

	FM-420	
	FERTILIZER METHODS	Chapter
		NITROGEN ANALYSIS
		Subject
	Ammoniacal Nitrogen (Urea Not Present) – Direct	

SCOPE: This is an analytical procedure for the determination of ammoniacal nitrogen in **fertilizer samples that do not contain urea, water soluble organic nitrogen, or water insoluble nitrogen.**

PRINCIPLE: The determination of ammoniacal nitrogen is achieved by making a fertilizer solution basic, distilling ammoniacal nitrogen into a standard acid trap and titrating the excess acid with standard base.

SAFETY: Each laboratory is responsible for maintaining a current file of the Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

APPARATUS & EQUIPMENT:

- Kjeldahl distillation unit
- Kjeldahl flasks, 650 mL or 800 mL
- Beaker, 300 mL
- Auto Titration System, or manual titration equipment (Class “A” glassware)

REAGENTS & CHEMICALS:

- Antifoam - Dow Corning Antifoam B
- Boiling stones, 8 to 14 mesh
- Deionized water q.s.
- Methyl Red indicator
- Methyl Red indicator solution – 0.5% in ethanol (5g in 1000 mL)
- Potassium acid phthalate (PAP) Certified A.C.S. grade or equivalent
- Sodium Hydroxide 50% Commercially available solution - 1.36 Specific gravity, or make 50% NaOH solution. **Caution: Strong Base, wear**

protective equipment.

- Standard acid
- Standard base
- THAM [Tris(Hydroxy Methyl) amminomethane] Certified A.C.S. Alkalimetric Standard

STANDARDS:

- 0.5N standard acid – THAM [Tris (Hydroxy Methyl) aminomethane] solution is titrated with 0.5N 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 (approximately pH 5.25) to double check 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.

**SAMPLE
PREPARATION****PROCEDURE:**

1. Weigh approximately 0.75 g of sample into 800 mL Kjeldahl flask.
2. Add 3 to 5 boiling stones.
3. Add antifoam sparingly.
4. Add 300 to 350 mL water.
5. 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.
6. Preheated distillation burner (low).
7. Add 5 mL sodium hydroxide solution to make contents of Kjeldahl flask 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!

8. Immediately connect flask to distillation bulb and rotate flask to mix

contents. Adjust heating element as necessary. Distill until receiving beaker contains 250 mL.

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

SAMPLE

ANALYSIS:

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

CALCULATIONS:

- Quick acid trap for a 0.70 to 0.75 g sample. Trap = Guarantee + 2 and round-up to next even number (minimum 4 trap)
- Acid trap for any weight sample

$$Trap = \left(\frac{(\% Guarantee)}{1} \times \frac{2.5}{1\%} \times \frac{weight}{1} \times \frac{1}{1.75g} \right) + 2(\text{round up to even number})$$

- Ammoniacal 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:


Signature

Date: 2/5/03

Bureau Chief

Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	06/30/98	Replaces N-200	W.M. Bell
Revision	02/05/03		W.M. Bell

REFERENCE:

- AOAC 15th Edition - *Method 892.01 "Nitrogen (Ammoniacal and Nitrate) in Fertilizer"*.
- National Plant Food Institute 2nd Edition - *Method 2.220 "Total Ammoniacal Nitrogen"*.