



Carbon Analysis: Organic and Inorganic Carbon in Soils

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Keywords: TC, TOC, TIC, organic, inorganic, carbon, earth, sediments, environmental

1 Introduction

The HORIBA EMIA-V Series allows simultaneous measurements of Carbon and Sulfur in different types of samples, ferrous metals, non-ferrous metals, inorganic and organic substances, soil, cement, clay, lime, etc. For many environmental applications, it is necessary to know not only the Total Carbon (TC) concentration in the sample, but especially the Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC). The aim of this application note is to explain the determination of organic and inorganic carbon in soils and how to prepare samples to eliminate inorganic carbon (carbonates) in these soils.

2 Principles

2.1 Principle of the Carbon Analyzer, EMIA Series

The sample is introduced in a ceramic crucible and heated in a high frequency furnace to react with the oxygen stream. Most of the carbon (C) contained in the sample is transformed into carbon dioxide (CO_2) and remaining into carbon monoxide (CO). See Figure 1 below.

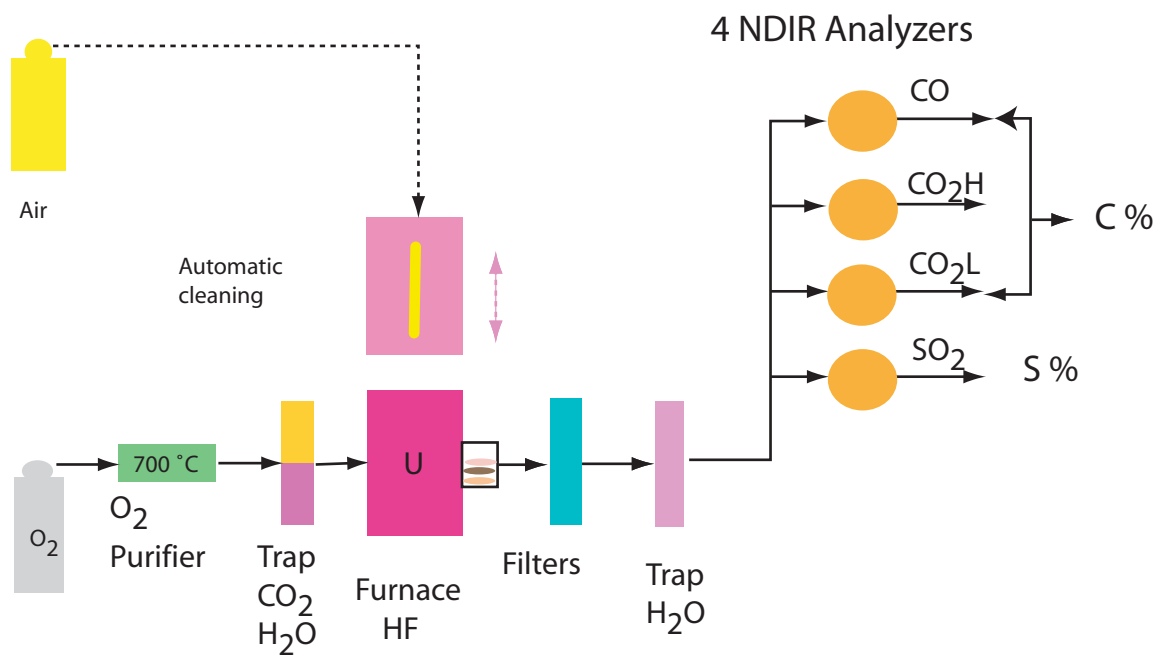


Figure 1: EMIA Series schematic



2.2 Principle of TOC, TIC, TC determination

In order to determine all specific concentrations for TOC, TIC and TC, the procedure for sample analysis is:

- TC: Direct analysis as it is in the crucible
- TOC: Analysis after complete removal of Inorganic Carbon with acid
- TIC: The TIC concentration will be determined by difference: $TIC = TC - TOC$

3 Sample preparation

Soil contains two different forms of carbon: Organic Carbon (TOC) and Inorganic Carbon (TIC). Before analyzing soil samples with the EMIA-V, sample preparation is used to eliminate Inorganic Carbon and measure the remaining carbon which is Organic Carbon. The sample preparation procedure is shown in Figure 2. Five separate samples should be prepared.

