

Nitrogen in Soil and Plant Tissue

LECO Corporation; Saint Joseph, Michigan USA

Instrument: FP628

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 Quick 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

Element Parameters

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

Burn Profile

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

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 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 5 results following the procedure outlined in the operator's instruction manual.
 - e. The standard deviation of the last 5 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 Quick Caps (502-382) will be used for sample analysis instead of Tin Foil Cups (502-186), the blank should be determined by analyzing empty Quick 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 Quick 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 Quick 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 Quick 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: 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. 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 Quick Cap Usage

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

Typical Results

(Based on a single standard calibration with 0.15 g of 502-092 EDTA)

Sample	Mass g	% Nitrogen
Soil 502-308*	0.2515	0.182
Lot: 1016	0.2507	0.179
0.183 ±0.010% N	0.2518	0.182
	0.2507	0.180
	0.2495	0.180
	0.2501	0.178
	0.2506	0.180
	0.2506	0.178
	0.2494	0.177
	0.2529	0.180
	X =	0.180
	s =	0.002

Soil 502-309*	0.2503	1.11
Lot: 1009	0.2535	1.11
1.12 ±0.04% N	0.2500	1.10
	0.2497	1.11
	0.2528	1.11
	0.2503	1.11
	0.2504	1.12
	0.2498	1.11
	0.2499	1.11
	0.2531	1.10
	X =	1.11
	s =	0.01

Sample	Mass g	% Nitrogen
Soil 502-062*	0.2495	0.183
Lot: 1015	0.2504	0.181
0.180 ±0.014% N	0.2509	0.182
	0.2515	0.182
	0.2500	0.178
	0.2512	0.183
	0.2518	0.182
	0.2493	0.179
	0.2492	0.182
	0.2522	0.178
	X =	0.181
	s =	0.002

Soil 502-814*	0.2506	1.96
Lot: 1001	0.2519	1.97
1.97 ±0.06% N	0.2515	1.97
	0.2502	1.96
	0.2500	1.96
	0.2503	1.96
	0.2500	1.96
	0.2500	1.96
	0.2504	1.96
	0.2513	1.96
	X =	1.96
	s =	0.01



Sample	Mass g	% Nitrogen
Orchard Leaves	0.2494	2.28
502-055**	0.2529	2.29
Lot: 1032	0.2497	2.28
2.28 ±0.04% N	0.2501	2.28
	0.2478	2.28
	0.2485	2.28
	0.2489	2.28
	0.2505	2.28
	0.2517	2.28
	0.2497	2.25
	X =	2.28
	s =	0.01

Tobacco 502-082**	0.2478	2.56
Lot: 1015	0.2509	2.56
2.56 ±0.04% N	0.2490	2.56
	0.2470	2.56
	0.2502	2.56
	0.2476	2.56
	0.2469	2.56
	0.2497	2.56
	0.2499	2.55
	0.2477	2.55
	X =	2.56
	s =	0.01

Alfalfa 502-273**	0.2517	3.89
Lot: 1022	0.2493	3.88
3.91 ±0.03% N	0.2488	3.88
	0.2540	3.89
	0.2485	3.88
	0.2502	3.89
	0.2502	3.89
	0.2489	3.89
	0.2505	3.88
	0.2510	3.89
	X =	3.89
	s =	0.01

*Samples were dried at 105°C for one hour prior to analysis.

**Samples were dried at 85°C for two hours prior to analysis.

LECO Corporation

3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • ISO-9001:2008 HQ-Q-994 • LECO is a registered trademark of LECO Corporation.