Application Note

078/2012

SpeedDigester K-436, K-439

KjelMaster K-375 with KjelSampler K-376

Comparison of different Kjeldahl Tablets for the Determination of Nitrogen and Protein in Milk according to the Kjeldahl Method

SHORT NOTE

Comparison of different Kjeldahl Tablets for the Determination of Nitrogen and Protein in Milk according to the Kjeldahl Method

The following introduces a simple and fast procedure for protein determination in milk according to the Kjeldahl method, as described in the DIN ISO 8968-1:2001, LFBG §64 L01.00-10/1, and AOAC 991.20 regulations. This Short Note compares the behaviour of the different types of Kjeldahl Tablets and summarizes their individual advantages. All the samples described in this ShortNote were digested together with sulfuric acid and different Kjeldahl Tablets with the SpeedDigester K-439 and then distilled and titrated with the Kjeldahl sampler system K-375/K-376.

Introduction

BUCHI offers different types of catalyst tablets for performing the standard Kjeldahl method. The Kjeldahl Tablet "Titanium" contains copper sulfate, titanium oxide and potassium sulfate. The tablet "Missouri" contains copper sulfate and potassium sulfate and the tablet "ECO" consists of a very low content of copper sulfate and potassium sulfate [1]. In this Short Note, milk was selected as sample material. The main focus of the experiments was to determine the individual behaviour of the different catalysts and to find out more about their influence on the total digestion time and the results. To see all results please refer to Application Note 078/2012.

Experimental

Instrumentation: SpeedDigester K-439,
KjelMaster K-375 with KjelSampler K-376

Samples: Strawberry milk drink with a labelled protein content of 3.0 g/100 ml.

Determination: Approx. 5 g of the homogenized milk drink were placed inside a sample tube. For every experiment with the different types of Kjeldahl Tablets, 2 tablets and the corresponding volume of sulfuric acid were added. The digestion was performed according to the "dairy products" method (K-439) with varied total digestion times of 85 and 115 min. After the digestion, the ammonia of the sample was distilled into a boric acid solution by steam distillation and titrated with sulfuric acid with the Kjeldahl sampler system K-375/K-376 (Table 1).

Table 1: Parameters for distillation and titration with the Kjeldahl sampler system K-375/K-376

Method Parameters			
H_2O volume	60 ml	Steam output	100%
NaOH volume	90 ml	Receiving solution	50 ml H_3BO_3 4%
Reaction time	5 s	Titration solution	H_2SO_4 0.1 mol/l
Dist. mode	Fixed time	Endpoint pH	4.65
Dist. time	180 s	Stirrer sp. titr.	7
Stirrer sp. dist.	5	Titr. algorithm	Optimal

Results

Figure 1 presents the protein contents of the milk drink determined with different types of tablets and after varying digestion times.

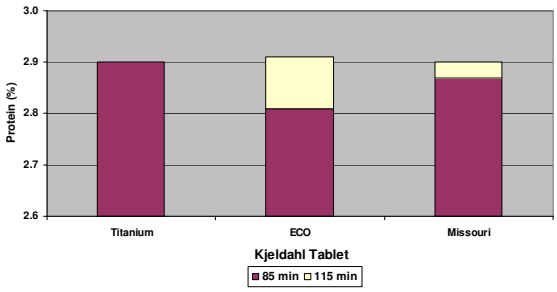


Figure 1: Results of the protein (%) determination in strawberry milk drink (n=3) with different catalysts after 85 and 115 min digestion time

Table 2 presents the time required by each catalyst for accomplishing a complete digestion. The experimental protein content [%] was re-calculated taking the density (1.030 g/ml) [2] into account in order to obtain the protein content as g/ 100 ml.

Table 2: Comparison of the complete digestion times for all catalysts

	Titanium	ECO	Missouri
Digestion time (min)	85	115	115
Protein content (%)	2.9	2.9	2.9
Protein g/100 ml	3.0	3.0	3.0
RSD (%)	0.24	0.04	0.13

Conclusion

Depending on the Kjeldahl Tablet used milk samples can be digested within 85 – 115 min. The fastest option is the tablet "Titanium" which digests the sample in only 85 min. With a total of 115 min. it takes the tablet "Missouri" longer to completely digest the sample. This catalyst is mentioned in some official methods [3]. The tablet "ECO" contains the lowest copper content of all described tablets. It also takes this catalyst 115 min. to fully digest the sample.

