Spectroquant®

Phosphate Cell Test



for the determination of orthophosphate and total phosphorus

USEPA approved for drinking water and wastewater

1. 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

2. Measuring range and number of determinations

Measuring range	Number of determinations
0.05 - 5.00 mg/l PO ₄ -P	
0.2 - 15.3 mg/l PO ₄ ³- 0.11 - 11.46 mg/l P₂O ₅	25

For programming data for selected photometers / spectrophotometers see www.service-test-kits.com.

3. Applications

This test measures only orthophosphate. Samples must be decomposed by digestion before total phosphorus can be measured (see section 6).

Sample material:

Groundwater and surface water, seawater

Drinking water

Wastewater

Nutrient solutions for fertilization

Soils after appropriate sample pretreatment

Food after appropriate sample pretreatment

4. Influence of foreign substances

This was checked in solutions containing 2 and 0 mg/l PO₄-P. The determination is not yet interfered with up to the concentrations of foreign substances given in the table.

	Concentrations of foreign substances in mg/l or $\%$						
Ag ⁺ AsO ₄ ³⁻ Ca ²⁺ Cd ²⁺ CN ⁻ Cr ³⁺ Cr ₂ O ₇ ²⁻ Cu ²⁺	1000 0.2 1000 1000 1000 1000 5 250		50 1000 10 1000 1000 1000 500 1000	Pb ²⁺ S ²⁻ SiO ₃ ²⁻ SO ₃ ²⁻ Zn ²⁺	2.5 1000 1000	EDTA Surfactants ¹⁾ COD (K-hydro phthalate) Na-acetate NaCl NaNO ₃ Na ₂ SO ₄	1000 100 ogen 150 ²⁾ 1 % 5 % 10 %

Reducing agents interfere with the determination.

1) tested with nonionic, cationic, and anionic surfactants



A higher COD may impair the efficacy of the digesting mixture in the determination of total phosphorus and thus result in false-low readings. Up to a maximum of 300 mg/l COD, this can be avoided by adding 2 doses of reagent P-1K instead of 1.

5. Reagents and auxiliaries

Please note the warnings on the packaging materials!

The test reagents are stable up to the date stated on the pack when stored closed at +15 to +25 $^{\circ}\text{C}.$

Package contents:

- 1 bottle of reagent P-1K
- 1 bottle of reagent P-2K
- 1 bottle of reagent P-3K 25 reaction cells
- 1 green dose-metering cap
- 1 blue dose-metering cap
- 1 sheet of round stickers for numbering the cells

Other reagents and accessories:

MQuant™ Phosphate Test, Cat. No. 110428,

measuring range 10 - 500 mg/l PO₄³ (3.3 - 163 mg/l PO₄-P)
MColorpHast[™] Universal indicator strips pH 0 - 14, Cat. No. 109535
Sulfuric acid 0.5 mol/l TitriPUR®, Cat. No. 109072
Spectroquant® CombiCheck 10, Cat. No. 114676

Phosphorus (total) standard solution CRM, 0.400 mg/l PO₄-P, Cat. No. 125046 Phosphorus (total) standard solution CRM, 4.00 mg/l PO₄-P, Cat. No. 125047 Hydrochloric acid 25 % for analysis EMSURE®, Cat. No. 100316

Pipette for a pipetting volume of 5.0 ml Thermoreactor

6. Preparation

Use only phosphate-free detergents to rinse glassware. Otherwise fill with hydrochloric acid (approx. 10 %) and leave to stand for several hours.

At the first use replace the screw caps of the reagent bottles P-1K and P-3K by the corresponding dose-metering caps:

Reagent P-1K: green dose-metering cap Reagent P-3K: blue dose-metering cap

Hold the respective reagent bottle **vertically** and, at each dosage, press the slide **all the way** into the dose-metering cap. **Before each dosage** ensure that the slide is completely retracted.



Reclose the reagent bottles with the corresponding screw caps at the end of the measurement series, since the function of the reagents is impaired by the absorption of atmospheric moisture.

- Analyze immediately after sampling.
- Digestion for the determination of total phosphorus (Wear eye protec-

Pretreated sample	5.0 ml	Pipette into a reaction cell.
Reagent P-1K	1 dose ¹⁾	Add, close the cell tightly, and mix.

Heat the cell at 120 $^{\circ}\text{C}^{\,2)}$ 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!

- 1) in the case of high COD values: 2 doses
- ²⁾ A digestion temperature of 100 °C may result in false-low readings (e.g. in the case of polyphosphates).
- Check the phosphate content with the MQuant[™] Phosphate Test. Samples containing more than 5.00 mg/l PO₄-P must be diluted with distilled water prior to digestion. Alternatively, it is also possible to use the Spectroquant® Phosphate Cell Test Cat. No. 114729 (measuring range 0.5 - 25.0 mg/l PO₄-P).
- The pH must be within the range 0 10. Adjust, if necessary, with sulfuric acid.
- Filter turbid samples.

7. Procedure

Pretreated sample (10 - 35 °C)	5.0 ml	Pipette into a reaction cell and mix or - after digestion for total phosphorus - shake the tightly closed cell vigorously after cooling.
Reagent P-2K ¹⁾ Reagent P-3K ¹⁾	5 drops ²⁾ 1 dose	Add, close the cell tightly, and mix. Add, close the cell tightly, and shake vigorously until the reagent is completely dissolved.

Leave to stand for 5 min (reaction time), then measure the sample in the photometer.

Notes on the measurement:

- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 0.80 0.95.
- The color of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.

8. Analytical quality assurance

recommended before each measurement series

To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, the phosphorus (total) standard solutions CRM, 0.400 mg/l PO $_4$ -P (Cat. No. 125046) and 4.00 mg/l PO $_4$ -P (Cat. No. 125047) - for the determination of total phosphorus - or Spectroquant 0 CombiCheck 10 can be used. Besides a **standard solution** with 0.80 mg/l PO $_4$ -P, CombiCheck 10 also contains an addition solution for determining sampledependent interferences (matrix effects).

Additional notes see under www.qa-test-kits.com.

Characteristic quality data:

In the production control, the following data were determined in accordance with ISO 8466-1 and DIN 38402 A51:

Standard deviation of the method (mg/l PO ₄ -P)	± 0.023
Coefficient of variation of the method (%)	± 1.0
Confidence interval (mg/l PO ₄ -P)	± 0.05
Number of lots	43

Characteristic data of the procedure:

Sensitivity: Absorbance 0.010 A corresponds to (mg/l PO ₄ -P)	0.02
Accuracy of a measurement value (mg/l PO ₄ -P)	max. ± 0.06

For quality and batch certificates for Spectroquant® test kits see the website.

9. Notes

- Reclose the reagent bottles immediately after use.
- Information on disposal can be obtained at www.disposal-test-kits.com. Merck KGaA, 64271 Darmstadt, Germany,



Tel. +49(0)6151 72-2440 www.analytical-test-kits.com EMD Millipore Corporation, 290 Concord Road, Billerica, MA 01821, USA, Tel. +1-978-715-1335

¹⁾ In the case of high chloride contents, it is recommended to switch the sequence of the reagents P-2K and P-3K.

²⁾ Hold the bottle vertically while adding the reagent!