Application Note

Instrument: CHN828

Determination of Carbon, Hydrogen, and Nitrogen in Flour and Plant Tissue

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Introduction

Flour is a fine particle powder created by milling or grinding a dry grain. The most common varieties of flour are made from wheat, although any grain can be used to make flour. Flour is typically used to make dough for a variety of bread products. Determination of Carbon and Nitrogen in flour provides information on the uniformity of the flour as well as the quality of the enrichment process.

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 the crop plant tissue 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. Testing for total Carbon and Nitrogen in plant tissue is useful in predicting fertilization needs and making fertilization management decisions for crops.

Instrument Model and Configuration

The LECO CHN828 is a combustion Carbon, Hydrogen, and Nitrogen determinator that utilizes a pure Oxygen environment in a vertical quartz furnace, ensuring complete combustion and superior analyte recovery. Combustion gases are swept from the furnace through an afterburner containing a reagent to scrub sulfur compounds from the gas stream prior to collection in the ballast volume. The combustion gases are collected in a ballast where they 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 an infrared cell (IR) for the detection of Carbon (CO_2) and a thermal conductivity cell (TC) for the detection of Nitrogen (N_2). A separate portion of the ballast gas is transferred to an IR cell for the detection of Hydrogen (H_2O).

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 the sensitivity of the detector. The CHN828 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. However, 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 CHN828 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.

Note: When changing carrier gas type, the flow needs to be adjusted following instructions provided in the 828 Series 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, Hydrogen, and Nitrogen results for flour and plant tissue 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 on the day of analysis and entered into the software to correct for moisture. Flour and plant tissue samples are typically dried at 80 °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. Reference Materials should be prepared according to the instructions provided by the certificate.

Accessories

502-186 Tin Foil Cups and 501-614 Spatula

Reference Materials

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

*General 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	Large (10 cm^3) or Small (3 cm^3)

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

Element Parameters*

	Helium			Argon		
Parameter	*C*	*H*	*N*	*C*	*H*	*N*
Integration Delay	4 s	**	4 s	5 s	**	5 s
Starting Baseline	15 s	**	15 s	15 s	**	15 s
Post Baseline Delay	0 s	**	14 s	0 s	**	15 s
Use Comparator	No	**	No	No	**	No
Integration Time	13 s	**	35 s	16 s	**	60 s
Use Endline	Yes	**	Yes	Yes	**	Yes
Endline Delay	19 s	**	25 s	23 s	**	30 s
Ending Baseline	15 s	**	15 s	15 s	**	15 s
Use H Correction	--	Yes	--	--	Yes	--
Use Profile Blank	--	--	--	--	--	Yes

**Refer to the 828 Series Operator's Instruction Manual for parameter definitions.*
***Hydrogen determination is performed using a "stop-flow" analysis technique; therefore, the element parameters for Hydrogen are not adjustable.*

Burn Profile

Burn Step	*Furnace Flow*	*Time*
1	5.00 L/min	30 s
2	1.00 L/min	30 s
3	5.00 L/min	End

Procedure

1. Prepare the 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 all three elements when utilizing Helium as a carrier gas, and less than or equal to 0.005% (50 ppm) for all three elements 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 an appropriate mass 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 reference material 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 CHN828 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. A multi-point calibration (fractional masses or multiple calibration materials) utilizing a linear, full regression calibration may be used if desired.
5. Analyze Samples.
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh an appropriate mass (~ 0.20 g) of a flour or plant tissue 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 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.

TYPICAL RESULTS

Data was generated utilizing linear, full regression calibrations for Carbon and Hydrogen determination, and a linear, force through origin calibration for Nitrogen determination, using fractional masses (~0.15 g to ~0.25 g) of LECO 502-896 (Lot 1007) LCRM EDTA (41.14% C, 5.53% H, 9.59% N). The calibrations were verified using LECO 502-642 (Lot 1020) LCRM Phenylalanine (65.45% C, 6.75% H, 8.46% N). Samples were dried at 80 °C for two hours prior to analysis and stored in a desiccator until used (within 24 hours). Results are reported on a dry basis.

