

**NITROGEN IN SOIL**

1. Introduction

This application describes the determination of nitrogen in soil using the combustion method acc. to Dumas.

2. Principle

The sample is combusted at high temperature 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
- AOAC Official Method 993.13 Nitrogen (Total) in Fertilizers, Combustion Method, First Action 1993, Final Action 1996
- DIN EN 13654-2: 2001 regarding Determination of nitrogen in soil improvement and culture media: Bodenverbesserungsmittel und Kultursubstrate - Bestimmung von Stickstoff - Teil 2: Verfahren nach Dumas

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 5.0¹⁾
- Oxygen 5.0
- Nitrogen 2.6
- DumaFoil Conditioned tin foil, cat. no. 14-0017
- DumaEdta (Ethylenediaminetetraacetic acid) C₁₀H₁₆N₂O₈, standard for calibration, min. purity > 99 %)
- 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 %

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

Regular soil is analysed without being milled or prepared in any other way.

Should the size of the particles be larger than 1 mm, then they must be milled by using a mortar mill.

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.



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7.2. Sample specific parameters

Recommended parameters for the analysis:

Initial weight: 100 to 300 mg
Category: B 0,8
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 covers the signal amplitudes of the unknown sample completely. When working with the initial sample weight recommended, peaks of about 1000 - 10000 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 : 65535
Software Version: DUMATHERM MANAGER V4.12
Submitter :
Operator : Serviceman

Table with 9 columns: Date, Time, Sample name, Weight [mg], Protein factor, Nitrogen Peak Area [mV*s], N Weight [mg], Nitrogen [%], Protein [%]. Contains 6 rows of sample data.

Calibration number for N (L-L-L)
and standard name :
Method : B 0,8
Series Name :

Summary table with 3 columns: Metric, Value, Status. Rows: Average (0,162, not defined), Standard Deviation (0,001, not defined), RSD [%] (0,757, not defined).

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Temperatures: Combustion Reactor 980 °C, Reduction Reactor 649 °C, Degassing Oven 299 °C
Flow Rates: He I 194,0 sccm, He II 199,0 sccm, O2 299,0 sccm
Times: Sample Delay 7 s, Sample Stop 11 s, Run Time 300 s

9. Comments

Regular solid soil is analysed without being milled or prepared in any other way.

