

Instrument: TruMac® N

Determination of Nitrogen in Soil and Plant Tissue

LECO Corporation; Saint Joseph, Michigan USA

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.

Nitrogen determination is performed using either the classical wet chemical method (Kjeldahl) or a combustion method. The Kjeldahl method is capable of handling macro samples sizes ~1 g, typically utilized for heterogeneous soil samples. Physical restrictions in sample encapsulation, and realities of handling the ash build up within a vertical furnace combustion nitrogen instrument, will often restrict the sample mass to ~200 mg or less. This makes the accurate analysis of heterogeneous soils products difficult. The LECO TruMac N combustion nitrogen determinator has been designed to handle macro sample sizes (~1 g) while maintaining a rapid analysis time with automatic postanalysis ash removal.

The TruMac N is a macro combustion nitrogen determinator that utilizes a pure oxygen environment in a ceramic horizontal furnace and large ceramic boats for the macro sample combustion process. It uses a combustion gas collection and handling system using helium or argon carrier gas and a thermal conductivity cell for the detection of nitrogen.

Sample Preparation

Samples must be of uniform consistency to produce suitable results. Typically samples should be ground to a fineness of <0.5 mm. Reference materials should be prepared as directed by the certificate prior to analysis.

Note: Nitrogen results for soil and plant tissue samples are generally reported on a dry basis. The materials can either be dried prior to analysis or the moisture content can be determined on the day of analysis and used to correct the values for moisture utilizing the instrument's software. Plant samples are typically dried between 80 °C and 85 °C for two hours prior to analysis. Soil samples are typically dried at 105°C for one hour prior to analysis. The dried samples should be stored in a desiccator and must be used for analysis within 24 hours.

Accessories 528-203 Crucibles

Calibration Samples

502-092 EDTA, 502-642 Phenylalanine, 501-050 Nicotinic Acid

Analysis Parameters*

Furnace Temperature	1100°C
TE Cooler Temperature	5°C
Dehydration Time	0 seconds
Purge Cycles	2 seconds

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 TruMac 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 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.

Element Parameters

	Helium	Argon
	10 cc and 3 cc	10 cc
Baseline Delay Time	6 seconds	6 seconds
Minimum Analysis Time	35 seconds	55 seconds
Endline Time	2 seconds	2 seconds
Conversion Factor	1.00	1.00
Significant Digits	5	5
TC Baseline Time	10 seconds	10 seconds

Burn Profile

Burn Cycle	Lance Flow	Purge Flow	Time (seconds)
1	Off	On	5 seconds
2	On	On	35 seconds
3	On	Off	END

Ballast Parameters

Ballast

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Equilibrate Pressure Time	4 seconds
High Precision	Yes
High Speed	No

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

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing 3 to 5 blanks (crucible is not required).
3. Determine blank.
 - a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
 - b. Place a 528-203 Crucible to the appropriate position of the autoloader.
 - c. Repeat steps 3a through 3b a minimum of three times.
 - d. Initiate the analysis sequence (F5).
 - e. Set the blank following the procedure outlined in the operator's instruction manual.

4. Calibrate.

- a. Weigh ~0.75 g of EDTA calibration sample into a 528-203 Crucible, enter mass and sample identification into Sample Login (F3).
- b. Transfer crucible to the appropriate position of the autoloader.
- c. Repeat steps 4a through 4b a minimum of three times. Initiate the analysis sequence (F5).
- d. Calibrate the instrument following the procedure outlined in the operator's instruction manual. Use single standard calibration.

Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. Research has shown that a properly functioning TruMac can be calibrated using several replicates of a single mass range (nominal 0.75 g) of EDTA utilizing a single standard calibration. This is a cost effective and simple process. The calibration can be verified by analyzing different compounds such as nicotinic acid (0.25 to 0.5 g) and/or phenylalanine (0.5 to 0.75 g).

5. Analyze Samples.

- a. Weigh ~1 g of soil or ~0.5 g of plant tissue sample into a 528-203 Crucible; enter mass and sample identification into Sample Login (F3).
- b. Transfer crucible to the appropriate position of the autoloader.
- c. Repeat steps 5a through 5b for each sample to be analyzed.
- d. Initiate the analysis sequence (F5).

Note: If soot (carbon black) is noticed in the primary filter (steel wool filter), reduce sample mass to prevent soot build-up in this filter. Soot can be produced when larger masses of some sample types are analyzed.

Typical Results

(based on single standard calibration using 0.75 g EDTA)

	3 cc Helium		10 cc Helium		10 cc Argon	
	Mass(g)	% N	Mass(g)	% N	Mass(g)	% N
LECO 502-062	1.0268	0.185	1.0234	0.184	1.0213	0.180
Lot: 1016	1.0436	0.184	1.0071	0.184	1.0230	0.174
Soil	1.0267	0.185	1.0370	0.183	1.0146	0.183
0.183% N ± 0.015	1.0129	0.185	1.0277	0.182	1.0576	0.179
	1.0457	0.183	1.0173	0.184	1.0013	0.178
	Avg = 0.184		Avg = 0.183		Avg = 0.179	
	s = 0.001		s = 0.001		s = 0.003	
LECO 502-309	1.0047	0.95	1.023	0.97	1.0289	0.94
Lot: 1013	1.0155	0.95	1.0222	0.95	1.0195	0.94
Soil	1.0610	0.95	1.0211	0.95	1.0253	0.93
0.94% N ± 0.03	1.0224	0.94	1.0240	0.96	1.0394	0.94
	1.0050	0.94	1.0151	0.94	1.0199	0.96
	Avg = 0.95		Avg = 0.95		Avg = 0.94	
	s = 0.005		s = 0.01		s = 0.01	
LECO 502-055	1.0025	2.05	1.0122	2.06	1.0109	2.06
Lot: 1035	1.0411	2.04	1.0228	2.06	1.0451	2.05
Orchard Leaves	1.0464	2.04	1.0373	2.06	1.0109	2.06
2.04% N ± 0.08	1.0330	2.04	1.0058	2.06	1.0706	2.06
	1.0088	2.04	1.0113	2.05	1.0045	2.05
	Avg = 2.04		Avg = 2.06		Avg = 2.06	
	s = 0.004		s = 0.004		s = 0.005	
LECO 502-082	1.0570	2.52	1.0188	2.50	1.0408	2.52
Lot: 1016	1.0006	2.49	1.0288	2.53	1.0380	2.52
Tobacco	1.0002	2.49	1.0155	2.53	1.0439	2.52
2.53% N ± 0.03	1.0029	2.52	1.0199	2.53	1.0117	2.50
	1.0044	2.53	1.0511	2.52	1.0565	2.52
	Avg = 2.51		Avg = 2.52		Avg = 2.52	
	s = 0.019		s = 0.013		s = 0.009	



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