Phosphorus, Reactive (Orthophosphate)

DOC316.53.01555

PhosVer[®] 3 HHC Method^{1, 2}

0.02 to 2.50 mg/L PO₄³⁻

Scope and application: For drinking water, wastewater and seawater.

- ¹ Adapted from Standard Methods for the Examination of Water and Wastewater.
- ² The HHC (high humidity climate) method for reactive phosphorus is recommended for use in humid environments.

☐ Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1	Instrument-sp	pecific	information
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Instrument	Sample cell orientation	Sample cell
DR6000	The fill line is to the right.	2495402
DR3800		
DR2800		<u>10 mL</u>
DR2700		
DR1900		
DR5000	The fill line is toward the user.	
DR3900		
DR900	The orientation mark is toward the user.	2401906

Before starting

Install the instrument cap on the DR900 cell holder before ZERO or READ is pushed.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

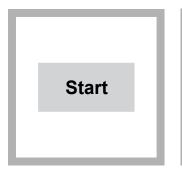
Description	Quantity
PhosVer 3 HHC Phosphate Reagent Powder Pillow	1
Ascorbic Acid Reagent Powder Pillow	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument- specific information on page 1.)	2

Refer to Consumable and replacement items on page 5 for order information.

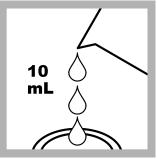
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
 - Let the sample temperature increase to room temperature before analysis.

Powder pillow procedure



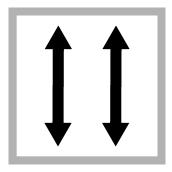
1. Start program **490 P React. PP**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Prepare the sample: Fill a sample cell with 10 mL of sample.



3. Add the contents of **one** PhosVer 3 HHC Phosphate Reagent Powder Pillow and **one** Ascorbic Acid Reagent Powder Pillow to the sample cell.

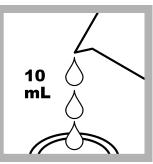


4. Immediately close the sample cell. Shake vigorously for 20–30 seconds. A blue color develops if phosphorus is in the sample.

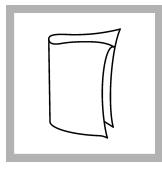


5. Start the instrument timer. A 2-minute reaction time starts.

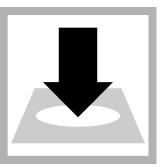
If the sample was digested using the Acid Persulfate digestion, a 10-minute reaction period is necessary.



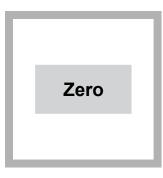
6. Prepare the blank: Fill a second sample cell with 10 mL of sample.



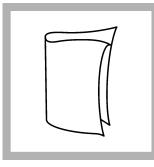
7. When the timer expires, clean the blank sample cell.



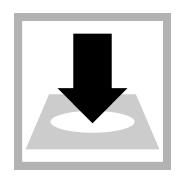
8. Insert the blank into the cell holder.



9. Push **ZERO**. The display shows 0.00 mg/L PO_4^{3-} .



10. Clean the prepared sample cell.



11. Insert the prepared sample into the cell holder.



12. Push **READ**. Results show in mg/L PO_4^{3-} .

Interferences

Interfering substance	Interference level
Aluminum	More than 200 mg/L
Arsenate	Interferes at any level
Chromium	More than 100 mg/L
Copper	More than 10 mg/L
Hydrogen Sulfide	Interferes at any level
Iron	More than 100 mg/L
Nickel	More than 300 mg/L
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary. A pH range of 2–10 is recommended.
Silica	More than 50 mg/L
Silicate	More than 10 mg/L
Turbidity or color	Samples with a high amount of turbidity can give inconsistent results. The acid in the reagents can dissolve some of the suspended particles and variable desorption of orthophosphate from the particles can occur.
Zinc	More than 80 mg/L

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample. Items to collect:

- Phosphate standard solution, 50 mg/L PO₄^{3–} ampule
- Ampule breaker
- Pipet, TenSette[®], 0.1–1.0 mL and tips
- Mixing cylinders, 25-mL (3)
- 1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
- 2. Go to the Standard Additions option in the instrument menu.
- 3. Select the values for standard concentration, sample volume and spike volumes.
- 4. Open the standard solution.
- Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.
- 6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
- 7. Select Graph to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 50 mg/L phosphate standard solution
- 100-mL volumetric flask, Class A
- 4-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water
- 1. Prepare a 2.00-mg/L phosphate standard solution as follows:
 - **a.** Use a pipet to add 4.00 mL of a 50-mg/L phosphate standard solution into the volumetric flask. (*Alternately, use one of the available mixed parameter standards. These standards contain 2.0 mg/L phosphate.*)
 - **b.** Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
- **2.** Use the test procedure to measure the concentration of the prepared standard solution.
- 3. Compare the expected result to the actual result.

Note: The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
490	2.00 mg/L PO ₄ ^{3–}	1.98–2.02 mg/L PO ₄ ^{3–}	0.02 mg/L PO ₄ ^{3–}

Summary of method

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. Ascorbic acid then reduces the complex, which gives an intense molybdenum blue color. The measurement wavelength is 880 nm for spectrophotometers (DR 1900: 710 nm) or 610 nm for colorimeters.

Consumable and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Reagent set, PhosVer® 3 HHC Method, contains:			2135100
PhosVer 3 HHC Phosphate Reagent Powder Pillows	1	100/pkg	2135069
Ascorbic Acid Reagent Powder Pillows	1	100/pkg	1457799

Required apparatus

Description	Quantity/test	Unit	ltem no.
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Phosphate Standard Solution, 10-mL Voluette [®] Ampule, 50 mg/L as PO ₄ ^{3–}	16/pkg	17110
Phosphate Standard Solution, 50-mg/L as PO ₄ ^{3–}	500 mL	17149
Phosphate Standard Solution, 1-mg/L as PO ₄ ³⁻	500 mL	256949
Drinking Water Standard, Mixed Parameter, Inorganic for F ⁻ , NO ₃ –N, PO ₄ ^{3–} , SO ₄ ^{2–}	500 mL	2833049
Wastewater Effluent Standard Solution, Mixed Parameter, for NH ₃ -N, NO ₃ -N, PO ₄ ^{3–} , COD, SO ₄ ^{2–} , TOC	500 mL	2833249
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Sampling bottle, with cap, low density polyethylene, 250 mL	12/pkg	2087076
Mixing cylinder, graduated, 25 mL	each	189640
Flask, volumetric, Class A, 100 mL, glass	each	1457442
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Paper, pH, 0–14 pH range	100/pkg	2601300
Phosphate Treatment Powder Pillows	100/pkg	1450199

Optional reagents and apparatus (continued)

Description	Unit	ltem no.
Phosphate Standard Solution, 10-mg/L as PO ₄ ³⁻	946 mL	1420416
Phosphate Standard Solution, 15-mg/L as PO ₄ ³⁻	100 mL	1424342
Phosphate Standard Solution, 100-mg/L as PO ₄ ³⁻	100 mL	1436832
Phosphate Standard Solution, 10-mL ampule, 500 mg/L as PO ₄ ^{3–}	16/pkg	1424210
Phosphate Standard Solution, 500-mg/L as PO ₄ ³⁻	100 mL	1424232
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet, TenSette [®] , 1.0–10.0 mL	each	1970010
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet tips for TenSette [®] Pipet, 1.0–10.0 mL	50/pkg	2199796
Pipet tips for TenSette [®] Pipet, 1.0–10.0 mL	250/pkg	2199725
Pipet, volumetric, Class A, 4.00 mL	each	1451504