	Helium 10 cm³				Helium 3 cm³				Argon 10 cm³				Argon 3 cm³			
	Mass (g)	% C	% H	% N	Mass (g)	% C	% H	% N	Mass (g)	% C	% H	% N	Mass (g)	% C	% H	% N
Wheat Flour	0.1995	45.46	6.47	3.09	0.2035	45.27	6.57	3.11	0.2055	45.66	6.49	3.11	0.1965	45.64	6.49	3.09
LECO 502-692	0.2043	45.47	6.42	3.09	0.2103	45.37	6.53	3.08	0.2077	45.59	6.45	3.10	0.1998	45.69	6.45	3.09
Lot 1003	0.2070	45.66	6.43	3.12	0.2015	45.27	6.58	3.07	0.2070	45.66	6.47	3.13	0.2034	45.76	6.42	3.12
45.44% ± 0.31% C	0.2006	45.37	6.50	3.09	0.2000	45.15	6.51	3.07	0.2008	45.60	6.48	3.09	0.1954	45.54	6.52	3.10
6.50% ±0.08% H	0.2040	45.55	6.42	3.10	0.2031	45.26	6.53	3.05	0.2074	45.72	6.43	3.11	0.1964	45.65	6.46	3.11
3.08% ±0.04% N	***Avg=***	***45.50***	***6.45***	***3.10***	***Avg=***	***45.26***	***6.55***	***3.08***	***Avg=***	***45.65***	***6.46***	***3.11***	***Avg=***	***45.66***	***6.47***	***3.10***
	s=	***0.11***	***0.03***	***0.01***	***s=***	***0.08***	***0.03***	***0.02***	***s=***	***0.05***	***0.02***	***0.01***	***s=***	***0.08***	***0.04***	***0.01***
Orchard Leaves	0.2003	48.71	6.16	1.36	0.2045	48.81	6.20	1.35	0.2030	48.75	6.21	1.36	0.1985	48.45	6.24	1.35
LECO 502-931	0.2050	48.84	6.12	1.36	0.2037	48.76	6.21	1.35	0.2016	48.61	6.19	1.34	0.2045	48.36	6.16	1.36
Lot 1001	0.2021	48.73	6.17	1.36	0.2038	48.83	6.20	1.35	0.2085	48.84	6.12	1.34	0.2035	48.59	6.18	1.38
48.62% ±0.26% C	0.2053	48.84	6.18	1.36	0.2037	48.86	6.24	1.34	0.2022	48.83	6.22	1.35	0.2015	48.68	6.20	1.40
6.18% ±0.06% H	0.2007	48.75	6.19	1.36	0.2093	48.68	6.18	1.35	0.2015	48.87	6.19	1.35	0.2009	48.72	6.21	1.36
1.37% ±0.03% N	***Avg=***	***48.77***	***6.17***	***1.36***	***Avg=***	***48.79***	***6.21***	***1.35***	***Avg=***	***48.78***	***6.18***	***1.35***	***Avg=***	***48.56***	***6.20***	***1.37***
	s=	***0.06***	***0.03***	***<0.01***	***s=***	***0.07***	***0.02***	***0.01***	***s=***	***0.10***	***0.04***	***<0.01***	***s=***	***0.15***	***0.03***	***0.02***
Corn Gluten	0.1995	52.07	7.04	11.00	0.1981	52.05	7.08	11.04	0.2063	52.07	7.00	11.02	0.1942	51.88	7.07	10.94
502-932	0.2055	52.04	7.00	10.99	0.2033	52.08	7.06	11.04	0.2063	52.06	7.07	10.99	0.2016	51.88	6.98	10.98
Lot 1001	0.2073	52.08	7.00	11.03	0.2023	52.01	7.03	11.04	0.2096	51.90	7.05	10.95	0.1987	51.71	7.06	10.96
51.87% ±0.27% C	0.2059	51.91	7.01	10.98	0.2058	51.96	7.00	11.02	0.2046	51.86	7.14	10.96	0.2026	51.85	6.99	10.93
