

Ortho-Phosphate Analysis Reagent Recipes

v1.0

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 (HCI) 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

This method is based on a an optimization by Drummond and Maher (1995) of the popular chemistry published by Murphy and Riley (1962). Phosphate is reacted with molybdenum to form 12-molybdophosphoric acid that when reduced by ascorbic acid forms a colored molybdenum blue compound that is measured colorimetricly.

Preparation

Reagent 1 - Molybdic Acid (500 ml)

Reagents and amounts:

- 1. Amonium Molybdate 4-hydrate $((NH_4)_6Mo_7O_{24} 4H_2O) 3.2 g$
- 2. Potassium Antimony Tartrate solution 8 ml
- 3. Concentrated Sulfuric Acid (36 N) 35 ml
- **4.** SDS solution 5 ml
- **5.** DIW 250 ml, 215 ml

Preparation:

While stirring, slowly add the sulfuric acid to 250 ml of DIW in a one liter flask. Then add the ammonium molybdate and continue stirring until all the molybdate is dissolved. While stirring, slowly add 8.0 ml of the potassium antimony tartrate solution and insure that it mixes completely. Add 215 ml of DIW followed by the SDS solution, mix well and store in a tightly sealed container.



Storage: Stable for 6 months at under refrigeration and 3 months at room temperature.

Potassium Antimony Tartrate ($C_8H_4K_2O_{12}Sb_2 - 3H_2O$) solution: Add 3.5 g potassium antimony tartrate to 200ml DIW and mix thoroughly. Stable for 6 months under refrigeration.

Sodium Dodecyl Sulfate (SDS, CH₃(CH₂)₁₁OSO₃Na) solution: Add 3 g of SDS to 100 ml of DIW and stir until fully dissolved. Stable for 6 months under refrigeration.

Note: If also running ammonium analysis on an EcoLAB, replace the ammonium molybdate with 4.5g of sodium molybdate (Na₂MoO₄ - 2H₂O). Sodium molybdate is very sensitive to ambient electrical fields and thus particles tend to "dance" around the weighing containers. It is recommended to weigh the salt inside a clean, dry bottle and seal until it is added to the acid. Wash the remaining crystals out of the bottle with the reagent.

Reagent 2 - Ascorbic Acid (500 ml)

Reagents and amounts:

- 1. Ascorbic Acid $(C_6H_8O_6) 4.5 g$
- **2.** DIW 500 ml

Preparation: Add the ascorbic acid to the DIW and stir until completely dissolved.

Storage: Stable for thee months under refrigeration in a tightly sealed container. Stable for one month or longer in the field when protected from light.

Air or Inert Gas

Reagent:

1. Air free of dust particles or inert gas such as argon.

References:

- L. Drummond and W. Maher: Determination of phosphorus in aqueous solution via formation of the phosphoantimonylmolybdenum blue complex Rexamination of optimium conditions for the analysis of phosphate. Analytica Chimica Acta 302 (1995) pp. 69 74.
- J. Murphy and J. P. Riley: A modified single solution method for the determination of phosphate in natural waters, Analytical Chimica Acta, 27 (1962) p. 31