References

[1] 070/2011 Technical Note
[2] Kessler, H.-G.: *Lebensmittel-und Bioverfahrenstechnik, Molkereitechnologie*, 4. Auflage 1996
[3] AOAC 991.20

For more detailed information please refer to Application Note 078/2012

1 Introduction

An easy and reliable method for determining nitrogen and protein contents in milk according to the Kjeldahl method, as described in the ISO 8968-1:2001, LFBG §64 L01.00-10/1, and AOAC 991.20 regulations, is introduced in chapter 8. All described samples were digested with the SpeedDigester K-436 and K-439. The distillation and boric acid titration are performed with the Kjeldahl sampler system K-375/K-376. In a first step, the new BUCHI Kjeldahl Tablets for performing standard Kjeldahl were compared with each other in order to learn more about the advantages of each type of tablet. For the experiments tryptophan was selected as a reference substance, whole milk and strawberry milk drink were chosen as sample products.

2 Equipment

- SpeedDigester K-436, K-439
  (the parameters used for K-436 are also valid for SpeedDigester K-425)
- Scrubber K-415 TripleScrub ^{ECO}
- KjelMaster K-375 with KjelSampler K-376
- Analytical balance (accuracy ± 0.1 mg)

3 Kjeldahl Tablets

Three different types of Kjeldahl Tablets are available for performing standard Kjeldahl. Due to the different weight of the tablets, different volumes of sulfuric acid had to be used during the experiments in order to obtain the optimal ratio of catalyst to sulfuric acid. The composition of each tablet, their weight and the used volume of sulfuric acid are summarized in Table 1.

Table 1: Composition of the Kjeldahl Tablets Titanium, ECO and Missouri

Name	Weight [g]	Potassium sulfate [%]	Copper sulfate pentahydrate [%]	Titanium(IV) oxide [%]	Sulfuric acid [ml]
Titanium	3.7	94.3	2.8	2.8	15
ECO	4	99.9	0.06	-	16
Missouri	5	99.6	0.4	-	20

4 Chemicals and Materials

- Sulfuric acid conc 98 %, Fluka (84727)
- Titanium, BUCHI Kjeldahl Tablet (11057980)
- Missouri, BUCHI Kjeldahl Tablet (11057982)
- ECO, BUCHI Kjeldahl Tablet (11057983)
- Sodium hydroxide 32 %, Brenntag (81980-452)
- Boric acid 4 %, 200 g boric acid, Brenntag (80948-155) diluted to 5 l with deionized water, pH adjusted to 4.65
- Sulfuric acid 0.1 mol/l, Fluka (35357) standard solution
- Neutralization solution for the Scrubber: 600 g sodium carbonate, calcined, technical, Synopharm (0179420) about 2 ml ethanol and a spatula tip of bromthymol blue, Fluka (18460) diluted to 3 l with distilled water
- DL-tryptophan (assay >99 %), Alfa Aesar (L05936)

5 Samples

- Whole milk with a labelled protein content of 3.5 g/100 ml
- Strawberry milk drink with a labelled protein content of 3 g/100 ml

The samples were purchased at a local supermarket.

6 Procedure

The determination of nitrogen and protein in whole milk and strawberry milk includes the following steps:

- Homogenization of the sample by shaking
- Digestion of the sample, using SpeedDigester K-439 or K-436
- Distillation and titration of the sample, using Kjeldahl sampler system K-375/K-376

6.1 Digestion method - tryptophan (verification of the method)

- Place approx. 0.18 g tryptophan in a 300 ml sample tube
- Add two tablets of the to be tested catalyst and the corresponding volume of sulfuric acid (98 %) described in Table 1
- Prepare additional blanks, chemicals without sample
- Carefully suspend the sample by gently swirling the tube
- Connect the Scrubber K-415 to the SpeedDigester K-436 or K-439 for absorbing the acid fumes created during digestion
- Insert the rack containing the samples into the preheated unit
- Digest the samples according to the parameters listed in Table 2 or 10, depending on the catalyst type

