Optimization of Combustion Methods for the Determination of Low Level Nitrogen/Protein in Food and Feed Products

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Introduction

Total protein in foods and feeds is determined using the total measured nitrogen content in the sample and a protein factor specific to the sample matrix. Nitrogen determination is commonly performed by one of two major methods: a classical wet chemistry (Kjeldahl) technique, or a combustion instrument-based (Dumas) technique. The combustion technique is gaining popularity due to several advantages including shorter analysis times, ease-of-operation, and improved safety characteristics.

Total nitrogen combustion instruments use a high-temperature furnace in an oxygen environment to combust the sample. Combustion gases are swept from the furnace and are scrubbed and processed to eliminate various components that would interfere with nitrogen determination. Nitrogen combustion gases, in the form of NO_x, are reduced to Nitrogen (N_2) prior to detection using a Thermal Conductivity (TC) detector.

Total nitrogen combustion instruments are limited to the amount of sample that can be analyzed. Samples containing low levels of nitrogen (dry corn starch, Neutral Detergent Insoluble Nitrogen (NDIN) filter bags, low level filtrate, beer, low level filtration media, etc.) can present a challenge for analysis due to sample characteristics and method limitations.

This poster presentation will cover the method optimization for low-level nitrogen determination utilizing combustion instruments. Optimization parameters studied include: sample mass, containment vessels, and correct use of atmospheric blanks. The data presented in this poster was obtained through the analysis of materials including starches, beer, nutritional drinks, and other low level nitrogen materials utilizing the LECO FP828P (Performance package) and LECO FP928 combustion instruments.

FP828P FP928

FP828P Theory of Operation

- The sample is weighed into a container, and is transferred to a sealed purge chamber where atmospheric gas is removed.
- The sample is then dropped into the furnace in a pure oxygen environment to ensure complete combustion.
- Combustion gases are swept from the furnace, moisture is removed, and the gasses are collected in a ballast.
- The gases equilibrate and mix within the ballast before an aliquot of the gas is extracted and introduced into a flowing stream of inert gas for analysis.
- A heated reduction tube, filled with copper, is used to convert NO_x to N_2 and to remove excess oxygen. Carbon dioxide (CO_2) is removed by LECOSORB® and water is removed by Anhydrone®.
- The aliquot gas is carried to a TC Cell to detect N_2.

FP928 Theory of Operation

- The sample is weighed into a combustion boat and is transferred into the furnace purge area where the atmospheric gasses are purged.
- The sample is transferred to the furnace in a pure oxygen environment to ensure complete combustion.
- Combustion gases are swept from the furnace, moisture is removed, and the gasses are collected in a ballast.
- The gases equilibrate and mix within the ballast before an aliquot of the gas is extracted and introduced into a flowing stream of inert gas for analysis.
- A heated reduction tube, filled with copper, is used to convert NO_x to N_2 and to remove excess oxygen. CO_2 is removed by LECOSORB, and water is removed by Anhydrone.
- The aliquot gas is carried to a TC Cell to detect N_2.

Sample Description

Liquids

• Liquid Permeate	• Orange Juice
• Specialty Beer	• Commercial Beer

Powders

• Lactose A	• Lactose B
• Waxy Corn Starch	

Analysis Parameters

Parameters	FP828P	FP928
Gas Type	Helium	Helium
Furnace Temperature	950 °C	1100 °C
Ballast Equilibrate Time	10 s	10 s
Ballast Filled Timeout	300 s	300 s
Aliquot Loop Equilibrate Time	6 s	6 s
Dose Loop Size	10 cc	10 cc

Burn Steps

Burn Step	FP828P	FP928
1	5.0 L/min Oxygen	Lance Flow- No/Furnace Flow - Yes
Time	30 s	5 s
2	1.0 L/min Oxygen	Lance Flow - Yes/Furnace Flow -Yes
Time	30 s	35 s
3	5.0 L/min Oxygen	Lance Flow - Yes/Furnace Flow - No
Time	End	End

Methodology

FP828P

Five replicates of each powder reference material and sample were weighed into three different containment vessels, for a total of 15 replicates per powder sample. Approximately 0.25 g was analyzed in a LECO 502-186 Tin Foil Cup*, 0.30 g in a 502-338 Small Gel Cap, and 0.40 g in a 502-382 Medium Gel Cap. The samples were analyzed utilizing 502-912 Corn Starch @ 755 ppm N_2 as a calibrant. A single point calibration curve was utilized.

Five replicates of each liquid reference material and sample were weighed into two different containment vessels for a total of 10 replicates per liquid sample. Samples and reference materials were weighed into 502-040 Small Tin Capsules (0.30 g) and 502-825 Large Tin Capsules (0.50 g) then analyzed utilizing 502-602 Ammonium Solution @ 1002 ppm N_2 as a calibrant. A single point calibration curve was utilized.

