Phosphorus, Reactive

DOC316.53.01117

Ascorbic Acid Rapid Liquid Method[1]

Method 10055

19 to 3000 µg/L PO_4^{3-} (LR)

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
DR6000	The flow path is to the right.	LZV899[1]	—
DR3800		5940400	LZV585 (B)
DR2800		5940400	LZV585 (B)
DR2700		5940400	LZV585 (B)
DR1900		LZV899	—
DR5000	The flow path is toward the user.	LZV479	—
DR3900		LZV899	—

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.
Prepare the reagent before use. Refer to Prepare the reagent on page 3.
Clean the Pour-Thru cell and all labware before use. Refer to Clean the labware on page 4 and Clean the Pour-Thru Cell on page 4.
The reaction time changes with the sample temperature. For best results run the test at room temperature (approximately 20 °C (68 °F)).
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
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.

[1] A cell cover (LZQ105) and a tube set (LZQ102) is also necessary.

Items to collect

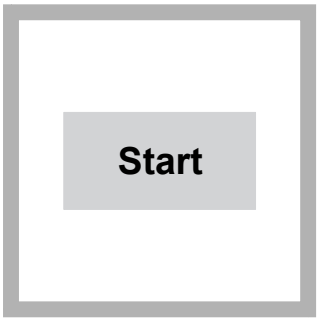
Description	Quantity
Ascorbic Acid reagent (prepare before use)	1 mL
Molybdate reagent	2 mL
Cylinder, graduated, polypropylene, 25 mL	1
Fixed volume micropipette, 1 mL	2
Flask, Erlenmeyer, Polymethylpentene, screw cap, 125 mL	2
Water, deionized	varies
Pour-Thru Module and Cell (For information about 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

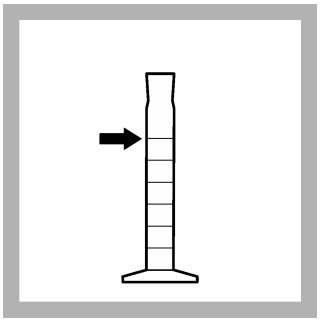
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples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

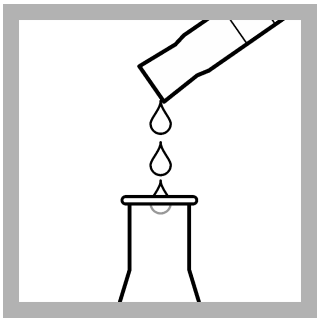


1. Start program **488 P React. LR RL**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

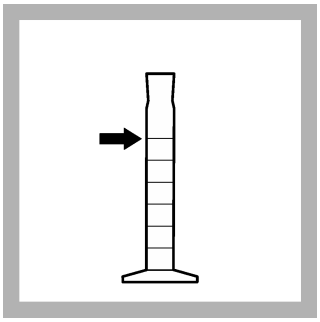
2. Rinse two clean Erlenmeyer flasks and one clean graduated cylinder three times with the sample.



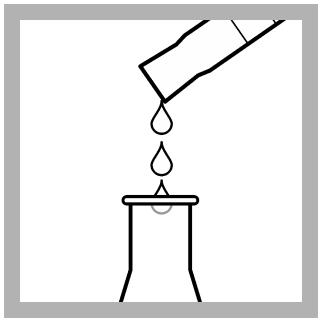
3. Fill the rinsed cylinder to the 25-mL mark with sample.



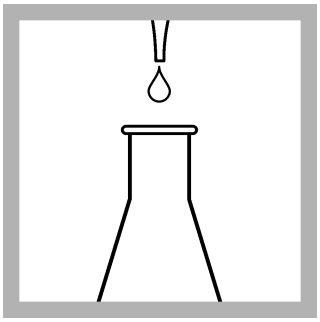
4. Pour the contents of the cylinder into one of the Erlenmeyer flasks.



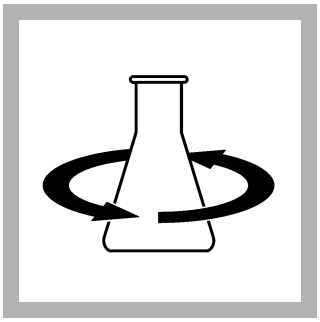
5. Measure a second 25-mL portion of sample into the graduated cylinder.



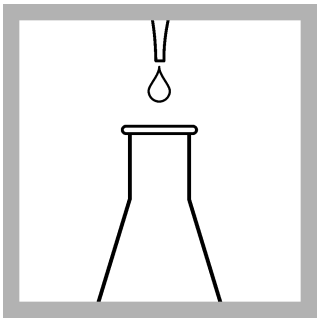
6. Pour the contents into the second Erlenmeyer flask.



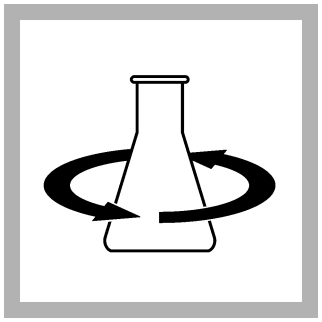
7. Use the micropipette to add 1.0 mL of Molybdate reagent to each Erlenmeyer flask.



