Application Note

Instrument: CNS928

Determination of Carbon, Nitrogen and Sulfur in Plant Tissue

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Introduction

Nitrogen and Sulfur are considered essential macronutrients for plant development, playing a key role in the formation of enzymes and proteins. Determination of carbon, nitrogen, and sulfur 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. Testing for carbon, nitrogen, and sulfur in plant tissue 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 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. Plant samples are typically dried between 80 °C and 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. For (Certified) 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 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
Use Monitor Cell	Yes
Monitor Cell Comparator	1.00%

*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

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	5 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

**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	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. Weigh ~1.0 g of 502-321 COM-CAT into a 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 through 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 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 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. (Certified) Reference Materials such as 502-897 BBOT and 502-657 Sulfamethazine can be analyzed up to 0.15 g, while (Certified) Reference Materials such as 502-082 Tobacco, 502-273 Alfalfa, and 502-931 Orchard Leaves 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 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 to be 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

Data was generated utilizing a single standard calibration (linear forced through origin) using ~0.25 g of 502-931 (Lot 1000) Orchard Leaves LCRM (49.54% C, 2.31% N, 0.442% S). Samples were analyzed as received and corrected for moisture. Moisture values were obtained using a TGM800 at 85 °C with a hold time of 2 hours.

	10 cm^3 Helium				3 cm^3 Helium				10 cm^3 Argon				3 cm^3 Argon			
	Mass (g)	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*	*Mass (g)*	*% C*	*% N*	*% S*
Tobacco LRM	0.2509	47.22	2.51	0.44	0.2525	47.30	2.52	0.45	0.2536	47.31	2.51	0.44	0.2527	47.09	2.45	0.45
LECO 502-082	0.2527	47.22	2.51	0.44	0.2543	47.24	2.50	0.45	0.2539	47.17	2.52	0.44	0.2514	47.34	2.49	0.44
Lot: 1018	0.2531	47.24	2.51	0.44	0.2508	47.32	2.52	0.45	0.2527	47.24	2.52	0.44	0.2510	47.27	2.52	0.45
% C = 47.27 ±0.23	0.2501	47.10	2.52	0.44	0.2525	47.11	2.52	0.44	0.2516	47.04	2.50	0.44	0.2514	47.35	2.49	0.45
% N = 2.48 ±0.04	0.2522	47.23	2.51	0.44	0.2527	47.29	2.52	0.45	0.2553	47.13	2.51	0.44	0.2525	47.30	2.48	0.44
% S = 0.46 ±0.04	***Avg =***	***47.20***	***2.51***	***0.44***	***Avg =***	***47.25***	***2.52***	***0.45***	***Avg =***	***47.18***	***2.51***	***0.44***	***Avg =***	***47.27***	***2.49***	***0.45***
	s =	***0.05***	***<0.01***	***<0.01***	***s =***	***0.07***	***0.01***	***<0.01***	***s =***	***0.10***	***0.01***	***<0.01***	***s =***	***0.11***	***0.03***	***0.01***
Alfalfa LRM	0.2538	45.02	3.66	0.36	0.2520	45.22	3.66	0.37	0.2532	45.00	3.65	0.37	0.2521	45.03	3.61	0.38
LECO 502-273	0.2541	45.02	3.63	0.36	0.2536	45.16	3.62	0.36	0.2532	45.01	3.64	0.37	0.2516	45.10	3.67	0.38
Lot: 1026	0.2516	45.02	3.63	0.36	0.2544	45.16	3.63	0.38	0.2517	44.96	3.64	0.37	0.2544	45.02	3.63	0.37
% C = 45.05 ±0.23	0.2528	45.06	3.64	0.36	0.2547	45.14	3.62	0.36	0.2522	44.99	3.64	0.37	0.2533	45.16	3.60	0.38
% N = 3.62 ±0.05	0.2517	45.05	3.63	0.36	0.2531	45.16	3.63	0.36	0.2527	45.05	3.64	0.37	0.2547	45.02	3.61	0.37
% S = 0.38 ±0.02	***Avg =***	***45.04***	***3.64***	***0.36***	***Avg =***	***45.17***	***3.63***	***0.37***	***Avg =***	***45.00***	***3.64***	***0.37***	***Avg =***	***45.07***	***3.63***	***0.38***
	s =	***0.02***	***0.01***	***<0.01***	***s =***	***0.03***	***0.01***	***0.01***	***s =***	***0.03***	***0.01***	***<0.01***	***s =***	***0.06***	***0.03***	***0.01***
Apple Leaves	0.2538	49.89	2.269	0.17	0.2525	49.91	2.283	0.18	0.2522	49.91	2.293	0.18	0.2530	49.91	2.280	0.17
NIST SRM 1515	0.2530	49.84	2.288	0.17	0.2542	49.97	2.277	0.17	0.2525	49.86	2.304	0.17	0.2529	49.87	2.203	0.18
Certified Value:	0.2538	49.89	2.274	0.17	0.2543	49.71	2.289	0.19	0.2519	49.85	2.295	0.17	0.2539	49.70	2.251	0.19
% N = 2.299 ±0.090	0.2528	49.99	2.280	0.17	0.2535	49.90	2.286	0.18	0.2520	49.93	2.278	0.17	0.2551	49.86	2.294	0.18
Informational Value:	0.2525	50.01	2.282	0.17	0.2535	49.75	2.270	0.18	0.2528	49.86	2.290	0.17	0.2531	49.93	2.331	0.18
% S = 0.18	***Avg =***	***49.92***	***2.278***	***0.17***	***Avg =***	***49.85***	***2.281***	***0.18***	***Avg =***	***49.88***	***2.292***	***0.17***	***Avg =***	***49.85***	***2.272***	***0.18***
	s =	***0.07***	***0.008***	***<0.01***	***s =***	***0.11***	***0.008***	***<0.01***	***s =***	***0.04***	***0.009***	***<0.01***	***s =***	***0.09***	***0.048***	***0.01***

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