Application Note

Instrument: TruMac® N

Determination of Nitrogen in Flour

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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. 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 TruMac N is a macro combustion nitrogen/protein determinator that utilizes a pure oxygen environment in a ceramic horizontal furnace and large ceramic boats for the macro sample combustion process. A thermoelectric cooler removes the moisture in the combustion gas without the use of chemical reagents. A 3 or 10 cc 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 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 uniform consistency to produce suitable results. Reference materials should be prepared as directed by the certificate prior to analysis.

Note: Nitrogen results for flour samples are generally reported on a dry basis. The material can either be dried prior to analysis or the moisture content can be determined on the day of analysis and used to correct the values for moisture utilizing the instrument's software. Flour 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.

Accessories 528-203 Crucibles

Calibration Samples

502-092 EDTA, 502-642 Phenylalanine, 501-050 Nicotinic Acid

*Analysis Parameters**

Furnace Temperature	1100°C
TE Cooler Temperature	5°C
Dehydration Time	0 seconds
Purge Cycles	2 seconds

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 TruMac is available in models that support 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, providing the best performance at the lower end of the nitrogen range. Helium models also offer the additional advantage of replacing the 10 cc aliquot loop with a 3 cc loop within the instrument's gas collection and handling system. The 10 cc aliquot loop optimizes the instrument for the lowest nitrogen range and best precision. The 3 cc aliquot loop extends reagent life expectancy by approximately three fold compared to the 10 cc aliquot loop, while providing the lowest cost per analysis with minimal impact on practical application performance (see Typical Results section).

Due to the recent history of low supply and general availability issues for helium gas, the argon model was developed to utilize argon as a carrier gas. Since 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 with argon as a carrier gas. The argon model has a similar practical application performance compared to the helium model, operating with equivalent instrument and method configurations (see Typical Results section).

Note: Changing carrier gas and aliquot loop size requires hardware changes within the instrument.

Element Parameters

	Helium	*Argon*
	10 cc and 3 cc	*10 cc*
Baseline Delay Time	6 seconds	6 seconds
Minimum Analysis Time	35 seconds	55 seconds
Endline Time	2 seconds	2 seconds
Conversion Factor	1.00	1.00
Significant Digits	5	5
TC Baseline Time	10 seconds	10 seconds

Burn Profile

Burn Cycle	*Lance Flow*	*Purge Flow*	*Time (seconds)*
1	Off	On	5 seconds
2	On	On	35 seconds
3	On	Off	END

Ballast Parameters

Ballast

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Equilibrate Pressure Time	4 seconds
High Precision	Yes
High Speed	No

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

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing 3 to 5 blanks (crucible is not required).
3. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Place a 528-203 Crucible to the appropriate position of the autoloader.
   c. Repeat steps 3a through 3b a minimum of three times.
   d. Initiate the analysis sequence (F5).
   e. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate.
   a. Weigh ~0.75 g of EDTA calibration sample into a 528-203 Crucible, enter mass and sample identification into Sample Login (F3).
   b. Transfer crucible to the appropriate position of the autoloader.
   c. Repeat steps 4a through 4b a minimum of three times.
   d. Initiate the analysis sequence (F5).
   e. Calibrate the instrument following the procedure outlined in the operator's instruction manual. Use single standard calibration.

   Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. Research has shown that a properly functioning TruMac can be calibrated using several replicates of a single mass range (nominal 0.75 g) of EDTA utilizing a single standard calibration. This is a cost effective and simple process. The calibration can be verified by analyzing different compounds such as nicotinic acid (0.25 to 0.5 g) and/or phenylalanine (0.5 to 0.75 g).
5. Analyze Samples.
   a. Weigh ~1 g of flour sample into a 528-203 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Transfer crucible to the appropriate position of the autoloader.
   c. Repeat steps 5a through 5b for each sample to be analyzed.
   d. Initiate the analysis sequence (F5).

   Note: If soot (carbon black) is noticed in the primary filter (steel wool filter), reduce sample mass to prevent soot build-up in this filter. Soot can be produced when larger masses of some sample types are analyzed.

*Typical Results**

Name	*Mass (g)*	*% Carbon*	*% Nitrogen*	*% Sulfur*
LECO 502-055	0.2536	50.33	2.04	0.152
Orchard Leaves	0.2512	50.34	2.04	0.150
@ 50.5% C, 2.04% N,	0.2547	50.33	2.06	0.157
0.146% S	0.2512	50.28	2.05	0.152
	0.2526	50.32	2.05	0.157
	Avg =	***50.32***	***2.05***	***0.154***
	s =	***0.02***	***0.01***	***0.003***
LECO 502-082	0.2538	46.01	2.56	0.603
Tobacco	0.2560	46.10	2.54	0.606
@ 46.16% C, 2.53% N,	0.2501	46.11	2.53	0.604
0.58% S	0.2545	46.02	2.53	0.595
	0.2572	45.94	2.52	0.597
	Avg =	***46.03***	***2.53***	***0.601***
	s =	***0.07***	***0.01***	***0.005***
LECO 502-273	0.2497	44.57	3.45	0.271
Alfalfa	0.2485	44.69	3.40	0.262
@ 44.8% C, 3.38% N,	0.2502	44.60	3.38	0.253
0.24% S	0.2514	44.67	3.39	0.244
	0.2496	44.60	3.38	0.240
	Avg =	***44.63***	***3.40***	***0.254***
	s =	***0.05***	***0.03***	***0.013***

**Based on a single standard, force through origin calibration utilizing LECO 502-897 BBOT. Results reported on a dry basis.*

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