NH_4^+ Analysis

Erica Kiss - updated Jan. 2007

amended from Parsons et al 1984. A Manual of Chemical and Biological Methods for Seawater Analysis

Sample Collection:

Nutrient samples: 5 mL each, filtered with 47 mm GF/F. Store in 6 mL polypropylene vials, frozen at -20C.

Porewater samples: usually 0.25 mL each, filtered with 0.45 μm syringe filters

Standards:

Prepare standard solutions to bracket your sample concentrations. Use 10 mM NH_4^+ stock (0.5349g NH_4Cl/1L ultrapure water) NH_4Cl FW=53.49g/mol

- 100 mL final volume safely makes 19 standard reps (5 mL each)
- Prepare fresh solutions on the day of analysis.
- Note that linearity begins to be lost at concentrations greater than 100 μM.

Final Conc.	**NH_4^+ amount**	**Volumetric Flask**
μmol/L	μL stock	Final vol. (mL)
0	0	----
1	10	100
3	30	100
6	60	100
12	120	100
18	180	100
24	240	100
30	300	100
60	600	100
100	1000	100

Sample Preparation:

Porewater samples: usually diluted 1:20; use 5 mL final volume of sample (0.25 mL filtered sample and 4.75 mL ultrapure water).

Reagents:

1. Phenol solution
    - Dissolve 20g analytical grade phenol in 200 mL Photrex® grade ethanol (Alcohol, Anhydrous, Reagent).
    - Store in an amber glass bottle in the refrigerator.
2. Sodium nitroprusside, $Na_2[Fe(CN)5NO]\cdot 2H_2O$, solution
    - Dissolve 1g sodium nitroprusside in 200 mL ultrapure water.
    - Store in an amber glass bottle in the refrigerator.
3. Alkaline reagent
    - Dissolve 100g sodium citrate, $HOC(COONa)(CH_2COONa)_2\cdot 2H_2O$, and 5g sodium hydroxide, NaOH, in 500 mL ultrapure water.
    - Store in the refrigerator.
4. Oxidizing reagent

- 4:1 Alkaline reagent:Clorox Bleach

The oxidizing reagent must be prepared on the day of analysis. It is best to buy fresh Clorox each time due to degradation.

Procedure:
Use re-pipettor bottles. Add reagents in the following order. **Cap and mix between each addition**:

1. 0.2 mL phenol soln.
2. 0.2 mL nitroprusside soln.
3. 0.5 mL oxidizing reagent

Store samples overnight at room temperature in the dark before running (or for a minimum of 4 hrs). Read absorbance at 640 nm using the 1 cm flow cell within 24 hours.