ULR Phosphorus, Reactive (Orthophosphate) and Total

DOC316.53.01321

Ascorbic Acid Method **Method 10209 Reactive and 10210 Total**

30 to 1500 µg/L PO_4^{3-}, 10 to 500 µg/L PO_4–P or 46 to 2300 µg/L P_2O_5 **TNTplus™ 843**

Scope and application: For wastewater, drinking water, boiler water, surface water and process water.

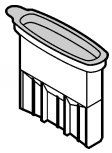
Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows requirements that can change between instruments, such as adapter and sample cell 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	Adapter	Sample cell orientation	50-mm sample cell
DR 6000	LZV902.99.00020	The thin side is to the right.	LZP341 
DR 5000	A23618	The thin side is toward the user.	
DR 3900, DR3800, DR 2800, DR 2700	—	The thin side is to the right.	

Before starting

Use the stored program with the DR 3900 and DR 6000. For other instruments, refer to the application note *Ultra Low Range Total and Reactive Phosphorus (Lit. No. 2097)* for calibration information.
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 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.
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 LR TNTplus 843 Reagent Set	1
DRB200 reactor with 13-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1

Items to collect (continued)

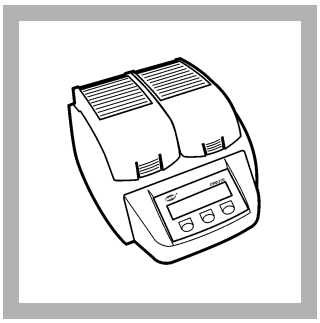
Description	Quantity
Pipet, adjustable volume, 0.2–1.0 mL	1
Pipet tips	1
Test tube rack	1

Refer to Consumables and replacement items on page 7 for order information.

Sample collection and storage

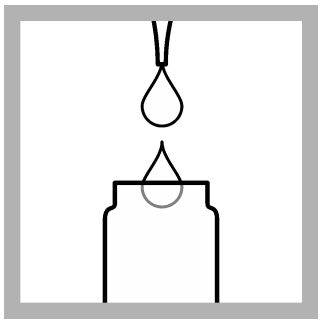
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Analyze the samples as soon as possible for best results.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent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

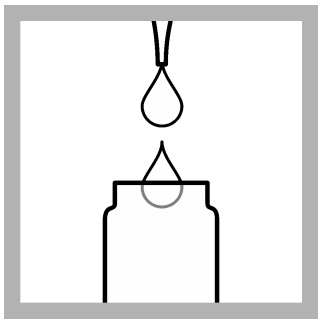


1. Set the DRB200 reactor power to on. Set the temperature to 100 °C.

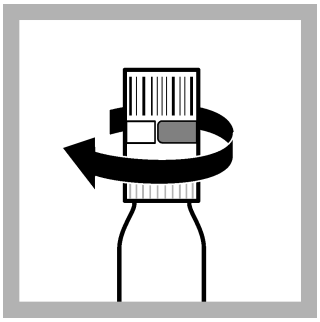
2. Carefully remove the lids from the DosiCap™ Zip caps. Remove the caps from the test vials.



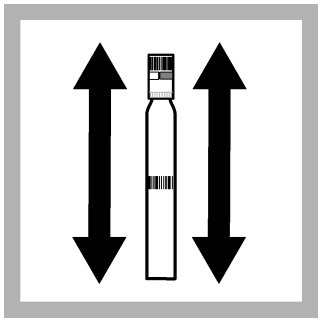
3. Prepare the blank: Use a pipet to add 3.5 mL of deionized water into one test vial.



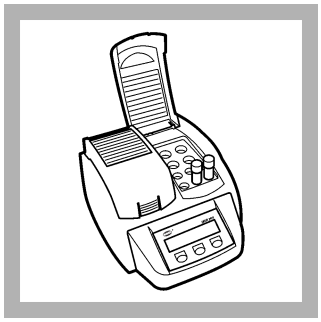
4. Prepare the sample: Use a pipet to add 3.5 mL of sample into the other test vial.



5. Turn the DosiCap Zip over the test vial so that the reagent side goes on the vial. Do this for each test vial, then tighten the caps on the vials.

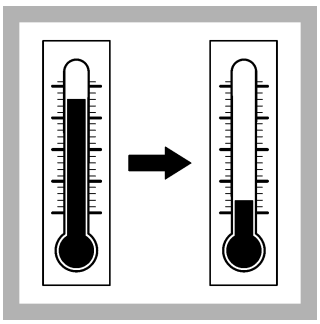


6. Shake each vial 2–3 times to dissolve the reagent in the cap. Look through the open end of the DosiCap to make sure that the reagent has dissolved.

