# **EZ1031 Phosphate Analyzer**

Method

Standard range: 0.1 to 10 mg/L PO<sub>4</sub>-P

EZ1031



# **Test preparation**

# **Before starting**

Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

Review the Safety Data Sheets (MSDS/SDS) before the bottles are filled or the reagents are prepared.

All chemicals must be of reagent grade, ACS grade or better<sup>1</sup>. The use of pro-analysis chemicals is recommended. Use of reagents that are not of sufficient quality can have a negative effect on the analyzer performance.

All EZ analyzers are put through long tests with standard solutions, reagents and dilution water prepared with Type I water or better water as specified in ASTM D1193-91.

To get the specifications shown on the data sheet, method and reagents sheet and acceptance test reports, the same water quality (or better) must be used to prepare the standard solutions, reagents and dilution water.

In addition, prepare the standard solutions for an EZ analyzer with water that does not contain the parameter to be measured or interferences for the method.

When operating the device, always make sure to follow the reagent recommendations given in Reagent consumption on page 3.

For longer-term storage, keep the reagents in a cold and dark place. Do not keep reagents longer than recommended. If applicable, keep the reagents in a refrigerator during measurements. Refer to Reagent consumption on page 3 for the reagent temperature.

The manufacturer recommends to replace the reagents, stock and standard solution at 28-day intervals unless specified differently in the sections that follow. Do not mix used reagents with freshly prepared reagents. If reagents, standards or DI water in the containers are replaced, discard all of the container contents in accordance with local, regional and national regulations. Rinse out all of the containers and then fill each container with freshly prepared new reagent.

# **Specifications**

Specifications are subject to change without notice.

Specification	Details
Analysis method	Colorimetric measurement vanadates yellow method compliant with APHA 4500-P (C)
Wavelength accuracy	450 nm
Parameter	Long description: Phosphate
	Short description (default): PO <sub>4</sub> -P
	Options: PO <sub>4</sub>
Unit	mg/L (default); ppm, ppb, μg/L
Precision	The precision value is found on the full-scale range for standard test solutions. Refer to Table 1.
Cleaning	Automatic; frequency is freely programmable

<sup>&</sup>lt;sup>1</sup> Analytical Reagent (AR), Guaranteed Reagent (GR), UNIVAR, AnalaR, Premium Reagent (PR), ReagentCertified ACS reagent, ACS Plus reagent, puriss p.a. ACS reagent, ReagentPlus<sup>®</sup>, TraceCERT<sup>®</sup>, Suprapur<sup>®</sup>, Ultrapur<sup>®</sup>, or better are also possible.

Specification	Details
Calibration	Automatic; 2-point, offset or slope; frequency freely programmable  Note: The manufacturer recommends that a calibration is done when the reagents are replaced.
Validation	Automatic; frequency is freely programmable
Interferences	Positive interferences are caused by silica [Si] and arsenate [As] when the sample is heated.
	Negative interferences are caused by arsenate [(AsO <sub>4</sub> ) <sup>3-</sup> ], fluoride [(F) <sup>-</sup> ], thorium [Th], bismuth [Bi], sulphide [(S) <sup>2-</sup> ], thiosulphate [(S <sub>2</sub> O <sub>4</sub> ) <sup>2-</sup> ], thiocyanate [(SCN) <sup>-</sup> ] or excess of molybdate.
	Blue color (n) is caused by ferrous iron $[(Fe)^{2+}]$ . The blue color has no effect on the results if the ferrous iron concentrations are less than 100 mg/L.
	lons that do not cause interference in concentrations until 1,000 mg/L are aluminium [(Al) <sup>3+</sup> ], ferric [(Fe) <sup>3+</sup> ], magnesium [(Mg) <sup>2+</sup> ], calcium [(Ca) <sup>2+</sup> ], barium [(Ba) <sup>2+</sup> ], strontium [(Sr) <sup>2+</sup> ], lithium [(Li) <sup>+</sup> ], sodium [(Na) <sup>+</sup> ], potassium [(K) <sup>+</sup> ], ammonium [(NH <sub>4</sub> ) <sup>+</sup> ], cadmium (II) [(Cd) <sup>2+</sup> ], manganese (II) [(Mn) <sup>2+</sup> ], lead (II) [(Pb) <sup>2+</sup> ], mercury (I) [(Hg) <sup>+</sup> ], mercury (II) [(Hg) <sup>2+</sup> ], tin (II) [(Sn) <sup>2+</sup> ], copper [(Cu) <sup>2+</sup> ], nickel [(Ni) <sup>2+</sup> ], silver (I) [(Ag) <sup>+</sup> ], uranium (IV) [(U) <sup>4+</sup> ], zirkonium (IV) [(Zr) <sup>4+</sup> ], arsenic [(AsO <sub>3</sub> ) <sup>-</sup> ], bromium [(Br) <sup>-</sup> ], carbonate [(CO <sub>3</sub> ) <sup>2-</sup> ], perchlorate [(ClO <sub>4</sub> ) <sup>-</sup> ], cyanide [(CN) <sup>-</sup> ], iodate [(IO <sub>3</sub> ) <sup>-</sup> ], silicate [(SiO <sub>4</sub> ) <sup>4+</sup> ], nitrate [(NO <sub>3</sub> ) <sup>-</sup> ], nitrite [(NO <sub>2</sub> ) <sup>-</sup> ], sulphate [(SO <sub>4</sub> ) <sup>2-</sup> ] and sulphite [(SO <sub>3</sub> ) <sup>2-</sup> ], pyrophosphate, molybdate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate and salicylate.
	If nitric acid (HNO <sub>3</sub> ) is used, chloride (CI) <sup>-</sup> causes interferences in concentrations of 75 mg/L. Large quantities of color and turbidity cause interferences in fats, oil, proteins, surfactants and tar.