6.2 Digestion method - samples

- Place approx. 5 g of the sample in a 300 ml sample tube
- Add two tablets of the to be tested catalyst and the corresponding volume of sulfuric acid (98 %) described in Table 1
- Prepare additional blanks, chemicals without sample
- Carefully suspend the sample by gently swirling the tube
- Connect the Scrubber K-415 to the SpeedDigester K-436 or K-439 for absorbing the acid fumes created during digestion
- Insert the rack containing the samples into the preheated unit
- Digest the samples according to the parameters listed in Table 2 or 10, depending on the catalyst type

Table 2: Temperature profile for the comparison of the Kjeldahl Tablets with the K-439

Step	Temperature [°C]	Time [min]
Preheating	480	-
1	480	10
2	550	10
3	490	variable [1]
Cooling	-	30

[1] In order to be able to determine the progression of the digestion, different digestion times were applied in the third digestion step (490 °C), with varying time periods of 65 min and 95 min. In total the digestion time, of step 1 to 3, was of 85 min and 115 min.

6.3 Distillation and titration

Distill the samples according to the parameters listed in Table 3

Table 3: Distillation and titration with the Kjeldahl sampler system K-375/K-376

Method parameters KjelMaster K-375			
H_2O volume	60 ml	Titration solution	H_2SO_4 0.1 mol/l
NaOH volume	90 ml[2] / 60 ml[3]	Sensor type	Potentiometric
Reaction time	5 s	Titration mode	Standard
Distillation mode	Fixed time	Measuring mode	Endpoint pH
Distillation time	180 s	Endpoint pH	4.65
Stirrer speed distillation	5	Stirrer speed titration	7
Steam output	100 %	Titration start volume	0 ml
Titration type	Boric acid	Titration algorithm	Optimal
Receiving solution vol.	50 ml		

[2] Volume of sodium hydroxide used for the comparison of the Kjeldahl Tablets

[3] Volume of sodium hydroxide used for the Application Note, chapter 8

6.4 Calculation

The results are calculated as a percentage of nitrogen. In order to calculate the protein content of the sample, the nitrogen content is multiplied with a sample-specific protein factor. The following equations (1), (2), and (3) are used to calculate the results. For the reference substance, the purity of the tryptophan is considered in equation (4).

$$w_N = \frac{(V_{Sample} - V_{Blank}) \cdot z \cdot c \cdot f \cdot M_N}{m_{Sample} \cdot 1000} \quad (1)$$

$$\%N = w_N \cdot 100\ \% \quad (2)$$

$$\%P = w_N \cdot PF \cdot 100\ \% \quad (3)$$

$$\%N_{Try} = \frac{\%N \cdot 100}{P} \quad (4)$$

w_N	: weight fraction of nitrogen
V_{Sample}	: amount of titrant for the sample [ml]
V_{Blank}	: mean amount of titrant for the blank [ml]
z	: molar valence factor (1 for HCl, 2 for H_2SO_4)
c	: titrant concentration [mol/l]
f	: titrant factor (for commercial solutions normally 1.000)
M_N	: molecular weight of nitrogen (14.007 g/mol)
m_{Sample}	: sample weight [g]
1000	: conversion factor [ml/l]
%N	: percentage of weight of nitrogen
%P	: percentage of weight of protein
PF	: sample-specific protein factor (6.38 for dairy products)
$\%N_{Try}$	: percentage of weight of nitrogen corrected for the purity of reference substance tryptophan (%)
P	: purity of the reference substance tryptophan (%)

7 Results for the comparison of the Kjeldahl Tablets

7.1 Recovery of tryptophan

The results of the nitrogen determination and the recovery of tryptophan (assay > 99 %) after 85 and 115 min, are presented in Table 4-5. The nominal value of tryptophan is 13.72 % of nitrogen.

