

# Short Note No. 180/2015

## Nitrogen & protein determination in milk powder

*KjelDigester K-449, KjelMaster K-375 with KjelSampler K-376:  
Nitrogen and Protein Determination in Milk Powder According to the Kjeldahl Method by Colorimetric Titration*

In this note a reliable and efficient method for the determination of total nitrogen and protein in milk powder, according to EN ISO 8968-1:2014 [1], AOAC 991.20 [2] and AOAC 930.29 [3] is presented. The samples are digested using the KjelDigester K-449. The distillation and boric acid titration are performed with the KjelMaster K-375 by colorimetric titration and the KjelSampler K-376.

### 1. Introduction

Protein determination is one of the key analyses performed in the food industry. The samples require digestion with sulfuric acid to convert nitrogen to ammonium sulfate. After conversion to ammonia through alkalization with sodium hydroxide, the sample is steam distilled into a boric acid receiver, followed by a colorimetric titration with sulfuric acid solution. The measured nitrogen content is multiplied by a sample-specific protein factor (6.38 for dairy products) to obtain the protein content.

### 2. Experimental

**Equipment:** KjelDigester K-449 / KjelMaster K-375 with KjelSampler K-376

**Samples:** Skimmed milk powder with a protein content of 33 g/100 g, and whole milk powder (reference material) with a declared protein content of 26.44 g/100 g.

**Determination:** The samples were placed directly into a sample tube as described in Table 1. Two Titanium tablets and 15 mL of sulfuric acid (conc. 98 %) were added.

Table 1: Sample Weight

Samples	Sample weight [g]
Skimmed milk powder	0.4
Whole milk powder	0.5

The digestion was performed using the K-449, applying the temperature profile specified in Table 2.

Table 2: Temperature profile for digestion with the K-449

Step	Temperature [°C]	Time [min]
1	350	0
2	420	120
Cooling	---	35

After digestion the ammonia of the sample was distilled into a boric acid solution by steam distillation and titrated with sulfuric acid (Table 3) performed with the KjelMaster system K-375 / K-376 by colorimetric titration using the Sher indicator.

Table 3: Parameters for distillation and titration with the KjelMaster system K-375 / K-376

Method Parameters			
H <sub>2</sub> O volume	50 mL	Receiving solution	60 mL H <sub>3</sub> BO <sub>3</sub> 4 %
NaOH volume	60 mL	pH receiver solution	4.65
Reaction time	5 s	Titration solution	H <sub>2</sub> SO <sub>4</sub> 0.1 mol/L
Dist. mode	Fixed time	Titration mode	Online
Dist. time	180 s	Titration start	90 sec
Stirrer speed distillation	5	Measuring mode	Setpoint
Steam output	100%	Titration start volume	0 mL
Titration type	Boric acid	Stirrer speed titration	10
Sensor type	Colorimetric	Titration algorithm	Optimal

For colorimetric titration it's necessary to determine the setpoint of the boric acid solution in advance to the blank and sample determination. This procedure including specific preparation for the sensor is described in the Technical Note 179/2015 [4] Colorimetric titration procedure using Sher indicator.

### 3. Results

The recoveries of the whole milk powder reference were 99.3 %, rsd = 0.3 % (n=5). The determined protein contents are presented in Table 4.

Table 4: Determined protein contents (rsd in brackets, n=5)

Product	Protein content [g/100 g]
Skimmed milk powder	33.3 (0.4)
Whole milk powder	26.3 (0.3)

### 4. Conclusion

The determination of nitrogen and protein in milk powder using the KjelDigester K-449 and KjelMaster system K-375 / K-376 by colorimetric titration provides reproducible results. These results correspond well to the declared protein contents and with the results determined by potentiometric titration shown in the Application Note 180/2015 with low relative standard deviations (rsd) [5].

### 5. References

- [1] EN ISO 8968-1:2014 Milk and milk products-Determination of nitrogen content Part 1: Block-digestion method
- [2] AOAC 991.20 Nitrogen (Total) in Milk
- [3] AOAC 930.29 Protein in Dried Milk
- [4] Technical Note 179/2015 Colorimetric titration procedure using Sher indicator
- [5] Application Note 180/2015 for detailed information Operation Manual of KjelDigester K-446/K-449 Operation Manual of KjelMaster system K-375/K376