Protocol for Persulfate Oxidation to Measure Total Nitrogen

Equipment:

- 20 mL PTFE screw-cap vials
- two-place balance
- Autoclave

Reagents:

0.05 *M* K_2SO_4 (8.714 g L^{-1}) for reagent blank
10 mg N/L as alanine in 0.05 *M* K_2SO_4 for standards
5% Alkaline potassium persulfate reagent
- Sodium hydroxide, NaOH (15 g L^{-1}) (low N)
- Potassium persulfate, $K_2S_2O_8$ (50 g L^{-1}) (low N)
- Boric Acid, H_3BO_4 (30 g L^{-1})

Note: To make the alkaline potassium persulfate, first dissolve low-N NaOH into DDI water, then add H_3BO_4, and finally add low-N $K_2S_2O_8$ and bring to volume with DDI water. Stable for up to a week at room temperature. Keep out of light or in a dark bottle.

Digestion Procedure:

1. Thaw the K_2SO_4 extracts.

2. Record the rack and vial number for each sample.

3. Invert the sample (K_2SO_4 extract) several times to mix. Pipette 2 mL of sample, 8 mL water, and 10 mL of persulfate reagent into each vial.

4. For every batch of samples include 1 reagent blank and 1 standard:

Blank = 2 mL of 0.05 *M* K_2SO_4
Standard = 2 mL of 10 mg N/L as alanine in 0.05 *M* K_2SO_4

5. Immediately after adding the persulfate reagent, tightly cap the vial to prevent gaseous loss of ammonia. Cap will "snap" into tightly-locked position, but be sure that it is flat. Caps will occasionally snap into place in a lopsided position, allowing vapor loss.

6. Place the capped vials into autoclave for 40 minutes inside an autoclave pan. Ensure the autoclave gets up to temperature.

NOTE: As little as 15 minutes may be sufficient for complete digestion of organic nitrogen, but more autoclave time is needed to ensure complete degradation of the persulfate. Otherwise, residual persulfate will prevent efficient reduction and capture of ammonia for ^{15}N diffusion, and shortens life of the cadmium column for Lachat nitrate analysis.

7. After autoclaving, allow sufficient time for the digests to cool and the outside of the vials to dry.