

Determination of Phosphate Concentration in Soil

Safety

Lab coats, safety glasses and enclosed footwear must be worn at all times in the laboratory.

Concentrated sulfuric acid is highly corrosive – wear rubber gloves and take care when handling. It will burn your skin, and leave a stain on your skin for some days. If you do splash some on your skin, wash it well with cold running water IMMEDIATELY. Make sure your teacher is told about it. Also, be careful of your clothes, as acid will burn holes in them. When diluting into water, ALWAYS dilute acid into water, and never add water to acid. When you heat your solutions with sulfuric acid, there is a chance that the flask could break. Make sure you have water ready to clean any spills. Your teacher or laboratory supervisor should be with you at all times when you are doing this.

Introduction

This method uses a complexation reaction to produce a coloured complex of molybdate and phosphorus. This complex is formed when phosphate (from your sample) is heated with ammonium molybdate in the presence of acid and excess ascorbate ions (which are to prevent the colour degrading as the molybdate oxidizes slowly). The coloured complex formed is dependent on the initial phosphate concentration in the sample.

The amount of phosphate present is determined by comparison of the blue colour with known standards of phosphate, subjected to the same reaction with molybdate reagent. From this information, the concentration of phosphate in the soil can be calculated.

Equipment Needed

250 mL conical flasks

250 mL and 100 mL volumetric flasks

Bunsen burner, or heating plate

A sample of soil, dried in a very low oven overnight (~50-70°C)

Solutions Needed

Concentrated Sulfuric Acid (see safety notes)

Ammonium molybdate solid: $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$

Ascorbic acid solid (Vitamin C)

Ammonium sulfate solid: $(\text{NH}_4)_2\text{SO}_4$

Method

Sample Preparation

1. Dig up a sample of soil and heat it overnight at about 50°C to dry it. You don't want it too hot or there might be some chemical change in the sample, but you want to dry it completely. Cover it with tinfoil and avoid breathing the dust that comes off the dry soil.
2. In a 250 mL volumetric flask, add about 50 mL of water. To this add 0.75 g of ammonium sulfate, dissolve, and then add SLOWLY 5 mL of concentrated sulfuric acid. The solution will get hot. Allow it to cool, then dilute to the mark on the flask with distilled water.
3. In a plastic flask, put 10 g of dry soil and 200 mL of your sulfuric acid / ammonium sulfate mixture and shake occasionally over 30 minutes.
4. Filter the soil sample through fine filter paper and put to one side. It should be clear, but may be slightly brown in colour.

Preparation of standard

1. Prepare standard phosphate solutions. First prepare a 300 ppm solution by accurately weighing about 0.220 g of solid KH_2PO_4 into a 500 mL volumetric flask, and diluting it to the mark. Pipette 10 mL of the standard phosphate into a 200, 250, 500 mL and a 1 L volumetric flask, and fill it to the mark. This will give you phosphate solutions of 15, 12, 6 and 3 ppm solution respectively. Pipetting 15 mL of your standard solution into a 1 L volumetric flask will give you a 4.5 ppm solution.
2. Label each solution with the concentration, and the date you made it.

Preparation of Complex

1. Dissolve 5 g of ammonium molybdate into 100 mL of water. Transfer this to a 500 mL volumetric flask. To this add very slowly 160 mL of concentrated sulfuric acid (**see safety notes**). If the flask becomes very hot, stop and wait for it to cool over 15 minutes. Once all the acid has been added, dilute the solution to 500 mL with water – add the water slowly with stirring.
2. Take 10 mL of sample in a 150 mL conical flask and add 20 mL of water, 2 mL of molybdate solution and a spatula of ascorbic acid crystals. Heat this slowly to boiling (a deep blue/green colour should develop) and then allow it to cool. Repeat this for all the standards.

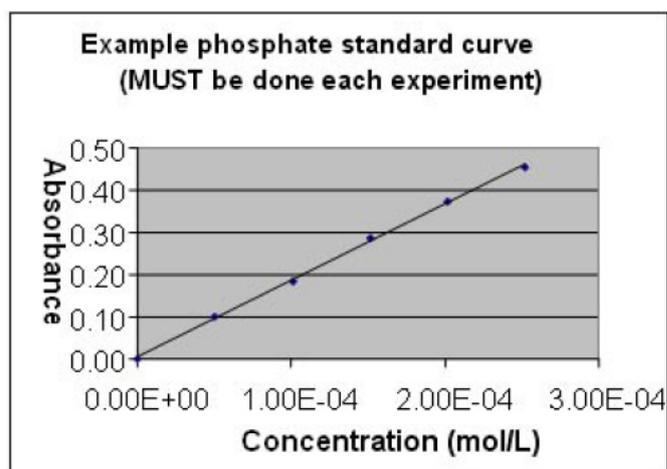
Colorimetric Analysis

This will depend on the colorimeter you have available to work with in your school. Your teacher should be able to guide you, but you need to make sure you are measuring at 650 nm. The instructions below are a good guide for what to do.

1. Fill a colorimetric tube with water (we call this a blank when you are working with colorimetry) and place it into the colorimeter. With the absorbance set to 650 nm (this is reddish light) take an absorbance reading. If there is a 'zero' adjust, or a 'blank' function on the colorimeter, use this water sample to zero the colorimeter.
2. Place your solution of lowest concentration (3 ppm from above) in the sample tube and take a reading. After recording the absorbance, wash the tube and repeat the measurement on the next most concentrated standard, until all the standards have been measured.
3. Place your sample into the colorimeter tube. Take an absorbance reading, and record.

Result Calculations

1. Draw a standard curve, by plotting on a graph the absorbance of your standard solutions (y-axis) versus the concentration of the standards (x-axis). This should be a straight line. Using the absorbance measurement of your fertilizer sample, read along the graph until you reach your curve, then read off the concentration which corresponds to your absorbance. This is the concentration of phosphate in your liquid sample [see example].
2. You will need to correct for any dilutions you performed.



Contact Us

If you have any questions or comments relating to this experiment, please contact us. Please note that this service is for senior school chemistry students in New Zealand only. We regret we are unable to respond to queries from overseas.

Outreach
College of Science
University of Canterbury
Private Bag 4800
Christchurch
New Zealand
Phone: +64 3 364 2178
Fax: +64 3 364 2490
Email: outreach@canterbury.ac.nz
www.outreach.canterbury.ac.nz