Nitrogen in Soil and Plant Tissue

LECO Corporation; Saint Joseph, Michigan USA

Instrument: FP628

Introduction

Nitrogen determination is one of the most important elements for plant development and is the nutrient that is most often found to be deficient for crop production in arable soils. The accurate and precise determination of nitrogen before the planting season is essential in the overall fertilization strategy including predicting the fertilization need, choosing the type and concentration of fertilizer for a particular soil plot. Nitrogen determination in plant tissue is an important diagnostic tool giving the grower an indication of plant nutritional health and nutrient uptake efficiency as well as an avenue of monitoring high value intensively managed crops like tobacco, cotton and fruits. Often a combination of nitrogen determination in both the soil and plant tissue will be used to correctly diagnose and/or correct a growth issue with the plant.

The LECO FP628 is a combustion nitrogen/protein determinator 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 with no metal oxidizer reagents in the primary or secondary furnace. A thermoelectric cooler removes the moisture in the combustion gas without the use of chemical reagents. A 3 or 10 cc volume of combustion gas is taken using a combustion gas collection and handling system. The combustion gas collection and handling system achieves a low cost-per-analysis by reducing the amount of chemical reagents used for scrubbing and converting the nitrogen oxide combustion gas to nitrogen. A thermal conductivity (TC) cell is used for the detection of nitrogen in the combustion gas.

Sample Preparation

Samples must be of uniform consistency to produce suitable results. Samples should be ground to pass a 0.5 mm screen. Nitrogen results for soil and plant tissue materials are normally reported on a dry basis. The materials can either be dried prior to analysis or the moisture content determined and the values corrected. Please see the note addendum at the end of this document for details on drying these materials.

Accessories

502-186 Tin Foil Cup, 502-382 Quik-Caps™

Calibration Samples

502-092 EDTA, 502-642 Phenylalanine, 501-050 Nicotinic Acid, or other suitable pure compounds.

*Analysis Parameters**

Furnace Temperature	950°C
Afterburner Temperature	850°C

Instrument Model and Configuration

Thermal conductivity detectors work by detecting changes in the thermal conductivity of the analytical gas compared to the constant thermal conductivity of the reference gas. The greater the difference between the thermal conductivity of the carrier gas and the analyte gas, the greater the sensitivity of the detector. The FP628 is available in models that support either the use of helium or argon as the instrument's carrier gas for the thermal conductivity cell.

When used as a carrier gas, helium provides the highest sensitivity, providing the best performance at the lower end of the nitrogen range. Helium models also offer the additional advantage of replacing the 10 cc aliquot loop with a 3 cc loop within the instrument's gas collection and handling system. The 10 cc aliquot loop optimizes the instrument for the lowest nitrogen range and best precision. The 3 cc aliquot loop extends reagent life expectancy by approximately three fold compared to the 10 cc aliquot loop, while providing the lowest cost-per-analysis with minimal impact on practical application performance (see Typical Results section).

Due to the recent history of low supply and general availability issues for helium gas, the argon model was developed to utilize argon as a carrier gas. Since the thermal conductivity difference between argon and nitrogen is not as great as the thermal conductivity difference between helium and nitrogen, the detector is inherently less sensitive with argon as a carrier gas. The argon model (10 cc aliquot only) has a similar practical application performance compared to the helium model, operating with equivalent instrument and method configurations (see Typical Results section).

Note: Changing carrier gas and aliquot loop size requires hardware changes within the instrument.

Method Selection

Both the helium and argon (10 cc aliquot only) models and aliquot loop size system configurations have the option of a High Precision method or High Throughput method. The High Precision method is optimized to deliver the best performance in terms of nitrogen results resulting in an analysis time of 4 minutes. The High Throughput method is optimized to deliver the fastest analysis time of 3.5 minutes (210 seconds) while maintaining instrument performance specifications and acceptable practical application performance (see Typical Results section).

Organic Application Note

Delivering the Right Results

Element Parameters - Helium Model

	High Precision (10 cc & 3 cc)	*High Throughput (10 cc)*
Analyze	Yes	Yes
Baseline Delay Time	4 seconds	4 seconds
Min. Analysis Time	40 seconds	40 seconds
Comparator Level	1.00	1.00
Endline Time	2 seconds	2 seconds
Conversion Factor	1	1
Significant Digits	5	5
TC Baseline Time	10 seconds	6 seconds

