

C,S determination in low carbon steel (with gas purification furnace)



Suitable analyzers

- ELEMENTRAC CS-i

Used accessories

- Ceramic crucibles (90149)
- Tungsten accelerator (90220)
- Suitable calibration material (NIST or other)
- Carrier gas purification furnace



Induction furnace

ELEMENTRAC CS-i

Application Settings

I) General

Sample type:	Advanced
Standby flow:	180 l/h
Lance Purging:	On
Purging while closing:	Yes
Open Furnace:	Yes
Furnace purge through:	Exhaust
Furnace purge time:	3 sec
Furnace purge flow:	180 l/h

Stabilizing

Lance valve:	On
Stabilize by time:	Off
Stability:	0.001
Minimum time:	30 sec
Maximum time:	60 sec

II) Analysis

Voltage:	100 %
Power duration:	180 sec
Flow:	180 l/h
Chamber only:	0 sec
Lance and chamber:	0 sec
Drift compensation:	on

Channel	Max time [sec]	Min time [sec]	Integration delay [sec]	Comparator factor [%]
Low C	90	25	6	0.1
Low S	90	25	6	0.1

III) Postwaiting

Postwaiting time:	10 sec
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Introduction

For standard sample analysis with 500 mg sample weight and a carbon content of 200 ppm or higher ELTRA recommends the standard carrier gas oxygen with a purity of 99.5% and the usage of foil wrapped crucibles (90149). These conditions allow a reliable measurement of the carbon and sulfur content.

For a safe measurement of lower carbon concentrations with a sample weight of 500 mg ELTRA recommends the usage of a carrier gas purification furnace (88200-9000) and the application of preheated crucibles which have been heated to 1000 °C in minimum for 1 hour and subsequently stored in a desiccator. The aforementioned provisions reduce the carbon blank values of the crucibles and the carrier gas significantly which prevents unwanted fluctuations in the carbon signal. The blank value of the carrier gas is mainly caused by gaseous hydrocarbons whereas the blank value of the crucibles is caused by different substances. These impurities of gaseous CO₂, hydrocarbons or carbonates increase the measured carbon content. This application note provides typical analysis data of measurements with and without carrier gas purification furnace to illustrate the resulting consequences for the calibration and measurement process. All measurements have been processed with preheated crucibles.

Sample preparation

The analysed certified reference materials have been analysed without any sample preparation. Customer samples may have a contaminated surface due to the previous drilling or sectioning process. In this case treatment with acetone may be useful to remove contaminations. Make sure that all acetone is evaporated before measurement.

Procedure

- Prepare ELTRA analyzer (e.g. exchange anhydron, sodium hydroxide, platin catalyst if necessary); clean the combustion tube, brush, heat shield, dust trap
- Run three warm up samples (e.g. steel samples (92400-3050) with a minimum weight of 500 mg; add 1.7 g tungsten)
- Calibrate the analyzer with suitable calibration material (NIST or other)
The procedure of analysis of pure iron should be like this:
 - (1) Weigh in approx. 500 mg into the crucible
 - (2) Add 1.7 g of tungsten accelerator (90220)
 - (3) Place the crucible on the pedestal and start analysis

Repeat steps (1) – (3) at least three times;
Mark the results and use the calibration function in the software.

➔ Now start the actual analysis.



Induction
furnace

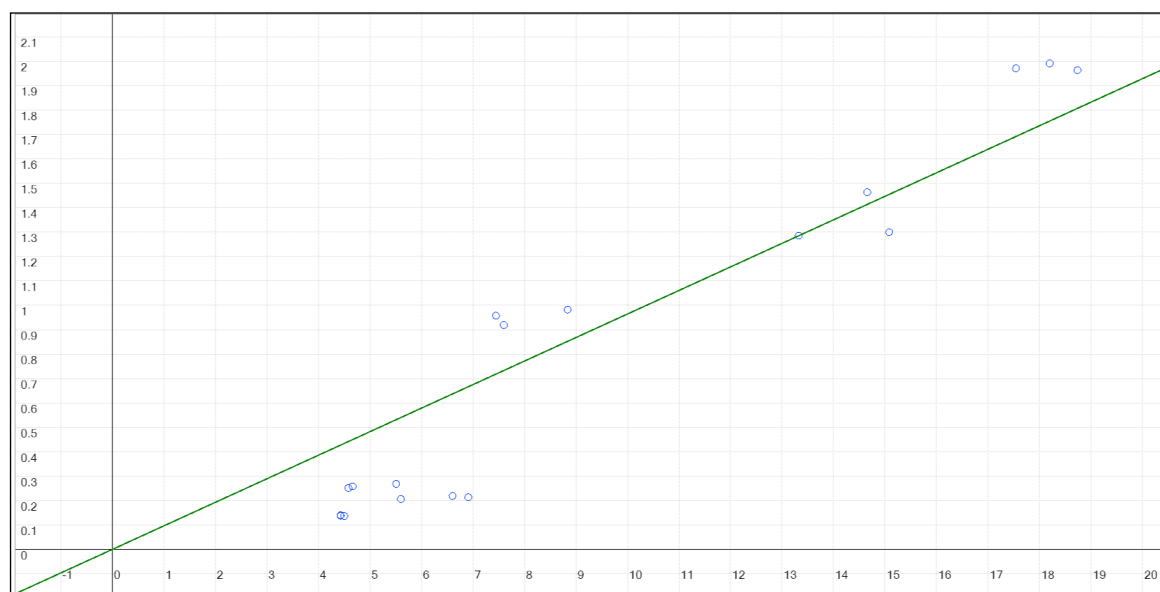
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Typical carbon results with preheated crucibles and no utilization of a carrier gas purification furnace

