

Determination of Phosphate in Water – Method 1 (High Level)

1. SCOPE

The method can be used for water samples up to 75ml in size containing from 100 to 1,000 micrograms of phosphate (PO_4). Method 2 should be used for smaller concentrations of phosphate.

2. PRINCIPLE

The method is based on the formation of a yellow vanadomolybdi-phosphoric acid from orthophosphates. Total phosphate is determined after hydrolysis of polyphosphates to orthophosphate.

3. REAGENTS

Molybdate-vanadate reagent. Dissolve 20g of ground ammonium molybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, in 400ml of warm water (Solution A). Dissolve 1g of ammonium metavanadate in about 300ml of warm water, cool and slowly add, with stirring, 140ml of concentrated nitric acid (Solution B). Add Solution A to Solution B, stirring continuously, and dilute to 1 litre with water. Store in a polyethylene bottle. The reagent is stable for at least 1 month. Dilute nitric acid. Dilute 200ml of concentrated nitric acid ($d_{20} = 1.42$) to 1 litre with water. Store in a polyethylene bottle. Phosphate standard solution. Dry a sufficient quantity of potassium dihydrogen orthophosphate for one hour at $100-105^\circ\text{C}$. Cool in a desiccator, weigh out 1.433g, dissolve in water and dilute to 1 litre. Store in a polyethylene bottle. Phosphate working solution. Dilute 10ml of the phosphate standard solution to 100ml in a one mark volumetric flask. 1ml contains 100 micrograms of phosphate (PO_4).

4. APPARATUS

100ml volumetric flasks; 150ml beakers; spectrophotometer for operation at 420nm; 4cm spectrophotometer cells.

5. PROCEDURE

5.1 *Preparation of calibration graph*

Into each of a series of 100ml volumetric flasks measure volumes of the phosphate working solution containing 0, 200, 400, 600, 800 and 1,000 μ g. Dilute each to about 50ml with water, add 5ml of the dilute nitric acid and mix. Add 10ml of the molybdate vanadate reagent to each, dilute to the 100ml mark and mix well. Adjust the temperatures of the flasks to a known temperature in a spectrophotometer with the wavelength set at 420nm, using a 4cm cell. Use water in the compensating cell. Correct the readings for the blank with no added phosphate and from the net absorbance obtained and the known phosphate contents of the solutions plot the calibration graph. The absorbance given by 1,000 μ g of phosphate in 100ml is approximately 0.7.

5.2 *Determination of orthophosphate*

Transfer a suitable volume (not exceeding 75ml) of the filtered sample to a 100ml of volumetric flask, neutralise any alkalinity with dilute nitric acid and add 5ml in excess. Mix. Add 10ml of the molybdate-vanadate reagent, dilute to the 100ml mark and mix well. Treat 75ml of water in a similar manner. Adjust the temperature of the flasks to within 1°C of the temperature of calibration and allow to stand for 20 minutes, measure the optical densities in the spectrophotometer with the wavelength set at 420nm and using 4cm cells. Use water in the compensating cell. If it is necessary to compensate for colour in the sample under test, measure an equal volume of the sample and proceed as before but using 7ml of dilute nitric acid instead of the molybdate-vanadate reagent. Measure the optical density against water, deduct the readings obtained on the blank and the colour compensating solutions and read off the phosphate content in microgrammes in the sample from the calibration graph.

5.3 *Determination of total phosphate*

Transfer a suitable volume (not exceeding 75ml) of the filtered sample to a 150ml beaker and dilute, if necessary, to about 75ml with water.

Neutralise any alkalinity with the dilute nitric acid and add 5ml in excess. Then boil gently for 30 minutes covering the beaker with a watch glass to prevent undue evaporation. Cool, transfer completely to a 100ml volumetric flask, add 10ml of the molybdate-vanadate reagent, dilute to the mark and mix well. Treat 75ml of water in a similar manner. Adjust the temperature of the flasks to within 1°C of the temperature of calibration and allow to stand for 10 minutes. Then measure the optical densities in the spectrophotometer with the wavelength set at 420nm and using 4cm cells. Use water in the compensating cell.

If it is necessary to compensate for colour in the sample under test, measure an equal volume of the sample and proceed as before but using 7ml

of dilute nitric acid instead of the molybdate-vanadate reagent. Measure the optical density against water, deduct the readings obtained on the blank and the colour compensating solutions and read off the phosphate content in microgrammes in the sample from the calibration graph.

6. EXPRESSION OF RESULTS

Results are expressed as mg of phosphate (PO_4) per litre.

7. REFERENCES

British Standard 2690, Part 3, p.12, 1966.