Application Short Note DUMATHERM

Nitrogen in Hard Cheese / Hartkäse

Applied method (e.g. AOAC, DIN, EN, ISO, EPA, ASTM, §64, company sop, etc.)

DIN EN ISO method 14891, IDF 185, Milk and Milk Products – Determination of nitrogen content – routine method using combustion according to Dumas principle.

Instruments

1	Analytical Balance (readability 0,1 mg or better)
2	Homogenizer, e.g. Grindomix GM200 Knife Mill or a regular Knife
3	DUMATHERM N Pro, standard configuration

Gases and Consumables

1	Helium and Oxygen, bottle gas, min. quality grade 5.0	
2	Nitrogen or compressed air as bottle gas, min. quality grade 2.6.	
3	DumaReact, Combustion Reactor, packed with catalyst,	14-0245
4	DumaTube, Quartz glass for reactor,	14-0203
5	DumaFoil, Tin Foil for packing the samples,	14-0017
6	DumaEDTA, Standard for Calibration, purity > 99 %,	14-0032

Method Settings

Sample Weight	200 mg
Packing of the sample	Tin foil
Combustion Method	A 1,8 (400 ml O_2 / min, 1.8 ml O_2 / mg sample
Protein Factor	6,38
Combustion Temperature	980 °C (alternatively 1030 °C)
Reduction temperature	650 °C (alternatively 750 °C)
Recommended Calibration Range	1 – 15 mg N absolute (measured with 10-150 mg EDTA)

Homogenization / Preparation

The sample is taken from the fridge and directly homogenized with a regular kitchen grinder (type Moulinette). The mashed material is thoroughly mixed again with a spatula and taken with a spatula for weighing into the tin foil.

The material should be at room temperature (≤ 20 °C) at most when it is weighed in.

Alternatively the sample is taken from the fridge and is knead at room temperature in the foil packaging to get a homogeneous sample material. The homogenized sample is then taken with a spatula for weighing.

Higher temperatures for combustion (1030 °C) and reduction (750 °C) will improve slightly the standard deviation. But as norm requirements are fulfilled already with the reduced temperatures, there is no need for the higher ones.

Example Results

Dumatherm Nitrogen / Protein Analyser

Serial Number: 0 Submitter :
Software Version: DUMATHERM MANAGER V4.17 Operator : Serviceman

Date	Time	Sample name	Weight [mg]	Moisture [%]	Protein factor	Nitrogen Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]	Protein [%]
27.05.2015	12:23:45	4889 Hartkäse MUVA	198.953		6.38	4.155E+04	10.571	5.313	33.90
27.05.2015	12:28:28	4889 Hartkäse MUVA	201.241		6.38	4.208E+04	10.707	5.320	33.94
27.05.2015	12:33:12	4889 Hartkäse MUVA	203.563		6.38	4.257E+04	10.819	5.315	33.91
27.05.2015	12:37:55	4889 Hartkäse MUVA	201.797		6.38	4.219E+04	10.721	5.313	33.90
27.05.2015	12:42:38	4889 Hartkäse MUVA	201.297		6.38	4.210E+04	10.699	5.315	33.91

Average	5.315	33.91
Standard Deviation	0.003	0.02
RSD [%]	0.058	0.06

Calibration number and standard name : for N EDTA 25-02-2014 (L-Q-Q)
Method : A 1,8
Series Name : Mai 15

Temperatures:		Flow Rates:		Times:	
Combustion Reactor	979 °C	He I	194.0 sccm	Sample Delay	7 s
Reduction Reactor	649 °C	He II	199.0 sccm	Sample Stop	11 s
Degassing Oven	299 °C	O_2	299.0 sccm	Run Time	Auto

The ISO norm requires for hard cheese of cheddar type a repeatability of at least 0,093 %N. Getting a standard deviation of 0,003 %N we exceed these requirements. No further increase of sample weight necessary.

Remarks

It is important to take a representative sample weight of the well homogenized sample material at room

The ISO norm suggests at least 200 mg as initial sample weight.