ELTRA®
a VERDER company

EXCELLENCE IN ELEMENTAL ANALYSIS

BASIC APPLICATION INFORMATION

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1 INTRODUCTION

This document provides basic application information for ELTRA elemental analyzers. The provided information is valid for the following ELTRA analyzer:

Type of analyzer	Current ELTRA ELEMENTRAC analyzer	Outdated ELTRA analyzer
ONH analyzer (Inert gas fusion)	ONH-p2	ELEMENTRAC ONH-p1 ONH 2000 ON 900 OH 900
CS analyzer (Induction)	CSi CSd (Induction part)	CS 800 CS 2000 (Induction part)
CS analyzer (Resistance)	CS-r/CHS-r CSd (Resistance part)	CS 580 CHS 580 CS 2000 (Resistance part)

Additional information about ELTRA analyzers is available in the following documents:

- Operation Manual
- Software Manual (ELEMENTS Software; UNI Software)
- Software Tutorial for selected functions (ELEMENTS Software)
- Application Notes (Various Analyzers)

For further information contact your local ELTRA agent or write an email: info@eltra.com

Disclaimer

This document was prepared according state of the art knowledge about the ELTRA analyzers and relevant standards (e.g. ASTM E 1019). ELTRA is not liable for typos in this document, changes in standards or damages in the analyzer due to wrong application of settings, consumables, CRMs or samples.

1.1 General recommendation

A reliable and precise determination of oxygen, nitrogen, hydrogen or carbon and sulfur does not only require correct settings in the analyzer or utilization of standard compliant consumables. In case of irritating results always the encompassing analysis process has to be considered.

Step	Possible error sources (example)
Sample taking	- Taking of wrong / not representative sample
Sample preparation	- Introduction of the element to be measured - Preparation of a not suitable sample shape, size, weight - Loss of the element to be analyzed due to application of too much heat
Storage	- Sample corrode, gets wet, alters, changes chemical composition
Preparation before analysis	- Maybe additional cleaning is required before analysis - Utilization of flux / accelerators
Analysis	- Correct application of power and channel settings
Report	- Report of the correct results

Errors in the beginning of the analytical process usually have a higher impact on the correctness and repeatability of results in comparison to errors at the end of the analytical process.

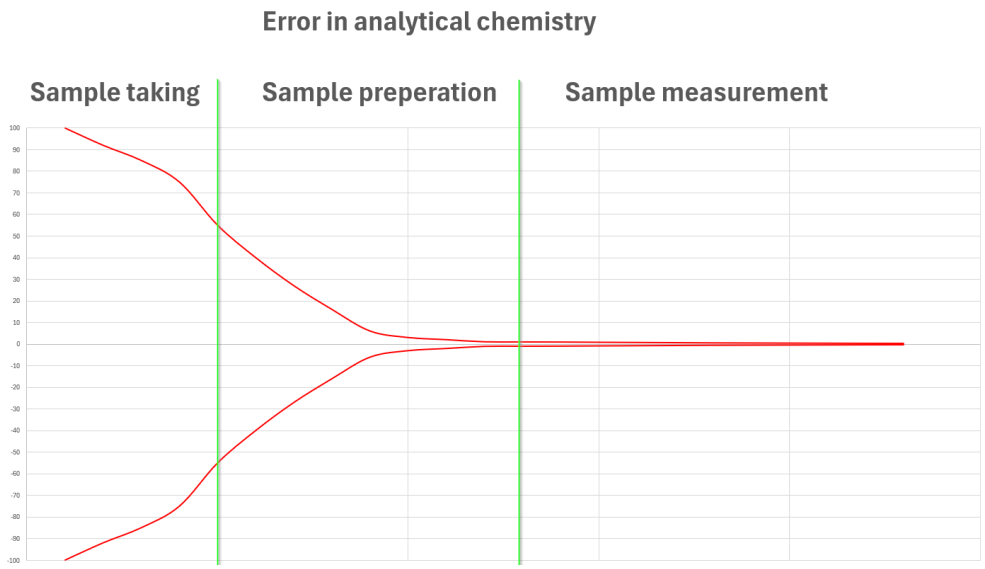


Illustration of errors in the analytical process

1.2 Before starting analysis

Before starting analysis evaluate the following details first:

Topic	Questions	Consider
Safety	Does the sample release substances during combustion / decomposition that can harm the user or analyzer?	Release of acids, halogens, metal vapors, etc.
Laboratory equipment	Can hazardous substances be weighed, applied and disposed safely? Are samples hygroscopic? Are suitable balances (e. g. for micro quantities) available?	Depending on the sample composition different accelerators, helping tools or a halogen trap has to be utilized. Are all required accessories in the laboratory?
Suitability of analysis method	Is the analytical technique used suitable for the analytical request?	See additional information at the analyzer chapters. E.g. an ELEMENTRAC ONH-p2 cannot process liquid samples.
Application - Hardware-	Before the analysis, it should be clarified whether the requested application can be performed (required tools for sample preparation, analysis and calibration)	Accelerators, auxiliary materials (graphite, sand), capsules, baskets are materials that may be required, as well as suitable calibration material.
Application -Software-	Define suitable settings and calibrate in the correct analysis range.	Analysis power, analysis time, calibration
Requirements of standards (See list in Annex)	Is a standard compliant analysis requested?	Please read the standard Intensively regarding - Sample taking - Sample preparation - Calibration - Measurement

