

Total Carbon and Total Organic Carbon determination in Sludge with CN802 Carbon Nitrogen analyzer

Reference:

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

UNI EN 15936:2012: Sludge, treated biowaste, soil and waste - Determination of total organic carbon (TOC) by dry combustion.

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



Introduction

Sewage sludge generated from wastewater treatment plants are being merited greater attention in light of their potential for improving soil properties and for providing important nutrient and trace element supplements that are essential for plant growth.

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 prevent 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 sewage sludge were analyzed using the CN 802 and the results obtained (as % Nitrogen) were compared with the statistical range accepted by WEPAL.

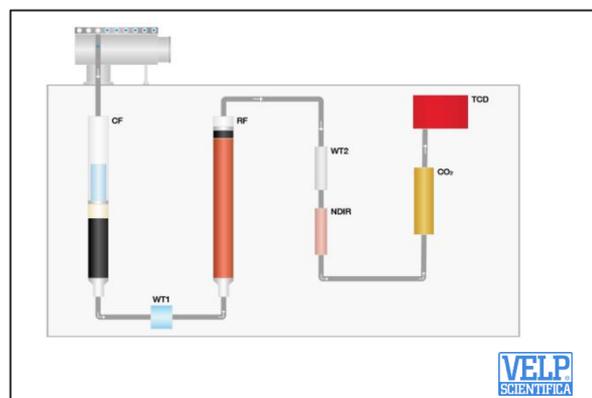
Organic and Total Carbon determination in sludge

The elemental analysis starts with a combustion furnace (CF) to burn the sample, obtaining a mixture of gas molecules.

Water is removed by a first physical trap (WT1 - **DriStep™**), 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 CO₂ concentration that the unit is able to convert in carbon quantity.

Then, the auto-regenerative CO₂ absorbers (CO₂) let pass only the elemental nitrogen that is detected by the **LoGas™** innovative 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 Argon: 190 ml/min

Flow rate MFC2 Argon: 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 curve creation.

Sample Preparation

Two reference sewage sludge samples and an unknown sludge sample have been analyzed.

Expected range on dry matter:

Wepal BIMEP 404 Sewage sludge sample	C % = 32.50 – 32.96
Wepal BIMEP 447 Sewage sludge sample	C % = 23.7 – 24.7
Unknown sludge sample	C % unknown

Use a sieve in order to remove any coarse parts; then, homogenize the sample and dry it in an oven at 105 °C.

Reference sludge samples are already homogeneous and they have been dried directly at 105 °C before the analysis.

Two different aliquots of each sludge samples have been analyzed:

- directly with CN 802 to determine the total carbon (TC)
- after acidification with HCl, for determining the total organic carbon (TOC).

Accordingly 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 tin 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 TC and TOC Results on Sludge sample

Results have been obtained with the following calibration curve: in a range of 0 – 70 mg C using the standard calcium carbonate (C% = 12) and acetanilide (C% = 71). The calibration curve has been checked as per the method B in the Standard UNI 15936, with the Mixture B. The data obtained are included in the tolerance admitted by the certificate of analysis. The table below shows the TC and TOC results, obtained by the CNSoft™.

Sludge Sample	Sample quantity (mg)	TC %	Sample quantity (mg)	TOC %
Wepal BIMEP 404	198.400	32.461	100.82	28.861
	200.500	32.345	100.78	28.258
	199.200	33.014	103.87	28.637
	Average ± SD%	32.607 ± 0.358	Average ± SD%	28.582 ± 0.305
Wepal BIMEP 447	99.780	24.080	101.37	21.485
	101.290	23.913	103.73	21.390
	99.590	23.924	99.26	21.361
	Average ± SD%	23.972 ± 0.093	Average ± SD%	21.412 ± 0.065
Unknown sludge	296.79	6.133	109.12	5.151
	298.21	6.129	105.44	4.385
	297.50	6.316	100.72	5.330
	Average ± SD%	6.193 ± 0.107	Average ± SD%	4.955 ± 0.502

Conclusion

VELP Scientifica CN 802 Carbon Nitrogen Analyzer is the ideal solution for the determination of total carbon (TC) and Total Organic Carbon (TOC) in sludge samples. The analyzer ensures reliable results in a fast easy and way with automatic calculation by the software CNSoft™.

All data obtained are acceptable and comparable to the expected values, demonstrating excellent repeatability and accuracy of the CN 802 Analyzer with no memory effect observed.

With high productivity and non-stop performances, CN 802 combustion apparatus is ideal for high throughput, both with Helium and Argon as carrier gas, being fully automated and requiring from 2-5 minutes per analysis.

Connecting the system to **VELP Ermes Cloud Platform** makes possible to easily monitor and control the analysis in real time via PC, smartphone or tablet.