

## Control Method Template

(Ref. SOP QMS-030)

### 4. Nitrate (BP method)

#### Reagents

1. 10% w/v solution of Potassium chloride in Distilled water
2. Diphenylamine,  $C_{12}H_{11}N$  [MWt 169.2]
3. Sulphuric acid concentrated
4. Distilled water (nitrate - free)
5. Potassium nitrate,  $KNO_3$  [MWt 101.1] AR grade
6. Diphenylamine solution  
Dissolve 0.1 g of Diphenylamine in 100 mL of concentrated Sulphuric acid

#### Preparation of Nitrate (0.2 ppm) Standard Solution

##### Nitrate standard solution (100 ppm $NO_3$ )

Dilute 1 mL of a 0.163% w/v solution of Potassium nitrate to 10 mL with water.

##### Nitrate standard solution (10 ppm $NO_3$ )

Dilute 1 mL of nitrate standard solution (100 ppm  $NO_3$ ) to 10 mL with water.

##### Nitrate standard solution (2 ppm $NO_3$ )

Dilute 1 mL of nitrate standard solution (10 ppm  $NO_3$ ) to 5 mL with water.

##### Nitrate standard solution (0.2 ppm $NO_3$ )

Dilute 0.5 mL of nitrate standard solution (2 ppm  $NO_3$ ) with 4.5 mL of Nitrate-free water to 5 mL.

#### Procedure

Pipette 5 mL of sample into a test tube and immerse in ice. Add 0.4 mL of a 10% w/v solution of Potassium chloride, 0.1 mL of Diphenylamine solution and dropwise with shaking add 5 mL of concentrated Sulphuric acid. Transfer the test tube to a water bath at 50°C and allow to stand for 15 minutes.

Any blue colour in the sample solution is not more intense than that in the Nitrate standard solution (0.2 ppm  $NO_3$ ) prepared at the same time and in the same manner.

### 5. Sulphate

#### Reagents

1. 2M Hydrochloric acid
2. Barium chloride,  $BaCl_2 \cdot 2H_2O$  [MWt 244.27]