Phosphorus, Reactive

DOC316.53.01114

Molybdovanadate Rapid Liquid Method[1]

Method 8114

0.3 to 45.0 mg/L PO_4^{3-} (HR)

Pour-Thru Cell

Scope and application: For treated and natural waters.

[1] 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.

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	Pour-Thru Kit	Adapter
DR 6000	The flow path is to the right.	LQV157.99.20002	—
DR 3800		5940400	LZV585 (B)
DR 2800		5940400	LZV585 (B)
DR 2700		5940400	LZV585 (B)
DR 1900		LZV899	—
DR 5000	The flow path is toward the user.	LZV479	—
DR 3900		LQV157.99.10002	—

Before starting

Refer to the instrument documentation for Pour-Thru cell and module assembly and installation. Make sure to install the Pour-Thru cell correctly.
To protect the Pour-Thru Cell from contamination when not in use, invert a small beaker over the top of the glass funnel.
Clean the Pour-Thru cell and all labware before use. Refer to Clean the labware on page 3 and Clean the Pour-Thru Cell on page 3.
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

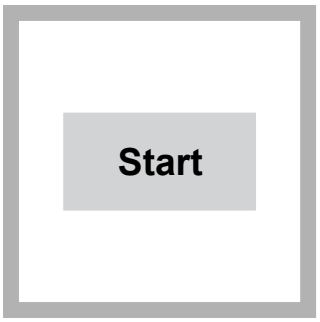
Description	Quantity
Molybdovanadate Reagent Solution	1 mL
Cylinder, graduated, polypropylene, 25-mL	1
Dispenser, adjustable volume, 1.0–5.0 mL	2
Flask, Erlenmeyer, Polymethylpentene, screw cap, 125-mL	2
Water, deionized	varies
Pour-Thru Module and Cell (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1

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

Sample collection and storage

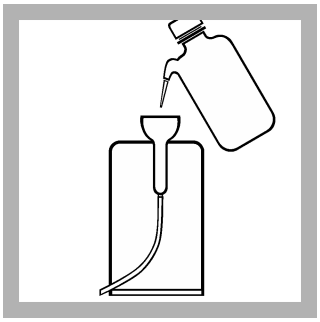
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples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



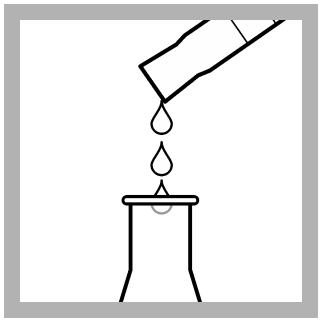
1. Start program **489 P React. Mo. HR RL**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

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

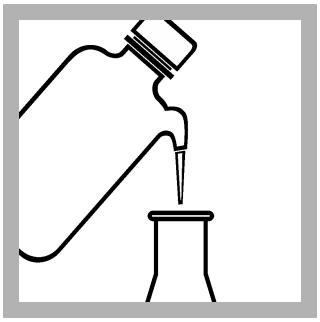


2. Flush the Pour-Thru Cell with at least 50-mL of deionized water.

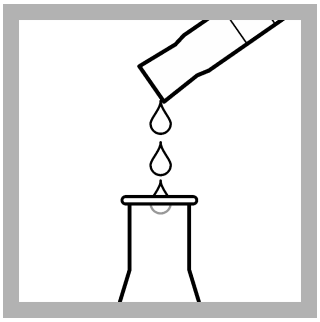
3. Rinse a clean plastic 125-mL Erlenmeyer flask and a 25-mL graduated cylinder with deionized water.



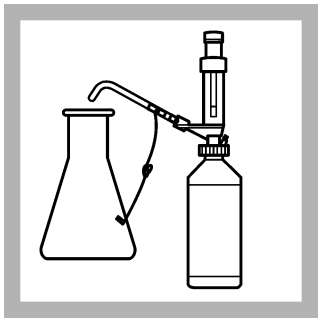
4. Prepare the blank: Measure 25 mL of deionized water in the graduated cylinder. Pour the water into the Erlenmeyer flask.



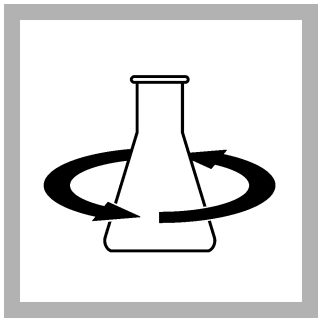
5. Rinse another clean plastic 125-mL Erlenmeyer flask with deionized water.



6. Prepare the sample: Measure 25 mL of sample in the graduated cylinder. Pour the sample into the second Erlenmeyer flask.

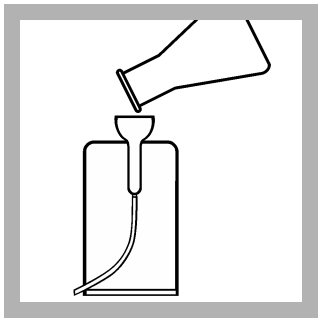


7. Use the bottle-top dispenser to add 1.0 mL of Molybdovanadate reagent to each flask.

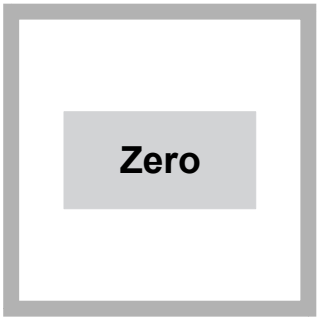


8. Swirl to mix.
A yellow color forms if phosphate is in the sample. A small amount of yellow can form in the blank because of the reagent color.