Table 4: Results of the recovery of nitrogen in tryptophan with K-439 after 85 min

Tryptophan	m_{Sample} [g]	V_{Sample} [ml]	$\%N_{Try}$	Recovery [%]	Average [%]	RSD [%]
Titanium[a]	0.1733	8.480	13.530	98.6	98.8	0.4
Titanium[a]	0.1858	9.139	13.623	99.3		
Titanium[a]	0.1816	8.872	13.522	98.6		
ECO[b]	0.1741	8.341	13.236	96.5	95.6	1.1
ECO[b]	0.1805	8.581	13.143	95.8		
ECO[b]	0.1939	9.080	12.963	94.5		
Missouri[c]	0.1820	8.778	13.335	97.2	96.8	0.3
Missouri[c]	0.1827	8.762	13.259	96.7		
Missouri[c]	0.1786	8.566	13.253	96.6		

[a] Blank 0.194 ml (n=3)
[b] Blank 0.198 ml (n=3)
[c] Blank 0.201 ml (n=3)

Table 5: Results of the recovery of nitrogen in tryptophan with K-439 after 115 min

Tryptophan	m_{Sample} [g]	V_{Sample} [ml]	$\%N_{Try}$	Recovery [%]	Average [%]	RSD [%]
ECO[b]	0.1770	8.652	13.522	98.6	98.3	0.4
ECO[b]	0.1822	8.880	13.490	98.3		
ECO[b]	0.1886	9.157	13.448	98.0		
Missouri[c]	0.1724	8.497	13.617	99.3	99.2	0.3
Missouri[c]	0.1916	9.381	13.558	98.8		
Missouri[c]	0.1891	9.317	13.641	99.4		

[b] Blank 0.194 ml (n=3); [c] Blank 0.201 ml (n=3)

7.2 Protein determination in milk

The results of the determination of nitrogen and protein contents in whole milk and strawberry milk drink after 85 and 115 min are presented in Tables 6-9.

Table 6: Results of the recovery of nitrogen and protein in strawberry milk drink with K-439 after 85 min

Milk drink	m_{Sample} [g]	V_{Sample} [ml]	%N	Protein [%]	Average [%]	RSD [%]
Titanium[a]	5.1331	8.545	0.456	2.91	2.9	0.2
Titanium[a]	5.0626	8.392	0.454	2.89		
Titanium[a]	5.0838	8.450	0.455	2.90		
ECO[b]	5.0963	7.932	0.425	2.71	2.8	3.1
ECO[b]	5.1436	8.432	0.448	2.86		
ECO[b]	5.1437	8.444	0.449	2.87		
Missouri[c]	5.1347	8.467	0.451	2.88	2.9	0.2
Missouri[c]	5.1361	8.460	0.450	2.87		
Missouri[c]	5.1399	8.449	0.450	2.87		

[a] Blank 0.086 ml (n=3)
[b] Blank 0.090 ml (n=3)
[c] Blank 0.098 ml (n=3)

The experimental protein content [%] was re-calculated taking the density of (1.030 g/ml) [2] into account in order to obtain the protein content as g/100 ml.

Table 7: Results of the recovery of nitrogen and protein in strawberry milk drink with K-439 after 115 min

Milk drink	m_{Sample} [g]	V_{Sample} [ml]	%N	Protein [%]	Average [%]	RSD [%]
ECO[b]	5.1212	8.529	0.456	2.91	2.9	0.0
ECO[b]	5.1004	8.495	0.456	2.91		
ECO[b]	5.1222	8.537	0.456	2.91		
Missouri[c]	5.0566	8.415	0.455	2.90	2.9	0.1
Missouri[c]	5.1903	8.615	0.454	2.90		
Missouri[c]	5.0821	8.459	0.455	2.90		

[b] Blank 0.194 ml (n=3)
[c] Blank 0.201 ml (n=3)

With the catalyst Titanium and Missouri a digestion time of 85 min is sufficient to have corresponding protein content to the labelling of the strawberry milk drink of 3.0 g/100 ml. To compare the results, the average in % was multiplied with the density of 1.030 g/ml. With the catalyst ECO an elongation of 30 min is necessary to show a complete digestion.

In Table 8 and 9, the results of the determination of nitrogen and protein in whole milk are presented.

