

Phosphorus, Reactive (Orthophosphate) and Total

DOC316.53.01125

Ascorbic Acid Method**Method 10209/10210****1.5 to 15.0 mg/L PO₄³⁻ or 0.5 to 5.0 mg/L PO₄-P (HR)****TNTplus™ 844****Scope and application:** For wastewater, drinking water, boiler water, surface water and process analysis.

Test preparation

Instrument-specific information

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

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

Table 1 Instrument-specific information for TNTplus vials

| Instrument | Adapters | Light shield |
|------------------|------------------------|--------------|
| DR 6000, DR 5000 | — | — |
| DR 3900 | — | LZV849 |
| DR 3800, DR 2800 | — | LZV646 |
| DR 1900 | 9609900 or 9609800 (A) | — |

Before starting

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before this test is started.

Review the safety information and the expiration date on the package.

The recommended sample pH is 2–10.

The recommended temperature for samples and reagents is 15–25 °C (59–77 °F).

The recommended temperature for reagent storage is 15–25 °C (59–77 °F).

The reagents that are used in this test contain molybdenum and are corrosive. Collect the reacted samples for proper disposal.

Use the DRB reactor with 13-mm wells for the digestion. If the reactor has 16-mm wells, insert adapter sleeves into the wells.

DR 1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.

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 |
|---|----------|
| Phosphorus, Reactive and Total HR TNTplus Reagent Set | 1 |
| DRB200 reactor with 13-mm wells | 1 |

Items to collect (continued)

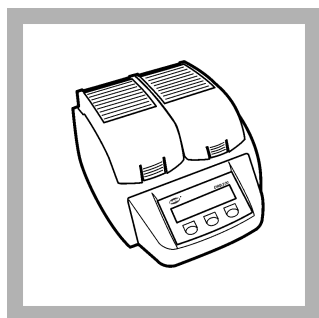
| Description | Quantity |
|--------------------------------------|----------|
| Pipet, adjustable volume, 0.2–1.0 mL | 1 |
| Pipet tips, for 0.2–1.0 mL pipet | 1 |
| Test tube rack | 1 |

Refer to [Consumables and replacement items](#) on page 7 for order information.

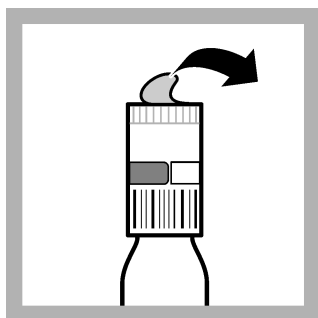
Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- 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 2 or less with concentrated sulfuric acid (approximately 2 mL per liter). Do not acidify samples to be analyzed only for reactive phosphorus. 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 (reactive phosphorus only: 48 hours).
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Test procedure—total phosphorus



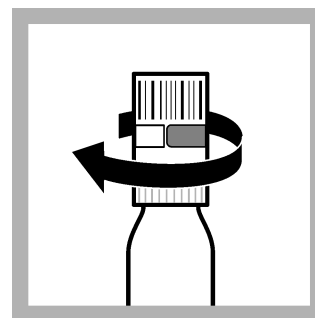
1. Set the DRB200 reactor power to on. Set the temperature to 100 °C.



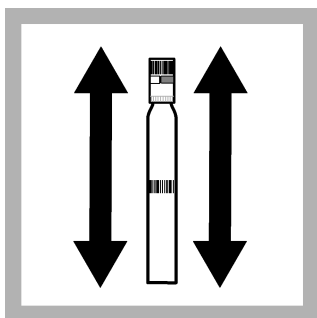
2. Carefully remove the lid from the DosiCap™ Zip cap. Remove the cap from the test vial.



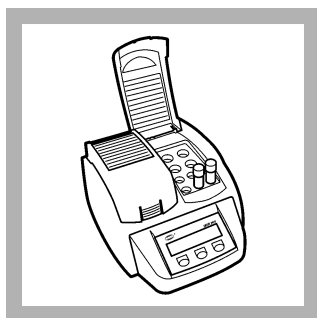
3. Use a pipet to add 0.5 mL of sample to the test vial.



