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EZ7750 Total Nitrogen, Nitrate & Nitrite Analyser

Method and reagent sheets

10/2021, Edition 1.01

Table of Contents

1. Legal information

Manufacturer: AppliTek NV/SA

Distributor: Hach Lange GmbH

The translation of the manual is approved by the manufacturer.

2. Analytical specifications

Please refer also to the respective technical datasheet at Hach Support Online.

Total Nitrogen, Nitrate & Nitrite - All specifications			
Analysis method	TN: Colorimetric measurement at 546 nm using hydrazine reduction and NEDD colour solution alter persulfate destruction in alkaline medium NO_3-N: Colorimetric measurement using NEDD colour solution after reduction with hydrazine solution. NO_2-N Colorimetric measurement using NEDD colour solution		
Parameter	Total nitrogen, Nitrate and Nitrite		
Cycle time	Standard measurement cycle time: 60 minutes - including digestion time of 10 minutes External dilution: + 5 – 10 min.		
Limit of detection (LOD)	≤ 0.25 mg/L TN, ≤0.20 mg/L NO_3-N, ≤0.10 mg/L NO_2-N		
Precision/Repeatability	Better than 4% full scale range for standard test solutions		
Cleaning	Automatic; frequency freely programmable		
Calibration	Automatic, 2-point; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences	Ions like antimony (III) [$(Sb)^{3+}$)], bismuth (III) [$(Bi)^{3+}$], chloroplatinate [$(PtCl_6)^{2-}$], gold (III) [$(Au)^{3+}$], iron (III) [$(Fe)^{3+}$], lead (II) [$(Pb)^{2+}$], mercury (II) [$(Hg)^{2+}$], metavanadate [$(VO_3)^{-}$] and silver (I) [$(Ag)^{+}$] can precipitate with nitrate. Presence of cupric [$(Cu)^{2+}$] may decompose the diazonium salt which results in a low result. Strong oxidizing agents. NCl_3 results in a false red color. Large amounts of color and turbidity interferes. Fats, oil, proteins, surfactants and tar.		
Measuring range	**Parameter**	**Low range (mg/L)**	**High range (mg/L)**
	TN	0.25	10
	NO_3-N	0.20	10
	NO_2-N	0.10	5.0

3. Analysis method

Summary

The determination of the total nitrogen, nitrate and nitrite concentration is based on three methods, combined in one analyzer.

The total nitrogen concentration is determined in the ‘TN’ method. The nitrate concentration is determined in the ‘NO3-N’ method. The nitrite concentration is determined in the ‘NO2’ method. The concentration of all parameters is determined alternately in the ‘Main’-method.

The calibration for total nitrogen is determined in the ‘TN’ method. The calibration for nitrate is determined in the ‘NO3-N’ method. The calibration for nitrite is determined in the ‘NO2-N’ method.

Remark

The methods cannot be started at the same time.

3.1 Total Nitrogen

Summary

The determination of the total nitrogen concentration in water is based on the reaction of nitrate with colour reagent in an acidic medium to form an intense blue coloured complex. The absorption is measured at 546 nm.

Analysis steps

The sample is mixed with the oxidation reagents Superoxi A and Superoxi B and heated to 120 °C (or up to 150 °C – programmable) in an oven during several minutes (standard 10 minutes; programmable up to 60 minutes). During this digestion process the organic and the inorganic nitrogen compounds are oxidized and converted to nitrate (NO_3-). After digestion, the nitrate is reduced to nitrite by adding a reducing reagent. The nitrite reacts in an acidic medium with the colour reagent to produce a violet azo-complex. The absorption at 546 nm is measured with a photometer and is proportional to the concentration of nitrogen in the sample. With the obtained absorbance values, the total nitrogen concentration is calculated according to Beer’s Law.

Calibration

The calibration procedure measures a REF1 TN solution (channel 9, REF1 valve) and a REF2 TN solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two point calibration.

3.2 Nitrate

Summary

Nitrate is converted to nitrite by adding reducing reagent. Nitrite reacts with the colour reagent in an acidic medium to form a violet coloured complex. The absorption is measured at a wavelength of 546 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling, the sulfamic acid is added. Next, the buffer and reducing reagent are added and the initial absorbance value is measured at 546 nm. Next, the colour solution is added and after respecting a stirring period – performed to obtain complete colour development – the final absorbance value is determined. With the obtained absorbance values, the nitrate concentration can be calculated according to Beer’s law.

Calibration

The calibration procedure measures a REF1 NO_3-N solution (channel 9, REF1 valve) and a REF2 NO_3-N solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two-point calibration.

3.3 Nitrite

Summary

Nitrite reacts with the colour reagent in an acidic medium to form a violet coloured complex. The absorption is measured at a wavelength of 546 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling the initial absorbance value is measured at 546 nm. Next, the colour solution is added and after respecting a stirring period – performed to obtain complete colour development – the final absorbance value is determined. With the obtained absorbance values, the nitrite concentration can be calculated according to Beer’s law.

