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EZ7705 Total Nitrogen analyser

Method and reagent sheets

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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 - All specifications			
Analysis method	Colorimetric measurement at 546 nm using hydrazine reduction and NEDD colour solution alter persulphate destruction in alkaline medium		
Parameter	Total nitrogen		
Cycle time	60 minutes incl. digestion of 10 minutes (standard)		
Limit of detection (LOD)	≤ 4 mg/L		
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 TN	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	4	100

3. Analysis method

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 diluted and 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.

The calibration is performed in the MAIN method.

Remark

The methods cannot be started at the same time.

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.

Total Nitrogen

Product	Consumption	Consumption/28 days A rata 1 analysis/60 min	Recommended containers
Superoxi A	~ 6.0 mL	< 5 L	Plastic – 10 L
Superoxi B	~ 3.0 mL	< 3 L	Plastic – 5 L
Buffer	~ 1.0 mL	< 1 L	Plastic – 2.5 L
Reducing reagent	~ 4.0 mL	< 3 L*	Plastic – 5 L
Colour	~ 1.0 mL	< 1 L	Glass – 2.5 L
REF1 solution	~ 0.5 L / calibration	/	Plastic – 1 L
REF2 solution	~ 0.5 L / calibration	/	Plastic – 1 L

*This solution is stable for maximum 2 weeks

4.2 Storage and quality of chemicals

Quality of chemicals

All chemicals should be of ACS grade or better. We recommend the use of pro analysis chemicals.

Quality of water

Reagent grade, nitrogen-free de-ionized water must be used to prepare the chemical solutions and for rinse purposes.

Storage of Reagents

While operating the instrument, keep in mind the ambient temperature conditions as stated in the data sheet of the instrument.

Store the reagents cold; Store the reagents in the dark; Refresh the reagents after one month (unless stated differently in the chapters below).

4.3 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

4.4 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.

4.5 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. 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

4.6 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.

4.7 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.

4.8 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium Nitrate	$NaNO_3$	84.99	7631-99-4	6.071 g

Preparation

1000 mg/L TN stock solution

Prepare a stock solution of 1000 mg/L TN: Dissolve accurately 6.071 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.

100 mg/L TN standard solution – REF2

Prepare a standard solution of 100 mg/L TN. Take accurately 100 mL of the 1000 mg/L TN stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

0 mg/L TN standard solution – REF1

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

4.9 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.