Phosphonates

DOC316.53.01470

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

Multiple ranges from 0.1 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

Before starting

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.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
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
Flask, volumetric, 50-mL	1
Goggles, UV safety	1
Cylinder, graduated, 5-mL	1
PhosVer® 3 Phosphate Reagent Powder Pillows, 10-mL	2
Potassium Persulfate Powder Pillow for Phosphonate	1
Sample cells, 25-mm (10mL)	2

Items to collect (continued)

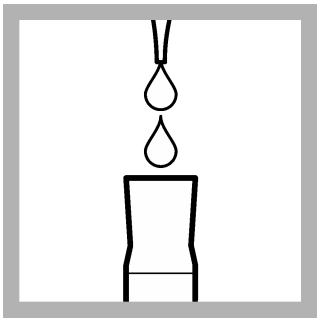
Description	Quantity
Water, deionized	varies
UV lamp with power supply	1

Refer to Consumables and replacement items on page 6 for order information.

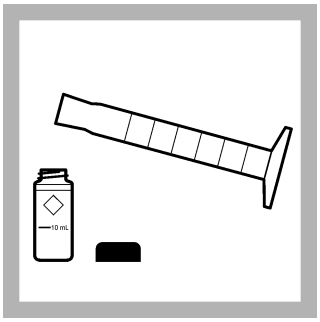
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) (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

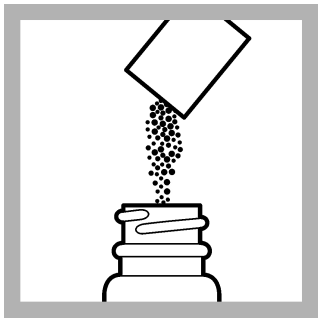


1. Select the sample size from Table 1 on page 4. Use a pipet to add the correct volume of sample into the supplied 50-mL volumetric flask or a 50-mL graduated cylinder. If necessary, dilute the sample to 50 mL with deionized water and mix well.

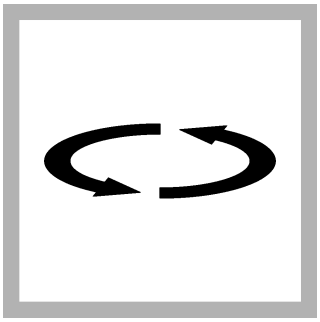


2. Prepare the blank: Fill a sample cell to the 10-mL mark with the diluted sample from step 1.

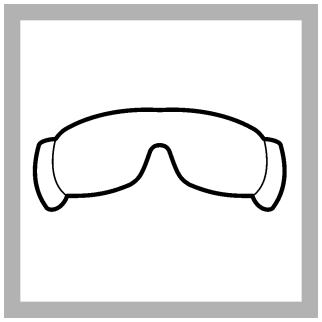
3. Prepare the digested sample: Pour 25-mL of the diluted sample from step 1 into a mixing bottle.



4. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the sample.



5. Swirl to mix.



6. Put on UV safety goggles.

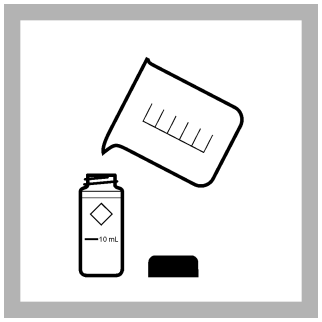
7. Put the ultraviolet lamp into the mixing bottle. Turn on the UV lamp.

8. Set and start a timer for 10 minute. A 10-minute reaction time starts.

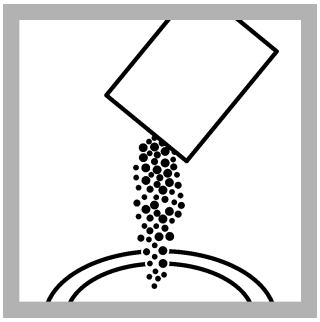
Phosphonates are converted to orthophosphate in this step.

