Phosphorus, Reactive (Orthophosphate)

DOC316.53.01116

Molybdovanadate Method[1] **Method 8114**

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.

Test preparation

Instrument specific information

The table in this section shows all of the instruments that have the program for this test. Table 1 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 corresponding information for this test.

Table 1 Instrument-specific information for test tubes

Instrument	Adapters	Light shield
DR 6000	—	—
DR 5000	—	—
DR 900	4846400	Cover supplied with the instrument
DR 3900	—	LZV849
DR 3800	—	LZV646
DR 2800	—	

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.
The blank vial that is prepared in the test procedure can be used more than once. At room temperature, the reagent blank is stable for a maximum of three weeks. Prepare a new blank vial when a new lot of reagent is used.
The 7-minute reaction time in the test procedure is for samples that are at 23 °C (73 °F). If the sample temperature is 13 °C (55 °F), wait 15 minutes. If the sample temperature is 33 °C (91 °F), wait 2 minutes.
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 and use any recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Use the Safety Data Sheets for disposal information for unused reagents. Consult the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Description	Quantity
High Range Reactve Phosphorus Test 'N Tube Vials	1
Light shield or adapter (For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.)	1

Items to collect (continued)

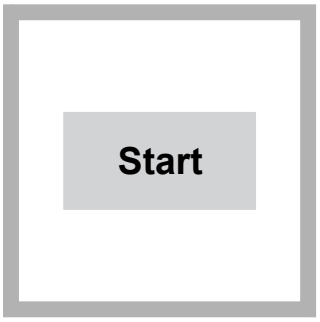
Description	Quantity
Pipet, TenSette®, 1.0- to 10.0-mL, with pipet tips	1
Test tube rack	1
Water, deionized	5 mL

Refer to Consumables and replacement items on page 5 for reorder information.

Sample collection and storage

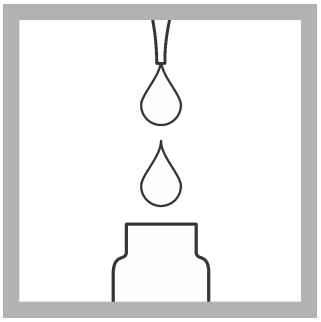
- Collect samples in clean glass or plastic bottles that have been cleaned with 1:1 hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples as soon as possible for best results.
- If prompt 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.

Molybdovanadate method for Test 'N Tubes

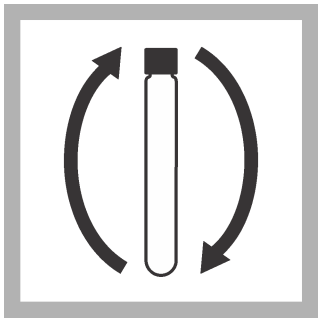


1. Start program **540 P React. HR TNT**. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

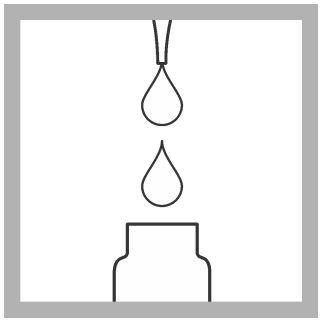
Note: *Although the program name may vary between instruments, the program number does not change.*



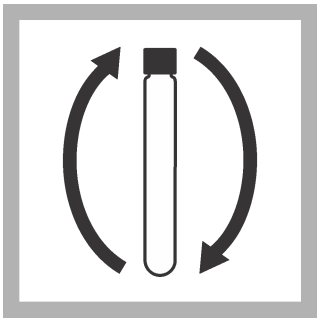
2. Prepare the blank: Add 5.0 mL of deionized water to a Reactive High Range Phosphorus Test 'N Tube Vial.



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



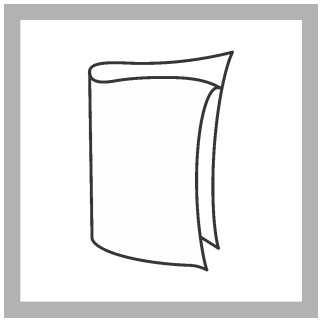
4. Prepare the sample: Add 5.0 mL of sample to a second Reactive High Range Phosphorus Test 'N Tube Vial.



