

Spectrophotometric Determination of



1. INTRODUCTION

Nitric acid and nitrates in air can be measured conveniently by means of the chromotropic acid (9.1) or brucine (9.2) methods. Likewise, mixtures of nitric oxide, nitrous anhydride and nitrogen tetroxide can be converted to equivalent nitrate and estimated as such (9.3). Other methods such as the phenol-disulfonic acid procedure, reduction to nitrite with subsequent measurement by means of a modified Griess reaction or the use of ultra-violet spectrophotometry have been proposed but are not recommended for general use because of lack of specificity or sensitivity or both. The chromotropic acid method is chosen in preference to the brucine procedure because the latter may suffer from interferences due to oxidants, other than nitrate, that may be present in the air.

2. SCOPE AND FIELD OF APPLICATION

This is a method for determining trace quantities of nitrate in the air suitable for establishing ambient air quality for spot checks, monitoring, or field surveys. Reliable results can be obtained over the range of 0.5-50 μ g of nitrate. Interferences due to substances such as nitrite, oxidizing agents, chloride and iron (III) which normally interfere in nitrate determinations have been eliminated.

3. PRINCIPLE

The method is based on the reaction of nitrate with chromotropic acid. The absorbance of the resulting yellow solution is proportional to the nitrate concentration. Potential interferences have been eliminated. Addition of excess sodium sulfite removes the interference due to oxidizing agents, and addition of urea eliminates nitrite as nitrogen gas. Chloride is masked by the addition of antimony (III).

4. REAGENTS

4.1 Standard nitrate solution

Dissolve 1.371g of reagent grade sodium nitrate in distilled water and dilute to one liter. This gives a nitrate concentration of 1mg/ml. A series of standards can be prepared by suitable dilutions.

4.2 Sulfuric acid

Use analytical reagent grade concentrated acid which is free from nitrate. (Readily checked by adding 1ml reagent to 10ml sulfuric acid. No color should be formed.)

4.3 Purified chromotropic acid

Prepare a saturated solution of the disodium salt of 1,8-dihydroxy-3,6-naphthalene disulfonic acid. Process twice using decolorizing charcoal. Crystallize the reagent from the filtered solution by adding sulfuric acid. Filter; wash several times with ethanol; and dry below 80°C. Prepare a 0.1% solution of the reagent in concentrated sulfuric acid. The reagent solution is colorless and is stable for two weeks.

4.4 Sulfite – urea solution

Dissolve 5g of urea and 4g of reagent grade anhydrous sodium sulfite and dilute to 100ml with distilled water.

4.5 Antimony solution

Heat 0.5g of antimony metal in 80ml of concentrated sulfuric acid until all the metal is dissolved. Cool the solution and add to 20ml of ice water. When kept overnight, if any salt crystallizes, redissolve by heating.

5. APPARATUS

5.1 High volume sampler

Spectrophotometer suitable for measurement of absorbance at 410nm and equipped with matched quartz cells.

6. SAMPLING

A high volume sampler is used for sample collection. Since nitrates are soluble in water, they can be eluted from the filter paper by repeated washing with a small amount of distilled water. Combine the washings and make up to 100ml with distilled water.

7. PROCEDURE

Pipet 2.5ml of each sample or standard solution into dry 10ml volumetric flasks. To each flask add 1 drop of sulfite-urea solution; place the flasks in a