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

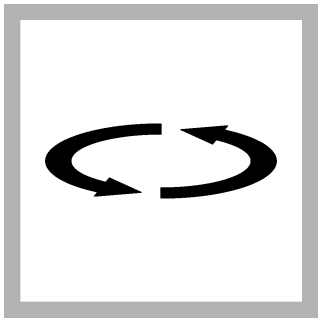
Note: *If the sample is hot, let the sample decrease to room temperature.*



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

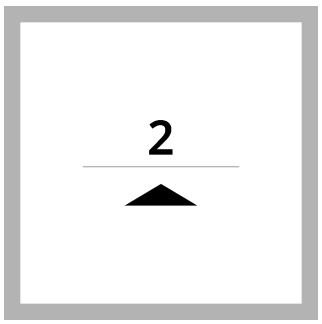


11. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to both the blank and the prepared sample.



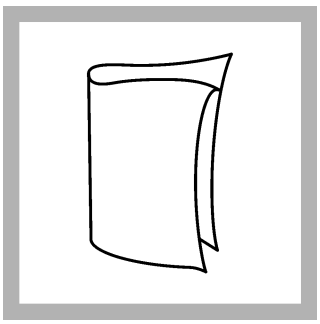
12. Immediately swirl both cells vigorously for 20–30 seconds to mix. Some powder may not dissolve.
A blue color will show if phosphate is in the sample. Both the sample and the blank may show color.

13. Set and start a timer for 2 minutes. A 2-minute reaction time starts.
If the sample temperature is less than 15 °C (59 °F), wait 4 minutes for color development.

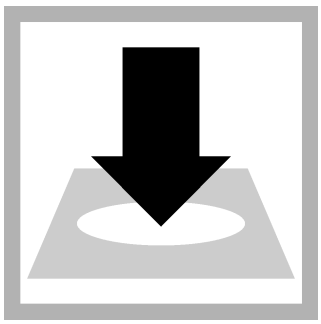


14. Set the instrument to channel 2.

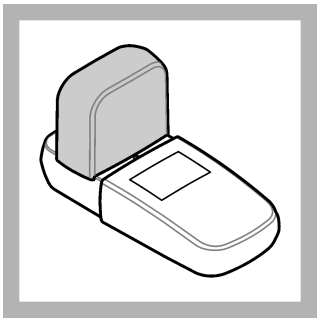
For DR300, push the up arrow button. For PCII, push the menu button, checkmark button, then the menu button again.



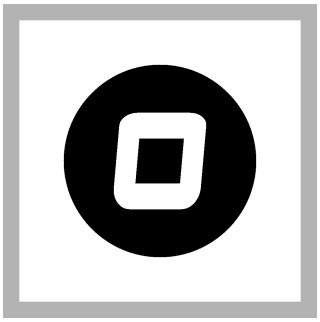
15. When the timer expires, clean the blank sample cell. Complete the rest of the steps in this procedure within 3 minutes.



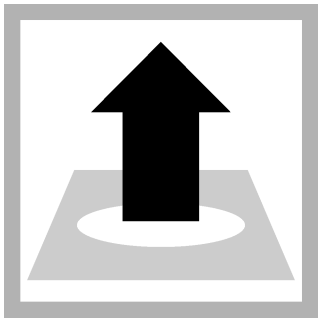
16. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



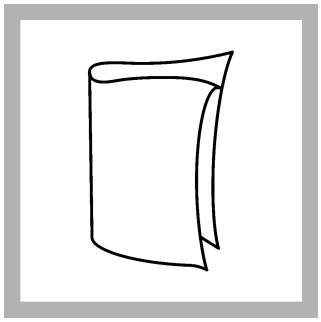
17. Install the instrument cap over the cell holder.



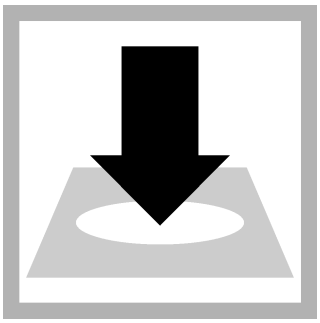
18. Push **ZERO**. The display shows "0.0"



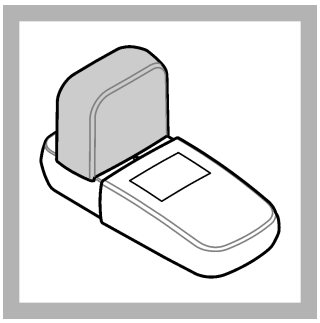
19. Remove the sample cell from the cell holder.