Element Parameters - Argon Model

	High Precision (10 cc)	*High Throughput (10 cc)*
Analyze	Yes	Yes
Baseline Delay Time	4 seconds	4 seconds
Min. Analysis Time	60 seconds	60 seconds
Comparator Level	1.00	1.00
Endline Time	2 seconds	2 seconds
Conversion Factor	1	1
Significant Digits	5	5
TC Baseline Time	10 seconds	6 seconds

Burn Profile

Burn Steps	*Time (seconds)*	*Furnace Flow*
1	90 seconds	High

Ballast Parameters

	High Precision	*High Throughput*
Equilibrate Time	30 seconds	10 seconds
Not Filled Timeout	300 seconds	300 seconds
Aliquot Loop Fill Pressure Drop	200 mm Hg	200 mm Hg
Equilibrate Pressure Time	8 seconds	4 seconds

**Refer to FP628 Operator's Instruction Manual for Method 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 at least five results following the procedure outlined in the operator's instruction manual.
   e. The standard deviation of the last five blanks should be less than or equal to 0.001% (10 ppm) for nitrogen. Additional blanks beyond the recommended 10 may need to be analyzed in order to achieve the recommended precision.

Note: If Quik-Caps (502-382) will be used for sample analysis instead of Tin Foil Cups (502-186), the blank should be determined by analyzing empty Quik-Caps due to their marginal level of nitrogen that must be accounted for.

3. Calibrate.
   a. Weigh ~0.15 g of EDTA into a 502-186 Tin Foil Cup and seal. 502-382 Quik-Caps may also be used for analysis (please see the note addendum for more information).
   b. Enter sample mass and identification into Sample Login (F3).
   c. Transfer sample to the appropriate position in the sample carousel.
   d. Repeat steps 3a through 3c a minimum of five times.
   e. Initiate the analysis sequence (F5).
   f. Calibrate the instrument using single standard calibration (fixed at origin) following the procedure outlined in the operator's instruction manual.

Note: An FP628 can be calibrated using several replicates of a single mass range (nominal 0.15 g) of EDTA utilizing a single standard calibration. The calibration can be verified by analyzing a pure compound that is different than the material used for calibration, such as phenylalanine (~0.1 g) or nicotinic acid (~0.1 g). Multi-point (fractional weight or multiple calibration samples) may also be used to calibrate if desired.

4. Analyze Samples.
   a. Weigh ~0.25 g of the soil or plant tissue sample into a 502-186 Tin Foil Cup and seal. 502-382 Quik-Caps may also be used for analysis (please see the note addendum for more information).
   b. Enter mass and sample identification into Sample Login (F3).
   c. Transfer sample to the appropriate position on the sample carousel.
   d. Repeat steps 4a through 4c for each sample to be analyzed.
   e. Initiate the analysis sequence (F5).
5. Atmospheric Blank.
   Some atmosphere will be trapped with the sample when it is encapsulated in the tin foil cup. Some atmosphere may also be present when using the Quik-Caps as well. This will cause biased nitrogen results at low nitrogen concentrations (particularly with soil samples), therefore an atmospheric blank should be determined and entered using the following procedure.
   a. Analyze an inert material such as LECO 501-427 Com-Aid several times using similar weights of the Com-Aid to the weight of samples being analyzed.
   b. Enter the actual weight of the Com-Aid (Com-Aid should be baked-off in a muffle furnace at ~1000°C for 15 minutes, allowed to cool, and stored for up to 24 hours in a desiccator until used). The nitrogen value obtained is considered the atmospheric blank and can be automatically compensated using the FP628 software. Refer to the operator's instruction manual for details regarding the setting of the atmospheric blank.

Notes

Sample Drying Instructions

- Soils—Samples dried at 105°C for one hour prior to analysis.
- Plant Tissues—Samples dried at 85°C for two hours prior to analysis.

502-382 Quik-Cap Usage

- Only one-half (the longer, narrower portion) of the Quik-Cap is used for analysis. Please discard the other half.
- A sample is analyzed in the open cap, no sealing is required.

