

	<b>FM-440</b>	
	<b>FERTILIZER METHODS</b>	Chapter
		NITROGEN ANALYSIS
		Subject
	Other Water Soluble Nitrogen B Kjeldahl	

**SCOPE:** This is an analytical procedure for the determination of water soluble nitrogen in fertilizer samples.

**PRINCIPLE:** The determination of water soluble organic nitrogen is achieved by using ferrous sulfate to convert nitrate to nitrite and then liberating the nitrite gas while using sulfuric acid to digest fertilizer samples. The ammoniacal product of the Kjeldahl digestion is distilled into a standard acid trap and the excess acid is titrated 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:**

- Beaker, 300 mL
- Kjeldahl digestion and distillation unit
- Kjeldahl flasks, 650 mL or 800 mL
- Auto titration system, or manual titration equipment
- Pipet, 100 mL

**REAGENTS & CHEMICALS:**

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

**acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment.**

- Standard acid
- Standard base
- THAM [Tris(Hydroxy Methyl) amminomethane] Certified A.C.S. Alkalimetric Standard
- Zinc metal - 20 mesh

#### STANDARDS:

- 0.5N standard acid B THAM [Tris(Hydroxy Methyl) amminomethane] solution is titrated with .5N H<sub>2</sub>SO<sub>4</sub> solution to a pH of 4.70 to determine exact normality.
- 0.2N standard base B 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 USING FILTRATE FROM METHOD FM-430:

1. Pipet 100 mL of filtrate into a Kjeldahl flask.
2. Add 3 to 5 boiling stones.
3. Add 2 g of FeSO<sub>4</sub>·7H<sub>2</sub>O.
4. Add antifoam sparingly if needed.
5. Preheated digestion burner.
6. Add 20 mL sulfuric acid. Place flask on preheated burner and digest on high (rotating flask occasionally to ensure complete digestion) for 60 minutes or until dense white fumes of sulfuric acid clear the bulk of the flask.
7. Remove flask from burner. Allow to cool for 8-10 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<sup>N</sup> C or below.
8. Add, to a 300 mL receiving beaker, 1 mL of standard 0.5N H<sub>2</sub>SO<sub>4</sub> 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 distilled water to immerse the tip of the distillate delivery tube. [Acid + water = approximately 50 mL B this is your acid trap.] Place the receiving beaker under the delivery tube.

9. Preheated distillation burner.
10. 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.

**NOTE: GO TO NEXT STEP NOW!**

11. 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 ABLASTING@ NH<sub>4</sub> 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:**

- Acid trap for 1 g weight sample.  
Trap = 2/3 (NH<sub>4</sub><sup>+</sup> WSN) and round up to next even number (minimum 4 trap)
- Acid trap for any weight sample (minimum 4 trap).

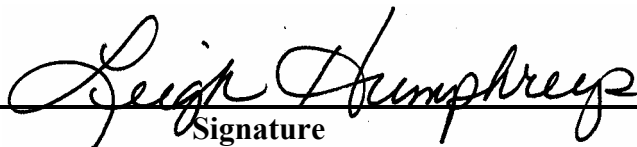
$$Trap = \left( \frac{(\% AM + WS Guarantee)}{1} \times \frac{2.5}{1\%} \times \frac{weight}{1} \times \frac{1}{1.75g} \right)$$

- Water insoluble nitrogen as percent nitrogen. Where:
  - C = Conversion factor for nitrogen = 1.40068 g/mL
  - D = Dilution factor = 250 mL/100 mL = 2.5
  - N<sub>a</sub> = Normality of standard acid in mL
  - N<sub>b</sub> = Normality of standard base in mL
  - V<sub>a</sub> = Volume of standard acid in mL
  - V<sub>b</sub> = Volume of standard base in mL
  - W = Weight of sample taken in g

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

**APPROVAL:**

Approved by: \_\_\_\_\_

  
Signature

Date: 2/4/03

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**Bureau Chief**

Title

**METHOD REVISION HISTORY:**

Version	Date	Description	Author
Original	06/11/98	Replaces N-400.2	W.M. Bell
Revision	02/04/03		W.M. Bell

**REFERENCE:**

- AOAC 15<sup>th</sup> Edition - *Method 930.01 "Robertson Method"*.
- AOAC 15<sup>th</sup> Edition - *Method 930.02 "Jones Modification of Robertson Method"*.