.04% H	0.0850	89.3	0.24	1.23
1.21% ±0.06% N	0.0877	89.4	0.24	1.21
	Avg =	***89.3***	***0.25***	***1.22***
	s =	***0.1***	***0.01***	***0.01***

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