

Selective nitrogen determination methods related to Kjeldahl

KjelMaster System K-375 / K-376, KjelDigester K-449, Scrubber K-415

1. Introduction

For nitrate containing fertilizers the AOAC 955.04 D standard describes a sample digestion method (TKN+) that overcomes the influences of nitrate in samples. For urea and / or nitrate containing samples such as fertilizer, soil, sludge, bio waste and related waste, dedicated nitrogen determination procedures have to be applied.

2. Experiment

Sample:

The samples can be seen in Table 2. For further information please download the full application note from the website.

Equipment:

KjelMaster System K-375 / K-376 with pH electrode, Sample tubes 300 mL (037377), KjelDigester K-449, Scrubber K-415 TripleScrub^{ECO}, Devarda splash protector (043335), Sample tubes 500 mL (043982)

Procedure:

The Samples are digested and afterwards distilled. After the Distillation the samples are titrated.

Table 1. Parameters and settings with the KjelMaster System K-375 / K-376.

Parameters	Settings TKN	Settings TKN+	Nitrate / Nitrite / Devarda
Reaction time	5 s	5 s	300 s
Distillation time	180 s	200 s	300 s
Titration type	Boric acid	Boric acid	Boric acid
Sensor type	Standard	Potentiometric	Potentiometric



3. Results

The Results of different nitrogen determinations are shown in Table 2.

Table 2. Results of different Nitrogen determinations (n=3).

Sample	TKN			TKN (TKN+)		Ammonium distillation		Devarda	
	Total Nitrogen w/w [%]	Total Kjeldahl Nitrogen w/w [%]	RSD [%]	Total Nitrogen w/w [%]	RSD [%]	N from ammonium [%]	RSD [%]	N from nitrate / nitrite origin [%]	RSD [%]
Glycine	18.66	18.66	0.06	18.66	0.14	0.00	-	0.00	-
Urea	46.65	46.65	0.02	46.65	0.04	0.00	2.36	0.00	14.59
Ammonium dihydrogen phosphate	12.18	12.18	0.08	12.18	0.23	12.18	0.08	0.00	0.00
Ammonium nitrate	35.01	17.50	1.54	35.01	0.35	17.50	0.26	17.50	0.87
½ glycine + ½ ammonium nitrate	26.83	18.08	0.81	26.83	0.34	8.75	0.47	8.75	0.65
½ glycine + ½ sodium nitrate	17.57	9.33	4.47	17.57	0.30	0.00	-	8.24	0.71
⅓ glycine + ⅓ ammonium nitrate + ⅓ ammonium dihydrogen phosphate	21.95	16.12	1.03	21.95	0.49	9.89	0.39	5.83	0.78
⅓ glycine + ⅓ sodium nitrate + ⅓ ammonium dihydrogen phosphate	15.78	10.28	4.01	15.78	0.17	4.06	0.88	5.49	1.00

4. Conclusion

The Kjeldahl method is applicable to amines, amides, amino acids and their derivatives but generally fails to give quantitative results when nitrogen is in N=N and N-O linkages [10]. Nevertheless, the nitrate (NO₃⁻) and nitrite (NO₂⁻) content is included in the result, when the TKN+ method according to AOAC 955.04 D is applied [7, 8, 11]. With the Devarda distillation, NO₃⁻ and NO₂⁻ are selectively determined [8, 10]. For further information please download the full application note from the website.

5. References

Application Note No. 252/2016: Selective nitrogen determination methods related to Kjeldahl

[1] http://petrowiki.org/Gas_as_fertilizer_feedstock

[2] Glibert, P.M., Harrison, J., Heil, C. et al.; Escalating Worldwide use of Urea – A Global Change Contributing to Coastal Eutrophication, Biogeochemistry, 77: 441, (2006).

[3] Bremner J. M., Recent research on problems in the use of urea as a nitrogen fertilizer, Volume 69 of the series. Developments in Plant and Soil Sciences pp 321-329 (1995).

[4] Panyachariwat N., Steckel H., Stability of urea in solution and pharmaceutical preparations, J Cosmet Sci. M ay-Jun;65(3):187-95, (2014).

[5] Standard Methods for the Examination of Water and Wastewater, 4500-Norg Nitrogen (organic), (1999).

[6] KjelOptimizer App: <http://www.buchi.com/en/service-support/scientific-mobile-apps>

[7] AOAC 955.04 for total nitrogen in nitrate containing fertilizers.

[8] BUCHI Application note 029/2010, Nitrogen Determination in Nitrate Containing Fertilizers according to AOAC 955.04-D (Kjeldahl Method) – IR Digestion.

[9] AOAC 2001.11 Protein (crude) in animal feed, forage (plant tissue), grain and oilseeds.

[10] <http://galbraith.com/wp-content/uploads/2015/08/E7-1-Nitrogen-by-the-Kjeldahl-MethodGLI-Method-Summary.pdf>

[11] BUCHI Application note 116/2013, Nitrogen determination in sodium nitrate according to the Devarda method.