TECHNICAL NOTE

elementar
EXCELLENCE IN ELEMENTS

ANKOM^{TDF}
DIETARY FIBER ANALYZER

Analysis of the dietary fiber content in food and animal feed

Introduction

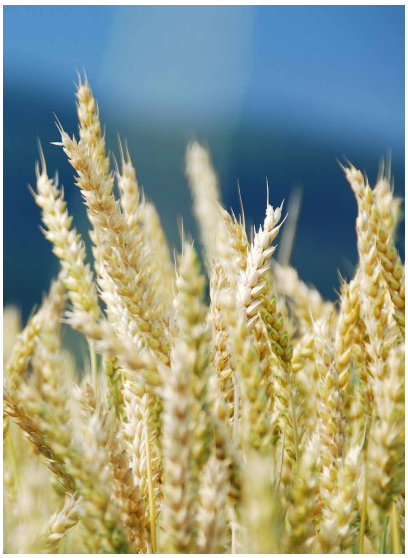
Dietary fiber aids digestion by increasing the bulk of food passing through the digestive system. It contributes to satiation, limiting the consumption of high-calorie alternatives. Therefore, the amount of dietary fiber in food and animal feed is a key nutrition factor to be considered when planning a diet. Dietary fiber is primarily found in grains, fruits, and vegetables and consists of long-chain carbohydrates. Depending on its affinity to water, dietary fiber is classified as either soluble (SDF) or insoluble (IDF). The sum of IDF and SDF is the total dietary fiber (TDF).

Determining the dietary fiber content of food and animal feed is a multi-step process involving several enzymes, buffer solutions, and filtering steps. The accepted methods include the slight modifications necessary for determining IDF and SDF or just TDF in a single sample. After appropriately digesting the sample, the remaining residue consists of dietary fiber, protein, and minerals (ash). Ash and protein are determined from duplicate samples and the fiber content is then computed by subtraction.

The protein content of the final digestion residue is determined from the total nitrogen content of the residue multiplied by the accepted factor. The Dumas combustion method has a long history of providing reliable, low-cost analysis of total nitrogen in a variety of samples, including the residue from dietary fiber analysis. A method that simplifies the interface between the sample digestion and protein determination would increase productivity and reduce costs for dietary fiber analysis. This note outlines a method for optimizing the interface between the **ANKOM^{TDF} Dietary Fiber Analyzer** and the **rapid MAX N exceed** Dumas analyzer from Elementar.

DIETARY FIBER ANALYSIS

ANKOM^{TDF} Dietary Fiber Analyzer
rapid MAX N exceed



Instrumentation

Much of the time-consuming dietary fiber analysis can be automated with the ANKOM^{TDF} Dietary Fiber Analyzer. This instrument automates the enzyme delivery, pH adjustments, heating, mixing, and filtering necessary to determine IDF, SDF, and/or TDF according to AOAC standards 985.29, 991.43, 2009.01, and 2011.25.

With over 110 years of experience producing elemental analyzers and more than 50 years of experience producing dedicated N/Protein analyzers, Elementar recently released the rapid MAX N exceed analyzer, which combines high-throughput and ease of operation with reliable determination of nitrogen, even at low concentrations and in difficult samples. The 90-position autosampler utilizes stainless steel crucibles that can hold up to 5 mL of liquid or 5 g of solid. Combining smart oxygen dosing right at the sample with Elementar's proprietary EAS REGAINER® and EAS REDUCTOR® technology, the rapid MAX N exceed has an unrivaled low price-per-sample.

Because the rapid MAX N exceed can measure up to 1 g of organic material, accurate, reproducible results can be obtained even from difficult samples, such as stringy or gritty foods that can be hard to homogenize. The sample ash is retained in the upright crucibles throughout the process and removed with the crucible after analysis, preventing diatomaceous earth (DE) blowing out and damaging the analyzer. This makes the Elementar rapid MAX N exceed the perfect instrument for analyzing the protein content in ANKOM^{TDF} Dietary Fiber Analyzer filter bags, as well as the total protein content of the original food and feed samples.

Method

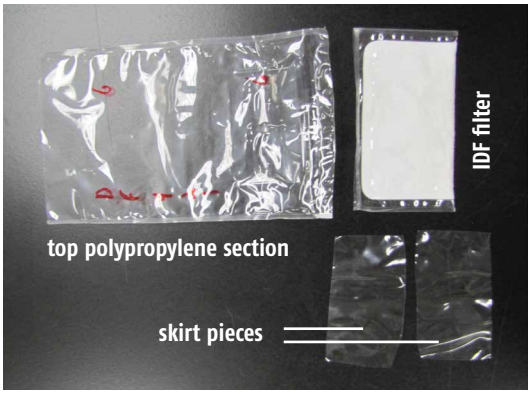
There are two options for transferring the samples from the ANKOM^{TDF} Dietary Fiber Analyzer to the rapid MAX N exceed for protein analysis: either with or without the filter bags. The polypropylene plastic of the filter bags is combustible, but must be divided in thirds to ensure complete combustion and avoid producing methane which can interfere with the nitrogen detection. Alternatively, the filter bag can be cut open and the contents removed. This allows for protein to be determined in one analysis, but may be more prone to sampling errors. This method works well for the steps that include DE, and is therefore recommended only for SDF or TDF portion analysis, but can also be used if DE is added to the IDF bag.

