APPLICATION NOTE

E-K-006-2023/A1

TKN Determination in Water and Wastewater

Reference**: Internal method based on 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), **KS 1000 Scrubber** (code F307A0660) and **UDK 169 Automatic Kjeldahl Nitrogen Protein Analyzer** (code F30200165)

NITROGEN DETERMINATION IN WATER
KJELDAHL METHOD

Introduction

Nitrogen-containing compounds act as nutrients in streams, rivers, and reservoirs. The major routes of entry of nitrogen into bodies of water are municipal and industrial wastewater, septic tanks, feed lot discharges, animal wastes (including birds and fish), runoff from fertilized agricultural fields and lawns and discharges from car exhausts. Bacteria in water quickly convert nitrites [NO_2^-] to nitrates [NO_3^-] and this process uses up oxygen. High nitrates in drinking water can cause digestive disturbances in people.
From an analytical point of view, the properties of water, wastewater 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 wastewaters 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, wastewater and sludge according to the Kjeldahl Method

The modern Kjeldahl method consists of a procedure of catalytically supported mineralization of organic material in a boiling mixture of sulfuric acid and sulfate salt at high temperatures 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 all the nitrogen concentrations in water samples, VELP suggests setting the following method: 60 minutes at 420°C. The below table shows the indicative sample volume depending on the nitrogen concentration.

Nitrogen concentration (mgN / l)	Sample volume (ml)
< 10	75
10-50	50
> 50	25

Sample

Urea diluted solutions with different concentrations of nitrogen: 5, 50 and 100ppm

Sample Digestion

- Stir the sample into a beaker using a VELP magnetic stirrer at 700 rpm;
- Put the proper amount 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 $CuSO_4 \cdot 5H_20$ Missouri)
  - Mineralization rod (Code A00000431 pack. 10 pcs) is placed in each test tube with the cava side down, in order to avoid the formation of bubbles, foam, violent shocks and acid splashes.
  - 8 ml concentrated sulphuric acid (96-98%)
- Prepare some blanks with all chemicals except the sample and mineralization rod;
- Connect the Digestion Unit to KS 1000 Scrubber (code F307A0660) to neutralize the acid fumes created during the digestion phase. For samples with low nitrogen content (< 50 ppm), it is recommended the use of the accessory code A00000477 Condensation kit for water samples. The accessory is strictly required when the total sample volume in the digestion unit is more than 500 ml.

Example:

Digestion Unit	Sample volume for each test tube
DK 6	> 80 ml
DK - DKL 8	> 60 ml
DKL 12	> 40 ml

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Distillation and Titration

Let the test tubes cool down to 50-60 °C.
Condition the **UDK 169** unit by performing the Automatic Check-up and Wash-down in the Menu-System.
Distil the samples by selecting the method "**27 – Total nitrogen in water**" in the operating manual. Using 0.1N Titrant solution.

Typical Results on Urea Solutions

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

Sample volume (ml)	mg N	ppm
75	0.39	5.17
75	0.40	5.29
75	0.38	5.12
Average ± SD%	**0.39 ± 0.01**	**5.20 ± 0.09**
Nitrogen Expected value: 5ppm		

Sample volume (ml)	mg N	ppm
50	2.49	48.72
50	2.49	49.66
50	2.50	50.02
Average ± SD%	**2.49 ± 0.01**	**49.867 ± 0.15**
Nitrogen Expected value: 50 ppm		

Sample volume (ml)	mg N	ppm
25	2.50	99.95
25	2.51	100.37
25	2.51	100.26
Average ± SD%	**2.51 ± 0.01**	**100.19 ± 0.22**
Nitrogen Expected value: 100 ppm		

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Conclusions

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

Benefits of the Kjeldahl method by using DKL 20 and UDK 169:

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

Connecting the UDK 169 to the VELP Ermes Cloud platform it IS possible to easily monitor and control the distillation step of the analysis in real-time via PC, smartphone or tablet, improving your laboratory experience.

Moreover, the UDK 169 can be used in combination with the **AutoKjel autosampler**, for a highly productive system, since it is capable of autonomously processing up to 24 samples with standard test tubes (250 ml) or 21 Jumbo test tubes (400 ml).

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