

## PHOSPHATE METHOD 4

### Using Hydroquinone and Ammonium Molybdate

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#### PRINCIPLE OF THE METHOD

The test consists of adding ammonium molybdate to an acid solution of the phosphate. The ammonium phosphomolybdate formed is then reduced to a lower state of oxidation resulting in a blue-coloured compound said to have the composition  $(\text{MoO}_2 \cdot 4\text{MoO}_3)_2 \cdot \text{H}_3\text{PO}_4$ . The reducing agent used in this method is hydroquinone (1, 4-dihydroxybenzene).

#### REAGENTS REQUIRED

1. **Sulphuric Acid solution.** To about 500ml. of deionised water slowly add 65ml. of concentrated sulphuric acid, cool and dilute the mixture to exactly 1 litre.
2. **Acid-Molybdate Solution.** Dissolve **without heating** 8.8g. of ammonium molybdate  $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O})$  in about 100ml. of deionised water. To about 300ml. of deionised water carefully add 30ml. concentrated sulphuric acid. Cool, then add the diluted acid to the ammonium molybdate solution and make up to exactly 500ml. with deionised water.
3. **Hydroquinone Solution. Either; - Dissolve completely** 5g. of hydroquinone in 500ml. of deionised water and then add 0.3ml. of concentrated sulphuric acid.  
**Or: -** Dissolve one quinol tablet in 50ml. of 0.01M sulphuric acid.  
This solution slowly darkens in colour, but will keep for about 3 weeks in an amber glass bottle in the dark. Quinol (Hydroquinone) tablets may be obtained from chemical suppliers e.g. VWR International.
4. **Carbonate/Sulphite.** Dissolve 130g. of anhydrous potassium carbonate  $(\text{K}_2\text{CO}_3)$  and 24g. of sodium sulphite  $(\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O})$  in 500ml. deionised water. This is stable up to 6 months.

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

#### THE STANDARD LOVIBOND COMPARATOR DISC 3/12

The disc covers the range 0 to 80mg /l. of phosphate as  $\text{PO}_4$  in steps of 10mg. based on a 5ml. sample. The equivalent in terms of phosphorus (P) is 0 to 26.1mg./l.

#### METHOD

1. Filter the sample to give a clear filtrate so that only soluble phosphate is determined.
2. Measure two 5ml. volumes of filtrate and add to two 50ml. Nessler cylinders or measuring cylinders. Label these as “test” and “blank”.
3. To the “test” sample add 2ml. of molybdate solution (reagent 2), swirl to mix then add 1ml. of hydroquinone solution (reagent 3) and swirl to mix.
4. To the “blank” sample add 2ml. of sulphuric acid (reagent 1), swirl to mix then add 1ml. of hydroquinone solution (reagent 3) and swirl to mix.
5. Adjust the temperature of both solutions to  $25^\circ\text{C} \pm 2^\circ\text{C}$  and allow to stand for 5 minutes.
6. During this time measure 2ml. of the carbonate/sulphite solution (reagent 4) into two more Nessler cylinders or measuring cylinders.

7. At the end of the 5 minute standing period pour the “test” solution into one of the carbonate/sulphite solutions, pouring backwards and forwards to mix it thoroughly. Pour the “blank” solution into the other carbonate/sulphite solution and mix in the same way.
8. Pour both solutions into each of two 13.5mm./10ml. moulded cells and then place these in the Comparator. The “blank” cell in the left-hand compartment and the “test” cell in the right-hand compartment.
9. **IMMEDIATELY** compare the colour against the disc by holding the Comparator facing a source of white light e.g. a Lovibond Daylight 2000 Cabinet or, failing this, North daylight. Rotate the disc until the nearest colour match is obtained.
10. The figure displayed in the bottom right-hand corner of the Comparator is the concentration of Phosphate as  $\text{PO}_4$  in mg./l. when 5ml. of the sample is taken for the test.

## **REVISION HISTORY**

<b>Date</b>	<b>Change Note</b>	<b>Issue</b>
17/12/02	36/460	2
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