Application Short Note DUMATHERM

Nitrogen/Protein in Organic Fertilizer (humus / pulp)

Gerhardt Analytical Systems

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

AOAC 993.13: Nitrogen (Total) in Fertilizers, DIN ISO 13878 (1998, Determination of the total nitrogen content by dry combustion (elemental analysis).

Instruments

1	Analytical Balance (readability 0,1 mg or better)
2	Homogenizer, e.g. Speed Rotor Mill, Pulverisette 14 (Fritsch), 1 mm sieve size
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,	14-0244
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	100 +/- 10 mg
Packing of the sample	Tin foil 14-0017
Combustion Method	C 2,2 (O_2 dosing 200 ml O_2 / min, 2.2 ml O_2 / mg sample
Protein Factor	6,25
Combustion temperature [°C]	1030
Reduction temperature [°C]	750
Recommended Calibration Range	1 – 25 mg N abs. (EDTA, 20 equidistant points, 10-250 mg)

Homogenization / Preparation / Weighing

The sample was measured as received. In case the level of homogenization is not resulting in acceptable repeatability's according to the above mentioned norm (s_r <= 0,11-0,26), it is recommended to grind the sample down to 1 mm sieve size with a speed rotor mill

Sample orignal, humus

Original sample, pulp

... weighing process

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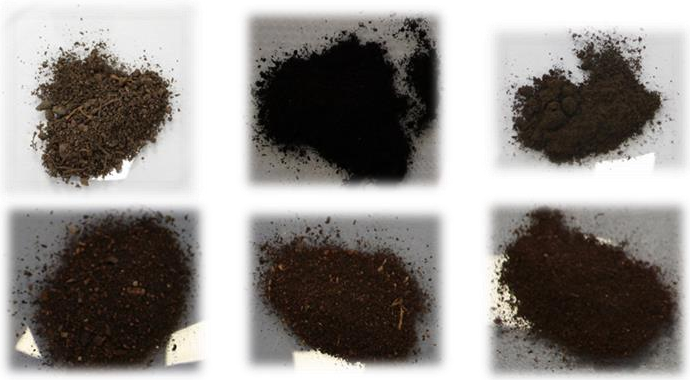
Homogenization – different tests were done to improve results.
Using a speed rotor mill, the sample was grinded to 2 and 1 mm sieve size to check repeatability. A sieve size of 1 mm was found to be suitable for this kind of sample.

Speed Rotor Mill

Humus / Pulp / Fruchtfleisch

Pomace / Trester



Original **2 mm** **1 mm**

Example Results

Results with the sample grinded to 1mm

Date	Time	Sample name	Weight [mg]	Protein factor	Nitrogen Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]	Protein [%]
16.09.2020	10:21:07	Pulp (7431)	100,200	6,25	9,909E+03	2,427	2,422	15,14
16.09.2020	10:26:10	Pulp (7431)	100,800	6,25	1,049E+04	2,570	2,550	15,94
16.09.2020	10:31:15	Pulp (7431)	100,900	6,25	9,479E+03	2,321	2,300	14,37
16.09.2020	10:36:24	Pulp (7431)	102,500	6,25	1,046E+04	2,562	2,500	15,62
16.09.2020	10:41:31	Pulp (7431)	101,000	6,25	9,324E+03	2,282	2,260	14,12
16.09.2020	10:46:38	Pulp (7431)	100,400	6,25	9,600E+03	2,350	2,341	14,63
16.09.2020	10:51:41	Pulp (7431)	99,300	6,25	9,658E+03	2,365	2,381	14,88
16.09.2020	10:56:44	Pulp (7431)	101,100	6,25	9,903E+03	2,425	2,399	14,99
16.09.2020	11:01:50	Pulp (7431)	100,200	6,25	9,724E+03	2,381	2,376	14,85
16.09.2020	11:06:58	Pulp (7431)	100,500	6,25	9,899E+03	2,424	2,412	15,08
16.09.2020	11:12:06	Pulp (7431)	99,900	6,25	9,476E+03	2,320	2,322	14,51
16.09.2020	11:17:13	Pulp (7431)	100,000	6,25	1,009E+04	2,471	2,471	15,45

					Average	2,395	14,97
Calibration number and standard name :	for N	PBS V 1 (L-Q-Q)	EDTA		Standard Deviation	0,084	0,53
					RSD [%]	3,516	3,52
Method :		C 2,2					
Sample Table :		Belastung ohne Stahl					

The method requirements of the two referring methods: AOAC 993.13 (1996), sr = 0,11-0,26 % N
DIN ISO 13878:1998, sr < 0,935 %. Both requirements were met with these conditions.

Remarks

It is important to take a representative sample portion of the well homogenized sample material.

Mix the sample before weighing thoroughly with a spatula to receive a representative sample portion for weighing.

It may can happen that the bottom connector tubing between left and right reactor gets dirty due to residues generated during combustion. This requires regular cleaning of the entire connector if residues are visible.

Alternatively the bottom connection with a larger diameter of 6 mm (14-0231 Upgrade Kit) should be used.