Dumas Application

A.2.1.1 Nitrogen Determination in Meat and Meat Products

C. Gerhardt GmbH & Co. KG
Cäsariusstraße 97
53639 Königswinter, Germany
☎+49 (0)2223 2999-0
✉ info@gerhardt.de
✉ application@gerhardt.de
@ http://www.gerhardt.de

1 Principle

The nitrogen contained in the sample is oxidized to nitrogen oxides in an oxygen atmosphere, at high temperatures and in the presence of a catalyst. Then the nitrogen oxides are reduced to nitrogen with the help of copper. The side products, water and carbon dioxide, are separated in specific traps. Last, the nitrogen is detected by a thermal conductivity detector (TCD) and its amount is determined using a calibration previously performed by analysing a suitable substance with known nitrogen content.

2 Methods

This application note is meant to be a guideline for the operation of your C. Gerhardt analysis system and has to be adapted to your sample matrix and the local circumstances in your laboratory.
The document is based on

- AOAC 992.15, Crude Protein in Meat and Meat Products, 1992.
- Amtliche Sammlung von Untersuchungsverfahren nach § 64 LFGB L07.00-68, Untersuchung von Lebensmitteln - Bestimmung des Rohproteingehaltes in Fleischerzeugnissen - Dumas-Verfahren, März 2021.

3 Gases and Consumables

The following gases and consumables are needed for the operation of DUMATHERM:

- Helium cylinder gas, quality grade min. 5.0
- Oxygen cylinder gas, quality grade min. 5.0
- Nitrogen or air cylinder gas, quality min. 2.6, resp. quality grade 1 acc. ISO 8573-1 (free from moisture, particles or oil)
- DumaCollect, ash insert with bottom (14-0015)
- DumaFoil, tin foil for sample wrapping (14-0017) or DumaFoil XL, tin foil specially designed for weighing in larger samples (14-0417)
- DumaSorb, absorbent for liquid samples (14-0022) or Super-absorber, absorbent special for low-salt and low-fat liquid samples (14-0295)
- DumaReact, prepacked combustion reactor filled with HT- and LT-catalyst (14-0244) or DumaReact, prepacked combustion reactor filled with chromium-free catalyst (14-0245)
- DumaTube, quartz tube for reactor (14-0203)
- DumaPad, wool pads for reduction reactor (14-0225)
- DumaCop, copper for reduction (14-0046)
- Water trap (14-0061) filled with adsorbent (14-0219) and wool (14-0243)
- CO2 adsorber tube (14-0021)
- DumaEDTA, calibration standard, purity > 99 % (14-0032)
- THAM, Tris(hydroxymethyl)aminomethan, purity > 99 %

4 Instruments

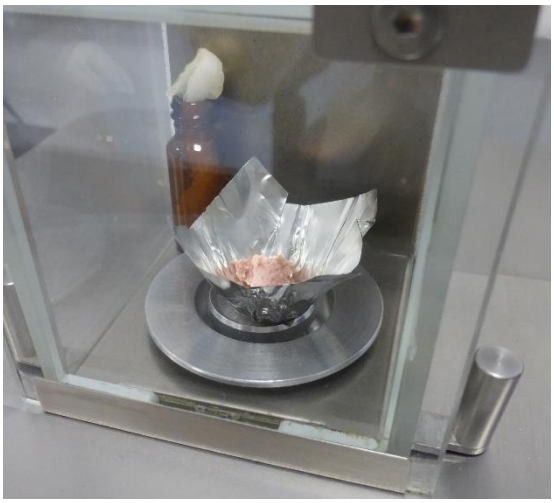
- Knife mill
- Analytical balance (accuracy at least 0.1 mg, preferably 0.01 mg)
- 14-0400, DUMATHERM DT N Pro, with starter kit and consumables

5 Procedure

5.1 Sample preparation and weighing

Parameter	Setting
Sample weight	200 mg +/- 10 mg
Sample packing	DumaFoil or DumaFoil XL Absorbent if needed (if fat or moisture content > 20 %)

- Take a representative sample.
- Mix and homogenize the sample for instance using a knife mill.
- Store the samples air-tight, to prevent deterioration or alteration of the composition.
- Mix the samples thoroughly again by hand.
- Prepare either the DumaFoil or the DumaFoil XL.
- Add some DumaSorb (ratio 1:3) if the sample has a high fat content (> 20 %). Add some Super-absorber (ratio 1:10) if the sample has a high moisture content (> 20 %).
- Press tare on the balance and add the sample (about 200 mg).



About 200 mg of liver sausage weighted in DumaFoil XL

- Take note of the first stable weight or transfer the weighing data from the balance automatically into the software DUMATHERM-Manager.
- Close the tin foil and place the sample in the transfer tray.

5.2 Daily Routine

Before the analysis, wake-up the DUMATHERM and perform the quality assurance described in the operating instructions of the instrument (Check-up maintenance, Check-up leaktest, Check-up blank value, Check-up standard).