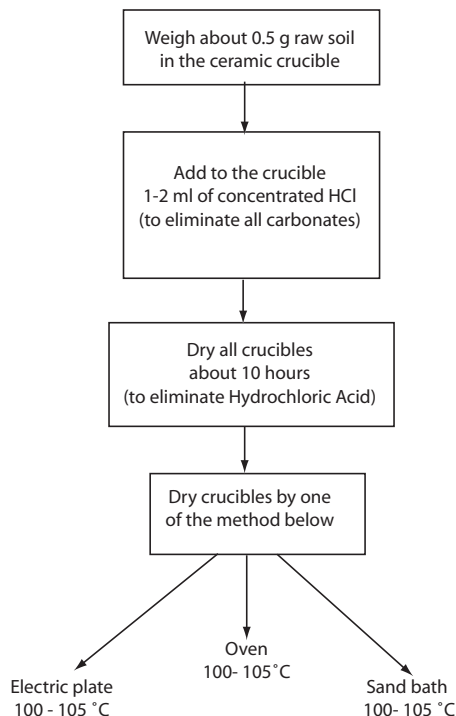


Figure 2: Sample preparation procedure

Five crucibles are prepared in the same way as the soil samples, i.e. only with hydrochloric acid. These will be used for the calibration under the same conditions as the sample to cancel any effect or interference from the acid.

It is very important to thoroughly dry all crucibles to avoid contamination from the hydrochloric acid. Hydrochloric acid can damage the infrared detector. To avoid detector contamination, it is recommended to use some nylon in the dust filter with the quartz wool and to use optional EMIA Halogen Trap. If the gas generated in the furnace contains hydrochloric acid, it will be trapped by the nylon.

4 Analytical conditions

To determine the Total Organic Carbon (TOC) in soil samples, an initial weight of about 0.5 g is used before adding hydrochloric acid. All crucibles are analyzed directly with the EMIA.

Table 1: Analytical conditions: standard conditions (see Figure 3)

Sample weight	About 0.5g (Weight before adding hydrochloric acid)
Blanks	Accelerators only 1g of Pure Iron, 2 g of Tungsten and 0.3g of Tin
Standard used for the Carbon calibration:	CaCO ₃ : 12% Carbon

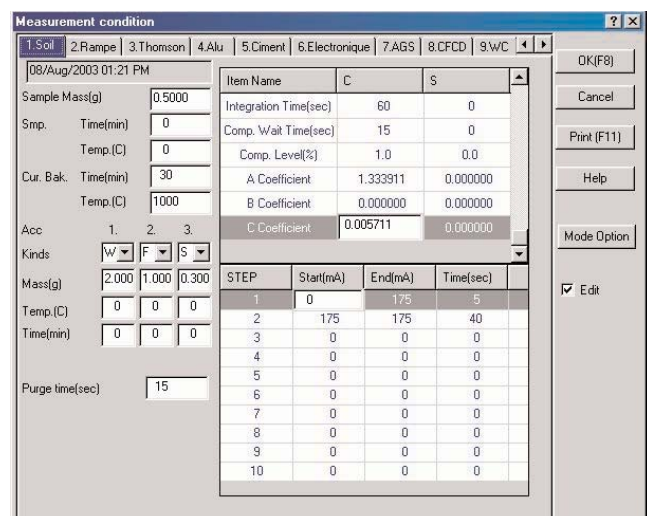


Figure 3: Analytical conditions screen



5 Typical results

5.1 Results from blanks

The blanks are prepared only with hydrochloric acid (for reference). This data will be used for blank shift in the calibration.

Table 2: Results for blanks

Blanks	Weight (g)	Mode	TOC (%)
	1.0000	1	0.0029
	1.0000	1	0.0023
	1.0000	1	0.0024
	1.0000	1	0.0025
	1.0000	1	0.0028
		Average	0.0026
		SD	0.0003

5.2 Test Protocol: Measurement of two soil samples

Two samples were analyzed, one with about 1.5% and the other with about 8% of Total Carbon (TC). The ratio TC/TIC is different for these two samples. Three replicates of both samples are analyzed directly to measure the Total Carbon concentration. Five replicates of both samples are prepared with hydrochloric acid to eliminate Total Inorganic Carbon (TIC). After drying, all crucibles are directly analyzed with the EMIA to determine the Total Organic Carbon (TOC). It is now easy to determine Total Inorganic Carbon concentrations (TIC = TC - TOC). Also, the ratio TC/TIC can be calculated.

