

C,S determination in soil

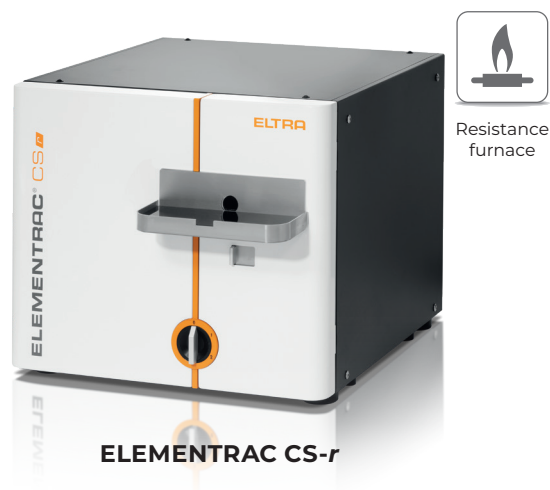


Suitable analyzers

- ELEMENTRAC CS-r

Used accessories

- Disposable procelain boat (90160)
- Suitable calibration material



Application Settings

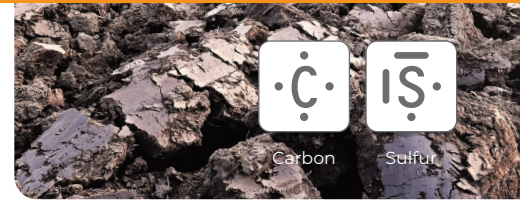
I) General

Temperature: 1350 °C (± 20 °C tolerance)
 Standby flow: Enable
 Stability: 0.03 V
 Minimum time: 40 sec
 Maximum time: 60 sec
 Flow: 180 l/h
 Analyzing: Voltage threshold

II) Analysis

Channel	Integration delay [sec]	Min time [sec]	Max time [sec]	Comparator level [mV]	Comparator peak [%]	Peak max [V]
Low C	0	75	300	20	0.1	8
Low S	0	75	300	20	0.1	8

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Introduction

With application of the recommended settings in the ELEMENTS software of the ELEMENTRAC CS-r total carbon and sulfur analysis of various soil samples is safe, reliable and compliant to international standards like ISO 15178 or DIN EN 15936. The reported deviation is valid for the analysed certified reference material but may not be representative for customer samples. The last chapter of this application notes provides additional information to improve repeatability and correctness of carbon and sulfur measurements which are processed with the ELEMENTRAC CS-r.

Sample preparation

Dry the sample to constant mass at 105°C (at least 1 hour).

Procedure

- Prepare and clean ELTRA analyzer (e.g. exchange anhydron, filter, boat stop); and set the furnace temperature to 1350 °C
- Run at least three warm up samples (e.g. ELTRA 92511-3020) with a medium sample weight of 200 mg until the results are consistent
- Calibrate the system with a suitable calibration material (NIST or other):
 - (1) Weigh in 200 mg of sample in a porcelain boat (90160)
 - (2) Start analysis (F5 Button)
 - (3) Wait until baseline is stable
 - (4) Load the sample into the furnace and wait until the PC calculates results
 - (5) Remove combustion boat

Repeat steps (1) – (5) at least three times;
Mark the results and use the calibration function in the software.

➔ Now start the actual analysis.