Table 1 Measuring ranges

Range code	Description	LOD (mg/L)	Range (mg/L)	Precision (%)	Cycle time (minutes)
A	10% of standard range	0.02	1	2	10
В	25% of standard range	0.05	2.5	2	10
С	50% of standard range	0.05	5	2	10
0	Standard range	0.1	10	2	10
V	Internal dispenser dilution (factor 5)	0.5	50	2	15
W	Internal dispenser dilution (factor 10)	1	100	2	15
Х	Internal dispenser dilution (factor 25)	2.5	250	2	15
Υ	Internal dispenser dilution (factor 50)	5	500	2	15
Z	Internal dispenser dilution (factor 75)	7.5	750	2	15
5	Internal dispenser dilution (factor 100)	10	1000	2	15

## **Summary of method**

## **Summary**

The determination of the phosphate 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. The yellow ammonium phosphorus vanamolybdate is measured at a wavelength of 450 nm.

## **Analysis steps**

The analysis vessel is cleaned and filled with clean sample. After the sample is collected, the initial absorbance value is measured. This measurement is done to correct a possible color contribution of the sample. The color solution is then added to the sample solution and the last absorbance value is found after a stirring time, which is done for complete color development. The absorbance values are used to find the phosphate concentration on the basis of Beer's law.

#### Calibration

The calibration procedure measures a REF1 PO<sub>4</sub>-P solution (Channel 9, REF1 valve) and a REF2 PO<sub>4</sub>-P solution (channel 10, REF2 valve).

#### **Validation**

The validation procedure measures a REF2 PO<sub>4</sub>-P solution (Channel 8, REF2 valve).

# Reagent consumption

Refer to Necessary reagents for EZ1031 on page 4 to collect the necessary items to prepare the reagents. The product name, formula, molecular weight, CAS No. and the necessary quantity to prepare 1 L of the reagents are given. Examine the consumption of the reagents (28 days) to adjust the necessary quantities.

## **Table 2 Reagent consumption**

Product information		Consumption		Recommendation			
Label	Code	Product	Each analysis	Per 28 days, a rata 1 analysis/10 minutes.	Use life	Containers	Store during operation
Reagent 1	Red	Reagent 1: Color	~ 0.5 mL	~ 2.1 L	28 days	Plastic; 2.5 L	Ambient temperature 10 to 30 °C (50 to 86 °F)

## **Table 3 Calibration standards**

Product information		oduct information Consumption Recommendation		
Label	Product	Per calibration	Use life	Containers
REF1	REF1 standard	~ 0.2 L	28 days	Plastic, 1 L (align with recommendation)
REF2	REF2 standard	~ 0.2 L	28 days	Plastic, 1 L (align with recommendation)

## **Table 4 Calibration recommendations**

Calibration	Time (minutes) ABC0 / VWXYZ5	Recommended frequency	Solutions
Offset	30/45	N.A.	REF1
2-point	60/90	Change of reagents (28 days)	REF1 and REF2

#### Table 5 Hach reagent kit

Label	Code	Product	Volume	Item no.
Reagent 1	Red	Reagent 1: Color	2 L (1x)	APPC1031-01-EU

# DI water consumption

The volumes shown in Table 6 are an estimation of the consumption for rinse and dilution water based on a standard operating procedure as given in the specifications of the EZ analyzer.

Note: Rinse water volumes can increase because of the sample matrix.

**Note:** The range codes A,B,C,0 are configured as default without the use of rinse and dilution water.

# Table 6 DI water consumption

Range code	Rinse water (mL/analysis) Type I	Dilution water (mL/analysis) Type I	Total (mL/analysis)	Per 28 days, a rata 1 analysis/15 minutes
A - B - C - 0	_	_	_	_
V - W - X - Y - Z - 5	51 mL	16 mL	67 mL	180 L

#### Rinse water

If the analyzer does a dilution, a DI water rinse must be used. If no dilution is done, use the sample to rinse. If there is a filter panel in front of the analyzer, make sure that the rinse water also flows through the filter.

