Determination of Nitrogen in Fertilizer

LECO Corporation; Saint Joseph, Michigan USA

Instrument: FP628

Introduction

Nitrogen is one of the most important elements for plant development and is the macronutrient that is most often found to be deficient in arable soils used for crop production. Nitrogen plays a key role in promoting lush, vigorous growth and development of plants that often leads to an increase in the yield from the plant. Fertilizers are utilized to re-introduce nitrogen back into arable soils. Fertilizers can be grouped by their makeup and/or origins into categories including inorganic and/or synthetic (including nitrates, ammonium, and urea) and organic (including compost materials and manures). The accurate and precise determination of nitrogen in all fertilizer types is not only important in the process of blending and preparing the fertilizer material for use, but also will have significant impact on the commercial value and guarantee of the fertilizer.

The LECO FP628 combustion nitrogen analyzer has been designed to easily handle the fertilizer sample and required sucrose addition (per AOAC 993.13), while maintaining a rapid analysis time.

The LECO FP628 is a combustion nitrogen analyzer that utilizes a pure oxygen environment in a vertical quartz furnace for the sample combustion process, resulting in an analysis time of 3.5 minutes without the use of metal oxidizer reagents in the primary or secondary furnace. A thermoelectric cooler removes the moisture from the combustion gas without the use of chemical reagents. The combustion gases are homogenized in a ballast and a 10 cc aliquot is taken using a combustion gas collection and handling system. The aliquot is swept through reagents using an inert carrier gas, such as helium or argon. This system achieves a low cost-per-analysis by reducing the amount of chemical reagents used for scrubbing and converting the nitrogen oxide compounds to nitrogen gas. A thermal conductivity (TC) cell is used for the detection of nitrogen, as N_2.

Accessories

502-186 Tin Foil Cup, 501-441 Sucrose (ground), 502-040 Tin Capsules

Reference Materials

LCRM®, LRM®, NIST, or other suitable reference materials.

Reference Method

AOAC 993.13

Analysis Parameters

Analysis Parameters	
Combustion Temperature:	950 °C
Afterburner Temperature:	850 °C

*Element Parameters**

	Nitrogen
Analyze	Yes
Baseline Delay Time	5 seconds
Minimum Analysis Time	40 seconds
Comparator Level	100.00
Endline Time	2 second
Conversion Factor	1.00
Significant Digits	5
TC Baseline Time:	10 seconds

Burn Profile

Burn Steps	*Time*	*Furnace* Flow
1	20 seconds	High
2	150 seconds	Medium
3	30 seconds	High

Macro Ballast Parameters

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Fill Pressure Drop	200 mm Hg
Equilibrate Pressure Time	8 seconds

**Refer to FP628 Operator's Instruction Manual for Parameter definitions.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Select 10 replicates.
   c. Initiate the analysis sequence (F5).
   d. Set the blank using the last 5 results following the procedure outlined in the operator's instruction manual.

   Note: blank precision should be <0.001%.
3. Calibrate/Drift Correct.
   a. Weigh ~0.05 gram of the selected reference material into a 502-186 Tin Foil Cup.
   b. Enter mass and sample identification into Sample Login (F3).

Organic Application Note

c. Add ~0.20 grams of ground LECO 501-441 Sucrose to the reference material and seal the 502-186 Tin Foil Cup.
d. Transfer sample to the appropriate position of the sample carousel.
e. Repeat steps 3a through 3d a minimum of five times for each calibration/drift sample used.
f. Initiate the analysis sequence (F5).
g. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
h. Verify the calibration by weighing ~0.05 grams of a different reference material into a 502-186 Tin Foil Cup.
i. Add ~0.20 grams of ground LECO 501-441 Sucrose to the reference material and seal the 502-186 Tin Foil Cup.
j. Transfer sample to the appropriate position of the sample carousel.
k. Initiate the analysis sequence (F5).

4. Analyze Samples (Solid Fertilizer).
   a. Weigh ~0.05 gram of the unknown sample into a 502-186 Tin Foil Cup.
   b. Enter mass and identification information into Sample Login (F3).
   c. Add ~0.20 grams of ground LECO 501-441 Sucrose to the unknown sample and seal the 502-186 Tin Foil Cup.
   d. Transfer sample to the appropriate position of the sample carousel.
   e. Initiate the analysis sequence (F5).
5. Analyze Samples (Liquid Fertilizer).
   a. Weigh ~0.10 gram of the unknown sample into a 502-040 Tin Capsule.
   b. Enter mass and identification information into the Sample Login (F3).
   c. Add ~0.20 grams of ground LECO 501-441 Sucrose to the unknown sample.
   d. Transfer sample to the appropriate position of the sample carousel.
   e. Initiate the analysis sequence (F5).

*Typical Results**

Name	*Mass (g)*	*% Nitrogen*
Ammonium Nitrate	0.0509	34.99
Aldrich Lot: 09016AR	0.0512	34.89
Theoretical: 35.0% N	0.0519	35.04
	0.0522	35.08
	0.0543	35.11
	Avg =	***35.02***
	s =	***0.09***

Name	*Mass (g)*	*% Nitrogen*
Urea	0.0531	46.75
Sigma-Aldrich Lot:	0.0548	46.63
SLBH9709V	0.0513	46.57
Theoretical: 46.65% N	0.0530	46.67
	0.0511	46.69
	Avg =	***46.66***
	s =	***0.06***

Potassium Nitrate	0.0558	13.86
Sigma-Aldrich Lot:	0.0512	13.80
MKBT3182V	0.0515	13.69
Theoretical: 13.85% N	0.0601	13.90
	0.0562	13.83
	Avg =	***13.81***
	s =	***0.08***

Liquid Fertilizer	0.1108	8.65
	0.1110	8.53
	0.1087	8.52
	0.1082	8.46
	0.1071	8.48
	Avg =	***8.53***
	s =	***0.07***

**Based on a single standard, force through origin calibration utilizing Sigma-Aldrich Lot: SLBH9709V Urea @ 46.65% N.*

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