4. Turn the DosiCap Zip over so that the reagent side goes on the test vial. Tighten the cap on the vial.



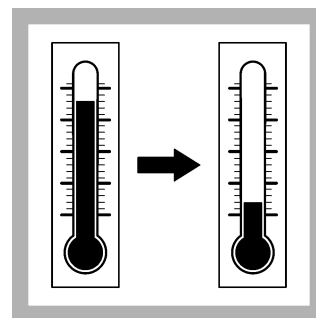
5. Shake the vial 2–3 times to dissolve the reagent in the cap.
Look through the open end of the DosiCap to make sure that the reagent has dissolved.



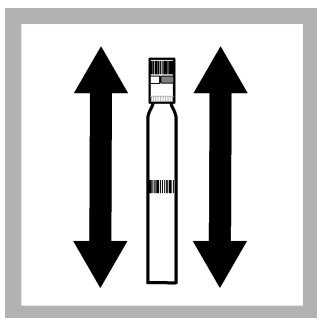
6. Insert the vial in the preheated DRB200 reactor. Close the lid.



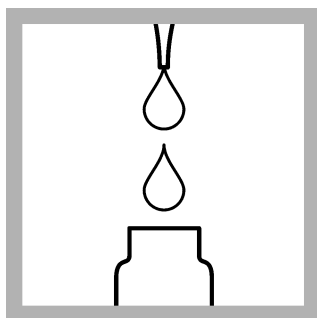
7. Keep the vial in the reactor for 1 hour.



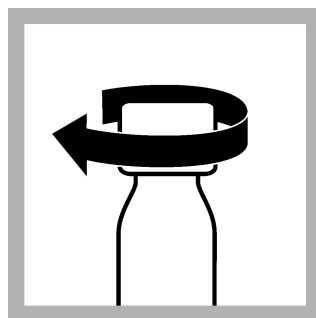
8. When the timer expires, carefully remove the vial from the reactor. Set the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



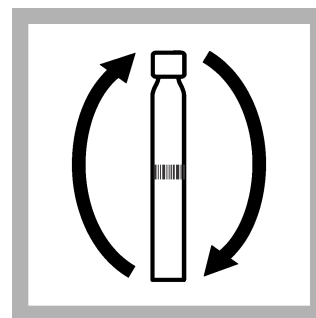
9. Shake the vial 2–3 times.



10. Use a pipet to add 0.2 mL of Solution B to the test vial. Immediately tighten the cap on the Solution B container.



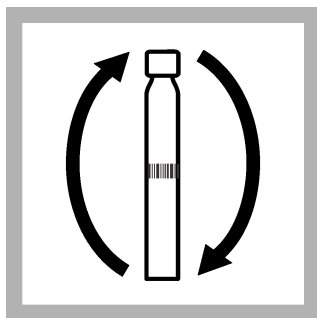
11. Put a grey DosiCap C on the vial.



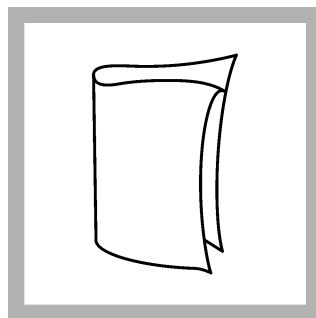
12. Tighten the cap on the vial and invert the vial 2–3 times.



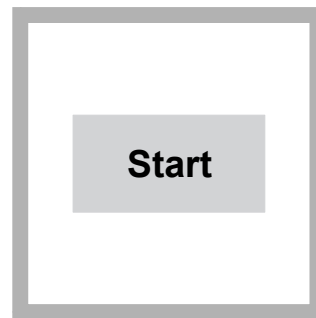
13. Start the reaction time of 10 minutes.



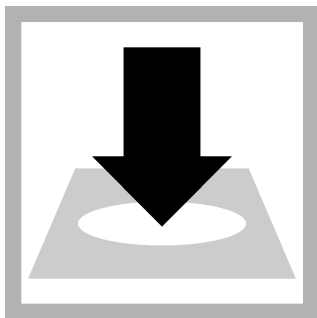
14. When the timer expires, invert the vial 2–3 times.



