

# Phosphorus, Reactive (Orthophosphate)

DOC316.53.01115

**Molybdovanadate Method<sup>1</sup>****0.3 to 45.0 mg/L PO<sub>4</sub><sup>3-</sup>****Method 8114****Reagent Solution****Scope and application:** For water and wastewater.<sup>1</sup> Adapted from Standard Methods for the Examination of Water and Wastewater.

## 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.

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

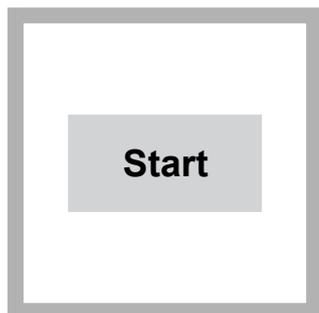
Description	Quantity
Molybdovanadate reagent	1.0 mL
Sample cells (For information about sample cells, adapters or light shields, refer to <a href="#">Instrument-specific information</a> on page 1.)	2

Refer to [Consumables and replacement items](#) on page 5 for order information.

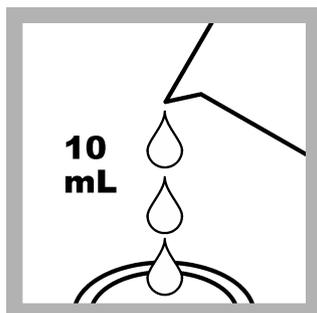
## Sample collection and storage

- 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.

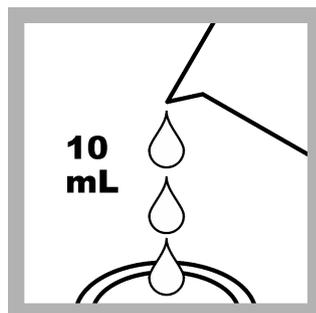
## Test procedure



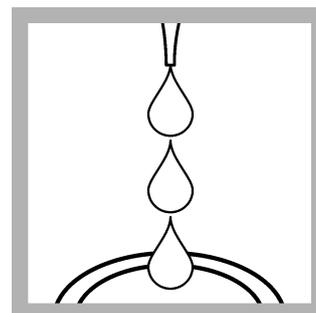
**1. Start program 480 P React. Mo.** For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



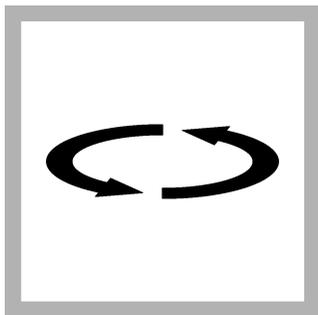
**2. Prepare the blank:** Fill a sample cell with 10 mL of deionized water.



**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.**

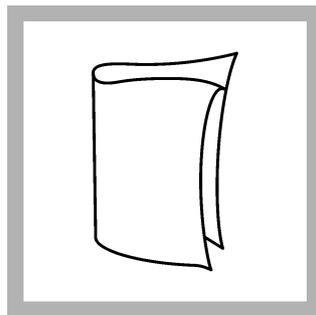


5. Swirl to mix.

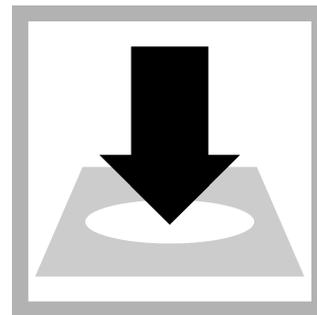


6. Start the instrument timer. A 7-minute reaction time starts.

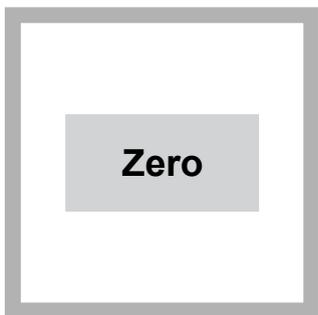
If the sample concentration is greater than 30 mg/L  $\text{PO}_4^{3-}$ , read at exactly 7 minutes or make a 1:1 dilution of the sample and repeat the test.



