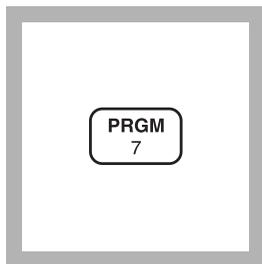


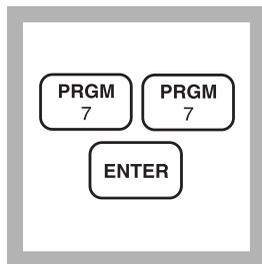
PHOSPHORUS, REACTIVE (0 to 45.0 mg/L PO₄³⁻) For water and wastewater**(Also called Orthophosphate) Molybdovanadate Method***
(Reagent Solution or AccuVac Ampuls)**Using Reagent Solution**

1. Enter the stored program number for high range phosphate (PO₄³⁻) reagent solution.

Press: **PRGM**

The display will show:

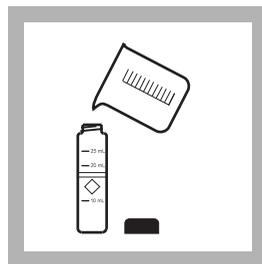
PRGM ?



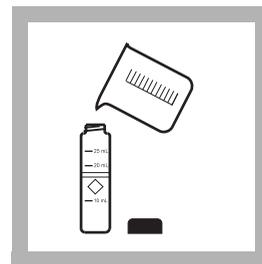
2. Press: **77 ENTER**

The display will show **mg/L, PO₄** and the **ZERO** icon.

*Note: For alternate forms (P, P₂O₅), press the **CONC** key.*

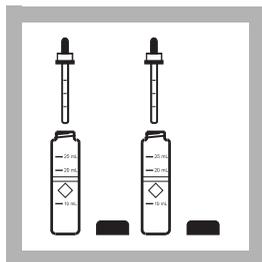


3. Fill a sample cell with 25 mL of deionized water (the blank).



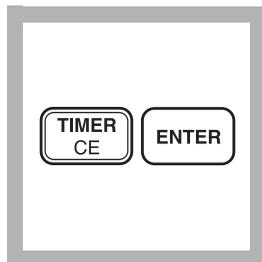
4. Fill another sample cell with 25 mL of sample (the prepared sample).

Note: For best results, the sample temperature should be 20-25 °C.



5. Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Cap the cells and invert to mix.

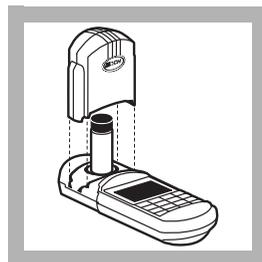
Note: A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank, because of the reagent.



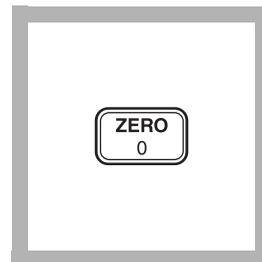
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



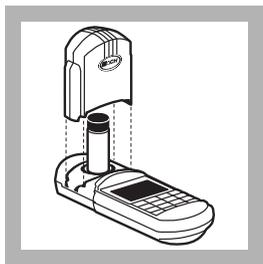
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

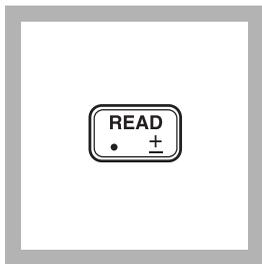
0.0 mg/L PO₄

* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

PHOSPHORUS, REACTIVE, continued



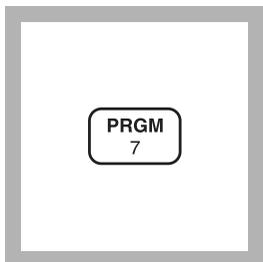
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**
The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.

Using AccuVac Ampuls

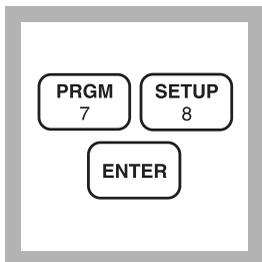


1. Enter the stored program number for high range phosphate (PO_4^{3-})-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?



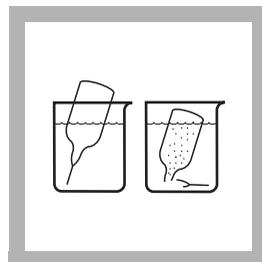
2. Press: **78 ENTER**
The display will show **mg/L, PO4** and the **ZERO** icon.

*Note: For alternate forms (P, P_2O_5), press the **CONC** key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

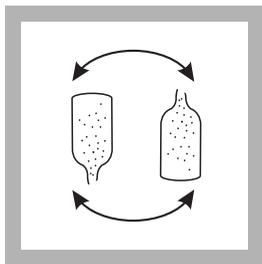
Note: For best results, sample temperature should be 20-25 °C.



4. Fill a Molybdo-vanadate Reagent AccuVac Ampul with sample. Fill a second AccuVac Ampul with deionized water (the blank).

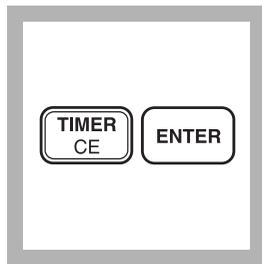
Note: Keep the tip immersed while the ampul fills completely.

