Agri - Food

Chemicals

Geo - Environment

Energy

Application Note

CNS analysis in soil samples

Instrument: ECS 80 series
Mode: CNS or CHNS

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NC TECHNOLOGIES
Innovative Elemental Analysis

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Introduction

Soil plays an important role in various operational and management areas, with implications and interests that vary according to the different uses.
Depending on the areas of use, it is necessary for its proper management and to identify the appropriate quality indicators to be used in the evaluation and study phase.
Area of use:

- agricolture
- fruit growing and viticulture
- woody crops
- protected horticulture
- nursery
- ornamental green
- sporty green

Soil quality indicators

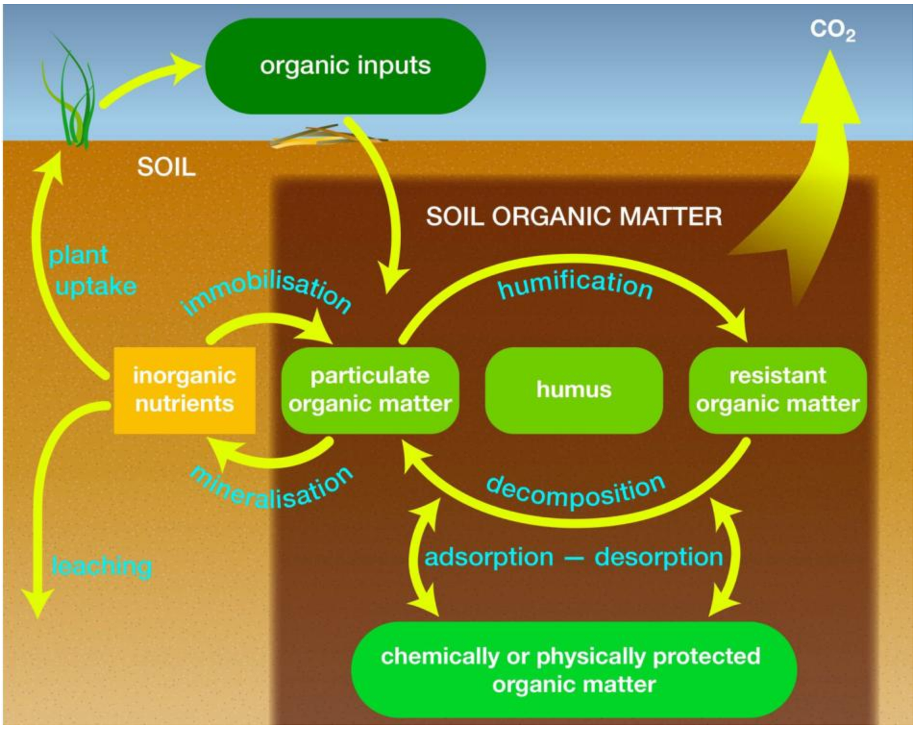
An indicator is a parameter, or a value derived from it, from which it is possible to obtain information about the state of a phenomenon.
There are several classes of parameters and indicators through which the characteristics of soils are evaluated depending on their use:

- physical characteristics
- chemical characteristics
- biological characteristics

Among the chemical characteristics there are some parameters, determinable through elemental analysis, which provide relevant indications.

Organic matter

Soil organic matter is a potential environmental indicator because it correlates with numerous aspects of the productivity and sustainability of agroecosystems and environmental conservation.
It is a reserve of nutrients and energy for microorganisms and plants and interferes with their availability in exchange mechanisms.



The term soil organic matter refers to all the organic matter in soil including living and non-living components.

The living components of soil organic matter includes active roots and living organisms, and non-living components include root exudates, decomposing plant and animal material, humus and charcoal.

Soil organic matter is largely made of carbon, oxygen, and hydrogen, but also contains many nutrients essential to the growth of plants such as nitrogen, phosphorus, sulphur, and other nutrients.