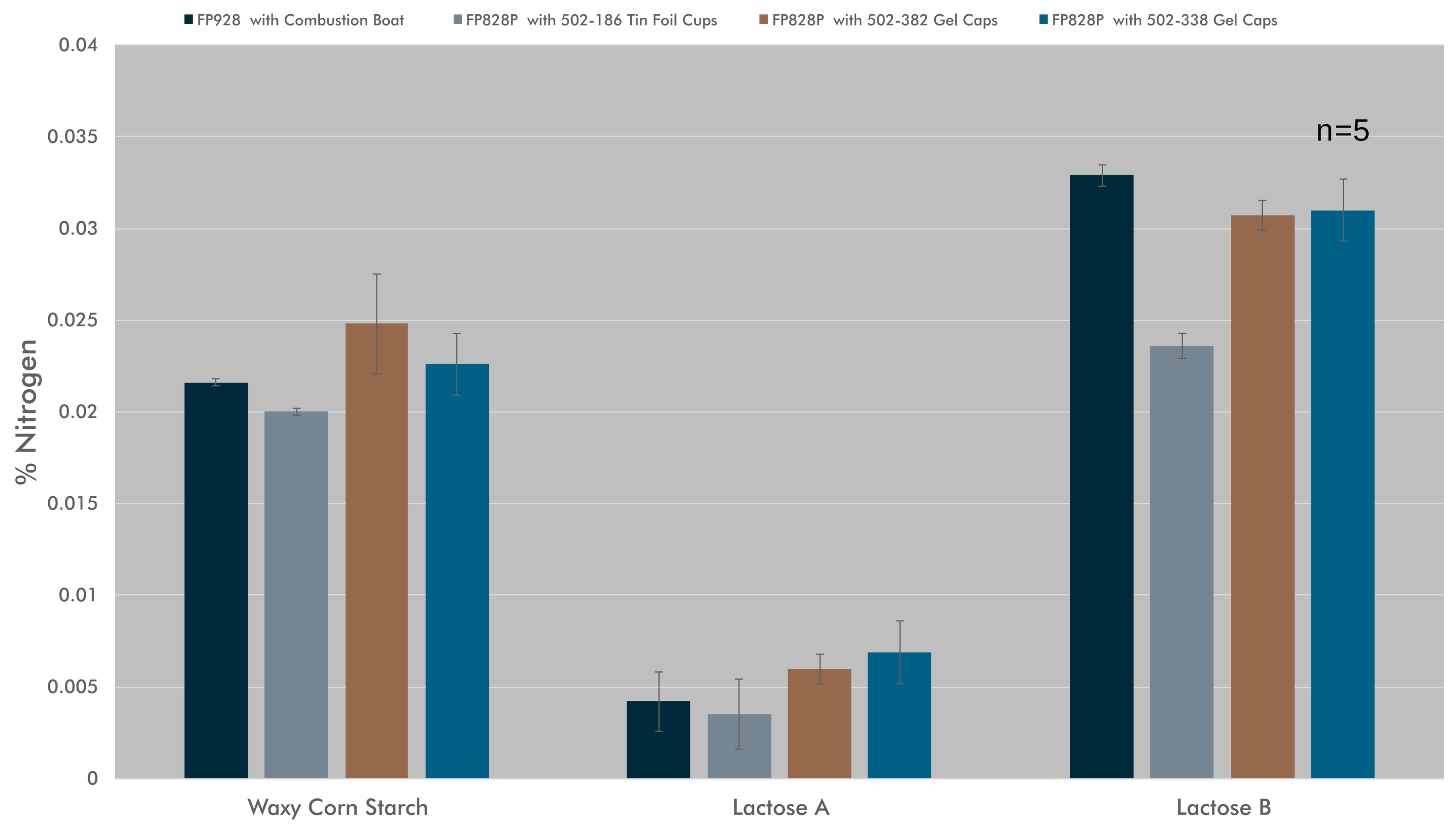
FP928

Five replicates of each powder reference material and sample were weighed into 528-203 Combustion Boats (~1.0 g), then analyzed utilizing 502-912 Corn Starch @ 755 ppm N_2 as a calibrant. A single point calibration curve was utilized.

Five replicates of each liquid reference material and sample were weighed into 528-203 Combustion Boats (~1.0 g) with LECO 502-343 Nickel Boat Liners, then analyzed utilizing 502-602 Ammonium Solution @ 1002 ppm N_2 as a calibrant. A single point calibration curve was utilized.

