

phosphate GenGard* GN8000 series

PhosVer 3 Persulfate UV Oxidation Method

0.5 to 25.0 mg/L Orthophosphate, Orthophosphate + Phosphonate and Phosphonate as PO_4^{3-}

scope and application: For water.




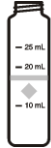
Test preparation

instrument specific information

Table 1 shows the sample cell and cell orientation requirements for the SUEZ instruments that can use this Analytical Procedure (AP).

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

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 2800 DR 1900	The fill line is to the right.	L2793.0002 
DR 900	The fill lines and diamond mark are toward the display.	L1976 

Find a contact near you by visiting www.suezwatertechnologies.com and clicking on "Contact Us."

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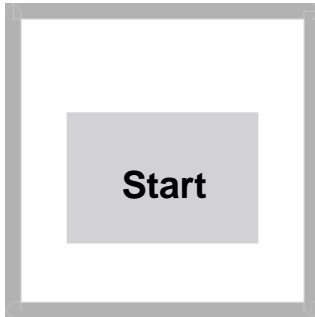
before starting

Clean all glassware with 6.0 N (50%) hydrochloric acid, then fully rinse with deionized water to remove contaminants.
Do not use a detergent that contains phosphate to clean glassware. The phosphate in the detergent will contaminate the sample.
Wear UV safety goggles while the UV lamp is on.
Do not touch the UV lamp surface with bare fingers. Fingerprints can damage the glass. Rinse the lamp and wipe with a soft, clean tissue between tests.
The UV digestion in this procedure is typically complete in less than 10 minutes. However, high-organic loaded samples or a weak lamp can cause incomplete phosphate conversion. To check conversion efficiency, use a longer digestion time and make sure that the readings do not increase.
Two UV lamps can connect to a single power supply with a cord adapter for digestion of two samples at the same time. A second UV lamp is necessary.
Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
Refer to the instrument user manual for timer operation instructions.
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.
Clean the external surface of the sample cells before insertion into the instrument cell holder. Use a damp towel and then a dry towel to remove fingerprints or other marks.
Highly buffered samples or extreme pH may exceed the buffering capacity of the reagent and require sample pre-treatment.
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.
Results that are not within the working range of this procedure are not valid.

sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (50%) hydrochloric acid and rinsed with deionized water.
- Do not use a commercial detergent 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 24 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.

procedure

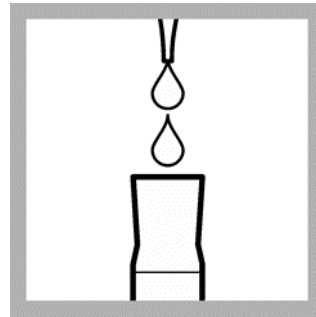


1. Start program 804 Phosphate GN8000. For information about sample cells, adapters or light shields, refer to Instrument specific information on page 1.

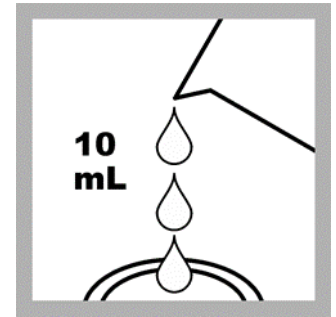
Note: Although the program name can be different between instruments, the program number does not change.



2. Use a syringe to push at least 25 mL of sample through a 0.22-micron filter membrane apparatus.



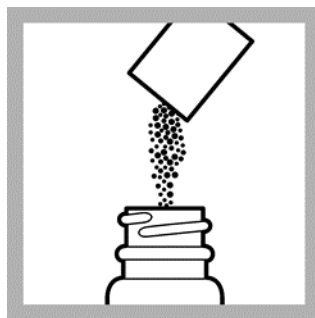
3. Use a pipet to add 5 mL of filtered sample into a 50-mL graduated mixing cylinder. Dilute the sample to 50 mL with deionized water and mix well.