Table 8: Results for the recovery of nitrogen and protein in whole milk with K-439 after 85 min

Whole milk	m_{Sample} [g]	V_{Sample} [ml]	%N	Protein [%]	Average [%]	RSD [%]
Titanium[a]	5.0600	9.768	0.530	3.38	3.4	0.3
Titanium[a]	5.0499	9.783	0.532	3.39		
Titanium[a]	5.0279	9.693	0.529	3.38		
ECO[b]	5.0091	9.557	0.523	3.34	3.3	0.5
ECO[b]	5.0326	9.521	0.519	3.31		
ECO[b]	5.0129	9.563	0.523	3.34		
Missouri[c]	5.0486	9.617	0.522	3.33	3.3	0.1
Missouri[c]	5.0543	9.607	0.521	3.33		
Missouri[c]	5.0223	9.554	0.522	3.33		

[a] Blank 0.194 ml (n=3)
[b] Blank 0.198 ml (n=3)
[c] Blank 0.201 ml (n=3)

Table 9: Results of the recovery of nitrogen and protein in whole milk with K-439 after 115 min

Whole milk	m_{Sample} [g]	V_{Sample} [ml]	%N	Protein [%]	Average [%]	RSD [%]
ECO[b]	5.0174	9.688	0.530	3.38	3.4	0.1
ECO[b]	5.0396	9.736	0.530	3.38		
ECO[b]	5.0645	9.803	0.532	3.39		
Missouri[c]	4.9668	9.564	0.528	3.37	3.4	0.3
Missouri[c]	5.0572	9.725	0.528	3.37		
Missouri[c]	5.0429	9.744	0.530	3.38		

[b] Blank 0.194 ml (n=3)
[c] Blank 0.201 ml (n=3)

To compare the results, the average in % was multiplied with the density of 1.030 g/ml. The labelled protein content for the whole milk is 3.5 g/100 ml. Just with the catalyst Titanium a complete digestion in 85 min is possible. For Missouri and ECO an elongation of 30 min is necessary to reach the labelled protein content of 3.5 g/100 ml.

7.3 Conclusion drawn from the comparison of the different types of Kjeldahl Tablets

With the reference material tryptophan, a good differentiation between the Kjeldahl Tablets is possible. Titanium is the only catalyst, with a recovery (98.8 %; rsd 0.4 %) within the specification of > 98 % [1], after a digestion time of 85 min. Missouri and ECO need an elongation of 30 min the reach the specification (Figure 1).

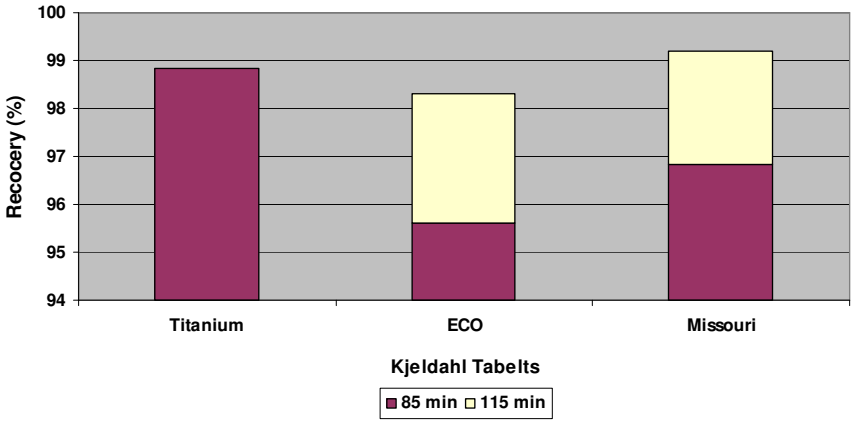
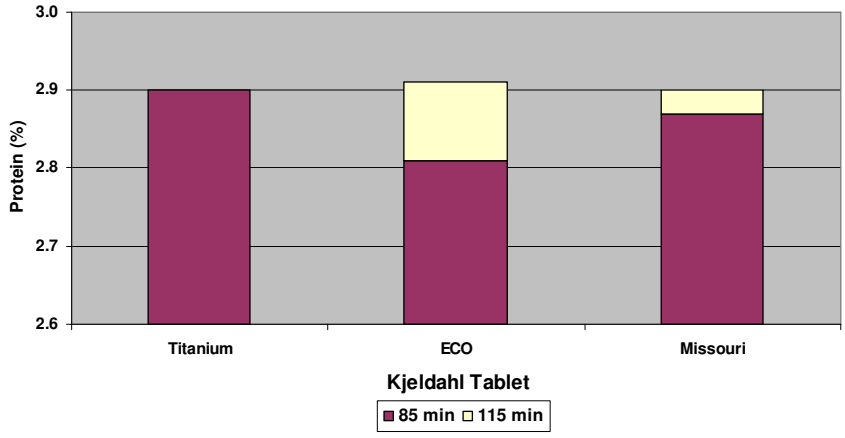


