Application Note

Instrument: FP828

Determination of Nitrogen in Fertilizer

LECO Corporation; Saint Joseph, Michigan USA

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 the formation of enzymes and proteins, which promotes lush vigorous growth and development in 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 FP828 is a combustion nitrogen/protein determinator that utilizes a pure oxygen environment in a vertical quartz furnace. A thermoelectric cooler removes moisture from the combustion gases before they are collected in a ballast. The gases equilibrate and mix in the ballast before a representative aliquot (3 cm^3 or 10 cm^3 volume) of the combustion gas is extracted and introduced into a flowing stream of inert gas (helium or argon) for analysis. The aliquot gas is carried to a thermal conductivity (TC) cell for the detection of nitrogen (N_2).

Instrument Model and Configuration

Thermal conductivity detectors work by detecting changes in the thermal conductivity of the analyte gas compared to a reference/carrier gas. The greater the difference between the thermal conductivity of the carrier gas and the analyte gas, the greater sensitivity of the detector. The FP828 supports either the use of helium or argon as the instrument's carrier gas. When used as a carrier gas, helium provides the highest sensitivity, and the best performance at the lower limit of the nitrogen range. Argon can also be used as a carrier gas. The thermal conductivity difference between argon and nitrogen is not as great as the thermal conductivity difference between helium and nitrogen, therefore the detector is inherently less sensitive when using argon as a carrier gas.

The FP828 offers the additional advantage of utilizing either a 10 cm^3 aliquot loop or a 3 cm^3 aliquot loop within the instrument's gas collection and handling system. The 10 cm^3 aliquot loop optimizes the system for the lowest nitrogen range and provides the best precision. The 3 cm^3 aliquot loop extends reagent life expectancy by approximately three-fold when compared to the 10 cm^3 aliquot loop, while providing the lowest cost-per-analysis with minimal impact on practical application performance (see Typical Results section).

Note: When changing carrier gas type, the flow needs to be adjusted following instructions provided in the 828 Series Operator's Instruction Manual. Base model instruments require manual changing of the dose loop.

Method Reference

AOAC 993.13 - Nitrogen (Total) in Fertilizers, Combustion Method.

Sample Preparation

Samples must be of a uniform consistency to produce suitable results. Reference materials should be prepared as directed by the certificate prior to analysis.

Accessories

502-186 Small Tin Foil Cups, 502-040 Tin Capsules or 502-825 Large Tin Capsules, commercially available ground sucrose, and 501-614 Spatula

Reference Materials

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

*Method Parameters**

Gas Type	Helium or Argon
Furnace Temperature	950 °C
Afterburner Temperature	850 °C
Nominal Mass	1.0000 g
Purge Cycles	2

*Ballast Parameters**

	Helium	*Argon*
Ballast Equilibrate Time	8 s	8 s
Ballast Not Filled Timeout	300 s	300 s
Aliquot Loop Fill Pressure Drop	200 mm Hg	175 mm Hg
Aliquot Loop Equilibrate Time	4 s	4 s
Dose Loop Size	10 cm^3 or 3 cm^3	10 cm^3 or 3 cm^3
Interleave Analysis	Yes	Yes
Sample Drop Detection	Disabled	Disabled

*Element Parameters**

	Helium	*Argon*
Integration Delay	4 s	5 s
Starting Baseline	15 s	15 s
Post Baseline Delay	14 s	15 s
Use Comparator	No	No
Integration Time	40 s	55 s
Use Endline	Yes	Yes
Endline Delay	25 s	30 s
Ending Baseline	15 s	15 s
Use Profile Blank	---	Yes

**Refer to 828 Series Operator's Instruction Manual for Parameter definitions.*

Burn Profile

Burn Step	*Furnace Flow*		*Time*
	Base Model	*Performance Model*	
1	High	5.0 L/min	20 s
2	Low	0.2 L/min	150 s
3	High	5.0 L/min	End

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the System.
   a. Select three to five replicates in the Login screen.
   b. Weigh ~0.05 g of a fertilizer reference or sample material into a 502-186 Tin Foil Cup.
   c. Enter conditioning sample mass and identification into the Login screen.
   d. Add ~0.2 g of ground sucrose to the conditioning sample in the tin foil cup and seal the cup by twisting the top edges of the foil together.
   e. Transfer the tin foil cup containing the conditioning sample and sucrose to the appropriate position in the sample carousel.
   f. Perform steps 2b through 2e for each conditioning sample to be analyzed.
   g. Initiate the analysis sequence.
3. Determine Blank.
   a. Select five or more Blank replicates in the Login screen.
   b. Initiate the analysis sequence.
   c. Set the blank using at least five blank results following the procedure outlined in the operator's instruction manual.