1.3 Basic Application information for all analyzer

A reliable analysis of carbon, sulfur, oxygen, nitrogen and hydrogen requires a "fitting" combination of consumables and settings in the software. The following table summarizes basic details for combustion analyzer:

	C/(H)/S analysis via Resistance furnace	**C/S analysis via Induction furnace**	**O/N/H analysis via electrode / impulse furnace**
Suitable samples	Coal, coke, oil, ores, building materials, plants, soil	Steel, iron, alloys, ceramics, ores, building materials	Steel, iron, alloys, ceramics
Not suitable samples	Steel, metals	Oils, fuels, plants	Coal, coke, food, liquid samples
Typical sample weights	60 – 500 mg	60 – 1000 mg	10 – 2000 mg
	"Hardware requirements"		
Sample carrier	Ceramic boats - Single use (small) - Reusable (small) - Reusable (big)	Ceramic crucibles: - Preheated? - Foil wrapped? - Backed? - Application of lids?	Graphite crucibles - Single - Inner+Outer - Tin/Nickel capsules for analysis of powder
Accelerator / Flux	-Quartz sand -Combsolid	- Tungsten - Iron - Iron (high purity) - Tin - Copper - ELTRACell	- Nickel baskets - Tin pellets - Nibbled Nickel - Graphite
	"Software settings"		
Applied temperature	Can be set in degree C; Typically from 600 – 1550°C	Cannot be set directly; combustion of sample & accelerator provides temperatures > 2000°C	Application of electrical power. Typically 3000 – 6000 W; Resulting temperatures up to 3000°C
Channel settings	- Element or channel on/off - Integration delay - Min & Max Time - Comparator factors		
	"Calibration"		
Suitable calibration material	Coal, coke, soil, ores, limestone, $CaCO_3$, ZnS, $BaSO_4$	Steel, cast iron, limestone, soil, ores, $CaCO_3$, ZnS; $BaSO_4$	Steel, titanium, copper, ceramics

Note:

The decision regarding application settings (accelerator, sample carrier, applied temperature) is usually based on the chemical composition of the sample. This does mean that ELTRA application notes usually describe the settings for a typical “base” like steel, iron or limestone. Depending on the analysed samples in the laboratory adjustments in the recommended settings may be required. E.g. the application of higher sample weights in the ONH-p2 could require the application of more power to assure a reliable analysis. Higher power could also be required when a sample is higher alloyed (e.g. stainless steel with high chromium content). Depending on the local situation, expected concentration other adjustments (stabilizing time, used accelerator and amount) may be required.

Comments about the applied sample weight

ELTRA elemental analyzers combust or fuse the applied sample and measure the released gases. Due to this working principle the application of a suitable sample weight is a very important detail. Please check if a standard or the laboratory operation procedure defines the application of special sample weight. The following table illustrates the general tendencies for the application of “low” and “high” sample weights:

	“High” sample weights	**“Low” sample weights**
Application (example)	C/S analysis of coal in the ELEMENTRAC CS-r	
Applied sample weight	500 mg coal in CS-r	50 mg coal in CS-r
Advantages	(Heterogenous) samples could be analyzed with a good repeatability and a low limit of detection	- Fast analysis time - Low maintenance frequency
Disadvantages	- Longer analysis time - Maybe applied mass is not compliant with standard - High consumption of consumables - Saturation of channels - High maintenance frequency	- Bad repeatability - Weighing errors - Maybe applied mass is not standard compliant

The recommended sample weights in the different application note consider the standard compliance of an analysis as well as a good repeatability. In addition, the consumption of chemicals and the resulting maintenance frequency should be reasonable. Depending on the sample to be analyzed adjustments may be required in applied sample weight, settings.

2 INERT GAS FUSION ANALYZER FOR “O/N/H” ANALYSIS

ELTRA inert gas fusion analyzers are well suited for the reliable oxygen, nitrogen and hydrogen analysis in inorganic samples like metals, alloys and ceramics.

Topic	Description
Current analyzer	ELEMENTRAC ONH-p2
Outdated analyzer	ONH-p1 ONH 2000, ON 900, OH 900
Typical sample	Iron, steel, copper, titanium, ceramics
Not supported samples	Fuels (coal, coke, oil), food, pharmaceuticals, any liquid samples
Typical sample weights	10 – 2000 mg
Typical analysis time	25 – 90 seconds (only peaks)
Typical cycle time	160 – 300 sec (total analysis time)

ELEMENTRAC ONH-p2 with optional autoloader and cleaner

General usage:

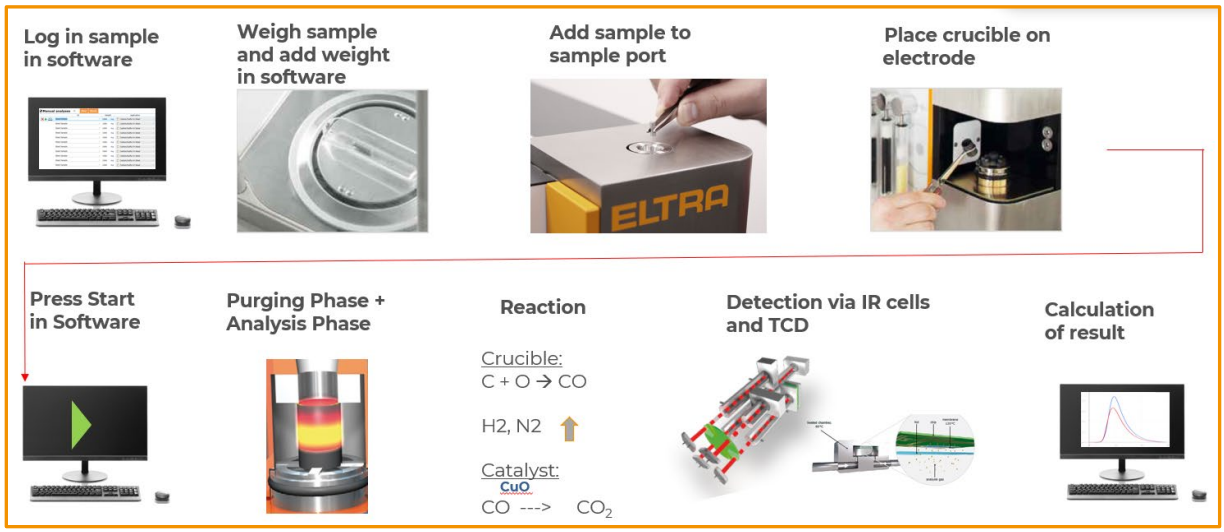


Illustration of the O/N/H analysis process

The sample is logged in with its sample weight in the software and the sample is applied to the sample port. A graphite crucible must be placed on the lower electrode. With starting the analysis, the furnace closes and the crucible is preheated. At the same time the sample port is flushed with carrier gas to remove atmospheric gases. After an outgassing and stabilizing phase the sample falls into the hot crucible and oxygen is released as CO, whereas nitrogen and hydrogen are released in its elemental form. The carrier gas led the released gases through a catalyst and subsequently to IR and/or TC cells.

2.1 Typical consumables and accessories

For ELTRA inert gas fusion analyzers different consumables are available at ELTRA which can be used for standard compliant analysis of different samples:

Part Number	Name	Intended use
88400-0213	Tweezer, curved for graphite crucible	Application of graphite crucibles
88400-0229	Tweezer 160 mm, acuate ending	Application of solid samples to the sample port
90180	Inner graphite crucible	Can be used for every application
90185	Outer graphite crucible	
90190	Single crucibles	Recommended for copper and steel analysis
90257	Nickel capsules 3.2 x 7 mm	Measuring of powders for O/N/H (small sample amounts)
90256	Nickel capsules 4.5 x 10 mm	Measuring of powders for O/N/H (medium sample amounts)
90252	Tin capsules 5 x 18 mm	Measuring of powders for N/H (large sample amounts)
90800	Graphite	Application of up to 30 mg in graphite crucible can improve repeatability and recovery of oxygen measurement
88600-0012	Nickel baskets	Required for O/N analysis in titanium
90251	Tin pellets	Required for H analysis in titanium
90258	Nickel	Can be used as flux for analysis of sample with a low melting point

90190 Crucibles

90256 Nickel capsules

90252 Tin capsules

2.2 Typical settings and required tools for standard applications

Typical applications for inert gas fusion analyzers are the O/N/H analysis of metals, alloys and ceramics. The following table summarizes the basic settings for these most important applications:

Element	Sample weight (mg)	Crucibles	Flux	Analysis power
Iron based materials: Steel, iron				
ON	1000	90190 or 90180+90185	NN	4000 – 5000 W
H	1000			3200 – 4200 W
Copper based materials				
O	1000	90190 or 90180+90185	NN	3000 – 5000 W
Titanium based materials				
ON	100	90180+90185	Nickel basket	5500 – 6000 W
H	100 – 250		Tin pellets	32000 – 4000 W
Ceramics				
ON	10 – 50	90180+90185	Graphite	5500 – 6000 W

Typical sample weight

For steel, copper and comparable samples the applicable sample weight could be different depending on the utilized analyzer, chemical composition of the sample and sample shape. Typically it is in the range between 250 and 3000 mg.

For refractories like titanium the maximum of applicable sample weight is limited to (100-130) mg for ON and approx. 250 mg for H analysis. Higher sample amounts may not release the embedded gases.

For ceramics the working range of the analyzer has to be considered. High sample weights can cause saturation of the IR or TC cell.