# **Necessary reagents for EZ1031**

Solutions	Products	Formula	MW (g/mol)	CAS No.	For each 1 L solution
Reagent 1 Color solution (red)	Ammonium-hepta- molybdate-tetrahydrate	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> * 4 H <sub>2</sub> O	1235.86	12054-85-2	25 g
	Ammonium (mono) metavanadate	NH <sub>4</sub> VO <sub>3</sub>	116.98	7803-55-6	1.25 g
	Nitric acid 65%	HNO <sub>3</sub>	63.01	7697-37-2	330 mL
Stock solution	Potassium dihydrogen phosphate	H <sub>2</sub> KO <sub>4</sub> P	136.09	7778-77-0	43.94 g
REF1 calibration standard	Solution of 0 mg/L $PO_4$ -P with deionized water.	_	_	_	_
REF2 calibration standard	10000 mg/L PO <sub>4</sub> -P stock solution	_	_	_	Refer to Table 7 on page 5.
Validation standard (optional)	10000 mg/L PO <sub>4</sub> -P stock solution	_	_	_	Refer to Table 7 on page 5.
Cleaning solution (optional)	Hydrochloric acid (36%)	HCI	36.46	7647-01-0	41.5 mL

# Reagent preparation

## Reagent 1: Color

To prepare the Reagent 1: Color, refer to Necessary reagents for EZ1031 on page 4 to collect the applicable items. To calculate the correct reagent quantity, refer to Reagent consumption on page 3.

- **1. Solution A:** Dissolve 25 g of ammonium molybdate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub> \* 4 H<sub>2</sub>O p.a., > 99%) in 300 mL of warm deionized water. Fully dissolve.
- 2. Solution B: Dissolve 1.25 g ammonium metavanadate (NH<sub>4</sub>VO<sub>3</sub>) in 300 mL deionized water until the mixture boils. Let the mixture become cool. Then, add 330 mL nitric acid (HNO<sub>3</sub> 65%). Wait for the temperature of Reagent 2 to decrease to room temperature.
- 3. Pour Solution A into Solution B to make the color solution.
- 4. Pour the color solution into a 1000-mL volumetric flask. Mix.
- 5. Add deionized water to the mark.

## **Calibration standards**

Calibrations are completed with two standards, 1) a REF1 standard, and 2) a REF2 standard.

#### Stock solution

To prepare a 10000 mg/L PO<sub>4</sub>-P stock solution, collect the applicable items. Refer to Necessary reagents for EZ1031 on page 4.

- 1. Dissolve 43.94 g of potassium dihydrogen phosphate (H<sub>2</sub>KO<sub>4</sub>P) in 500 mL water.
- 2. Mix the solution.
- 3. Pour the solution into a 1000-mL volumetric flask. Mix.
- 4. Add deionized water to the mark.

#### **REF1** calibration standard

Prepare a REF1 calibration standard of 0-mg/L PO<sub>4</sub>-P with deionized water.

#### **REF2** calibration standard

To prepare the REF2 calibration standard, collect the applicable items. Refer to Necessary reagents for EZ1031 on page 4. Dilute the stock solution as necessary for the selected measuring range. Refer to Table 7.

- 1. Accurately pour x mL of the 10000 mg/L (PO<sub>4</sub>-P) stock solution into a 1000-mL volumetric flask. Refer to Stock solution on page 4.
- 2. Mix the solution.
- **3.** Add deionized water to the mark.

Table 7 Calibration standard preparation

Range code	Concentration REF 2 (mg/L)	x = Stock standard (mL) <sup>2</sup>
A	1	0.1
В	2.5	0.25
С	5	0.5
0	10	1
V	50	5
W	100	10
Х	250	25
Y	500	50
Z	750	75
5	1000	100

#### Validation standard

By default, the REF2 standard is used as the validation standard. Refer to REF2 calibration standard on page 5.

The automatic validation procedure is not enabled by default. Before a validation is started, connect the validation standard to the REF2 sample line.

# Cleaning solution

By default, the automatic cleaning procedure is not enabled.

The cleaning procedure must prevent the collection of chemicals in the analyzer. For an accurate cleaning procedure, examine the cleaning solution and the cleaning interval for each application. Make sure that the cleaning procedure is sufficient. Change the cleaning procedure if necessary.

The manufacturer recommends to use a 0.5 M hydrochloric acid (HCI) solution. Refer to Necessary reagents for EZ1031 on page 4. Prepare the solution as given in the steps that follow or use a commercially available solution.

- 1. Prepare a 0.5 M hydrochloric acid (HCI) solution.
- 2. Dilute 41.5 mL of hydrochloric acid (HCl 36%) in 500 mL of deionized water.
- 3. Pour the solution into a 1000-mL volumetric flask. Mix.
- **4.** Add deionized water to the mark.

<sup>&</sup>lt;sup>2</sup> Quantity to add to each 1 L of solution