For further information refer to the C. Gerhardt document “Guideline DUMATHERM N Pro daily routine”.

5.3 Combustion of the sample

For the combustion of meat and meat products we recommend the following settings:

Parameter	Setting	
Combustion Method	B 1,4 (with 1,4 ml O_2 / mg sample and a dosing speed of 300 ml/min) For samples with high fat content (> 20%), more oxygen is needed, and a slower combustion should be used. For instance, C 1,8 (with 1,8 ml O_2 / mg sample and a dosing speed of 200 ml/min)	
Combustion temperature	With DumaReact 14-0244: 980 °C	With DumaReact 14-0245: 1030 °C
Reduction temperature	With DumaReact 14-0244: 650 °C	With DumaReact 14-0245: 750 °C

5.4 Calibration

The calibration chosen must cover the working range. Using an initial weight as recommended, a calibration performed with EDTA from 1 mgN to 10 mgN is usually sufficient. The minimum requirement for the correlation factor R2 is a value ≥ 0,9999.

For further information refer to the C. Gerhardt document “Guideline DUMATHERM N Pro calibration”.

6 Example results: liver sausage

200 mg of liver sausage were weighted and then combusted with the method B1,4. The following results were obtained:

Dumatherm Nitrogen / Protein Analyser

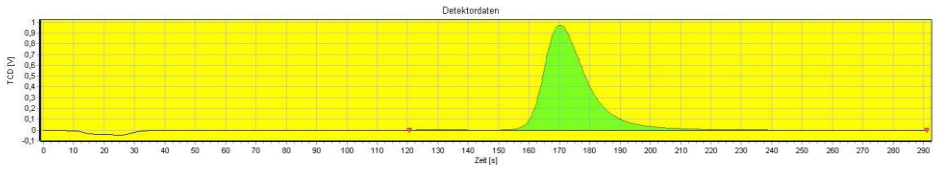
Serial Number : 1005 Submitter :
Software Version: DUMATHERM MANAGER V8.11 Operator : Administrator

Date	Time	Sample name	Weight [mg]	Moisture [%]	Protein factor	Nitrogen Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]	Protein [%]
28.02.2022	12:04:33	7948	213,303	0,00	6,25	1,734E+04	4,480	2,100	13,13
28.02.2022	12:09:56	7948	212,305	0,00	6,25	1,674E+04	4,326	2,037	12,73
28.02.2022	12:15:08	7948	206,752	0,00	6,25	1,681E+04	4,343	2,101	13,13
28.02.2022	12:20:36	7948	238,773	0,00	6,25	1,918E+04	4,952	2,074	12,96
28.02.2022	12:25:41	7948	217,023	0,00	6,25	1,731E+04	4,470	2,060	12,87
28.02.2022	12:30:59	7948	203,588	0,00	6,25	1,612E+04	4,166	2,046	12,79

Average	2,070	12,94
Standard Deviation	0,027	0,17
RSD [%]	1,294	1,29

Calibration number and standard name : for N TGIF EDTA 1 - 20 mg EDTA
Method : B1,4
Sample Table : 7948

Results obtained for the sample 7948



Example of peak plot

According to AOAC 992.15 for meat and meat products (with 10-20% crude protein), the repeatability standard deviation obtained with the results of the interlaboratory study is s_r = 0,12-0,41 % (for % Protein). The results achieved with the DUMATHERM are compliant.

COMPREHENSIVE APPLICATION DATA BASE

C. Gerhardt offers a wide range of application notes for many methods and procedures. Please contact our application lab team via application@gerhardt.de for deeper information on:

- Nitrogen in food and feed samples according to Kjeldahl and Dumas
- Crude fibre, ADF and NDF in feed
- Fat in food and feed
- Alcohol determination
- Total cyanide in water
- Trace metal in soil and sludge
- COD determination in water
- Total nitrogen determination in water, soil and plants
- Many more application notes on request.

An excerpt from our product portfolio

Fully AUTOMATIC HYDROLYSIS

HYDROTHERM – automatic acid hydrolysis system for fat determination according to Weibull-Stoldt. When combined with SOXTHERM, HYDROTHERM is an ideal system solution for total fat determination.

Fully AUTOMATIC FAT EXTRACTION

SOXTHERM – automatic fast extraction system for fat determination.

Fully AUTOMATIC WATER STEAM DISTILLATION

VAPODEST – fast distillation system for Kjeldahl nitrogen/ protein determination and steam distillation as sample preparation for further analysis.

COMPLETELY AUTOMATIC NITROGEN ANALYSIS

DUMATHERM – nitrogen/protein determination of solid and liquid samples according to the Dumas combustion method. A fast and convenient alternative to the classic Kjeldahl method for almost all sample matrices.

AUTOMATED CRUDE FIBRE DETERMINATION

FIBRETHERM – completely automated processing of the boiling and filtration processes for determining crude fibre, ADF and NDF.

www.gerhardt.de