Crucibles

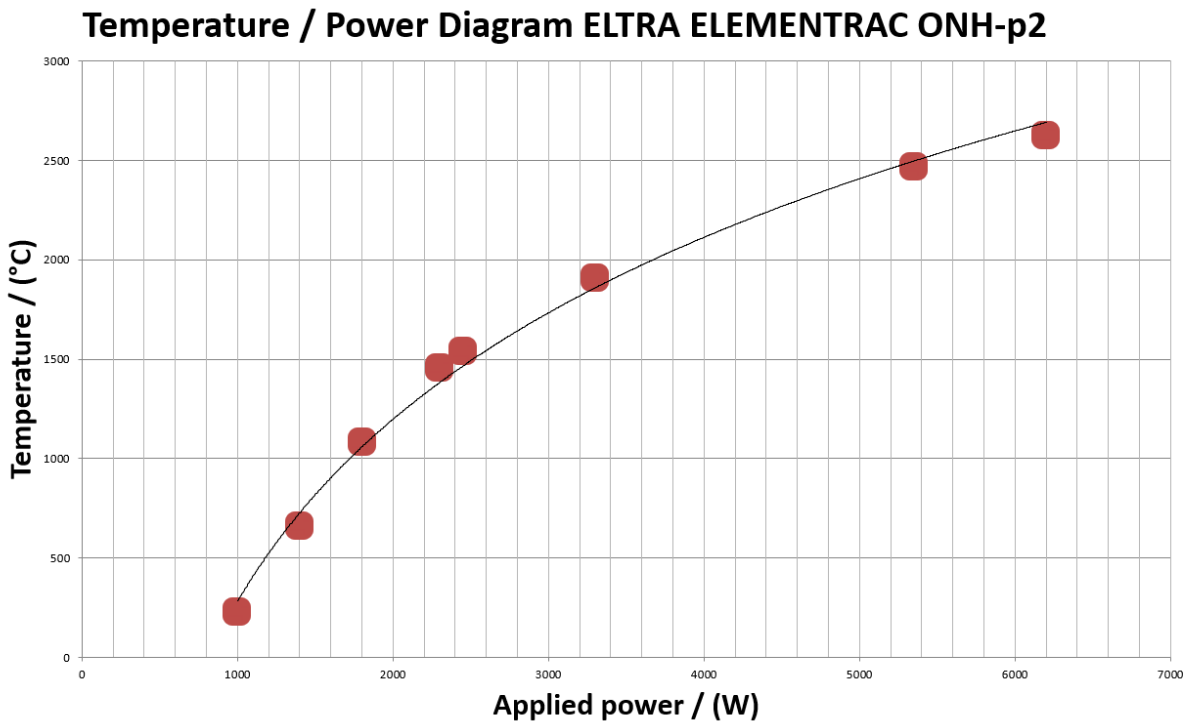
The combination of inner and outer crucibles is suitable for alle applications and provides in general the best repeatability. Single crucibles (90190) require in general less analysis power (-300W) and are recommended for copper and steel applications. The application of higher power like 5000 W or more can cause cracks in the crucible and sticking on upper electrode.

Flux

Flux material sometimes is required for standard compliant analysis, e.g. O/N or H analysis in titanium. The flux material reduces the melting point of the sample and assures the complete release of the embedded gases. For oxygen analysis in ceramics or other samples with high oxygen content (0.1 % and higher) additional graphite could improve the repeatability of measurements because the additional fine divided graphite provides more surface for reaction with oxygen of the sample. The application of nickel (90258) could be recommended when the analysis of samples with a low melting point is required (e.g. tin, zinc). For such applications, the melting point of the sample is increased by the application of nickel.

Analysis power

The application of the correct analysis power should assure the complete release of the embedded gases (e.g. N_2). For unknown samples the melting point of the sample could be a good orientation for the application of the correct analysis power:



Temperature / Power Diagram for ONH-p2

Metal	Melting point (°C)	Required power for melting (W)
Tin	231	1000
Copper	1085	1800
Nickel	1455	2200
Iron	1538	2300
Chromium	1907	3200
Niobium	2469	5350
Molybdenum	2623	6500

Caution!
A melting of the sample does not automatically mean that the embedded gases are released. See example below:

Example:
Pure copper melts at an applied power of 1800 W. But the provided temperature at 1800 W is not sufficient to release the oxygen of the sample. At 1800 W the oxygen of the copper sample cannot react with the graphite of the crucible. An analysis power of 3200 W in minimum assures a melting of the sample as well as the reaction of the present oxygen with graphite.

The applied analysis power for an application should be adjusted due to

- Sample amount (more power for more sample)
- Oxygen content of the sample (more power when more oxygen is present)
- Temperature of furnace (consider the temperature of the cooling water)
- High content of other elements (e.g. Mn)

Note:
The applied analysis power should be the same as the applied power for stabilization.

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Suitable calibration material for ONH analysis

Application	Can also be used for	Recommended calibration material
O/N/H analysis in steel	Nickel, cobalt (+500 W)	Steel pins 91100-100x for O/N 91400-100x for H Steel powder for high O 91500-1001 Copper pins for O 91100-100x
Copper	Brass, bronze	Copper pins for O 91100-100x
O/N/H analysis in titanium	Ti6Al4V; Zr;Hf	Titanium pins for ONH 91205-100x Titanium pins for H 91305-100x

2.3 Usage of different crucibles

Inner and outer crucibles are the recommended crucibles for most applications. They assure a reliable and repeatable measurement. For steel and copper analysis single crucibles may be used, but the usage of single crucibles for high temperature applications (e.g. O/N analysis in titanium) can cause trouble like bubbling and cracking of crucibles. For working with inner and outer crucibles just place the inner crucible (90180) into an outer crucible (90185):

Illustration of usage of inner and outer crucible

After the measurement just give the inner crucible into waste. The outer crucible can be used approximately 10 times.

