Application Note

Instrument: CNS928

Determination of Carbon, Nitrogen and Sulfur in Soil

LECO Corporation; Saint Joseph, Michigan USA

Introduction

Nitrogen and Sulfur are considered essential macronutrients for proper 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. While carbon in almost any form is a benefit to the soil, it helps enormously if it is accompanied by the right ratios of nitrogen and sulfur. Testing to determine the content of carbon, nitrogen, and sulfur 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 carbon, nitrogen, and sulfur in arable soils can provide valuable information regarding fertilization needs that can be used to make fertilization management decisions for the soil.

The LECO CNS928 is a macro combustion carbon, nitrogen, and sulfur determinator that utilizes a pure oxygen environment in a high-temperature horizontal ceramic combustion furnace designed to handle macro sample mass. The combustion gases are collected in a ballast where the gases equilibrate and mix 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 non-dispersive infrared (NDIR) cells for the detection of carbon (as CO_2) and of sulfur (as SO_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 CNS928 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 for nitrogen determination.

The CNS928 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 CNS928 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: Carbon, nitrogen, and sulfur results for soil 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. The dried samples should be stored in a desiccator and must be used for analysis within 24 hours. For Reference materials, follow the sample drying instructions directed by the provided certificate.

Accessories

528-203 Ceramic Combustion Boats*, 502-321 COM-CAT™, 761-929 Crucible Tongs, and 501-614 Spatula

**Note: For optimal precision, ceramic combustion boats should be baked in a muffle furnace at 1,000 °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	1450 °C
Dehydration Time	0 s
Nominal Mass	1.0000 g
Purge Cycles	3
Use Monitor Cell	Yes
Monitor Cell Comparator	10.00%

*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

Burn Profile**

Burn Step	*Lance Flow*	*Furnace Flow*	*Time*
1	No	Yes	5 s
2	Yes	Yes	End

Element Parameters**

For Helium	*Carbon*	*Nitrogen*	*Sulfur*
Wait for Baseline Stability	Yes	—	Yes
Integration Delay	15 s	0 s	15 s
Starting Baseline	1 s	10 s	1 s
Post Baseline Delay	3 s	25 s	5 s
Use Comparator	No	No	No
Integration Time	22 s	50 s	22 s
Use Endline	Yes	Yes	Yes
Endline Delay	0 s	30 s	0 s
Ending Baseline	10 s	5 s	10 s

For Argon	*Carbon*	*Nitrogen*	*Sulfur*
Wait for Baseline Stability	Yes	—	Yes
Integration Delay	15 s	2 s	15 s
Starting Baseline	1 s	10 s	1 s
Post Baseline Delay	3 s	25 s	5 s
Use Comparator	No	No	No
Integration Time	22 s	65 s	22 s
Use Endline	Yes	Yes	Yes
Endline Delay	0 s	28 s	0 s
Ending Baseline	10 s	5 s	10 s
Use Profile Blank	—	No	—

***Refer to CNS928 Operator's Instruction Manual for Parameter definitions.*

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) utilizing helium as a carrier gas, and less than or equal to 0.005% (50 ppm) utilizing argon as a 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. Weigh ~1.0 g of 502-321 *COM-CAT* into a pre-baked 528-203 Ceramic Combustion Boat.
   c. Transfer the ceramic combustion boat containing *COM-CAT* to the appropriate position in the autoloader.
   d. Repeat steps 3b and 3c a minimum of five times.
   e. Initiate the analysis sequence.
   f. 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 ~0.05 g to 0.25 g of a suitable reference material into a pre-baked 528-203 Ceramic Combustion Boat.
   c. Enter sample mass and identification into the login screen.
   d. Add ~1.0 g of 502-321 *COM-CAT* into the ceramic combustion boat containing the sample and thoroughly mix the *COM-CAT* with the sample.
   e. Transfer the ceramic combustion boat containing the sample mixed with *COM-CAT* to the appropriate position in the autoloader.
   f. Repeat steps 4b through 4e a minimum of five times for each calibration/drift sample used.
   g. Initiate the analysis sequence.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

   Note: A multi-point calibration (fractional mass or multiple calibration samples) may be used to calibrate if desired. Typically, the LECO CNS928 can be calibrated utilizing a single standard calibration (linear, forced through origin calibration) using a pure compound. This is a cost effective and simple process. Pure compound (Certified) Reference Materials such as BBOT and Sulfamethazine can be analyzed up to 0.15 g, while (Certified) Reference Materials such as Soils, Calcium Carbonate or Synthetic Carbons can be analyzed up to 0.25 g.
5. Analyze Samples.
   a. Select the desired number of sample replicates in the login screen.
   b. Weigh ~0.25 g of the unknown sample into a pre-baked 528-203 Ceramic Combustion Boat.
   c. Enter sample mass and identification into the login screen.
   d. Add ~1.0 g of 502-321 *COM-CAT* into the ceramic combustion boat containing the sample and thoroughly mix the *COM-CAT* with the sample.
   e. Transfer the ceramic combustion boat containing the sample mixed with *COM-CAT* to the appropriate position in the autoloader.
   f. Repeat steps 5b through 5e for each sample being analyzed.
   g. Initiate the analysis sequence.

