

## Determination of Phosphate in Water – Method 2 (Low Level)

### 1. SCOPE

The method can be used for water samples up to 50ml in size containing from 5 to 100 micrograms. Method 1 should be used for higher concentrations of phosphate.

### 2. PRINCIPLE OF METHOD

The method is based on the reaction of orthophosphate in acid solution with ammonium molybdate to form phosphomolybdic acid and the subsequent reduction of this by stannous chloride to the molybdenum blue complex. Total phosphate is determined after hydrolysis of polyphosphates to orthophosphates.

### 3. REAGENTS

#### 3.1 *Ammonium molybdate sulphuric acid mixed reagent*

Dissolve 25g of ground ammonium molybdate in approximately 200ml of water without heating (Solution A). Add 280ml of concentrated sulphuric acid to 400ml of water and cool (Solution B). Add Solution A to Solution B, cool, and dilute to 1 litre. Store in a polyethylene bottle.

#### 3.2 *Stannous chloride*

Dissolve 2.5g of stannous chloride in glycerol and dilute to 100ml with glycerol. The solution is stable for several weeks. Phosphate standard solution. Dry a sufficient quantity of potassium dihydrogen orthophosphate for one hour at 100-105°C. Cool in a dessicator, weight out 1.433g, dissolve in water and dilute to 1 litre. Store in a polyethylene bottle.

#### 3.3 *Phosphate working solution*

Dilute 5ml of the phosphate standard solution to 500ml in a volumetric flask. 1ml contains 10 micrograms of phosphate ( $\text{PO}_4$ ).

#### 3.4 *Dilute nitric acid*

Dilute 200ml of concentrated nitric acid to 1 litre with water. Store in a polyethylene bottle.

### 3.5 Sodium hydroxide

5% w/v solution. Store in a polyethylene bottle.

## 4. APPARATUS

100ml standard flasks; spectrophotometer for operation at 690nm; 4cm spectrophotometer cells.

## 5. PROCEDURE

### 5.1 Preparation of calibration graph

Measure accurately volumes of the phosphate working solution containing 0, 10, 20, 40, 60, 80 and 100 $\mu$ g phosphate into a series of 100ml standard flasks and add water to make the volume of solution in each flask 50ml. Add 2.5ml of the ammonium molybdate reagent and mix. Add 4 drops of stannous chloride from a dropping pipette, mix and dilute to the mark. Allow to stand for 10 to 12 minutes. Measure the absorbance of the solution at a known temperature between 20°C and 25°C in a spectrophotometer at the wavelength corresponding to maximum absorption (approximately 690nm, but the exact wavelength should be checked for each spectrophotometer) using a 4mm cell. Use water in the compensating cell. Deduct the reading for the blank with no added phosphate and plot a calibration graph of optical density against known phosphate content. The absorbance given by 100 $\mu$ g in 100ml is approximately 1.1 and increases by about 1% per 1°C temperature rise.

### 5.2 Determination of orthophosphate

Transfer a suitable volume (containing less than 100 $\mu$ g phosphate) of the filtered sample into a 100ml volumetric flask and dilute to 50ml if necessary. To a second 100ml volumetric flask add 50ml of water. Treat both in a similar manner. Add 2.5ml of ammonium molybdate reagent and mix. Add 4 drops of stannous chloride reagent from a dropping pipette, mix and dilute to the mark. Allow to stand for 10 to 12 minutes. Measure the absorbances of the solutions at a temperature within 1°C of that at which the calibration graph is prepared in the spectrophotometer using the wavelength already determined and 4cm cells. Use water in the compensating cell. If it is necessary to compensate for colour of the sample under test, measure an equal volume of the sample and proceed as before but using 5ml of dilute nitric acid instead of the molybdate sulphuric acid reagent. Measure the absorbance of the solution against water, deduct the readings obtained on the blank and the colour compensating solutions and read off the phosphate content in microgrammes from the calibration graph.

### 5.3 *Determination of total phosphate*

Transfer a suitable volume (containing not more than 100 $\mu$ g PO<sub>4</sub>) of the filtered sample to a 150ml beaker. Dilute to 50ml if necessary. To a second 150ml beaker add 50ml of water. Treat both in a similar manner. Add 5ml of dilute nitric acid and boil gently for 30 minutes covering the beaker with a watch glass to prevent undue evaporation. Cool and neutralise with sodium hydroxide, 5% w/v, to pH7. Transfer the solution to a 100ml volumetric flask. Dilute to about 50ml if necessary. Add 2.5ml of ammonium molybdate and proceed as given for orthophosphate.

## 6. EXPRESSION OF RESULTS

Results are expressed as mg of phosphate (PO<sub>4</sub>) per litre.

## 7. REFERENCES

British Standard 2690 Part 3, p.14, 1966.