

APPLICATION NOTE

F&F-D-002-2016/A1

Nitrogen Determination in Sport Powder according to the Dumas method (He/Ar as Carrier)

Reference: ISO 14891:2002 Milk and Milk Products

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





NITROGEN DETERMINATION IN SPORT POWDER DUMAS COMBUSTION METHOD

Introduction

Bodybuilders may supplement their diets with protein for reasons of convenience, lower cost (relative to meat and fish products) and to avoid the concurrent consumption of carbohydrates and fats. Protein supplements are sold in ready-to-drink shakes, bars, meal replacement products, bites, oats, gels and powders. Protein powders are the most popular and may have flavoring added for palatability. The powder is usually mixed with water, milk or juice and is generally consumed immediately before and after exercising, or in place of a meal.

Different sport powder products were chosen to demonstrate the suitability of the DUMAS method for Nitrogen/ Protein analysis by using Helium and Argon as carrier gas.

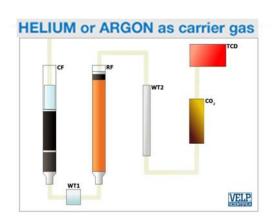
Nitrogen Determination in Sport Powders

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 adsorbers (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 ~ 30 - 50 mg Sport Powder into the tin foil according to the nitrogen content. 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** The ENERGY BAR method shows the following parameters:

Protein factor: 6.25 O₂ flow rate: 400 ml/min O₂ factor: 1.8 ml/mg

Press to start the analysis.

Analysis time: from 3 minutes for one run.



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Results on Sport Powder

Results have been obtained with the following calibration curve: in a range of 0 - 90 mg N with 8 measurements of EDTA standard (N% = 9.57) (Code A00000149) for both carrier gas.

The data obtained are included in the tolerance admitted by the EDTA certificate.

The table below shows the nitrogen/protein reproducibility of four sport powders analyzed for three consecutive times using Helium and Argon as carrier gas and a small sample weight (30 - 50 mg).

The sample weight is always the same with Helium and Argon as carrier gas.

	HEL	IUM as Carrier	Gas	ARGON as Carrier Gas			
Sample	Nitrogen %	Protein %	RSD%	Nitrogen %	Protein %	RSD%	
Sport powder 1	13.103 13.072 13.127	81.894 81.700 82.221	0.21	13.024 13.188 13.127	81.403 82.426 82.046	0.63	
Sport powder 2	11.629 11.630 11.479	72.679 72.690 71.742	0.75	11.621 11.569 11.597	72.633 72.305 72.478	0.22	
Sport powder 3	12.002 12.040 12.024	75.011 75.247 75.149	0.15	12.018 12.061 12.020	75.110 75.381 75.128	0.20	
Sport powder 4	14.173 14.126 14.110	88.583 88.288 88.186	0.23	14.136 14.165 14.073	88.351 88.530 87.959	0.33	

In order to prove the performance of two different NDA 702 Dumas Nitrogen Analyzer by using helium and argon carrier gas, four sport powder samples were analyzed for three consecutive times.

	HELIU	IM as Carrie	r Gas	ARGON as Carrier Gas				
Sample	NDA 702_A	NDA 702_A	NDA 702_B	NDA 702_B	NDA 702_A	NDA 702_A	NDA 702_B	NDA 702_B
	Average N %	RSD%	Average N %	RSD%	Average N %	RSD%	Average N %	RSD%
Sport powder 1	13.09	0.21	13.02	0.55	13.11	0.63	13.13	0.42
Sport powder 2	11.57	0.75	11.54	0.21	11.59	0.22	11.54	0.88
Sport powder 3	12.02	0.15	12.00	0.08	12.03	0.20	12.08	0.69
Sport powder 4	14.14	0.23	14.04	0.08	14.12	0.33	14.01	0.38



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Conclusion

The determination of nitrogen and protein in sport powder using NDA 702 provides reliable and reproducible results by using both Helium and Argon as carrier gas.

The data confirm the complete combustion of the sample with no memory effect observed when changing sample.

Results are extremely reliable, as demonstrated by the RSD, both using helium and 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.

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 excellent 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.