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| | FM-445 | |
| | FERTILIZER METHODS | Chapter |
| | | NITROGEN ANALYSIS |
| | | Subject |
| | Water Insoluble Nitrogen – Kjeldahl | |

SCOPE: This is an analytical procedure for the determination of water insoluble nitrogen in fertilizer samples. This method does not give the degree to which the water insoluble nitrogen is available.

PRINCIPLE: The determination of Kjeldahl water insoluble nitrogen is achieved by the use of sulfuric acid and Kel Pac's to digest the insoluble nitrogen. The product of the Kjeldahl digestion is distilled into a standard acid trap and titrated with standard base.

APPARATUS & EQUIPMENT:

- Beaker, 300 mL
- Kjeldahl digestion and distillation unit
- Kjeldahl flasks, 650 mL or 800 mL
- Auto titration system, or manual titration equipment

REAGENTS & CHEMICALS:

- Antifoam – Dow Corning Antifoam B
- Boiling stones, 8 to 14 mesh
- Deionized water
- Kel Pac #2
- Methyl Red indicator solution – 0.5% in ethanol (5 g in 1000 mL)
- Sodium Hydroxide solution – 1.36 Specific gravity [42° to 44° Baume']. Dissolve 1 kg of sodium hydroxide in deionized water, stir until cool. Add water and stir until hydrometer in cool solution reads between 42° and 44° Baume'.

NOTE: CAN USE COMMERCIALY AVAILABLE 50% NaOH SOLUTION.

- Sulfuric Acid – reagent grade
- Standard acid
- Standard base
- Zinc metal – granular, 20 mesh

STANDARDS:

- 0.5N standard acid– THAM [Tris(Hydroxy Methyl) amminomethane] solution is titrated with .5N H₂SO₄ solution to a pH of 4.70 to determine exact normality.
- 0.2N standard base– PAP [Potassium acid phthalate] solution is titrated with 0.2N NaOH solution to a pH of 8.60 to determine exact normality. Then standard acid is titrated with standard base to the methyl red end point (approx. pH 5.25) to doublecheck results.
- An internal standard is weighed and analyzed with each set to check for completeness of digestion, completeness of distillation, and the precision and accuracy of the titration.
- Class "A" glassware is used throughout method.

**SAMPLE
PREPARATION****PROCEDURE USING RESIDUE FROM METHOD FM-430:**

1. Add the filter paper containing the insoluble residue to the Kjeldahl flask.
2. Add 3 to 5 boiling stones.
3. Add 1 Kel Pac.
4. Add antifoam sparingly if needed.
5. Add 40 mL of concentrated H₂SO₄, place flask on preheated burner and digest on high [rotating flask occasionally to ensure complete digestion] for 75 min.
6. Allow to cool for 8 to 10 minutes, then swirl flask a few times to prevent solidification of digestate. After further cooling, add 300 to 350 mL water, and cool to 25° C or below.
7. Add, to a 300 mL receiving beaker, 1 mL of standard 0.5N H₂SO₄ for each 7 mg of nitrogen in the sample, plus at least 2 mL excess acid. Add 5 drops of methyl red indicator solution and sufficient deionized water to immerse the tip of the distillate delivery tube. [Acid + water = approximately 50 mL – this is your acid trap]. Place the receiving beaker under the delivery tube.
8. Add 2 to 3 g of 20 mesh zinc and sufficient sodium hydroxide solution (at least 60 mL) to make contents of Kjeldahl flask strongly alkaline. Tilt the flask when adding sodium hydroxide solution to layer the sodium hydroxide solution under the acid mixture without agitation. **GO TO NEXT STEP NOW!**
9. Immediately connect flask to distillation bulb, and rotate flask to mix contents.

NOTE: IF SOLUTION COLOR IS GRAY THEN MORE CAUSTIC MAY BE NEEDED TO MAKE SOLUTION STRONGLY ALKALINE! DISTILL UNTIL RECEIVING BEAKER CONTAINS 250 mL.

NOTE: WHEN USING 650 mL KJELDAHL FLASK, WARM FLASK VERY SLOWLY TO KEEP FROM “BLASTING” NH₄ BUBBLE THROUGH ACID TRAP.

SAMPLE ANALYSIS:

Titrate distillate in receiving beaker with 0.2N standard NaOH to the methyl red end point (approximately pH 5.25). Correct for detected nitrogen in reagent blank if applicable.

CALCULATIONS:

1. Acid trap for any weight sample (minimum 4 trap).

$$Trap = \left(\frac{(\% \text{ Ammoniacal } N + \text{ Water Soluble } N \text{ Guarantee})}{1} \times \frac{2.5}{1\%} \times \frac{\text{weight}}{1} \times \frac{1}{1.75g} \right)$$

2. Water insoluble nitrogen as percent nitrogen. Where:
 C = Conversion factor for nitrogen = 1.40068 g/mL
 N_a = Normality of standard acid in mL
 N_b = Normality of standard base in mL
 V_a = Volume of standard acid in mL
 V_b = Volume of standard base in mL
 W = Weight of sample taken in g

$$N\% = \frac{[(N_a \times V_a) - (N_b \times V_b)] \times C}{W}$$

APPROVAL:

Approved by: Leigh Humphreys Date: 11/1/01
 Signature

Bureau Chief
 Title

METHOD REVISION HISTORY:

| Version | Date | Description | Author |
|----------------|-------------|--------------------|---------------|
| Original | 11/01/01 | Replaces N-400.3 | W.M. Bell |
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REFERENCE:

- AOAC 15th Edition -*Method 930.01 "Robertson Method"*
- AOAC 15th Edition -*Method 930.02 "Jones Modification of Robertson Method"*