CRM Name	Certified carbon content (ppm)	Measured carbon content (N=3) ppm
502-401	2.6 ± 0.5	8.1 ± 0.2
JSS 003-8	4 ± 0.6	11.5 ± 1
502-704	5 ± 1	9.3 ± 0.6
ECRM 285-2	18 ± 2	14.5 ± 1
ECRM 284-3	25 ± 3	26 ± 1.9
502-402	39.1 ± 0.9	34.6 ± 1.6

The data have been obtained by the application of a linear regression with forced zero crossing in the ELEMENTS software. Especially the samples with a very low carbon content below 10 ppm show significant higher results than certified. In sum the linearity of the calibration curve seems to be not reliable.



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Typical carbon results with preheated crucibles and connected carrier gas purification furnace

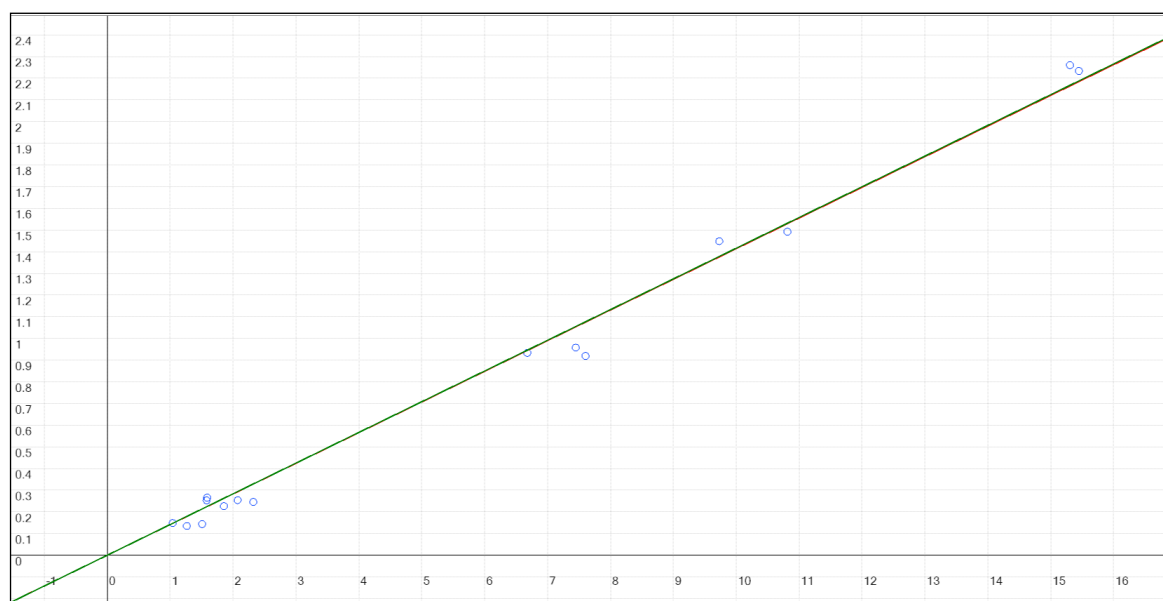
In the following the measurements have been repeated with utilization of a carrier gas purification furnace. The inlet of the furnace was connected with the general oxygen supply and the furnace outlet was connected to the gas inlet of the ELEMENTRAC CS-i.