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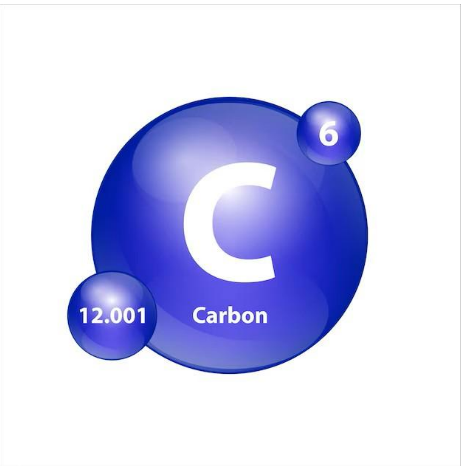
Assessing soil organic matter

Soil organic matter (SOM) is commonly but incorrectly used interchangeably with soil organic carbon (SOC).
Soil organic matter differs in that it includes other elements such as hydrogen, oxygen, phosphorous, sulphur and nitrogen whereas soil organic carbon is a measure of the organic carbon content of soils.

The importance of the elements



Soil organic matter is approximately 58% carbon.
Soil carbon analysis is important because soil carbon plays a critical role in soil health and fertility, and it is also a significant contributor to global carbon cycles. Understanding the amount and distribution of soil carbon can help us better understand the impacts of land use and management practices on soil health and can also help inform efforts to mitigate climate change by increasing carbon sequestration in soil.

Nitrogen is required for healthy plant growth and physiological development, and less than optimal amounts will constrain potential yield and protein quality. Oversupply can also adversely affect plant growth and yield. Farmers should aim to apply nitrogen to meet crop demand in order to reduce environmental losses and maximise fertiliser efficiency. Plants will use nitrogen to increase vegetative growth and therefore set up yield potential, this then increases their photosynthetic area in order to increase the amount of carbohydrate production and fill grain.

The C/N ratio in soil is the ratio of organic carbon to nitrogen and is a key indicator of soil health, microbial activity, and nutrient availability. A healthy, balanced ratio is ideal for microbial life and efficient decomposition, with optimal ranges often cited between ~12:1 and 24:1. A low ratio (less carbon, more nitrogen) can lead to quicker decomposition and nitrogen release, while a high ratio (more carbon, less nitrogen) can cause microbes to temporarily "tie up" available nitrogen from the soil to break down the carbon-rich material, potentially starving plants

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Soil mineralisation, recycling of crop residues and leaching are the critical soil processes determining the levels of plant available sulfur during the growing season.
Most sulfur exists in soil within soil organic matter. Sulfur supply relies on the concentration of organic matter and the rate of breakdown to maintain nutrient supply. Organic matter can mineralise organic sulfur as sulfate (SO42-) where the carbon to sulfur ratio is sufficiently narrow to result in net supply. Sulfate that is not absorbed by plants is subject to leaching so that in coarse textured soils under high rainfall, supplies of available sulfur are normally limited.

We can therefore see how the analysis of the elements carbon, nitrogen and sulphur are of significant importance in the study of soils.

International standard norms

There are several analytical techniques for determining carbon, nitrogen, and sulfur in soil samples.
In particular, some international standards that define some of these techniques can be considered:

- **ISO 15178** - Soil quality — Determination of total sulfur by dry combustion
- **ISO 13878** - Soil quality — Determination of total nitrogen content by dry combustion
- **ISO 10694** - Soil quality — Determination of organic and total carbon after dry combustion

International
Organization for
Standardization

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Elemental analysers

The “dry combustion” is defined, usually, as a device that burn the sample in oxygen, or in an oxygen/carrier gas mixture, under conditions such that it is converted into ash and gaseous products of combustion. These consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or oxides of nitrogen, oxides and oxyacids of sulfur and hydrogen halides. The products of combustion are treated to ensure that any hydrogen associated with sulfur or halides products of combustion are liberated as water vapour. Oxides of nitrogen are reduced to nitrogen, and those products of combustion which would interfere with the subsequent gas-analysis procedures are removed. The carbon dioxide, water vapour and nitrogen mass fractions of the gas stream are then determined quantitatively by appropriate instrumental gas analysis procedures.
The sulfur content of a soil sample, is determined by heating the sample to a temperature of at least in a stream of oxygen-containing gas. Organically and inorganically bound sulfur is converted to SO_2. This reaction may in some cases need a higher temperature or the addition of catalysts, modifiers or accelerators. At temperatures < 1350°C, SO_3 may be formed in the presence of excess oxygen. This SO_3 has to be reduced to SO_2 using an appropriate reagent, e. g. copper. The SO_2 arising from the combustion is measured by infrared spectrometry, thermal conductivity or other suitable detection technique.
This is the high-temperature catalytic combustion.

