APPLICATION NOTE

E-K-001-2015/A1

TKN Determination in Water, Waste Water and Sludge according to the Kjeldahl method

Reference: **AOAC 973.48 Nitrogen (Total) in Water; EPA 351.3; Standard Method EN 25663, ISO 5663-1984**

Tested with **VELP Scientifica DKL 20 Automatic Kjeldahl Digestion Unit** (Code S30100210) and **UDK 169 Automatic Kjeldahl Analyzer with AutoKjel Autosampler** (Code S30200160)

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NITROGEN DETERMINATION IN WATER
KJELDAHL METHOD

Introduction

Nitrogen ends up in the environment mainly through agricultural processes, and thereby also ends up in water. The main source of nitrogen compounds in water are inorganic fertilizers that mainly contain nitrates, but also ammonia, ammonium, urea and amines. After fertilization, crops take up a relatively small part of added nitrogen compounds, namely 25-30%. The residue ends up in groundwater and surface water through soil. Organic fertilizers (such as guano) mainly contain nitrogen as proteins, urea or amines, which have different mechanisms of absorption. Nitrogen compounds are also applied in several different industries. Thereby other nitrogen compounds, such as nitrous oxide applied in anaesthetics, can be produced. Nitric acid, urea, hydrazine and amines are other products from nitrogen industries. Nitrogen compounds are by-products of colouring and synthetic agent production.

From an analytical point of view, the properties of water, waste water and sludge samples can vary considerably depending upon their source; in unpolluted waters, low nitrogen levels make it necessary to use large sample volumes, whereas high concentrations of surface active agents in some waste waters can also cause foaming during the initial step of the digestion so the volume required by the analysis is lower.

Total Kjeldahl Nitrogen Determination in Water, Waste Water and Sludge and according to the Kjeldahl Method

The modern Kjeldahl method consists in a procedure of catalytically supported mineralization of organic material in a boiling mixture of sulfuric acid and sulfate salt at high temperature until SO_3 fumes are given off and the solution turns limpid. During the process the organically bonded nitrogen is converted into ammonium sulfate.

Alkalizing the digested solution liberates ammonia which is quantitatively steam distilled and determined by titration.
This method covers the determination of total Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

For low nitrogen content samples, VELP suggests to follow the pre-defined digestion method n° 26 with the following temperature ramps: 60 minutes a 150 °C, 60 minutes at 250 °C and 120 minutes at 370 °C.
For high nitrogen content samples, VELP suggests to set the following temperature ramps: 60 minutes at 150 °C, 60 minutes at 250 °C, 60 minutes at 370 °C and 60 minutes at 420 ° C.
Regarding the distillation and titration step, the concentration of titrant solution (0,01N or 0,1N) can be modified depending on the properties of water sample.

Sample

Sludge coming from a waste water treatment plant Expected Nitrogen range: > 500 mg N/l (0.05% TKN)

Sample Digestion

Stir the sample into a beaker using a VELP magnetic stirrer at 700 rpm.
Put 10 ml of sample into a 250 ml test tube, by using a pipette.
For each sample, add in the test tube:

- 2 catalyst tablets Kjtabs VCM (code A00000274; 3.5 g K_2SO_4, 0.1 g $CuSO_4$*$5H_20$ Missouri)
- 2 antifoam tablets VS (code A00000283)
- 20 ml concentrate sulphuric acid (96-98%)

Prepare some blanks with all chemicals and without the sample.
Connect the Digestion Unit to a proper Aspiration Pump (JP code F30620198) and a Fume Neutralization System (SMS Scrubber code F307C0199) to neutralize the acid fumes created during digestion phase.
Since the analyzed sample is very rich of nitrogen, it has been digested for 60 minutes at 150 °C, 60 minutes at 250 °C, 60 minutes at 370 °C and 60 minutes at 420 ° C.

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Distillation and Titration

Let the test tubes cool down to 50-60 °C.
Condition the **UDK 169 with AutoKjel Autosampler** unit by performing the Automatic Check-up and Wash-down in the Menu-System.

Distill the samples according to the following parameters (pre-defined method n° 26):

- H_2O (dilution water): 50 ml
- NaOH (32 %): 70 ml
- H_3BO_3 (4 % with indicators): 30 ml
- H_2SO_4 (0.1 N) as titrant solution
- Protein factor: 0

Distillation & Titration analysis time: from 4 minutes for one test.

Typical Results on Sludge

The results are automatically calculated by UDK 169 as percentage of nitrogen.

Sample volume (ml)	TKN %
10	0.071
10	0.071
10	0.071
10	0.070
10	0.068
10	0.071
Average ± SD%	**0.070 ± 0.001**
RSD% *	**1.722**
Nitrogen Expected range: > 500 mgN /l (0.05% TKN)	

***** RSD% = (Standard Deviation * 100) / Average

The complete procedure was verified by using 5 ml of glycine standard solution (3%) containing 28 mg of nitrogen. as reference substance. The obtained recovery falls into the expected range: between 98 % and 102 %.

Conclusions

The determination of TKN in water, waste water and sludge using VELP DKL 20 and UDK 169 gives reliable and reproducible results in accordance with the expected range.

Benefits of Kjeldahl method by using DKL 20 and UDK 169 with AutoKjel Autosampler are:

- High level of precision and reproducibility
- Maximum productivity and full automation
- Worldwide official method
- Reliable and easy method
- Time saving
- Moderate running costs

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