Method 1: Combustion with filter bag

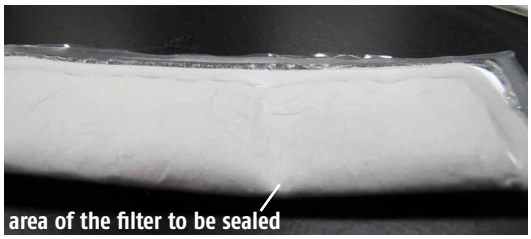
1. When the IDF and SDF bags are removed from the TDF instrument and rinsed with acetone, they are heat sealed just above the filter. The first step in preparation for ash/protein analysis is to cut the bag just above the seal.

a. The SDF bag is simply cut away from the upper bag section.

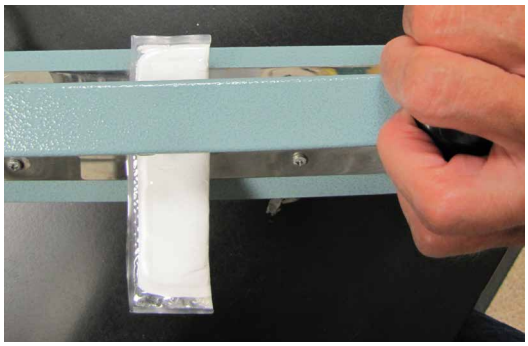
b. The IDF bag has a strip of polypropylene referred to as a "skirt" that covers both sides of the filter. This section of polypropylene is cut away.



2. The next step is to heat seal the two layers of the filter together to create at least three equal sections of filter. The SDF filter contains not only sample residue but also DE. The filter needs to be manipulated so that the mixture is away from the area to be sealed.



3. To seal the filter, the heat sealer should be set to 5. With the filter in place on the heat sealer, the arm of the sealer is pressed down. A click will be heard and the red light will be lit. It is at this time the heat is on. In a second or two, another click will be heard and the light will go off. At this point the heat is deactivated but the sealer arm should remain pressed down for 3-4 more seconds to allow the filter material to cool.



4. Repeat steps 2 and 3 to seal the filter again, creating three fairly equal sections.

5. Carefully cut down the middle of the sealed area of the filter. The seal on both sides of the cut will help to keep the sample residue and DE inside the filter.

6. The three portions of the bag are now each placed in their own crucible in the Teflon sample tray and heated to 120 °C for 30 minutes. While still hot, the bags are pressed into their crucibles with the steel pressing tool to remove any air that would contribute to the blank.

7. The prepared samples are now ready for analysis in the rapid MAX N exceed. The "TDF Bags" measurement mode optimizes temperatures and flows to ensure complete combustion and detection of nitrogen in samples containing up to 500 mg of total carbon (bag plus sample carbon).

8. The total protein in the residue is calculated simply as the sum of the absolute protein in the three bag parts.

Method 2: Combustion without filter bag

1. When the SDF/TDF bags are removed from the TDF instrument and rinsed with acetone, they are heat sealed just above the filter. The first step in preparation for ash/protein analysis is to cut the bag just above the seal for easier handling.

2. The contents of the bag are mixed thoroughly and the bag is then cut open.

3. The contents of the bag are then transferred into an empty, weighed crucible. Be careful to not scrape out and plastic fibers from the filter portion of the bag.

4. The total protein in the residue is calculated as follows:

$$TP_{residue} = \frac{TP_{sample} \times (m_{residue} + m_{DE})}{m_{sample}}$$

where:

$TP_{residue}$ is the total protein in the residue
TP_{sample} is the total protein in the measured sample
$m_{residue}$ is the total mass of residue
m_{DE} is the mass of diatomaceous earth used
m_{sample} is the mass of the measured sample

Method Validation Data

A key challenge of these methods is to reduce the blank protein (nitrogen) introduced with the DE and plastic filter bags. To illustrate the reliability of these methods, a series of blank measurements were conducted in 18–20 replicates. The values below are given as the mean blank ± one standard deviation.

	METHOD	VALUE [mg]
IDF Protein Blank	1	2.2 ± 0.4
IDF Overall Blank	1	-8.5 ± 1.5
IDF Protein Blank	2	2.6 ± 0.3
IDF Overall Blank	2	-9.2 ± 0.7
SDF Protein Blank	2	6.9 ± 2.3
SDF Overall Blank	2	-5.9 ± 1.9

In order to validate the complete process, three standard substances were analyzed for TDF and compared to the accepted values. Each sample was measured in six replicates. The values below are given as mean TDF percentage ± one standard deviation. The peanut butter sample required an additional initial defatting step via three hour soxhlet extraction.

SAMPLE	METHOD	MEASURED [%]	ACCEPTED [%]
Bran ERM 542	IDF: 2 SDF: 2	31.3 ± 0.5	30.5
Cereal SRM 3233	IDF: 2 SDF: 2	8.8 ± 0.3	9.0 ± 1.2
Peanut Butter SRM 2387	IDF: 1 SDF: 2	6.1 ± 0.3	5.57

Summary

The fast and environmentally-friendly Dumas method provides a reliable measurement of the protein content of digestion residue in dietary fiber analysis. With a well-developed and customized method, the interface between the **ANKOM^{TDF} Dietary Fiber Analyzer** and the **rapid MAX N exceed** Dumas analyzer from Elementar automates and optimizes what is otherwise a labor-intensive and time-consuming process.

The results of this coupled method show reliable results over a variety of applications and analysis needs. Furthermore, the rapid MAX N exceed expands the capabilities of an analysis lab to measure protein in an extensive variety of food and feed samples, handling liquids and solids, as well as plastic filter bags, with reliability and ease.

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