Calibration

The calibration procedure measures a REF1 NO_2-N solution (channel 9, REF1 valve) and a REF2 NO_2-N solution (channel 10, REF2 valve) to adapt the slope and offset factors by means of a two-point calibration.

4. Reagents

⚠ CAUTION

⚠ Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Read the safety data sheet from the supplier before bottles are filled or reagents are prepared. For laboratory use only. Make the hazard information known in accordance with the local regulations of the user.

⚠ CAUTION

⚠ Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

4.1 Reagent overview and consumption

In the tables below, the products that are needed to prepare the reagents are listed. The product name, the formula, the molecular weight, the CAS No. and the amount needed to prepare 1 liter of the reagents is given. Check the consumption of the reagents (28 days) to adapt the volumes needed.

Product	**Consumption**	**Consumption/28 days A rata 1 analysis/60 min**	**Recommended containers**
Superoxi A (TN)	~ 6.0 mL	~ 4.1 L	Plastic – 5 L
Superoxi B (TN)	~ 3.0 mL	~ 2.1 L	Plastic – 2.5 L
Colour solution (NO_2-N)	~ 1.0 mL / analysis	~ 2.1 L	Glass-Amber – 2.5 L
Colour solution (NO_3-N)	~ 1.0 mL / analysis		
Colour solution (TN)	~ 1.0 mL / analysis		
Buffer solution (NO_3-N)	~ 0.5 mL / analysis	~ 1.01 L	Plastic – 2.5 L
Buffer solution (TN)	~ 1.0 mL / analysis		
Reducing reagent (NO_3-N)	~ 4 mL / analysis	~ 5.4 L*	Plastic – 5 L
Reducing reagent (TN)	~ 4 mL / analysis		
Sulfamic acid solution (NO_3-N)	~ 0.25 mL / analysis	~ 0.5 L	Plastic – 2.5 L
REF1 solution (NO_2-N, NO_3-N, TN)	~ 1.5 L / calibration	/	Plastic – 2.5 L
REF2 solution (NO_2-N, NO_3-N, TN)	~ 1.5 L / calibration	/	Plastic – 2.5 L

*This solution is stable for maximum 2 weeks

4.2 DI-water overview and consumption

	Rinse water (mL/analysis) Type I			**Dilution water (mL/analysis) Type I**			**Total (mL/analysis)**	**Consumption/28 days A rata 1 analysis / 60 min**
	TN	**NO_2-N**	**NO_3-N**	**TN**	**NO_2-N**	**NO_3-N**	**TN +NO_2-N+ NO_3-N**	**TN +NO_2-N +NO_3-N**
0	50 mL	50 mL	50 mL	10 mL	10 mL	10 mL	180 mL	121 L

Remark

The indicated volumes are an estimation of the consumption for rinse and dilution water, based on a standard operating procedure, as defined in the specifications of the EZ analyser. Please be aware that, depending on the sample matrix, the rinse water volumes might increase.

4.3 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of Reagent grade, ACS grade or better (*). The use of pro analysis chemicals is recommended. Poor quality of the reagents can affect the analyser performance.

> (*) Analytical Reagent (AR), Guaranteed Reagent (GR), UNIVAR, AnalaR, Premium Reagent (PR), ReagentCertified ACS reagent, ACS Plus reagent, puriss p.a. ACS reagent, ReagentPlus®, TraceCERT®, Suprapur®, Ultrapur®, or better are also possible.

Quality of DI-water

All EZ analysers are tested with standard solutions, reagents and dilution water prepared using type I water or better as defined by ASTM D1193-91.

To achieve the specifications as stated on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used for the preparation of the standard solutions, reagents and dilution water.

Additionally the water used for the preparation of the standard solutions for an EZ analyser must be free of the parameter or any of the interferences for the method of that EZ analyser.

Storage of Reagents

While operating the instrument, keep in mind the reagent requirements as stated in the reagent overview, the chapters below and/or in the data sheet of the instrument.

⚠ CAUTION	
⚠	For longer-term storage: Store the reagents cold; Store the reagents in the dark; If applicable: Store the reagents in a fridge during operation

⚠ CAUTION	
⚠	Refresh the reagents after one month (unless stated differently in the chapters below). Do not mix old reagents with freshly prepared reagents. Remove old reagents from the container before adding freshly prepared reagents.

4.4 Superoxi A solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium peroxodisulfate	$Na_2S_2O_8$	238.11	7775-27-1	15 g

Preparation

Dissolve 15 g of sodium peroxodisulfate ($Na_2S_2O_8$) in 500 ml of de-ionized water using a volumetric flask of 1000 ml. Mix and add de-ionized water up to the grade mark.