Sample Results

Powders

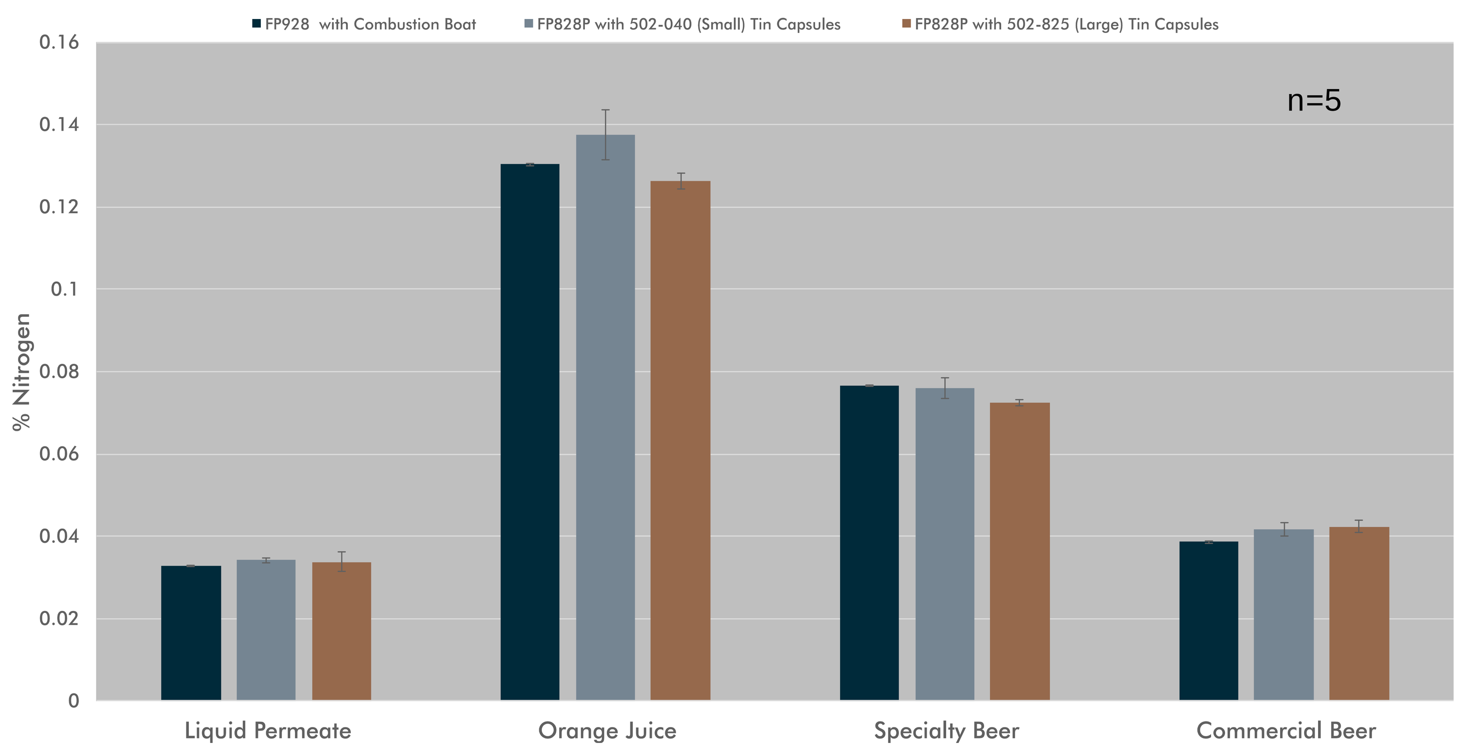


Standard Deviations of Powder Results

Sample	FP928 w/ Combustion Boat	FP828P w/ 502-186 Tin Foil Cups*	FP828P w/ 502-382 Medium Gel Caps	FP828P w/ 502-338 Small Gel Caps
Waxy Corn Starch	0.0002	0.0016	0.0006	0.0009
Lactose A	0.0027	0.0019	0.0007	0.0017
Lactose B	0.0002	0.0008	0.0008	0.0017

*Atmospheric blank applied (See note in conclusions)

Liquids



Standard Deviations of Liquid Results

Sample	FP928 w/ Combustion Boat	FP828P w/502-040 Tin Capsule (Small)	FP828P w/502-825 Tin Capsule (Large)
Liquid Permeate	0.0002	0.0006	0.0024
Orange Juice	0.0002	0.0061	0.0019
Specialty Beer	0.0001	0.0025	0.0007
Commercial Beer	0.0004	0.0016	0.0014

Conclusions

Powders

In general, the results are comparable to one another. Improved precision is typically obtained with the FP928 due to the larger sample mass and the elimination of the atmospheric interference. On the FP828P, the samples analyzed in the Tin Foil Cups had decreased precision due to the probable influence of the atmospheric blank (see note below). Increasing the sample mass using medium gel caps did improve the precision. Some variation can be attributed to sample inhomogeneity with certain materials.

Liquids

All results for each tested methodology match closely to one another. The FP928 demonstrated superior precision due to larger sample masses analyzed.

The objective of this poster was to establish method optimization parameters and procedures that can be utilized when analyzing materials containing low levels of Nitrogen. Liquids containing low concentrations of Nitrogen appear to run very well on both the FP828P and FP928. Increasing sample mass does improve precision for liquids. The powder samples were more problematic due to sample inhomogeneity and issues with atmospheric blank.

Note

Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. 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 masses of sucrose to the mass of samples being analyzed. Enter the actual mass of the sucrose. The nitrogen value obtained is considered the atmospheric blank, and can be automatically compensated for using the FP828P software.