

APPLICATION NOTE

E-CN-001-2021/A1

Total Carbon, Nitrogen and TOC Determination in reference soils and sediment samples

Reference:

ISO 10694: Soil quality - Determination of organic and total carbon after dry combustion (Elemental analysis);

ISO 13878: Soil quality - Determination of total nitrogen content by dry combustion (Elemental analysis)

EN13654-2: Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method

Gazzetta ufficiale Official Italian Method 248.1999

Tested with VELP Scientifica CN 802 Carbon Nitrogen Analyzer (Code F30800090)





TOTAL CARBON, NITROGEN AND TOC DETERMINATION IN REFERENCE SOILS AND SEDIMENT SAMPLES

Introduction

A carbon-to-nitrogen ratio (C:N ratio) is a ratio of the mass of carbon to the mass of nitrogen in a substance. For example, if we have a C:N ratio of 24:1, this means we have 24 units of carbon to 1 unit of nitrogen. The C:N ratio is important because it has a direct impact on residue decomposition and also nitrogen cycling in soils.

It can, amongst other things, be used as indicator for nitrogen limitation of plants and other organisms. As a rule of thumb, the higher the ratio, the longer it takes for the material to decompose. Likewise, the smaller the ratio is, the more rapidly the plant material will decompose. This also has a direct relationship with the amount of nitrogen that is tied up in the soil that will be available to the next growing plant.

The Total Carbon "TC" could be divided into Total Organic Carbon "TOC" and Total Inorganic Carbon "TIC", that are the concentrations of the total carbon in organic and inorganic compounds, respectively.

TOC determination is important because high levels in soils prevents the anaerobic digestion process and limit the subsoils nitrogen enrichment.

The performance of the VELP CN 802 was evaluated by participating in the **Proficiency Testing program** organized by **WEPAL** (Wageningen Evaluating Programs for Analytical Laboratories).

Samples of soil were analyzed using the CN 802 and the results obtained (as C %, N % and TOC %) were compared with the statistical range accepted by WEPAL.

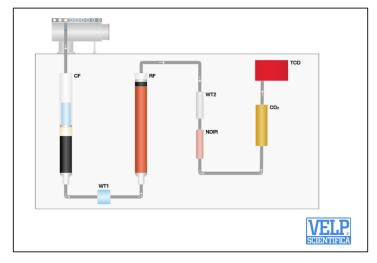
Carbon, nitrogen and Total Carbon determination in soils and sediments

The elemental analysis starts with a combustion (CF) to burn the sample, obtaining elemental compounds.

Water is removed by a first physical trap (WT1 - $DriStep^{TM}$), placed after the combustion, and a second chemical one (WT2). Between the two, the elemental substances passed through a reduction furnace (RF).

VELP exclusive **Non-Dispersive Infrared NDIR Detector** accurately measures the CO_2 concentration that the unit is able to convert in carbon quantity. Then, the autoregenerative CO_2 absorbers (CO_2) let pass only the elemental nitrogen that is detected by the innovative **LoGasTM** Thermal Conductivity Detector (TCD) with no requirement for a reference gas.

The CN 802 is controlled via PC through the intuitive CNSoft™.



CN 802 Preliminary Operations (daily)

Follow the operating manual to start the CN 802 and check that the following parameters are set:

Temperature Combustion reactor (Code A00000158): 1030 °C Temperature Reduction reactor (Code A00000226): 650 °C

Flow rate MFC1 He: 190 ml/min Flow rate MFC2 He: 220 ml/min

Condition the system by testing 2 EDTA standard (Code A00000149) and 3 to 5 empty tin foils (Code A00000153) as Check up. Verify the calibration curves with one or more tests as Standard by testing the same standard used for the curves creation.



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Sample Preparation

Reference soil and sediments samples:

Wepal ISE 2019-4 Sediment Wepal ISE 2019-4 Clay soil Wepal ISE 2019-4 Sandy soil Wepal ISE 2019-4 Forrest Sandy soil

Expected values on dry matter:

C%=6.981±0.245 N%=0.372±0.019 TOC%=5.882±0.495 C%=2.539±0.092 N%=0.142±0.011 TOC%=1.503±0.199 C%=1.724±0.105 N%=0.109±0.009 TOC%=1.667±0.153 C%=5.224±0.620 N%=0.168±0.023 TOC%=5.119±0.854

All samples have been dried at 105 °C before the analysis.

Two different aliquots of each samples have been analyzed:

- directly with CN 802 to determine the total carbon and nitrogen (%C and %N).
- after acidification with HCI, for determining the total organic carbon (TOC).

According to official references (i.e ISO 10694), TOC can be determined after removing carbonate minerals by acidification of the sample with HCl 2N, using the Silver foil (code A00000371).

Analysis Procedure

Fill the following fields in the database: Sample name, Weight, Method, Sample type, Calibration number

Create a new customizable method with the following parameters:

Protein factor: none O₂ flow rate: 300 ml/min O₂ factor: 1.6 ml/mg

Press (to start the analysis.

Analysis time: from 3 minutes for one run.

Typical C, N and TOC Results on sediment and soil samples

Carbon Calibration: the Carbon results have been obtained with the calibration curve using the standard Acetanilide (C% = 71), in a range of 0 - 35 mg C.

Nitrogen Calibration: the Nitrogen results have been obtained with the calibration curve using the standard aspartic acid $(C_4H_7NO_4)$, in the range of 0 - 1.5 mg N.

The calibration curve has been checked as per the method B in the Standard UNI 15936, with the Control Mixture B. The data obtained are included in the tolerance admitted by the certificate of analysis. The table below shows the %C, %N and TOC results, obtained by the CNSoft™.

Sample	C % (Average ± SD%)	%N (Average ± SD%)	%TOC (Average± SD%)
Wepal Sediment Riverclay	7.023 ± 0.041	0.370 ± 0.007	6.107 ± 0.002
Wepal Clay soil	2.577 ± 0.012	0.138 ± 0.002	1.556 ± 0.075
Wepal Sandy soil	1.762 ± 0.040	0.111 ± 0.003	1.705 ± 0.015
Wepal Forrest Sandy soil	5.193 ± 0.074	0.152 ± 0.002	5.006 ± 0.121

n = 10



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Conclusion

The participation of VELP at the **Proficiency Testing program** organized by **WEPAL** highlights the reliability of the results given by the **CN 802 Carbon Nitrogen Analyzer**.

The determination of percentage of carbon (%C), nitrogen (%N) and the Total Organic Carbon (TOC) in soil and sediment samples is one of the most common applications, where CN 802 can perform precise, fast and reliable results, as certified by **WEPAL**.

All the obtained data, in fact, are acceptable and comparable to the expected values, demonstrating excellent repeatability and accuracy of the CN 802 Analyzer.

The main advantages of VELP Combustion analyzer:



- Rapid analysis in 3-4 minutes per samples
- Precision and reproducibility guaranteed by the lowest LOD of 0.001 mgN with Helium and the RSD lower than 0.5% (EDTA)
- Low cost per analysis thanks to innovative technology and genuine consumables
- Fast set up of the instrument
- Easy maintenance
- Intuitive software User Interface
- Connectivity to VELP Ermes Cloud Platform for remote Application and Service support, remote access to the instruments, alerts and notifications and much more.