

	FM-430	
	FERTILIZER METHODS	Chapter
		NITROGEN ANALYSIS
		Subject
	Breakdown Nitrogen Preparation	

SCOPE:

This is a standard analytical preparation for the determination of the individual nitrogen components of fertilizer samples using methods

- FM-435 Ammoniacal Nitrogen (Urea Present) – Kjeldahl
- FM-440 Other Water Soluble Nitrogen – Kjeldahl
- FM-445 Water Insoluble Nitrogen – Kjeldahl
- FM-451 Nitrate Nitrogen – Flow Injection
- FM-461 Urea Nitrogen – Flow Injection by Dimethylaminobenzaldehyde
- FM-462 Urea Nitrogen – Flow Injection by Urease
- FM-470 Ammoniacal Nitrogen – Flow Injection
- FM-471 Improved Ammoniacal Nitrogen – Flow Injection

PRINCIPLE:

The preparation for individual break-down nitrogen determinations is achieved by dissolving and/or filtering a fertilizer sample with 250 mL of water.

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.

**APPARATUS &
EQUIPMENT:**

- Balance, minimum accuracy to 0.001 g
- Dilution container, 1 or 2 L as needed
- Filter paper, 11 cm Whatman No. 2
- Flask, 250 mL volumetric
- Funnels short-stemmed, 60°C, 60 mm diameter
- Transfer pipet, disposable

**REAGENTS &
CHEMICALS:**

- Deionized water - 20 - 25°C
- Methanol - 95%

STANDARDS:

1. An internal standard is weighed and analyzed with each set to check precision and accuracy.
2. Class "A" glassware is used throughout method.

**SAMPLE
PREPARATION:****FILTERED OR LONG SAMPLE** Preparation Procedure:

NOTE: THIS PROCEDURE IS APPLICABLE TO LIQUID SAMPLES WITH WATER INSOLUBLE NITROGEN GUARANTEES.

1. Weigh approximately 1 g of sample into Whatman # 2 filter paper in a 60 mm, 60°C, short-stemmed funnel over a 250 mL volumetric flask.
2. Wet sample with 2 to 3 mL of methanol, to prevent clumping.
3. Wet the sample residue with 200 mL of deionized water in 20 to 30 mL portions, allowing sample to drain completely between washings.
4. Bring volume of filtrate to 250 mL, stopper and shake. Save for methods:
 - FM-435 Ammoniacal Nitrogen (Urea Present) – Kjeldahl
 - FM-440 Other Water Soluble Nitrogen – Kjeldahl
 - FM-451 Nitrate Nitrogen – Flow Injection
 - FM-461 Urea Nitrogen – Flow Injection by Dimethylaminobenzaldehyde
 - FM-462 Urea Nitrogen – Flow Injection by Urease
 - FM-470 Ammoniacal Nitrogen – Flow Injection
 - FM-471 Improved Ammoniacal Nitrogen – Flow Injection
5. Save filter and residue for method FM-445 Water Insoluble Nitrogen – Kjeldahl.

UNFILTERED OR SHORT SAMPLE Preparation Procedure **for samples that do not contain water insoluble nitrogen:**

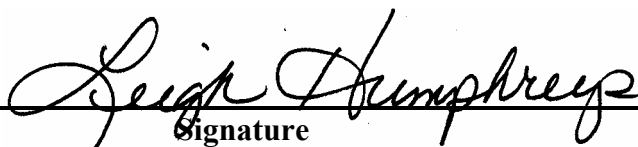
1. Weigh approximately 1 g of ground sample into a 250 mL volumetric flask.
2. Wet sample with 2 to 3 mL of methanol, to prevent clumping.
3. Add approximately 200 mL of water, stopper and shake.
4. Bring volume to 250 mL with deionized water, stopper and shake. Save for methods:
 - FM-460 Urea Nitrogen – Continuous Segmented Flow
 - FM-475 Urea Nitrogen – Flow Injection by Dimethylaminobenzaldehyde

WEIGHING LIQUID SAMPLES Preparation Procedure:

1. Check liquid samples for crystals using a glass rod. Crystals are easily detected by rubbing the glass rod on the bottom of the container.
2. If crystals are present dilute with enough hot deionized water to completely dissolve all crystals.
 - a. Weigh the sample and its container.
 - b. Weigh the dilution container.
 - c. Pour the sample into the dilution container.
 - d. Rinse sample container by filling half full with hot deionized water, cap and shake. Pour into dilution container and repeat.
 - e. Make sure all particles are transferred from sample bottle and dissolved.
 - f. Cap dilution container and shake.
 - g. Weigh dilution container again, and record this total diluted weight.
 - h. Subtract the original dilution container weight from the total diluted weight. This is the diluted weight of sample; record this number.
 - i. Clean and dry the original sample container.
 - j. Weigh the clean and dry original sample container.
 - k. Subtract the weight of the clean sample container from the weight of the sample and the container. This is the weight of liquid sample.
 - l. Divide the diluted weight of sample by the weight of the liquid sample, and record this number as your dilution multiplier.
 - m. Allow sample to cool to room temperature before weighing.
3. Shake liquid sample until well mixed. **GO IMMEDIATELY TO THE NEXT STEP!**
4. Using a disposable transfer pipet, remove approximately 1 g of sample from the middle of the container.
5. **Transfer entire contents** of disposable transfer pipet to weigh dish or 250 mL volumetric flask. Weigh sample and proceed with either filtered or unfiltered sample procedure.

APPROVAL:

Approved by: _____


Signature

Date: 11/12/01

Bureau Chief

Title

METHOD REVISION HISTORY:

Version	Date	Description	Author
Original	11/12/01	Replaces N-400	G. H. Huang

REFERENCE:

AOAC 15th Edition - *Method 945.01 "Nitrogen [Water Insoluble] in Fertilizers - Method I"*.