The carrier gas purification furnace oxidizes hydrocarbons which may be present in the oxygen to CO₂ and water which subsequently are removed by the gas purification tube of the CS-i.

Without gas purification furnace the gaseous hydrocarbons are oxidized to CO₂ at the surface of the hot crucible and falsify the released CO₂ content of the sample. When samples with medium or high carbon content are combusted the additional blank values of the carrier gas is negligible. But for low concentrations the purity of the carrier gas is relevant. The following data show a significantly improved correctness of results.

CRM Name	Certified carbon content (ppm)	Measured carbon content (N=3) ppm
502-401	2.6 ± 0.5	3.2 ± 0.65
JSS 003-8	4 ± 0.6	4.9 ± 0.4
502-704	5 ± 1	4.4 ± 0.2
ECRM 285-2	18 ± 2	19.5 ± 1.5
ECRM 284-3	25 ± 3	24.7 ± 1
502-402	39.1 ± 0.9	38 ± 0.5

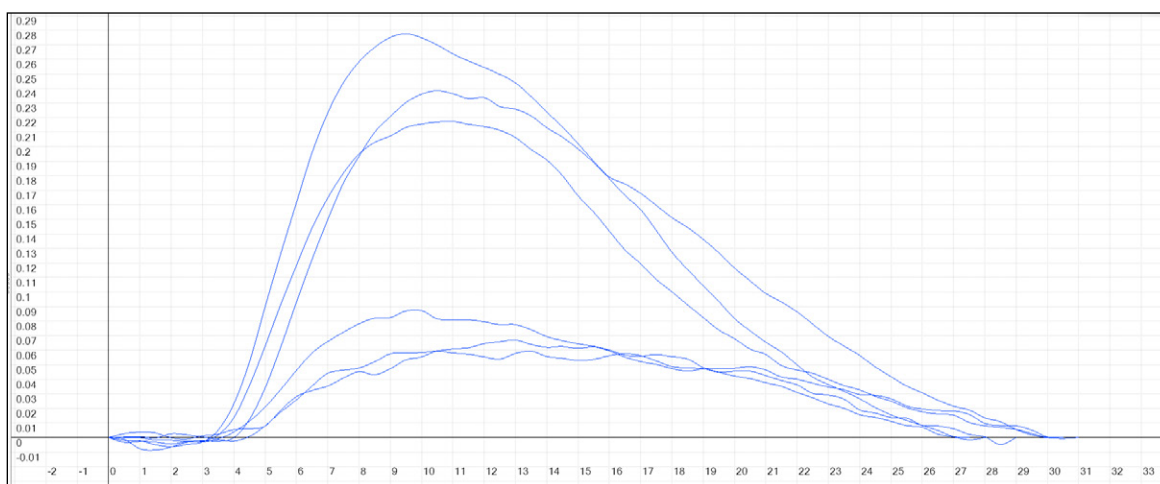
With utilization of a carrier gas purification furnace the linearity of the calibration curve was improved significantly. Esp. samples with a very low carbon content below 10 ppm do not show an offset.



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The following screenshot may illustrate the reason for the improved linearity of the carbon signal in the lower concentration range:



It shows the response of the low carbon channel of the JSS 003-8 sample. The higher intensities (0.22-0.28V) have been obtained without utilization of the carrier gas purification furnace, whereas the lower intensities (0.05-0.08V) have been measured with utilization of the carrier gas purification furnace.

In both cases a sample weight of approx. 500 mg has been combusted. The additional peak height is caused by contaminations in the carrier gas.

Sulfur measurements with carrier gas purification furnace

Due to the fact that oxygen does not contain any species which could cause a sulfur blank value the utilization of a carrier gas purification furnace does not affect sulfur measurements at all. The following samples have been measured three times with and three times without utilization of the carrier gas purification furnace.

