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DETERMINATION OF PROTEIN IN FOODS: COMPARISON OF THE DUMAS AND KJELDAHL METHODS

What is the best way to determine protein? To give a reasonable answer to this question in the article, let's compare the methods in detail, in particular, in terms of accuracy, safety, performance, cost and features of operation.

Classic or popular?

Protein content is one of the most important quality indicators that determine its nutritional value. The classic method for determining protein is the Kjeldahl method, but the Dumas method has recently become increasingly popular. So, Dumas or Kjeldahl? This article answers that question.

Which of the methods is arbitrage?

Both of these methods – both Dumas and Kjeldahl – are official, that is, arbitral methods. What does it mean? That they are recognized and approved by the world's leading bodies and organizations and their results are beyond doubt.

In Europe and in the United States, in recent decades, the Dumas method has become increasingly popular, gradually replacing the Kjeldahl method. You will understand why later.

Dumas: the history of the method

The method was developed by the French chemist Jean-Baptiste Dumas. The scientist invented it in 1831 during his studies. Subsequently, the method was named after him. Interestingly, the method was invented much earlier than the Kjeldahl method, but due to the difficulties with its implementation - the lack of gases of proper purity, the necessary reagents and catalysts - did not gain popularity. Gradually, the method was modified and automated. Automatic devices were

developed to implement it. From that moment on, the Dumas method began to compete with the Kjeldahl method as an arbitration method for determining protein in foods.

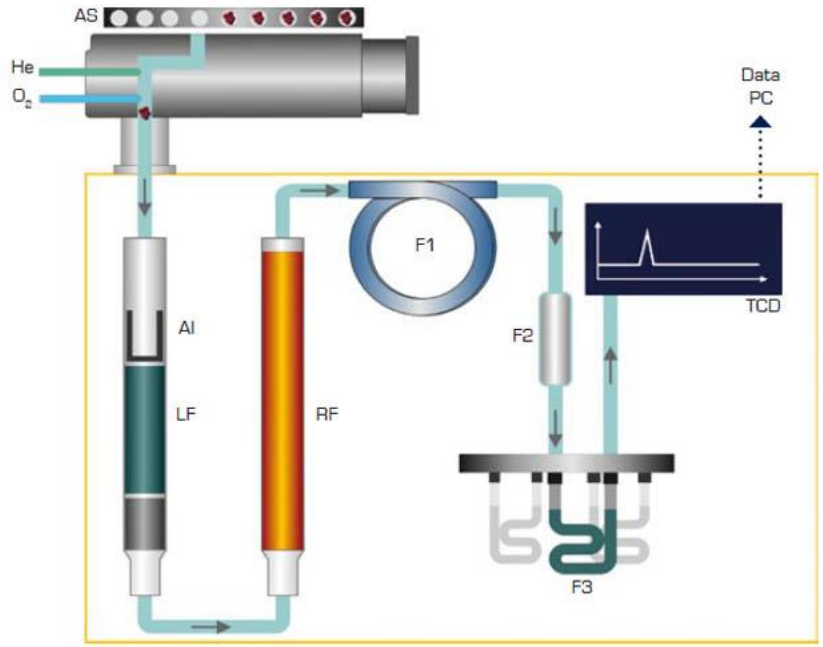
What are the stages of the Dumas method?

It begins with sample preparation. This stage is quite simple. For solid samples, they just need to be ground to a grind size of 0.5 mm. For liquids – stir.

Next are the main stages. This is the incineration of a sample at a temperature of more than 1000 °C. After that, the sample breaks down into elementary particles such as nitrogen oxides, water and carbon dioxide.

