

**Total Nitrogen**  
**Alkaline Persulfate Oxidation**  
(Rev B, 01 June 00)

**NOTE:** For **Total Dissolved N** filter samples through  $\leq 1.0$   $\mu\text{M}$  filter before processing

For **Total N** filter samples through  $\leq 1.0$   $\mu\text{M}$  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 $\mu\text{L}$  – 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 N contamination. Rinse all glassware with ultrapure water before use.

NaOH, 3.75 M stock solution - Dissolve 75 g of low N NaOH in ~400mL of ultrapure water (Nanopure, Milli-Q, etc). Allow to cool and Q.S. to 500mL. Store in a tightly sealed glass or, preferably, a polypropylene bottle.

Oxidizing Reagent - Add 50g low N potassium persulfate and 30g of boric acid to 100mL of 3.75 N NaOH in a 1 L flask. Bring to 1 L with ultrapure water. Prepare enough for samples, blanks, and controls plus  $\geq 600\text{mL}$  to prepare standards.

Urea Stock Standard (100 ppm N) - Dissolve 0.1073 g urea in ultrapure water to 500ml total volume.

**Procedure:** 1) Prepare at least 2 replicates each of controls, blanks, and samples in 25 mL glass test tubes as follows

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

Blanks: 5.00mL low N 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 ultrapure water (use new container if necessary). This will be called "Sample Matrix".

- 5) Prepare NO<sub>3</sub>-N standards and instrument QC in Sample Matrix according to the following table.

Stock Conc. 100 ppm NO <sub>3</sub> -N		Stock Recipe: 0.3034g Sodium Nitrate per 500 mL				
Final Working Standard Conc (ppm)		Vol. Flask Volume (mL)		Volume of Stock to Add (mL)		
2.000		50		1.000		
0.800		50		0.400		
0.200		100		0.200		
0.050		500		0.250		
0.020		100		1.0 mL of 2.0ppm Working Standard		
0.010		100		0.5 mL of 2.0ppm Working Standard		
External Source QC (0.1 ppm): See "Quality Control Preparation Table"						

- 6) Dilute controls 1:50 with Sample Matrix.
- 7) Analyze for NO<sub>3</sub>-N on TrAAcs using TPN manifold and 'TotalN' input file template. Use Sample Matrix as blank for standard curve.

**Calculations:**

Corrected Target Value for Sample Interference Check

$$[(\text{Avg. Measured Control 'A' Conc.} \times 50) - \text{Avg. Measured Blank Conc.}] + [\text{Avg. Measured Representative Sample Conc.} \times \text{Dil Factor}]$$

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

Corrected Interference Check Concentration

$$2 \times [(\text{Control 'B' Avg. Measured Conc.} \times 50) - (0.5 \times \text{Avg. Measured Blank Conc.})]$$

% Recovery of Interference Check

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

Corrected Measured Urea Concentration

$$2 \times [(\text{Avg. Measured Control 'A' Conc.} \times 50) - \text{Avg. Measured Blank Conc.}]$$

% Recovery of Urea N

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