15. Clean the vial.

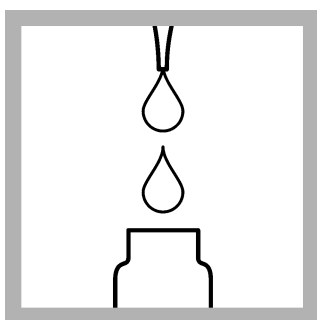


16. DR 1900 only: Select program 844. Refer to [Before starting](#) on page 1.

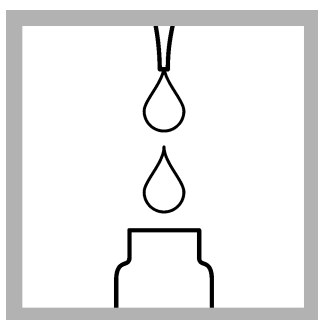


17. Insert the vial into the cell holder. DR 1900 only: Push **READ**. Results show in mg/L PO_4^{3-} .

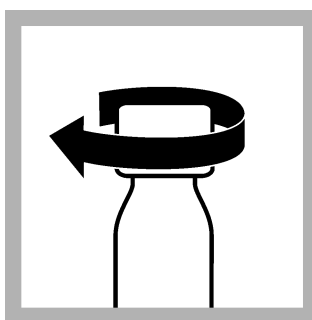
Test procedure—reactive phosphorus



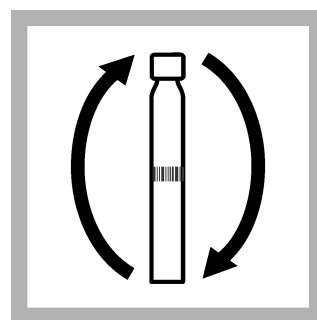
1. Use a pipet to add 0.5 mL of sample to the test vial.



2. Use a pipet to add 0.2 mL of Solution B to the test vial. Immediately tighten the cap on the Solution B container.



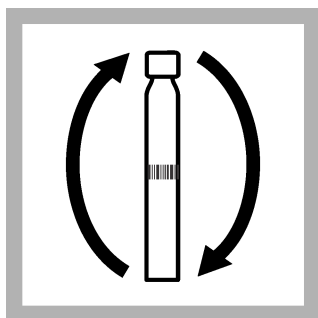
3. Put a grey DosiCap C on the vial.



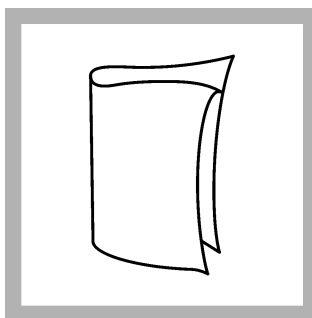
4. Tighten the cap on the vial and invert the vial 2–3 times.



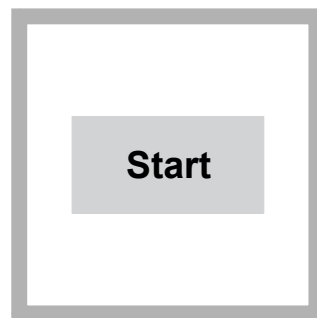
5. Start the reaction time of 10 minutes.



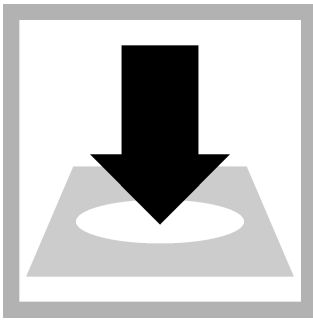
6. When the timer expires, invert the vial 2–3 times.



7. Clean the vial.



8. DR 1900 only: Select program 844. Refer to [Before starting](#) on page 1.



9. Insert the vial into the cell holder. DR 1900 only: Push

READ.

Results show in mg/L
PO₄³⁻.

Reagent blank correction

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. Measure the reagent blank value when a new lot of reagent is used.

1. Use deionized water as the sample in the test procedure to measure the reagent blank value.
2. Set the reagent blank function to on. The measured reagent blank value is shown.
3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.

Note: As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.

Sample blanks

Samples with color or turbidity can cause high results. Samples without color or turbidity do not require sample blanks. The digestion in the total phosphate test procedure usually removes all color and turbidity. A sample blank is not required. To adjust for color or turbidity in the reactive phosphate test procedure, use the steps that follow to find the sample blank.