All our elemental analysers of the 80 series are compliance to the mentioned norms.
Depending on the chemical elements you are interested in, you can choose one of the analyser here below.

ECS 8020

ECS 8024 NC
SOIL SPECIAL

ECS 8040

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Analytical suggestions

Since the chemical characteristics of soils are not always the same, the concentration of elements can vary significantly from one type of sample to another.
Furthermore, the quantity of sample to be analyzed, to obtain good repeatability, depends on the homogeneity of the sample itself:

- ✓ if the sample is homogeneous, the amount of sample to be analyzed can be limited (**micro weight**)
- ✓ if the sample is inhomogeneous, the amount of sample to be analyzed must be increased to achieve good repeatability (**semi-macro weight**)

The elemental analysis golden rule

Even if an instrument can analyse up to 200 mg or more, the most important things is to weight the minimum amount of sample to get repeatability analysis.
In this case I get good results but saving consumption of helium, oxygen and chemical reagents.

How to prepare a sample

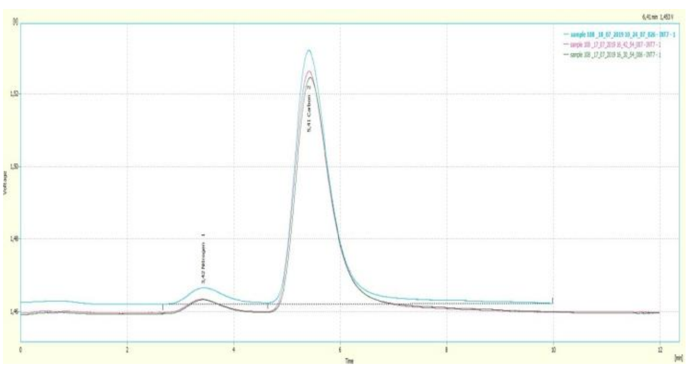
Especially in the case of inhomogeneous samples, the sample must be ground to obtain an adequate granulometry but above all to make it as homogeneous as possible:

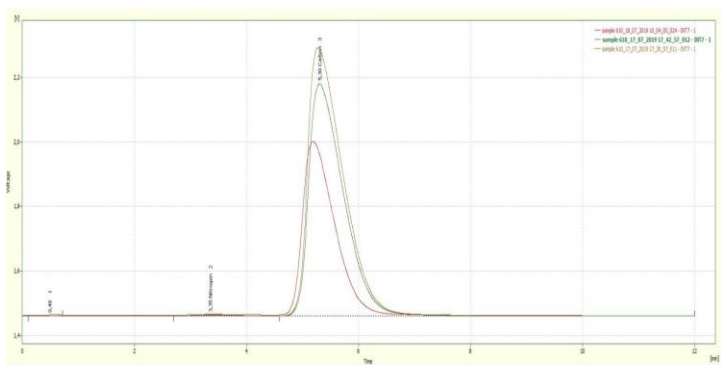
- ➢ preparation to obtain adequate granulometry (mills, …….)
- ➢ introduce sample into tin foil
- ➢ weigh the sample with adequate balance (5 or 6 digits)
- ➢ form a tablet
- ➢ introduce into the auto sampler