Note:

Single crucibles can only be used once. For operating with autoloader only single crucibles (Different part number: 88400-0471) can be used. For analysis of refractories with an autoloader and single crucibles different settings and a chiller are required (see application note 1102).

2.4 ONH analysis of different shaped samples

Depending on the shape of the sample different requirements for a correct analysis has to be considered:

- Solid samples must be tested if they fit into crucibles and sample port (esp. special shaped samples or application of high sample weights)
- Granule and chipped samples must be tested for their applicable maximum sample weight. Maybe a capsule is required when the chipped sample contains a lot of fine divided dust.
- Powders always must be analysed by the utilization of a capsule

2.4.1 Analysis of solid samples

Samples which are shaped as pin (nominal weight of 1g) usually do not cause any trouble in the analyzer. The application of unusual shaped samples (example below) always has to be tested if they fit into the sample port and utilized crucible. Samples with a critical shape could stick in the furnace or maybe cause a short circuit.

Illustration of a critical sample shape: Maybe not applicable in furnace and crucible

2.4.2 Analyse of granule, chipped samples

Chipped or drilled iron based samples can be applied directly to the sample port of the ONH analyzer. Depending on the sample shape and the utilized analyzer die applicable sample weight is different. Please test if the applied sample weight and shape may block the sample port.

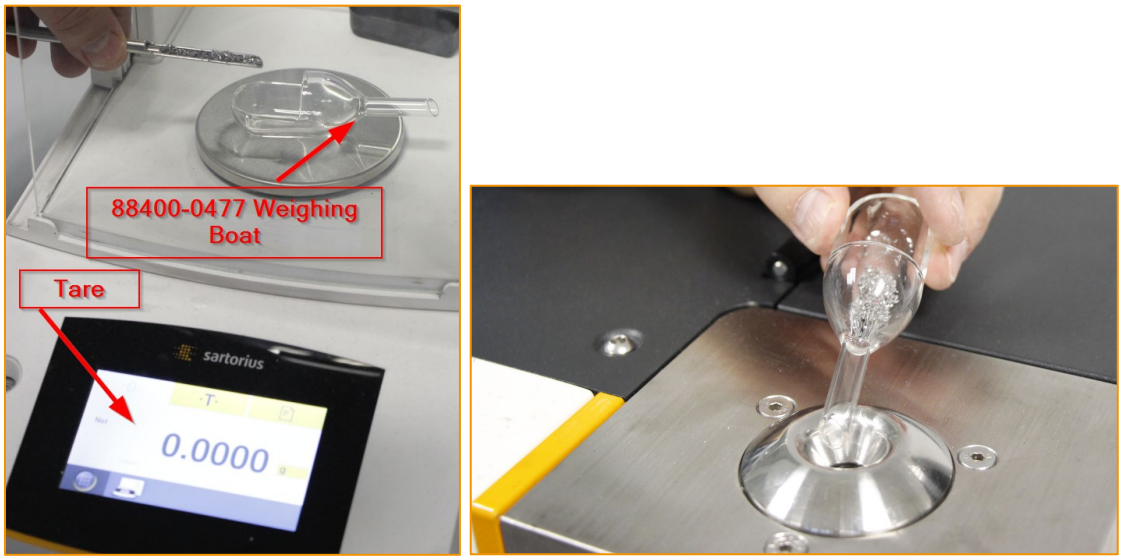
Examples of different shaped samples: test required regarding applicable sample weight

ELTRA recommends to use a weighing boat to apply iron based granulates

Part number	Description
88400-0477	Weighing boat
23110	Spatula 6

Application procedure for analyzing chipped samples

- Place the weighing boat on the balance and tare
- Weigh the sample and enter the weight in the software
- Fill in the granule or chipped sample in the sample port
- ELEMENTRAC ONH-p2: Switch of the setting: “Purging while closing”
- Start analysis



Weighing and application of granule /chipped sample to ONH analyzer

2.4.3 Analysis of powdered samples

Powdered samples cannot be applied directly to the furnace. The sample has to be filled into capsules. The application of powders without capsules could cause several issues like minor determination of O/N/H due to insufficient sample transport to the crucible. The utilization of capsules differs depending on the utilized analyzer and chemical composition of sample

