Dumas Application

A.3.3.1 Nitrogen Determination in Soil

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1 Principle

Nitrogen is used in agriculture as a fertilizer to achieve high yields of good quality, to supply sufficient nutrients and to maintain soil fertility. Too much nitrogen, on the other hand, inhibits root growth and can lead to soil acidification.

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.

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

- DIN EN 13654-2: 2001, Determination of nitrogen in soil improvement and culture media: Bodenverbesserungsmittel und Kultursubstrate - Bestimmung von Stickstoff - Teil 2: Verfahren nach Dumas.
- DIN ISO 13878:1998-11, Soil quality - Determination of total nitrogen content by dry combustion (elemental analyses).
- AOAC Official Method 993.13 Nitrogen (Total) in Fertilizers, Combustion Method, First Action 1993, Final Action 1996.

3 Gases and Consumables

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

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 DumaCollect, ash insert with bottom (Order number 14-0015)
3.6 DumaReact, prepacked combustion reactor filled with HT- and LT-catalyst (Order number 14-0244)
3.7 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)
3.8 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-Realyzer, chapter 10.4.
3.9 REAL-N Spiral adsorber for CO_2 (Order number 14-4085)
3.10 DumaEDTA, calibration standard, purity > 99 % (Order number 14-0032)
3.11 THAM, Tris(hydroxymethyl)aminomethane, purity > 99 %

4 Instruments

- Mortar grinder
- 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 soil sample should be representative and homogeneous. If the material still contains plant material and/or has a particle size higher than 1 mm, it is recommended to grind it further with a mortar grinder.

Soil sample preparation

Just before weighing, the sample is mixed thoroughly again by hand. Then, the DumaFoil (3.4) is tared and about 200 mg of sample are added. After having closed the tin foil, the weight is either noted or automatically transferred from the balance into the software REAL-OS. Last the sample is placed in the transfer tray.

Higher sample weights can be analyzed if required. In this case, the DumaFoil XL (3.4) is recommended.

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_2 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.

The presence of inorganic compounds in soil leads to a shorter lifetime of the crucible, depending on the sample weight chosen. Adjust the lifetime of the crucible in the REAL-OS (lifetime advised: 40 samples).

5.3 Combustion of the sample

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

Parameter	Setting
Combustion method	B 0.4 (with 0.4 ml O_2 / mg sample and a dosing speed of 300 ml/min)
Combustion temperature	With DumaReact (3.6): 980 °C
Reduction temperature	With DumaReact (3.6): 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, $H_2NC(CH_2OH)_3$) (3.11) in 100 ml of distilled water.

The minimum requirement for the correlation factor R2 is a value ≥ 0.999.

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

5.5 Example of results

The soil sample 8991 was analyzed using the combustion method B0,4 and a THAM 0,5%N calibration.

Sample Name	Weight [mg]	Nitrogen Peak Area [mV*s]	N Weight [mg]	Nitrogen [%]
8991	199,712	1,5E+03	0,385	0,193
8991	200,495	1,4E+03	0,375	0,187
8991	200,380	1,5E+03	0,391	0,195
8991	200,488	1,5E+03	0,385	0,192
8991	200,695	1,5E+03	0,383	0,191
8991	199,957	1,4E+03	0,373	0,187

Mean nitrogen content = 0,191%N
Standard deviation = 0,003%N

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- Crude fibre, ADF and NDF in feed
- Fat in food and feed
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- Trace metal in soil and sludge
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- Total nitrogen determination in water, soil and plants
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