

PHOSPHATE METHOD 9

Using Ammonium Molybdate, Potassium Antimonyl Tartrate and Ascorbic Acid

PRINCIPLE OF THE METHOD

Ammonium Molybdate and Potassium Antimonyl Tartrate react in acid solution with *ortho*-phosphates to form phosphomolybdic acid. This is reduced to the intensely coloured Molybdenum blue by Ascorbic acid and the colour so formed is compared with Lovibond permanent colour glass standards.

REAGENTS REQUIRED

1. **Sulphuric Acid 2.5M.** Slowly add 135ml. of concentrated Sulphuric Acid (S.G.1.84) to 800ml. of deionised water with continuous stirring and cooling. (Caution). Cool and dilute to 1 litre.
2. **Sulphuric Acid 0.5M.** Dilute 20ml. of 2.5M Sulphuric Acid to 100ml. with deionised water.
3. **Sodium Hydroxide 1M.** Dissolve 4g. Sodium Hydroxide in 80ml. of deionised water (Caution). Cool and dilute to 100ml.
4. **Potassium Antimonyl Tartrate Solution.** Dissolve 1.371g. Potassium Antimonyl Tartrate ($\text{KSbOC}_4\text{H}_4\text{O}_6$) (Care-Poison) in 400ml. of deionised water and dilute to 500 ml. in a volumetric flask.
5. **Ammonium Molybdate Reagent.** Add 6g. Ammonium Molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) to 100ml. of warm deionised water and stir occasionally until dissolved. To this solution add 500ml. of 2.5M Sulphuric Acid (Reagent 1) and 50ml. of Potassium Antimonyl Tartrate solution (Reagent 4) and dilute to 1 litre.
6. **Ascorbic Acid** ($\text{C}_6\text{H}_8\text{O}_6$) 0.1g is used for preparation of both the “test” sample & the “blank”
7. **Ammonium Persulphate** ($(\text{NH}_4)_2\text{S}_2\text{O}_8$).
8. **Phenolphthalein Indicator.** Dissolve g. Phenolphthalein in 60ml. of Ethanol (industrial methylated spirit) or Propan-2-ol and add 40ml. deionised water.

All chemicals used in the preparation of reagents should be of analytical reagent quality.

THE STANDARD LOVIBOND NESSLERISER DISC NMD

This Disc covers the range 10 to 100 μg . of Phosphate calculated as PO_4 in steps of 10 μg ., omitting the 90 step. This is equivalent to 1 to 10mg./l. of PO_4 based on a 10ml. sample.

METHOD

a) *ortho*-Phosphate

1. Measure 10ml., or other suitable volume of filtered sample, not exceeding 50ml, into a 100ml. volumetric flask or measuring cylinder. Dilute to about 75ml. with deionised water and add 10ml. of the Ammonium Molybdate solution (Reagent 5) and 0.1 g. Ascorbic Acid (Reagent 6). Dilute to 100ml. with deionised water, mix thoroughly and allow to stand for 15 minutes at a temperature between 10 and 30°C. this is the “test” solution.
2. At the same time carry out a “blank” determination using deionised water in place of the sample, still adding the reagents in the amounts given in 1 above.
3. Fill a 50ml. Nessler cylinder to the mark with the “test” solution and place it in the right-hand side of a Lovibond Nessleriser 2150. Fill another cylinder with the “blank” solution and place it in the left-hand side.

Place the Nessleriser facing North daylight or failing this, use the Lovibond Daylight 2000 unit and compare the solution with the disc, rotating the latter until a colour match is obtained.

$$\textit{ortho}\text{-Phosphate as PO}_4 = \frac{\text{Disc reading in } \mu\text{g.}}{\text{Vol. of sample in ml.}} \text{ mg./l.}$$

b) Total Inorganic Phosphate

1. Transfer a volume of filtered sample, not exceeding 50ml., into a 150ml. beaker and dilute to 50ml. with deionised water. Add 50ml. of deionised water to another beaker to act as a "blank". To each beaker add 10ml. 0.5M Sulphuric Acid and boil gently for 20 minutes, maintaining the volume above 25ml. by addition of deionised water as necessary.
2. Cool, add 2 drops of Phenolphthalein indicator followed by 1M Sodium Hydroxide until a faint pink colour persists. Transfer both solutions to volumetric flasks or measuring cylinders and dilute to about 75ml. with deionised water and add 10ml. of Ammonium Molybdate solution (Reagent 5) and 0.1g. Ascorbic Acid (Reagent 6) and mix. Dilute to the 100ml. mark with deionised water, mix thoroughly and allow to stand for 15 minutes at a temperature between 10°C and 30°C.
3. Transfer 50ml. volumes of the solutions to Nessler cylinders and match against the disc as in a) above.

$$\text{Total Inorganic Phosphate as PO}_4 = \frac{\text{Disc reading in } \mu\text{g.}}{\text{Vol. of sample in ml.}} \text{ mg./l.}$$

c) Total Phosphorus

1. Transfer a volume of filtered sample, not exceeding 50ml. into a 150ml. beaker and dilute to 50ml. with deionised water. Add 50ml. of deionised water to another beaker to act as a "blank". To each beaker add 10ml. 0.5M Sulphuric Acid and 0.1g. Ammonium Persulphate and boil gently for 20 minutes, maintaining the volumes above 25ml. by the addition of deionised water as necessary.
2. Cool and add 2 drops of Phenolphthalein indicator followed by 1M Sodium Hydroxide until a faint pink colour persists. Transfer both solutions to volumetric flasks or measuring cylinders, dilute to about 75ml. with distilled water and add 10ml. of Ammonium Molybdate solution (Reagent 5) and 0.1g. Ascorbic Acid (Reagent 6) and mix. Dilute to 100ml. with deionised water, mix thoroughly and allow to stand for 15 minutes at a temperature between 10°C and 30°C.
3. Transfer 50ml. of each of the the solutions to Nessler cylinders and match the disc as in a) above.

$$\text{Total Phosphorus as PO}_4 = \frac{\text{Disc reading in } \mu\text{g.}}{\text{Vol. of sample in ml.}} \text{ mg./l.}$$

IMPORTANT NOTE

The degradation of some Organophosphorus compounds may be incomplete if the test portion contains more than 2mg. organic matter or 0.1g. chloride as NaCl. Recovery of product present can be checked by analysing portions of sample with and without known additions of product.

1. Acknowledgement is made to Nalfloc Limited for assistance in preparing this disc and for permission to quote their Laboratory methods.
2. Derived Values:

Condensed Phosphate (*poly*-phosphates) = Total Inorganic Phosphate – *ortho*-phosphate

Organophosphorus = Total Phosphorus - Total Inorganic Phosphate

OR

In the absence of condensed Phosphate:

Organophosphorus = Total Phosphorus – *ortho*-phosphate

REVISION HISTORY

Date	Change Note	Issue
18/06/02	36/460	2
11/04/05	CA243	3
22/11/07	JC129	4