
Nitrogen Determination in Rice Flour according to the Dumas method (He/Ar as Carrier)

Reference: **AOAC 992.23** Crude Protein in Cereal Grains and Oilseeds

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



Introduction

Rice flour Certified Reference Material (CRM), traceable to NIST standard (SRM), is 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.

Nitrogen Determination in Rice Flour

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 adsorbers (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.

Sample Preparation

Using a spatula, put ~ 150 mg rice flour Certified Reference Material (CRM) 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**

The CEREAL MEAL 1 method shows the following parameters:

Protein factor: 6.25

O₂ flow rate: 400 ml/min

O₂ factor: 1.6 ml/mg

Press  to start the analysis.

Analysis time: from 3 minutes for one run.

Typical Results on Rice Flour

Results have been obtained with the following calibration curve: in a range of 0 - 5.9 mg N with 5 measurements of EDTA standard (N% = 9.57) (Code A00000149).

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

HELIUM as Carrier Gas		ARGON as Carrier Gas	
Sample quantity (mg)	Nitrogen %	Sample quantity (mg)	Nitrogen %
150.98	1.329	150.24	1.376
150.14	1.348	150.29	1.365
150.51	1.349	150.58	1.335
150.78	1.353	150.67	1.367
150.50	1.389	151.27	1.337
151.25	1.345	150.63	1.316
150.69	1.339	150.58	1.366
152.24	1.348	149.98	1.371
150.44	1.339	151.00	1.357
151.12	1.348	150.99	1.331
Average ± SD	1.349 ± 0.016		1.352 ± 0.021
RSD% *	1.168		1.520

Nitrogen Certified Value (Nitrogen %): 1.38 ± 0.05

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

Conclusion

The results show the reproducibility of 10 consecutive tests on rice flour Certified Reference Material (CRM), traceable to NIST standard (SRM).

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

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.