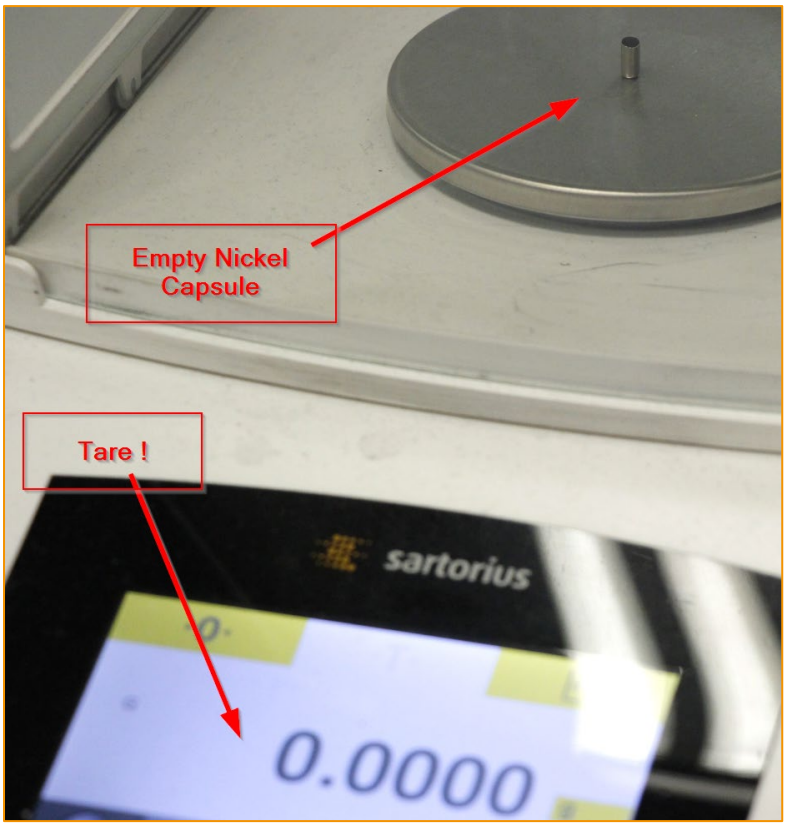
Applicable sample weight	Analyzer	Suitable capsules	Workflow
Iron, copper, nickel based powder			
100-500 mg	ELEMENTRAC ONH-p2	90256 / 90257	Fill powder in capsule, apply to sample port without sealing
	ONH-p1; ONH-2000	90256 / 90257	Fill powder in capsules, seal it and apply to sample port
Titanium based powder			
100 mg for ON; 250 mg for H analysis	ELEMENTRAC ONH-p2	90256	Fill powder in capsule. - ON analysis: place open capsule in basket and apply basket with capsule to furnace - H analysis: place open capsule in furnace
	ONH-p1; ONH-2000	90256	Fill powder in capsule. - ON analysis: seal capsule and apply it to basket. Apply basket with capsule to sample port -H analysis: place sealed capsule in sample port

Recommended tools for ONH powder analysis

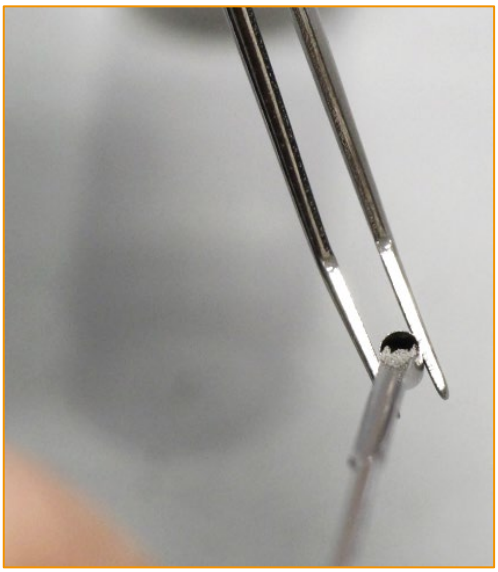
Part number	Description
88400-0213	Tweezer, curved for handling of graphite crucible
88400-0229	Tweezer 160 mm, acuate ending
90257 or 90256	Nickel capsule
88400-0476	Micro spatula
NN	Tongs to seal the capsule
88600-0012	Nickel baskets: Required for O/N analysis in titanium
90251	Tin pellets: Required for H analysis in titanium

In the following the workflow for the analysis of powders is illustrated.

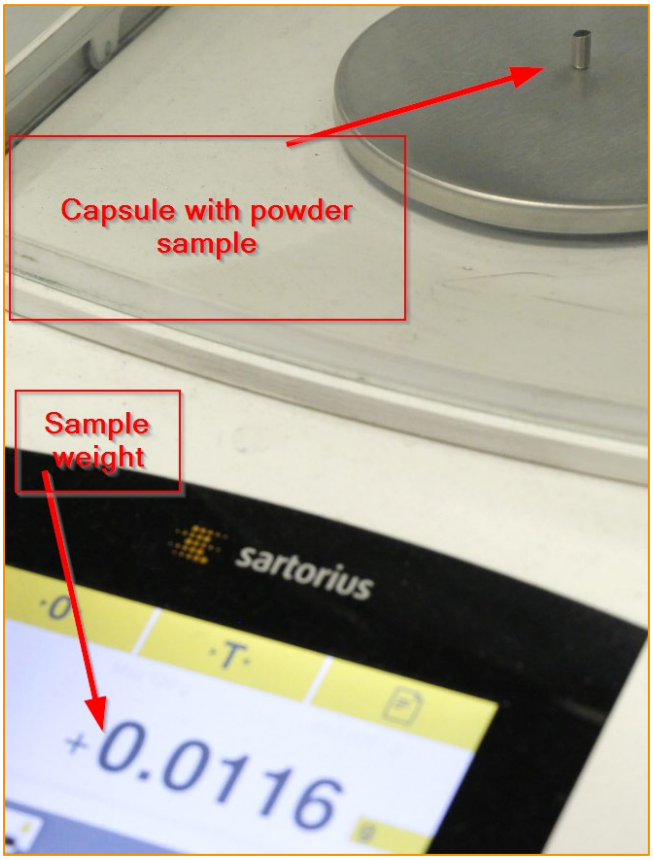
- Place the empty (nickel) capsule on the balance and tare



- Fill in the sample by utilization of a (micro) spatula



- Weigh the filed capsule again and type in the weight in the software



- **Note:**
  Depending on the utilized analyzer and composition of the sample the subsequent workflow is different. For analysis of powders made from copper, steel, iron and comparable materials the capsule can be applied directly to the sample port. In case of titanium analysis the nickel capsule has to be filled in a nickel basket.

