APPLICATION NOTE

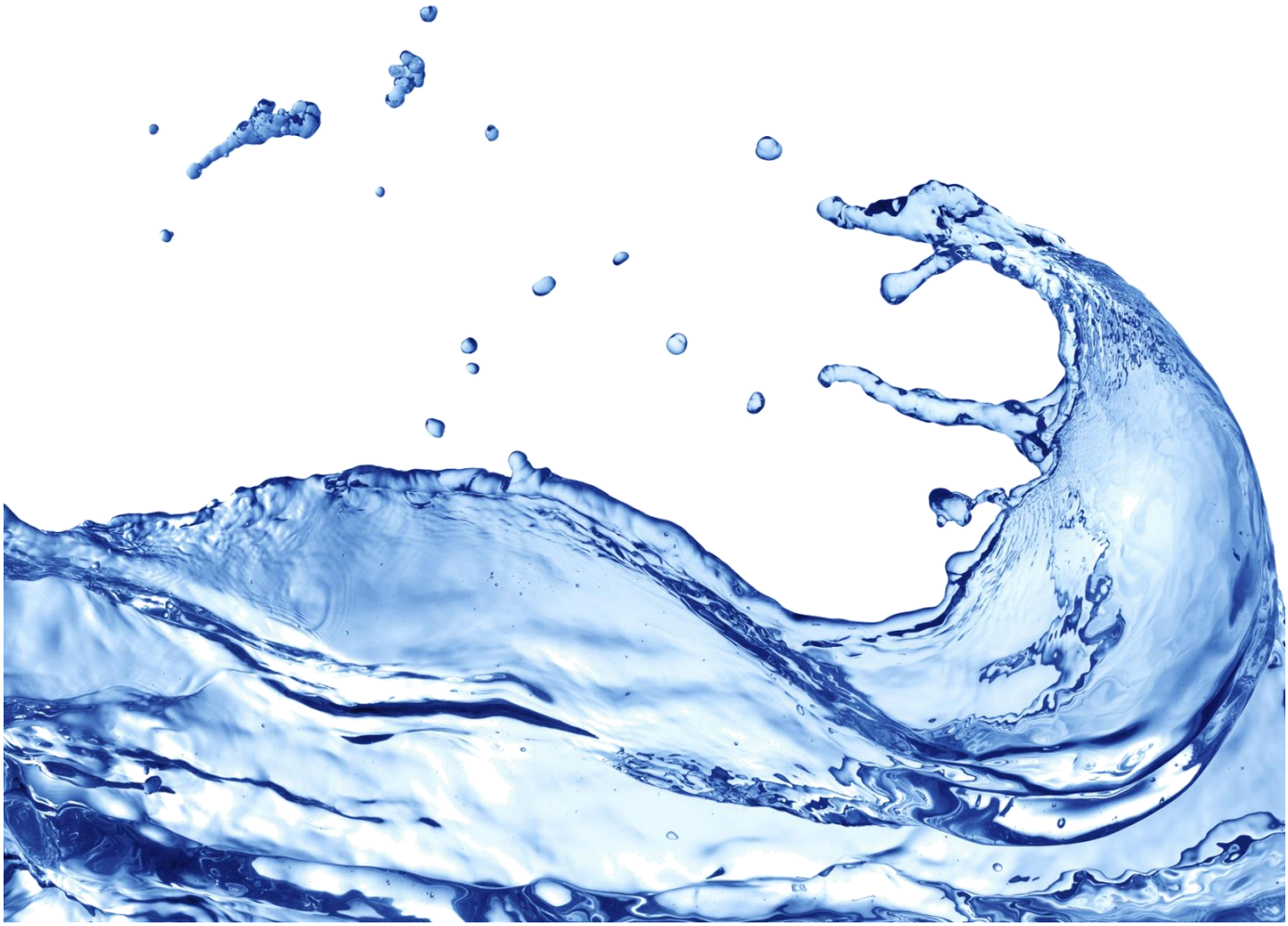
F&F-K-001-2018/A1

N/Ammonia Determination in water

according to the Kjeldahl method

Reference: **ISO 5664; EPA method n° 1690; APAT IRSA 4030 C; Standard Method 4500 NH_3**

Tested with **VELP Scientifica UDK 129 Kjeldahl Distillation unit** (Code F30200120)



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N/AMMONIA DETERMINATION IN WATER
UDK DISTILLATION UNIT

Introduction

Water is the most important resource on Earth, for humans and other living organisms alike. Despite this, nearly half of the world's population does not have access to drinking water of acceptable standard. Groundwater is usually a good source of drinking water since water is naturally purified when it is slowly percolating through soil. The use of groundwater as a source for drinking water has expanded much in modern times and today makes up 25 to 30% of the total water extraction of the world.
Between the 1960s and the 1990s groundwater extraction increased and concerns were raised about possible degradation of the quality of the groundwater (Trafford et al., 1996).

