Carbon, Hydrogen, and Nitrogen in Coal

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CHN628

Introduction

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

The LECO CHN628 is a combustion elemental carbon, hydrogen, and nitrogen instrument that utilizes only pure oxygen in the furnace, ensuring complete combustion and superior recovery of the elements of interest. A combustion gas collection and handling system lowers the overall cost-per-analysis and extends reagent lifetimes. Helium carrier gas sweeps the combustion gas to separate infrared cells utilized for the detection of H_2O and CO_2, while a thermal conductivity cell is used for the detection of nitrogen.

Method Reference ASTM D 5373

Sampling and Sample Preparation

A representative, uniform sample is required. Samples should be prepared in accordance to ASTM D2013. Coal reference materials such as those offered by LECO and NIST are properly prepared.

Accessories 502-186 Tin Foil Cup

Calibration

LECO 502-642 Phenylalanine, 502-092 EDTA, or other suitable pure compound

Note: ASTM Method D 5373-08 requires pure compounds be used for calibration.

Analysis Parameters

Combustion Furnace Temperature	950°C
Afterburner Temperature	850°C

Element Parameters

	Nitrogen	*Carbon*	*Hydrogen*
Analyze	Yes	Yes	Yes
Baseline Delay Time	10	0	0
Minimum Analysis Time	40	20	40
Comparator Level	100.00	100.00	100.00
Endline Time	2	1	1
Conversion Factor	1.00	1.00	1.00
Significant Digits	5	5	5

IR Baseline Time	1
TC Baseline Time	10

Burn Profile

Burn Steps	*Time*	*Furnace Flow*
1	30	High
2	180	Medium
3	30	High

Macro Ballast Parameters

Ballast

Equilibrate Time	30
Not Filled Timeout	600

Aliquot Loop

Equilibrate Pressure Time	8
Fill Pressure Drop	250

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Select 10 replicates.
   c. Initiate the analysis sequence (F5).
   d. Set blank using the last 5 results following the procedure outlined in the operator's instruction manual. Note: blank precision should be <0.001%.
3. Calibrate
   a. Weigh ~0.1 g of pure compound calibration sample (EDTA, Phenylalanine, BBOT, etc.) into a 502-186 Tin Foil Cup and seal.
   b. Enter sample mass and identification into Sample Login (F3).
   c. Transfer sample to the appropriate position of the sample carousel.
   d. Repeat steps 3a through 3c a minimum of five times.
   e. Initiate the analysis sequence (F5).
   f. Calibrate the instrument using single standard calibration (fixed at origin) following the procedure outlined in the operator's instruction manual.
   g. Verify the calibration by analyzing ~0.1 g of a pure compound different than the material used for calibration. For example, if Phenylalanine was used for calibration, verify the calibration using EDTA or BBOT.

   Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. Typically single-point calibration using a pure compound provides a suitable and cost-effective calibration. Refer to the operator's instruction manual for details regarding multi-point calibration.
4. Analyze Sample
   a. Weigh ~0.08 to 0.1 g of coal sample into 502-186 Tin Foil Cup and seal.
   b. Enter sample mass and identification into Sample Login (F3).
   c. Transfer sample to the appropriate position on the sample carousel.
   d. Initiate the analysis sequence (F5).

   Note: Coal should be analyzed "as received". Analytical values are corrected for moisture after analysis. Moisture should be determined within the same day the coal is analyzed.

Organic Application Note

Delivering the Right Results

Typical Results

(Based on single standard calibration with 0.1 g of 502-642 Phenylalanine)

Sample	*Mass (g)*	*Carbon*	*Hydrogen*	*Nitrogen*
LECO 502-092	0.1010	41.09	5.63	9.58
EDTA Lot 1055	0.1070	41.06	5.58	9.57
41.06 ±0.09% C	0.1027	41.08	5.56	9.58
5.55 ±0.03% H	0.1004	41.11	5.57	9.58
9.56 ±0.03% N	0.1010	41.05	5.56	9.57
	X =	***41.08***	***5.58***	***9.58***
	s =	***0.02***	***0.03***	***0.01***
LECO 502-680	0.0851	80.57	4.07	1.06
lvb Coal*	0.0868	80.68	4.04	1.07
81.4 ±2.3% C	0.0857	80.60	4.00	1.07
4.06 ±0.26% H	0.0859	80.62	4.02	1.07
1.10 ±0.15% N	0.0860	80.49	3.98	1.07
	X =	***80.59***	***4.02***	***1.07***
	s =	***0.07***	***0.04***	***0.003***
LECO 502-681	0.0852	77.79	4.92	1.46
hvAb Coal**	0.0859	77.70	4.93	1.46
78.4 ±0.9% C	0.0859	77.82	4.92	1.46
4.98 ± 0.14% H	0.0861	77.84	4.94	1.46
1.46% ±0.08% N	0.0856	77.81	4.91	1.46
	X =	***77.81***	***4.92***	***1.46***
	S =	***0.07***	***0.01***	***0.001***

**Low Volatile Bituminous Coal, Alberta Canada.*
***High Volatile A Bituminous Coal, West Virginia, USA.*

LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • ISO-9001:2008 HQ-Q-994 • ***LECO is a registered trademark of LECO Corporation.***

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