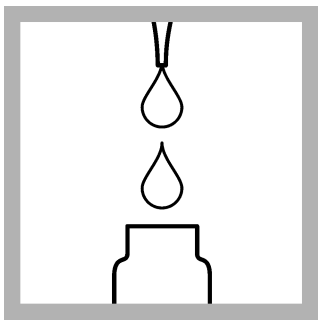


7. Insert the vials in the preheated DRB200 reactor. Close the lid.

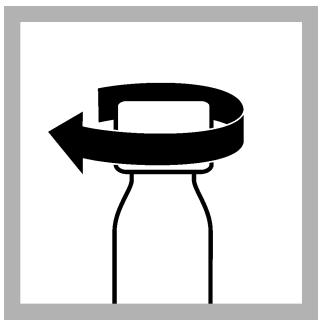
8. Keep the vials in the reactor for 1 hour.



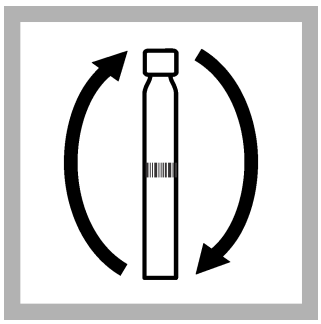
9. When the timer expires, carefully remove the vials from the reactor. Set the vials in a test tube rack. Let the temperature of the vials decrease to room temperature.



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

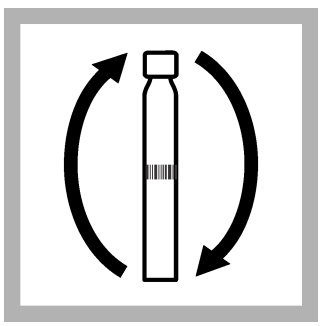


11. Put a grey DosiCap C on each vial.

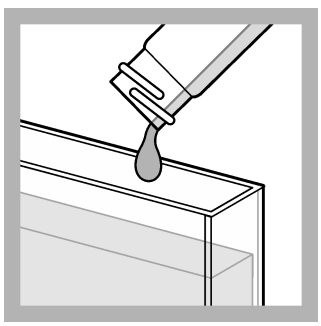


12. Tighten the cap on the vials and invert the vials 2–3 times.

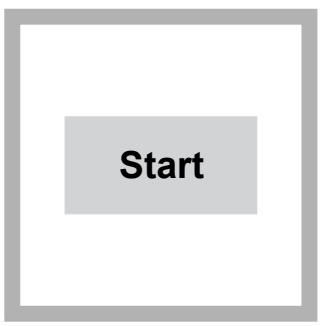
13. Start the reaction time of 10 minutes.



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

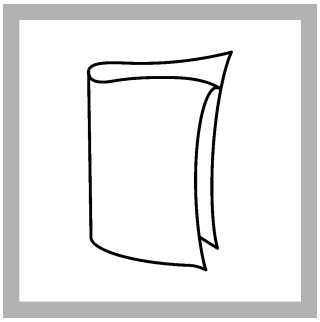


15. Pour the digested blank into the sample cell.

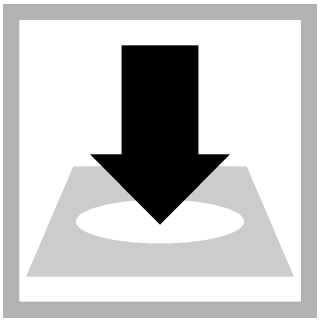


16. Start program **538 ULR Phosphate**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

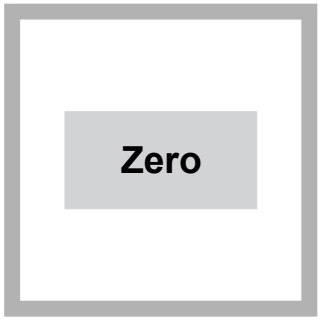
Note: *Although the program name can be different between instruments, the program number does not change.*



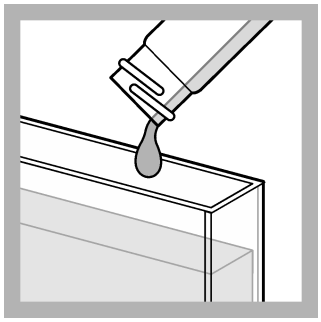
17. Clean the blank sample cell.



