

	FM-405	
	FERTILIZER METHODS	Chapter
		NITROGEN ANALYSIS
		Subject
	Total Nitrogen – Kjeldahl	

SCOPE: This is an analytical procedure for the determination of total nitrogen in all fertilizer samples.

PRINCIPLE: The determination of total nitrogen is achieved by converting all nitrogen into the ammoniacal form using chromium metal, hydrochloric acid, Popes mixture #2 or Kel Pac #2, and sulfuric acid to digest fertilizer samples. The 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 Health and Safety Act (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 digestion and distillation unit
- Kjeldahl flasks, 650 mL or 800 mL
- Beaker, 300 mL
- Auto Titration System, or manual titration equipment

REAGENTS & CHEMICALS:

- Boiling stones, 8 to 14 mesh
- Chromium metal, 100 mesh, low nitrogen
- Deionized water
- Hydrochloric acid (Certified A.C.S. grade)
- Methyl Red indicator solution - 0.5% in ethanol (5 g in 1000 ml)
- PAP (Potassium Acid Phthalate) (Certified A.C.S. grade)
- Popes mixture #2 or Kel Pac #2
- Sodium hydroxide (Certified A.C.S. grade)
- Sulfuric acid (H₂SO₄)(Certified A.C.S. grade) **Caution – strong acid. Avoid breathing vapors and skin contact. Use in a fume hood and wear protective equipment (H₂SO₄)**

- THAM [Tris(Hydroxy Methyl) amminomethane] (Certified A.C.S. grade Alkalimetric Standard)
- Sodium hydroxide solution - 1.36 Specific gravity (42^N to 44^N Baume'). Dissolve 1 kg of sodium hydroxide in water, stir until cool. Add water and stir until hydrometer in cool solution reads between 42^N and 44^N Baume'. Option: Use commercially available 50% NaOH solution.
- Dilute sulfuric acid (1:1); slowly add 625 mL H₂SO₄ to 300 mL D.I. water, cool and dilute to 1000 mL, mix and store in stoppered container.
- Standard acid
- Standard base
- Zinc metal - 20 mesh

STANDARDS:

1. .5N standard acid – A THAM [Tris(Hydroxy Methyl) amminomethane] solution is titrated with .5N H₂SO₄ solution to a pH of 4.70 to determine exact normality.
2. .2N standard base – A PAP (Potassium Acid Phthalate) solution is titrated with .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.
3. 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.
4. Class "A" glassware is used throughout method.

SAMPLE PREP PROCEDURE:

- Weigh sample, 0.3 - 2.2 g into Kjeldahl flask.
NOTE: Adjust sample weight so that sample does not contain more than 7 mg of nitrate.
- Add 3-5 boiling stones, 1.2 g chromium metal, and 35 mL water. Let stand for 10 minutes, with occasional gentle swirling to dissolve all nitrate salts.
- Add 10 mL hydrochloric acid and let stand for at least 30 seconds, but not more than 10 minutes. (5-10 minutes best).
- Place flask on preheated burner. After heating 3-5 minutes, remove from heat and let cool.
- Add 22 g of Popes mixture #2 or one Kel Pac #2.
- Add 40 mL dilute sulfuric acid. Place flask on preheated burner and heat on med-high until dense white fumes of sulfuric acid clear the bulk of the flask. Heat another 5-10 minutes (**about 15 minutes total**).
- Digestion is now complete for samples containing ammoniacal, nitrate, and urea nitrogen. Swirl flask gently and continue digestion (**on high**) for remainder of hour or until digestion is complete (1 hour 15 minutes maximum time).

- 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 300-350 mL water and cool to 25°C or below.
- 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 distilled water to immerse the tip of the distillate delivery tube. *Acid + H₂O = approximately 50 mL - this is your acid trap. Place the receiving beaker under the delivery tube.*
- Add 2-3 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 in order to layer the sodium hydroxide solution under the acid mixture without agitation.

GO TO NEXT STEP NOW!

- Immediately connect flask to distillation bulb and rotate flask to mix contents. 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 .2N 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 .7 to .75 g sample.

Trap = (Total Nitrogen Guarantee) + 2 and round-up to next even number.

Acid trap for any weight sample

$$Trap = \left(\frac{(\%Total\ Nitrogen\ Guarantee)}{1} \times \frac{2.5}{1\%} \times \frac{weight}{1} \times \frac{1}{1.75g} \right)$$

Total percent nitrogen:

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

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

In many cases it may be more convenient to calculate the total nitrogen content of the sample. If all nitrogen compounds present in the sample have been determined by analysis using Methods FM-415, FM-420, FM-425, FM-435, FM-440, FM-445, FM-451, FM-462 and/or FM-471 then total nitrogen may be calculated as the sum of the nitrogen percents contributed by each component.

APPROVAL:

Approved by: Leigh Humphreys Date: 8/16/02
Signature

Bureau Chief
Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	06/05/98	Replaces N-100	W.M. Bell
Revised	08/16/02		W.M. Bell

REFERENCE:

AOAC 15th Edition – Method 978.02 “Nitrogen (Total) in Fertilizers”