

Nitrate Analysis

Reagents:

1. Concentrated NH_4Cl : 100 g NH_4Cl per 500 ml DI water
2. Diluted NH_4Cl : Dilute 50 ml of concentrate NH_4Cl to 2 L of Volumetric flask with DI water
3. Diazotizing reagent: 0.5g sulfanilamide in 100 ml of 2.4 M HCl, store it in refrige.
4. Coupling reagent: Dissolve 0.3 g of N-(1-naphthyl)-ethylenediamine dihydrochloride in 100 ml of 0.12 M HCl. Store it in brown bottle in refrigerator.
5. Standard nitrate solution: 0.3609 g of KNO_3 /1 L, then dilute 20 ml of this solution to 500 ml that contains 2 μg of $\text{NO}_3\text{-N}$ ml^{-1} .

Preparation of Cd column before carrying out $\text{NO}_3\text{-}$ analyses every time:

1. Add 1ml Concentrated NH_4Cl , and use the outlet to lower the level until it is even with the top of the column.
2. Add 75 ml of dilute NH_4Cl to the upper reservoir.
3. Allow NH_4Cl to flow through the column at a rate of 110 ml/min until even to the top of Cd beads.

Analysis the samples:

1. Open the stopcock to drain excess NH_4Cl from the column until the liquid level is even with the top of the column.
2. Pipette 1 ml of concentrated NH_4Cl onto the top of the column.
3. Pipette an aliquot of the sample (2-5 ml) onto the column. Record the volume of sample used.
4. Place a 100 ml volumetric flask under the outlet stopcock. Open the stopcock to allow the sample to drain into the cadmium column. Immediately rinse the inside of the reservoir with 2 mL diluted NH_4Cl .
5. Add 75 mL dilute NH_4Cl to to the upper reservoir of the cadmium column. Then open the stopcock to let the liquid flow through the column at a rate of 110 ml/min (1.83 ml/sec) until the level of liquid is about 1 cm above the cadmium granules. It should take about 42 seconds for the liquid to drain to the top of the cadmium granules.
6. Add 2 ml of diazotizing reagent to the contents of the flask and mix thoroughly. Let stand for 5 minutes.
7. Add 2 ml coupling reagent and then bring to volume with DI water. Mix thoroughly.
8. Measure absorbance at 540 nm.

Preparation standard solution:

Pipette 1 ml concentrated NH_4Cl onto the top of the column, pipette 0, 1, 2, 3, 5, and 7.5 ml of 2 $\mu\text{g}/\text{ml}$ of $\text{NO}_3\text{-N}$ to Cd column, and then follow step 5-8.

Note: check for air bubbles, never let the liquid fall below the top of Cd level.

Reference:

Dorich and Nelson, 1984. p:1155-1157. *In* D. L. Sparks. Methods of soil analysis. Part 3-chemical methods. ASA and SSSA, Madison, WI.