

Instrument: CN828

Determination of Carbon and Nitrogen in Soil

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Introduction

Carbon content in soils can represent the presence of organic matter and is used to estimate nitrogen availability from the natural decay of organic materials, especially when using organic fertilizers. Nitrogen is one of the most important elements for plant development and is the nutrient that is most often found to be deficient for crop production in arable soils. Nitrogen is considered an essential macronutrient for proper plant development, playing a key role in the formation of enzymes and proteins. While carbon in almost any form is a benefit to the soil, it helps enormously if it is accompanied by the right ratio of nitrogen. Testing to determine the content of carbon and nitrogen in the soil allows growers to adjust these ratios, if needed, through fertilization. Often a combination of carbon and nitrogen determination in both the crop plant tissue and surrounding soil will be used to diagnose and correct any nutritional-related growth issues. Testing for total carbon and nitrogen in arable soils can provide valuable information regarding fertilization needs that can be used to make fertilization management decisions for the soil.

Instrument Model and Configuration

The LECO CN828 is a combustion carbon and nitrogen determinator that utilizes a pure oxygen environment in a vertical quartz furnace, ensuring complete combustion and superior analyte recovery. A thermoelectric cooler removes moisture from the combustion gases before they are collected in a ballast. The combustion gases equilibrate and mix in the ballast before a representative aliquot (3 cm³ or 10 cm³ 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 a non-dispersive infrared (NDIR) cell for the detection of carbon (as CO₂) and a thermal conductivity cell (TC) for the detection of nitrogen (N₂).

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 sensitivity of the detector. The CN828 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. 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.

The CN828 offers the additional advantage of utilizing either a 10 cm³ aliquot loop or a 3 cm³ aliquot loop within the instrument's gas collection and handling system. The 10 cm³ aliquot loop optimizes the system for the lowest nitrogen range and provides the best

precision. The 3 cm³ aliquot loop extends reagent life expectancy by approximately three-fold when compared to the 10 cm³ aliquot loop, while providing the lowest cost-per-analysis.

Note: When changing carrier gas type, refer to the 828 Series Operator's Instruction Manual for the procedure on setting the gas flow rate. The aliquot loop size is changed by selecting the desired aliquot loop size in the software's Method Parameters.

Method Reference

ISO 13878: Soil Quality - Determination of Total Nitrogen Content by Dry Combustion.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Reference materials should be prepared as directed by the certificate, prior to analysis.

Note: Carbon and Nitrogen results for soil samples are typically reported on a dry basis in order to avoid a reporting bias due to fluctuations in moisture levels. Therefore, either the material can be dried prior to analysis, or the moisture content can be determined and entered into the software to correct for moisture. Soil samples are typically dried at 105 °C for one hour prior to analysis. The dried samples should be stored in a desiccator and must be used for analysis within 24 hours. For Soil Reference Materials, follow the sample drying instructions provided by the certificate.

Accessories

502-186 Tin Foil Cups, 501-427 Com-Aid, and 501-614 Spatula

Reference Materials

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

Method Parameters*

Gas Type	Helium or Argon
Furnace Temperature	950 °C
Afterburner Temperature	850 °C
Nominal Mass	1.0000 g
Purge Cycles	3
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
Interleave Analysis	Yes
Sample Drop Detection	Disabled
Dose Loop Size**	10 cm ³ or 3 cm ³

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

**Due to the low nitrogen levels typically found in soils, the 10 cm³ dose loop is recommended for this application when utilizing argon as a carrier gas.

Element Parameters[†]

Parameter	Helium		Argon	
	Carbon	Nitrogen	Carbon	Nitrogen
Integration Delay	4 s	4 s	4 s	4 s
Starting Baseline	15 s	15 s	15 s	15 s
Post Baseline Delay	0 s	14 s	0 s	20 s
Use Comparator	No	No	No	No
Integration Time	18 s	50 s	25 s	65 s
Use Endline	Yes	Yes	Yes	Yes
Endline Delay	15 s	20 s	15 s	20 s
Ending Baseline	15 s	15 s	15 s	15 s
Use Profile Blank	--	--	--	Yes

[†]Refer to the 828 Series Operator's Instruction Manual for Parameter definitions.

Burn Profile

Burn Step	Furnace Flow	Time
1	5.00 L/min	20 s
2	1.00 L/min	180 s
3	5.00 L/min	End

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the System.
 - a. Select five or more Blank replicates in the Login screen.
 - b. Initiate the analysis sequence.
3. Determine Blank.
 - a. Select five or more Blank replicates in the Login screen.
 - b. Initiate the analysis sequence.
 - c. Set the blank 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 both carbon and nitrogen when utilizing Helium as a carrier gas, and less than or equal to 0.005% (50 ppm) for both carbon and nitrogen when utilizing Argon as a carrier gas. Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.
4. Calibrate/Drift Correct.
 - a. Select the desired number of calibration/drift replicates in the Login screen (minimum of five).
 - b. Weigh 0.05 g to 0.25 g of a suitable reference material into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize entrapped atmosphere by twisting the top edges of the foil together.
 - c. Enter sample mass and identification into the Login screen.
 - d. Transfer the tin foil cup containing the reference material to the appropriate position in the sample carousel.
 - e. Perform steps 4b through 4d a minimum of five times.
 - f. Initiate the analysis sequence.
 - g. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

- h. Verify the calibration/drift correction by analyzing an appropriate mass of another/different suitable reference material and confirm that the results are within the acceptable tolerance range.

