Nitrogen & protein determination in milk powder

SpeedDigester K-439, Kjel Line and MultiDist:
Nitrogen and protein determination in milk powder according to the Kjeldahl method with Kjeldahl tablets and H_2O_2

1. Introduction

An easy and reliable method for the determination of total nitrogen and protein in milk powder, according to Kjeldahl is introduced below. The samples are digested using the SpeedDigester K-439 using Kjeldahl Titanium Tablets in combination with H_2O_2. A steam distillation protocol followed by a suitable boric acid titration is performed with the MultiKjel together with the Metrohm Eco Titrator. Coupling the new MultiKjel system and the Eco Titrator results in excellent performance with ease and speed of the analysis.

2. Experiment

Sample:
Milk powder 20.54 ± 0.729 g/100 g

Equipment:
MultiKjel coupled with Metrohm EcoTitrator (11K36531210), BasicKjel (11K36521110), SpeedDigester K-439(1154392000)

Procedure:
Weigh samples and reference substance and samples into nitrogen free paper boats. Add 15 ml Conc. H_2SO_4 and 2 Titanium Kjeldahl tablets. (For H_2O_2 assisted digestion refer to Table 1) Distillation and Titration parameters are as presented in Table 2. (For detailed procedure please download AN752/2021)

Table 1. Digestion parameters on SpeedDigester K-439

Step	Standard Kjeldahl digestion		H_2O_2 assisted accelerated digestion	
	Temperature [°C]	Time [min]	Temperature [°C]	Time [min]
Preheating	400	0	400	0
1	490	120	490	60
Cooling	-	35	-	35
Total Time	-	155	-	95

Table 2. Distillation parameters on Kjel line

Parameter	After standard Kjeldahl digestion	After H_2O_2 assisted digestion
H_2O Volume	60mL	50mL
NaOH Volume	63mL	45mL
Distillation Time	180s	180s
Titration Type	Boric Acid Titration	Boric Acid Titration
H_3BO_3 Volume	60 mL (4%)	60 mL (4%)
Sensor type	Potentiometric (pH)	Potentiometric (pH)
Endpoint pH	4.65	4.65
Titrant	H_2SO_4 0.1mol/L	H_2SO_4 0.1mol/L

3. Results

The results correspond well to the certified reference values with low relative standard deviations. The protein content results for the milk sample are presented in Table 3. All measured recovery rates for reference substance Tryptophan were within specifications. (Recoveries ≥ 98 %, RSD ≤ 1 %, n=3)

Table 3: Determined protein contents (rsd in brackets, n=5).

Product	Protein content [g/100 g] (RSD)	Digestion method	Kjel line instrument
Milk Powder	20.61 (0.12%)	Standard Kjeldahl	MultiKjel
Milk Powder	20.57 (0.20%)	H_2O_2 assisted	MultiKjel
Milk Powder	20.584 (0.38%)	Standard Kjeldahl	BasicKjel

4. Conclusion

The determination of nitrogen and protein in milk powder using the SpeedDigester K-439 and Kjel Line instruments provides reliable and reproducible results. These results correspond well to the certified reference-values with low relative standard deviations. On coupling with Eco-Tirator, MultiKjel systems offer easy automation without any manual handling after distillation and titration. Distillation with BasicKjel followed by separate titration provides a cost-friendly alternative with equally quantitative results. Digestion time can be reduced to half by H_2O_2 assisted digestion protocol.

5. References

Kjeldahl Optimizer App, AN752/2021, Operation Manual Kjel-/DistLine