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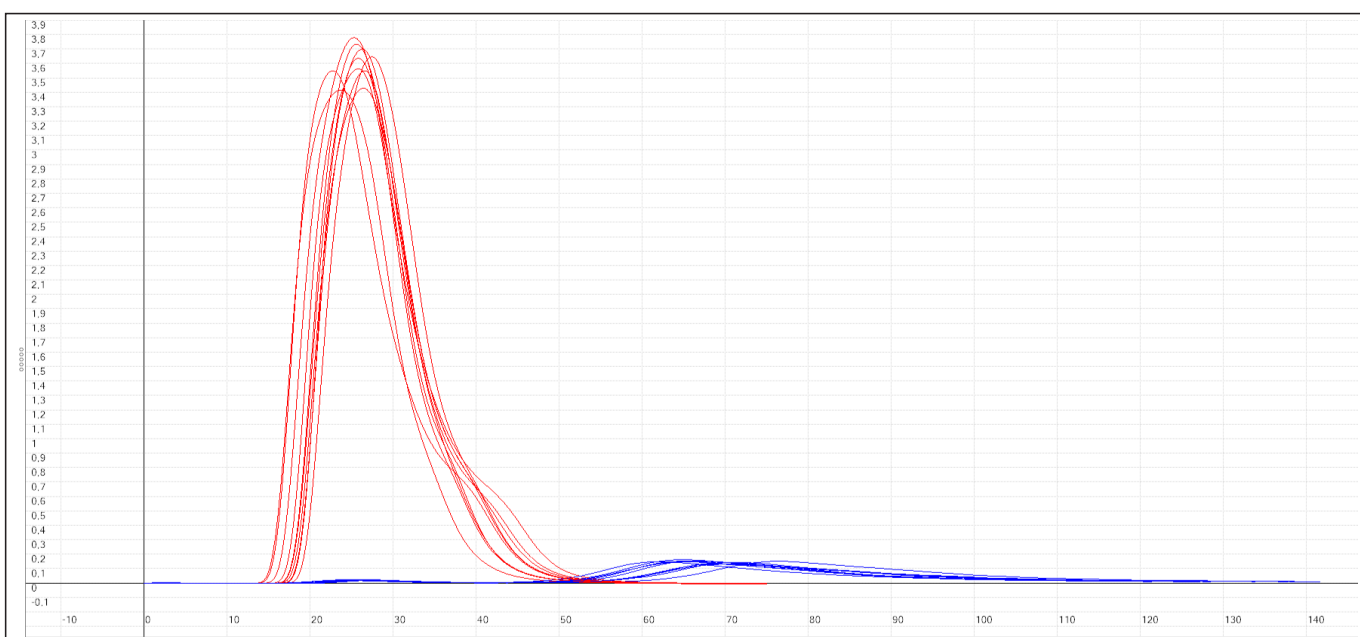


Typical results

Soil B2184 (Elemental Micro analysis) *1		
Weight (mg)	Carbon (%)	Sulfur (%)
209.5	2.29	0.031
228.1	2.28	0.031
226.9	2.27	0.031
208.3	2.29	0.031
203.5	2.28	0.031
212.7	2.29	0.031
201.9	2.30	0.032
213.2	2.28	0.030
201.3	2.31	0.030
203.0	2.27	0.032
Average values		
	2.29	0.031
Deviation / Relative deviation (%)		
	0.01 / 0.5%	0.001 / 1.7%
*1 = certified values: C: 2.29 ± 0.07 % S: 0.031 % (informative)		



Resistance furnace



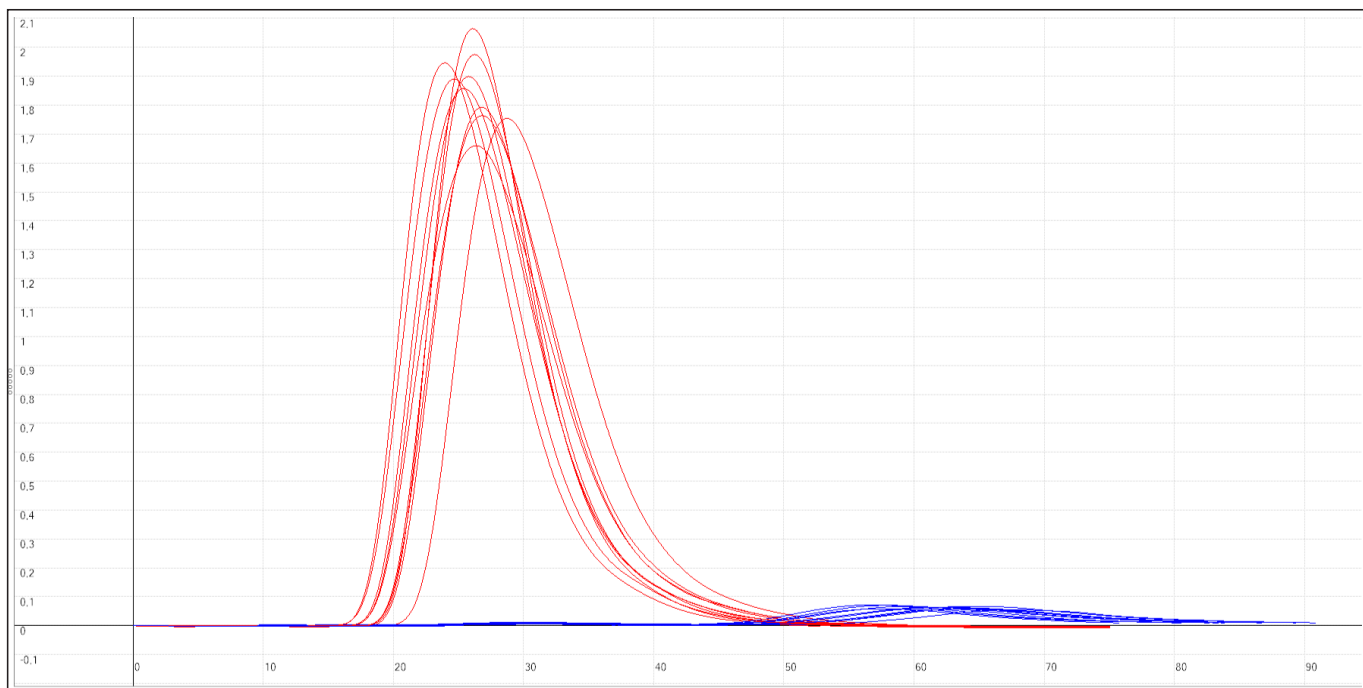
Subject to technical modification and errors

