C10 - Total Kjeldahl Nitrogen (TKN) determination

1. Principles

TKN or KN is the sum of the concentration (mg / L) of ammonia nitrogen and organic nitrogen in water.
First stage : the sample is mineralised with sulphuric acid and selenium as catalyst : organic nitrogen (urea, proteins…) is transformed in ammonium salt NH_4^+ .
Second stage : the whole quantity of ammonium salt (initial and from organic nitrogen mineralization) is transformed in ammonia NH_3 by addition of soda lye NaOH and is distillated in a boric acid solution.
Ammonia is dosed by titrimetry with acid.

2. Procedure :

2.1. Sample volume

When the approximate nitrogen concentration is known, sample volume can be chosen thanks to this board :

KN concentration (mg/L)	Sample volume * (mL) **V_0 mL**
until 10	250
10 to 20	100
20 to 50	50
50 to 100	25

* when hydrochloric solution HCl 0.02 mol / L is used for the final titration.

2.2. Dosage

Mineralization must be carried out under a fume hood.

Introduce the sample in the adapted flask
Add 10 mL of sulphuric acid (1.84 g/mL) and 5 +/- 0.5g catalyst
Add some glass beads
Boil the sample until white fume appears
Carry on heating during 2 hours at 440°C.

Cool the sample gently

Introduce 50 mL of indicator (grey – violet coloured boric acid and Tashiro indicator) in an Erlenmeyer flask or in the receiving flask of the distillation apparatus. The end of the cooler must be immersed in the indicator solution.

Set the distillation apparatus : introduction of 100 to 200 mL of deionized water and 50 mL of soda lye in the ammonium solution.

Heat the ammonium solution and distillate NH_3 : Tashiro indicator turns green (5 to 10 min).

Titrate the distillate with HCl 0.02 mol/L until solution turns grey-violet : **V_1 mL**.

Blank : carry on this experiment with deionized water : **V_2 mL**.

$$KN\ (mg / L) = (V_1 - V_2 / V_0) * C * 14.01 * 1000$$

C is the molar concentration (mol / L) of the hydrochloric acid

3. Practical work

Carry out the determination of KN with the two samples : “raw water NH4” and “raw water Norg”)

4. Report

For each sample, note C, V_0, V_1 and V_2 .
Determinate the KN concentration (mg / L).