   Note: The standard deviation of the last five blanks should be less than or equal to 0.001% (10 ppm) when utilizing Helium as a carrier gas, and less than or equal to 0.005% (50 ppm) when utilizing Argon as a carrier gas. Additional blanks beyond the recommended five may be required in order to achieve the recommended precision.
4. Calibrate/Drift Correct.
   a. Select the desired number of calibration/drift replicates in the Login screen (minimum of five).
   b. Weigh ~0.05 g of NIST 913b Uric Acid or another suitable reference material into a 502-186 Tin Foil Cup.
   c. Enter sample mass and identification into the Login screen.
   d. Add ~0.2 g of ground sucrose to the reference material in the tin foil cup and twist the top edges of the foil cup together to seal.
   e. Transfer the tin foil cup containing the reference material and sucrose to the appropriate position in the sample carousel.
   f. Perform steps 4b through 4e a minimum of five times.
   g. Initiate the analysis sequence.
   h. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
   i. Verify the calibration or drift correction by analyzing an appropriate mass of another suitable reference material and confirm that the results are within the acceptable tolerance range.
5. Analyze Samples (Powder/Granular Samples).
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh ~0.05 g of the sample into a 502-186 Tin Foil Cup.
   c. Enter sample mass and identification information into the Login screen.
   d. Add ~0.2 g of ground sucrose to the reference material in the tin foil cup and twist the top edges of the foil cup together to seal.
   e. Transfer the tin foil cup containing the sample and sucrose to the appropriate position in the sample carousel.
   f. Perform steps 5b through 5e for each sample to be analyzed.
   g. Initiate the analysis sequence.
6. Analyze Samples (Liquid Samples)**
   a. Select the desired number of sample replicates in the Login screen.
   b. Weigh an appropriate mass (typically between 0.10 g and 1.0 g) of the sample into a 502-040 Tin Capsule or a 502-825 Large Tin Capsule.
   c. Enter sample mass and identification information into the Login Screen.
   d. Transfer the capsule containing the sample to the appropriate position in the sample carousel.
   e. Perform steps 6b through 6d for each sample to be analyzed.
   f. Initiate the analysis sequence.

   ***Most liquid fertilizers do not require the addition of sucrose. However, liquid fertilizers containing high levels of nitrates do require the addition of sucrose. Refer to AOAC Method 993.13 Nitrogen (Total) in Fertilizers, Combustion Method for further details.*

LECO Corporation | 3000 Lakeview Avenue | St. Joseph, MI 49085 | Phone: 800-292-6141 | 269-985-5496
info@leco.com • www.leco.com | ISO-9001:2015 Certified | *LECO is a registered trademark of LECO Corporation.*

TYPICAL RESULTS

Data was generated utilizing a linear, forced through origin calibration using ~0.05 g of NIST 913b Uric Acid (33% N). Alternatively, LECO 503-530 LCRM Urea may be used for calibration. The calibration was verified using LECO 502-896 (Lot 1002) EDTA (9.57% N).