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Typical results

Soil B2180 (Elemental Micro analysis; BN291717) *2		
Weight (mg)	Carbon (%)	Sulfur (%)
198.8	0.832	0.014
199.0	0.830	0.011
198.6	0.823	0.013
201.3	0.825	0.014
204.5	0.832	0.014
200.8	0.839	0.018
201.5	0.831	0.015
203.2	0.822	0.015
207.3	0.829	0.014
202.6	0.837	0.014
Average values		
	0.830	0.014
Deviation / Relative deviation (%)		
	0.006 / 0.7%	0.002 / 11.8%
*2 = certified values: C: 0.83 ± 0.05 % S: 0.014 % (informative)		



Subject to technical modification and errors

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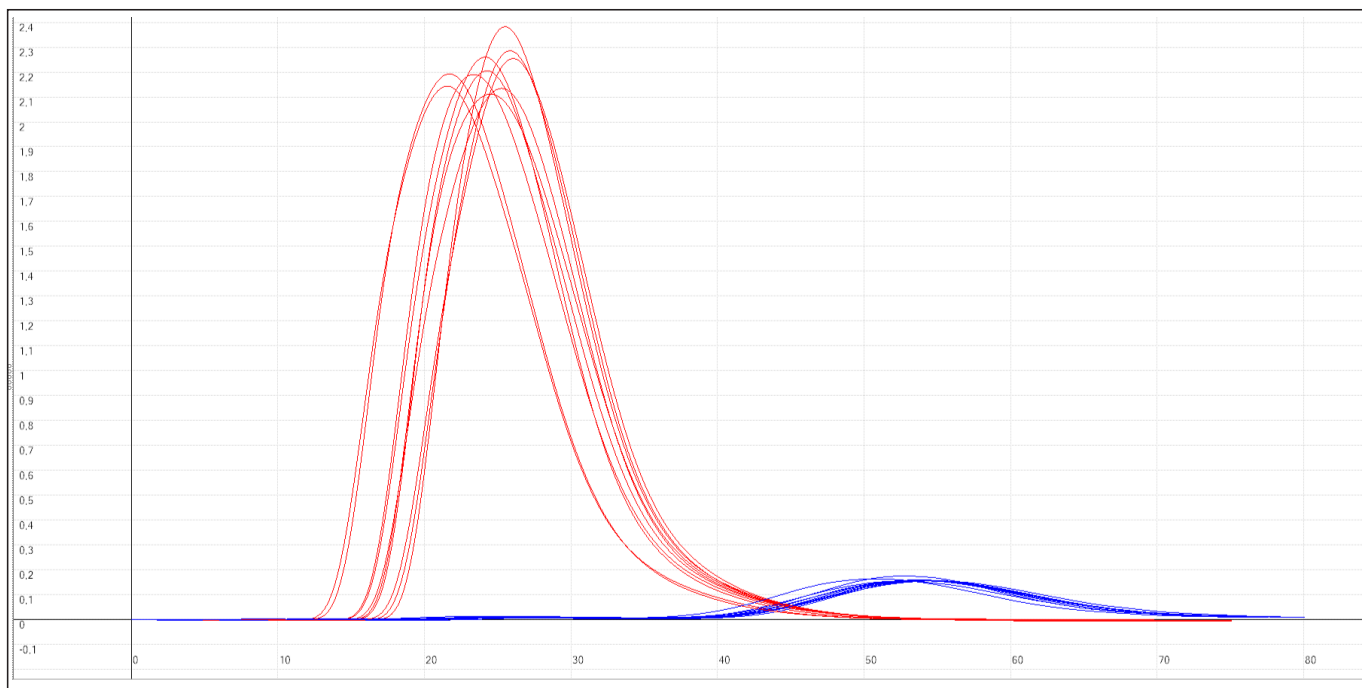


