

Phosphorus, Reactive (Orthophosphate)

DOC316.53.01115

Molybdovanadate Method¹

Method 8114

0.3 to 45.0 mg/L PO₄³⁻

Reagent Solution or AccuVac[®] Ampuls

Scope and application: For water and wastewater.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater.



Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

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

Table 1 Instrument-specific information for reagent addition





Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

For best results, the sample temperature should be 20–25 °C (68–77 °F).

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

Reagent solution

Description	Quantity
Molybdovanadate reagent	1.0 mL
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

AccuVac Ampuls

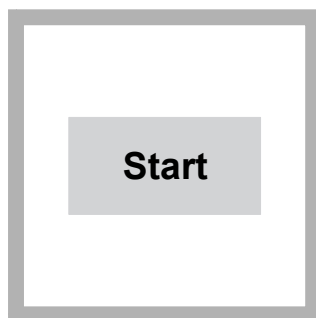
Description	Quantity
Molybdovanadate reagent AccuVac [®] Ampuls	2
Beaker, 50-mL	1
Stoppers for 18 mm tubes and AccuVac Ampuls	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection and storage

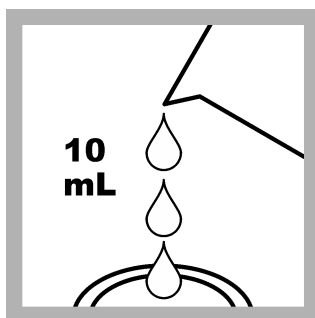
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples 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.

Reagent solution procedure

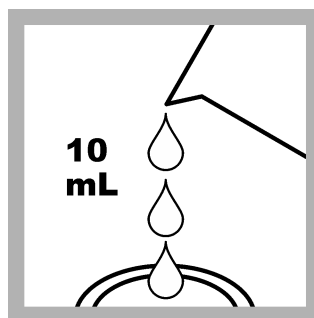


1. Start program **480 P React. Mo**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

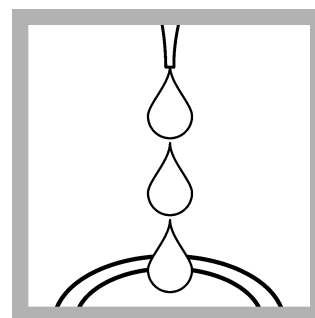
Note: Although the program name can be different between instruments, the program number does not change.



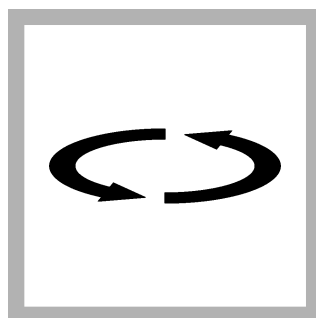
2. **Prepare the blank:** Fill a sample cell with 10 mL of deionized water.



3. **Prepare the sample:** Fill a second sample cell with 10 mL of sample.



4. Add 0.5 mL of Molybdovanadate reagent to each cell.

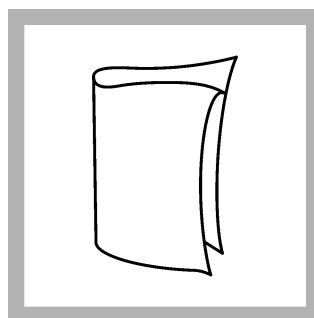


5. Swirl to mix.

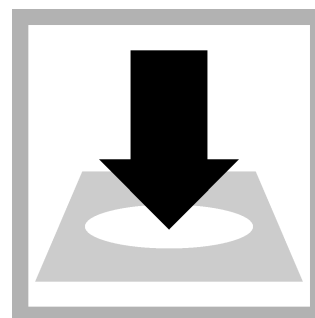


6. Start the instrument timer. A 7-minute reaction time starts.

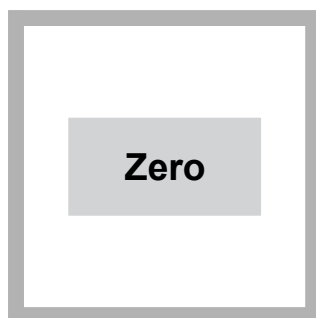
If the sample concentration is greater than 30 mg/L PO_4^{3-} , read at exactly 7 minutes or make a 1:1 dilution of the sample and repeat the test.



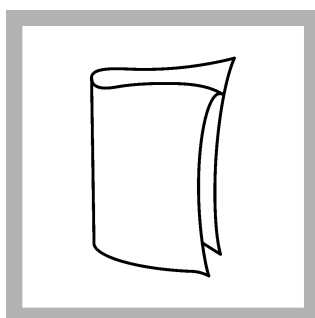
7. When the timer expires, clean the blank sample cell.



