

TKN determination in water and waste water using colorimetric titration

KjelDigester K-449, KjelMaster K-375 with Optrode and KjelSampler K-376:

Colorimetric determination of Total Kjeldahl Nitrogen in water and waste water and determination of Limit of Detection LOD and Limit of Quantification LOQ

1. Introduction

A reliable method for the colorimetric determination of Total Kjeldahl Nitrogen (TKN) in water and waste water using the Optrode sensor, according to ISO 5663 [1], DIN EN 25 663 [7] and the methods listed in 40 CFR part 136.3 [6], is presented. In this study, the Limit of Detection and Limit of Quantification, which are a benchmark for the method's sensitivity, were determined for water samples. To test the system's performance, the nitrogen recovery of different concentrations of a urea reference sample was tested.

2. Experiment

Sample:

Urea stock solution: 1.0719 g urea diluted to 1000 mL with deionized water and about 0.5 mL concentrated sulfuric acid for preservation. Resulting nitrogen concentration of the stock solution: 0.500 mg N/mL

Equipment:

KjelDigester K-449 (the parameters used are also valid for the K-446)
User Protection Shield (BUCHI 11057889)
Scrubber K-415 TripleScrub^{ECO} with TKN Set (BUCHI 11057333)
KjelMaster K-375 with colorimetric sensor
Optrode-KjelSampler K-376 (the parameters used are also valid for the K-377)

Procedure:

The samples are digested and then distilled. After the distillation a colorimetric titration is followed.

Table 1. Parameters and Setting of the K-375 / K-376.

Parameters	Samples
Reaction time	5 s
Distillation time	180 s
Titration type	Boric acid
Sensor type	Colorimetric



3. Results

The Results of the determination of TKN are shown in Table 2.

Table 2. Results of the recovery of TKN in urea stock solution with different volumes (n=3).

Sample	Volume [ml]	TKN in sample [mg/L]	Ø Measured TKN [mg/L]	Ø Recovery [%]
Urea stock solution	200	0.5	0.504	100.7
Urea stock solution	25	50	49.775	99.6

4. Conclusion

The determination of TKN (Total Kjeldahl Nitrogen) in water using the KjelDigester K-449, the KjelMaster system K-375 / K-376 and the colorimetric sensor Optrode provides reliable and reproducible results. The recovery of the urea stock solution was excellent with low standard deviations. According to DIN, the LOD is 0.044 mg nitrogen/L and the LOQ is 0.131 mg nitrogen/L for 200 mL sample volume. For further information please download the full application note from the website.

5. References

Application Note No. 338/2018: TKN determination in water and waste water using colorimetric titration

- [1] ISO 5663, Water quality – Determination of Kjeldahl nitrogen – Method after mineralization with selenium
- [2] Standard Methods for the Examination of Water and Waste Water, 4500-NorgB, nitrogen (organic) macro-Kjeldahl Method, <https://www.standardmethods.org/doi/full/10.2105/SMWW.2882.090>, accessed April 1st 2019
- [3] Standard Methods for the Examination of Water and Waste Water, 4500-NH3, nitrogen (ammonia), titrimetric method, <https://www.standardmethods.org/doi/full/10.2105/SMWW.2882.087>, accessed April 1st 2019
- [4] Technical Note No. 335/2018 Colorimetric titration with Optrode sensor
- [5] DIN 32 645 Nachweis-, Erfassungs- und Bestimmungsgrenze
- [6] 40 CFR Part 136.3 table IB, Guidelines Establishing Test Procedures for the Analysis of Pollutants; Total Kjeldahl Nitrogen, <https://ecfr.io/Title-40/pt40.25.136>, accessed on March 29th 2019.
- [7] DIN EN 25 663, Determination of Kjeldahl Nitrogen in Water
- [8] ASTM D3590-11, Standard Test Methods for Total Kjeldahl Nitrogen in Water
- [9] AOAC 973.48, Nitrogen (Total) in Water, Kjeldahl Method
- [10] PAI-DK01, Nitrogen, Total Kjeldahl by Block Digestion, Steam Distillation, Titrimetric Detection, revised 12/22/94, Perstorp Analytical