7.06% ±0.09% H	0.2059	51.92	7.03	10.96	0.1990	51.93	7.08	11.03	0.2018	51.77	7.15	10.95	0.1989	51.89	7.03	11.02
11.01% ±0.08% N	***Avg=***	***52.00***	***7.02***	***10.99***	***Avg=***	***52.00***	***7.05***	***11.03***	***Avg=***	***51.93***	***7.08***	***10.97***	***Avg=***	***51.84***	***7.03***	***10.97***
	s=	***0.08***	***0.02***	***0.03***	***s=***	***0.06***	***0.03***	***0.01***	***s=***	***0.13***	***0.06***	***0.03***	***s=***	***0.08***	***0.04***	***0.04***
Alfalfa	0.1964	44.07	5.87	2.92	0.2015	44.04	5.68	2.92	0.2022	44.02	5.91	2.88	0.2015	43.98	5.83	2.85
LECO 502-983	0.2058	44.10	5.78	2.92	0.1994	43.97	5.70	2.93	0.2037	43.99	5.84	2.91	0.2113	43.92	5.68	2.93
Lot 1001	0.2073	43.93	5.73	2.91	0.1990	43.93	5.64	2.92	0.1969	44.00	5.88	2.91	0.2060	43.82	5.73	2.92
43.73% ±0.44% C	0.2063	43.94	5.80	2.91	0.1959	43.96	5.68	2.93	0.2006	43.96	5.79	2.90	0.2049	43.81	5.78	2.85
2.89% ±0.06% N	0.1999	43.77	5.77	2.90	0.2000	43.68	5.64	2.90	0.1904	43.68	5.80	2.89	0.1986	43.76	5.85	2.88
	Avg=	***43.96***	***5.79***	***2.91***	***Avg=***	***43.91***	***5.67***	***2.92***	***Avg=***	***43.93***	***5.84***	***2.90***	***Avg=***	***43.86***	***5.78***	***2.89***
	s=	***0.13***	***0.05***	***0.01***	***s=***	***0.14***	***0.03***	***0.01***	***s=***	***0.14***	***0.05***	***0.01***	***s=***	***0.09***	***0.07***	***0.04***
Tobacco	0.2025	48.08	5.85	3.29	0.2047	48.49	5.82	3.30	0.1964	48.33	5.90	3.27	0.2075	48.46	5.92	3.29
LECO 502-984	0.2110	47.93	5.76	3.32	0.1956	48.35	5.90	3.29	0.2012	48.20	5.86	3.27	0.2105	47.93	5.90	3.32
Lot 1000	0.2048	47.88	5.80	3.33	0.2011	48.07	5.78	3.26	0.2087	48.36	5.83	3.29	0.2085	48.10	5.99	3.26
48.33% ±0.45% C	0.2019	47.93	5.78	3.32	0.2056	48.34	5.79	3.29	0.2024	48.25	5.92	3.26	0.1951	47.95	6.14	3.27
3.29% ±0.03% N	0.2088	48.01	5.74	3.32	0.2044	48.21	5.81	3.27	0.2044	48.28	5.89	3.28	0.2090	47.91	5.93	3.29
	Avg=	***47.96***	***5.79***	***3.32***	***Avg=***	***48.29***	***5.82***	***3.28***	***Avg=***	***48.29***	***5.88***	***3.27***	***Avg=***	***48.07***	***5.98***	***3.29***
	s=	***0.08***	***0.04***	***0.01***	***s=***	***0.16***	***0.05***	***0.02***	***s=***	***0.06***	***0.03***	***0.01***	***s=***	***0.23***	***0.1***	***0.02***



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