

Nitrogen (Total/Kjeldahl)

1. Application

This method covers the digestion of samples for Nitrogen (Total/Kjeldahl)

2. Summary of Methods

Total nitrogen (Org N + NH₄-N + NO₃-N, NO₂-N) digested with sulfuric acid, metal catalyst, salicylic acid.

Total Kjeldahl Nitrogen (Org N + NH₄-N) digested with sulfuric acid and metal catalyst.

3. Safety

Each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should be made available to all personnel involved in the chemical analysis.

4. Interferences

- 4.1 Samples must not consume more than one fifth of the sulfuric acid during the digestion. The buffer will accommodate a range of 5.7 to 7.0% (v/v) H₂SO₄ in the diluted digestion sample without any change in signal intensity.
- 4.2 Samples with particles remaining after digestion will require filtering prior to analysis by FIA.

5. Sample Collection, Preservation and Handling

- 5.1 Soil and plant samples are dried at 55°C, 65°C, respectively. The dried soil is then ground to pass a 12 mesh screen and plant tissue is ground to pass a 2 mm screen.
- 5.2 Water sample are stored at 4°C.

6. Apparatus and Materials

- 6.1 Scale 0.001 g
- 6.2 QuickChem 8000 Automated Ion Analyzer
- 6.3 Block Digester (Easy digest 40/20) Westco Scientific Instruments
- 6.4 75 ml digestion tubes
- 6.5 Vortex Mixer

7. Reagents (FIA 7.1-7.4) (N Digestion 7.5-7.7)

Flow Injection:

- 7.1 Buffer – Dissolve 65.0 sodium hydroxide, 50.0 g sodium potassium tartrate and 26.8 g sodium phosphate dibasic heptahydrate in deionized water (10 megohm) and dilute to 1 liter. Degas the buffer solution by passing He at 140 kPa through a helium degassing tube for one minute.
- 7.2 Color Reagent – Dissolve 150.0 g sodium salicylate and 1.0 g sodium nitroprusside in deionized water and dilute to 1 liter. Degas the solution with He.
- 7.3 Hypochlorite Solution – In a 1 L volumetric flask, add 60.0 ml regular Chlorox bleach (5.25% sodium hypochlorite), dilute to 1 L with deionized water.
- 7.4 Carrier – In a 1 L volumetric flask containing approximately 600 ml deionized water, add 70.0 ml of sulfuric acid, 30.0 g of potassium sulfate, and 2.5 g of copper sulfate. Dilute to 1 L with deionized water.

Nitrogen Digestion:

- 7.5 Conc. H₂SO₄
- 7.6 Metal Catalyst (digestion tablet – potassium sulfate 93%, cupric sulfate 7%)
- 7.7 Salicylic acid (75 g salicylic acid/2.5 L H₂SO₄) is used when including NO₃-N + NO₂-N

8. Methods

- 8.1 Weigh out 0.15-0.20 g of dried plant tissue or 0.45-0.5 g of soil into a clean, dry digestion tube. Carry a (LRB) blank through all steps of the procedure (see 10.1).
- 8.2 For Total Kjeldahl N (Org N + NH₄-N): To each tube add 1 (metal catalyst) digestion tablet and 3.5 ml of concentrated H₂SO₄.
- 8.3 For Total N (Org N + NH₄-N + NO₃-N + NO₂-N): To each tube add 1 (metal catalyst) digestion tablet and 3.5 ml of H₂SO₄ with Salicylic acid.
- 8.4 Place tubes in a block digester. Set temperature 160°C and time 1 to 20 minutes. Set temperature to 380°C and time 240 minutes.
- 8.5 Remove the samples from the block and allow 15 minutes for cooling.
- 8.6 Fill with deionized water to 50.0 ml. If samples are not run immediately, they should be covered to prevent evaporation.
- 8.7 Transfer \approx 7 ml of digested solution to FIA tubes.
- 8.8 Determine the ammonium concentration by FIA.

9. Calculations

The nitrogen content is calculated using the formula:

$$\text{ppm N} = 50/W_S X C_D \quad (\text{for soil sample})$$

$$\% \text{ N} = 50/W_S X C_D / 10,000 \quad (\text{for plant sample})$$

where W_S = Weight of sample (g)
 C_D = Concentration in the digest (mg N/l)

10. Quality Control

- 10.1 Laboratory Reagent Blank (LRB) – At least one LRB must be analyzed with each batch of samples in order to assess contamination from the laboratory environment. If LRB values exceed the method detection limit, laboratory or reagent contamination should be suspected, take correction action before continuing the analysis.
- 10.2 Laboratory Fortified Blank (LFB) – At least one LFB must be analyzed with each batch of samples. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required control limits of 90-110%, the analyte is judged out of control, take corrective action for continuing analysis.
- 10.3 Instrument Performance Check Solution (IPC) – For all determinations, a mid-range check standard and a calibration blank must be analyzed immediately after daily instrument calibration, after every tenth sample, and at the end of the sample run. This process verifies that the instrument is within 10% of calibration. If the IPC solution indicates that the calibration is outside of present limits, take corrective action before continuing analysis.

11. Reporting

- 11.1 Data is reported as mg/l of N for soil and % N for plant tissue on a dry weight basis.
- 11.2 Detection limit = 0.01 mg/l

12. References

- 12.1 Lachat Instruments 1995. Total Kjeldahl Nitrogen in Soil/Plant. QuikChem Method 13-107-06-2-D.
- 12.2 Jaromir Ruzicka 1983. Flow Injection Analysis – From Test Tube to Integrated Microconduits. Analytical Chemistry 55: 1040A-1053A.
- 12.3 Bremner, J. M. 1965. Total nitrogen. In C. A. Black et al. (ed.) Methods of soil analysis, Part 2. Agronomy 9:1149-1178. Am. Soc. Of Agron., Inc., Madison, Wis.