1. Do the test procedure, but do not add the DosiCap C.
2. Put the cap on the vial, but do not remove the foil. Use the side of the cap that does not have the reagent.
3. Subtract the value from the final procedure step from the initial sample value to get the corrected sample concentration.

Note: Alternatively, samples that contain only turbidity can be filtered through a membrane filter, then analyzed.

Interferences

Table 2 shows that the ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found. Verify the measurement results with sample dilutions or standard additions.

Table 2 Interfering substances

| Interfering substance | Interference level |
|-------------------------------|--------------------|
| SO ₄ ²⁻ | 20 g/L |
| Cl ⁻ | 10 g/L |
| Ca ²⁺ | 1000 mg/L |

Table 2 Interfering substances (continued)

| Interfering substance | Interference level |
|--|--------------------|
| K ⁺ , Na ⁺ | 4000 mg/L |
| NO ₃ ⁻ | 500 mg/L |
| Mg ²⁺ | 400 mg/L |
| Co ²⁺ , Fe ²⁺ , Fe ³⁺ , Zn ²⁺ , Cu ²⁺ , Ni ²⁺ , NO ₂ ⁻ , Cd ²⁺ , NH ₄ ⁺ , Mn ²⁺ , Al ³⁺ , CO ₃ ²⁻ | 200 mg/L |
| I ⁻ | 100 mg/L |
| SiO ₂ | 50 mg/L |
| Hg ²⁺ | 40 mg/L |
| Pb ²⁺ | 20 mg/L |
| Ag ⁺ , Sn ⁴⁺ | 10 mg/L |
| Cr ³⁺ | 5 mg/L |
| Cr ⁶⁺ | 1 mg/L |

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Phosphate Standard Solution, 10-mg/L PO₄³⁻ or Wastewater Influent Standard Solution, Mixed Parameter (contains 10-mg/L PO₄³⁻)

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 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 |
|---------|---|---|--|
| barcode | 10.0 mg/L PO ₄ ³⁻ | 9.5–10.5 mg/L PO ₄ ³⁻ | — |

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates in the total phosphorus procedure by heating with acid and persulfate. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample. The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. The measurement wavelength is 880 nm (DR 1900: 714 nm).

Consumables and replacement items

Required reagents

| Description | Quantity/Test | Unit | Item no. |
|---|---------------|--------|----------|
| Phosphorus, Reactive and Total HR TNTplus Reagent Set | 1 | 25/pkg | TNT844 |

Required apparatus

| Description | Quantity/test | Unit | Item no. |
|---|---------------|---------|-----------|
| DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block | 1 | each | DRB200-01 |
| DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block | 1 | each | DRB200-05 |
| Pipet, adjustable volume, 0.2–1.0 mL | 1 | each | BBP078 |
| Pipet tips, for 0.2–1.0 mL pipet | 2 | 100/pkg | BBP079 |
| Test tube rack | 1 | each | 1864100 |
| Light shield, DR 3800, DR 2800, DR 2700 | 1 | each | LZV646 |
| Light shield, DR 3900 | 1 | each | LZV849 |

Recommended standards

| Description | Unit | Item no. |
|--|--------|----------|
| Phosphate Standard Solution, 10-mg/L as PO_4^{3-} | 946 mL | 1420416 |
| Wastewater Influent Standard Solution, Mixed Parameter, for $\text{NH}_3\text{-N}$, $\text{NO}_3\text{-N}$, PO_4 , COD, SO_4 , TOC | 500 mL | 2833149 |

Optional reagents and apparatus

| Description | Unit | Item no. |
|---|------------|----------|
| Filter membrane, 0.45-micron, 25-mm | 100/pkg | 2514101 |
| Flask, volumetric, Class A, 1000 mL glass | each | 1457453 |
| Hydrochloric Acid Solution, 6.0 N (1:1) | 500 mL | 88449 |
| Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials | 5/pkg | 2895805 |
| Sampling bottle with cap, low density polyethylene, 500-mL | 12/pkg | 2087079 |
| Sodium Hydroxide Standard Solution, 5.0 N | 100 mL MDB | 245032 |
| Sulfuric Acid, concentrated, ACS | 500 mL | 97949 |



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