TYPICAL RESULTS - High Precision Method*

	3 cc Helium		10 cc Helium		10 cc Argon	
	Mass(g)	*% N*	*Mass(g)*	*% N*	*Mass(g)*	*% N*
502-062	0.2484	0.194	0.2700	0.196	0.2531	0.197
Lot: 1016	0.2578	0.191	0.2650	0.193	0.2466	0.196
Soil	0.2503	0.205	0.2720	0.197	0.2417	0.199
0.183% N ±0.015	0.2508	0.195	0.2750	0.197	0.2564	0.199
	0.2500	0.196	0.2651	0.197	0.2438	0.199
	Avg =	***0.196***	***Avg =***	***0.196***	***Avg =***	***0.198***
	s =	***0.005***	***s =***	***0.002***	***s =***	***0.001***
502-309	0.2492	0.97	0.2486	0.98	0.2565	0.92
Lot: 1013	0.2461	0.94	0.2459	0.98	0.2500	0.92
Soil	0.2472	0.93	0.2491	0.96	0.2566	0.92
0.94% N ±0.03	0.2415	0.95	0.2553	0.98	0.2483	0.94
	0.2586	0.95	0.2474	0.97	0.2569	0.94
	Avg =	***0.95***	***Avg =***	***0.97***	***Avg =***	***0.93***
	s =	***0.01***	***s =***	***0.01***	***s =***	***0.01***
502-055	0.2435	2.08	0.2375	2.03	0.2546	2.05
Lot: 1035	0.2416	2.07	0.2360	2.04	0.2512	2.04
Orchard Leaves	0.2480	2.07	0.2391	2.02	0.2589	2.06
2.04% N ±0.08	0.2438	2.06	0.2374	2.03	0.2429	2.05
	0.2409	2.06	0.2388	2.05	0.2449	2.04
	Avg =	***2.07***	***Avg =***	***2.04***	***Avg =***	***2.05***
	s =	***0.01***	***s =***	***0.01***	***s =***	***0.01***
502-082	0.2481	2.53	0.2474	2.54	0.2548	2.52
Lot: 1016	0.2543	2.52	0.2562	2.55	0.2450	2.50
Tobacco	0.2471	2.53	0.2423	2.55	0.2469	2.51
2.53% N ±0.03	0.2552	2.53	0.2462	2.55	0.2445	2.50
	0.2529	2.53	0.2521	2.56	0.2498	2.52
	Avg =	***2.53***	***Avg =***	***2.55***	***Avg =***	***2.51***
	s =	***0.01***	***s =***	***0.01***	***s =***	***0.01***

TYPICAL RESULTS - High Throughput Method*

	3 cc Helium		10 cc Helium		10 cc Argon	
	Mass(g)	*% N*	*Mass(g)*	*% N*	*Mass(g)*	*% N*
502-062	0.2675	0.187	0.2605	0.189	0.2459	0.187
Lot: 1016	0.2592	0.186	0.2615	0.181	0.2499	0.183
Soil	0.2528	0.183	0.2659	0.187	0.2527	0.182
0.183% N ±0.015	0.2635	0.184	0.2654	0.186	0.2574	0.186
	0.2624	0.183	0.2563	0.184	0.2445	0.189
	Avg =	***0.185***	***Avg =***	***0.185***	***Avg =***	***0.185***
	s =	***0.002***	***s =***	***0.003***	***s =***	***0.003***
502-309	0.2426	0.96	0.2559	0.96	0.2555	0.93
Lot: 1013	0.2471	0.95	0.2549	0.97	0.2502	0.92
Soil	0.2500	0.96	0.2560	0.96	0.2453	0.94
0.94% N ±0.03	0.2497	0.94	0.2462	0.95	0.2426	0.96
	0.2508	0.92	0.2596	0.97	0.2459	0.95
	Avg =	***0.94***	***Avg =***	***0.96***	***Avg =***	***0.94***
	s =	***0.02***	***s =***	***0.01***	***s =***	***0.02***
502-055	0.2471	2.05	0.2426	2.05	0.2568	2.05
Lot: 1035	0.2572	2.06	0.2467	2.04	0.2571	2.05
Orchard Leaves	0.2450	2.08	0.2400	2.02	0.2522	2.05
2.04% N ±0.08	0.2407	2.08	0.237	2.04	0.2488	2.06
	0.2519	2.06	0.2412	2.03	0.2503	2.07
	Avg =	***2.07***	***Avg =***	***2.03***	***Avg =***	***2.06***
	s =	***0.02***	***s =***	***0.01***	***s =***	***0.01***
502-082	0.2543	2.53	0.2534	2.55	0.2450	2.53
Lot: 1016	0.2481	2.53	0.2445	2.56	0.2444	2.53
Tobacco	0.2505	2.53	0.2421	2.54	0.2429	2.53
2.53% N ±0.03	0.2510	2.50	0.2407	2.54	0.2468	2.52
	0.2424	2.51	0.2411	2.55	0.2500	2.52
	Avg =	***2.52***	***Avg =***	***2.55***	***Avg =***	***2.52***
	s =	***0.01***	***s =***	***0.01***	***s =***	***0.01***

**Based on a single standard calibration with 0.25 g of 502-092 EDTA weighing into 502-186 Tin Foil Cups*

Organic Application Note

LECO Corporation | 3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
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