

Urea Nitrogen in Fertilizer Samples

1. Scope:

This procedure is to be used for the analysis of urea nitrogen in fertilizer. This method determines the amount of ammoniacal nitrogen + urea.

2. Principle:

Samples are prepared as described in RA-SP-SMPL-PREP. 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.

3. Safety:

All laboratory safety rules for sample preparation and analysis shall be followed. Read the SDS for all materials before use.

Gloves, eye protection, a face shield, and a lab coat shall be worn when handling hazardous materials and corrosive 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 and Supplies (equivalents are acceptable):

- 4.1 Analytical balance capable of measuring to 0.0001g
- 4.2 Kjeldahl digestion/distillation Unit
- 4.3 Kjeldahl flasks, 800 mL
- 4.4 Rubber stoppers, size 7
- 4.5 Erlenmeyer flasks, 500 mL

5. Reagents (equivalents are acceptable)

- 5.1 Urease powder (Fisher Scientific U21)
- 5.2 Magnesium oxide (Fisher Scientific M68-3)
- 5.3 Calcium chloride
- 5.4 2% Boric acid solution with indicator (Fisher Scientific 1064-1)

- 5.5 Standardized sulfuric acid, 0.5N
- 5.6 Monopotassium phosphite
- 5.7 Dipotassium phosphate

6. Sample Analysis:

- 6.1 Prepare calcium chloride solution by adding 25g CaCl_2 in a 100mL volumetric flask and filling to the line with water.
- 6.2 Prepare phosphate buffer 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.
- 6.3 For samples containing <5% urea, weigh 2g of sample into a Kjeldahl flask (record the weight). For samples containing $\geq 5\%$ urea, weigh 1g of sample.
- 6.4 Prepare a blank consisting of 250mL water and process using the same procedure as the samples.
- 6.5 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 2 hours at room temperature.
- 6.6 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 in the boric acid solution.
- 6.7 Turn on the cooling system for the Kjeldahl unit and turn on the burners for the units that will be used.
- 6.8 Add 5mL CaCl_2 solution (for defoaming) and $\geq 2\text{g}$ 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.
- 6.9 Distill ~100mL (100mL minimum) into the boric acid solution. The total volume will be ~200mL.
- 6.10 Titrate with standardized sulfuric acid to the red endpoint.
- 6.11 The result includes both ammoniacal and urea nitrogen.

NOTE: If boric acid with indicator is not commercially available, it may be prepared by dissolving 200mg methyl red in 100mL isopropyl alcohol, dissolving 100mg

methylene blue in 50mL isopropyl alcohol, combining the 2 solutions then adding 10mL to 1L of boric acid.

7. QA/QC:

- 7.1 A reagent blank shall be run with each set.
- 7.2 A QC sample shall be digested and analyzed with each set and should be a similar matrix to the samples. An acceptable QC sample is a Magruder test sample with a known mean and standard deviation. Results shall be within 2 standard deviations of the assigned value.
- 7.3 The reporting limit is 0.1%.

8. Calculations:

$$8.1 \quad \% \text{ Ammoniacal + urea} = \frac{(A - B) * N * 1.4007}{W}$$

Where:

A = Amount of acid used to titrate the sample (mL)
B = Amount of acid used to titrate the reagent blank (mL)
N = Normality of the standardized sulfuric acid
W = Sample weight (g)
1.4007 = Conversion factor

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

$$\% \text{ urea} = \% \text{ ammoniacal + urea} - \% \text{ ammoniacal}$$

9. References:

- 9.1 Official Methods of Analysis of AOAC, 17th ed., Method 941.04, Chapter 4.3.01
- 9.2 Official Methods of Analysis of AOAC, 19th ed., Method 955.04, Chapter 2.4.03
- 9.3 Standard Methods for the Examination of Water and Wastewater, 20th ed., Method 4500-NH3 C, Chapter 4 – 105
- 9.4 Methods of Soil Analysis, Part 3 - Chemical Methods, SSSA, Regular Kjeldahl Method, page 1104

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