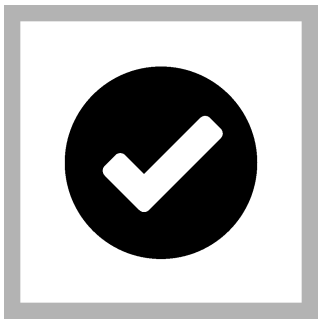
20. Clean the prepared sample cell.



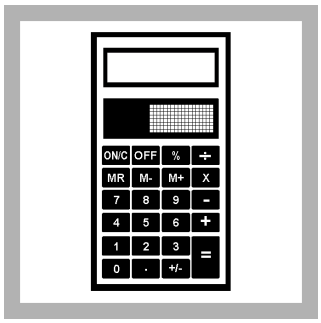
21. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



22. Install the instrument cap over the cell holder.



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



24. Multiply the results by the applicable sample volume multiplier in Table 1 on page 4 for the phosphonate concentration. Refer to Table 2 on page 4 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 1). Use the multiplier to adjust the test result (in mg/L PO_4^{3-}) for the sample volume that was used.

Table 1 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 conversion factor in Table 2.

Table 2 Conversion factors by phosphonate type

Phosphonate type	Conversion factor
DETPMPA	1.207
EDTMPA	1.148

Table 2 Conversion factors by phosphonate type (continued)

Phosphonate type	Conversion factor
HEDPA	1.085
HMDTMPA	1.295
HPA	1.49
NTP	1.05
PBTC	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
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

Standard additions method

Use the standard additions method 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, 10-mL Voluette® Ampule, 50 mg/L PO_4^{3-}
- Graduated cylinder, 50-mL
- Ampule breaker
- Volumetric flasks, 50-mL (3x)
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare three spiked samples: use the graduated cylinder to add equal portions of fresh sample to the three volumetric flasks. Refer to Table 1 on page 4 to select the sample volume. Use the same volume that was used when the test was done on samples.
2. Use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to the three water samples.
3. Add deionized water to the three volumetric flasks to the 50-mL mark. Mix well.
4. Use the test procedure to measure the concentration of each of the spiked samples.

   The expected phosphate concentration reading increase is 1.0 mg/L for each 0.1 mL of standard that is added (due to a dilution factor of 10 in the calibration).

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.*

Summary of method

In the phosphonate procedure, a UV-catalyzed oxidation converts phosphonate to orthophosphate. The orthophosphate is then quantified with the PhosVer 3 method. The orthophosphate concentration of the sample is subtracted by doing the orthophosphate test on the blank.

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

Consumables and replacement items (continued)

Description	Quantity/test	Unit	Item no.
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.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606
Flask, volumetric, polypropylene, 50-mL	1	each	1406041
Mixing cylinder, graduated, 50 mL, with glass stopper	1	each	189641
Bottle, mixing, square glass, 29-mL	1	6/pkg	43906
UV safety goggles	1	each	2113400
Cylinder, graduated, 5-mL	1	each	50897
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
Phosphate Standard Solution, 50-mg/L, 10-mL Voluette® Ampules	16/pkg	17110

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
Sodium Hydroxide Standard Solution, 5 N	100 mL MDB	245032
Thermometer, non-mercury, –10 to +225 °C	each	2635700
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Graduated cylinder, 50 mL	each	50841
Flask, volumetric, 50 mL	each	1457441
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
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
Paper, pH, 0–14 pH range	100/pkg	2601300
UV Lamp, shortwave, pencil type	each	2671000
Power supply, 115 V/60 Hz	each	2670700
Power supply, 220 V/50 Hz	each	2670702

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

Optional reagents and apparatus (continued)

Description	Unit	Item no.
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, 100-mg/L as PO_4	100 mL	1436832
Phosphate Standard Solution, 10-mL ampule, 500 mg/L as PO_4	16/pkg	1424210
Phosphate Standard Solution, 500-mg/L as PO_4	100 mL	1424232

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