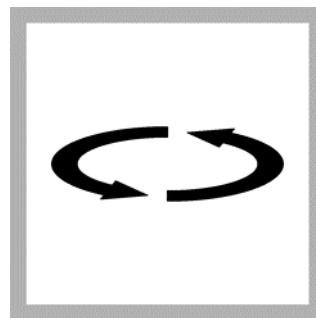
4. Fill two sample cells to the 10-mL mark with the diluted sample from step 3. Label one cell as the blank and the other cell as o-PO_4^{3-} .



5. Prepare the digested sample: Fill a mixing bottle to the 25-mL mark with the diluted sample from step 3.



6. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the 25-mL sample.



7. Swirl to mix.



8. Put on UV safety goggles.



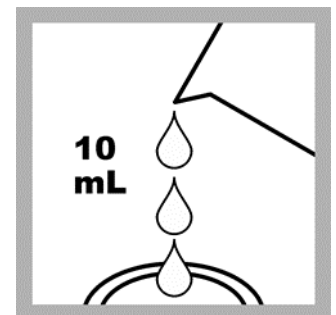
9. Put the ultraviolet lamp into the mixing bottle. Turn on the UV lamp. Make sure that only the glass portion of the lamp is in contact with the sample.



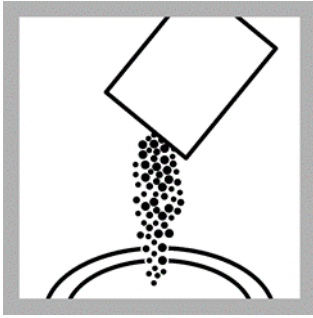
10. Start the instrument timer. A 10-minute reaction time starts. Phosphonates are converted to orthophosphate in this step.



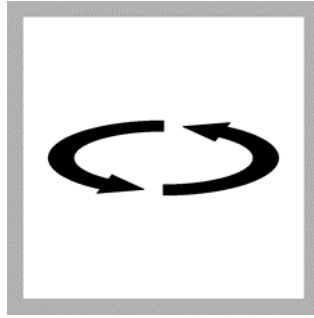
11. When the timer expires, turn off the UV lamp. Remove the UV lamp from the sample.



12. Fill a third sample cell to the 10-mL mark with the digested sample. Label this cell as $\text{(o-PO}_4 + \text{Phosphonate)}$.



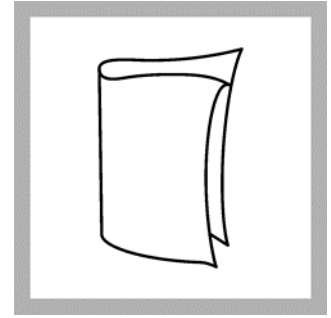
13. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to both the $o\text{-PO}_4$ and ($o\text{-PO}_4$ + Phosphonate) labeled cells.



14. Immediately swirl both cells vigorously for 20–30 seconds to mix. Some powder may not dissolve. A blue color shows if phosphate is present. Both sample cells may show color.



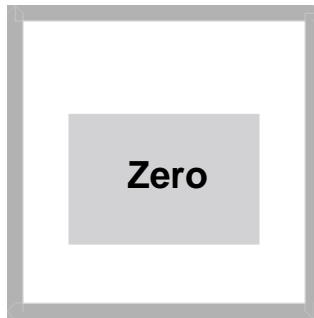
15. Start the instrument timer. A 2-minute reaction time starts. If the sample is colder than 15 °C, wait 4 minutes for color development.



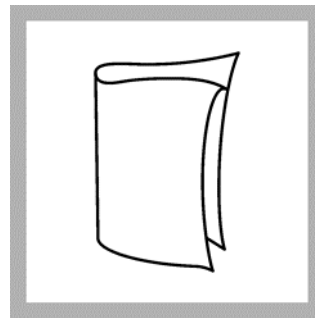
16. When the timer expires, clean the blank. Complete the rest of the steps in this procedure within 3 minutes.



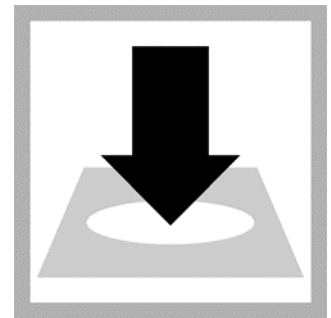
17. Insert the blank into the cell holder.



