Carbon/Nitrogen in Coal, Coke, Carbon Black, and Graphite

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruMac® CN

Sample Preparation

Samples must be of uniform consistency to produce suitable results, preferably minus 60 mesh. Carbon and nitrogen results for coal, coke and select reference materials are typically reported on a dry basis. Therefore, either the materials must be dried prior to analysis or the moisture content determined and the values corrected. Please see the note addendum at the end of this document for details on drying these materials.

Accessories

528-203 Crucibles

Calibration Samples

502-092 EDTA, 502-642 Phenylalanine, 502-654 BBOT, 502-634 High Purity Graphite Powder

*Analysis Parameters**

Furnace Temperature	1350°C
TE Cooler Temperature	5°C
Dehydration Time	0 seconds
Purge Cycles	3 seconds

Element Parameters

	Carbon	*Nitrogen*
Baseline Delay Time	0 seconds	6 seconds
Minimum Analysis Time	15 seconds	35 seconds
Comparator Level	100.00	100.00
Endline Time	2 seconds	2 seconds
Conversion Factor	1.00	1.00
Significant Digits	5	5
TC Baseline Time	—	10 seconds
IR Analysis Stabilize Comparator	0	—
IR Baseline Time	1 second	—

Burn Profile

Burn Cycle	*Lance Flow*	*Purge Flow*	*Time*
1	Off	On	5 seconds
2	On	On	5 seconds
3	On	Off	END

Ballast Parameters

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Equilibrate Pressure Time	4 seconds
High Precision	Yes
High Speed	No

**Refer to TruMac Operator's Instruction Manual for Method Parameter definitions.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing 3-5 blanks (crucible is not required).
3. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Place a 528-203 Crucible to the appropriate position of the autoloader.
   c. Repeat steps 3a through 3b a minimum of three times.
   d. Initiate the analysis sequence (F5).
   e. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate.
   a. Weigh ~0.50 g of EDTA calibration sample into a 528-203 crucible, enter mass and sample identification into Sample Login (F3).
   b. Transfer crucible to the appropriate position of the autoloader.
   c. Repeat steps 4a through 4b a minimum of three times.
   d. Initiate the analysis sequence (F5).
   e. Calibrate the instrument following the procedure outlined in the operator's instruction manual.

   Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. A TruMac can be calibrated using several replicates of a single mass range (nominal 0.50 g) of EDTA utilizing a single standard calibration. This is a cost-effective and simple process. The calibration can be verified by analyzing different compounds such as BBOT (0.25 g), phenylalanine (0.25 g) and/or high purity graphite (0.25 g).
5. Analyze Samples.
   a. Weigh ~0.25 g sample into a 528-203 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Transfer crucible to the appropriate position of the autoloader.
   c. Repeat steps 5a through 5b for each sample to be analyzed.
   d. Initiate the analysis sequence (F5).

Organic Application Note

Delivering the Right Results

Notes

- If soot (carbon black) is noticed in the primary filter (steel wool filter), reduce sample mass to promote complete combustion and prevent soot build-up in this filter.
- Carbon and nitrogen results for coal, coke and select reference materials are typically reported on a dry basis. Therefore, either the materials must be dried prior to analysis or the moisture content determined and the values corrected.
  - Graphite—Samples must be dried at 105°C for one hour prior to analysis.
  - Carbon Black—Samples must be dried at 125°C for one hour prior to analysis.
  - Coal and Coke—A moisture correction must be determined by oven drying a separate 1 g sample @ 107 ±3°C for one hour.

Typical Results

(Based on a single standard calibration with 0.5 g of 502-092 EDTA)

Sample	Mass g	% N	% C
Graphite*	0.2510	0.0043	100.2
	0.2523	0.0054	100.0
	0.2503	0.0040	100.1
	0.2560	0.0061	100.1
	0.2551	0.0039	100.2
	0.2515	0.0034	100.4
	0.2504	0.0044	100.3
	0.2531	0.0044	100.2
	0.2500	0.0048	100.2
	0.2509	0.0038	100.0
	X=	***0.0045***	***100.2***
	s=	***0.0008***	***0.12***
Carbon Black**	0.2564	0.2080	94.16
	0.2507	0.2121	93.86
	0.2504	0.2194	93.44
	0.2550	0.2158	93.50
	0.2506	0.2132	93.59
	0.2550	0.2207	93.62
	0.2506	0.2106	93.61
	0.2505	0.1965	93.61
	0.2522	0.2009	93.83
	0.2504	0.1970	94.15
	X=	***0.2094***	***93.74***
	s=	***0.0088***	***0.26***
Coal***	0.2597	1.515	77.61
	0.2488	1.458	77.67
	0.2536	1.446	77.61
	0.2506	1.455	77.70
	0.2543	1.457	77.76
	0.2520	1.448	77.67
	0.2563	1.456	77.81
	0.2520	1.455	77.48
	0.2579	1.452	77.60
	0.2526	1.455	77.46
	0.2569	1.457	77.55
	X=	***1.459***	***77.63***
	s=	***0.019***	***0.11***
Pet Coke***	0.2508	1.514	87.95
	0.2513	1.515	87.95
	0.2518	1.514	87.94
	0.2511	1.513	87.99
	0.2533	1.514	88.00
	0.2548	1.514	88.05
	0.2537	1.514	88.15
	0.2518	1.513	88.04
	0.2507	1.511	87.92
	0.2510	1.515	88.15
	X=	***1.514***	***88.01***
	s=	***0.001***	***0.08***
Met Coke***	0.2609	1.053	88.57
	0.2587	1.054	88.57
	0.2555	1.053	88.49
	0.2500	1.054	88.45
	0.2596	1.052	88.40
	0.2516	1.052	88.41
	0.2680	1.052	88.29
	0.2547	1.054	88.54
	0.2657	1.053	88.48
	0.2516	1.049	88.26
	X=	***1.053***	***88.45***
	s=	***0.001***	***0.11***

**Samples must be dried at 105°C for one hour prior to analysis.*
***Samples must be dried at 125°C for one hour prior to analysis.*
****A moisture correction must be determined by oven drying a separate 1 g sample @ 107 ±3°C for one hour.*

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