

N/Protein Determination in Soils according to the Dumas method (He/Ar as Carrier)

Reference: AOAC 993.13 Nitrogen (Total) in Fertilizers ISO 13878:1998 Soil quality -- Determination of total nitrogen content by dry combustion ("elemental analysis")

Tested with VELP Scientifica NDA 702 Dual Carrier Gas Dumas Nitrogen Analyzer (Code F30800080)



Introduction

The nitrogen in plants is a part of numerous compounds, including proteins, enzymes, nucleic acids, chlorophyll and vitamins essential for all biological functions.

It is taken from soil or from air, and it can be used only through a specified and complex chemical process called nitrogen cycle.

The performance of the VELP NDA 702 was evaluated by participating in the Proficiency Testing program organized by WEPAL (Wageningen Evaluating Programs for Analytical Laboratories).

Samples of soil were analyzed using the NDA 702 and the results obtained (as % Nitrogen) were compared with the statistical range accepted by WEPAL.

Nitrogen Determination in Soils

The Dumas method starts with a combustion furnace (CF) to burn the sample, obtaining elemental compounds.

Water is removed by a first physical trap (WT1 - **DriStep™**), placed after the combustion, and a second chemical one (WT2). Between the two, the elemental substances passed through a reduction furnace (RF).

The auto-regenerative CO₂ absorbers (CO₂) let pass only the elemental nitrogen that is detected by the **LoGas™** innovative Thermal Conductivity Detector (TCD) with no requirement for a reference gas.

The NDA 702 is controlled through the intuitive **DUMASoft™**.



NDA 702 Preliminary Operations (daily)

Follow the operating manual to start the NDA 702 and check that the following parameters are set:

Temperature Combustion reactor (Code A00000158): 1030 °C

Temperature Reduction reactor (Code A00000226): 650 °C

Flow rate MFC1 Carrier gas (He/Ar): 190 ml/min

Flow rate MFC2 Carrier gas (He/Ar): 220 ml/min

Condition the system by testing 2 EDTA standard (Code A00000149) and 3 to 5 empty tin foils (Code A00000153) as Check up.

Verify the calibration curve with one or more tests as Standard by testing the same standard used for the curve creation. Use the quartz ash insert (Code A00000161).

Sample Preparation

Using a spatula, put 300 mg of Soils Reference Material into the tin foil.

Close the tin foil, obtaining a capsule and load the capsule into the autosampler.

Analysis Procedure

Fill the following fields in the database: **Sample name, Weight, Method, Sample type, Calibration number**

Use the "Soil" method with the following parameters:

O₂ flow rate: 400 ml/min

O₂ factor: 0.7 ml/mg

Press to start the analysis.

Analysis time: from 3 minutes for one run.

Typical Results on Soil Reference Material

Results have been obtained with the following calibration curve: in a range of 0 – 1,2 mg N with 5 measurements of Rice Flour standard (N% = 1,38) (Code A00000235).

Sample	HELIUM as Carrier Gas		ARGON as Carrier Gas		Range of %N
	quantity (mg)	N %	quantity (mg)	N%	
Soil 1 (code WEPAL 2015.4_870)	299.10	0.165	299.85	0.161	
	305.75	0.163	301.53	0.161	
	300.22	0.164	302.52	0.162	
	Average ± SD%	0.164 ± 0.001	Average ± SD%	0.161 ± 0.001	0.146 - 0.166
	RSD% *	0.609	RSD% *	0.358	
Soil 2 (code WEPAL 2015.4_861)	303.53	0.163	301.74	0.159	
	300.34	0.164	304.62	0.161	
	300.16	0.162	303.05	0.162	
	Average ± SD%	0.163 ± 0.001	Average ± SD%	0.161 ± 0.002	0.150 - 0.180
	RSD% *	0.613	RSD% *	0.951	
Soil 3 (code WEPAL 2015.4_860)	304.38	0.169	304.35	0.166	
	300.00	0.170	304.11	0.166	
	301.34	0.169	305.11	0.168	
	Average ± SD%	0.169 ± 0.001	Average ± SD%	0.167 ± 0.001	0.147 - 0.175
	RSD% *	0.341	RSD% *	0.693	
Soil 4 (code WEPAL 2015.4_919)	305.95	0.153	305.95	0.156	
	300.13	0.152	301.85	0.153	
	299.78	0.150	302.36	0.154	
	Average ± SD%	0.152 ± 0.002	Average ± SD%	0.154 ± 0.002	0.139 - 0.165
	RSD% *	1.007	RSD% *	0.990	

* RSD% = (Standard Deviation * 100) / Average

Conclusion

Results are extremely reliable and reproducible, as demonstrated by the RSD, by using helium or argon as carrier gas, with the same conditions (method and sample weight) since the goal is to obtain < 2.0% relative standard deviation, as requested by official methods. The obtained values fell within the expected nitrogen range of each standard material certified by WEPAL, demonstrating the high performance of NDA702.

Helium remains the best choice for premium accuracy but its shortages and interruptions are affecting any related product or instrument, including elemental analyzers. Argon, the best alternative available, has demonstrated to be a valid substitute, ensuring optimal results. VELP Scientifica NDA 702 Dual Carrier Gas Dumas Nitrogen Analyzer is the perfect response to simple, fast and precise nitrogen/protein determination, both with Helium and Argon as carrier gas.