Phosphonates

DOC316.53.01109

PhosVer 3 with Persulfate UV Oxidation[1] — Method 8007

Multiple ranges from 0.02 to 125 mg/L PO_4^{3-} — Powder Pillows

Scope and application: For boiler and cooling water, wastewater and seawater.

[1] Adapted from Blystone, P., Larson, P., A Rapid Method for Analysis of Phosphate Compounds, International Water Conference, Pittsburgh, PA. (Oct 26-28, 1981)

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-specific information

Instrument	Sample cell orientation	Sample cell
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
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.
Clean all glassware with 6.0 N (1:1) hydrochloric acid, then fully rinse with deionized water to remove contaminants.
Do not use a detergent that contains phosphate to clean glassware. The phosphate in the detergent will contaminate the sample.
Wear UV safety goggles while the UV lamp is on.
Do not touch the UV lamp surface with bare fingers. Fingerprints can damage the glass. Rinse the lamp and wipe with a soft, clean tissue between tests.
The UV digestion in this procedure is normally complete in less than 10 minutes. However, high-organic loaded samples or a weak lamp can cause incomplete phosphate conversion. To check conversion efficiency, use a longer digestion time and make sure the readings do not increase.
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

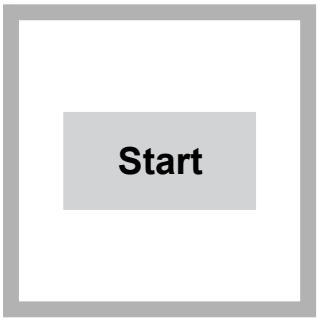
Description	Quantity
Bottle, square, with 25-mL mark	1
Cylinder, graduated mixing, 50-mL	1
Goggles, UV safety	1
Pipet, serological, 10-mL	1
PhosVer® 3 Phosphate Reagent Powder Pillows, 10-mL	2
Potassium Persulfate Powder Pillow for Phosphonate	1
Pipet filler, safety bulb	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2
Water, deionized	varies
UV lamp with power supply	1

Refer to Consumables and replacement items on page 7 for order information.

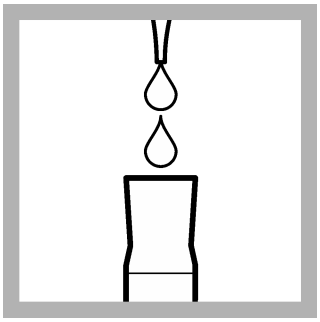
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (50%) hydrochloric acid and rinsed with deionized water.
- Do not use a commerical detergent to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 24 hours.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

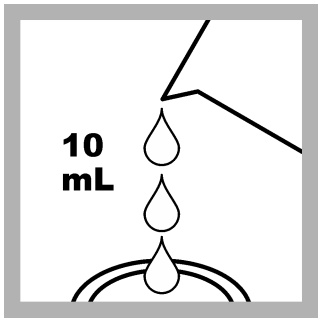
Powder pillow procedure with UV photolysis



1. Start program **501 Phosphonates**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

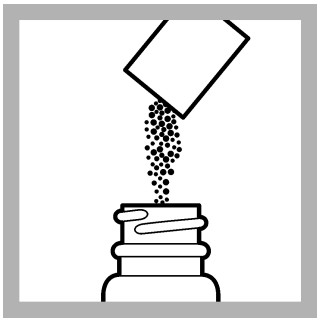


2. Select the sample size from Table 2 on page 4. Use a pipet to add the correct volume of sample into a 50-mL graduated cylinder.
If necessary, dilute the sample to 50 mL with deionized water and mix well.

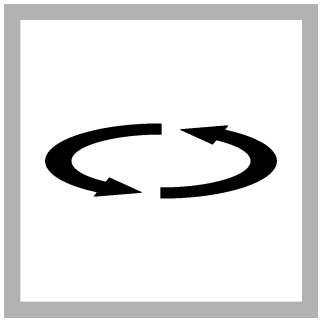


3. Prepare the blank: Fill a sample cell to the 10-mL mark with the diluted sample from step 2.

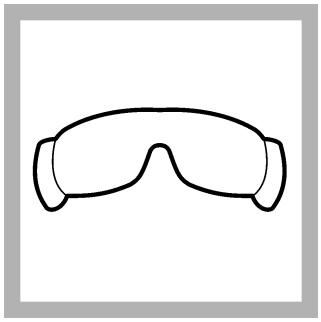
4. Prepare the digested sample: Fill a mixing bottle to the **25-mL mark** with the diluted sample from step 2.



