

Total Phosphorous Persulfate Oxidation

(Rev B, 01 June 00)

NOTE: For **Total Dissolved P** filter samples through ≤ 1.0 μM filter before processing

For **Total P** filter samples through ≤ 1.0 μM filter after digestion

Materials:

- 25ml glass test tubes and glass scintillation vials, dram vials, or 10 mL beakers for lids (acid-washed & ashed)
- Adjustable auto-pipettors and tips (ranges 50 μL – 5 mL)
- Autoclavable test tube rack(s)
- Acid washed glassware (vol. Flasks, beakers, erlenmeyer flasks, etc)

Reagents: Note: Wear gloves and use dedicated glassware and utensils when preparing reagents and processing samples to avoid P contamination. Rinse all glassware with ultrapure water before use.

Oxidizing Reagent - Dissolve 50g potassium persulfate in appx. 800mL ultrapure water in a 1 L vol flask. Bring to 1 L with water. Prepare enough for samples, blanks, and controls plus $\geq 500\text{mL}$ to prepare standards.

Digestion Control (D.C.) Stock Standard (50 ppm P) - Dissolve 0.2470 g of glycerol 2-phosphate pentahydrate, disodium salt in ultrapure water to 500ml total volume.

Working D.C. Standard (10 ppm) - Add 5 mL 50 ppm stock to a 25 mL vol flask. QS w/ ultrapure water.

Procedure:

- 1) Prepare at least 2 replicated each of controls, blanks, and samples in 25 ml glass test tubes as follows

Controls: A. 5.00mL D.C. Stock + 5.00mL Oxidizing Reagent (Ox Rgt)
B. 2.50mL D.C. Stock + 2.50mL Representative Sample + 5.00mL Ox Rgt

Blanks: 5.00mL ultrapure water + 5.00mL Ox Rgt

Samples: 5.00mL sample + 5.00mL Ox Rgt

- 2) Place inverted vial/beaker over each tube. Weigh each tube. Record weights.
- 3) Autoclave controls, blanks, samples, and all excess Ox Rgt (in foil covered Erlenmeyer flask) for 30 minutes on liquid cycle (250 F). The volume of Ox Rgt to be autoclaved should be measured first with a graduated cylinder, and then recorded.
- 4) After allowing to cool to room temp, re-weigh tubes and bring back to original weight with ultrapure water. Also, re-measure volume of Ox Rgt and bring to 2 X's its original volume with water (use new container if necessary). This will be called "Sample Matrix".
- 5) Prepare $\text{PO}_4\text{-P}$ standards and instrument QC in Sample Matrix using one of the following sets of standards/QC's depending on expected concentration range:

Calibration Standards

Range: High (expected sample concentration of 0.4 – 4.0 ppm P)							
Stock Conc: 100 ppm PO ₄ -P				Stock Recipe: 0.2199g Potassium Phosphate, Monobasic per 500 mL			
Final Working Standard Conc (ppm)		Vol. Flask Volume (mL)			Volume of Stock to Add (mL)		
	2.000			50			1.000
	1.600			50			0.800
	1.200			50			0.600
	0.800			100			0.800
	0.400			100			0.400
	0.200			100			0.200

Range: Low (expected sample concentration of 10 - 400 ppb P)							
Stock Conc: 10 ppm PO ₄ -P				Stock Recipe: 1.000 mL of 100 ppm Stock per 10 mL			
Final Working Standard Conc (ppb)		Vol. Flask Volume (mL)			Volume of Stock to Add (mL)		
	200.0			50			1.000
	100.0			100			1.000
	50.0			100			0.500
	20.0			250			0.500
	10.0			250			0.250
	5.0			500			0.250

External Source QC's: See "Quality Control Preparation Table"

- 6) If working in the high range, dilute controls 1:3 with Sample Matrix.
If working in the low range, dilute controls 1:50 with Sample Matrix.
- 7) Analyze for PO₄-P on TrAAcs using appropriate template ("LOWSRP" or "HIGHSRP"). Use Sample Matrix as blank for standard curve.

Calculations:

Corrected Target Value for Sample Interference Check

$$[(\text{Avg. Measured Control 'A' Conc.} \times \text{Dil Factor}) - \text{Avg. Measured Blank Conc.}] + [\text{Avg. Measured Sample Conc.} \times \text{Dil Factor}]$$

Note: This formula already accounts for 1:2 dilution with Ox Rgt

Corrected Interference Check Concentration

$$2 \times [(\text{Control 'B' Avg. Measured Conc.} \times \text{Dil Factor}) - (0.5 \times \text{Avg. Measured Digestion Blank Conc.})]$$

% Recovery of Interference Check

$$(\text{Corrected Interference Check Conc.} \div \text{Corrected Target Value}) \times 100\%$$

Corrected Measured D.C. Concentration

$$2 \times [(\text{Avg. Measured Control 'A' Conc.} \times \text{Dil Factor}) - \text{Avg. Measured Blank Conc.}]$$

% Recovery of Digestion Control

$$(\text{Corrected Measured D.C. Conc.} \div 50 \text{ ppm}) \times 100\%$$