Phosphorus, Total

DOC316.53.01123

Molybdovanadate with Acid Persulfate Digestion Method[1] — Method 10127

1.0 to 100.0 mg/L PO_4^{3-} (HR) — Test 'N Tube™ Vials

Scope and application: For water and wastewater.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater, (4500 B-C).

Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows adapter and light shield requirements for the instruments that use them.

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

Table 1 Instrument-specific information for test tubes

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800, DR 2700	—	LZV646
DR 1900	9609900 (D[1])	—
DR 900	4846400	Cover supplied with the instrument

[1] The D adapter is not available with all instrument versions.

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
DR 3900, DR 3800, DR 2800 and DR 2700: Install the light shield in Cell Compartment #2 before this test is started.
Reagent blanks can be used more than once, but should not be used more than one day.
The reagent that is used in this test is corrosive. Use protection for eyes and skin and be prepared to flush any spills with running water.
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
Total High Range Phosphorus Test 'N Tube Reagent Set	1
DRB200 Reactor	1
Funnel, micro	1
Light shield or adapter (For information about sample cells, adapters or light shields, refer to Items to collect on page 1.)	1
Pipet, TenSette®, 1.0- to 10.0-mL, with pipet tips	1

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Items to collect (continued)

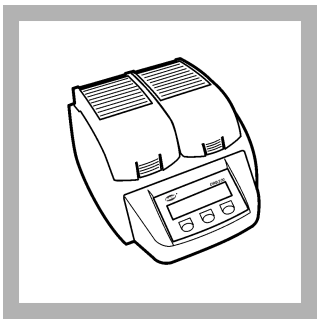
Description	Quantity
Test tube rack	1
Water, deionized	varies

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

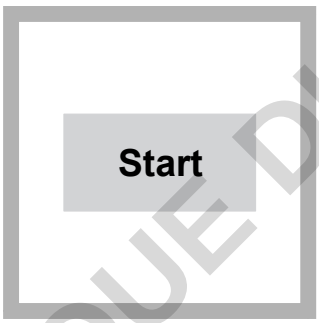
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 1:1 hydrochloric acid and rinsed with deionized water.
- Analyze the samples as soon as possible for best results.
- Do not use a detergent that contains phosphate 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 (about 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 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution caused by the volume additions.

Molybdovanadate method, acid persulfate digestion

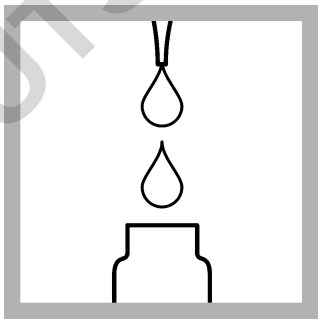


1. Start the DRB200 Reactor. Preheat to 150 °C. Refer to the DRB200 manual.

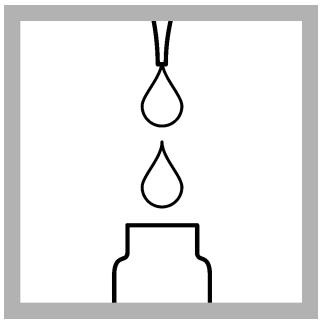


2. Start program **542 P Total HR TNT**. For information about sample cells, adapters or light shields, refer to Items to collect on page 1.

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

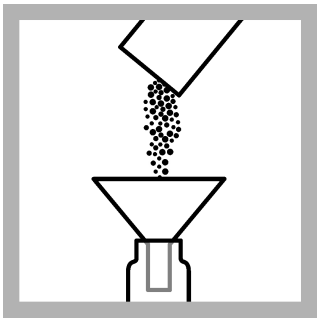


3. Prepare the blank: Add 5.0 mL of deionized water to a Total Phosphorus Test Vial.

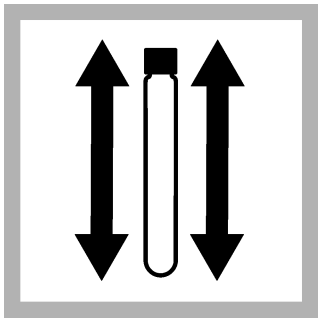


4. Prepare the sample: Add 5.0 mL of sample to a Total Phosphorus Test Vial.

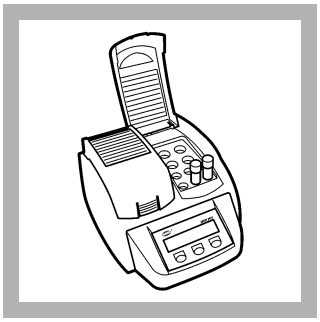
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5. Add the contents of one Potassium Persulfate Powder Pillow to each vial.