	10 cm^3 Helium		**3 cm^3 Helium†**		**10 cm^3 Argon**		**3 cm^3 Argon†**	
	Mass (g)	*% N*	*Mass (g)*	*% N*	*Mass (g)*	*% N*	*Mass (g)*	*% N*
Urea	0.0519	46.6	0.0520	46.7	0.0496	46.7	0.0493	46.5
Reagent Grade	0.0490	46.7	0.0496	46.6	0.0544	46.6	0.0546	46.6
Theoretical Value: 46.6% N	0.0497	46.5	0.0503	46.6	0.0500	46.7	0.0468	46.5
	0.0510	46.6	0.0510	46.5	0.0519	46.5	0.0503	46.7
	0.0529	46.6	0.0522	46.6	0.0497	46.6	0.0505	46.8
	0.0493	46.6	0.0531	46.6	0.0543	46.6	0.0491	46.5
	0.0514	46.7	0.0517	46.7	0.0505	46.6	0.0522	46.6
	0.0490	46.6	0.0507	46.7	0.0503	46.7	0.0511	46.7
	0.0506	46.7	0.0493	46.5	0.0516	46.4	0.0516	46.5
	0.0511	46.8	0.0502	46.6	0.0513	46.7	0.0505	46.6
	Avg =	***46.6***	***Avg =***	***46.6***	***Avg =***	***46.6***	***Avg =***	***46.6***
	s =	***0.1***	***s =***	***0.1***	***s =***	***0.1***	***s =***	***0.1***
Ammonium Sulfate	0.0500	21.3	0.0574	21.3	0.0557	21.3	0.0545	21.3
Reagent Grade	0.0530	21.3	0.0541	21.2	0.0538	21.3	0.0484	21.2
Theoretical Value: 21.2% N	0.0518	21.3	0.0524	21.2	0.0532	21.1	0.0475	21.1
	0.0493	21.2	0.0535	21.3	0.0530	21.2	0.0508	21.1
	0.0493	21.2	0.0542	21.2	0.0549	21.2	0.0540	21.1
	0.0518	21.2	0.0541	21.2	0.0524	21.2	0.0559	21.2
	0.0507	21.2	0.0537	21.2	0.0489	21.3	0.0500	21.1
	0.0503	21.2	0.0548	21.2	0.0545	21.2	0.0481	21.0
	0.0531	21.2	0.0521	21.2	0.0573	21.2	0.0514	21.1
	0.0516	21.3	0.0512	21.3	0.0514	21.2	0.0493	21.1
	Avg =	***21.2***	***Avg =***	***21.2***	***Avg =***	***21.2***	***Avg =***	***21.1***
	s =	***<0.1***	***s =***	***0.1***	***s =***	***0.1***	***s =***	***0.1***
Potassium Nitrate	0.0502	13.9	0.0522	13.9	0.0510	13.8	0.0552	13.7
Reagent Grade	0.0515	13.8	0.0508	13.8	0.0510	13.9	0.0535	13.9
Theoretical Value: 13.85% N	0.0536	13.8	0.0486	13.9	0.0481	13.9	0.0538	13.8
	0.0535	13.9	0.0490	13.9	0.0538	13.8	0.0543	13.8
	0.0479	13.8	0.0504	13.8	0.0550	13.8	0.0531	13.8
	0.0520	13.8	0.0533	13.9	0.0549	13.8	0.0552	13.8
	0.0499	13.8	0.0526	13.8	0.0500	13.8	0.0564	13.8
	0.0503	13.8	0.0532	13.8	0.0478	13.9	0.0533	13.9
	0.0502	13.9	0.0497	13.9	0.0518	13.9	0.0486	13.8
	0.0519	13.8	0.0491	13.9	0.0482	13.9	0.0521	13.7
	Avg =	***13.8***	***Avg =***	***13.9***	***Avg =***	***13.8***	***Avg =***	***13.8***
	s =	***<0.1***	***s =***	***<0.1***	***s =***	***0.1***	***s =***	***0.1***
LECO 502-602	1.0003	0.097	1.0057	0.102	0.5245	0.010	0.4984	0.103
Lot# CT-0859	1.0007	0.100	1.0052	0.096	0.5318	0.096	0.4994	0.090
Ammonium Solution	1.0005	0.098	1.0032	0.097	0.5422	0.096	0.4889	0.079
0.10% N	0.9901	0.095	1.0046	0.097	0.5224	0.101	0.4930	0.099
	1.0095	0.100	1.0030	0.095	0.5273	0.096	0.4925	0.104
	1.0150	0.101	1.0663	0.096	0.5320	0.099	0.5006	0.095
	1.0011	0.095	1.0283	0.099	0.5055	0.096	0.4975	0.085
	1.0017	0.100	1.0220	0.092	0.5793	0.102	0.4950	0.102
	1.0016	0.097	1.1001	0.097	0.5355	0.095	0.4995	0.099
	1.0559	0.101	1.0334	0.097	0.5360	0.094	0.4991	0.106
	Avg =	***0.098***	***Avg =***	***0.097***	***Avg =***	***0.097***	***Avg =***	***0.096***
	s =	***0.002***	***s =***	***0.003***	***s =***	***0.003***	***s =***	***0.009***
LECO 502-601	1.0632	0.011	1.0847	0.012	---	††	---	††
Lot# 0006538211	1.0749	0.010	1.0612	0.011	---	††	---	††
Ammonium Solution	1.0641	0.010	1.0541	0.009	---	††	---	††
0.010% N	1.0599	0.011	1.0563	0.010	---	††	---	††
	1.0744	0.010	1.1004	0.012	---	††	---	††
	1.0680	0.009	1.0806	0.010	---	††	---	††
	1.0665	0.010	1.0936	0.011	---	††	---	††
	1.0892	0.010	1.0485	0.011	---	††	---	††
	1.0654	0.010	1.0677	0.011	---	††	---	††
	1.1086	0.011	1.0019	0.009	---	††	---	††
	Avg =	***0.010***	***Avg =***	***0.011***	***Avg =***	***---***	***Avg =***	***---***
	s =	***0.001***	***s =***	***0.001***	***s =***	***---***	***s =***	***---***

†Utilizing the 3 cm^3 dose loop, when analyzing materials with low nitrogen concentrations, may result in poor precision. LECO recommends utilizing the 10 cm^3 dose loop when analyzing materials with lower nitrogen concentrations.

††Results were below the lower nitrogen detection range of the instrument.