Application Note

Instrument: CN928

Determination of Carbon/Nitrogen in Soils and Plant Tissue

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Introduction

Determination of carbon and nitrogen concentrations in crop plant tissue provides an important diagnostic tool to the grower, giving an indication of nutritional health and nutrient uptake efficiency from the soil, as well as providing an avenue for monitoring high-value, intensively managed crops such as tobacco, cotton, and fruits. 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. Nitrogen is considered an essential macronutrient for plant development, playing a key role in the formation of enzymes and proteins. 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. Testing for total carbon and nitrogen in arable soils is most useful just before the planting season in order to predict fertilization needs, and make fertilization management decisions for the soil.

The LECO CN928 is a macro combustion carbon and nitrogen/protein determinator that utilizes a pure oxygen environment in a high-temperature horizontal ceramic combustion furnace designed to handle macro sample mass. A thermoelectric cooler removes moisture from the combustion gases before they are collected in a ballast. The gases equilibrate and mix in the ballast before a representative aliquot (3 cm^3 or 10 cm^3 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 detection of carbon (as CO_2), and a thermal conductivity cell (TC) for the detection of nitrogen (N_2).

Instrument Model and Configuration

Thermal conductivity detectors work by detecting changes in the thermal conductivity of the analyte gas compared to the constant thermal conductivity of the 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 CN928 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. Argon can also be used as a carrier gas. 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 CN928 offers the additional advantage of utilizing either a 10 cm^3 aliquot loop or a 3 cm^3 aliquot loop within the instrument's gas collection and handling system. The 10 cm^3 aliquot loop optimizes the system for the lowest nitrogen range and provides the best precision. The 3 cm^3 aliquot loop extends reagent life expectancy by approximately three fold when compared to the 10 cm^3 aliquot loop, while providing the lowest cost-per-analysis with minimal impact on practical application performance (see Typical Results section).

Note: When changing carrier gas type, the flow needs to be adjusted following instructions provided in the CN928 Operator's Instruction Manual. The aliquot loop size is changed by selecting the desired aliquot loop size in the software's Method Parameters.

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: Nitrogen and Carbon results for soil and plant tissue samples are typically reported on a dry basis. Therefore, either the materials 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 and Plant samples are typically dried at 85 °C for two hours prior to analysis. The dried samples should be stored in a desiccator and must be used for analysis within 24 hours.

Accessories

528-203 Ceramic Combustion Boats*, 761-929 Crucible Tongs, and 501-614 Spatula

**Note: For optimal precision, ceramic combustion boats should be baked in a muffle furnace at 1000 °C for a minimum of 40 minutes. Once the ceramic combustion boats have cooled, they should be transferred to a desiccator for storage. If the ceramic combustion boats are not used within twenty-four hours, they should be re-baked. After baking, handle ceramic combustion boats with clean tongs only; do not use fingers.*

Reference Materials

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

*Analysis Parameters**

Gas Type	Helium or Argon
Furnace Temperature	1350 °C
Dehydration Time	0 s
Nominal Mass	1.0000 g
Purge Cycles	3

*Ballast Parameters**

Ballast Equilibrate Time	10 s
Ballast Not Filled Timeout	300 s
Aliquot Loop Fill Pressure Drop	200 mm Hg
Aliquot Loop Equilibrate Time	4 s
Dose Loop Size	10 cm^3 or 3 cm^3

Element Parameters*

	Helium (10 cm^3 & 3 cm^3)		Argon (10 cm^3 & 3 cm^3)	
	Carbon	Nitrogen	Carbon	Nitrogen
Wait for Baseline Stability	Yes	---	Yes	---
Integration Delay	20 s	0 s	15 s	9 s
Starting Baseline	1 s	10 s	1 s	10 s
Post Baseline Delay	0 s	28 s	5 s	20 s
Use Comparator	No	No	No	No
Integration Time	22 s	40 s	22 s	61 s
Use Endline	No	Yes	Yes	Yes
Endline Delay	---	30 s	0 s	30 s
Ending Baseline	---	5 s	10 s	5 s
Use Profile Blank	---	No	---	No

**Refer to CN928 Operator's Instruction Manual for Parameter definitions.*

Burn Profile

Burn Step	Lance Flow	Furnace Flow	Time
1	No	Yes	5 s
2	Yes	Yes	5 s
3	Yes	No	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 (ceramic combustion boat is not required).
   b. Initiate the analysis sequence.