Determination of ammonia in water sample

This method describes the procedure for the determination of ammonia-N in drinking, ground, and surface water; domestic and industrial waste; and biosolids (municipal sewage sludge). Distillation of ammonia from the sample is followed by spectrophotometric analysis.
Concentration range: 0.4 – 4 mg/l. If the concentration of nitrogen in ammonia is more than 4mg/L, it's necessary to dilute the sample to 200 ml with distilled water to obtain as max concentration of 4 mg/L.

Sample

CRM synthetic water certified value: 15.0000 mg/l

Reagents and equipment

- Volumetric flasks marked at 200 ml
- Volumetric flasks marked at 50 ml
- Spectrophotometer UV-VIS. Absorbance reading at 420 nm
- Cuvettes with optical path 10 mm
- Dechlorinating reagent—Dissolve 0.35 g sodium thiosulfate ($Na_2S_2O_3$*$5H_2O$) in reagent water and dilute to 100 mL.
- Borate buffer: Dissolve 9.5 g sodium tetraborate decahydrate ($Na_2B_4O_7$*10 H_2O) in about 500 ml of distilled water. Add 88 mL of 0.1N NaOH solution and dilute to 1 L with reagent water.
- Boric acid 2 %: dissolve 20 g of boric acid in 1000 ml distilled water
- NaOH 6M: dissolve 24 g of NaOH in water and dilute to 100 ml
- NaOH 1 M: dissolve 40 g of NaOH in water and dilute to 1000 ml
- H_2SO_4 1N: carefully add 28 ml H_2SO_4 concentrated (d=1.84) to 500 ml of water and dilute to 1000 ml
- Ammonia standard solutions: dissolve 3.819 ml of ammonium chloride (NH_4Cl), dried at 110 °C, in 1 liter of distilled water (1 ml = 1 mg N-NH_3). Dilute 10 ml of this solution to 1 l with distilled water (10 mg/l N-NH_3).
- Nessler reagent for photometric reading
- Seignette salt: dissolve 50 g of sodium tartrate and potassium tetrahydrate in 30 ml of distilled water. Boil to remove ammonia residues, and after cooling dilute to 100 ml with distilled water.
- It's possible to perform this test with standard test tubes (300 ml) or with the optional 1 liter test tube (code A00001083)

Sample Preparation

- Clean the distillation unit by using 200 mL of deionized water with 20 mL of borate buffer solution; distill and check if adding to 10 mL of distillate, the Nessler reagent, there is color formation. In case of color formation it's necessary to wash the distillation unit.
Wash the unit 4-5 times before starting the analysis.

- Pour 200 ml of water sample into the test tube. If necessary, remove the chlorine using Dechlorinating reagent. 0.5 ml of this solution will neutralize 1 mg/L of residual chlorine in a 200 mL sample aliquot.
If necessary neutralize the sample using acid (H_2SO_4 1N) or base (NaOH 1M).

- Add 10 ml of borate buffer and adjust pH to 9.5 using NaOH 6 M

Distillation and Photometric reading

Distill the samples with the following parameters (Set a customizable method):

- Distillation time: 10 min
- NaOH: 0 ml

As receiving solution, 50 ml of Boric acid (2 % p/v) has been used in a flask marked at 200 ml.

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Push START to begin the distillation.
Stop the analysis manually when the volume of distillate is a little bit less than 200 mL (it is important not to overcome 200 ml).
Add deionized water to dilute the distillation solution to 200 ml.

For spectrophotometric measurement, take 50 ml of distillate and add 5 drops of stabilizing solution of Seignette salt and mix well. Add 2 ml of Nessler reactive, mix well and wait for 15 min before the absorbance reading in 10 mm cuvette at 420 nm. Compare the results with a calibration curve in the range of 0.4 - 4.0 mg /l $N\text{-}NH_4^+$.

Typical Results on CRM synthetic water

The results are calculated as mg/l ammonia nitrogen:

Sample	Sample quantity (ml)	mg/l $N\text{-}NH_4^+$
CRM synthetic water	200	14.25
	200	14.20
	200	14.15
	200	14.60
	200	14.65
	200	14.25
	200	14.25
	200	14.35
	200	14.35
	200	14.60
	Average ± SD%	14.37 ± 0.18
	RSD% *	1.28
	Recovery %	96

Certified value: 15.0000 mg/l
***** RSD% = (Standard Deviation * 100) / Average

The complete procedure was verified by using 5 ml of glycine standard solution (3%) containing 28 mg of nitrogen, as reference substance.

Acknowledgments

A special thanks goes out to Dr. Maria Teresa Dabraio of the third party laboratory *Studio Analisi Chimiche e Ambientali S.A.S.* for their contribution in the analysis.

Conclusions

The obtained results are reliable and reproducible in accordance with the expected values: all data fulfill the expected range of 80-120 %, adopted by the laboratory, in accordance to the official references.

Benefits of ammonia distillation by using UDK 129 are:

- High level of precision and reproducibility
- High productivity
- Worldwide official method
- Time saving
- Affordable equipment cost
- Moderate running costs

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