We recommend to use of Sodium peroxodisulfate with following specifications:

Product	Brand	Product No.	Specification
Sodium peroxodisulfate	Honeywell	71890	Purum p.a. ≥ 99.0% (RT) – Low Nitrogen concentration

4.5 Superoxi B solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium Hydroxide	NaOH	40.00	1310-73-2	10 g

Preparation

Dissolve 10 g of sodium hydroxide (NaOH) in 500 mL of de-ionized water using a volumetric flask of 1000 mL. Mix and add de-ionized water.

4.6 Reducing reagent

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Copper(II) sulfate pentahydrate	$CuSO_4 * 5H_2O$	249.69	7758-99-8	-
Hydrazine sulfate	$N_2H_4 * H_2SO_4$	130.12	10034-93-2	1 g

Preparation

Dissolve 0.5 g of copper sulfate ($CuSO_4 * 5H_2O$) in 50 mL of de-ionized water using a volumetric flask of 100 mL. Mix and add de-ionized water up to the grade mark.

This solution is stable for > 1 month.

Take 1 mL of the copper sulfate solution and add to 100 mL de-ionized water in a volumetric flask of 1000 mL. Add 1 g of hydrazine sulfate ($N_2H_4 * H_2SO_4$). Dissolve and add de-ionized water up to the grade mark.

This solution is stable for maximum 2 weeks. Store the reagent in a closed (brown) bottle and in a fridge during operation to prolong stability up to one month.

4.7 Buffer solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium Hydroxide	NaOH	40.00	1310-73-2	20 g

Preparation

Dissolve 20 g of sodium hydroxide (NaOH) in 500 mL of de-ionized water using a volumetric flask of 1000 mL. Mix and add de-ionized water up to the grade mark.

4.8 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Phosphoric acid 85%	H_3PO_4	98.00	7664-38-2	100 mL
Sulfanilamide	$H_2NC_6H_4SO_2NH_2$	172.20	63-74-1	10 g
N-(1-Naphthyl) ethylenediamine dihydrochloride	$C_{12}H_{16}Cl_2N_2$	259.17	1465-25-4	0.5 g

Preparation

Dilute 100 mL of phosphoric acid (H_3PO_4, 85%) in 400 mL de-ionized water using a volumetric flask of 1000 mL. Add 10 g of sulfanilamide ($H_2NC_6H_4SO_2NH_2$) and dissolve completely. Add 0.5 g N-(1-naphthyl) ethylenediamine dihydrochloride ($C_{12}H_{16}Cl_2N_2$) and dilute with de-ionized water to the grade mark.

This solution is stable for maximum 2 weeks. Store the reagent in a closed (brown) bottle and in a fridge during operation to prolong stability up to one month.

4.9 Sulfamic acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sulfamic acid	NH_2SO_3H	97.09	5329-14-6	60 g

Preparation

Dissolve 60 g sulfamic acid (NH_2SO_3H) in 400 mL de-ionized water using a volumetric flask of 1000 mL. Mix and fill up with de-ionized water to the grade mark.

4.10 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium nitrate	$NaNO_3$	84.99	7631-99-4	3.035 g

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium nitrite	$NaNO_2$	69.00	7632-00-0	2.464 g

Preparation

500 mg/L NO_3-N stock solution

Prepare a stock solution of 500 mg/L NO_3-N/TN: Dissolve accurately 3.035 g sodium nitrate ($NaNO_3$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

500 mg/L NO_2-N stock solution

Prepare a stock solution of 500 mg/L NO_2-N: Dissolve accurately 2.464 g sodium nitrite ($NaNO_2$) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

15 mg/L TN (10 mg/L NO_3-N & 5 mg/L NO_2-N) standard solution – REF2

Prepare a standard solution of 15 mg/L TN. Take accurately 20 mL of the 500 mg/L NO_3-N stock solution and 10 mL of the 500 mg/L NO_2-N stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

0 mg/L TN, NO_3-N & NO_2-N standard solution – REF1

Prepare a standard solution of 0 mg/L NO_3-N. Use de-ionized water.

4.11 Cleaning solution (facultative)

The cleaning procedure should prevent any build-up of chemicals in the analyser. To obtain an effective cleaning procedure one has to test the cleaning solution and the cleaning interval for each application. Perform the selected cleaning solution and interval for a trial period, check then the effectiveness of the procedure and change if necessary.

Change Information	
Date: 22/10/2021	Previous version: Edition 5 to Edition 1.01
Reason for Change	
- Addition of water consumption - Addition of information reagents - Change in reagent recipe	
Description of Change	
- Addition of estimated consumption of water for rinse and dilution (chapter 4.2) - Addition of extra information regarding storage and quality of reagents (chapter 4.3) - Change in recipe for sulfamic acid solution (chapter 4.10)	