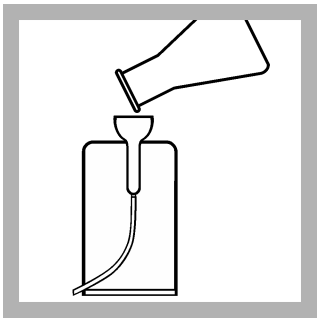
9. Start the instrument timer. A 7-minute reaction time starts.
If the sample concentration is more than 30 mg/L PO_4^{3-}, read at exactly 7 minutes or make a 1:1 dilution of the sample and start the test again.



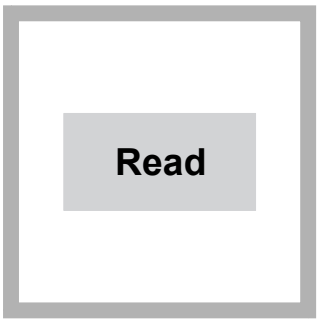
10. When the timer expires, pour the contents of the flask that contains the blank into the Pour-Thru Cell.



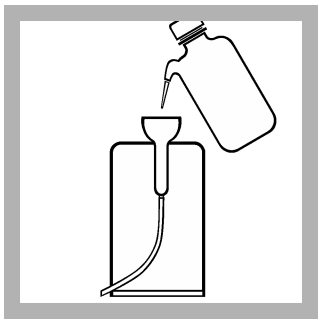
11. When the flow stops, push **ZERO**. The display shows 0.0 mg/L PO_4^{3-}.



12. Pour the prepared sample into the Pour-Thru Cell.



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



14. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Clean the labware

Fully clean all containers that are used in this test to remove possible traces of phosphate.

1. Clean containers (do not use phosphate detergents), then rinse with high quality deionized water.
2. Soak for 10 minutes with a 1:25 dilution of Molybdovanadate Reagent in deionized water.
3. Fully rinse with deionized water. Keep the containers tightly closed when not in use. Use these containers only for phosphate analysis. If the containers are rinsed and closed after each use, only occasional treatment is necessary.
4. Fill the Pour-Thru Cell with this same mixture of reagent and water, then let it stay in the Pour-Thru Cell for several minutes before use. Rinse with deionized water.

Clean the Pour-Thru Cell

The Pour-Thru Cell can collect a buildup of products with color, especially if the reacted solutions stay in the cell for long periods of time after measurement.

1. Rinse the Pour-Thru Cell with 1:5 dilution of ammonium hydroxide to remove the color.
2. Fully rinse with deionized water.
3. Put a cover on the Pour-Thru Cell funnel when it is not in use.

Interferences

Table 2 shows a list of substances, interference levels and type of interference. Table 3 shows a list of substances that do not interfere in concentrations less than 1000 mg/L.

Table 2 Interfering substances

Interfering substance	Interference level
Arsenate	Negative interference. Positive interference if the sample is warm when the reagent is added.
Bismuth	Negative interference.
Fluoride	Negative interference.
Iron, Ferrous	Causes a blue color which interferes at more than 100 mg/L
Molybdate	Negative interference.
Silica	Positive interference if the sample is warm when the reagent is added.
Sulfide	Negative interference. Sulfide interference can be removed by oxidation with Bromine Water as follows: **1.** Measure 25 mL of sample into a flask. **2.** Add Bromine Water, one drop at a time with constant swirling until a permanent yellow color shows. **3.** Add Phenol Solution, one drop at a time just until the solution is colorless. Continue with step 7 of the test procedure.
Thiocyanate	Negative interference.
Thiosulfate	Negative interference.
Thorium	Negative interference.
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.

Table 3 Noninterfering substances at low concentrations (less than 1000 mg/L)

Pyrophosphate	Tetraborate	Benzoate
Citrate	Lactate	Formate
Oxalate	Tartrate	Salicylate
Al^{3+}	Selenate	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
Zr^{4+}	AsO_3^{-}	Br^{-}
CO_3^{2-}	ClO_4^{-}	CN^{-}
IO_3^{-}	Fe^{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, Voluette Ampule, 500-mg/L as PO_4^{3-}
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips

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:

- 10.0-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
489	10.0 mg/L PO_4^{3-}	9.9–10.1 mg/L PO_4^{3-}	0.34 mg/L PO_4^{3-}

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to give a phosphomolybdate complex. Vanadium in the sample causes a yellow vanadomolybdophosphoric acid to form. The intensity of the yellow color is proportional to the phosphate concentration. The measurement wavelength is 430 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Molybdovanadate Reagent Solution	2 mL	500 mL	2076049
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, polypropylene, 25-mL	1	each	108140
Dispenser, adjustable volume, 1.0–5.0 mL	2	each	2563137
Flask, Erlenmeyer, Polymethylpentene, screw cap, 125-mL	2	each	2089843

Recommended standards

Description	Unit	Item no.
Phosphate Standard Solution, 10-mg/L as PO_4	946 mL	1420416
Phosphate Standard Solution, 500-mg/L, 10-mL Voluette® Ampules	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.
Ammonium Hydroxide, 58%	500 mL	10649
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Bromine Water, 30-g/L	29 mL	221120
Filter paper, folded, 3–5-micron, 12.5-cm	100/pkg	69257
Funnel, poly, 65-mm	each	108367
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	88449
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696

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