

Reagent and standard matrix/extractant recipes for inorganic nitrogen extraction and analysis

### **AMMONIUM ANALYSIS**

#### **Sodium salicylate solution (store in refrigerator):**

Dissolve in 50ml DI H<sub>2</sub>O on a stirplate:

6.8 g sodium salicylate: Fisher # S395-500

5 g sodium citrate: Fisher # BP327-1

5 g sodium tartrate: Fisher # BP352-500

0.025 g sodium nitroprusside: Fisher # S350-100

Use a volumetric flask to bring to 100ml with DI H<sub>2</sub>O.

#### **1.5M NaOH (store on lab bench):**

6 g sodium hydroxide (Fisher #S318-1)

Bring to 100ml in a volumetric flask with DI H<sub>2</sub>O.

#### **Bleach/NaOH solution (mix fresh every day; makes enough for approx. 2 plates):**

0.4 ml bleach

19.60 ml 1.5M NaOH

#### **Stock Ammonium Solution (100 ppm) (store on lab bench):**

0.23585 g ammonium sulfate: Fisher # A938-500

500 ml ultrafiltered DI water

### **NITRATE ANALYSIS \*Make these solutions in a fume hood\***

#### **Vanadium solution (store in freezer)**

3.3ml 2% sulfanilamide solution (recipe below)

3.3 ml 0.2% NED solution (recipe below)

400 ml DI H<sub>2</sub>O

Premix the sulfanilamide solution, NED solution, and water before making the vanadium chloride solution.

Add 50ml saturated vanadium chloride solution (recipe below)

Store the solution in amounts that are reasonable for one time use (e.g., a 20ml scintillation vial holds enough for 1.5 plates or a 40ml septa vial holds enough for 2 plates). To store the solution, partition it into opaque scintillation vials or rubber septa vials that are covered in aluminum foil. Purge the solution with nitrogen before storing. This should be done immediately upon making the solution as it is very susceptible to oxygen.

When purging a scintillation vial, have nitrogen air blown into the vial to displace any empty head space with nitrogen. When purging a septa vial, insert two needles into the septa, one that is attached to a nitrogen source and another that has nothing attached. As the nitrogen is pumped into the empty head space, the displaced air flows out through the second needle; cycle nitrogen through the vial for about 3 minutes to ensure that it has been purged. When using this method, try to limit the number of times insert a needle into the rubber septa, as overused septas can leak. The vials can be stored in the freezer for up to 1 year. If making this solution in a nitrogen box, you can use a Schlenk flask to store the solution.

**1M HCl**

83.3 mL of concentrated HCl

Bring to 1L with DI H<sub>2</sub>O

\*\*Remember to always add acid to water

**2% sulfanilamide solution**

0.2g sulfanilamide

10ml 1.0M HCl

**0.2% NED solution**

0.02g N-(1-naphthyl)-ethylenediamine dihydrochloride

10ml DI H<sub>2</sub>O

**Saturated vanadium chloride solution**

\*\*Vanadium chloride powder is very reactive with air! It releases a chlorine gas when exposed to air. Prepare solution ideally in a nitrogen box but if not available a fume hood, and work quickly and carefully. The compound as well as the solution should also be stored in the dark as it breaks down in light and under nitrogen. Vanadium III chloride is a corrosive – wear gloves, goggles, and a lab coat.

0.35g vanadium (III) chloride (Fisher # AC19700-0250)

50ml 1.0M HCl.

Swirl the solution for 1-2 minutes. The solution should turn a blue to blue green color. Gravity-filter the solution using a funnel that has a cotton ball in its neck, and pre-wet the cotton ball with 1.0M HCl.

**Stock nitrate solution (100ppm) (store on lab bench)**

0.3609 g potassium nitrate

500 ml ultrafiltered DI water

**STANDARD EXTRACTANTS/MATRIX SOLUTIONS:****2.0M KCl**

149.1 g KCl

1 L DI H<sub>2</sub>O

**0.5M K<sub>2</sub>SO<sub>4</sub> (used in extractions for microbial biomass N)**

87.13g K<sub>2</sub>SO<sub>4</sub>

1 L DI H<sub>2</sub>O