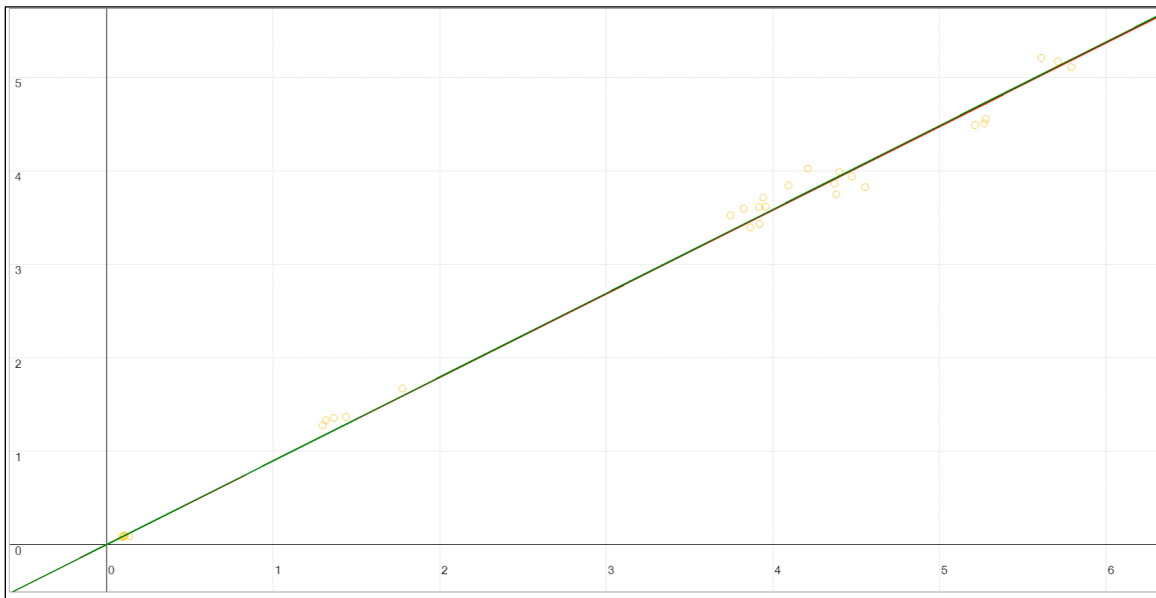
The resulting six measurements of every CRM have been used for a linear regression in the low sulfur channel of the ELEMENTS software. All certified reference materials have been measured within their certified values and deviations.

CRM Name	Certified sulfur content (ppm)	Measured Sulfur content (N=6) ppm
JSS 003-8	1.66 ± 0.5	1.6 ± 0.1
ECRM 285-2	25 ± 2	23.0 ± 0.8
ECRM 284-3	66 ± 3	67 ± 2.5
ECRM 088-2	70 ± 4	69 ± 2
502-402	89.4 ± 3.1	90 ± 3

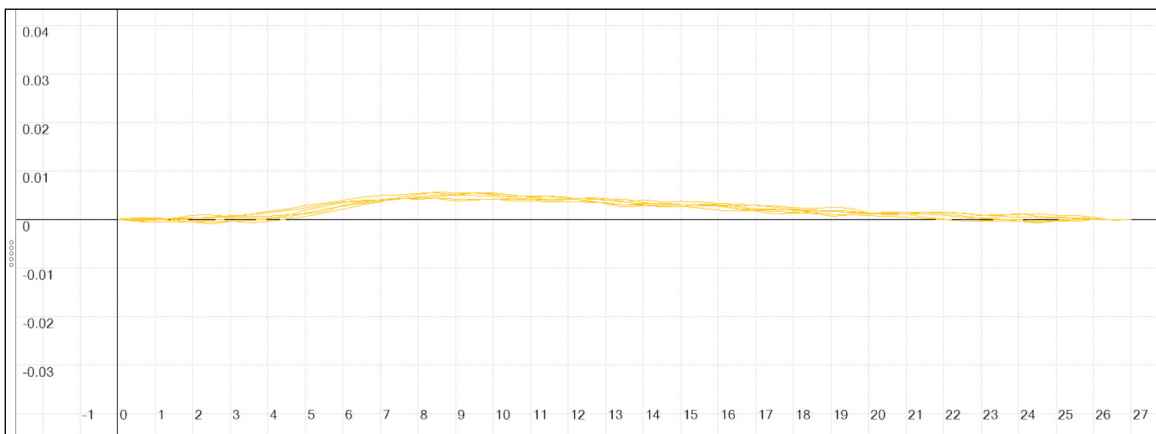
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The linear regression curve with forced zero crossing displays a good linearity over a wide concentration range and no general offset for lower sulfur concentrations.



The following screenshot illustrates that the peak height does not depend on the utilization of a carrier gas purification furnace. The screenshot shows the response of the low sulfur channel for the JSS 003-8 sample with a sample weight of approx. 500 mg. In contrast to the carbon measurements of JSS 003-8 the sulfur peak height of each three measurements with and without utilization of a carrier gas purification furnace is comparable.



Recommendation

When multiple samples below a carbon concentration of 100 ppm have to be analyzed the utilization of a carrier gas purification furnace is reasonable to improve the repeatability and correctness of carbon measurements. A reduced blank value assures a more reliable calibration and provides more safety when unknown sample concentrations are measured.