

APPLICATION NOTE F&F-D-007-2014/A3

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

Reference: AOAC 997.09 Nitrogen in Beer, Wort, and Brewing Grains Protein (Total)

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





N/PROTEIN DETERMINATION IN MALT DUMAS COMBUSTION METHOD

Introduction

Beer is the product of the alcoholic fermentation by yeast of extracts of malted barley.

The production of alcohols other than ethanol is linked with nitrogen uptake by yeast.

The yeast requires nitrogen in order to make protein and other nitrogenous cell components.

For this reason the monitoring of the protein content during the brewing process is important to ensure the survival, growth, and productivity of the yeast used to convert sugars to ethanol and carbon dioxide.

Moreover the protein content is an important criteria in evaluating the quality of beer: water-soluble barley proteins play a major role in the formation, stability, and texture of head foams.

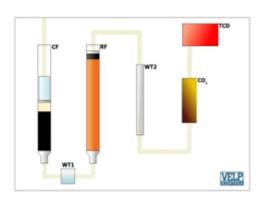
Protein Determination in barley malt

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 via PC 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 (He/Ar): 190 ml/min Flow rate MFC2 (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 ~ 200 mg of finely grinded sample directly 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.



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Typical Results on Barley Malt

The obtained results are in accordance with the expected value. Results have been obtained with the following calibration curve: in a range of 0 - 9.46 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)	Protein %	Sample quantity (mg)	Protein %
199.94	10.490	200.87	10.416
200.94	10.197	201.63	10.348
200.72	10.284	200.39	10.542
200.00	10.266	200.25	10.192
200.72	10.099	200.85	10.297
200.70	10.340	201.10	10.164
200.87	10.028	201.41	10.473
202.16	9.943	200.12	10.203
200.40	10.150	200.15	10.160
202.50	10.047	201.15	10.372
Average ± SD	10.184 ± 0.165		10.317 ± 0.136
RSD% *	1.618		1.316

Protein Expected Value: 9.8-10.8 %

Protein Factor: 6.25

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.

^{*} RSD% = (Standard Deviation * 100) / Average