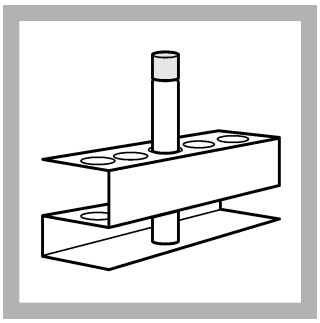
6. Put the cap on the vial. Shake to dissolve the powder.



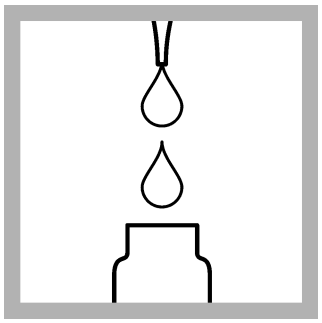
7. Insert the vial into the reactor. Close the reactor.



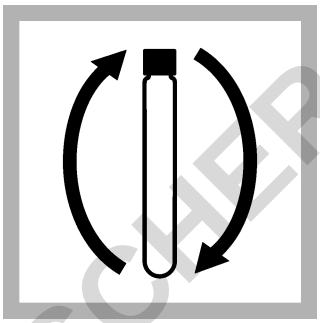
8. Start the instrument timer. A 30-minute reaction time starts.



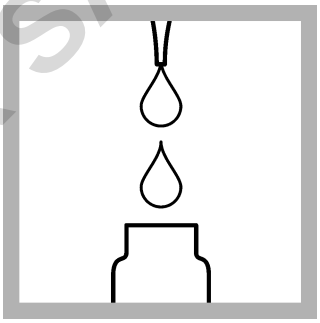
9. When the timer expires, carefully remove the hot vials from the reactor. Set the vials in a test tube rack. Let the vials cool to room temperature.



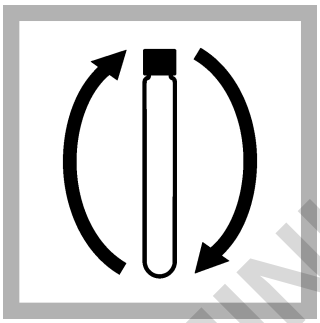
10. Add 2 mL of 1.54 N Sodium Hydroxide Standard Solution to each vial.



11. Put the cap on the vial. Invert to mix.



12. Use a polyethylene dropper to add 0.5 mL of Molybdovanadate Reagent to each vial.

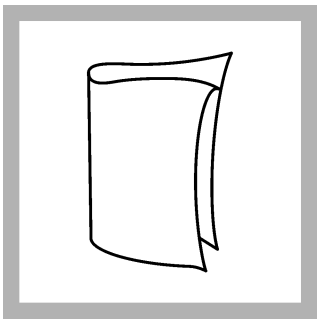


13. Put the cap on the vial. Invert to mix.

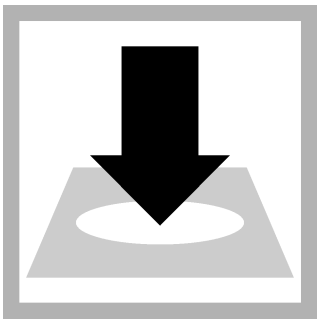


14. Start the instrument timer. A 7-minute reaction time starts.

Measure the sample between seven and nine minutes after the addition of the Molybdovanadate Reagent.

