

APPLICATION NOTE

E-D-001-2015/A1

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

Reference: **AOAC 992.23** Crude Protein in Cereal Grains and Oilseeds; **AACC 46-30** Crude Protein Combustion Method; **ICC 167** Determination of crude protein in grain and grain products for food and feed by the Dumas Combustion Principle

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





N/PROTEIN DETERMINATION IN PLANTS DUMAS COMBUSTION METHOD

Introduction

Nitrogen is the most important nutritional element for plants, since it is involved in building of their proteins, nucleic acids and other several cell components. It is taken from soil or from air, and it can be used only through a specified and complex chemical process called nitrogen cycle.

Four Plants WEPAL samples (Wageningen Evaluating Programs for Analytical Laboratories, Wageningen University, Netherlands), are used to evaluate the reliability of the analytical method for the nitrogen and protein determination by using helium and argon as carrier gas with NDA 702. They were tested without any pre-treatment.

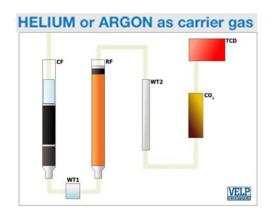
Nitrogen Determination in Plants

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 CO_2 absorbers (CO_2) let pass only the elemental nitrogen that is detected by the **LoGas**TM 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.

Sample Preparation

Using a spatula, put from 50 mg to 100 mg of Plants 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 "Plants" method with the following parameters:

O₂ flow rate: 400 ml/min O₂ factor: 1.4 ml/mg

Press o to start the analysis.

Analysis time: from 3 minutes for one run.



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Typical Results on Plants Reference Material

Results have been obtained with the following calibration curve: in a range of 0 - 4 mg N with 5 measurements of EDTA standard (N% = 9.57) (Code A00000149).

Sample	HELIUM as Carrier Gas		ARGON as Carrier Gas		Certified Average%N
	Quantity (mg)	N %	Quantity (mg)	N%	Range of %N
Broccoli IPE 132	50.24	3.744	50.21	3.771	3.79%
	50.63	3.661	50.49	3.908	3.56 - 4.01 %
	50.67	3.728	50.28	3.875	
	Average ± SD%	3.711 ± 0.044	Average ± SD%	3.851 ± 0.072	
	RSD% *	1.187	RSD% *	1.857	
Tulip IPE 175	100.87	1.301	101.29	1.333	1.30%
	101.40	1.293	101.77	1.328	1.26 - 1.34 %
	100.70	1.300	101.17	1.312	
	Average ± SD%	1.298 ± 0.004	Average ± SD%	1.324 ± 0.011	
	RSD% *	0.336	RSD% *	0.828	
Cord Grass IPE 955	100.05	2.044	100.42	2.034	2.01%
	101.15	2.006	100.81	2.042	1.87 - 2.14 %
	100.42	2.071	100.45	2.061	
	Average ± SD%	2.040 ± 0.033	Average ± SD%	2.046 ± 0.014	
	RSD% *	1.600	RSD% *	0.678	

^{*} 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.