Figure 1: Recovery of tryptophan with different types of Kjeldahl Tablets and digestion times.

In Figure 2 the behaviour of the Kjeldahl Tablets with the sample material strawberry milk drink and whole milk are presented.



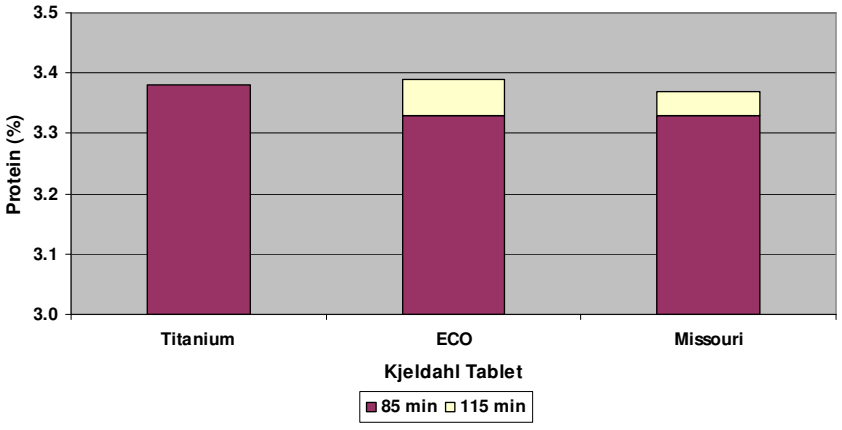


Figure 2: Up: Protein content (%) of strawberry milk drink (n=3) using different Kjeldahl Tablets and digestion times; Below: Protein content (%) of whole milk (n=3) using different Kjeldahl Tablets and digestion times.

The results of the samples are also presented in Table 6-9. The behaviour is similar to the results with the tryptophan. Just the catalyst Titanium is able to digest the samples in 85 min. The catalyst ECO and Missouri need an elongation of 30 min. It is possible, that the combination of copper and titanium helps to digest the ring-bonded nitrogen faster, than the copper alone. In the official regulations [1], a combination of copper sulfat and potassium sulfat, similar to the catalyst Missouri is described.

Table 10: Comparison of the Kjeldahl Tablets

Kjeldahl Tablet	Total digestion time [min]	Advantage
Titanium	85	Shortest total digestion time
ECO	115	Lowermost copper content
Missouri	115	Similar to AOAC 991.20

For the digestion with the K-439 and the K-436, the fastest Kjeldahl Tablet, “Titanium”, was used for the following application.

8 Results for the digestion conducted with the SpeedDigester K-439 and K-436

The following chapter describes the digestion of the milk samples whole milk and strawberry milk drink which were conducted with the SpeedDigester K-436 and K-439. The tablet “Titanium” was the only catalyst used for this experiment, the total digestion time was of 85 min.

The preparation of the samples and the calculation of the results are similar to the ones described in Chapter 7. Two tablets “Titanium” and 15 ml of sulfuric acid (98 %) were used (Table 1) for the experiment. The total time of the digestion conducted with the K-439 was of 85 min respective 90 min for K-436. All parameters are specified in Table 11.