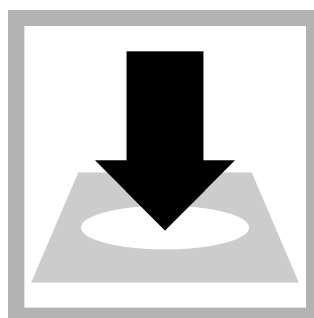
8. Insert the blank into the cell holder.



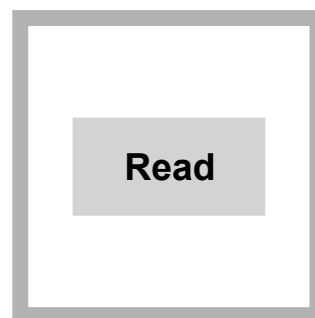
9. Push **ZERO**. The display shows 0.0 mg/L PO_4^{3-} .



10. Clean the prepared sample cell.

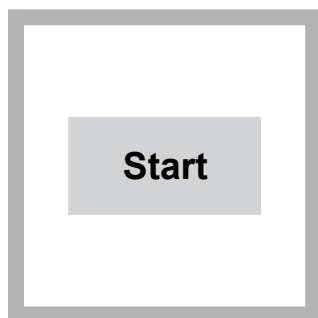


11. Insert the prepared sample into the cell holder.



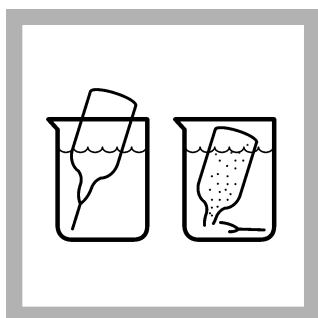
12. Push **READ**. Results show in mg/L PO_4^{3-} .

AccuVac Ampuls procedure

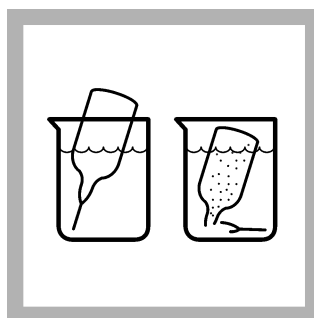


1. Start program **482 P React. Mo. AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

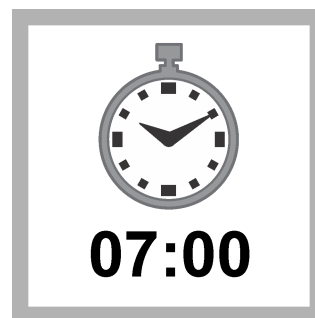
Note: Although the program name can be different between instruments, the program number does not change.



2. **Prepare the blank:** Pour at least 40 mL of deionized water into a 50-mL beaker. Fill an AccuVac Ampul with deionized water. Keep the tip immersed while the AccuVac Ampul fills completely.

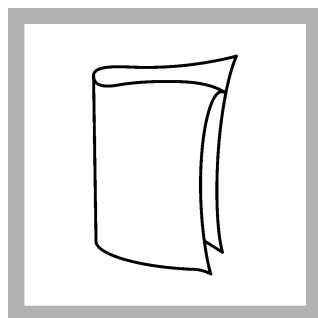


3. **Prepare the sample:** Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

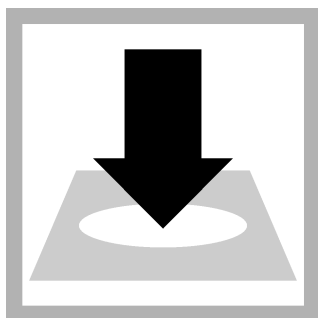


4. Start the instrument timer. A 7-minute reaction time starts.

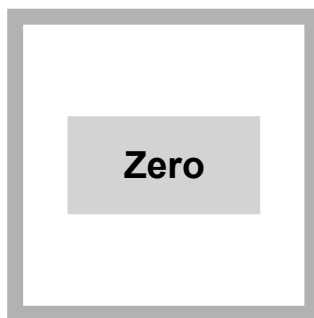
If the sample concentration is greater than 30 mg/L PO_4^{3-} , read at exactly 7 minutes or make a 1:1 dilution of the sample and repeat the test.



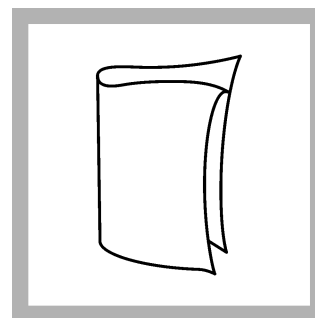
5. When the timer expires, clean the blank AccuVac Ampul.



