NITROGEN IN CHOCOLATE

1. Introduction
This application describes the determination of nitrogen/protein in chocolate using the combustion method acc. to Dumas.

2. Principle
The sample is combusted in an atmosphere of pure oxygen. The resulting nitrogen oxides are reduced with the help of copper. After separating the side products, carbon dioxide and water, the detection of the nitrogen is done using a calibrated thermal conductivity detector.

3. Reference method
- C. Gerhardt laboratory application

This application document is intended to be a guide to assist users in the initial use of C. Gerhardt analytical equipment. It is not a definitive method. Users may have to adapt this method to suit their own analytical requirements.

4. Gases and consumables required
- Helium 5.0[1)]
- Oxygen 5.0
- Nitrogen 2.6
- DumaFoil, Conditioned tin foil, cat. no. 14-0017
- DumaEDTA, ((Ethylenediaminetetraacetic acid) $C_{10}H_{16}N_2O_8$, standard for calibration, min. purity > 99 %[2)]), cat. no. 14-0032
- DumaReact, Prepacked combustion reactor, filled with HT and LT catalyst, cat. no. 14-0244
- DumaCop, Copper for reduction, cat. no. 14-0046
- DumaTube, Quartz tube for reactor, cat. no. 14-0203
- DumaPads, Quartz wool pads, 30 pcs small, 30 pcs large, cat. no. 14-0225
- DumaCollect, Ash insert with bottom, cat. no. 14-0015

[1)] The given qualities are minimum qualities and present at 5.0 a purity degree of at least 99,999 %
[2)] The calibration standard used should have a nitrogen content in the range of the unknown sample

5. Instruments
- Analytical balance, precision 0.1 mg
- DUMATHERM DT N40+, 40-place, with Starter Kit, cat. no. 14-0000 or
- DUMATHERM DT N2, 2-place, with Starter Kit, cat. no. 14-0003

▪ Sample preparation
Common chocolate is cut into 150 mg–squares and without further comminution used for the combustion analysis.

7. Analysis
7.1. General parameters
Prior to the analysis of an unknown sample, the DUMATHERM has to be activated according to the recommended quality control (a stable blank value has to be reached, check of a standard as unknown sample). The flow rates for the gases used are preset in the software (also see print out of results on the next page). The initial sample weights should be as consistent as possible (+/- 10 mg) and should correspond to the recommended initial weight.

7.2. Sample specific parameters

Recommended analysis parameters:

Initial weight:	140 to 170 mg
Category:	C 1.0
Sample type:	unknown
Combustion temperature:	990 °C
Ash insert:	ceramic

7.3. Calibration

In order to calculate the results of the analysis a calibration must be used, which coves the signal amplitudes of the unknown sample completely. When working with the initial sample weight recommended, signal amplitudes of about 8000 - 12000 mVs are reached. Thus, a calibration of 1 to 5 mg N absolute, ideally measured with EDTA in the weighing range of 10 to 50 mg, is necessary.

8. Sample data

Dumatherm Nitrogen / Protein Analyser

Serial Number : 19 Submitter:
Software Version: DUMATHERM MANAGER V2.04d Operator: Küppers

Date	Time	Sample name	Weight [mg]	Standard name	Category	Protein factor	Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]	Protein [%]
17.09.2007	13:31:58	Schokolade LVU 2002	184,700		C 1,0		9,692E+03	2,322E+00	1,257	
17.09.2007	13:36:18	Schokolade LVU 2002	196,300		C 1,0		1,027E+04	2,456E+00	1,251	
17.09.2007	12:45:55	Schokolade LVU 2002	160,300		C 1,0		8,238E+03	1,982E+00	1,237	
17.09.2007	12:13:32	Schokolade LVU 2002	159,000		C 1,0		7,961E+03	1,917E+00	1,239	

Average	1,246	
Standard Deviation	0,010	
RSD [%]	0,779	

Calibration # : 30 (Cubic, With Zero)
Analysis Conditions for Method : Factory
Sample Table : Proben August 2007

Temperatures:

Combustion Reactor	990 °C
Reduction Reactor	649 °C
Degassing Oven	298 °C

Times:

Sample Delay	5 s
Sample Stop	9 s
Run Time	Auto

Flow Rates:

He I	194 sccm
He II	200 sccm
O_2	197 sccm