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Nitrogen (Total/Kjeldahl)

1. Application (analytes and matrices)

This method covers the digestion and analysis of plant tissue, soil and other aqueous and solid materials for Nitrogen (Total/Kjeldahl).

2. Method Sensitivity

1.0 to 100 mg N/L in digest, 0.03 to 2.5% N in plant tissue and 0.01 to 1.25% N in soil
2.0 – 20 mg N/L in aqueous samples.

3. Summary of Methods

Total nitrogen (Org N + NH_4-N + NO_3-N, NO_2-N) digested with sulfuric acid, metal catalyst and salicylic acid.
Total Kjeldahl Nitrogen (Org N + NH_4-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. Potential 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) H_2SO_4 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 Aqueous samples are stored at 4°C.

6. Apparatus and Analytical Instruments

6.1 Scale 0.001 g
6.2 Lachat QuickChem 8000 Flow Injection Analyzer (FIA)
6.3 Block Digestor (Easy digest 40/20) Westco Scientific Instruments
6.4 75 ml digestion tubes
6.5 Vortex Mixer

7. Consumables, Standards and Reagents

Sample Digestion:

7.1 Conc. H_2SO_4
7.2 Metal Catalyst (digestion tablet – potassium sulfate 93%, cupric sulfate 7%)
7.3 Salicylic acid (75 g salicylic acid/2.5 L H_2SO_4) is used when including NO_3-N + NO_2-N.

Flow Injection analyzer

7.4 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.
7.5 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 Helium gas.
7.6 Hypochlorite Solution – In a 1 L volumetric flask, add 60.0 ml regular Chlorox bleach (5.25% sodium hypochlorite), dilute to 1 L with de-ionized water.
7.7 Carrier – In a 1 L volumetric flask containing approximately 600 ml de-ionized 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 de-ionized water.

8. Procedures for digestion

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 + NH_4-N): To each tube add 1 (metal catalyst) digestion tablet and 3.5 ml of concentrated H_2SO_4.
8.3 For Total N (Org N + NH_4-N + NO_3-N + NO_2-N): To each tube add 1 (metal catalyst) digestion tablet and 3.5 ml of H_2SO_4 with Salicylic acid.
8.4 Place tubes in a block digestor. 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 de-ionized water to 50.0 ml. If samples are not run immediately, they should be covered to prevent evaporation.
8.7 Transfer $\underset{\sim}{}$ 7 ml of digested solution to FIA tubes.
8.8 Determine the ammonium concentration by flow injection analyzer.

9. Calibration, Standardization , Analysis and Calculations

9.1 The instrument is calibrated using a set of 7 calibration standards of the following concentrations 100, 50, 25, 10, 5, 1, 0 mg/L of carrier solution. The standards are prepared from primary stock standards purchased from an outside vendor. Date of preparation, lot number, initials of the person who prepared the standards and expiration date of standards are marked on the standards containers. Working standards are kept in a fridge at 0-4ºC.

9.2 Correlation coefficient of standard curve of calibrators must be at least 0.995 for the calibration test to pass.

9.3 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/I)

10. Quality Control

10.1 Method Blank (MB) – At least one MB must be analyzed with each batch of samples in order to assess contamination from the laboratory environment. If MB values exceed the method detection limit, laboratory or reagent contamination should be suspected, take correction action before continuing the analysis.

10.2 Laboratory Control Sample (LCS) – At least one LCS must be analyzed with each batch of sample. Calculate accuracy as percent recovery. If the recovery of the analyte falls outside the required control limits of 90-110%, the analyte is judged out of control, take corrective action before continuing analysis.

10.3 Replicates and Spikes_ At least 10% of samples of a sample batch are replicates and spikes. If spikes and replicates do not fall within the specified limits of ±10% of true value then re-digest and analyze sample batch including QC samples.

10.3 Continuing Calibration Verification (CCV) – For all determinations, a mid-range check standard 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 CCV standard indicates that the calibration is outside of present limits, take corrective action before continuing analysis.

11. Data Assessment and Reporting of Results

11.1 Data are accepted when quality control samples fall within the set limits. Corrective actions of re-digestion and/or re-analyzing of samples and quality control samples will take place to handle out of control data.

11.2 Data are reported as mg/l of N for water and soil and as a % N for plant tissue on a dry weight basis.

12. References

12.1 EPA method 351.2 Determination of Total Kjeldahl Nitrogen by semi-automatic colorimetry. Revision 2.0. August 1993.