



✓ Method 8190

PhosVer 3 with Acid Persulfate Digestion

(0.00 to 3.50 mg/L PO₄³⁻)

Test 'N Tube™ Vials

(0.00 to 1.10 mg/L P)

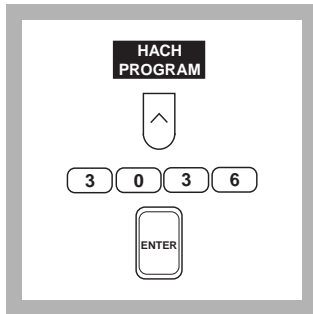
Scope and Application: For water, wastewater and seawater. USEPA accepted for reporting wastewater analyses. The estimated detection limit for program number 3036 is 0.06 mg/L PO₄³⁻.



1. Turn on the COD Reactor. Heat to 150 °C. Place the plastic shield in front of the reactor.

Note: Ensure safety devices are in place to protect the analyst from splattering should leakage occur.

Note: See COD Reactor Manual for temperature adjustment instructions.



2. Press the soft key under **HACH PROGRAM**. Select the stored program for Test 'N Tube total phosphorus by pressing **3036** with the numeric keys.

Press: **ENTER**

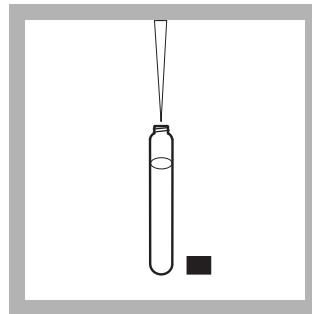
Note: If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.



3. The display will show: **HACH PROGRAM: 3036 P Total As. TNT**

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

Note: Determine a reagent blank for each new lot of each reagent as follows: Prepare a reagent blank by repeating Steps 4 through 18, using deionized water as the sample. 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.

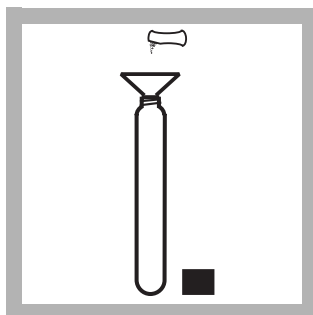


4. Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial.

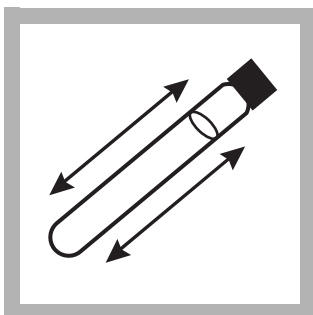
Note: For proof of accuracy, use a 1.0 mg/L Phosphate (0.33 mg/L P) Standard Solution in place of the sample (see **OPTIONAL REAGENTS AND STANDARDS**).

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

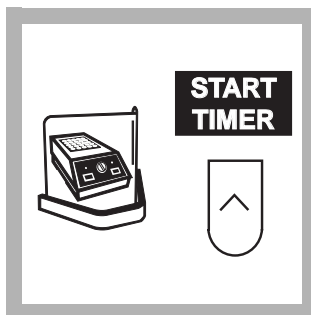
PHOSPHORUS, Total, continued



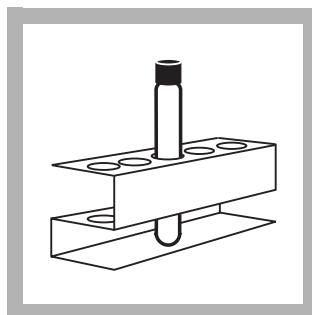
5. Using a funnel, add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



6. Cap tightly and shake to mix.

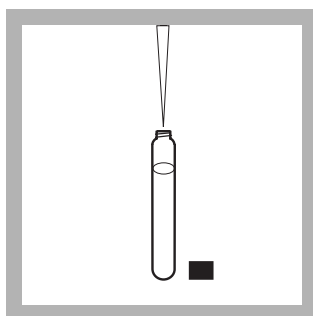


7. Place the vial in the COD Reactor, and start a 30-minute heating period by pressing the soft key under **START TIMER**.



8. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

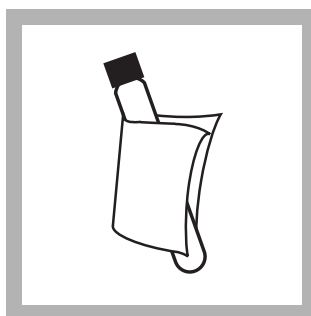
Note: Tubes will be hot.



9. Using a TenSette Pipet, add 2 mL of 1.54 N Sodium Hydroxide Standard Solution to the vial. Cap and mix.

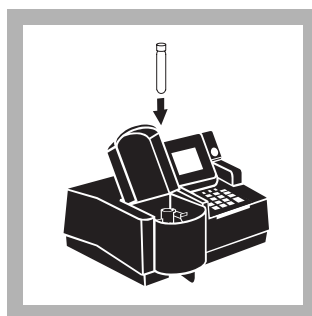


10. Insert the Test Tube Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

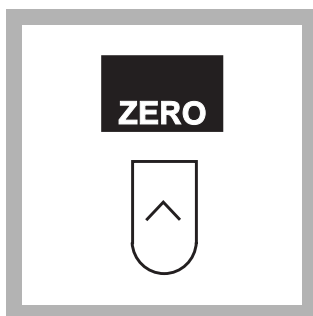


11. Clean the outside of the vial with a towel.

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



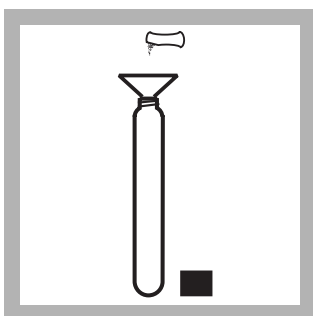
12. Place the sample vial in the cell holder and close the light shield.



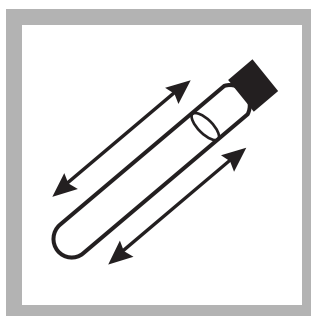
13. Press the soft key under **ZERO**.

The display will show:

0.00 mg/L PO₄³⁻

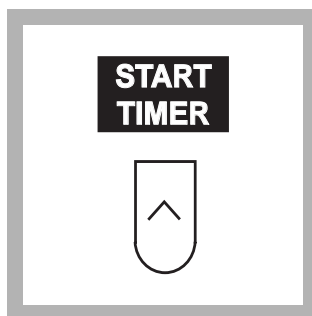


14. Using a funnel, add the contents of one PhosVer 3 Powder Pillow to the vial.



15. Cap tightly and shake to mix for 10-15 seconds.

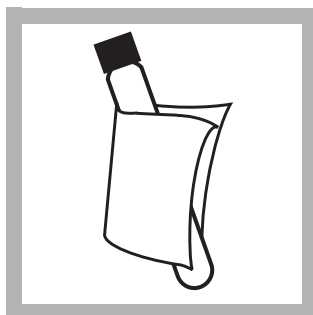
Note: The powder will not dissolve completely.



16. Press the soft key under **START TIMER**. A 2-minute waiting period will begin.

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

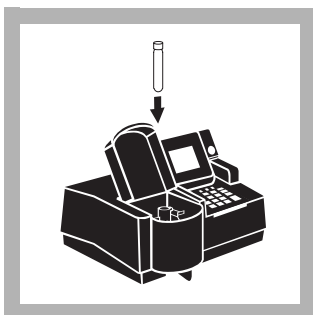
Note: For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



17. After the timer beeps, clean the outside of the sample vial with a towel.

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

Note: Read the sample 2-8 minutes after the addition of the PhosVer 3 reagent.