5.2.1- Results for Sample 1

Table 3: Determination of Total Carbon concentration in Sample 1

Sample 1	Weight (g)	Mode	TC (%)
	0.5036	1	1.5044
	0.4996	1	1.4467
	0.4932	1	1.4611
		Average	1.4704
		SD	0.0300
		RSD	2.0420

Table 4: Determination of Total Organic Carbon in Sample 1

Sample 1	Weight (g)	Mode	TOC (%)
	0.5000	1	0.8448
	0.5077	1	0.8251
	0.5007	1	0.8171
	0.5007	1	0.8480
	0.4994	1	0.8238
		Average	0.8318
		SD	0.0138
		RSD	1.65 %

Table 5: Determination of Total Inorganic Carbon and the ratio TC/TIC for Sample 1

Sample 1	TC%	TOC %	TIC %	TC/TIC
	1.4707	0.8448	0.6259	2.35
	1.4707	0.8251	0.6456	2.28
	1.4707	0.8171	0.6536	2.25
	1.4707	0.8428	0.6227	2.36
	1.4707	0.8238	0.6469	2.27

5.2.2- Results for Sample 2

Table 6: Determination of Total Carbon concentration in Sample 2

Sample 2	Weight (g)	Mode	TC (%)
	0.5064	1	8.1596
	0.5091	1	8.1595
	0.5021	1	8.1491
		Average	8.1561
		SD	0.0060
		RSD	0.0740

**Table 7: Determination of Total Organic Carbon in Sample 2**

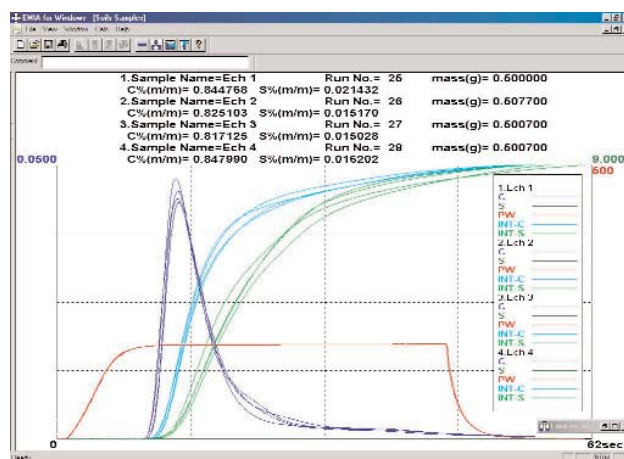
Sample 2	Weight (g)	Mode	TOC (%)
	0.5000	1	1.9820
	0.5000	1	1.9647
	0.4998	1	1.9853
	0.4996	1	2.0006
	0.4996	1	2.0077
		Average	1.9881
		SD	0.0168
		RSD	0.85 %

Table 8: Determination of Total Inorganic Carbon and the ratio TC/TIC for Sample 2

Sample 2	TC%	TOC %	TIC %	TC/TIC
	8.1561	1.9820	6.1741	1.32
	8.1561	1.9647	6.1914	1.32
	8.1561	1.9853	6.1708	1.32
	8.1561	2.0006	6.1555	1.33
	8.1561	2.0077	6.1484	1.33

6 Comparison of extraction curves

The overlay and comparison of Carbon extraction curves shows that the extraction and the combustion of samples shown in Figure 4 are correct and efficient.

**Figure 4: Carbon extraction curves**

7 Conclusion

The results are repeatable for all measurements. The combustion and the extraction are complete and efficient in all cases. It is possible to determine individually the concentration of Total Carbon, Total Inorganic Carbon and Total Organic Carbon in soil samples.

The sample preparation should be done very carefully, to avoid possible contamination of the detector from the hydrochloric acid. A halogen trap and a nylon reagent in the dust filter should be used to trap the contamination and halogen gas.



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