Urea Nitrogen in Fertilizer Samples

1. Scope:

This procedure is to be used for the analysis of urea nitrogen in fertilizer.

2. Principle:

The amount of urea is determined by analyzing samples for ammoniacal + urea nitrogen. Ammoniacal nitrogen is determined separately and is subtracted from the ammoniacal & urea result. Samples are prepared as described in RA-SP-SMPL-PREP.

3. Safety:

The SDS for all chemicals shall be read before performing any part of this method. Gloves, safety glasses, and a lab coat shall be worn when handling hazardous materials and reagents.

Sulfuric acid is very toxic and extremely corrosive and shall be used in a fume hood. The acidification of samples containing reactive materials may result in the release of toxic gases and can be exothermic. Sample acidification and digestion shall be performed carefully in a fume hood.

4. Equipment, Reagents, and Supplies (equivalents are acceptable):

4.1 Kjeldahl distillation Unit
4.2 Kjeldahl flasks – 800 mL
4.3 Rubber stoppers – Size 7
4.4 Erlenmeyer flasks – 500 mL
4.5 Urease powder (Fisher Scientific cat# U21)
4.6 Magnesium oxide (Fisher Scientific cat# M68-3)
4.7 Calcium chloride solution prepared by adding 25g $CaCl_2$ in a 100mL volumetric flask and filling to the line with water
4.8 2% Boric acid solution with indicator (Fisher Scientific cat# 1064-1)
4.9 Defoaming solution (Spectrum cat# A1302)
4.10 Standardized sulfuric acid solution - 0.5N (Fisher Scientific cat # SA215)
4.11 Phosphate buffer prepared by adding 13.61g KH_2PO_4 and 17.42g K_2HPO_4 in a 200mL volumetric flask and filling to the line with water

5. Sample Analysis

5.1 For samples containing <5% urea, weigh 2g into a labeled Kjeldahl flask (record the weight). For samples containing ≥ 5% urea, weigh 1g of sample.

5.2 Prepare a blank consisting of 250mL water and process using the same procedure as the samples.

5.3 Add 0.25g urease and 10mL phosphate buffer to the Kjeldahl flasks. Wash down the sides of each flask with 250-300mL water (except for blank), stopper tightly, and gently swirl to mix. Let stand ≥ 1 hour at room temperature.

5.4 For each sample, add 100mL 2% boric acid solution with indicator to a labeled 500mL Erlenmeyer flask and place beneath each unit of the Kjeldahl distillation unit with the glass tube immersed into the boric acid solution.

5.5 Turn on the cooling system for the Kjeldahl unit and turn on the burners for the units that will be used.

5.6 Add a few drops of defoaming solution (enough so foaming stops), 5mL $CaCl_2$ solution, and ≥ 2g MgO to the Kjeldahl flask. Rinse the neck with water and immediately connect the flask to the condenser of the Kjeldahl unit. Gently swirl the flask to mix the contents.

5.7 Distill ~100mL (100mL minimum) into the boric acid solution. The total volume will be ~200mL.

5.8 Titrate with standardized sulfuric acid solution to the endpoint (color change to bright red).

5.9 The result includes both ammoniacal and urea nitrogen.

6. Calculations:

$$\%\ \text{ammoniacal \& urea} = \frac{(S - B) * 0.5 * 1.4007}{\text{Sample Weight (g)}}$$

Where:

S = Amount (mL) of sulfuric acid used to titrate sample
B = Amount (mL) of sulfuric acid used to titrate blank
0.5 = Normality of the sulfuric acid solution
1.4007 = Conversion factor

Use the ammoniacal nitrogen method to determine the amount of ammoniacal nitrogen present in the sample.

% urea = % ammoniacal & urea – % ammoniacal

7. References:

7.1 Official Methods of Analysis of AOAC, 17th ed., Method 941.04, Chapter 4.3.01

7.2 Official Methods of Analysis of AOAC, 19th ed., Method 955.04, Chapter 2.4.03

7.3 Standard Methods for the Examination of Water and Wastewater, 20th ed., Method 4500-NH3 C, Chapter 4 – 105

7.4 Methods of Soil Analysis, Part 3 - Chemical Methods, SSSA, Regular Kjeldahl Method, page 1104

Approvals:

Written By:

Stacy Aylesworth — 2/15/21
Stacy Aylesworth — Date
Senior Environmental Scientist (Supervisor)

Approved By:

Maryam Khosravifard — 2/19/21
Maryam Khosravifard — Date
Environmental Program Manager I

Sarva Balachandra — 3/16/21
Sarva Balachandra — Date
Quality Assurance Officer

Revision Log:

Date	What was Revised? Why?