   Note: The standard deviation of the last three blanks should be less than or equal to 0.001% (10 ppm) using He carrier gas; or 0.005% (50 ppm) using Argon carrier gas. Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.
3. Determine Blank.
   a. Select five Blank replicates in the Login screen.
   b. Place 528-203 Ceramic Combustion Boats in the appropriate positions in the autoloader.
   c. Initiate the analysis sequence.
   d. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate/Drift Correct.
   a. Select the desired number of calibration/drift replicates in the Login screen (minimum of five).
   b. Weigh ~0.50 g of a suitable reference material into a 528-203 Ceramic Combustion Boat.
   c. Enter sample mass and identification into the Login screen.
   d. Transfer the Ceramic Combustion Boat containing the sample to the appropriate position in the autoloader.
   e. Repeat steps 4b through 4d a minimum five times for each calibration/drift sample used.
   f. Initiate the analysis sequence
   g. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

   Note: A multi-point calibration (fractional weight or multiple calibration samples) may be used to calibrate if desired. Typically, the CN928 can be calibrated utilizing several replicates of a single mass range (nominal 0.50 g) of 502-896 EDTA utilizing a single standard calibration (linear forced through origin calibration). This is a cost effective and simple process. The calibration can be verified by analyzing different compounds such as 502-688 nicotinic acid (0.10 to 0.25 g), 502-642 phenylalanine (0.10 to 0.25 g), and/or (~ 0.50 g) of a known flour or soil standard.
5. Analyze Samples.
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh ~0.50 g of the unknown sample into a 528-203 Ceramic Combustion Boat.
   c. Enter mass and identification information into the Login screen.
   d. Transfer the Ceramic Combustion Boat containing the sample to the appropriate position in the autoloader.
   e. Repeat steps 5b through 5d for each sample to be analyzed.
   f. Initiate the analysis sequence.

TYPICAL RESULTS

Data was generated utilizing a single standard calibration using 0.50 g of LECO 502-896 (Lot 1001) EDTA (41.00% C and 9.56% N). The calibration was verified using 502-688 (Lot 1001) Nicotinic Acid (58.53% C, 11.37% N). Samples were analyzed using baked-off ceramic combustion boats.

