

Short Note No. 118/2013

TKN determination in water and waste water

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

Determination of TKN (Total Kjeldahl Nitrogen) in water and waste water according to the Kjeldahl Method

The determination of TKN (Total Kjeldahl Nitrogen) in water and waste water is a routine procedure for quality assurance. A simple and fast procedure for the TKN determination in water, according to ISO 5663, DIN EN 25 663 and 40 CFR part 136.3, is introduced below. The sample is digested with sulfuric acid and the Kjeldahl Tablet Titanium using the KjelDigester K-449, followed by distillation and titration with the KjelMaster system K-375/K-376.

1. Introduction

TKN is one of the key parameters for the evaluation of water and its pollution. The samples require digestion with sulfuric acid to convert nitrogen into ammonium sulfate. After conversion to ammonia through the alkalization with sodium hydroxide, the sample is distilled into a boric acid receiver by steam distillation, followed by a titration with sulfuric acid solution.

2. Experimental

Equipment: KjelDigester K-449 / KjelMaster K-375 with KjelSampler K-376

Samples: Urea stock solution 0.493 mg N/mL, surface water from lake constance and surface water from pond threelinden.

Determination: The samples were added directly into a sample tube, depending on the nitrogen content as described in Table 1. One Titanium tablet, boiling aids to avoid bumping and 8 mL of sulfuric acid (conc. 98 %) were added. Before starting the digestion the user protection shield must be affixed.

Table 1: Volume for each sample

Nitrogen content	Sample volume [mL]	Digestion Time
50 – 100 mg N/L	25	50
20 – 50 mg N/L	50	70
10 - 20 mg N/L	100	90
< 10 mg N/L	200	160

The digestion was performed using the K-449, applied by the parameters specified in Table 2, the total digestion time depends on the sample volume described in Table 1. The method was verified by using an urea stock solution as the reference.

Table 2: Temperature profile for digestion with the K-449

Step	Temperature [°C]	Time [min]
1	250	0
2	420	See Table 1
Cooling	---	25

After digestion the ammonia of the sample was distilled into a boric acid solution by steam distillation and titrated with sulfuric acid (Table 3) performed by the KjelMaster system K-375/K-376.

Table 3: Parameters for distillation and titration with the KjelMaster system K-375/K-376.

H ₂ O volume	50 mL	Receiving solution	50 mL H ₃ BO ₃ 2 % + KCl
NaOH volume	40 mL	Titration solution	H ₂ SO ₄ 0.01 mol/l
Reaction time	5 s	Titration mode	Standard
Dist. mode	fixed time	Endpoint pH	4.65
Dist. time	180 s	Stirrer sp. Tir.	7
Stirrer sp. dist	5	Tit. algorithm	Optimal
Steam output	100%		

3. Results

The determined TKN values of the urea stock solution and the recoveries are presented in Table 4.

Table 4: Determined TKN contents and recoveries (rsd in brackets, n=5)

Sample volume	TKN [mg/L]	Recovery [%]
25 mL	59.1	99.9 (0.6)
	78.3	99.4 (0.4)
	97.9	99.4 (0.4)
50 mL	29.4	99.6 (0.3)
	39.2	99.6 (0.2)
	49.0	99.5 (0.3)
100 mL	12.2	99.3 (0.5)
	14.7	99.3 (0.4)
	19.6	99.3 (0.0)
200 mL	1.2	97.0 (4.3)
	2.4	98.5 (1.0)
	4.9	98.7 (0.3)
	9.8	99.2 (0.2)

4. Conclusion

The determination of TKN in water and waste water with sulfuric acid and the Kjeldahl Tablet Titanium using the KjelDigester K-449 and KjelMaster system K-375/K-376 provides reliable and reproducible results.

5. References

ISO 5663
DIN EN 25 663
40 CFR part 136.3

Operation Manual of KjelDigester K-446/K-449
Operation Manual of KjelMaster system K-375/K376

For more detailed information and safety considerations please refer to the Application Note no. 118/2013.