



✓ Method 8048

PhosVer 3 Method

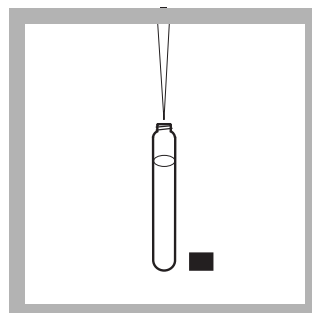
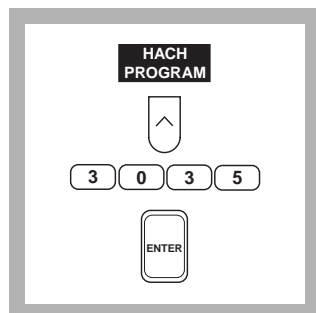
(0.00 to 5.00 mg/L PO₄³⁻)

Test 'N Tube™ Vials

(0.00 to 1.60 mg/L P)

Scope and Application: For water, wastewater and seawater. USEPA accepted for reporting wastewater analysis*. The estimated detection limit for program number 3035 is 0.02 mg/L PO₄³⁻.

* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P E for wastewater.



1. Press the soft key under **HACH PROGRAM**.

Select the stored program number for Test 'N Tube reactive phosphorus by pressing **3035** with the numeric keys.

Press: **ENTER**

2. The display will show: **HACH PROGRAM: 3035 P React. As. TNT**

The wavelength (λ), **890 nm**, is automatically selected.

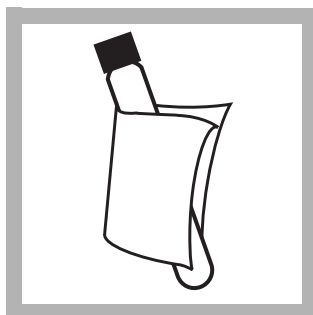
Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 12, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under **OPTIONS, (MORE)**, and then **BLANK:OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.

3. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive Phosphorus Test 'N Tube Dilution Vial. Cap and mix.

Note: For non-preserved samples with extreme pH, see Interferences.

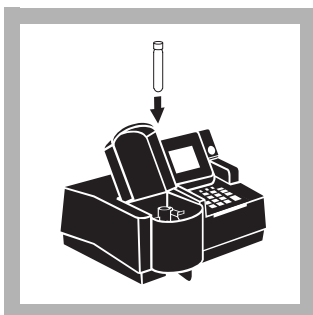
Note: For proof of accuracy, use a 1.0-mg/L Phosphate (0.33-mg/L P) Standard Solution in place of the sample (see **OPTIONAL REAGENTS**).

4. Insert the Test Tube Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

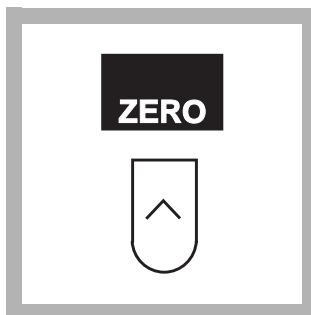


5. Clean the outside of the vial with a towel.

Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.



6. Place the vial into the cell holder and close the light shield.



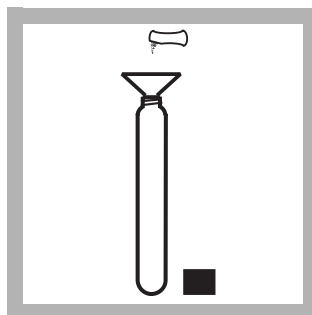
7. Press the soft key under **ZERO**.

The display will show:

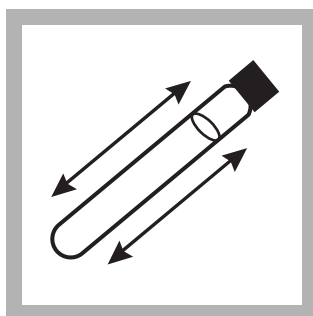
0.00 mg/L PO₄³⁻

Note: If you are using a reagent blank correction, the display will show the correction.

Note: For alternate concentration units press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.

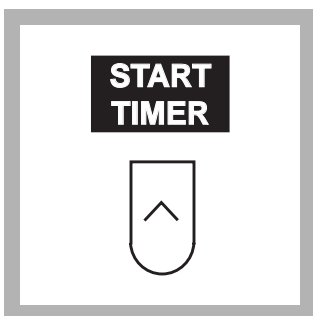


8. Using a funnel, add the contents of one PhosVer 3 Phosphate Powder Pillow to the vial.



9. Cap the vial tightly and shake for 10-15 seconds.

Note: The powder will not dissolve completely.