	10 cm^3 Helium			3 cm^3 Helium			10 cm^3 Argon			3 cm^3 Argon		
	Mass (g)	*% C*	*% N*	*Mass (g)*	*% C*	*% N*	*Mass (g)*	*% C*	*% N*	*Mass (g)*	*% C*	*% N*
Soil LRM	0.5077	22.7	1.95	0.5063	22.7	1.95	0.4989	22.3	1.94	0.5000	22.4	1.92
LECO 502-814*	0.5014	22.7	1.95	0.5084	22.7	1.95	0.5091	22.4	1.94	0.4957	22.5	1.94
Lot: 1002	0.4993	22.7	1.95	0.5044	22.7	1.95	0.5055	22.4	1.95	0.4946	22.5	1.93
% C = 22.6 ±0.3	0.5083	22.6	1.94	0.4968	22.7	1.93	0.5101	22.4	1.95	0.4951	22.4	1.93
% N = 1.93 ±0.05	0.5049	22.7	1.95	0.5031	22.7	1.95	0.5043	22.3	1.94	0.5002	22.4	1.91
	Avg =	***22.7***	***1.95***	***Avg =***	***22.7***	***1.95***	***Avg =***	***22.4***	***1.94***	***Avg =***	***22.4***	***1.93***
	s =	***0.1***	***<0.01***	***s =***	***<0.1***	***0.01***	***s =***	***<0.1***	***<0.01***	***s =***	***<0.1***	***0.01***
Soil LCRM	0.4937	3.82	0.331	0.5199	3.85	0.324	0.4987	3.75	0.346	0.5072	3.84	0.306
LECO 502-697**	0.5048	3.84	0.332	0.5150	3.83	0.321	0.5000	3.78	0.341	0.5059	3.84	0.304
Lot: 1000	0.5017	3.82	0.332	0.5203	3.81	0.320	0.4990	3.77	0.346	0.4999	3.85	0.331
% C = 3.82 ±0.07	0.5021	3.81	0.330	0.5158	3.86	0.322	0.4958	3.76	0.340	0.5014	3.85	0.339
% N = 0.323 ±0.032	0.5085	3.80	0.331	0.5257	3.90	0.325	0.4995	3.77	0.338	0.5056	3.82	0.348
	Avg =	***3.82***	***0.331***	***Avg =***	***3.85***	***0.322***	***Avg =***	***3.77***	***0.342***	***Avg =***	***3.84***	***0.326***
	s =	***0.02***	***0.001***	***s =***	***0.03***	***0.002***	***s =***	***0.01***	***0.004***	***s =***	***0.01***	***0.020***
Soil LRM	0.5049	11.98	0.97	0.5047	12.09	0.96	0.5014	11.93	0.96	0.4982	11.72	0.92
LECO 502-309**	0.5047	12.02	0.97	0.5060	12.05	0.95	0.5009	11.90	0.97	0.4961	11.78	0.96
Lot: 1012	0.5079	11.88	0.96	0.5002	12.24	0.96	0.5054	11.89	0.95	0.4939	11.92	0.96
% C = 11.98 ±0.44	0.5017	11.96	0.96	0.5031	12.09	0.95	0.5049	11.92	0.97	0.5054	11.82	0.98
% N = 0.93 ±0.04	0.5041	11.87	0.96	0.4985	12.02	0.95	0.5110	11.89	0.97	0.5089	11.97	0.96
	Avg =	***11.94***	***0.96***	***Avg =***	***12.10***	***0.95***	***Avg =***	***11.91***	***0.97***	***Avg =***	***11.84***	***0.96***
	s =	***0.07***	***0.01***	***s =***	***0.08***	***0.01***	***s =***	***0.02***	***0.01***	***s =***	***<0.01***	***0.02***
Tobacco LRM	0.5040	47.44	2.49	0.5064	47.20	2.49	0.5044	47.44	2.51	0.5065	47.11	2.48
LECO 502-082†	0.5145	47.36	2.48	0.4990	47.13	2.49	0.4909	47.43	2.51	0.5024	47.23	2.50
Lot 1018	0.5127	47.37	2.48	0.4983	47.08	2.48	0.5035	47.45	2.51	0.4973	47.15	2.47
% C = 47.27 ±0.23	0.5138	47.27	2.47	0.5045	47.24	2.49	0.5102	47.41	2.51	0.5019	47.21	2.50
% N = 2.48 ±0.04	0.5037	47.44	2.48	0.5034	47.16	2.49	0.5081	47.40	2.51	0.4945	47.14	2.48
	Avg =	***47.38***	***2.48***	***Avg =***	***47.16***	***2.49***	***Avg =***	***47.43***	***2.51***	***Avg =***	***47.17***	***2.49***
	s =	***0.07***	***<0.01***	***s =***	***0.06***	***<0.01***	***s =***	***0.02***	***<0.01***	***s =***	***0.05***	***0.01***
Alfalfa LRM	0.4967	45.12	3.60	0.4926	44.96	3.61	0.4955	45.16	3.64	0.5062	44.88	3.60
LECO 502-273†	0.5005	45.14	3.61	0.4960	45.02	3.63	0.5011	45.13	3.64	0.5060	44.88	3.59
Lot 1026	0.5016	45.03	3.60	0.4953	44.87	3.60	0.5014	45.09	3.63	0.5026	44.87	3.62
% C = 45.05 ±0.23	0.5058	45.17	3.61	0.5053	44.89	3.62	0.5038	45.10	3.63	0.4973	44.84	3.63
% N = 3.62 ±0.05	0.5018	45.23	3.61	0.5029	44.92	3.61	0.5012	45.10	3.62	0.4913	44.94	3.61
	Avg =	***45.14***	***3.61***	***Avg =***	***44.93***	***3.61***	***Avg =***	***45.12***	***3.63***	***Avg =***	***44.88***	***3.61***
	s =	***0.08***	***0.01***	***s =***	***0.06***	***0.01***	***s =***	***0.03***	***0.01***	***s =***	***0.04***	***0.02***

**Sample was dried at 105°C for two hours prior to analysis, and stored in a desiccator until used for analysis (within 24 hours).*
***Sample was dried at 105°C for one hour prior to analysis, and stored in a desiccator until used for analysis (within 24 hours).*
†Sample was dried at 85°C for two hours prior to analysis, and stored in a desiccator until used for analysis (within 24 hours).

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