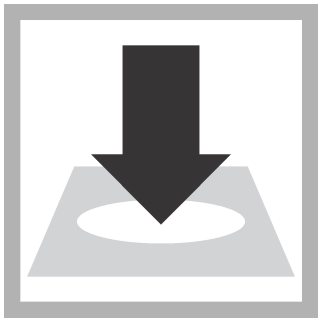
5. Put the cap on the vial. Invert to mix.

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

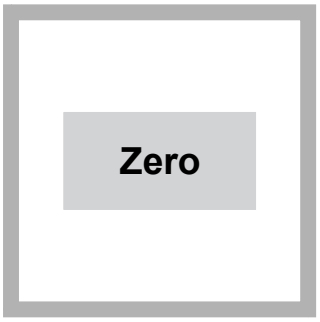
Measure the prepared sample within two minutes after the timer expires.



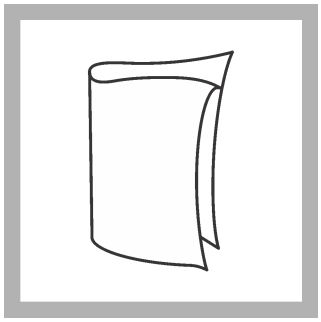
7. When the timer expires, clean the blank.



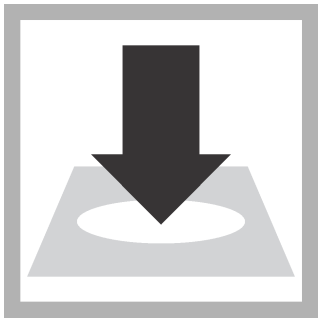
8. Insert the blank into the cell holder.



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



10. Clean the sample vial.



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

12. 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. 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	Causes a negative interference at more than 1000 mg/L.
Silica	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.
Sulfide	Causes a negative interference. Correct for this interference as follows: **1.** Measure 50 mL of sample into an Erlenmeyer flask. **2.** Add Bromine Water drop-wise with constant swirling until a permanent yellow color remains. **3.** Add Phenol Solution drop-wise 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 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	Causes a negative interference.
Temperature	Temperatures below 18 °C (64 °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^{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:

- 10-mL Voluette® Ampule of Phosphate Standard Solution, 500-mg/L 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, reagents and instrument.

Items to collect:

- 50-mg/L PO_4^{3-} 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 may get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
540	50.0 mg/L PO_4^{3-}	49.1–50.9 mg/L PO_4^{3-}	0.7 mg/L PO_4^{3-}

Summary of method

Orthophosphate reacts with molybdate in an acid 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.
High Range Reactive Phosphorus Test 'N Tube™ Reagent Set	—	50 vials	2767345
Includes:			
Reactive High Range Phosphorus Test 'N Tube Vials (not sold separately)	1	50/pkg	—
Water, deionized	varies	100 mL	27242

Required apparatus

Description	Quantity/test	Unit	Item no.
Pipet, TenSette®, 1.0- to 10.0-mL	1	each	1970010
Pipet Tips, for TenSette Pipet 1970010	varies	50/pkg	2199796
Test tube rack	1	each	1864100

Recommended standards and apparatus

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

Optional reagents and apparatus

Description	Unit	Item no.
Silica Standard Solution, 1-mg/L SiO_2	500 mL	110649
Bromine Water, 30 g/L	29 mL	221120
Cylinder, mixing, 25-mL	each	189640
Hydrochloric Acid, 6.0 N 1:1, 50%	500 mL	88449
Phenol Solution, 30-g/L	29 mL	211220
Dropper, LDPE, 0.5 –1.0 mL	20/pkg	2124720
Pipet tips for TenSette Pipet 1970010	250/pkg	2199725
Paper, pH, 0–14 pH range	100/pkg	2601300
Thermometer, non-mercury, -10 to +225 °C	each	2635700
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Filter paper, folded, 12.5-cm	100/pkg	69257
Funnel, poly, 65-mm	each	108367
Beaker, 50-mL	each	50041H
Finger cots	2/pkg	1464702

Optional standards

Description	Unit	Item no.
Phosphate Standard Solution, 3-mg/L as PO_4	946 mL	2059716
Phosphate Standard Solution, 10-mg/L as PO_4	946 mL	1420416
Phosphate Standard Solution, 15-mg/L as PO_4	100 mL	1424342
Phosphate Standard Solution, 30-mg/L as PO_4	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, 10-mL Ampule, 500 mg/L as PO_4	16/pkg	1424210
Phosphate Standard Solution, 500-mg/L as PO_4	100 mL	1424232

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