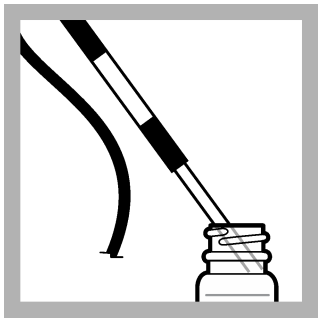
5. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the 25-mL sample.



6. Swirl to mix.



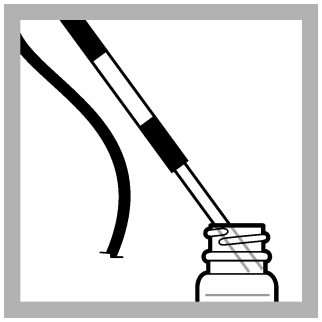
7. Put on UV safety goggles.



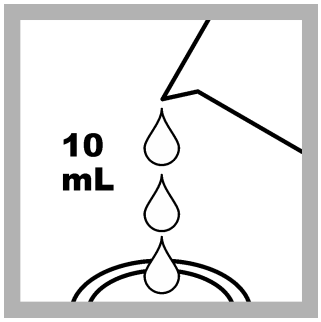
8. Put the ultraviolet lamp into the mixing bottle. Turn on the UV lamp.

9. Start the instrument timer. A 10-minute reaction time starts.

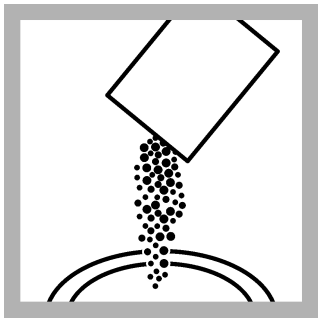
Phosphonates are converted to orthophosphate in this step.



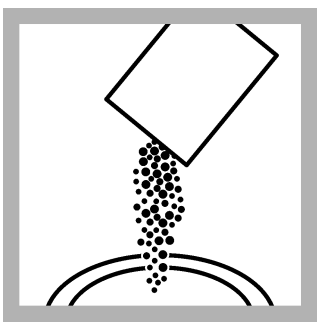
10. When the timer expires, turn off the UV lamp. Remove the UV lamp from the sample.



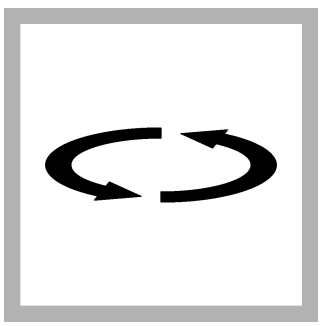
11. Prepare the sample: Fill a second sample cell to the 10-mL mark with the digested sample.



12. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the blank.



13. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the prepared sample.

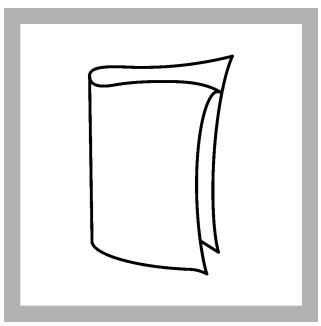


14. Immediately swirl both cells vigorously for 20–30 seconds to mix. Some powder may not dissolve.

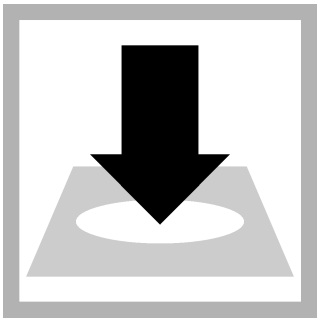
A blue color shows if phosphate is present. Both the sample and the blank may show color.

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

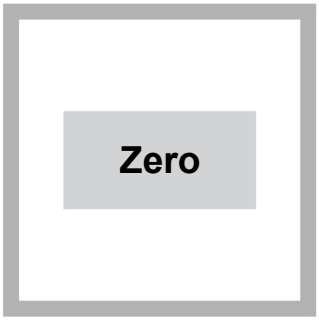
If the sample temperature is less than 15 °C (59 °F), wait 4 minutes for color development.