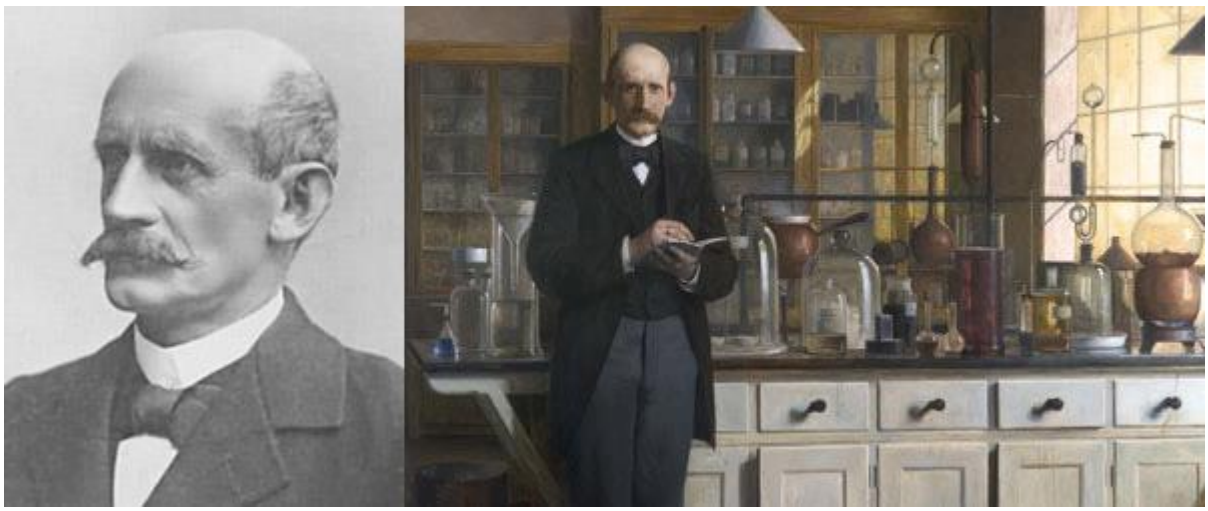
Next comes the reduction stage, during which the nitrogen oxides are oxidized, and pure nitrogen, water and carbon dioxide remain at the output.

Then carbon dioxide and water are extracted from the resulting mixture. What remains is pure nitrogen, which, in fact, is determined. Knowing the amount of nitrogen, we can find out the amount of protein by multiplying the amount of nitrogen by the appropriate factor.



AS - Autosampler; AI - Ash Insert; LF - Combustion Furnace; RF - Reduction furnace; F1 - Membrane system (Nafion); F2 - Absorption trap; F3 - self-regenerating adsorption trap; TCD - Thermal Conductivity Detector.

Kjeldahl: the history of the method



Johan Kjeldahl, in the Carlsberg laboratory in the 1880s

The method was developed by the Danish chemist Johan Kjeldahl at the end of the 18th century, when he was working in the Carlsberg laboratory. He was tasked with developing a fast, reproducible method for determining protein in grain. This was necessary to eliminate certain technological problems in the brewing process associated with different protein content in the initial raw material. He successfully coped with this task and invented a method, that was later called after his name. Due to its high accuracy, reproducibility and ease of implementation, the method immediately gained popularity and wide application. And today it is the most famous and widely used method for the determination of protein in food products all over the world.

What are the stages of the Kjeldahl method?

Like the Dumas method, the Kjeldahl method consists of three main stages: mineralization, distillation, titration. And for each of these stages, separate devices are needed, namely: mineralizer, steam distiller and titrator. But there are already devices that combine the process of distillation and titration.

Let's compare these two methods

Let's analyze the following criteria:

- accuracy,
- safety,
- performance,
- investment and exploitation.

Accuracy

If we talk about accuracy, we can see that the detection limit is better in the Dumas method. At the same time, both methods provide excellent reproducibility.

It should also be noted that the Kjeldahl method determines only organic nitrogen, while the Dumas method determines both organic and inorganic nitrogen. Therefore, as a rule, the Dumas method gives higher results than the Kjeldahl method. Although not always. After all, if we are talking about inorganic nitrogen, then these are, as a rule, nitrates and nitrites. And they may either not be present at all in the sample under study, or they may be in very small quantities. For example, if we are talking about cereal crops, then nitrates and nitrites are concentrated in the stem of the plant, and in the grain they are present in very small quantities or are completely absent.

