

## **Ammonia Nitrogen in Soil, Plant Tissue, and Water**

### **1. Application (analytes and matrices)**

In this procedure nitrogen, in the form of ammonia ions, in water, soil, sludges, sediments or plant tissue samples and analyzed by flow injection analyzer.

### **2. Method Sensitivity**

The approximate working ranges are 0.03 to 2.00 mg/N (as NH<sub>4</sub>)/L in water, and 0.25 - 40 mg/Kg for solids. This range might be extended by diluting the sample prior to analysis.

### **3. Summary of Methods**

KCl is used to extract NH<sub>4</sub><sup>-</sup>-N from the soil and tissue samples. While water samples are analyzed directly without extraction. The ammonium ions in sample are converted into ammonia by in-line neutralization with a concentrated buffer. The ammonia thus produced is heated with salicylate and hypochlorite to produce a blue color which is read at 630 nm.

### **4. Safety and Waste Management**

- 4.1 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.
- 4.2 All laboratory wastes, excess reagents and samples must be disposed of in a manner that is consistent with applicable rules and regulations.
- 4.3 Waste disposal guidelines are described in the University of Wisconsin Chemical Safety and Disposal Guide.

### **5. Sample Collection, Preservation and Handling**

- 5.1 Samples are collected by clients in clean containers and submitted to the laboratory for analysis.
- 5.2 A chain-of-custody form is submitted with the samples. If samples were not preserved by client they will be preserved immediately at the lab. Soil and plant tissue samples are dried at 55C and 65C, respectively.
- 5.3 Drying is followed by grinding and extraction by 2N KCl solution.
- 5.4 Aqueous solutions are preserved by addition of 1 mL of concentrated sulfuric acid per liter of sample and kept in a fridge at 0-5C until analyzed.

## **6. Potential Interferences**

- 6.1 Color and turbidity may interfere with results. Turbidity is removed by manual filtration. Sample color may be corrected for by running the sample through the manifold without the color reagent and subtracting the result obtained from the result using the color reagent.

## **7. Equipment and Analytical Instruments**

- 7.1 Weigh boat (metal or glass)  
7.2 Erlenmeyer flasks (50-ml)  
7.3 Pipette bank (15-ml)  
7.4 Time-controlled, oscillating shaker.  
7.5 Filter paper, 9-cm (Whatman No. 2 or equivalent)  
7.6 Funnel tubes (15-ml)  
7.7 Glass test tubes (6.2-ml)  
7.8 Flow injection analyzer  
7.9 XYZ sampler

## **8. Consumable Supplies, Reagents and Standards**

- 8.1 All reagents and standards should be stored in the appropriate bottles and labeled with the following information: identity, supplier, lot number, date, preparer's initials and concentrations. Use de-ionized water for all solutions.
- 8.2 2 N KCl-solution (1044.40 g of KCl to 7 liters of de-ionized water).
- 8.3 15 N Sodium hydroxide solution (150 g of NaOH added slowly to 250 mL of DI water and swirled until dissolved).
- 8.4 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.
- 8.5 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.
- 8.6 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.

## **9. Method of Extraction and Analysis**

- 9.1 Weigh out 1.50 g of soil or .25 g of tissue into a weigh boat.  
9.2 Transfer sample to a 50-ML Erlenmeyer flask.  
9.3 Add 15-ml of 2 N KCl solution using constant suction pipette.  
9.4 Shake for 15 minutes on oscillating shaker.  
9.5 Filter immediately.  
9.6 Pipette 5-ml of filtrate into glass test tube.

- 9.7 Analyze by flow injection.
- 9.8 The sample preparation procedure is taken from section 7.1.1 of Lachat QuikChem Method 12-107-06-1-A.

## **10 Calibration, Standardization and Calculations**

- 10.1 The instrument is calibrated using a set of 6 calibration standards of the following concentrations 20, 10, 2, 1, .3, 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 preserved in concentrated sulfuric acid and kept in a fridge at 0-4°C.
- 10.2 Correlation coefficient of standard curve of calibrators must be at least 0.995 for the calibration test to pass. Sample concentration is calculated from a regression equation by plotting response versus standard concentration

## **11. Quality Control**

- 11.1 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.
- 11.2 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.
- 11.3 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  $\pm 10\%$  of true value then re-digest and analyze sample batch including QC samples.
- 11.4 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.

## **12. Data Assessment and Reporting of Results**

- 12.1 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.
- 12.2 Data are reported as mg/l of N for water and mg/L N for solids on a dry weight basis.

### **13. References**

- 13.1 EPA method 350.1. Determination of Ammonia Nitrogen by automated colorimetry. Revision 2.0. August 1993.