DOC312.53.94498

EZ7652 Total Nitrogen and Total Phosphorus analyser

Method and reagent sheets

12/2020, Edition 1.0

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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.

TN/TP - All specifications			
Analysis method	**TN:** UV photometric measurement at 220 nm after persulfate destruction in alkaline medium, based on APHA 4500-NO3 (B) & China GB 11894-89 **TP:** Colorimetric measurement at 700 nm using ascorbic acid reduction and molybdate color solution after persulfate destruction in acidic medium, based on with APHA 4500-P & China GB 11893-89		
Parameter	Total nitrogen (TN), Total phosphorus (TP)		
Cycle time	Standard measurement cycle time for both TN & TP: 60 minutes		
Limit of detection (LOD)	≤ 0.5mg/L (TN); ≤ 0.01 mg/L (TP)		
Precision/Repeatability	Better than 3% full scale range for standard test solutions		
Cleaning	Automatic (self-cleaning)		
Calibration	Automatic, 2-point; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences TN	The main interferences are Br^- and I^-. When the amount of I^- is 2.2 fold of the amount of TN, or the amount of Br^- is 3.4 fold of the amount of TN, this will interfere on the test results. Dissolved organic matter, surfactants, nitrite [NO_2^-] and chromium (VI) [$(Cr)^{6+}$] interfere. Various inorganic substances not normally found in natural water, such as chlorite [ClO_2^-] and chlorate [ClO_3^-], may interfere. Turbidity can be compensated by 0.45 µm filtration.		
Interferences TP	Arsenic (V) [$(As)^{5+}$], chromium (VI) [$(Cr)^{6+}$], nitrite [NO_2^-] , copper (II) [$(Cu)^{2+}$] > 10 mg/L, iron (III) [$(Fe)^{3+}$] > 10 mg/L, sulfide [$(S)^{2-}$] > 2 mg/L and vanadium [$(V)^{5+}$], silica [$(Si)^{4+}$] > 60 mg/L. 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	0.5	20
	TP	0.01	2

3. Analysis method

Summary

The determination of total nitrogen and total phosphorus is based on two methods, combined in one analyser.

The total nitrogen concentration is determined in the ‘TN’ method. The total phosphorus concentration is determined in the ‘TP 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 total phosphorus is determined in the ‘TP’ method.

Remark

The methods cannot be started at the same time.

3.1 Total Nitrogen

Analysis steps

The sample is mixed with persulfate solution, sodium hydroxide solution and digested. During this digestion process the organic and the inorganic nitrogen compounds are converted to nitrate (NO_3^-). During digestion, the initial absorbance value is measured on water. The sample is then transferred into the analysis vessel and a HCl solution is added. The absorption at 220 nm is measured. 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 Total Phosphorus

Analysis steps

The sample is mixed with persulfate solution and digested. During the digestion process, the organic and the inorganic phosphorus compounds are converted to orthophosphate (PO_4^{3-}). After digestion, the sample is transferred into the analysis vessel and a reducing reagent is added. The initial absorbance value is measured. The orthophosphate reacts with the colour reagent forming phosphomolybdic acid. This acid is reduced to an intensely coloured molybdenum blue complex by means of the reducing reagent (ascorbic acid). The absorption at 700 nm is measured. With the obtained absorbance values, the total phosphorus concentration is calculated according to Beer’s Law.

Digestion:

Phosphorus + high temperature + reagents $\rightarrow PO_4^{3-}$ (orthophosphate)

Analysis:

PO_4^{3-} + color reagent → phosphomolybdic acid

Phosphomolybdic acid + reducing reagent → molybdenum blue

Calibration

The calibration procedure measures a REF1 TP solution (channel 9, REF1 valve) and a REF2 TP 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
Persulfate (TN)	~ 2.5 mL	~ 3.4 L	Plastic – 5 L
Persulfate (TP)	~ 2.5 mL		
NaOH (TN)	~ 1.0 mL	~ 1 L	Plastic – 2.5 L
NaOH (TP)	~ 0.5 mL		
HCl (TN)	~ 1 mL	~ 1 L	Plastic – 2.5 L
Reducing reagent (TP)	~ 1.5 mL	~ 1 L * (~ 0.5 L / 14 days)	Glass, Amber – 1 L
Colour (TP)	~ 1.5 mL	~ 1 L	Glass, Amber – 1 L
REF1 solution (TN & TP)	~ 1.0 L / calibration	/	Plastic – 1 L
REF2 solution (TN & TP)	~ 1.0 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 and phosphate-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 Persulfate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium peroxodisulfate	$Na_2S_2O_8$	238.11	7775-27-1	40 g

Preparation

Dissolve 40 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 Sodium hydroxide solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium hydroxide	NaOH	40.00	1310-73-2	4 g

Preparation

Dissolve 4 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.

4.5 Hydrochloric acid solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Hydrochloric acid 37%	HCl	36.46	7647-01-0	83 mL

Preparation

Dilute 83 mL of hydrochloric acid (HCl, 37%) 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.

4.6 Reducing reagent

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ascorbic acid	$C_6H_8O_6$	176.12	50-81-7	20 g

Preparation

Dissolve 20 g of ascorbic acid ($C_6H_8O_6$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. dissolve completely and dilute to 1litre with de-ionized water. mineralized water.

This solution is stable for maximum 2 weeks.

4.7 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium-hepta-molybdate-tetrahydrate	$(NH_4)_6Mo_7O_{24} * H_2O$	1235.86	12054-85-2	10 g
Potassium antimony tartrate trihydrate	$C_8H_4K_2O_{12}Sb_2 * 3\ H_2O$	667.87	28300-74-5	0.5 g
Sulfuric acid 96%	H_2SO_4	98.08	7664-93-9	75 mL

Preparation

Dissolve 10 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24}.H_2O$) in 400 mL of de-ionized water and dissolve completely. Add 0.5 g potassium antimony tartrate trihydrate ($C_8H_4K_2O_{12}Sb_2$. 3 H_2O) and dissolve completely. Add 75 mL sulfuric acid (H_2SO_4 95-97 %) and dilute the solution to 1 litre with de-ionized water. Cool down before use.

The colour solution should be colourless. If the colour of the solution is green or yellow, it can’t be used.

4.8 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium Nitrate	$NaNO_3$	84.99	7631-99-4	60.71 g
Potassium dihydrogen phosphate	H_2KO_4P	136.09	7778-77-0	4.394 g

Preparation

10000 mg/L TN stock solution

Prepare a stock solution of 10000 mg/L TN: Dissolve accurately 60.71 g sodium nitrate ($NaNO_3$) in 600 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

1000 mg/L TP stock solution

Prepare a stock solution of 1000 mg/L TP: Dissolve accurately 4.394 g Potassium dihydrogen phosphate (H_2KO_4P) in 300 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

TN & TP standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 10000 mg/L TN stock solution and y mg/L of the 1000 mg/L TP stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade. Store this solution in a plastic container.

Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
20 mg/L TN	20 mg/L TN	2 mL
2 mg/L TP	2 mg/L TP	2 mL

TN & TP standard solution – REF1

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

Change Information	
Date:	
Reason for Change	
Description of Change	