PHOSPHORUS, REACTIVE, continued

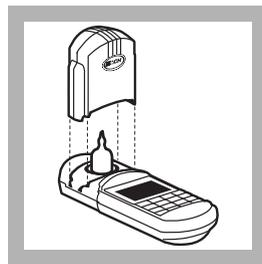


5. Invert the ampul several times to mix, then wipe off any liquid or fingerprints.

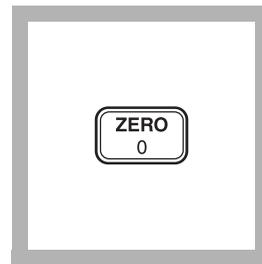
Note: A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank because of the reagent.



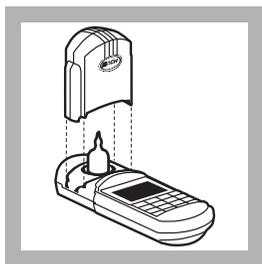
6. Press: **TIMER ENTER**
A five-minute reaction period will begin.



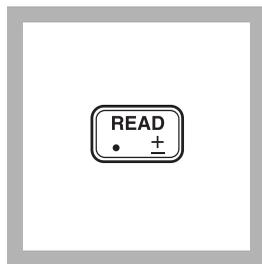
7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**
The cursor will move to the right, then the display will show:
0.0 mg/L PO₄



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**
The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

PHOSPHORUS, REACTIVE, continued

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

Accuracy Check

Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as PO_4^{3-} .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and invert to mix well.
- d) For analysis with AccuVac Ampuls, transfer the spiked samples to clean, dry 50-mL beakers to facilitate filling of the ampuls. For analysis with reagent solution, transfer the spiked samples to 25-mL sample cells.
- e) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L PO_4^{3-} .
- f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Obtain a Hach Phosphate Standard Solution, 10.0 mg/L as phosphate. Using this solution as the sample, perform the phosphate procedure as described above.

Standard Adjust

To adjust the calibration curve using the reading obtained with the

10.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* for more information.

PHOSPHORUS, REACTIVE, continued

Method Performance

Precision

In a single laboratory using a standard solution of 30.0 mg/L PO_4^{3-} , two lots of reagent, and the instrument, a single operator obtained a standard deviation of ± 0.1 mg/L PO_4^{3-} for the reagent solution method and a standard deviation of ± 0.2 for the AccuVac Ampul method.

Estimated Detection Limit

The estimated detection limit for program 77 is 0.3 mg/L PO_4^{3-} and 0.4 mg/L PO_4^{3-} for program 78. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatment

Interfering Substance	Interference Level and Treatment
Arsenate	Only interferes if sample is heated.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if sample is heated.
Sulfide	Causes a negative interference. Remove interference as follows: <ol style="list-style-type: none"> 1. Measure 50 mL of sample into an erlenmeyer flask. 2. Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops. 3. Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with step 4 of the procedure (step 3 if using the AccuVac procedure).
Extreme pH or highly buffered samples	May exceed buffering capacity of reagents. See Section 1, <i>pH Interferences</i> . Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause negative interference
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+} , Fe^{3+} , Mg^{2+} , Ca^{2+} , Ba^{2+} , Sr^{2+} , Li^+ , Na^+ , K^+ , NH_4^+ , Cd^{2+} , Mn^{2+} , NO_3^- , NO_2^- , SO_4^{2-} , SO_3^{2-} , Pb^{2+} , Hg^+ , Hg^{2+} , Sn^{2+} , Cu^{2+} , Ni^{2+} , Ag^+ , U^{4+} , Zr^{4+} , AsO_3^- , Br^- , CO_3^{2-} , ClO_4^- , CN^- , IO_3^- , SiO_4^{4-} .	

PHOSPHORUS, REACTIVE, continued

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

REQUIRED REAGENTS AND APPARATUS (using Reagent Solution)

Description	Quantity Required		Cat. No.
	Per Test	Units	
Molybdovanadate Reagent	2.0 mL	100 mL* MDB	20760-32
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Water, deionized.....	25 mL	4 L	272-56

REQUIRED REAGENTS AND APPARATUS (using AccuVac Ampuls)

Molybdovanadate Reagent AccuVac Ampuls	2	25/pkg	25250-25
Beaker, 50 mL.....	2	each	500-41H
Water, deionized.....	25 mL	4 L	272-56

OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L.....	29 mL*	2211-20
Hydrochloric Acid Solution, 1:1 (6.0 N).....	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 10.0 mg/L as PO ₄ ³⁻	946 mL	14204-16
Phosphate Standard Solution, Voluette Ampule, 500 mg/L as PO ₄ ³⁻ , 10 mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Sulfuric Acid, ACS	500 mL*	979-49
Wastewater Influent Standard, Inorganic (NH ₃ -N, NO ₃ -N, PO ₄ , COD, SO ₄ , TOC).....	500 mL	28331-49

OPTIONAL APPARATUS

AccuVac Snapper Kit.....	each	24052-00
Ampule Breaker Kit.....	each	21968-00
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, mixing, 25-mL.....	each	20886-40
Dispenser, fixed volume, 1.0 mL Repipet Jr.....	each	21113-02
Flask, erlenmeyer, 50 mL	each	505-41
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Paper, 1 to 11 pH units.....	5 rolls/pkg	391-33
pH Meter, <i>Sensio</i> TM 1, portable with electrode	each	51700-10

* Contact Hach for larger sizes.

PHOSPHORUS, REACTIVE, continued

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Pipet, serological, 2.0 mL.....	each	532-36
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 19700-01 TenSette Pipet	1000/pkg	21856-28
Thermometer, -20 to 110 °C.....	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.