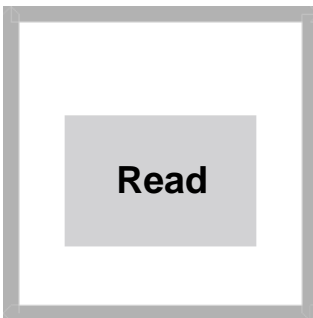
18. Push **ZERO**. The display shows 0.00 mg/L PO_4^{3-} .



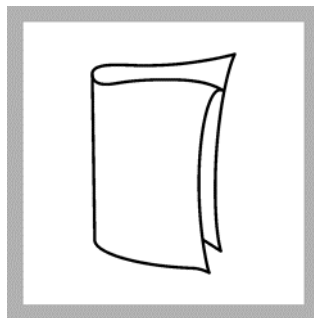
19. Clean the cell labeled $o\text{-PO}_4^{3-}$.



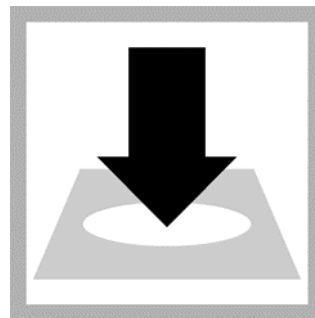
20. Insert the $o\text{-PO}_4^{3-}$ cell into the cell holder.



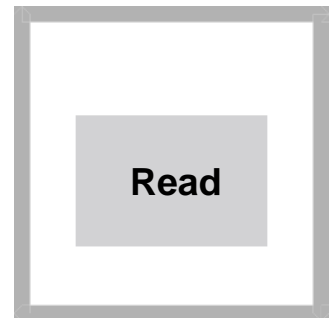
21. Push **READ**. Results show in mg/L PO_4^{3-} . The $o\text{-PO}_4^{3-}$ measured in this step is due to the $o\text{-PO}_4^{3-}$ in the make-up water and reversion of the fed phosphonate.



22. Clean the cell labeled $o\text{-PO}_4$ + Phosphonate.



23. Insert the $o\text{-PO}_4$ + Phosphonate cell into the cell holder.



24. Push **READ**. Results show in mg/L PO_4^{3-} . The $o\text{-PO}_4$ + phosphonate result should be equal to or greater than the $o\text{-PO}_4$ result. If it is not, repeat the test. The phosphonate concentration (that which has not reverted) in mg/L PO_4 = step 24 – step 21.

interferences

Interfering substance	Interference level (5-mL sample)
Aluminum	100 mg/L
Arsenate	Interferes at all levels
Benzotriazole	10 mg/L
Bicarbonate	1000 mg/L
Bromide	100 mg/L
Calcium	5000 mg/L
CDTA	100 mg/L
Chloride	5000 mg/L
Chromate	100 mg/L
Copper	100 mg/L
Cyanide	100 mg/L (Increase the UV digestion to 30 minutes.)
Diethanoldithiocarbamate	50 mg/L
EDTA	100 mg/L
Iron	200 mg/L
Nitrate	200 mg/L
NTA	250 mg/L
Orthophosphate	15 mg/L
Phosphites and organophosphorus compounds	Reacts quantitatively. Metaphosphates and polyphosphates do not interfere.
Silica	500 mg/L
Silicate	100 mg/L
Sulfate	2000 mg/L
Sulfide	Interferes at all levels
Sulfite	100 mg/L
Thiourea	10 mg/L
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.

accuracy check

standard solution method

The accuracy of the ortho-phosphate portion of the test or of the entire procedure (including the digestion) can be tested.