Typical results

Soil NCS ZC 73002 *3		
Weight (mg)	Carbon (%)	Sulfur (%)
200.2	1.06	0.021
201.5	1.08	0.021
199.9	1.06	0.020
199.4	1.06	0.022
201.2	1.07	0.021
203.8	1.06	0.021
199.6	1.06	0.023
198.8	1.06	0.022
201.6	1.07	0.023
199.6	1.08	0.023
Average values		
	1.07	0.022
Deviation / Relative deviation (%)		
	0.007 / 0.7%	0.001 / 5.2%
*3 = certified values: C: 1.07 ± 0.06 % S: 0.0217 ± 0.0023 %		



Resistance furnace



Subject to technical modification and errors

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The ELEMENTRAC CS-r: Application recommendations

The following chapter gives recommendation for the application of different sample carrier (boats) and describes typical errors and solutions of carbon and sulfur analysis.

Usage of boats

ELTRA offers different kinds of sample carrier (boats) for the ELEMENTRAC CS-r:

Article number	Description	No. of boats in packing unit	Recommended applications
90160	Small ceramic, disposable boats	1000	Fuels, cement, ore, soil, TOC (Tmax 1450 °C)
90153	Wide ceramic, re-usable boats	500	Fuels, soil, plastics
88600-0011	Small ceramic, re-usable boats	500	Fuels, soil, ore, cement, plastics
88400-0502	Re-usable Inconel boat: 13.5 mm height	1	Soil, synthetic products (Tmax 1350 °C)
88400-0503	Re-usable Inconel boat: 9 mm height	1	Soil, synthetic products (Tmax 1350 °C)

The disposable boats (90160) are suitable for all applications and can be disposed after one usage. The re-usable wide ceramic boats (90153) can be used several times (10 times in maximum), but have to be cleaned after usage. Depending on the application re-usable boats could be contaminated with carbon and sulfur during their first use and could show a higher carbon and sulfur blank value in subsequent measurements. Typical critical applications which can contaminate the boat are the analysis of heavy crude oil, bitumen and coal tar. For a reliable carbon and sulfur analysis of these samples the usage of disposable boats is recommended.

Application of the bigger 90153 boats in general show positive effects for the analysis of fast burning samples like oil and plastics and can improve the repeatability of carbon & sulfur measurements.

88600-0011 are small high quality re-usable boats and provide best possible repeatability for carbon, sulfur and hydrogen measurements (ELEMENTRAC CHS-r series).

Inconel boats (88400-0502, 88400-0503) are intended for analysis of samples with a very low carbon content (soil, synthetic products) below 1%.

Minor determination of sulfur

A minor determination of sulfur can have several reasons. Please make sure that no glass wool is applied on top of the anhydron (magnesium perchlorate). The glass wool can absorb moisture which is released from the sample during combustion. The condensed moisture is an ideal trap for the sulfur dioxide which is also released during combustion. Water saturated anhydron has to be replaced to avoid absorption of sulfur dioxide.