Some examples

Example n. 1

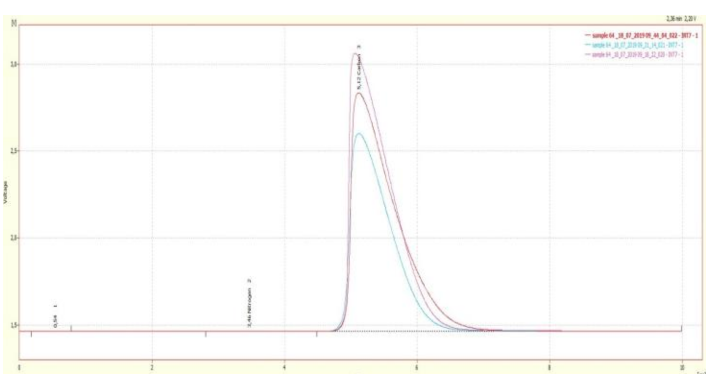
In these sample analyses of three different types of samples, we analyzed the concentrations of carbon and nitrogen.
You can see that all three sample types have a low nitrogen concentration and a variable carbon concentration.
The low nitrogen concentration (close to the lower detection limit of the instrument: a few micrograms), leads to a marked uncertainty in the relative standard deviation, although the analyses for each sample were repeated three times.





Sample ID	Weight (mg)	% N	% C
Sample 108	66,200	0,021	0,138
Sample 108	62,300	0,017	0,148
Sample 108	67,400	0,017	0,139
Average		0,018	0,142
Std Dev		0,002	0,006
RDS %		12,597%	3,888%

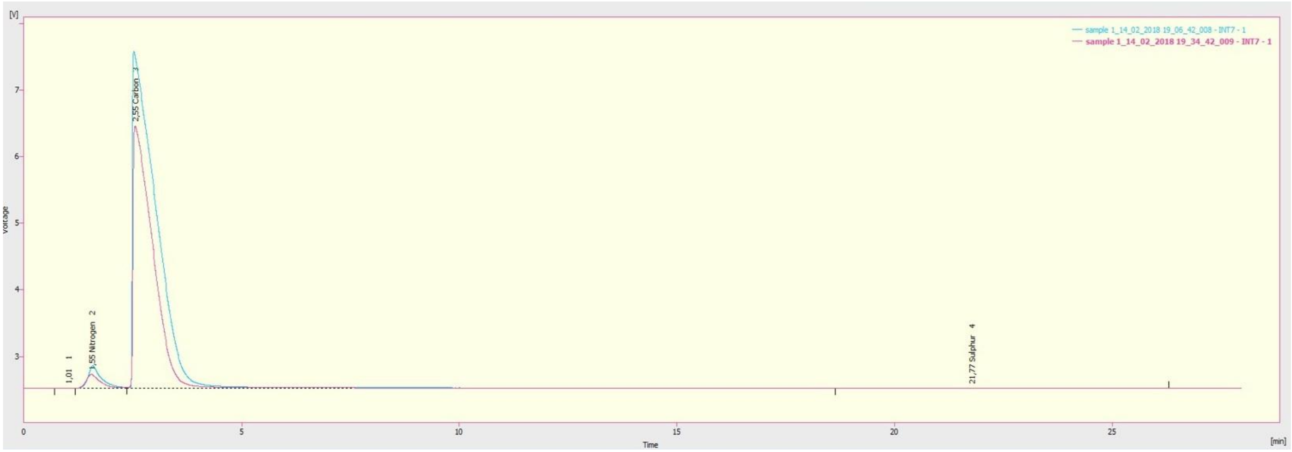
Sample ID	Weight (mg)	% N	% C
Sample 610	37,500	0,009	1,621
Sample 610	53,800	0,009	1,645
Sample 610	63,700	0,010	1,627
Average		0,009	1,631
Std Dev		0,001	0,012
RDS %		6,186%	0,766%



Sample ID	Weight (mg)	% N	% C
Sample 64	20,100	0,019	9,095
Sample 64	15,000	0,025	9,169
Sample 64	22,300	0,020	8,865
Average		0,021	9,043
Std Dev		0,003	0,159
RDS %		15,068%	1,753%

Example n. 2

In this example, we analyzed soil samples, determining the concentrations of nitrogen, carbon, and sulfur.
For both sample types, the concentrations of the three elements are quite low; for this reason, we weighed samples using a semi-macro analyzer.
As you can see, the closer we get to the instrument's detection limit (micrograms), the greater the measurement error.

