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EZ1031 Phosphate analyzer

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.

Phosphate - All specifications			
Analysis method	Colorimetric measurement using vanadate yellow method (450 nm) conform with APHA 4500-P (C)		
Parameter	PO ₄ -P		
Cycle time	Standard measurement cycle time: 10 minutes Internal dilution: + 5 min. External dilution: + 5 – 10 min.		
Limit of detection (LOD)	≤ 20 µg/L		
Precision/Repeatability	Better than 3% 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	Positive interference is caused by silica [Si] and arsenate [As] if the sample is heated. Negative interferences are caused by arsenate [(AsO ₄) ³⁻], fluoride [(F) ⁻], thorium [Th], bismuth [Bi], sulphide [(S) ²⁻], thiosulphate [(S ₂ O ₄) ²⁻], thiocyanate [(SCN) ⁻] or excess of molybdate. Blue colour is caused by ferrous iron [(Fe) ²⁺] but this does not affect results if ferrous iron concentrations is less than 100 mg/L. Ions that do not interfere in concentrations up to 1,000 mg/L are aluminium [(Al) ³⁺], ferric [(Fe) ³⁺], magnesium [(Mg) ²⁺], calcium [(Ca) ²⁺], barium [(Ba) ²⁺], strontium [(Sr) ²⁺], lithium [(Li) ⁺], sodium [(Na) ⁺], potassium [(K) ⁺], Ammonium [(NH ₄) ⁺], cadmium (II) [(Cd) ²⁺], manganese (II) [(Mn) ²⁺], lead (II) [(Pb) ²⁺], mercury (I) [(Hg) ⁺], mercury (II) [(Hg) ²⁺], tin (II) [(Sn) ²⁺], copper [(Cu) ²⁺], nickel [(Ni) ²⁺], silver (I) [(Ag) ⁺], uranium (IV) [(U) ⁴⁺], zirconium (IV) [(Zr) ⁴⁺], arsenic [(AsO ₃) ⁻], bromine [(Br) ⁻], carbonate [(CO ₃) ²⁻], perchlorate [(ClO ₄) ⁻], cyanide [(CN) ⁻], iodate [(IO ₃) ⁻], silicate [(SiO ₄) ⁴⁻], nitrate [(NO ₃) ⁻], nitrite [(NO ₂) ⁻], sulphate [(SO ₄) ²⁻] and sulphite [(SO ₃) ²⁻], pyrophosphate, molybdate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate and salicylate. If nitric acid (HNO ₃) is used, chloride [(Cl) ⁻] interferes from 75 mg/L. Large amounts of colour and turbidity interferes. Fats, oil, proteins, surfactants and tar.		
Measuring ranges	% of range - Dilution		Low range (mg/L)
	A	10% of standard range	0.020
	B	25% of standard range	0.050
	C	50% of standard range	0.050
	0	standard range	0.100
	1	internal MP dilution (factor 4)	0.800
	2	Internal MP dilution (factor 8)	1.600
	W	internal dispenser dilution (factor 10)	1
	X	internal dispenser dilution (factor 25)	2,5
	Y	internal dispenser dilution (factor 50)	5
	Z	internal dispenser dilution (factor 75)	7,5
	5	internal dispenser dilution (factor 100)	10
		High range (mg/L)	

3. Analysis method

Summary

The determination of the Ortho-Phosphate (PO_4^{3-}) concentration in water is based on vanadate/molybdate method or yellow method. In an acidic medium, the ortho-phosphate ions react with ammonium molybdate and ammonium vanadate to form yellow ammonium phosphoric vanamolybdate which is measured at 450 nm.

Analysis steps

The analysis vessel is cleaned and filled with fresh sample. After sampling the initial absorbance value is measured at 450 nm. This measurement is performed to correct for any colour contribution of the sample itself. Next, the colour solution is added to the sample solution and after respecting a stirring period, the final absorbance value is measured. With the obtained absorbance values, the Ortho-Phosphate (PO_4^{3-}) concentration is calculated according to Beer's Law.

Calibration

The calibration procedure measures a REF1 $\text{PO}_4\text{-P}$ solution (channel 9, REF1 valve) and a REF2 $\text{PO}_4\text{-P}$ 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.