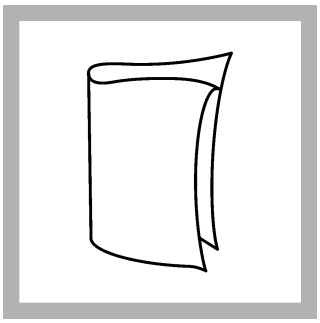
18. Insert the blank into the cell holder.



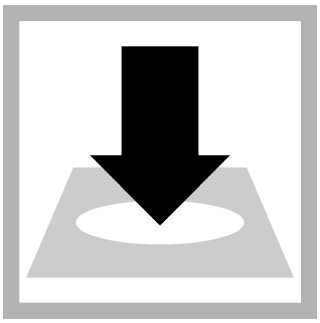
19. Push **ZERO**. The display shows 0 µg/L PO_4.



20. Pour the digested sample into a second sample cell.



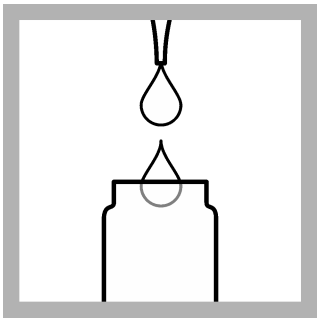
21. Clean the sample cell.



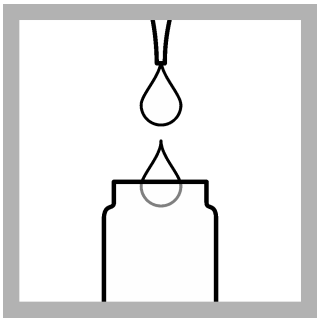
22. Insert the sample cell into the cell holder.

23. Push **READ**. Results show in µg/L PO_4.

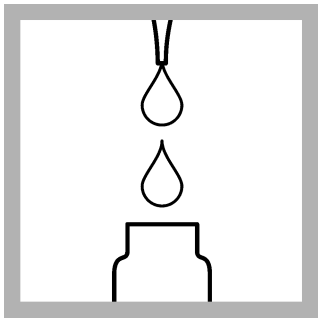
Test procedure—Reactive phosphorus



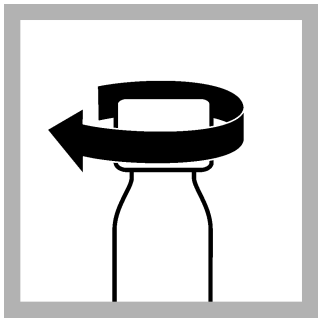
1. Prepare the blank: Use a pipet to add 3.5 mL of deionized water into one test vial.



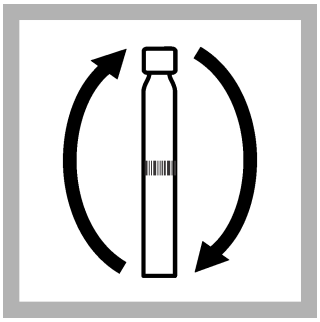
2. Prepare the sample: Use a pipet to add 3.5 mL of sample into the other test vial.



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

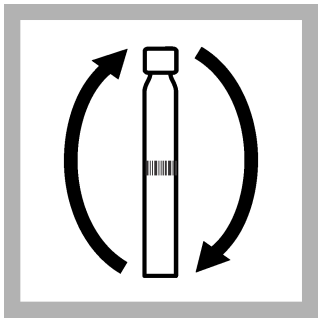


4. Put a grey DosiCap C on each vial.

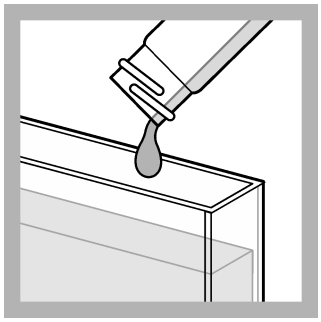


5. Tighten the cap on the vials and invert the vials 2–3 times.

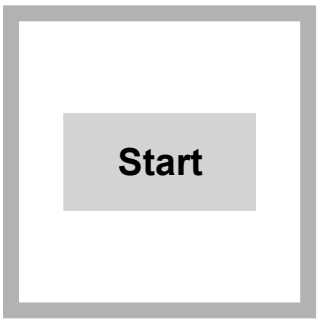
6. Start the reaction time of 10 minutes.



