

TKN determination in water and wastewater

SpeedDigester K-439, Kjel Line and MultiDist:

Steam distillation and Potentiometric determination of Total Kjeldahl Nitrogen (TKN) according to the Kjeldahl method in water and wastewater along with determination of Limit of Detection (LOD) and Limit of Quantification (LOQ)

1. Introduction

An easy and reliable method for the determination of Total Kjeldahl Nitrogen (TKN) in water and wastewater according to Kjeldahl is introduced below. The samples are digested using the SpeedDigester K-439 using Kjeldahl Titanium Tablets. A steam distillation protocol followed by a suitable boric acid titration is performed with the MultiKjel together with the Metrohm Eco Titrator. To test the performance, nitrogen recovery in different concentrations of urea solutions was tested.

2. Experiment

Sample:

Urea stock solution 1: ~ 0.5 mg N / mL simulating TKN concentrations in surface water

Urea stock solution 2: ~ 1.5 mg N / mL simulating TKN concentrations in wastewater

Equipment:

MultiKjel coupled with Metrohm EcoTitrator (11K36531210), K-439 SpeedDigester (1154392000)

Procedure:

Pour measured Water-Sample into a Sample Tube. Add 15 ml Conc. H₂SO₄ and 2 Titanium Kjeldahl tablets (Digestion in Table 1). Distillation parameters are as presented in Table 2.

(For detailed procedure please refer to AN755/2021)

Table 1. Digestion parameters on SpeedDigester K-439

Step	Standard Kjeldahl digestion	
	Temperature [°C]	Time [min]
Preheating	250	0
1	350	15
2	490	125
Cooling	-	35
Total Time	-	175

Table 2. Distillation parameters on Kjel Line

Parameter	Distillation and titration parameters
H ₂ O Volume	50 mL
NaOH Volume	40 mL
Distillation Time	180 s
Titration Type	Boric Acid Titration
H ₃ BO ₃ Volume	60 mL (2 %)
Sensor type	Potentiometric (pH)
Endpoint pH	4.65
Titrant	H ₂ SO ₄ 0.01mol/L

3. Results

The results of TKN determination and recovery for the urea solutions are presented in Table 3.

Table 3: Determined Nitrogen contents (rsd in brackets, n=6).

Product	Recovery (RSD)	Digestion method	Kjel line instrument
TKN (0.5 ppm) in urea solution (200 mL)	93.002% (1.502%)	Standard Kjeldahl	MultiKjel
TKN (1 ppm) in urea solution (200 mL)	101.380% (1.103%)	Standard Kjeldahl	MultiKjel
TKN (60 ppm) in urea solution (25 mL)	100.395% (0.278%)	Standard Kjeldahl	MultiKjel

4. Conclusion

The determination of TKN (Total Kjeldahl Nitrogen) in water using the SpeedDigester K-439 and MultiKjel system provides reliable and reproducible results. The recovery of the urea stock solution was excellent with low standard deviations. According to DIN 32 645, the LOD is 0.036 mg nitrogen/L and the LOQ is 0.108 mg nitrogen/L for 200 mL sample volume.

5. References

Kjeldahl Optimizer App, AN755/2021, Operation Manual Kjel Line, Operation Manual Dist Line