A minor sulfur determination can also be caused by an insufficient analysis temperature. Sulfur determination in general requires a minimum furnace temperature of 1350 °C. Some samples (ores, building materials, sulfates) would profit from a higher analysis temperature of 1450 °C and the application of combsolid (88600-0008) which

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provides additional oxygen during the combustion process. Depending on the chemical composition of the sample a larger analysis time may be required for a correct sulfur measurement.

Irritating results

Irritating results in general can be caused by handling errors during the analysis process, sample heterogeneity and sample preparation, the maintenance status of the analyzer and not suitable software settings. More detailed explanations can be found in the following:

a) Handling of samples

Analysis step	Recommendation	Comment
Applying sample on sample carrier	Spread sample over the whole sample carrier and use comparable sample weights (e.g. 350 mg ± 20 mg)	Different distributed samples on a boat and a big variation of the applied sample weight cause different combustion behaviour and a bad repeatability.
Applying high volumes and sample weights	Try to reduce sample volume and weight to a reasonable amount and assure a slow and careful sample introduction into the furnace.	When high sample amounts and volumes are applied the risk of sample loss increases. Sample can be blown on the ceramic tube of the furnace during combustion or the sample introduction process. This can cause irritating results and damage of the combustion tube.
Sample introduction into the hot furnace	Assure a smooth and constant sample introduction.	A smooth and constant sample introduction within 4-6 seconds assures a constant and reliable combustion. Make sure that only one combustion boat is in the furnace.

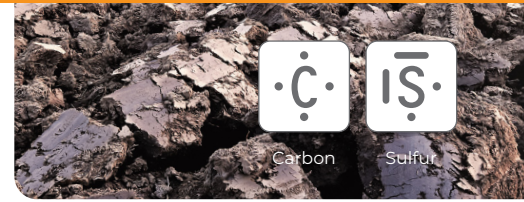
b) Sample heterogeneity and sample preparation

Sample heterogeneity and sample preparation cannot be influenced by the ELEMENTRAC CS-r. When heterogenous samples have to be analyzed the application of higher sample weights may be suitable to improve the repeatability of carbon and sulfur measurements. However, the maximum applicable sample volume and the working range of the analyzer have to be taken into account when the sample weight is increased.

A homogeneous size distribution of the sample can improve repeatability and correctness of carbon and sulfur measurements. The following exemplary results (soil) illustrate the big impact of a reliable sample preparation. A soil sample was analyzed four times with a typical sample weight of 200 mg but alternative sample size distributions:

Soil < 2 mm		Soil < 1 mm		Soil < 0.25 mm	
% Carbon	% Sulfur	% Carbon	% Sulfur	% Carbon	% Sulfur
4.23 ± 0.75 (17.8%)	0.033 ± 0.007 (20.1%)	4.10 ± 0.07 (1.6%)	0.033 ± 0.001 (3.2%)	4.20 ± 0.05 (1.1%)	0.037 ± 0.001 (2.4%)

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For professional consultation regarding sample preparation contact your local RETSCH agent (www.retsch.com).

c) Maintenance status of the analyzer

As mentioned in the previous chapter (minor determination of sulfur) worn Anhydron can falsify the sulfur measurement. When a single replacement of Anhydron was not suitable to improve the correctness and repeatability of results ELTRA recommends to inspect the following analyzer parts (see operation manual):

- Particle filter
- Boat stop
- Combustion tube
- Gas tubes inside the ELEMENTRAC CS-r

d) Software settings

Irritating results can be caused by wrong application settings in combination with the combustion behaviour of the sample.

Sometimes the carbon and sulfur concentration is measured in different channels (low/high carbon or sulfur). Due to slight variation in sample weight, sample distribution, sample introduction speed the low channel of one element can be saturated and the carbon or sulfur result is calculated in the high channel accordingly. Due to different calibration factors the calculated results may be different.

When samples have been already processed evaluate only the results of the high channel and look at the corresponding column in the ELEMENTS software. Otherwise switch off the low channel in this application or reduce the sample weight.

Sometimes the applied minimum analysis time is not sufficient for a complete and reliable integration of the complete peak. When minor determination of carbon and sulfur occur an adjustment of the minimum analysis time may be suitable. The following screenshot may illustrate the challenge. The measurement seems to be completed after 45 seconds, but later on the sample releases a significant amount of sulfur (blue peak).

