Carbon, Hydrogen, and Nitrogen in Hydrocarbons

LECO Corporation; Saint Joseph, Michigan USA

Instrument: CHN628

Introduction

Carbon, Hydrogen, and Nitrogen determination is part of the ultimate analysis of hydrocarbon materials, helping to characterize the materials and provide information that can be utilized in calculating material/energy balances and efficiencies, as well as emissions potentials if the material is utilized as a fuel. The Carbon, Hydrogen, and Nitrogen results for oil materials are also utilized to evaluate the refining potentials and yields in the petrochemical industry.

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 lifetime. 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.

Accessories

502-186 Tin Foil Cup, 501-427 Com-Aid™

Calibration Samples

502-654 BBOT (2,5-di (5-tert-butylbenzoxazol-2-yl) thiophene), 502-642 Phenylalanine, 501-050 Nicotinic Acid, or other suitable pure compounds.

*Analysis Parameters**

Furnace Temperature	950°C
Afterburner Temperature	850°C

Element Parameters

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

IR Baseline Time	1 second
TC Baseline Time	10 seconds

Burn Profile

Burn Steps	*Time (seconds)*	*Furnace Flow*
1	40 seconds	High
2	30 seconds	Medium
3	30 seconds	High

Ballast Parameters

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Fill Pressure Drop	200 mm Hg
Equilibrate Pressure Time	8 seconds

**Refer to CHN628 Operator's Instruction Manual for Analysis Parameter definitions.*

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 the blank using at least 5 results following the procedure outlined in the operator's instruction manual.
   e. The standard deviation of the last 5 blanks for all three elements should be less than or equal to 0.002% (20 ppm). Additional blanks beyond the recommended 10 may need to be analyzed in order to achieve the recommended precision.
3. Calibrate.
   a. Weigh ~0.1 g of a pure compound calibration sample (BBOT, Phenylalanine, Nicotinic Acid, 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 in 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 material different than the material used for calibration.

Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. Typically, a single-point calibration using a pure compound provides a suitable and cost-effective calibration. Refer to the operator's instruction manual for details regarding the multi-point calibration procedure.

Organic Application Note

4. Analyze Samples.
   a. Weigh ~0.07 g of a hydrocarbon (oil) into a 502-186 Tin Foil Cup.
   b. Enter mass and sample identification into Sample Login (F3).
   c. Add ~0.3 g of 501-427 Com-Aid on top of sample and seal the foil cup.
   d. Transfer sample to the appropriate position on the sample carousel.
   e. Repeat steps 4a through 4d for each sample to be analyzed.
   f. Initiate the analysis sequence (F5).

Note: If the viscosity of the hydrocarbon (oil) makes it difficult to seal the tin foil due to capillary action of the hydrocarbon along the creases of the tin foil cup, the following alternative sampling procedure may be undertaken:

1. Before weighing ~0.07 g of the hydrocarbon into the tin foil cup, add ~0.2 g of the 501-427 Com-Aid into the foil cup and create a small depression (crater) in the center and tare the balance.
2. Weigh ~0.07 g of the hydrocarbon (oil) into the depression made in the 501-427 Com-Aid.
3. Enter mass and sample identification into Sample Login (F3).
4. Add remaining ~0.1 g of the 501-427 Com-Aid on top of the sample and seal the foil cup.
5. Transfer sample to the appropriate position on the sample carousel.
6. Initiate the analysis sequence (F5).

Typical Results

(Based on a single standard calibration with 0.1 g of 502-654 BBOT)

Sample	*Mass (g)*	*% Carbon*	*% Hydrogen*	*% Nitrogen*
Residual Fuel Oil				
LECO 502-816	0.0717	86.3	10.52	0.322
86% Carbon	0.0721	86.1	10.49	0.324
10.6% Hydrogen	0.0702	85.9	10.50	0.316
0.31% Nitrogen	0.0723	86.2	10.52	0.298
	0.0726	86.2	10.51	0.325
	0.0719	86.1	10.52	0.318
	0.0732	86.3	10.54	0.332
	0.0728	86.3	10.56	0.283
	0.0715	86.5	10.57	0.301
	0.0709	86.3	10.57	0.321
	X=	***86.2***	***10.53***	***0.314***
	s=	***0.2***	***0.03***	***0.015***

Sample	*Mass (g)*	*% Carbon*	*% Hydrogen*	*% Nitrogen*
Paraffin Oil				
LECO 501-439	0.0755	86.92	13.77	-
86.34% Carbon	0.0784	86.80	13.73	-
13.80% Hydrogen	0.0728	86.83	13.84	-
<0.03% Nitrogen	0.0800	87.08	13.79	-
	0.0798	86.72	13.77	-
	0.0782	86.94	13.83	-
	0.0753	86.90	13.88	-
	0.0723	87.00	13.92	-
	0.0762	86.97	13.83	-
	0.0782	87.05	13.88	-
	X=	***86.92***	***13.82***	
	s=	***0.11***	***0.06***	

LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
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