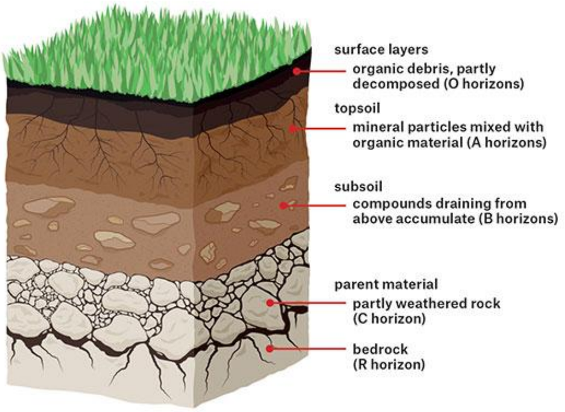


Sample ID	Weight (mg)	% N	% C	%S
Sample 1	247,269	0,150	1,690	0,029
Sample 1	159,035	0,147	1,686	0,027
Sample 1	250,539	0,150	1,722	0,031
Average		0,149	1,699	0,029
Std Dev		0,002	0,020	0,002
RDS %		1,162%	1,161%	6,897%

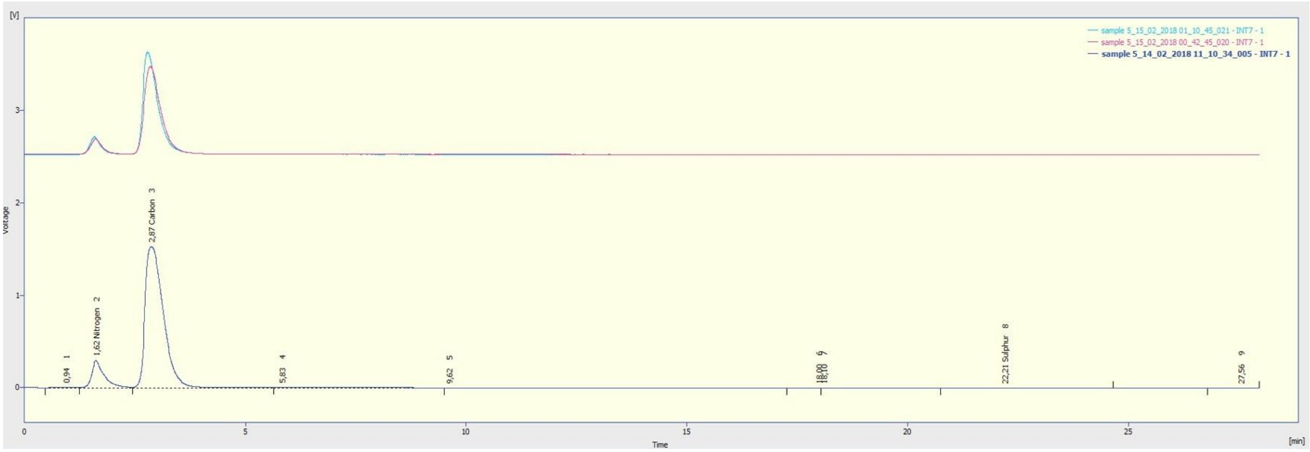
Theoretical values:

N: 1000-1500 ppm
C: 1,4-1,8 %
S: 200-400 ppm

The low carbon content (and that of other elements) might suggest that the samples were taken at depth. Indeed, considering the soil profile as a function of depth, organic matter decreases if the soil is deep.



Example n. 2



Sample ID	Weight (mg)	% N	% C	%S
Sample 5	277,224	0,065	0,209	
Sample 5	259,183	0,065	0,209	0,006
Sample 5	477,248	0,062	0,199	0,010
Average		0,064	0,206	0,006
Std Dev		0,002	0,006	0,003
RDS %		2,706%	2,807%	47,140%

Theoretical values:

N: 400-650 ppm
C: 0,16-0,24 %
S: < 100 ppm

Conclusion

Soil is of great importance for human needs but also for environmental health. Chemical elements contribute to understanding soil use, and therefore elemental analyzers can help characterize them.
NC Technologies can provide a full range of analyzers useful for studying this important resource.