Note: Typically, the CN828 can be calibrated using several replicates of a single mass range of a suitable reference material utilizing a linear, force through origin calibration. This is a cost-effective and simple process.

5. Analyze Samples.
 - a. Select the desired number of sample replicates in the Login screen.
 - b. Weigh ~0.25 g of the soil sample into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize entrapped atmosphere 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 5b through 5d for each sample to be analyzed.
 - f. Initiate the analysis sequence.
6. Atmospheric Blank Determination.

Note: Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. This will cause biased nitrogen results at low nitrogen concentrations (particularly with soil samples). Therefore, an atmospheric blank should be determined and entered using the following procedure.

- a. Select the desired number of sample replicates in the Login screen (minimum of three).
- b. Weigh a similar mass (to the mass of the samples being analyzed) of an inert material such as LECO 501-427 Com-Aid^{††} into a 502-186 Tin Foil Cup and seal the cup in a manner to minimize entrapped atmosphere by twisting the top edges of the foil together.
- c. Enter the mass of the Com-Aid into the Login screen.
- d. Transfer the tin foil cup containing the Com-Aid to the appropriate position in the sample carousel.
- e. Perform steps 6b through 6d for each Com-Aid sample to be analyzed.
- f. Initiate the analysis sequence.
- g. The average nitrogen value obtained is considered the atmospheric blank and can be automatically compensated for using the CN828 software[‡].

^{††}Com-Aid should be baked-off in a muffle furnace at ~1000 °C for 15 minutes, allowed to cool, and stored for up to 24 hours in a desiccator prior to use.

[‡]Refer to the 828 Series Operator's Instruction Manual for details regarding the setting of the atmospheric blank.

TYPICAL RESULTS

Data was generated utilizing a linear, force through origin calibration using ~0.10 g of 502-694 Soil LCRM Lot# 1002 (13.50% C, 1.13% N). Soil samples were dried at 105 °C for one hour prior to analysis and stored in a desiccator until use. An atmospheric blank was determined and utilized to compensate for entrapped atmosphere using the instrument's software.

	Helium 10 cm ³			Helium 3 cm ³			Argon 10 cm ³		
	Mass (g)	% C	% N	Mass (g)	% C	% N	Mass (g)	% C	% N
Soil LCRM	0.2516	3.75	0.321	0.2525	3.75	0.324	0.2530	3.75	0.325
LECO 502-697	0.2525	3.75	0.317	0.2504	3.78	0.322	0.2526	3.76	0.323
Lot: 1001	0.2536	3.76	0.320	0.2532	3.78	0.331	0.2534	3.78	0.322
% C = 3.79 ± 0.05	0.2511	3.75	0.320	0.2551	3.77	0.320	0.2540	3.75	0.322
% N = 0.328 ± 0.038	0.2535	3.74	0.320	0.2525	3.76	0.325	0.2537	3.74	0.326
	Avg =	3.75	0.320	Avg =	3.77	0.324	Avg =	3.76	0.324
	s =	0.01	0.002	s =	0.01	0.004	s =	0.01	0.002
Soil LCRM	0.2543	0.736	0.096	0.2525	0.732	0.093	0.2525	0.738	0.104
LECO 502-962	0.2510	0.724	0.096	0.2541	0.727	0.106	0.2527	0.728	0.106
Lot: 1000	0.2521	0.716	0.098	0.2536	0.713	0.106	0.2535	0.714	0.105
% C = 0.717 ± 0.027	0.2520	0.719	0.099	0.2512	0.711	0.095	0.2548	0.705	0.094
% N = 0.101 ± 0.009	0.2510	0.722	0.102	0.2522	0.722	0.108	0.2528	0.721	0.096
	Avg =	0.724	0.098	Avg =	0.721	0.102	Avg =	0.721	0.101
	s =	0.008	0.003	s =	0.009	0.007	s =	0.012	0.006
San Joaquin Soil	0.2536	1.15	0.118	0.2522	1.12	0.095	0.2535	1.12	0.105
NIST SRM 2709	0.2547	1.13	0.114	0.2548	1.11	0.114	0.2535	1.12	0.110
	0.2522	1.13	0.112	0.2527	1.13	0.110	0.2542	1.12	0.102
	0.2541	1.12	0.113	0.2548	1.14	0.110	0.2528	1.12	0.131
	0.2525	1.12	0.108	0.2541	1.13	0.102	0.2537	1.12	0.106
	Avg =	1.13	0.113	Avg =	1.12	0.106	Avg =	1.12	0.111
	s =	0.01	0.003	s =	0.01	0.008	s =	< 0.01	0.011



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