- ELEMENTRAC ONH-p2: Direct application of a capsule to the sample port without sealing (iron, copper, nickel and comparable samples)

- ONH2000 and ONH-p1: a sealing of the capsule is required due the rotating locks in the sample port:

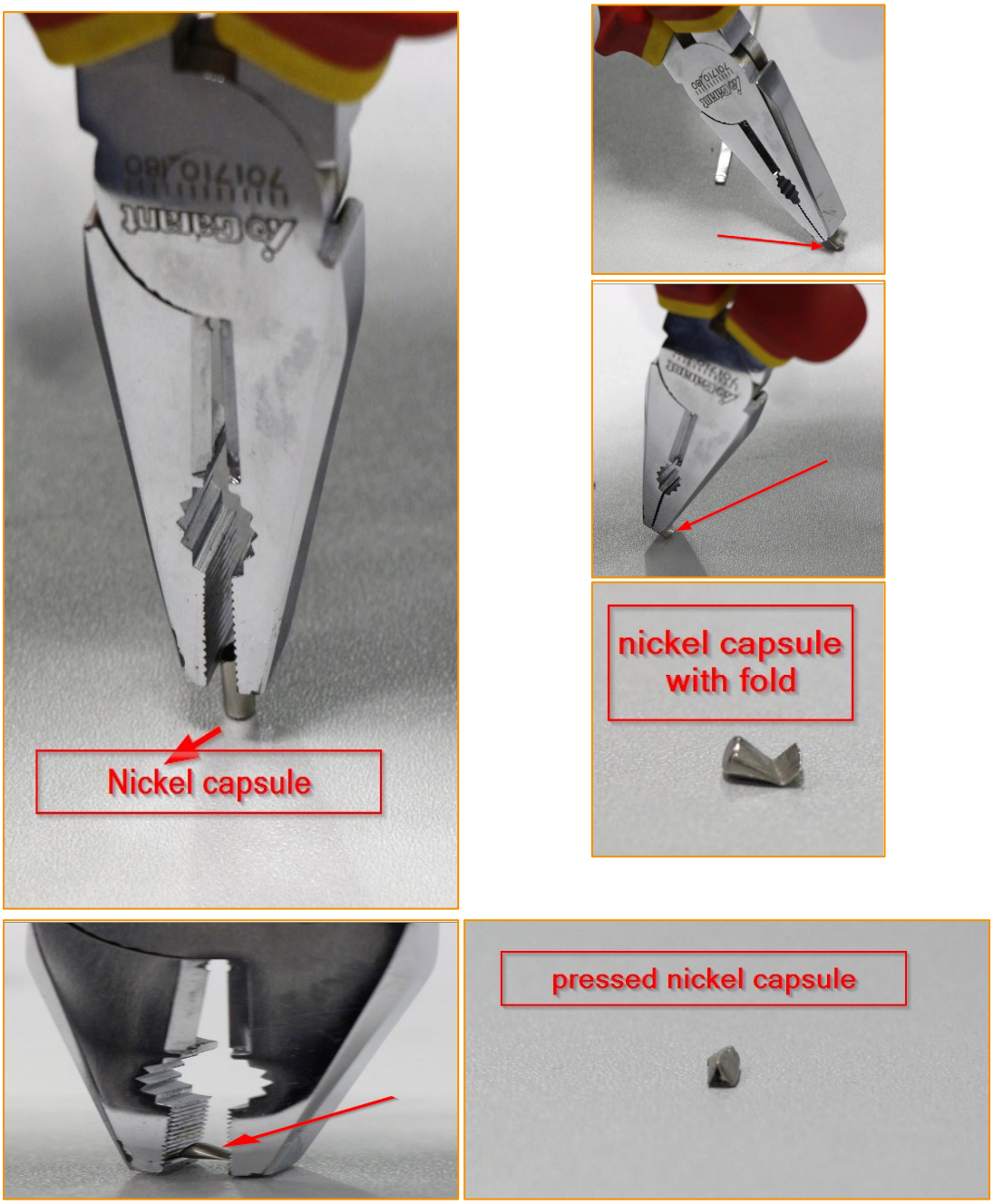


Illustration of sealing a capsule

- Iron, copper, nickel based samples (and comparable) can be applied directly to the sample port. Titanium based samples require flux (see chapter 2.5).

2.5 Usage of flux material

Some materials like titanium require for a correct and standard compliant analysis the application of flux like nickel baskets or tin pellets. The correct usage is explained in the following.

