

Carbon, Hydrogen, and Nitrogen in Coke and Graphite

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruSpec[®] CHN

Method Reference

ASTM D 5373

Sampling and Sample Preparation

It is essential that a representative and uniform sample be analyzed. Samples should be prepared in accordance to ASTM D2013. Coke reference materials such as those offered by LECO and NIST are properly prepared.

Accessories 502-186 Tin Foil Cup

Sample Mass 0.05 to 0.09 gram

Calibration

LECO 502-642 Phenylalanine, 502-092 EDTA, or other suitable reference materials.

Analysis Parameters

Combustion Furnace Temperature 950°C
Afterburner Temperature 850°C

Element Parameters

	Nitrogen	Carbon	Hydrogen
Analyze	Yes	Yes	Yes
Minimum Analysis Time (s)	30	NA	NA
Comparator Level	1.00	NA	NA
Endline Time (s)	1	NA	NA
Conversion Factor	1.00	1.00	1.00
Significant Digits	5	5	5
IR Analysis Stabilize Comparator		0.00	
IR Baseline Delay Time (s)		5	
IR Baseline Time (s)		2	
IR Pressure Stabilization Comparator		0.00	
IR Stop Flow Time (s)		5	
TC Baseline Delay Time (s)		5	
TC Baseline Time (s)		2	

Burn Profile

Burn Steps	Time	Furnace Flow
1	15 seconds	High
2	300 seconds	Medium
3	5 seconds	High

Macro Ballast Parameters

Ballast

Equilibrate Time 30 seconds
Not Filled Timeout 600 seconds

Aliquot Loop

Fill Time 20 seconds
Equilibrate Pressure Time 4 seconds



Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine and calibrate systems blank as outlined in the operator's instruction manual.
3. Instrument must be calibrated as outlined in the operator's instruction manual.
4. Perform Drift Correction as outlined in the operator's instruction manual.

Note: Drift should be performed at the start of every day or when the check standard doesn't return the correct result(s).

Weigh a 0.05 to 0.09 gram coke sample into 502-186 Tin Foil Cup, seal, enter sample identification and mass into Sample Login (F3), place into the appropriate position of the sample carousel, and proceed with analysis.

Note: Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil. This will cause biased nitrogen results at low nitrogen concentrations. Therefore, an atmospheric blank should be determined and entered. The atmospheric nitrogen blank can be determined by analyzing several replicates of LECO 501-073 Graphite Powder (dried at 105°C for 1 hour prior to use), using weights similar to those of the samples being analyzed. The average nitrogen value of the 501-073 is considered the atmospheric blank and can be automatically compensated using the TruSpec software. Refer to the operator's instruction manual for details regarding the setting of the atmospheric blank.

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Organic Application Note

Typical Results (Dry Basis)

Sample	Weight g	Carbon %	Hydrogen %	Nitrogen %
LECO	0.0855	92.58	3.90	1.43
502-452 @	0.0850	92.96	3.97	1.43
93.30 % C	0.0845	92.93	3.96	1.43
3.94 % H	0.0856	93.10	4.02	1.43
1.43 % N	0.0852	92.91	3.99	1.42
Pet. Coke	0.0858	93.12	3.99	1.43
	0.0844	92.92	3.95	1.42
	0.0852	93.22	3.98	1.44
	0.0851	92.86	3.99	1.43
	0.0850	92.91	3.98	1.44
	X =	92.95	3.97	1.43
	s =	0.17	0.03	0.01

Note: Results based on multi-point calibration with LECO and NIST reference materials.