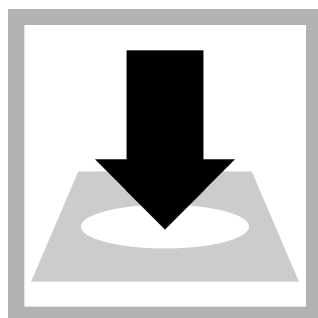
6. Insert the blank AccuVac Ampul into the cell holder.



7. Push **ZERO**. The display shows 0.0 mg/L PO_4^{3-} .



8. Clean the AccuVac Ampul.



9. Insert the prepared sample AccuVac Ampul into the cell holder.



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

Interferences

Table 3 shows the interferences and interference levels. Table 4 shows the substances that do not interfere at or below the indicated levels.

Table 3 Interfering substances

Interfering substance	Interference level
Arsenate	Causes a positive interference if the sample is warm when the reagent is added. The sample can be gently warmed to room temperature without interference.
Iron, ferrous	Causes a blue color which interferes at more than 100 mg/L
Molybdate	Negative interference at more than 1000 mg/L
Silica	Positive interference if the sample is warm when the reagent is added. The sample can be gently warmed to room temperature without interference.
Sulfide	Negative interference. Correct for this interference as follows: <ol style="list-style-type: none"> 1. Measure 50 mL of sample into an Erlenmeyer flask. 2. Add Bromine Water by drops with constant swirling until a permanent yellow color remains. 3. Add Phenol Solution by drops until the yellow color just disappears. Use this sample in the test procedure.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. The pH should be approximately 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Negative interference
Temperature	Temperatures below 20 °C (68 °F) cause a negative interference. Temperatures above 25 °C (77 °F) cause a positive interference. The sample can be gently warmed to room temperature without interference.

Table 4 Substances that do not interfere at less than 1000 mg/L

Pyrophosphate	Tetraborate	Selenate	Benzoate
Citrate	Oxalate	Lactate	Tartrate
Formate	Salicylate	Al ³⁺	Fe ³⁺
Mg ²⁺	Ca ²⁺	Ba ²⁺	Sr ²⁺
Li ⁺	Na ⁺	K ⁺	NH ₄ ⁺
Cd ²⁺	Mn ²⁺	NO ₃ ⁻	NO ₂ ⁻
SO ₄ ²⁻	SO ₃ ²⁻	Pb ²⁺	Hg ⁺
Hg ²⁺	Sn ²⁺	Cu ²⁺	Ni ²⁺
Ag ⁺	U ⁴⁺	Zr ⁴⁺	AsO ₃ ⁻
Br ⁻	CO ₃ ²⁻	ClO ₄ ⁻	CN ⁻
IO ₃ ⁻	SiO ₄ ⁴⁻	—	—

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, 500 mg/L PO₄³⁻ 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.
5. 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 25-mL portions of fresh sample. Mix well.

Note: For AccuVac® Ampuls, add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution to three 25-mL portions of fresh sample.

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:

- 10-mg/L Phosphate Standard Solution

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 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 slight 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
480	30.0 mg/L PO ₄ ³⁻	29.6–30.4 mg/L PO ₄ ³⁻	0.3 mg/L PO ₄ ³⁻
482	30.0 mg/L PO ₄ ³⁻	29.7–30.3 mg/L PO ₄ ³⁻	0.3 mg/L PO ₄ ³⁻

Summary of method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. In the presence of vanadium, yellow molybdovanadophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration. The measurement wavelength is 430 nm for spectrophotometers or 420 nm for colorimeters.

Consumables and replacement items

Note: Product and Article numbers may vary for some selling regions. Contact the appropriate distributor or refer to the company website for contact information.

Required reagents

Description	Quantity/Test	Unit	Item no.
Molybdovanadate Reagent Solution	1.0 mL	100 mL MDB	2076032
OR			
Molybdovanadate Reagent AccuVac [®] Ampuls	2	25/pkg	2525025
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Phosphate Standard Solution, 10-mg/L as PO ₄	946 mL	1420416
Phosphate Standard Solution, 10-mL ampule, 500 mg/L as PO ₄	16/pkg	1424210
Wastewater Influent Standard Solution, Mixed Parameter, for NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , TOC	500 mL	2833149

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac [®] Drainer	each	4103600
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Bromine Water, 30-g/L	29 mL	221120
Mixing cylinder, graduated, 25-mL	each	2088640
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Paper, pH, 0–14 pH range	100/pkg	2601300
Phenol Solution, 30-g/L	29 mL	211220
Phosphate Standard Solution, 3-mg/L as PO ₄ ³⁻	946 mL	2059716
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	100 mL	1436832
Phosphate Standard Solution, 500-mg/L as PO ₄	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
Thermometer, non-mercury, –10 to +225 °C	each	2635700



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