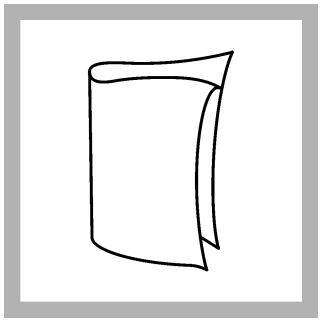
16. When the timer expires, clean the blank sample cell. **Complete the rest of the steps in this procedure within 3 minutes.**



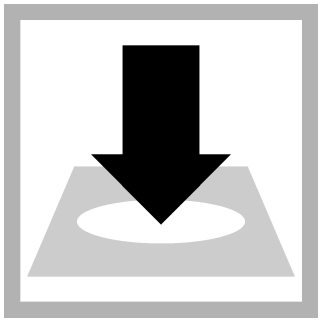
17. Insert the blank into the cell holder.



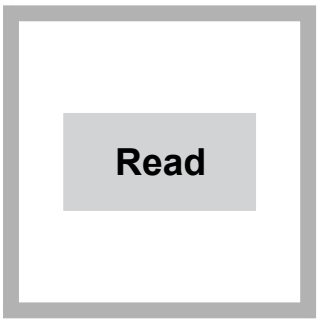
18. Push **ZERO**. The display shows 0.00 mg/L PO_4^{3-}.



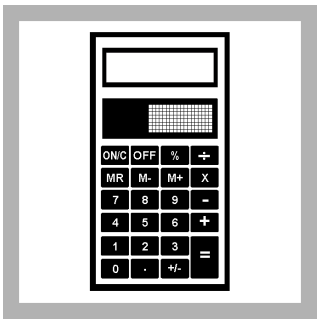
19. Clean the prepared sample cell.



20. Insert the prepared sample into the cell holder.



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



22. Multiply the results by the applicable sample volume multiplier in Table 2 on page 4 for the phosphonate concentration. Refer to Table 3 on page 5 to report results as the phosphonate compound.

Select the sample volume and multiplier

Use the expected phosphonate concentration to select a sample volume (refer to Table 2). Use the multiplier to adjust the test result (in mg/L PO_4^{3-}) for the sample volume that was used.

Table 2 Expected phosphonate range with multiplier

Expected range (mg/L phosphonate)	Sample volume (mL)	Multiplier
0–2.5	50	0.1
0–5	25	0.2
0–12.5	10	0.5
0–25	5	1
0–125	1	5

Convert phosphate to phosphonate

To convert the final test result from mg/L PO_4^{3-} to active phosphonate, multiply the final test result by the applicable PO_4 factor in Table 3.

Table 3 Conversion factors by phosphonate type

Acronym	Chemical name	MW g/mol	P/mol	PO_4 factor
DETPMPA	Diethylene triamine penta (methylene phosphonic acid)	573.2	5	1.207
EDTMPA	Ethylene diamine tetra (methylene phosphonic acid)	436.13	4	1.148
HEDPA	Hydroxyethylene diphosphonic acid	206.02	2	1.085
HMDTMPA	Hexamethylene diamine tetra (methylene phosphonic acid)	492.23	4	1.295
HPA	Hydroxyphosphono acetic acid	156.03	1	1.64
NTP	Nitrilotrimethylphosphonic acid	299.05	3	1.05
PBTC	Phosphonobutane tricarboxylic acid	270.1	1	2.84

Interferences

Interference levels decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10 mL, copper will begin to interfere above 50 mg/L.

Interfering substance	Interference level (5-mL sample)
Aluminum	100 mg/L
Arsenate	Interferes at all levels
Benzotriazole	10 mg/L
Bicarbonate	1000 mg/L
Bromide	100 mg/L
Calcium	5000 mg/L
CDTA	100 mg/L
Chloride	5000 mg/L
Chromate	100 mg/L
Copper	100 mg/L
Cyanide	100 mg/L (Increase the UV digestion to 30 minutes.)
Diethanoldithiocarbamate	50 mg/L
EDTA	100 mg/L
Iron	200 mg/L
Nitrate	200 mg/L
NTA	250 mg/L
Orthophosphate	15 mg/L
Phosphites and organophosphorus compounds	Reacts quantitatively. Metaphosphates and polyphosphates do not interfere.
Silica	500 mg/L
Silicate	100 mg/L
Sulfate	2000 mg/L

Interfering substance	Interference level (5-mL sample)
Sulfide	Interferes at all levels
Sulfite	100 mg/L
Thiourea	10 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.

