



DOC312.53. 94523

EZ7864 Total Phosphorus and Phosphate analyser

Method and reagent sheets

02/2022, Edition 1.01

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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.

TP/PO ₄ -P - All specifications			
Analysis method	TP: Colorimetric measurement at 700 nm using ascorbic acid reduction and molybdate color solution after persulfate destruction in acidic medium, based on APHA 4500-P & China GB 11893-89 PO ₄ -P: Colorimetric measurement using molybdate blue method (700 nm) based on APHA 4500-P		
Parameter	Total phosphorus (TP), Phosphate (PO ₄ -P)		
Cycle time	Standard measurement cycle time: 30 minutes; One measurement cycle for both TP & PO ₄ -P External dilution: + 5 – 10 min.		
Limit of detection (LOD)	≤ 0.005 mg/L		
Precision/Repeatability	Better than 2% full scale range for standard test solutions		
Cleaning	Automatic (self-cleaning)		
Calibration	Automatic, 2-point; frequency freely programmable		
Validation	Automatic; frequency freely programmable		
Interferences	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)
	TP	0.05	10
	PO ₄ -P	0.05	10

3. Analysis method

Summary

The determination of both total phosphorus and phosphate is combined in two methods. The concentration of all parameters is determined alternately and always started in the 'Main'-method. The total phosphorus and phosphate concentration cannot be determined separately.

Analysis steps


- 1) Start of the TP measurement cycle:
The analysis vessel is cleaned and filled with fresh sample. Next, the sample is mixed with a persulfate solution and digested. During the digestion process, the inorganic and the organic phosphorus compounds are converted to orthophosphate (PO_4^{3-}).
- 2) Start of the $\text{PO}_4\text{-P}$ measurement cycle:
During the digestion process for TP, the analysis vessel is cleaned again and filled with fresh sample. After sampling, the reducing reagent is added and the initial absorbance value is measured at 700 nm. Next, the colour solution is added and after respecting a stirring period, the final absorbance value is measured. With the obtained absorbance values, the Phosphate concentration is calculated according to Beer's Law.
- 3) Continuation of the TP measurement cycle:
After digestion, the sample is transferred into the analysis vessel and a reducing reagent is added. The initial absorbance value is measured at 700 nm. Next, the colour solution is added and after respecting a stirring period, the final absorbance value is measured. With the obtained absorbance values, the total phosphorus concentration is calculated according to Beer's Law.


Calibration

The calibration for both parameters runs simultaneously and is started in the 'Main'-method.

The calibration procedure measures a REF1 TP or $\text{PO}_4\text{-P}$ solution (channel 9, REF1 valve) and a REF2 TP or $\text{PO}_4\text{-P}$ 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/30 min	Recommended containers
Persulfate (TP)	~ 2.5 mL / analysis	~ 3.4 L	Plastic – 5 L
NaOH (TP)	~ 0.5 mL / analysis	~ 0.7 L	Plastic – 2.5 L
Reducing reagent* (TP)	~ 1.5 mL / analysis	~ 4.1 L (~ 2.1 L / 14 days)	Glass, Amber – 2.5 L
Reducing reagent* (PO ₄ -P)	~ 1.5 mL / analysis		
Colour (TP)	~ 1.5 mL / analysis	~ 4.1 L	Plastic, Dark – 5 L
Colour (PO ₄ -P)	~ 1.5 mL / analysis		
REF1 solution (TP & PO ₄ -P)	~ 1.0 L / calibration	/	Plastic – 1 L
REF2 solution (TP & PO ₄ -P)	~ 1.0 L / calibration	/	Plastic – 1 L

*This solution is stable for maximum 2 weeks

4.2 DI-water overview and consumption

	Rinse water (mL/analysis)		Dilution water (mL/analysis)		Total (mL/analysis)	Consumption/28 days A rata 1 analysis / 30 min
	TP	PO ₄ -P	TP	PO ₄ -P	TP + PO ₄ -P	TP + PO ₄ -P
0	75	60	5	10	150	201.6 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 to prolong stability.

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 Persulfate solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Sodium peroxodisulfate	Na ₂ S ₂ O ₈	238.11	7775-27-1	40 g

Preparation

Dissolve 40 g of sodium peroxodisulfate (Na₂S₂O₈) 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 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.

4.6 Reducing reagent

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ascorbic acid	C ₆ H ₈ O ₆	176.12	50-81-7	20 g

Preparation

Dissolve 20 g of ascorbic acid (C₆H₈O₆) 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. Store the reagent in a fridge during operation to prolong stability.

4.7 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium-hepta-molybdate-tetrahydrate	$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot \text{H}_2\text{O}$	1235.86	12054-85-2	10 g
Potassium antimony tartrate trihydrate	$\text{C}_8\text{H}_4\text{K}_2\text{O}_{12}\text{Sb}_2 \cdot 3 \text{H}_2\text{O}$	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 ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot \text{H}_2\text{O}$) in 400 mL of de-ionized water and dissolve completely. Add 0.5 g potassium antimony tartrate trihydrate ($\text{C}_8\text{H}_4\text{K}_2\text{O}_{12}\text{Sb}_2 \cdot 3 \text{H}_2\text{O}$) 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 after preparation. 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
Potassium dihydrogen phosphate	$\text{H}_2\text{KO}_4\text{P}$	136.09	7778-77-0	4.394 g

Preparation

1000 mg/L P stock solution

Prepare a stock solution of 1000 mg/L P: Dissolve accurately 4.394 g Potassium dihydrogen phosphate ($\text{H}_2\text{KO}_4\text{P}$) in 500 mL de-ionized water using a volumetric flask of 1000 mL. Fill up to 1 litre with de-ionized water.

TP & $\text{PO}_4\text{-P}$ standard solution – REF2

Prepare a 10 mg/L P standard solution for calibration: Take accurately 10 mL of the 1000 mg/L P 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.

TP & $\text{PO}_4\text{-P}$ standard solution – REF1

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

Change Information	
Date: 03/03/2022	Previous version: Edition 1.00 to Edition 1.01
Reason for Change	
- Correction of DOC ID	
Description of Change	
- Correction of DOC ID from DOC312.53. 94524 to DOC312.53. 94523	