Kjeldahl

Detection limits = 0.1 mg N

Reproducibility ≤1%

Determination of organic nitrogen only

Dumas

Detection limits = 0.001 mg N

Reproducibility ≤1%

Determination of both organic and inorganic nitrogen - nitrates and nitrites

The Dumas method determines total nitrogen, including inorganic nitrogen, for this reason it usually gives a slightly higher value than the Kjeldahl method.

Safety

If we talk about safety, then the undoubted advantage is on the side of Dumas. After all, the implementation of this method does not require the use of so-called wet chemistry. All reagents are non-toxic and do not require special permits for their use. However, to implement this method, high-purity gases will be required, namely oxygen for burning the sample and helium or argon as a carrier gas.

Kjeldahl

Traditional wet chemistry

Reagents:

- Sulfuric acid, 98%
- NaOH, 32-35%
- Boric acid, 4%
- Catalysts
- Titrant

Operator contact with heating elements

Дюма

Dry chemistry

Reagents and gases:

- Catalysts
- Copper for N recovery
- Compressed air, 99,6%
- Oxygen, 99,99%
- Helium / Argon, 99,99%

No operator contact with heating elements

Analysis Time and Performance

As for the analysis time, we can see that using the Kjeldahl method, we get the first results only after two hours. Using the Dumas method - after 3-4 minutes. But it should be noted here that using, for example, a 20-position mineralizer, it is possible to analyze 20 samples in less than 4 hours. Using the Dumas method, we will only need 3-4 minutes to analyze a single sample.

By productivity. If we talk about the Kjeldahl method, it directly depends on the capacity of the mineralizer and the degree of automation of the distiller and can fluctuate between 18 and 60 analyzes per day. The Dumas method allows up to 120 analyzes per day. It should also be noted that the Kjeldahl method allows only samples with the same matrix to be analyzed simultaneously in a batch, while the Dumas method - with different matrices.

Analysis Time and Performance

Kjeldahl

Not less than 2 hours

Productivity:

From 18 to 60 analyses per day

- If max. 60 analyses per day, then about 15000 per year
- If max. 18 analyses per day, then about 3000 analyses per year.

Samples with the same matrix in a batch.

Dumas

3-4 minutes per sample

Productivity:

Maximum 120 analyses per day, or about 25000 analyses per year.

High productivity, ease of operation (works without supervision).

Samples with different matrices in a batch.

Investment

In the figure you can compare the numbers. But it should be noted that the cost of an automatic system for the implementation of the Kjeldahl method can reach 26 thousand euros, and if equipped with an autosampler will be even more expensive than a Dumas analyzer.

Investment and Operation

Kjeldahl

Cost:

From 8 to 51 thousand euros

Higher cost of analysis.

Operation:

- Consists of several successive operations
- Work in series
- Requires operator intervention

Dumas

Cost:

From 35 to 56 thousand euros

20% lower cost of analysis

Operation:

- Sample needs to be prepared and weighed
- Safe operation
- Unattended operation

Operation

Here, too, the complete advantage is on the side of Dumas.

- The implementation of the Kjeldahl method consists of several sequential operations. It works only in batches. It is impossible to mineralize different types of samples at the same time. Even the most automated system requires operator intervention.
- In the case of Dumas, the operator only needs to prepare and weigh the sample. All stages of the analysis are automatic. The instrument can operate continuously and simultaneously with different types of samples. Does not require operator supervision.

Source:

https://apk.hlr.ua/ru/articles/opredelenie-belka-v-pishhevyix-produktax-sravnenie-metodov-dyuma-i-keldalya

Bogdan Bondaruk – Head of research direction “Chemical Analysis of Food Products by Arbitrage Methods”

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