10. Press the soft key under **START TIMER**.

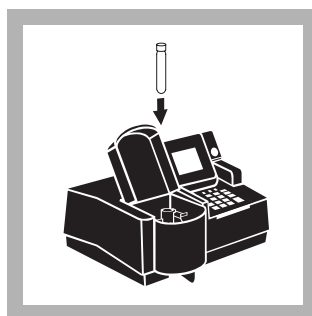
A 2-minute reaction time will begin.

Note: Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.



11. Clean the outside of the vial with a towel.

Note: Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.



12. When the timer beeps, place the vial into the cell holder and close the light shield. The results in mg/L PO₄³⁻ (or chosen units) will be displayed.

Note: Results may be expressed as phosphorus (P) or as phosphorus pentoxide (P₂O₅). Press the soft keys under **OPTIONS** and then **FORM:** to scroll through the available options.

Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Sulfide	Greater than 6 mg/L. Remove sulfide interference as follows: <ol style="list-style-type: none"> 1. Measure 25 mL of sample into a 50-mL beaker. 2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color appears. 3. Swirling constantly, add Phenol Solution drop-wise just until the yellow color disappears. Proceed with <i>step 1</i> of the phosphorus procedure.
Turbidity	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

Sample Collection, Storage and Preservation

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 48 hours by filtering immediately and storing at 4 °C. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method

Note: Clean glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. Do not use phosphate detergents to clean glassware.

- a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under **OPTIONS, (MORE)** and then **STD ADD**.
- b. Press **ENTER** to accept the default sample volume (mL), 25.0.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 50.00.
- d. Press the soft key under **ENTRY DONE**.
- e. Snap the neck off a Phosphate 2-mL Ampule Standard, 50-mg/L as PO_4^{3-} .
- f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.
- g. Analyze each standard addition sample as described above (use a 5-mL aliquot of the spiked sample as the sample). Accept the standard additions reading by pressing the soft key under **READ** each time. Each addition should reflect approximately 100% recovery.
- h. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.
- i. See Section 1.4.1 *Standard Additions* for more information.

Method Performance

Precision

Standard: 5.00 mg/L PO_4^{3-}

Program	95% Confidence Limits
3035	4.20–5.80 mg/L PO_4^{3-}

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
3035	0.01 mg/L PO_4^{3-}

For more information on derivation and use of Hach’s estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 3035

Portion of Curve	ΔAbs	$\Delta\text{Concentration}$
Entire Range	0.010	0.061 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

Calibration Standard Preparation

To perform a phosphate calibration using the Test 'N Tube method, prepare calibration standards containing 1.0, 2.0, 4.0, and 5.0 mg/L phosphate as follows:

- a. Into four different 50-mL Class A volumetric flasks, pipet 1.0, 2.0, 4.0, and 5.0 mL of a 50-mg/L Phosphate Standard Solution (Cat. No. 171-49) using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the Test 'N Tube method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

Summary Of Method

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to *Section 1*.

Pollution Prevention and Waste Management

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

PHOSPHORUS, Reactive, continued

REQUIRED REAGENTS

Reactive Phosphorus Test 'N Tube Reagent Set 50 tests 27425-45
Includes: (1) 21060-46, (50) Reactive Phosphorus Dilution Vials*

Description	Quantity Required		Cat. No.
	Per Test	Unit	
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Reactive Phosphorus Test 'N Tube Dilution Vials	1	50/pkg	*

REQUIRED APPARATUS

DR/4000 Test Tube Adapter.....	1	each	48189-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	1	50/pkg	21997-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30-g/L.....	29 mL**	2211-20
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phenol Solution, 30-g/L	29 mL	2112-20
Phosphate Standard Solution, 1-mg/L as PO ₄ ³⁻	500 mL	2569-49
Phosphate Standard Solution, PourRite™ Ampule, 50-mg/L as PO ₄ ³⁻ , 2-mL	16/pkg	171-20H
Phosphate Standard Solution, PourRite™ Ampule, 50-mg/L as PO ₄ ³⁻ , 10-mL	16/pkg	171-10
Phosphate Standard Solution, 50-mg/L.....	500 mL	171-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Flask, volumetric, Class A, 50-mL	each	14574-41
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette® Pipet	1000/pkg	21997-28
Pipet, volumetric, Class A, 2.00-mL.....	each	14515-36
PourRite™ Ampule Breaker	each	24846-00

* These items are not sold separately.

** Larger sizes available.



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