Ammonium Analysis Reagent Recipes

v1.100

Safety

Consult the Material Safety Data Sheet for each reagent before handling or preparing the reagents. Reagents must be made by appropriately trained and qualified personnel equipped with proper safety equipment (e.g. safety glasses, gloves, lab jacket, etc.) within a properly equipped work area (e.g. fume hood, eyes wash, etc.). **Green Eyes LLC (Green Eyes) is not responsible or liable for accidents during reagent preparation or other activities related to testing or deploying Green Eyes equipment.**

General Preparation Considerations

For optimal results, all reagents should be made with high quality (18 M ohm/cm) deionized water (DIW) in labware previously acid washed with a 10% (1.2 N) Hydrochloric Acid (HCl) solution. After acid washing, labware should be rinsed three times with DIW. Reagent storage bottles should also be acid washed and DIW rinsed. Reagent salts or solutions used should be of reagent grade or better unless otherwise specified.

Method Description

Ammonium (NH_4^+) and Ammonia (NH_3) are both determined by the Berthelot Reaction in which an indophenol blue chromophore forms when ammonium and ammonia react with sodium phenoxide (phenol and sodium hydroxide) followed by the addition of sodium hypochlorite. Sodium citrate is used to prevent precipitation of calcium and magnesium hydroxides at the elevated reaction pH.

Preparation

Reagent 1 – Complexing Reagent (1000 ml)

Reagents and amounts:

1. Sodium Citrate [$HOC(COONa)(Ch_2OONa)_2 \cdot 2H_20$] – 140 g
2. Sodium Hydroxide (NaOH) – 5 g
3. DIW – 1000 ml

Preparation: Add 800 ml of DIW to a clean 1000ml flask. Add the sodium citrate and stir until dissolved. Then add the sodium hydroxide and make to a final volume of 1000 ml. Mix well before transferring to a bottle or reagent bag.

Storage: Store in a refrigerator until use. Stable for 2 months or more.

Reagent 2 – Color forming reagent (250ml)

Reagents and amounts:

1. Phenol (C_6H_5OH) solution (88%) - 8.75 ml
2. Sodium Nitroprusside Dihydrate [$Na_2Fe(CN)_5NO \cdot 2H_20$] – 0.1 g
3. DIW – 250 ml

Preparation: Phenol is a toxic chemical. All staff working with it should read the MSDS and observe proper safety procedures to eliminate skin contact, inhalation or ingestion. In a fume hood, add 200 ml of DIW to a 250 ml flask. Add the phenol solution and mix well (magnetic stirrer recommended). Add the nitroprusside and 50 ml of DIW, and mix well.

Storage: Store protected from light in refrigerator. Reagent is stable for two months or more.

Reagent 3 – Oxidizer (250ml)

Reagents and amounts:

1. Sodium Hypochlorite (commercial bleach with 5.25% free chlorine) – 12.5 ml
2. DIW – 240 ml

Preparation: Add 240 ml of DIW to a clean flask and then add the hypochlorite. Mix well.

Storage: When protected from light the reagent is stable for two months or more at room temperature.

Wash – 91% Isopropyl Alcohol

Reagent:

1. Pharmacy grade isopropyl alcohol (91% preferred) – no dilution or additional reagents

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Air or Inert Gas

Reagent:

1. Air free of ammonia (gas) and particles or inert gas such as argon.

References:

J. D. H. Strickland and T. R. Parsons: A Practical Handbook of Seawater Analysis. Ottawa: Fisheries Research Board of Canada, Bulletin 167, 2nd Ed., 1972. 293 pp.

L Solorzano: Determination of ammonia in natural waters by the phenolhypochlorite method. Limnol. Oceangr. Vol.14(5). 1969. pp. 799-801.