Product	Consumption	Consumption/28 days A rata 1 analysis/10 min	Recommended containers
Colour solution	~ 0.5 mL / analysis	~ 1.5 L	Plastic – 2.5 L
REF1 solution	~ 0.5 L / calibration	/	Plastic – 1 L
REF2 solution	~ 0.5 L / calibration	/	Plastic – 1 L

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 / 10 min
A	N.A.	N.A.	N.A.	N.A.
B	N.A.	N.A.	N.A.	N.A.
C	N.A.	N.A.	N.A.	N.A.
0	N.A.	N.A.	N.A.	N.A.
1	60 mL	15 mL	75 mL	302 L
2	60 mL	15 mL	75 mL	302 L
W	60 mL	15 mL	75 mL	302 L
X	60 mL	15 mL	75 mL	302 L
Y	60 mL	15 mL	75 mL	302 L
Z	60 mL	15 mL	75 mL	302 L
5	60 mL	15 mL	75 mL	302 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 Colour solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Ammonium-hepta-molybdate-tetrahydrate	$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	1235.86	12054-85-2	25 g
Ammonium (mono) metavanadate	NH_4VO_3	116.98	7803-55-6	1.25 g
Nitric acid 65%	HNO_3	63.01	7697-37-2	330 mL

Preparation

Solution A:

Dissolve 25 g of ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ p.a., > 99%) in 300 mL of warm de-ionized water and dissolve completely.

Solution B:

Dissolve 1.25 g ammonium metavanadate (NH_4VO_3) by heating to boiling in 300 mL de-ionized water. Cool down and add 330 mL nitric acid (HNO_3 65%). Cool solution B to room temperature.

Pour Solution A into Solution B, mix and dilute to 1000 mL with de-ionized water.

4.5 Calibration solution

Products	Formula	MW (g/mol)	CAS No.	1 litre solution
Potassium dihydrogen phosphate	H ₂ KO ₄ P	136.09	7778-77-0	43.94 g

Preparation

10000 mg/L PO₄-P stock solution

Prepare a stock solution of 10000 mg/L PO₄-P. Dissolve 43.94 g of H₂KO₄P in 500 mL water and dilute to 1 litre with de-ionized water

PO₄-P standard solution – REF2

Prepare a standard solution for calibration according to the following table: take accurately x mL of the 10000 mg/L PO₄-P stock solution and transfer into a volumetric flask of 1000 mL. Add de-ionized water up to the mark grade.

	Measuring range	Concentration REF2	Amount of stock solution to add to 1 litre
A	1.0 mg/L PO ₄ -P	1.0 mg/L PO ₄ -P	0.10 mL
B	2.5 mg/L PO ₄ -P	2.5 mg/L PO ₄ -P	0.25 mL
C	5.0 mg/L PO ₄ -P	5.0 mg/L PO ₄ -P	0.50 mL
0	10 mg/L PO ₄ -P	10 mg/L PO ₄ -P	1.0 mL
1	40 mg/L PO ₄ -P	40 mg/L PO ₄ -P	4.0 mL
2	80 mg/L PO ₄ -P	80 mg/L PO ₄ -P	8.0 mL
W	100 mg/L PO ₄ -P	100 mg/L PO ₄ -P	10 mL
X	250 mg/L PO ₄ -P	250 mg/L PO ₄ -P	25 mL
Y	500 mg/L PO ₄ -P	500 mg/L PO ₄ -P	50 mL
Z	750 mg/L PO ₄ -P	750 mg/L PO ₄ -P	75 mL
5	1000 mg/L PO ₄ -P	1000 mg/L PO ₄ -P	100 mL

PO₄-P standard solution – REF1

Prepare a standard solution of 0 mg/L PO₄-P. Use de-ionized water.

4.6 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: 21/09/2021	Previous version: Edition 7 to Edition 1.01
Reason for Change	
- Addition of extra ranges to the portfolio of EZ1031 - Addition of water consumption - Addition of information reagents - Change in description of parameter conform Hach regulations	
Description of Change	
- Addition of extra ranges for internal dispenser dilution: 10x, 25x, 50x, 75x (chapter 4.5) - 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 of description of parameter from P-PO₄ to PO₄-P	