NITROGEN IN GRAIN

1. Introduction
This application describes the determination of nitrogen of grain flour, using the combustion method acc. to Dumas.

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

3. Reference method
- C. Gerhardt laboratory application
- DIN EN ISO 16634, Determination of the content of total nitrogen and raw protein in wheat and feed using the combustion method acc. to Dumas
- AOAC 992.23 Crude Protein in Cereal Grains and Oilseeds
- ICC Standard method no. 167, 2000

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

6. Sample preparation
Common grain flour is used for analysis without further sample preparation. When working with grain kernels, they have to be pulverized to flour using a mesh size of 1 mm.

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 pre-set in the software (also see print out of results on 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 for N-contents lower than 3.5 %:

Initial weight:	130 to 160 mg
Category:	B 1.0
Sample type:	unknown
Combustion temperature:	990 °C
Protein factor:	5.70 (or other)

- Recommended analysis parameter for N-contents higher than 3.5 %:

Initial weight:	130 to 160 mg
Category:	B 1.3
Sample type:	unknown
Combustion temperature:	990 °C
Protein factor:	5.70 (or other)

7.3. Calibration

In order to calculate the results of the analysis a calibration must be used, which covers the signal height of the unknown sample. Using the recommended initial sample weight for wheat samples, peak areas of about 12000 - 15000 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. If the absolute amount of nitrogen in the sample exceeds the 5-mg-limit, a calibration of 5 - 25 mg N absolute has to be used.

8. Sample data

Dumatherm Nitrogen / Protein Analyser

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

Date	Time	Sample name	Weight [mg]	Standard name	Category	Protein factor	Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]	Protein [%]
10.05.2007	17:25:57	1600	153,600		B 1,0	5,70	1,405E+04	3,388E+00	2,206	12,57
10.05.2007	17:29:38	1600	151,500		B 1,0	5,70	1,380E+04	3,329E+00	2,197	12,52
10.05.2007	17:33:15	1600	148,900		B 1,0	5,70	1,358E+04	3,277E+00	2,201	12,55
10.05.2007	17:36:59	1600	159,000		B 1,0	5,70	1,448E+04	3,488E+00	2,194	12,50

Average	2,199	12,54
Standard Deviation	0,005	0,03
RSD [%]	0,236	0,23

Calibration # : 15 (Cubic, With Zero)
Analysis Conditions for Method : Method_1
Sample Table : Ostern 2007 Mai

Temperatures:

Combustion Reactor	990 °C
Reduction Reactor	650 °C
Degassing Oven	299 °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	294 sccm

9. Comments

Common grain flour is used for analysis without further sample preparation. When grain kernel is present it has to be pulverized to flour using a mesh size of 1 mm.
A standard deviation of 1,324% has to be reached, according to the DIN standard above. Thus, the analysis with DUMATHERM meets the requirements of this standard.