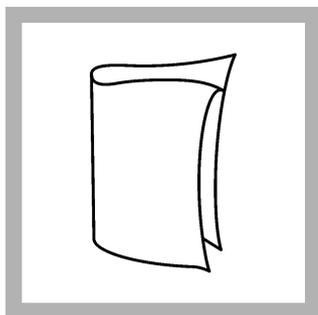
7. When the timer expires, clean the blank sample cell.



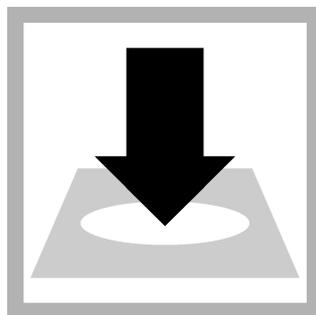
8. Insert the blank into the cell holder.



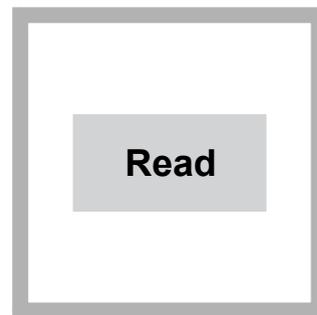
9. Push **ZERO**. The display shows 0.0 mg/L  $\text{PO}_4^{3-}$ .



10. Clean the prepared sample cell.



11. Insert the prepared sample into the cell holder.



12. Push **READ**. Results show in mg/L  $\text{PO}_4^{3-}$ .

## Interferences

Table 2 shows the interferences and interference levels. Table 3 shows the substances that do not interfere at or below the indicated levels.

**Table 2 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"> <li>1. Measure 50 mL of sample into an Erlenmeyer flask.</li> <li>2. Add Bromine Water by drops with constant swirling until a permanent yellow color remains.</li> <li>3. Add Phenol Solution by drops until the yellow color just disappears.</li> </ol> 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 pretreatment may be necessary. The pH should be approximately 7.

**Table 2 Interfering substances (continued)**

Interfering substance	Interference level
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 3 Substances that do not interfere at less than 1000 mg/L**

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

## 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 PO<sub>4</sub><sup>3-</sup> 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.
  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 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
480	30.0 mg/L PO <sub>4</sub> <sup>3-</sup>	29.6–30.4 mg/L PO <sub>4</sub> <sup>3-</sup>	0.3 mg/L PO <sub>4</sub> <sup>3-</sup>

## 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
Water, deionized	varies	4 L	27256

### Recommended standards and apparatus

Description	Unit	Item no.
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Phosphate Standard Solution, 10 mg/L as PO <sub>4</sub> <sup>3-</sup>	946 mL	1420416
Phosphate Standard Solution, 10-mL ampule, 500 mg/L as PO <sub>4</sub> <sup>3-</sup>	16/pkg	1424210
Wastewater Influent Standard Solution, Mixed Parameter, for NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> <sup>3-</sup> , COD, SO <sub>4</sub> <sup>2-</sup> , TOC	500 mL	2833149

### Optional reagents and apparatus

Description	Unit	Item no.
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

Optional reagents and apparatus (continued)

Description	Unit	Item no.
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 <sub>4</sub> <sup>3-</sup>	946 mL	2059716
Phosphate Standard Solution, 15-mg/L as PO <sub>4</sub> <sup>3-</sup>	100 mL	1424342
Phosphate Standard Solution, 30-mg/L as PO <sub>4</sub> <sup>3-</sup>	946 mL	1436716
Phosphate Standard Solution, 50-mg/L, 10-mL Voluette® Ampules	16/pkg	17110
Phosphate Standard Solution, 100-mg/L as PO <sub>4</sub> <sup>3-</sup>	100 mL	1436832
Phosphate Standard Solution, 500-mg/L as PO <sub>4</sub> <sup>3-</sup>	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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