

METHOD #: 354.1 Approved for NPDES (Issued 1971)
TITLE: Nitrogen, Nitrite (Spectrophotometric)
ANALYTE: CAS # N Nitrogen 7727-37-9
NO₂ Nitrite
INSTRUMENTATION: Spectrophotometer
STORET No. Total 00615

1.0 Scope and Application

- 1.1 This method is applicable to the determination of nitrite in drinking, surface and saline waters, domestic and industrial wastes.
- 1.2 The method is applicable in the range from 0.01 to 1.0 mg NO₂-N/L.

2.0 Summary of Method

- 2.1 The diazonium compound formed by diazotation of sulfanilamide by nitrite in water under acid conditions is coupled with N-(1-naphthyl) ethylenediamine dihydrochloride to produce a reddish-purple color which is read in a spectrophotometer at 540 nm.

3.0 Sample Handling and Preservation

- 3.1 Samples should be analyzed as soon as possible. They may be stored for 24 to 48 hours at 4°C.

4.0 Interferences

- 4.1 There are very few known interferences at concentrations less than 1,000 times that of the nitrite; however, the presence of strong oxidants or reductants in the samples will readily affect the nitrite concentrations. High alkalinity (>600 mg/L) will give low results due to a shift in pH.

5.0 Apparatus

- 5.1 Spectrophotometer equipped with 1 cm or larger cells for use at 540 nm.
- 5.2 Nessler tubes, 50 mL or volumetric flasks, 50 mL.

6.0 Reagents

- 6.1 Distilled water free of nitrite and nitrate is to be used in preparation of all reagents and standards.
- 6.2 Buffer-color reagent: To 250 mL of distilled water, add 105 mL conc. hydrochloric acid, 5.0 g sulfanilamide and 0.5 g N-(1-naphthyl) ethylenediamine dihydrochloride. Stir until dissolved. Add 136 g of sodium

acetate (CH₃COONa•3H₂O) and again stir until dissolved. Dilute to 500 mL with distilled water. This solution is stable for several weeks if stored in the dark.

- 6.3 Nitrite stock solution: 1.0 mL = 0.10 mg NO₂-N. Dissolve 0.1493 g of dried anhydrous sodium nitrite (24 hours in desiccator) in distilled water and dilute to 1000 mL. Preserve with 2 mL chloroform per liter.
- 6.4 Nitrite standard solution: 1.0 mL = 0.001 mg NO₂-N. Dilute 10.0 mL of the stock solution (6.3) to 1000 mL.

7.0 Procedure

- 7.1 If the sample has a pH greater than 10 or a total alkalinity in excess of 600 mg/L, adjust to approximately pH 6 with 1:3 HCl.
- 7.2 If necessary, filter the sample through a 0.45 u pore size filter using the first portion of filtrate to rinse the filter flask.
- 7.3 Place 50 mL of sample, or an aliquot diluted to 50 ml, in a 50 mL Nessler tube; hold until preparation of standards is completed.
- 7.4 At the same time prepare a series of standards in 50 mL Nessler tubes as follows:

mL of Standard Solution 1.0 mL = 0.001 mg NO ₂ -N	Conc., When Diluted to 50 ml, mg/L of NO ₂ -N
0.0	(Blank)
0.5	0.01
1.0	0.02
1.5	0.03
2.0	0.04
3.0	0.06
4.0	0.08
5.0	0.10
10.0	0.20

- 7.5 Add 2 mL of buffer-color reagent (6.2) to each standard and sample, mix and allow color to develop for at least 15 minutes. The color reaction medium should be between pH 1.5 and 2.0.
- 7.6 Read the color in the spectrophotometer at 540 nm against the blank and plot concentration of NO₂-N against absorbance.

8.0 Calculation

- 8.1 Read the concentration of NO₂-N directly from the curve.
- 8.2 If less than 50.0 mL of sample is taken, calculate mg/L as follows:

$$\text{NO}_2 - \text{N, mg/L} = \frac{\text{mg/L from std. curve} \times 50}{\text{mL sample used}}$$

9.0 Precision and Accuracy

9.1 Precision and Accuracy data are not available at this time.

Bibliography

1. Standard Methods for the Examination for Water and Wastewater, 14th Edition, p 434, Method 420, (1975).