7. When the timer expires, invert the vials 2–3 times.

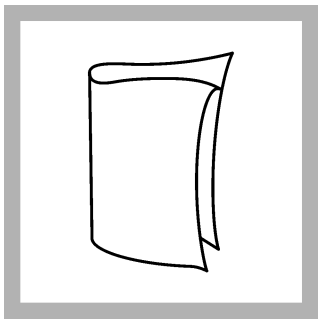


8. Pour the blank into the sample cell.

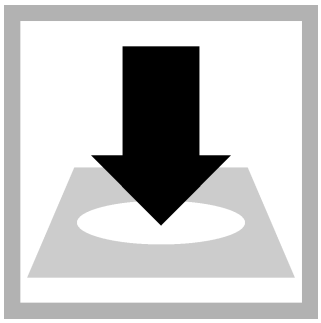


9. Start program **538 ULR Phosphate**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

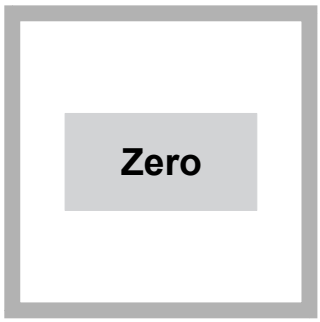
Note: *Although the program name can be different between instruments, the program number does not change.*



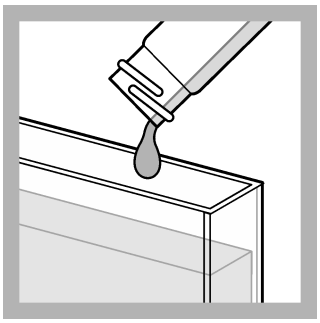
10. Clean the blank sample cell.



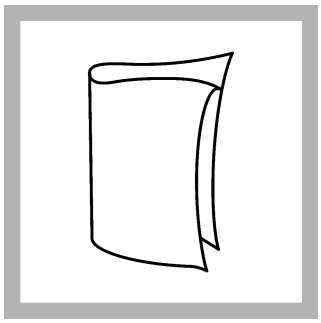
11. Insert the blank into the cell holder.



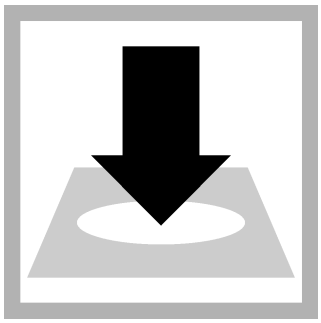
12. Push **ZERO**. The display shows 0 µg/L PO_4.



13. Pour the sample into a second sample cell.



14. Clean the sample cell.



15. Insert the sample cell into the cell holder.

16. Push **READ**. Results show in µg/L PO_4.

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-}	5000 mg/L
Cl^-	2000 mg/L
K^+, Na^+	1000 mg/L
NO_3^-	500 mg/L
Ca^{2+}	250 mg/L
Mg^{2+}	100 mg/L
CO_3^{2-}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+. Mn^{2+}, Al^{3+}, SiO_2	50 mg/L
Sn^{4+}, Hg^{2+}	5 mg/L
Ag^+, Pb^{2+}	2.5 mg/L
Cr^{3+}	1 mg/L
Cr^{6+}	0.5 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, 1000-µg/L (1-mg/L) as PO_4^{3-} (326 µg/L as PO_4–P)

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
538	200 µg/L PO_4–P	197.3–202.7 µg/L PO_4–P	3.01 µg/L PO_4–P

Summary of method

The total phosphorus digestion procedure uses acid, persulfate and heat to change organic and condensed inorganic phosphates (meta-, pyro- or other polyphosphates) to reactive orthophosphate. Then the reactive phosphorus measurement procedure measures the concentration of orthophosphate in the sample. The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex. The antimonyl phosphomolybdate complex is reduced by ascorbic acid to phosphomolybdenum blue.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Phosphorus, Reactive and Total LR TNTplus Reagent Set	1	25/pkg	TNT843

Required apparatus

Description	Quantity/test	Unit	Item no.
Cuvette, semi-micro, 5-cm	varies	10/pkg	LZP341
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20001
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20005
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Test tube rack	1	each	1864100

Recommended standards

Description	Unit	Item no.
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	256949

Optional reagents and apparatus

Description	Unit	Item no.
Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials	5/pkg	2895805
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Finger cots	2/pkg	1464702
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Pipet, serological, 2-mL	each	53236
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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