Supelco®
Analytical Products

Application Note

Phosphorus (total) in effluents

Photometric determination using the PMB method after decomposition in a thermoreactor

Introduction

Phosphorous is a key nutrient that can have adverse environmental effects when it is present at high levels. In excess, it can upset marine ecosystems by causing an excessive growth of algae and plants in a process termed eutrophication.[1] As a result, testing phosphorous in effluents is critical to maintaining the environment. In this application note, we describe a photometric determination of total phosphorous using Crack-Sets for sample digestion in a thermoreactor followed Spectroquant® Phosphate Test Kits.

Experimental

Method

In sulfuric solution orthophosphate ions react with molybdate ions to form molybdophosphoric acid. Ascorbic acid reduces this to phosphomolybdenum blue (PMB) that is determined photometrically. The method is analogous to EPA 365.2+3, APHA 4500-P E, and DIN EN ISO 6878.

Reagents and Instruments

Cat. No.	Product Description
Test Kits	
1.14543	Phosphate Cell Test (o-phosphate and total phosphorous) Method: photometric, PMB 0.05 - 5.00 mg/l PO_4-P; 0.2 - 15.3 mg/l PO_4^{3-}; 0.11 - 11.46 mg/l P_2O_5 Spectroquant® or
1.14729	Phosphate Cell Test (o-phosphate and total phosphorous) Method: photometric, PMB 0.5 - 25.0 mg/l PO_4-P 1.5 - 76.7 mg/l PO_4^{3-}; 1.1 - 57.3 mg/l P_2O_5 Spectroquant® or
1.14848	Phosphate Test (o-phosphate) Method: photometric, PMB 0.0025 - 5.00 mg/l PO_4-P; 0.0077 - 15.3 mg/l PO_4^{3-}; 0.0057 - 11.46 mg/l P_2O_5 Spectroquant® or
1.14688	Crack Set 10C for the digestion of lead, cadmium, iron, copper, nickel, phosphorus (total) and zinc 25 digestions Spectroquant® or

Reagents and Instruments (continued).

Cat. No.	Product Description
Test Kits	
1.14688	Crack Set 10C for the digestion of lead, cadmium, iron, copper, nickel, phosphorus (total) and zinc 25 digestions Spectroquant® or
1.14687	Crack Set 10 for the digestion of lead, cadmium, iron, copper, nickel, phosphorus (total) and zinc 100 digestions Spectroquant® or
1.09137	Sodium hydroxide solution c(NaOH) = 1 mol/l (1 N) Titripur® Reag. Ph Eur, Reag. USP or
1.09072	Sulfuric acid c(H_2SO_4) = 0.5 mol/l (1 N) Titripur® Reag. Ph Eur, Reag. USP
Instruments	
1.73026	Spectroquant® VIS Spectrophotometer Prove 100 plus or
1.73027	Spectroquant® UV/VIS Spectrophotometer Prove 300 plus or
1.73028	Spectroquant® UV/VIS Spectrophotometer Prove 600 plus or
1.09748	Spectroquant® Photometer NOVA 30 or
1.09751	Spectroquant® Photometer NOVA 60 or
1.06752	Spectroquant® Photometer NOVA 60A or
1.73632	Spectroquant® Colorimeter Move 100 or
	Thermoreactor with 120°C digestion temperature
Materials	
1.14946	Rectangular cells 10 mm or
1.14947	Rectangular cells 20 mm or
1.14944	Rectangular cells 50 mm or
1.14724	Empty cells with screw caps 16 mm Spectroquant®
1.09535	pH-indicator strips pH 0 - 14 Universal indicator non-bleeding pH 0 - 1 - 2 - 3 - 4 - 5 - 6 - 7 - 8 - 9 - 10 - 11 - 12 - 13 - 14 MQuant®

Also first generation Prove instruments are compatible and preprogrammed with this method.

Analytical Approach

Variant 1

Cat. No.	Product Description
1.14543	Spectroquant® Phosphate Cell Test
1.14729	Spectroquant® Phosphate Cell Test

Variant 2

Cat. No.	Product Description
1.14848	Spectroquant® Phosphate Test
1.14688	Crack-Set 10C

Variant 3

Cat. No.	Product Description
1.14848	Spectroquant® Phosphate Test
1.14687	Crack-Set 10
1.14724	Empty round cells 16 mm with screw caps

Hints:

Sewage water containing complexing agents like EDTA or Cyanide must be digested with potassium peroxodisulfate (= $K_2S_2O_8$). Digestion frees the PO_4-P from these complexes. When the COD concentration is >300 mg/l to 500 mg/l double the quantity of potassium peroxodisulfate.

Sample preparation using Variant 1:

Pipette 5.0 ml sample into a reaction cell. Add 1 dose reagent P-1K (= $K_2S_2O_8$), close the cell tightly, and mix. Heat the cell at 120°C in the preheated thermoreactor for 30 min. Allow the closed cell to cool to room temperature in a test-tube rack. Do not cool with cold water! Check the pH of the solution with Universal indicator strips. Adjust the pH with sodium hydroxide solution or sulfuric acid within the range 0 - 10.

Sample preparation using Variant 2:

Pipette 10.0 ml sample into a digestion cell. Add 1 dose reagent R-1K (= $K_2S_2O_8$), close the cell tightly, and mix. Heat the cell at 120°C in the preheated thermoreactor for 30 min. Allow the closed cell to cool to room temperature in the cell rack. Do not cool with cold water! Add 3 drops of reagent R-2K to the cool cell and mix. Check the pH of the solution with Universal indicator strips. Adjust the pH with sodium hydroxide solution or sulfuric acid within the range 0-10.

Sample preparation using Variant 3:

Pipette 10.0 ml sample into a digestion cell. Add 1 dose reagent R-1K (= $K_2S_2O_8$), close the cell tightly, and mix. Heat the cell at 120°C in the preheated thermoreactor for 30 min. Allow the closed cell to cool to room temperature in the cell rack. Do not cool with cold water! Add 3 drops of reagent R-2K to the cool cell and mix. Check the pH of the solution with Universal indicator strips. Adjust the pH with sodium hydroxide solution or sulfuric acid within the range 0-10

Phosphate content in mg/l PO_4-P = analysis value in mg/l PO_4-P

Analysis

Determine with the above-mentioned test kits.

Calculation

Phosphorus (total) content in mg/l P = analysis result in mg/l P

References

1. European Environment Agency. Eutrophication. Updated 2016. Accessed Oct 18, 2021. **eea.europa.eu/publications/signals-2000/page014.html**

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