- **o-PO₄³⁻**—use 25 mL of Phosphate Standard Solution, 10 mg/L as PO₄³⁻, instead of the sample in the test procedure. The result should be 10 mg/L as PO₄³⁻.
- **Entire procedure (o-PO₄³⁻ + Phosphonate)**—use 25 mL of Phosphate Standard Solution Complex, 10 mg/L as PO₄³⁻, in place of the sample in the test procedure. The result should be 10 mg/L as PO₄³⁻.

summary of method

This method is directly applicable to cooling tower samples that are treated with GenGard GN8000 series products. The test measures the amount of ortho-phosphate (o- PO₄³⁻), orthophosphate plus phosphonate (o- PO₄³⁻ + phosphonate), and phosphonate concentrations in the sample. Phosphonates are organophosphorous compounds which can revert to o- PO₄³⁻ in a cooling tower. The o- PO₄³⁻ in the sample is measured directly when a PhosVer 3 reagent is added to a 10X diluted filtered sample. The o- PO₄³⁻ +phosphonate in the sample is measured directly when a PhosVer 3 reagent is added to a 10X diluted filtered sample that has been digested with UV/persulfate, which converts the phosphonate to o- PO₄³⁻. The o- PO₄³⁻ in the sample reacts with the molybdate and ascorbic acid in the PhosVer 3 reagent to produce the molybdenum blue color. The measurement wavelength is 880 nm for spectrophotometers (DR 1900: 710 nm) or 610 nm for colorimeters.

In most applications of GN8000 series products, the level of ortho-phosphate (o- PO₄³⁻) must be known to ensure adequate levels for good mild steel corrosion protection. To determine the actual feed rate of a GN8000 series product based on tested phosphate residuals, the orthophosphate plus phosphonate test result is required.

Required reagents*

Description	Quantity/test	Unit	Code
Hydrochloric Acid Solution, 6 N (50%)	varies	60 mL	L247.0060
PhosVer 3 Phosphate Reagent Powder Pillow ¹ , 10-mL	2	100/pkg	L2325
Potassium Persulfate Powder Pillow for Phosphonate	1	100/pkg	L2045
Water, deionized	varies	4000 mL	L243.4000

¹ PhosVer is a registered trademark of Hach Company.

*Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use any recommended personal protective equipment.

Required apparatus

Description	Quantity/test	Unit	Code
Bottle, square, with 25-mL mark	1	each	L2633A
Pipetter, adjustable volume, 1.00–10.00 mL	1	each	L1089
Tips for L1089 Pipetter, 10-mL	varies	200/pkg	L20002
Pipetter, Socorex Calibra 832.10, 1.0–10.0 mL (EMEA only)	1	each	L8035
Tips for L8035 Pipetter, Socorex 312.10, 10 mL (EMEA only)	varies	100/pkg	L8036
Mixing cylinder, graduated, 50-mL, with glass stopper	1	each	L2631
Filter membrane, 0.22-micron, 25-mm	1	100/pkg	L774
Filter holder, 25-mm membrane filter	1	each	L773
Cord adapter, for dual UV lamps (for 110 V power supply)	1	each	L2669
Lamp, UV (for 110 V power supply)	1	each	L2666
Syringe, plastic 50-mL	1	each	L775
UV lamp, shortwave 254 nm (EMEA, for 220 V power supply)	1	each	L2138
Lamp Kit, 110 V (includes power supply and UV Lamp L2666)	1	each	L2692
Power supply for UV lamp, 220 V (EMEA, requires UV lamp L2138)	1	each	L2693.0020
UV safety goggles	1	each	L2668

Spectrophotometers and colorimeters (select one)

Description	Quantity/Test	Unit	Code
DR 1900 Spectrophotometer with accessories	1	each	L1901
Sample cells, 10-mL square, matched pair	2	2/pkg	L2793.0002
DR 900 Colorimeter with accessories	1	each	L901
Sample cells, 10-20-25-mL, with cap	3	6/pkg	L1976

Optional reagents and apparatus

Description	Unit	Code
Pipetter, Socorex Calibra 822.1000, 100–1000 μL (EMEA only)	each	L8034
Tips for L8034 Pipetter, Socorex 319.1000B, 1000- μL (EMEA only)	250/pkg	L8037
Pipet, plastic graduated, 1/10, 10-mL	each	L395
Safety bulb, rubber	each	L1575
Phosphate Standard Solution, 10-mg/L as PO_4^{3-}	500 mL	L1869.0500
Phosphate Standard Solution Complex, 10-mg/L PO_4	1000 mL	L477.1000