Kjeldahl vs Dumas—Overview and Comparison

The protein content is one of the key parameters in food and feed analysis and is essential for the characterization of the nutritional value of the material. Other important constituents are fat, fiber, carbohydrates, and moisture and ash content. In addition to nutrition labeling requirements, protein content can also play important roles in food processing. For example, the protein content in wheat flour determines the best use for the flour, with lower protein (~8%) typically being used for cakes or pastries and higher protein flours (~12%) being used for bread. There are a few spectroscopic techniques including near-infrared reflectance (NIR) and transmittance (NIT) that can predict protein content and other constituents; however, these methods are considered secondary methods that require extensive calibration work and are highly sensitive to even small matrix changes, which limits the scope of the NIR calibrations. More common methods for protein determination are the classical wet chemical Kjeldahl method and the combustion-based Dumas method. Both methods actually determine nitrogen and not the direct protein value. The protein value is calculated from the nitrogen value of the material using nitrogen-to-protein conversion factors and assumes that the nitrogen source in biological matrixes is protein. The nitrogen-to-protein factor can vary depending upon the food material and is often specified by governmental and international standard methods. For example: the nitrogen-to-protein multiplication factor for milk and dairy is 6.38, corn and cereals is 5.8, meat and fish is 6.25, soy beans is 5.71, and mushrooms is 4.17.

Figure 1: Two typical Dumas instrument from LECO, the 828 and 928 series

The Kjeldahl method was the dominant method since the late nineteenth century, when it was developed by Johan Kjeldahl who managed the lab of the Carlsberg Brewery in Copenhagen and wanted a reliable method to determine and evaluate the protein contents in the incoming barley brewing materials. The Kjeldahl nitrogen determination method is a classical wet chemistry digestion and titration method. Since the development of the method in 1883, it has been widely accepted for the total protein determination in feeds and food materials. Generally, the method uses the following steps:

1. Digestion of the sample in concentrated sulfuric acid with a catalyst converting the nitrogen within the sample to ammonium sulfate $(NH_4)_2SO_4$. The sample size is typically between 100 mg and one gram.
2. Neutralization of the digestate using concentrated sodium hydroxide (NaOH) converting the ammonium sulfate to form NH_3.
3. Distillation of the NH_3 into a standard acid to be back-titrated with a standardized alkali for quantification.

While the Kjeldahl method has compatibility with nearly all food materials and wide acceptance in the food and feed analytical and regulatory communities, there are many disadvantages to this method. First of all, the Kjeldahl method can have an incomplete recovery of nitrogen. The best and most effective Kjeldahl catalysts contain mercury or selenium and are no longer used due to environmental concerns and local regulations. Less effective $CuSO_4/TiO_4$-based Kjeldahl catalysts are typically used instead, resulting in a lower nitrogen recovery during the digestion step. The analysis times are long and can take multiple hours, depending on the exact procedure used and sample material being tested. The method involves several steps that use concentrated acids and bases, requiring trained laboratory technicians equipped with the proper safety equipment, use of fume hoods, and proper waste disposal challenges.

The combustion-based Dumas method was developed in the early 19th century by the French chemist Jean-Baptist Dumas. Even though the Dumas method is older, the Kjeldahl method was the dominant method for the first 100 years of use, as a combustion method required a higher level of technical skill compared to wet chemical methods. This has changed with advancements in modern analytical instrumentation, making the combustion nitrogen determination a well-established reference method.

Typically, the Dumas method determines nitrogen with the following steps:

1. The sample is combusted with oxygen, transferring the protein-nitrogen to a mixture of N_2/NO_x gas.
2. The NO_x is reduced to N_2 and other combustion gases (like excessive O_2, CO_2, and water vapor) are removed.
3. Nitrogen is detected with a thermal conductivity (TC) detector. The calculation of the protein content using the determined nitrogen content and a protein factor from this point on works identically to the Kjeldahl method.

Modern Dumas analyzers like the LECO 828 and 928 series use either tin foil, capsules, or combustion boats for sample introduction. Solid, liquid, or slurry samples can be entered without any change of the setup; additional liquid samplers or special procedures are not necessary. The systems run without matrix dependency due to the sample combustion taking place in a pure oxygen environment. Typical analysis times are ~3 minutes, and the nitrogen oxide reduction reagent chemicals can last up to 4000 analyses, depending on how the system is configured.

Figure 2: Combustion capsules, foils, and boats

The Dumas and Kjeldahl methods have been compared in many studies and papers over the years. Table 1 shows some typical values in comparison tests. (Example data collected from comparison and commissioning tests.)

Table 1: Comparison of Dumas and Kjeldahl Results for Typical Food Products

Food Type	*Nitrogen Content Dumas [%]*	*Nitrogen Content Kjeldahl [%]*
Milk powder	4.98	4.96
Raw milk	3.58	3.55
Blood meal	14.44	14.39
Wheat flour	2.66	2.67
Infant food	1.98	1.96
Ham	3.17	3.12
Rice flour	1.48	1.49

As seen in Table 1, the Dumas and Kjeldahl values agree for a wide variety of food materials tested. For some samples, the Kjeldahl method shows slightly lower values than Dumas. The overall difference for typical food and feed samples is normally less than 1-2% relative.

Many food laboratories are using the Dumas method instead of or in addition to the Kjeldahl method as an excellent method for the determination of protein in food and feed. Table 2 shows a small list of examples of official methods using the Dumas method in food analysis, showing that the Dumas method is gaining more and more importance compared to the Kjeldahl method. The only disadvantage to using the Dumas method over the Kjeldahl method is the higher cost of a Dumas-method instrument than a Kjeldahl method one, but the difference evens out quite quickly when the running costs are taken into account.

Table 2: Partial List of International Standards for the Dumas Method

Standard	*Description*	*Issuing Body*
AOAC 992.23	Crude protein in cereal grains and oils	AOAC
AOAC 992.15	Crude protein in meat and meat products	AOAC
AOAC 992.03	Crude protein in animal feed	AOAC
BA4E-93	Crude protein in oil seeds	AOCS
ISO 14891:2008	Determination of nitrogen content — Routine method using combustion	ISO
ISO 16634-1:2008	Determination of the total nitrogen content by combustion…Oilseeds and animal feeding stuffs	ISO
ICC Norm 167	Determination of crude protein in grain and grain products for food and feed after Dumas	ICC
OIV-MA-AS323-02A	Quantification of total nitrogen according to the Dumas method (musts and wines)	OIV
BVL L 03.00-27: and others	§ 64 LFGB German Food Law	Official Methods Germany

The advantages of the Dumas combustion method can be summarized with the following categories:

Speed: 3 minutes with Dumas vs hours of time with Kjeldahl

Throughput: Easily >150 samples can be analyzed in a 9-to-5 day with one LECO Dumas instrument

Cost per Analysis: The running costs of a combustion Dumas instrument will be far lower compared to classical wet chemistry Kjeldahl method

Environmental and Safety Issues: Combustion Dumas instruments eliminate the use of concentrated acids and bases, the need to dispose of the waste, and the fume hood requirements of the wet chemical Kjeldahl method

Efficiency and Automation: After the weighed sample is placed into the combustion Dumas instrument autoloader, all subsequent stages of the analysis are automated, with the result being calculated by the instrument's software and ready for transmission to a laboratory information management system (LIMS)

Standard Compliance: The combustion Dumas method is a well-established reference method as shown with the examples in Table 2.

These advantages will continue to drive the future replacement of the wet chemical Kjeldahl method with the combustion Dumas method within the food and feed industries.

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.*

LECO
EMPOWERING RESULTS