Table 11: Parameters for the digestion with K-439 und K-436

	K-439		K-436	
Step	Temperature [°C]	Time [min]	Heating Level	Time [min]
Preheating	480	-	8.5	10
1	480	10	8.5	10
2	550	10	9.5	15
3	490	65	8.5	65
Cooling	-	30	-	30

- If the liquid inside the sample tube is not clear and of a blue-green color after the digestion, digest for an additional 30 min as described in step 3
- Let the samples cool down to room temperature

NOTE: If the samples are placed in the cooling position it takes them approx. 30 min to cool down; if they are left in the heating chamber it takes at least 60 min.

8.1 Digestion with the Kjeldahl Tablet Titanium and K-439

8.1.1 Recovery of tryptophan

The results of the recovery of nitrogen in tryptophan (assay > 99 %) are presented in Table 12. The nominal value of tryptophan is 13.72 % of nitrogen. The determined values are within the specification of > 98 % [1].

Table 12: Results of the recovery of nitrogen in tryptophan with K-439

Tryptophan	m_{Sample} [g]	V_{Sample} [ml]	$\%N_{Try}$	Recovery [%]
Sample 1	0.1733	8.48	13.530	98.6
Sample 2	0.1858	9.139	13.623	99.3
Sample 3	0.1816	8.872	13.522	98.6
Average	-	-	**13.56**	**98.8**
Rsd [%]	-	-	**0.1**	**0.1**

The mean blank volume for this sample was 0.194 ml (n = 3).

8.1.2 Protein determination in milk

The results of the determination of nitrogen in strawberry milk drink and whole milk are presented in Tables 13 and 14.

Table 13: Results of the determination of nitrogen and protein in strawberry milk drink with K-439

Milk drink	m_{Sample} [g]	V_{Sample} [ml]	%N	%P
Sample 1	5.1331	8.545	0.456	2.91
Sample 2	5.0626	8.392	0.454	2.89
Sample 3	5.0838	8.45	0.455	2.90
Average	**-**	**-**	**0.455**	**2.9**
Rsd [%]	**-**	**-**	**0.2**	**0.2**

The mean blank volume for this sample was 0.194 ml (n = 3).

Table 14: Results of the determination of nitrogen and protein in whole milk with K-439

Whole milk	m_{Sample} [g]	V_{Sample} [ml]	%N	%P
Sample 1	5.0600	9.768	0.530	3.38
Sample 2	5.0499	9.783	0.532	3.39
Sample 3	5.0279	9.693	0.529	3.38
Average	**-**	**-**	**0.530**	**3.4**
Rsd [%]	**-**	**-**	**0.3**	**0.3**

The mean blank volume for this sample was 0.194 ml (n = 3).

The experimental protein content [%] was re-calculated taking the density (1.030 g/ml) into account in order to obtain the protein content as g/100 ml. For the strawberry milk drink, the protein content is 3.0 g/100 ml, for the whole milk 3.5 g/100 ml. The values correspond to the labelled ones, described in chapter 5.

8.2 Digestion with Kjeldahl Tablet Titanium and the K-436

8.2.1 Recovery of tryptophan

The results of the recovery of nitrogen in tryptophan (assay > 99 %) are presented in Table 15. The nominal value of tryptophan is 13.72 % nitrogen. The determined values are within the specification of > 98 % [1].

Table 15: Results of the recovery of nitrogen in tryptophan with K-436

Tryptophan	m_{Sample} [g]	V_{Sample} [ml]	$\%N_{Try}$	Recovery [%]
Sample 1	0.1755	8.632	13.597	99.1
Sample 2	0.1817	8.964	13.650	99.5
Sample 3	0.1886	9.267	13.605	99.2
Average	**-**	**-**	**13.62**	**99.3**
Rsd [%]	**-**	**-**	**0.2**	**0.2**

The mean blank volume for this sample was 0.199 ml (n = 3).

8.2.2 Protein determination in milk

The results of the determination of protein in strawberry milk drink and whole milk are presented in Tables 16 and 17.