8. Swirl to mix.

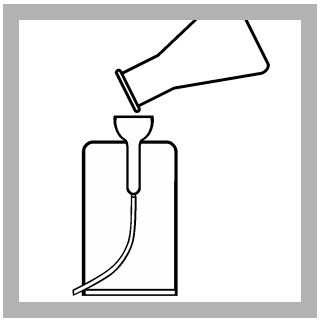


9. Prepare the sample: Use the micropipette to add 1.0 mL of prepared Ascorbic Acid reagent to one of the Erlenmeyer flasks. The remaining Erlenmeyer flask is the blank.

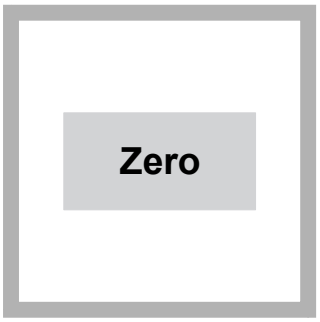


10. Swirl to mix.

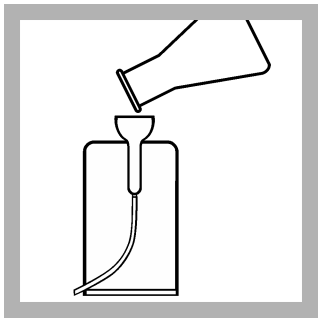
11. Start the instrument timer. A 5-minute reaction time starts.



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

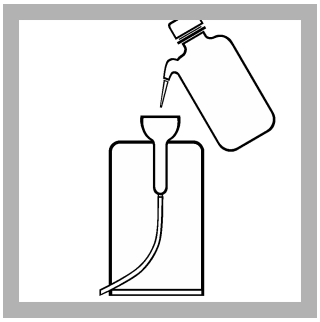


13. When the flow stops, push **ZERO**. The display shows 0 µg/L PO_4^{3-}.



14. Pour the contents of the Erlenmeyer flask that contains the prepared sample into the Pour-Thru Cell.

15. Push **READ**. Results show in µg/L PO_4^{3-}.



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

Prepare the reagent

Prepare the ascorbic acid reagent before use.

1. Open one 450-mL bottle of Ascorbic Acid Reagent Dilution Solution and put a powder funnel on the bottle. Pour the contents of one 48-g bottle of Ascorbic Acid Reagent Powder into the dilution solution.
2. Invert the bottle several times and swirl until the powder is completely dissolved.

A yellow color can develop with time, but the color does not have an effect on the results. This solution gives accurate results for a minimum of 1 month after preparation if kept at 20–25 °C (68–77 °F).

3. Write the date of preparation on the Ascorbic Acid Reagent bottle. Discard any remaining solution after 1 month.
4. Do not mix fresh reagent with previously prepared reagent. Use of this reagent after 1 month can result in high reagent blanks and low values at high concentration.

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 Molybdate 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 a 1:5 dilution of Ammonium Hydorxide 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

Interfering substance	Interference level
Aluminum	More than 200 mg/L
Arsenate	Interferes at any level
Chromium	More than 100 mg/L
Copper	More than 10 mg/L
Hydrogen Sulfide	Interferes at any level
Iron	More than 100 mg/L
Nickel	More than 300 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.
Silica	More than 50 mg/L
Silicate	More than 10 mg/L
Turbidity or color	Samples with a high amount of turbidity can give inconsistent results. The acid in the reagents can dissolve some of the suspended particles and variable desorption of orthophosphate from the particles can occur.
Zinc	More than 80 mg/L

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, 50-mg/L (50,000-µg/L) as PO_4^{3-} or 15-mg/L (15,000-µg/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:

- 1.000-mg/L (1000-µg/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
488	1000 µg/L PO_4^{3-}	970–1030 µg/L PO_4^{3-}	21 µg/L PO_4^{3-}

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphate/molybdate complex. Ascorbic acid then chemicaly reduces the complex, which makes an intense molybdenum blue color. Results include orthophosphate in the sample, plus a small fraction of condensed phosphate that can be hydrolyzed to orthophosphate

during the test. The measurement wavelength is 880 nm (710 nm in DR1900 instruments).

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Rapid Liquid Low Range Phosphorus Reagent Set, includes:	—	—	2678600
Ascorbic Acid Reagent Dilution Solution	1 mL	450 mL	2599949
Ascorbic Acid Reagent Powder	varies	48 g	2651255
Molybdate Reagent Solution	2 mL	500 mL	2599849
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, polypropylene, 25 mL	1	each	108140
Fixed volume micropipette, 1 mL	2	each	2946010
Flask, Erlenmeyer, Polymethylpentene, screw cap, 125 mL	2	each	2089843
Funnel, powder	1	each	2264467

Recommended standards

Description	Unit	Item no.
Drinking Water Standard, Mixed Parameter, Inorganic for F^-, NO_3–N, PO_4^{3-}, SO_4^{2-}	500 mL	2833049
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	256949
Phosphate Standard Solution, 3-mg/L as PO_4^{3-}	946 mL	2059716
Phosphate Standard Solution, 50-mg/L, 10-mL Voluette® Ampules	16/pkg	17110
Phosphate Standard Solution, 15-mg/L as PO_4^{3-}	100 mL	1424342
Wastewater Effluent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4^{3-}, COD, SO_4^{2-}, TOC	500 mL	2833249

Optional reagents and apparatus

Description	Unit	Item no.
Ammonium Hydroxide, 58%	500 mL	10649
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Sampling bottle, 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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