Accuracy check

Digestion method

To validate the full procedure with the digestion, prepare a solution that has a known concentration of a phosphonate compound. Use the test procedure to measure the concentration of the phosphonate solution.

Standard solution method

To validate the colorimetric portion of the procedure (without digestion), use a phosphate standard solution for the sample and deionized water for the blank. Add the PhosVer 3 reagent directly to 10 mL of the phosphate standard solution and to the blank. The expected result is 10 times the value of the standard solution due to a built-in dilution factor of 10 in the calibration.

Items to collect:

- Phosphate Standard Solution, 1 mg/L (the expected result is 10 mg/L if 10 mL is used)

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard calibration 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
501	2.00 mg/L PO_4^{3-}	1.97–2.03 mg/L PO_4^{3-}	Refer to Sensitivity on page 6.

Sensitivity

The sensitivity depends on the sample volume. Sensitivity is expressed as PO_4^{3-} in Table 4. To express as a specific phosphonate, refer to Table 3 on page 5.

Table 4 Sensitivity per sample volume

Range (mg/L phosphonate)	Sample volume (mL)	Concentration change per 0.010 Abs change
0–2.5	50	0.02 mg/L PO_4^{3-}
0–5	25	0.04 mg/L PO_4^{3-}
0–12.5	10	0.10 mg/L PO_4^{3-}
0–25	5	0.20 mg/L PO_4^{3-}
0–125	1	1.00 mg/L PO_4^{3-}

Summary of method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV-catalyzed oxidation of phosphonate to orthophosphate. The orthophosphate reacts with the molybdate in the PhosVer 3 reagent to form a mixed phosphate/molybdate complex. This complex is reduced by the ascorbic acid in the PhosVer 3, which gives a blue color that is proportional to the amount of phosphonate in the original sample. The orthophosphate in the original sample is removed when the blank is used to set the zero concentration. The measurement wavelength is 880 nm for spectrophotometers (DR 1900: 710 nm) or 610 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Water, deionized	varies	4 L	27256
Phosphonate Reagent Set, 10 mL	1	100 tests	2429700
Includes:			
PhosVer® 3 Phosphate Reagent Powder Pillow[1], 10 mL	1	100/pkg	2106069
Potassium Persulfate Powder Pillow for Phosphonate	1	100/pkg	2084769

Required apparatus

Description	Quantity/test	Unit	Item no.
Bottle, square, with 25-mL mark	1	each	1704200
Beaker, polypropylene, 50 mL, low form	1	each	108041
Mixing cylinder, graduated, 50 mL, with glass stopper	1	each	189641
UV safety goggles	1	each	2113400
Pipet, serological, graduated, 10 mL	1	each	53238
Pipet filler, safety bulb	1	each	1465100
UV lamp with power supply, 115 VAC	1	each	2082800
OR			
UV lamp with power supply, 230 VAC	1	each	2082802

Recommended standards

Description	Unit	Item no.
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	256949

Optional reagents and apparatus

Description	Unit	Item no.
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	88449
Sulfuric Acid, concentrated, ACS	500 mL	97949
Thermometer, non-mercury, –10 to +225 °C	each	2635700
Paper, pH, 0–14 pH range	100/pkg	2601300
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800

[1] PhosVer is a registered trademark of Hach Company.

Optional reagents and apparatus (continued)

Description	Unit	Item no.
PhosVer 3 Phosphate Reagent Powder Pillows, 10 mL	1000/pkg	2106028
UV Lamp, shortwave, pencil type	each	2671000
Power supply, 115 V/60 Hz	each	2670700
Power supply, 220 V/50 Hz	each	2670702
Phosphate Standard Solution, 3-mg/L as PO_4^{3-}	946 mL	2059716
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, 30-mg/L as PO_4^{3-}	946 mL	1436716
Phosphate Standard Solution, 50-mg/L, 10-mL Voluette® Ampules	16/pkg	17110
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

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