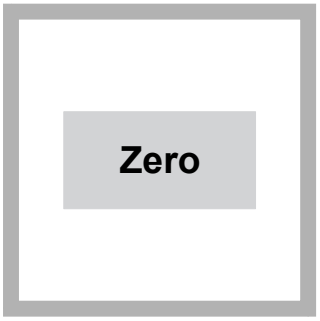


15. Clean the blank vial.

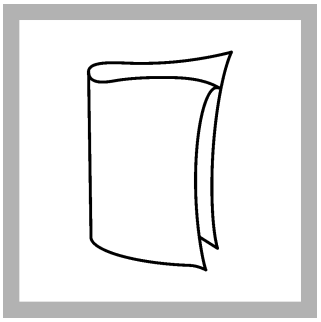


16. Insert the vial into the 16-mm cell holder.

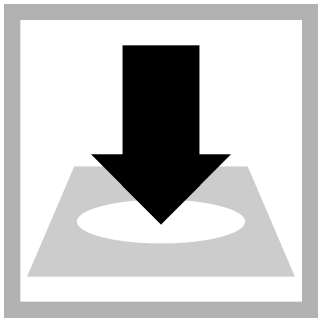
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17. Push **ZERO**. The display shows 0.0 mg/L PO_4^{3-}.



18. Clean the sample vial.



19. Insert the vial into the 16-mm cell holder.



20. Push **READ**. Results show in mg/L 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. Let the sample temperature decrease to room temperature after the digestion.
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. Let the sample temperature decrease to room temperature after the digestion.
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.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Negative interference
Turbidity	Sample turbidity can cause inconsistent results because the acid in the reagents can dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Temperature	Temperatures below 20 °C (68 °F) cause a negative interference. Temperatures above 25 °C (77 °F) cause a positive interference. Let the sample temperature decrease to room temperature after the digestion.

Table 3 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-}	—	—

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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 10-mL Ampule Standard, 500-mg/L as PO_4^{3-}
- Ampule breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips
- Mixing cylinders, 10-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 10-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:

- 50-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
542	50 mg/L PO_4^{3-}	49.4–50.6 mg/L PO_4^{3-}	0.7 mg/L PO_4^{3-}

Summary of method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphates by heating with acid and persulfate. Orthophosphate reacts with molybdate in an acid

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medium to produce a mixed phosphate/molybdate complex. In the presence of vanadium, yellow molybdovanadophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration. Test results are measured at 420 nm.

Pollution prevention and waste management

Reacted samples contain molybdenum and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Total High Range Phosphorus Test 'N Tube™ Reagent Set	—	50 vials	2767245
Includes:			
Molybdovanadate Reagent (not sold separately)	0.5 mL	25 mL	—
Potassium Persulfate Powder Pillow	1 pillow	50/pkg	2084766
Sodium Hydroxide, 1.54 N	varies	100 mL	2743042
Total Phosphorus Test Vials (not sold separately)	1	50/pkg	—
Water, deionized	varies	100 mL	27242

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 110 VAC option, 15 x 16-mm wells	1	each	LTV082.53.40001
DRB 200 Reactor, 220 VAC option, 15 x 16-mm wells	1	each	LTV082.52.40001
Dropper, measuring, 0.5-mL and 1.0-mL plastic	2	20/pkg	2124720
Funnel, micro, poly	1	each	2584335
Light shield, DR 3900	1	each	LZV849
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Pipet, TenSette®, 1.0–10.0 mL	1	each	1970010
Pipet Tips, for TenSette® Pipet, 1.0–10.0 mL	2	250/pkg	2199725
Test tube rack	1	each	1864100

Recommended standards and apparatus

Description	Unit	Item no.
Phosphate Standard Solution, 10-mL ampule, 500 mg/L as PO_4	16/pkg	1424210
Phosphate Standard Solution, 50-mg/L as PO_4^{3-}	500 mL	17149
Wastewater Influent Standard Solution, Mixed Parameter, for NH_3-N, NO_3-N, PO_4, COD, SO_4, TOC	500 mL	2833149
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800

Optional reagents and apparatus

Description	Unit	Item no.
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053
Sulfuric Acid, concentrated, ACS	500 mL	97949

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Optional reagents and apparatus (continued)

Description	Unit	Item no.
Molybdovanadate Reagent	100 mL	2076032
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
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
Water, deionized	4 L	27256
Thermometer, non-mercury, –10 to +225 °C	each	2635700
Finger cots	2/pkg	1464702
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Mixing cylinder, graduated, 25-mL	each	189640
Funnel, micro	each	2584335

Optional standards

Description	Unit	Item no.
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 Voluette® Ampule, 50-mg/L as PO_4	16/pkg	17110
Phosphate Standard Solution, 500-mg/L as PO_4	100 mL	1424232

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