Application Note

LEO EMPOWERING RESULTS

Instrument: CHN828

Determination of Carbon, Hydrogen, and Nitrogen in Coal

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Introduction

Carbon, Hydrogen, and Nitrogen determination is part of the ultimate analysis of coal fuel material, aiding in characterizing the material and providing information that can be utilized in calculating material/energy balances and efficiencies, as well as emission potentials for the coal fuel. The Carbon, Hydrogen, and Nitrogen results for a coal material are also utilized to evaluate the reactivity potential for its use in a liquefaction or gasification process.

Instrument Model and Configuration

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 detection of Hydrogen (H_2O).

Thermal conductivity detectors work by detecting changes in the thermal conductivity of the analyte gas compared to a reference/carrier gas. The greater the difference between the thermal conductivity of the carrier gas and the analyte gas, the greater the sensitivity of the detector. The CHN828 supports either the use of Helium or Argon as the instrument's carrier gas. When used as a carrier gas, Helium provides the highest sensitivity, and the best performance at the lower limit of the Nitrogen range. Argon can also be used as a carrier gas. The thermal conductivity difference between Argon and Nitrogen is not as great as the thermal conductivity difference between Helium and Nitrogen, therefore the detector is inherently less sensitive when using Argon as a carrier gas.

Note: When changing carrier gas type, the flow needs to be adjusted following instructions provided in the 828 Series Operator's Instruction Manual.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Samples should be prepared in accordance to ASTM D2013. Reference materials should be prepared as directed by the certificate prior to analysis. In accordance with ASTM D5373, coal 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 coal samples are analyzed.

Accessories

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

Method Reference

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

Reference Materials

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

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

Burn Profile

Burn Step	*Furnace Flow*	*Time*
1	5.00 LPM	30 s
2	1.00 LPM	180 s
3	5.00 LPM	End

Method Parameters*

Gas Type	Helium or Argon
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	No
Sample Drop Detection	Disabled

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

Element Parameters*

	Helium			Argon		
Parameter	*Carbon*	*Hydrogen*	*Nitrogen*	*Carbon*	*Hydrogen*	*Nitrogen*
Integration Delay	4 s	**	4 s	5 s	**	5 s
Starting Baseline	15 s	**	15 s	15 s	**	15 s
Post Baseline Delay	0 s	**	14 s	0 s	**	15 s
Use Comparator	No	**	No	No	**	No
Integration Time	13 s	**	35 s	16 s	**	60 s
Use Endline	Yes	**	Yes	Yes	**	Yes
Endline Delay	19 s	**	25 s	23 s	**	30 s
Ending Baseline	15 s	**	15 s	15 s	**	15 s
Use Hydrogen Correction	--	Yes	--	--	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.*

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 coal reference or sample material into a 502-186 Tin Foil Cup and seal the cup 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 and less than or equal to 0.005% (50 ppm) for all three elements when utilizing Argon 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 three).
      ii. Weigh an appropriate mass of a suitable reference material (pure compound) into a 502-186 Tin Foil Cup and seal the cup 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 acceptable tolerance range on the certificate.

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

   †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.

3. Drift Correction
   a. Condition the System
      i. Select two to five replicates in the Login screen.
      ii. Weigh ~0.1 g of a coal reference or sample material into a 502-186 Tin Foil Cup and seal the cup 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, and less than or equal to 0.005% (50 ppm) for all three elements when utilizing Argon as a carrier gas. Additional blanks beyond the recommended five 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 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 acceptable tolerance range on the certificate.

4. Sample Analysis
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh ~0.1 g of the coal sample into a 502-186 Tin Foil Cup and seal the cup 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 a linear, full regression calibration for Carbon determination, and linear, force through origin calibrations for Hydrogen and Nitrogen determination using fractional masses (0.05 g to 0.140 g) of LECO 502-642 (Lot 1019) Phenylalanine LCRM (65.47% C, 6.74% H, and 8.46% N). Drift Correction was performed utilizing ~0.1 g of LECO 502-642 (Lot 1019) Phenylalanine. The calibrations and drift corrections were verified using LECO 502-896 (Lot 1003) EDTA LCRM (41.08% C, 5.52% H, 9.58% N). Coal samples were analyzed as received and corrected for moisture in accordance with ASTM D5373.

	10 cm^3 Helium				**10 cm^3 Argon**			
	Mass (g)	*% C*	*% H*	*% N*	*Mass (g)*	*% C*	*% H*	*% N*
LECO 502-680	0.1006	82.3	4.24	1.19	0.1000	82.3	4.31	1.21
Lot# 20300	0.1081	82.5	4.23	1.19	0.1036	82.1	4.38	1.19
Prox-Plus Coal	0.0991	82.2	4.31	1.18	0.0960	81.6	4.44	1.21
82.1% ±1.2% C	0.0981	82.4	4.29	1.19	0.1012	82.3	4.30	1.22
4.26% ±0.36% H	0.1038	82.5	4.24	1.21	0.0982	82.2	4.29	1.22
1.17% ±0.10% N	***Avg =***	***82.4***	***4.26***	***1.19***	***Avg =***	***82.1***	***4.34***	***1.21***
	s =	***0.1***	***0.04***	***0.01***	***s =***	***0.3***	***0.07***	***0.01***
LECO 502-681	0.1062	77.2	5.06	1.47	0.1071	77.4	4.97	1.46
Lot# 20152	0.1029	77.2	5.04	1.47	0.1065	77.4	5.06	1.47
Prox-Plus Coal	0.1052	77.1	5.01	1.48	0.1000	77.3	5.09	1.48
76.9% ±1.0% C	0.1076	77.2	5.03	1.47	0.1052	77.4	5.05	1.45
4.96% ±0.33% H	0.1049	77.0	5.04	1.46	0.0990	77.9	5.12	1.47
1.43% ±0.08% N	***Avg =***	***77.1***	***5.04***	***1.47***	***Avg =***	***77.5***	***5.06***	***1.47***
	s =	***0.1***	***0.02***	***0.01***	***s =***	***0.2***	***0.05***	***0.01***
LECO 502-682	0.1004	70.2	4.86	1.56	0.1059	70.6	4.90	1.57
Lot# 20099	0.0972	70.5	4.93	1.57	0.1064	70.5	4.93	1.56
Prox-Plus Coal	0.0967	70.3	4.89	1.56	0.0947	70.5	5.00	1.56
71.1% ±1.7% C	0.1050	70.4	4.85	1.57	0.1022	70.5	4.90	1.56
4.93% ±0.38% H	0.1022	70.5	4.91	1.57	0.1014	70.5	4.92	1.55
1.55% ±0.10% N	***Avg =***	***70.4***	***4.89***	***1.57***	***Avg =***	***70.5***	***4.93***	***1.56***
	s =	***0.1***	***0.03***	***0.01***	***s =***	***<0.1***	***0.04***	***0.01***

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