

# Dumas Application

## A.3.1.2.1 Nitrogen Determination in Milk

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## 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. The nitrogen oxides are then reduced to nitrogen with the help of copper. The side products, water and carbon dioxide, are separated in specific traps. Finally, the nitrogen is detected by a thermal conductivity detector (TCD) and its quantity is determined on the basis of a previously performed calibration by analysing a suitable substance with a known nitrogen content. The nitrogen content is converted to protein content using the referring protein factor.

## 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.

This document is based on

- ISO 14891:2002 | IDF 185:2002, Milk and milk products — Determination of nitrogen content — Routine method using combustion according to the Dumas principle.
- Amtliche Sammlung von Untersuchungsverfahren nach LMBG, § 35 LMBG L 02.00-24, Determination of nitrogen content in milk products, Procedure according to Dumas, January 1997.

## 3 Gases and Consumables

The following items and gases are required for the operation of N-Realizer:

- 3.1 Helium cylinder gas, quality grade min. 5.0
- 3.2 Oxygen cylinder gas, quality grade min. 5.0
- 3.3 Compressed air, class 3 as per ISO 8573-1  
Alternative to compressed air: nitrogen cylinder gas, quality grade 2.6 (99.6 %, oil and water-free)
- 3.4 DumaFoil, tin foil for sample wrapping (Order number 14-0017) or DumaFoil XL, tin foil specially designed for weighing in larger samples (Order number 14-0417)
- 3.5 DumaSorb, absorbent for liquid samples (Order number 14-0022) or Super-absorber, absorbent special for low-salt and low-fat liquid samples (Order number 14-0295)
- 3.6 DumaCollect, ash insert with bottom (Order number 14-0015)
- 3.7 DumaReact, prepacked combustion reactor filled with HT- and LT-catalyst (Order number 14-0244)
- 3.8 DumaTube, quartz tube for reactor (Order number 14-0203), DumaPad, wool pads for reduction reactor (Order number 14-0225), DumaCop, copper for reduction (Order number 14-0007)
- 3.9 Water trap filled with adsorbent silica gel and wool (Order number 14-0217, 14-0219, 14-0243)  
**Note:** A mixture of 10 g silica gel (Order number 14-0219) and 30 g magnesium perchlorate (CAS 10034-81-8, ThermoFisher Scientific, 99% wasserfrei, ACS, 011636.36) can be prepared and used for the water trap to extend its lifetime. For further details, refer to the operating instructions of N-Realizer, chapter 10.4.
- 3.10 REAL-N Spiral adsorber for CO<sub>2</sub> (Order number 14-4085)
- 3.11 DumaEDTA, calibration standard, purity > 99 % (Order number 14-0032)
- 3.12 THAM, Tris(hydroxymethyl)aminomethane, purity > 99 %

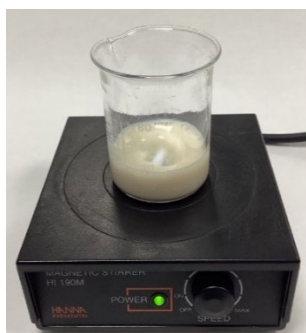
#### 4 Instruments

- Water bath
- Magnetic stirrer
- Analytical balance (accuracy at least 0.1 mg, preferably 0.01 mg)
- N-Realyzer basic unit, with starter kit and consumables, Order number 14-4000

#### 5 Procedure

##### 5.1 Sample preparation and weighing

The sample must be representative and homogeneous. The sample is warmed in a water bath set at 38°C-40°C and agitated using a magnetic stirrer during the weigh in.



*Stirring of the milk during the weighing*

Then, DumaFoil (3.4) is tared and some Super-absorber (3.5) is added with the ratio 1:10 (1 mg of Super-absorber for 10 mg of sample). The balance is tared again and 150 mg of sample are added using a syringe. The first stable weight is either noted or automatically transferred from the balance into the software REAL-OS.



*Weighing of 150 mg of milk in a DumaFoil*

After having waited a few seconds to let the Super-absorber react with the sample, the tin foil is closed and placed in the transfer tray.

**Note:** In case of whole milk, the use of DumaSorb (3.5) with the ratio 1:3 (1 mg of DumaSorb for 3 mg of sample) may be required.

### 5.2 Daily Routine

Before the analysis, perform the quality assurance described in the operating instructions of N-Realyzer (Check-up consumables, Check-up leak test, Check-up blank value, Check-up standard).

Check-up consumables	For further details about the handling of consumables, refer to the operating instructions of N-Realyzer, chapter 8.1.2, chapter 10.2 (reactors), chapter 10.3 (crucibles), chapter 10.4 (water trap) and chapter 10.5 (CO <sub>2</sub> adsorbers).
Check-up leak test	For further details about the leak test, refer to the operating instructions of N-Realyzer, chapter 8.1.3.
Check-up blank value	For further details about the blanks, refer to the operating instructions of N-Realyzer, chapter 8.1.4.
Check-up standard	For further details about the standards, refer to the operating instructions of N-Realyzer, chapter 8.1.4.

### 5.3 Combustion of the sample

For the combustion of milk, we recommend the following settings:

Parameter	Setting
Combustion method	C 1.2 (with 1.2 ml O <sub>2</sub> / mg sample and a dosing speed of 200 ml/min)
Combustion temperature	With DumaReact (3.7): 980 °C
Reduction temperature	With DumaReact (3.7): 650 °C

**Note:** For further information about the optimization of the combustion method, refer to the operating instructions of N-Realyzer, chapter 13.2.

### 5.4 Calibration

The selected calibration must cover the working range. Using a sample weight as recommended, a calibration performed with a THAM solution 0,5%N till 2,5 mgN is sufficient.

**Note:** A THAM solution 0.5%N is prepared by weighing 4.324 g of THAM (tris(hydroxymethyl)aminomethane, H<sub>2</sub>NC(CH<sub>2</sub>OH)<sub>3</sub>) (3.12) in 100 ml of distilled water.

The minimum requirement for the correlation factor R<sup>2</sup> is a value ≥ 0.999.

**Note:** It is also possible to use a milk reference sample as standard for the calibration.

**Note:** For further information about the calibration, refer to the operating instructions of N-Realyzer, chapter 8.2 and 13.1.

## 6 Comparison of Dumas and Kjeldahl results

“Recent interlaboratory studies and several proficiency testing studies show that results for milk, which has not been predried, obtained using the Dumas method are not fully comparable to those obtained with the Kjeldahl method, as specified in ISO 8968-1 | IDF 20-1, and do not fulfil the precision limits of the latter method.

However, reported precision data for yogurt, evaporated milk, sweetened condensed milk, cream cheese, Cheddar cheese, Swiss cheese, Parmesan cheese, non-fat dry milk, whey protein concentrate, sodium caseinate and dried skimmed milk (under repeatability and reproducibility conditions) obtained using the Dumas method are equal or even better than those obtained with the Kjeldahl method for the same test sample.

Mean values obtained using the Dumas method for test samples of milk and milk products (for raw and whole milk, skimmed milk powder, whey powder, whole milk powder, whey protein concentrate, cheese and whey) are generally 1 % to 3 % (relative) higher than those obtained with the Kjeldahl method for the same test sample.”

**Source:** ISO 14891:2002 | IDF 185:2002, Milk and milk products — Determination of nitrogen content — Routine method using combustion according to the Dumas principle.



### COMPREHENSIVE APPLICATION DATA BASE

C. Gerhardt offers a wide range of application notes for many methods and procedures.

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- 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.

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