Application Note

Instrument: FP828

Determination of Nitrogen/Protein in Flour

LECO Corporation; Saint Joseph, Michigan USA

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. The protein content in the flour is one of the primary constituents that determines the best use for the flour, with lower-protein flour (~8%) typically being used for cakes and pastries, mid-range protein flours (~10%) being categorized as all-purpose, and higher-protein flours (~12%) being referred to as bread flour.

The accurate and precise determination of protein not only plays a role in the characterization of nutritional or dietary value in flours, but is also the key to determining the category or quality of the flour. Protein in flour and other food products is most commonly calculated using the measured nitrogen in the sample and a protein factor multiplier (protein factors vary according to the sample matrix).

The LECO FP828 is a combustion nitrogen/protein determinator that utilizes a pure oxygen environment in a vertical quartz furnace for the sample combustion process resulting in an analysis time of 2.8 minutes with no reagents in the primary or secondary furnace. A thermoelectric cooler removes the moisture in the combustion gas without the use of chemical reagents. A 3 or 10 cm^3 volume of combustion gas is taken using a combustion gas collection and handling system. The combustion gas collection and handling system achieves a low cost-per-analysis by reducing the amount of chemical reagents used for scrubbing and converting the nitrogen oxide in the combustion gas to nitrogen. A thermal conductivity (TC) cell is used for the detection of nitrogen in the combustion gas.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results.

Dry sample at 85 °C for two hours prior to analysis. The dried sample should be stored in a desiccator and must be used for analysis within 24 hours.

Accessories

502-186 Small Tin Foil Cup, 502-397 Large Tin Foil Cup, 502-338 Small Gel Caps, 502-382 Medium Gel Caps, 502-810 Large Gel Caps

Reference Materials

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

Analysis Parameters

Gas Type:	Argon or Helium
Combustion Temperature:	950 °C
Afterburner Temperature:	850 °C
Nominal Mass:	1.0000
Purge Cycles:	3

Instrument Model and Configuration

Thermal conductivity detectors work by detecting changes in the thermal conductivity of the analytical gas compared to the constant thermal conductivity of the reference 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 FP828 supports either the use of helium or argon as the instrument's carrier gas for the thermal conductivity cell. When used as a carrier gas, helium provides the highest sensitivity, and the best performance at the lower end 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; the detector is inherently less sensitive when using argon as a carrier gas.

The FP828 offers the additional advantage of choosing a 10 cm^3 aliquot loop or a 3 cm^3 loop within the instrument's gas collection and handling system. The 10 cm^3 aliquot loop optimizes the instrument for the lowest nitrogen range and best precision. The 3 cm^3 aliquot loop extends reagent life expectancy by approximately three fold 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: Aliquot loop size changes require hardware updates within the instrument base model.

Element Parameters

	Helium	Argon
Integration Delay	4 s	5 s
Starting Baseline	15 s	15 s
Post Baseline Delay	14 s	16 s
Use Comparator	No	No
Integration Time	35 s	41 s
Use Endline	Yes	Yes
Endline Delay	25 s	30 s
Endline Baseline	15 s	15 s
Use Profile Blank	No	No

Burn Profile

	FP828	FP828P
Furnace Flow	High	4.00 lpm

Ballast Parameters

Equilibrate Time:	20 s
Not Filled Timeout:	300 s
Aliquot Loop Fill Pressure Drop:	200 mm Hg
Aliquot Loop Equilibrate Time:	6 s
Dose Loop Size:	Large (10 cm^3) or Small (3 cm^3)
Interleave Analysis:	Yes
Sample Drop Detection:	Disabled

**Refer to FP828 Operator's Instruction Manual for Method Parameter definitions.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Select 10 replicates and Log-In Blanks.
   b. Initiate the analysis sequence.
   c. Set the blank using at least five results following the procedure outlined in the operator's instruction manual.

   Note: Blank precision for nitrogen should be <0.001%.
3. Calibrate/Drift Correct.
   a. Weigh ~0.25 g of a suitable reference material into a Tin Foil Cup/Gel Cap. Seal the Tin Foil Cup if used.
   b. Enter sample mass and identification into Standard Login.
   c. Transfer sample to the appropriate position in the sample carousel.
   d. Repeat steps 3a through 3c a minimum of five times for each calibration/drift sample used.
   e. Initiate the analysis sequence.
   f. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
   g. Verify calibration by analyzing ~0.1 to 0.25 g of corn flour, barley, or wheat flour.
4. Analyze Samples.
   a. Weigh ~0.25 g of the unknown sample into a Tin Foil Cup/Gel Cap. Seal the Tin Foil Cup if used.
   b. Enter mass and identification information into Sample Login.
   c. Transfer sample to the appropriate position of the sample carousel.
   d. Repeat steps 4a through 4c for all unknown samples.
   e. Initiate the analysis sequence.
5. Atmospheric Blank.
   Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. Some atmosphere may also be present when using the gel caps as well. This will cause biased nitrogen results at low nitrogen concentrations, therefore an atmospheric blank should be determined and entered using the following procedure: Analyze reagent grade sucrose (finely ground) several times using similar weights of the sucrose to the weight of samples being analyzed. Enter the actual weight of the sucrose. The nitrogen value obtained is considered the atmospheric blank and can be automatically compensated using the FP828 software. Refer to the operator's instruction manual for details regarding the setting of the atmospheric blank.