Table 16: Results of the determination of nitrogen and protein in strawberry milk drink with K-436

Milk drink	m_{Sample} [g]	V_{Sample} [ml]	%N	%P
Sample 1	5.0527	8.43	0.456	2.91
Sample 2	5.2099	8.659	0.455	2.90
Sample 3	4.9259	8.227	0.457	2.91
Average	**-**	**-**	**0.456**	**2.91**
Rsd [%]	**-**	**-**	**0.0**	**0.0**

The mean blank volume for this sample was 0.199 ml (n = 3).

Table 17: Results of the determination of nitrogen and protein in whole milk with K-436

Whole milk	m_{Sample} [g]	V_{Sample} [ml]	%N	%P
Sample 1	4.9758	9.602	0.529	3.38
Sample 2	5.0644	9.74	0.528	3.37
Sample 3	5.0349	9.756	0.532	3.39
Average	**-**	**-**	**0.530**	**3.38**
Rsd [%]	**-**	**-**	**0.4**	**0.4**

The mean blank volume for this sample was 0.199 ml (n = 3).

The experimental protein content [%] was re-calculated taking the density (1.030 g/ml) into account in order to obtain the protein content as g/100 ml. For the strawberry milk drink, the protein content is 3.0 g/100 ml, for the whole milk 3.5 g/100 ml. These values correspond to the labelled ones, described in chapter 5.

9 Comparison to Standard Methods

The standard methods DIN EN ISO 8968-1:2001 and LFGB §64 L01.00-10/1 are identical. AOAC 991.20 Part “Traditional Method” does not use sucrose and water as blanks. All other parts are identical with DIN EN ISO 8968-1:2001 and LFGB §64 L01.00-10/1. The differences to this application are shown in Table 18.

Table 18: Differentiation to the standard methods

	This application note	Standard methods	Notes/Impact
Sample tube	300 ml	500 - 800 ml	No impact
Catalyst	2 x 3.7 g Tablets cont. - 47.7 % K_2SO_4 - 2.8 % TiO_2 - 1.8 % $CuSO_4$	15 g K_2SO_4 + 1 ml $CuSO_4$ Solution[1)]	Easy to handle specially in routine analytics. The choice of catalyst does not influence the result, but the digestion time
Sulfuric acid	15 ml	25 ml	No impact. Same ratio of sulfuric acid / catalyst
Water	60 ml	300 – 400 ml	The K-375 generates steam in a separated vessel; therefore it is not necessary to add such a high amount of water to the digested sample as described in the standard methods.
Sodium hydroxide	60 ml (Conc.: 32 %)	75 ml (Conc.: 50 %)	No impact. Same ratio of sodium hydroxide / sulfuric acid. Sodium hydroxide 32% is gentler to the pump than higher concentrated alkali.
Blank with sucrose for determination of samples	no	DIN EN ISO and LFGB §64 use 0.85 g and 5 ml water for blank in addition to the chemicals.	No difference observed between blanks with and without sucrose.
Titration	Boric acid titration	AOAC: back titration	No impact. The results are equal with boric acid titration or back titration.

[1)] 5 g $CuSO_4$ x 5 H_2O diluted to 100 ml deionized Water

10 Conclusion

The determination of nitrogen and protein contents in whole milk and strawberry milk drink with the help of the different types of Kjeldahl Tablets provides reliable and reproducible results which correspond to the labelled values of the sample products with low relative standard deviations. The total digestion time can vary between 85 and 115 min depending on the type of Kjeldahl Tablets and digestion unit used and can therefore be adapted to meet the individual needs. Two important characteristics of the latest version of the fully-automatic Kjeldahl sampler system, KjelMaster K-375 and KjelSampler K-376, are the short processing time as well as the reduced need for user attendance. In combination with the unique SpeedDigester models, the time needed for sample analysis is significantly reduced and the throughput is therefore increased.

11 References

[1] AOAC 991.20
[2] Kessler, H.-G.: *Lebensmittel-und Bioverfahrenstechnik*, *Molkereitechnologie*, Verlag A. Kessler, Freising, 4. Auflage 1996
DIN EN ISO 8968-1:2001
LFGB §64 L01.00-10/1

Operation manual of SpeedDigester K-425 / K-436
Operation manual of SpeedDigester K-439
Operation manual of Scrubber K-415
Operation manual of Kjeldahl sampler system K-375/K-376

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