   Note: If soot (carbon black) is observed in the primary filter (steel wool filter), the sample mass should be reduced to prevent further sooting. Some sample types will produce soot when analyzed at larger sample masses, and reducing the sample mass will prevent soot from building up in the primary filter.

TYPICAL RESULTS

Nitrogen and sulfur data were generated utilizing a single standard calibration (linear, forced through origin calibration) using ~0.10 g of LECO 502-897 (Lot 1002) BBOT LCRM (72.55% C, 6.52% N, 7.49% S). Carbon data was generated utilizing a linear, full regression calibration using ~0.25 g of LECO 502-902 (Lot: 1001), Calcium Carbonate LCRM (11.99% C) and a variety of synthetic carbon reference materials covering the carbon range of the soil samples being analyzed. Soil samples were dried at 105 °C for one hour prior to analysis. Samples were analyzed using pre-baked ceramic combustion boats.

	10 cm³ Helium				3 cm³ Helium				10 cm³ Argon				3 cm³ Argon			
	Mass (g)	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*
Soil LCRM	0.2555	10.84	0.84	0.131	0.2532	10.68	0.83	0.133	0.2546	10.86	0.86	0.126	0.2518	10.81	0.83	0.143
LECO 502-694	0.2563	10.72	0.83	0.127	0.2525	10.82	0.85	0.118	0.2551	10.98	0.87	0.124	0.2540	10.73	0.83	0.115
Lot: 1000	0.2518	10.57	0.82	0.122	0.2512	10.86	0.85	0.125	0.2529	10.79	0.84	0.125	0.2519	10.80	0.83	0.128
% C = 10.80 ±0.26	0.2544	10.70	0.85	0.124	0.2531	10.58	0.83	0.126	0.2547	10.79	0.87	0.125	0.2525	10.63	0.88	0.123
% N = 0.86 ±0.03	0.2564	10.75	0.85	0.125	0.2528	10.66	0.83	0.131	0.2541	10.78	0.87	0.121	0.2534	10.57	0.88	0.120
% S = 0.124 ±0.013	***Avg =***	***10.72***	***0.84***	***0.126***	***Avg =***	***10.72***	***0.84***	***0.127***	***Avg =***	***10.84***	***0.86***	***0.124***	***Avg =***	***10.71***	***0.85***	***0.126***
	s =	***0.10***	***0.01***	***0.003***	***s =***	***0.12***	***0.01***	***0.006***	***s =***	***0.08***	***0.01***	***0.002***	***s =***	***0.11***	***0.03***	***0.011***
Soil LCRM	0.2531	0.948	0.094	0.014	0.2503	0.921	0.096	†	0.2534	0.924	0.099	0.015	0.2531	0.934	†	†
LECO 502-062	0.2518	0.945	0.089	0.017	0.2558	0.942	0.097	†	0.2519	0.926	0.106	0.017	0.2531	0.951	†	†
Lot: 1018	0.2525	0.939	0.086	0.016	0.2519	0.938	0.092	†	0.2513	0.928	0.108	0.014	0.2547	0.931	†	†
% C = 0.924 ±0.025	0.2518	0.940	0.086	0.015	0.2520	0.917	0.097	†	0.2552	0.929	0.104	0.017	0.2519	0.939	†	†
% N = 0.093 ±0.010	0.2556	0.934	0.092	0.021	0.2549	0.929	0.089	†	0.2541	0.918	0.111	0.014	0.2550	0.938	†	†
% S = 0.017 ±0.004	***Avg =***	***0.941***	***0.089***	***0.016***	***Avg =***	***0.930***	***0.094***	***†***	***Avg =***	***0.925***	***0.106***	***0.015***	***Avg =***	***0.939***	***†***	***†***
	s =	***0.005***	***0.004***	***0.003***	***s =***	***0.011***	***0.004***	***†***	***s =***	***0.004***	***0.005***	***0.001***	***s =***	***0.008***	***†***	***†***
San Joaquin Soil	0.2512	1.12	0.085	0.081	0.2556	1.14	0.098	0.072	0.2526	1.11	0.107	0.078	0.2557	1.12	†	0.090
NIST SRM 2709	0.2525	1.13	0.086	0.079	0.2532	1.12	0.105	0.076	0.2543	1.11	0.105	0.079	0.2537	1.11	†	0.084
	0.2540	1.12	0.084	0.082	0.2532	1.12	0.102	0.076	0.2520	1.11	0.106	0.078	0.2529	1.13	†	0.094
	0.2543	1.13	0.083	0.076	0.2543	1.11	0.106	0.071	0.2515	1.10	0.112	0.078	0.2527	1.11	†	0.090
	0.2527	1.12	0.078	0.080	0.2510	1.13	0.108	0.075	0.2513	1.10	0.090	0.081	0.2540	1.12	†	0.080
	Avg =	***1.12***	***0.083***	***0.080***	***Avg =***	***1.12***	***0.104***	***0.074***	***Avg =***	***1.10***	***0.104***	***0.079***	***Avg =***	***1.12***	***†***	***0.088***
	s =	***<0.01***	***0.003***	***0.002***	***s =***	***0.01***	***0.004***	***0.002***	***s =***	***0.01***	***0.008***	***0.001***	***s =***	***0.01***	***†***	***0.006***

†Results were below the Method Detection Limit.

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
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