Notes

- The FP828 can be calibrated using several replicates of a single mass (nominal 0.25 g) of EDTA utilizing a linear forced through origin calibration. The calibration can be verified by analyzing a pure compound that is different than the material used for calibration, such as phenylalanine (~0.1 g), nicotinic acid (~0.1 g), or corn, barley or wheat flour (~0.1 to 0.25 g). Multipoint (fractional weight or multiple calibration samples) may also be used to calibrate if desired.

*TYPICAL RESULTS**

	10 cm³ Helium			3 cm³ Helium			10 cm³ Argon			3 cm³ Argon		
	Mass(g)	*% N*	*% Protein*	*Mass(g)*	*% N*	*% Protein*	*Mass(g)*	*% N*	*% Protein*	*Mass(g)*	*% N*	*% Protein*
Corn Flour LRM	0.2484	1.056	6.60	0.2530	1.050	6.56	0.2596	1.088	6.80	0.2517	1.123	7.02
LECO 501-563†	0.2513	1.048	6.55	0.2475	1.043	6.52	0.2594	1.061	6.63	0.2523	1.102	6.88
Lot 1016	0.2506	1.046	6.54	0.2484	1.050	6.56	0.2409	1.090	6.81	0.2505	1.091	6.82
% N = 1.06	0.2558	1.044	6.52	0.2455	1.047	6.54	0.2572	1.074	6.71	0.2470	1.054	6.59
± 0.03	0.2468	1.055	6.59	0.2513	1.047	6.54	0.2457	1.081	6.76	0.2548	1.085	6.78
†Protein Factor = 6.25	***Avg =***	***1.050***	***6.56***	***Avg =***	***1.048***	***6.55***	***Avg =***	***1.079***	***6.74***	***Avg =***	***1.091***	***6.82***
	s =	***0.005***	***0.03***	***s =***	***0.003***	***0.02***	***s =***	***0.012***	***0.07***	***s =***	***0.025***	***0.16***
Wheat Flour LCRM	0.2571	2.666	15.2	0.2587	2.655	15.1	0.2425	2.643	15.1	0.2560	2.672	15.2
LECO 502-692††	0.2529	2.669	15.2	0.2478	2.648	15.1	0.2556	2.657	15.1	0.2527	2.677	15.3
Lot 1000	0.2508	2.669	15.2	0.2497	2.650	15.1	0.2468	2.643	15.1	0.2481	2.648	15.1
% N = 2.66	0.2436	2.664	15.2	0.2493	2.646	15.1	0.2415	2.665	15.2	0.2438	2.660	15.2
± 0.05	0.2430	2.676	15.3	0.2438	2.633	15.0	0.2481	2.659	15.2	0.2554	2.701	15.4
†Protein Factor = 5.70	***Avg =***	***2.669***	***15.2***	***Avg =***	***2.646***	***15.1***	***Avg =***	***2.653***	***15.1***	***Avg =***	***2.672***	***15.2***
	s =	***0.004***	***0.03***	***s =***	***0.008***	***0.04***	***s =***	***0.010***	***0.05***	***s =***	***0.020***	***0.11***
Rice Flour LCRM	0.2406	1.576	9.38	0.2472	1.550	9.22	0.2542	1.556	9.26	0.2589	1.572	9.35
LECO 502-907‡	0.2506	1.572	9.35	0.2546	1.541	9.17	0.2477	1.562	9.29	0.2461	1.568	9.33
Lot 1000	0.2421	1.577	9.38	0.2413	1.537	9.14	0.2460	1.553	9.24	0.2525	1.509	8.98
% N = 1.56	0.2507	1.569	9.34	0.2600	1.543	9.18	0.2467	1.561	9.29	0.2526	1.550	9.22
± 0.05	0.2574	1.571	9.35	0.2458	1.541	9.17	0.2550	1.568	9.33	0.2459	1.567	9.32
†Protein Factor = 5.95	***Avg =***	***1.573***	***9.36***	***Avg =***	***1.542***	***9.18***	***Avg =***	***1.560***	***9.28***	***Avg =***	***1.553***	***9.24***
	s =	***0.003***	***0.02***	***s =***	***0.005***	***0.03***	***s =***	***0.006***	***0.03***	***s =***	***0.026***	***0.15***

**LECO and NIST standard reference materials were used for calibration utilizing a linear force through origin calibration curve.*

†Protein factors are provided by the United States Department of Agricultures, circular #183.

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

info@leco.com • www.leco.com | ISO-9001:2015 Q-994 | *LECO is a registered trademark of LECO Corporation.*