18. Place the prepared sample vial into the cell holder and close the light shield. Results in mg/L PO_4^{3-} (or chosen units) will be displayed.

Note: Results may be expressed as phosphorus (P) or as phosphorus pentoxide (P_2O_5). Press the soft keys under **OPTIONS** and then **FORM**: to scroll through the available options.

Important Note: The test range for total phosphate is limited to 0 to 3.5 mg/L PO_4^{3-} . Values greater than 3.5 mg/L may be used to estimate dilution ratios, but should NOT be used for reporting purposes. If the value is greater than 3.5 mg/L, dilute the sample and repeat the digestion and colorimetric test.

Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	Interferes at any level
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
pH, excess buffering	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Sulfide	Greater than 90 mg/L
Turbidity (large amounts) or color	May cause inconsistent results because the acid in the powder pillow may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	Greater than 80 mg/L

Store the PhosVer 3 Phosphate Reagent Powder Pillows in a cool, dry environment.

Sample Collection, Storage and Preservation

Collect samples in plastic or glass bottles that have been acid washed 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.

PHOSPHORUS, Total, continued

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with H₂SO₄ (2 mL per L) and storing at 4 °C. Before analyzing samples, warm to room temperature and neutralize.

Accuracy Check

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

Standard Additions Method

- 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.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 50.0.
- d. Press the soft key under **ENTRY DONE**.
- e. Snap the neck off a Phosphate 2-mL Ampule Standard, 50-mg/L as PO₄³⁻.
- 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: 3.00 mg/L PO₄³⁻

Program	95% Confidence Limits
3036	294–3.06 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
3036	0.06 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: 3036

Portion of Curve	Δ Abs	Δ 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 0.5, 1.0, 1.5, 2.0, and 2.5 mg/L phosphate as follows:

- a. Into five different 100-mL Class A volumetric flasks, pipet 1, 2, 3, 4, and 5 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, Total, continued

REQUIRED REAGENTS AND STANDARDS

Total Phosphorus Test 'N Tube Reagent Set 50 tests 27426-45
Includes: (1) 272-42, (1) 20847-66, (1) 21060-46, (1) 27430-42, (50) Total and Acid Hydrolyzable Test Vials*

Description	Quantity Required		Cat. No.
	Per Test	Unit	
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate powder Pillows.....	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N.....	2 mL	100 mL	27430-42
Total and Acid Hydrolyzable Test Vials.....	1	50/pkg	*
Water, deionized		4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

COD Reactor, 115/230 VAC (U.S.A. and Canada).....	1	each	45600-00
COD Reactor, 115/230 VAC (Europe)	1	each	45600-02
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	1000/pkg	21997-28
Safety Shield, laboratory bench	1	each	23810-00
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS AND STANDARDS

Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, 1-mg/L as PO ₄ ³⁻	500 mL	2569-49
Phosphate Standard Solution, 2-mL PourRite Ampule, 50-mg/L as PO ₄ ³⁻	20/pkg	171-20H
Sodium Hydroxide Standard Solution, 5.0 N.....	1 liter	2450-53
Sulfuric Acid, ACS.....	500 mL	979-49
Total and Acid Hydrolyzable Test 'N Tube Reagent Set	50/pkg	27427-45

OPTIONAL EQUIPMENT AND SUPPLIES

Flask, volumetric, Class A, 50-mL	each	14574-41
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
pH Meter, <i>sensio</i> TM <i>I</i> , portable	each	51700-00
Pipet, volumetric, Class A, 0.5-mL.....	each	14515-34
Pipet, volumetric, Class A, 1.00-mL.....	each	14515-35
Pipet, volumetric, Class A, 3.00-mL.....	each	14515-03
Pipet, volumetric, Class A, 4.00-mL.....	each	14515-04
Pipet, volumetric, Class A, 5.00-mL.....	each	14515-37
Pipet Filler, safety bulb.....	each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96

* These items are not sold separately.



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