Kjeldahl vs Dumas: Comparative Study of Nitrogen/Protein Analysis Methods

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Introduction

Nitrogen analysis plays a crucial role in determining protein content in food and feed products, essential for nutritional labeling, quality control, and regulatory compliance. Two widely used methods for nitrogen analysis are the Kjeldahl method and the combustion (Dumas) method which differ in principles but share the measurement of nitrogen content.

The classic manual Kjeldahl method has been a recognized reference method since around 1883 and was considered unchallenged for a long time. The Dumas method, much older (1833) has only been recognized in recent decades.

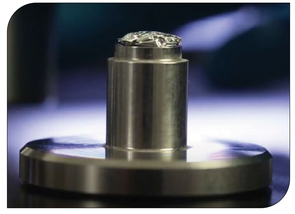
The Kjeldahl method involves wet chemical digestion of the sample using concentrated acids and catalysts, followed by distillation and titration to measure nitrogen content. It is a long-established method, highly reliable for various food and feed matrices. However, it is labor-intensive, requires hazardous chemicals, and generates significant waste, raising concerns about environmental sustainability.

In contrast, the combustion method (Dumas) uses high-temperature combustion to convert organic nitrogen into nitrogen gas (N_2), which is measured by a thermal conductivity detector. This method is faster and fully automated, making it ideal for high-throughput laboratories. It is also more environmentally friendly, as it avoids the use of hazardous chemicals and produces less waste.

This study compares the performance of both methods in terms of accuracy, efficiency, and suitability for a wide variety of sample types. The N-Realyzer was used for the combustion (Dumas) method, and the KJELDATHERM/ VAPODEST system was utilized for the Kjeldahl method, providing a comprehensive evaluation of both techniques.

Experimental

The samples were prepared as required for each technique. The N- Realyzer with the REAL-OS user interface was used for the Dumas experiments, while the KJEDLATHERM and VAPODEST and Titroline 5000 were used to perform the Kjeldahl experiments.



The homogeneous sample is introduced into the combustion tube of the N-Realyzer and undergoes oxidation to nitrogen oxides in an oxygen atmosphere, at elevated temperatures, and in the presence of a catalyst. The nitrogen oxides are then reduced back to nitrogen using copper as a reducing agent. During this process, side products such as water and carbon dioxide are removed by specific traps. Finally, the amount of nitrogen is detected using a thermal conductivity detector (TCD), and its concentration is determined by referencing a calibration performed previously with a substance of known nitrogen content.

General Preparation for the Dumas Samples

- Solid samples must have PS of <=1mm for best homogeneity.
- Liquid samples (e.g. milk/plant-based milks) were heated to 38-40 °C to ensure homogeneity, and then cooled to 25°C under mixing.
- Liquid samples (e.g. soy sauce/ other beverages) were mixed prior to weighing.
- Superabsorber was added to aqueous samples.
- Dumasorb was added for fatty aqueous samples.
- Sample weights 50-500 mg were used depending on sample type.
- Samples were weighed into tin foil and a an airtight package was created with the Dumapress.
- Transfer sample to numbered sample transfer plate, place on sample tray of X-Y autosampler of N-Realyzer

Parameter	Setting
Combustion Temperature	980 °C
Reduction Temperature	650 °C
Oxygen Dosage	Flow Rate: 200-300 ml/min; Total Oxygen: Avg 100-300 ml In exceptional cases higher

Preparation for the Kjeldahl Samples

- Liquid samples were heated to 38-40 °C to ensure homogeneity, and then cooled to 25°C under mixing.
- Liquid samples (e.g. milk/plant-based milks) were heated to 38-40 °C to ensure homogeneity, and then cooled to 25°C under mixing.
- Sample weight 0.1-10 grams were placed on weighing paper, put into 250 ml digestion tubes, and add chemicals; 20 ml of concentrated sulfuric acid, 2 Kjelcat-Tablets containing 10g of potassium sulphate, and 1g of copper sulfate. Use sulfuric acid to wash down any residue from the glass walls. These are then put in the KJELDATHERM and digestion parameters set to ensure a clear solution, followed by appropriate cooling. Post digestion the samples were put in the VAPODEST for a steam distillation followed by an end point titration on the Titroline 5000.

Results

Sample	Proficiency test Organization	% Nitrogen Dumas	SD	% NItrogen Kjeldahl	SD	Delta Dumas-Kjeldahl	Ring test mean value % N Kjeldahl	Upper limit Kjeldahl	Lower Limit Kjeldahl
Gluten Meal	GAFTA	3.592	0.015	3.528	0.007	0.064	3.560	3.766	3.354
Basmati Rice	GAFTA	1.380	0.008	1.350	0.004	0.030	1.363	1.321	1.404
Soy Meal	GAFTA	7.699	0.020	7.691	0.096	0.008	7.602	7.702	7.503
Barley	GAFTA	1.255	0.010	1.230	0.026	0.025	1.210	1.249	1.170
Boiled Sausage	LVU	2.318	0.007	2.295	0.012	0.023	2.259	2.381	2.126
Corn Chip	FAPAS	1.447	0.017	1.652	0.016	-0.005	1.660	1.722	1.598
Meat	AOAC	1.823	0.092	1.794	0.021	0.029	1.801	1.834	1.748
Porridge	FAPAS	1.916	0.008	1.860	0.010	0.054	1.820	1.960	1.690
Infant Formula	FAPAS	1.719	0.007	1.675	0.006	0.044	1.670	1.800	1.550
Yogurt	MUVA	0.771	0.002	0.770	0.003	0.001	0.771	0.781	0.761
High Temperature Pasteurized Milk	MUVA	0.551	0.003	0.549	0.002	0.002	0.553	0.559	0.547
Whey protein powder	MUVA	4.889	0.005	4.871	0.007	0.018	4.830	4.848	4.814

The Dumas method demonstrates equivalent or better precision compared to the Kjeldahl method, even with smaller sample sizes. Standard deviations for Dumas results fall within the allowable tolerances of recognized norms, including DIN ISO 14891 for dairy products and AOAC 992.15 for meat products. Furthermore, Dumas values meet the precision criteria of Kjeldahl norms, confirming both methods provide highly precise and comparable nitrogen and protein determinations.

Conclusion

The comparison highlights that there is no single "reference method" for nitrogen determination; instead, laboratories can choose between two distinct methods, each offering unique advantages.

The Kjeldahl method is highly versatile, accommodating a wide range of sample types and larger sample weights, making it ideal for applications requiring broad applicability and robustness, and reduced through-put. In contrast, the Dumas method excels in speed, delivering rapid results with minimal operator input, and requires less laboratory space, making it well-suited for high-throughput environments or facilities with limited space.

Both methods provide high precision and standard-compliant nitrogen determination, ensuring reliable results. The choice between the two depends on the specific needs and priorities of the laboratory, such as sample type, throughput requirements, and available resources.

Acknowledgements

For over 150 years, C. Gerhardt has been offering a wide range of food and beverage solutions such as the N-Realyzer state of the art, high through-put combustion analyzer for your nitrogen/protein analysis; HYDROTHERM automated acid hydrolysis and SOXTHERM® extraction system for fat determination; KJELDATHERM block digestion system and VAPODEST 500® steam distillation system for Kjeldahl nitrogen determination; or the FIBRETHERM® for feed fiber analysis. These automated and easy-to-operate instruments provide a safe and time-efficient alternative to manual processes, and improve analyses by removing human error.

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