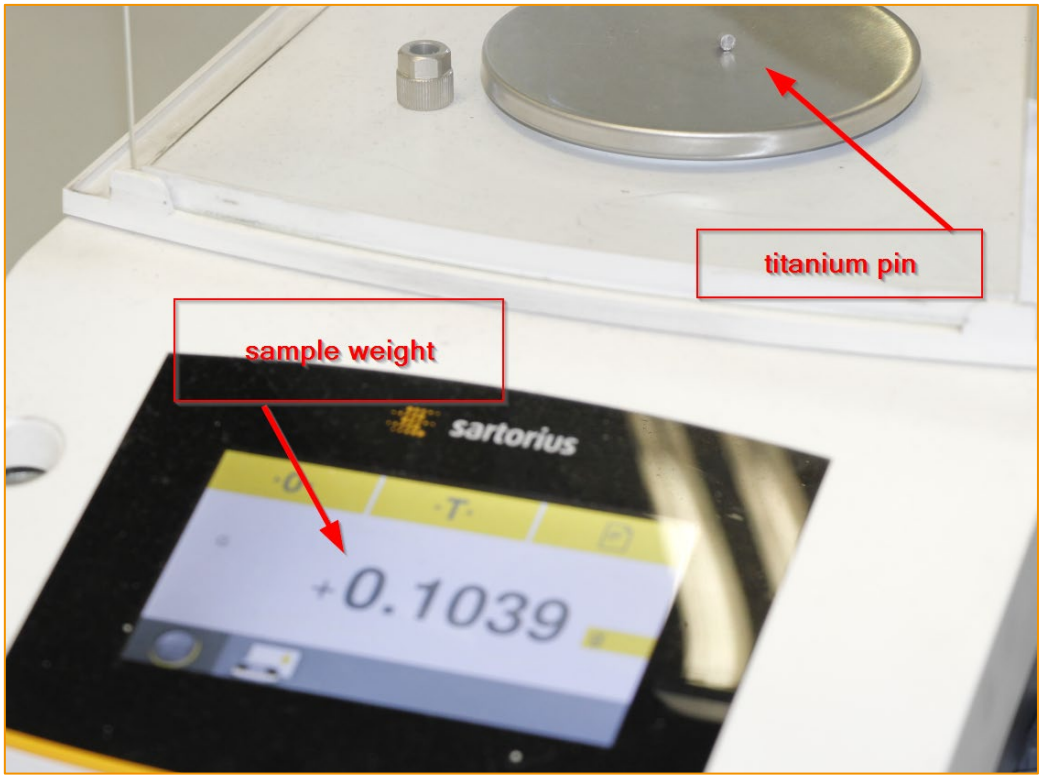
2.5.1 ON Analysis in titanium samples

For reliable and standard compliant O/N analysis of refractories the following consumables are required:

Part number	Description
90250, or **88600-0012**	Nickel basket, 1 g Nickel basket, 1 g high purity

Illustration of the workflow:

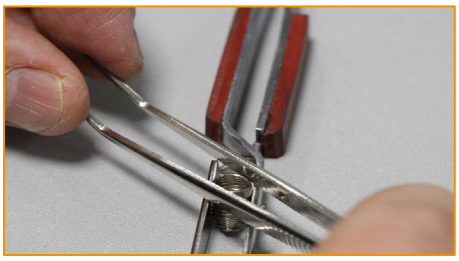
- Tare the balance and weigh the sample or CRM (<=130 mg)

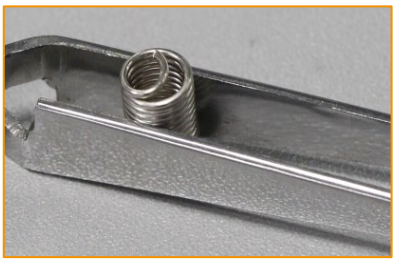


- Edit the sample weight in the software
- Take a nickel basket and apply sample in the basket

- ELEMENTRAC ONH-p2:
  Direct application of basket + sample to the sample port

- ELEMENTRAC ONH-p1 and ONH-2000: Sealing of the basket is required due the rotating sample lock





2.5.2 H analysis in titanium samples

For correct and standard compliant H analysis in refractories the following consumables are required:

Part number	Description
90251	Tin pellets

Illustration of the Workflow

- Weigh the sample or CRM (maximum sample weight approx. 250 mg)
- Log in the CRM or sample with its weight in the software
- Apply the sample to the sample port
- Fill in 2 tin pellets (90251) in the inner crucible and place the inner crucible on the electrode tip

Application of tin pellets to a graphite crucible (H in titanium analysis

2.6 Typical errors of ONH analysis

Error	Possible reason (in order of relevance)
Irritating Nitrogen results	- Filter (chemicals) before TC cell is saturated - Too low applied power (esp. for titanium, high alloyed steel samples) or too low minimum furnace temperature - Worn copper oxide catalyst (red/pink coloured)
Irritating oxygen results	- Dirty or worn upper or lower electrode or dirty furnace (clean upper electrode or replace it, clean furnace) - Bubbling (see picture below); add 30 mg graphite to the crucible or increase post waiting time to assure a cold furnace) - Too low furnace temperature (45°C in minimum) - Worn copper oxide catalyst (red/pink coloured) - Blank values of capsules and baskets - Contaminations on surface of sample (Recommendation: clean it with acetone)
Irritating oxygen and nitrogen results	- General cleanness of furnace and electrodes - Replace glass wool at furnace outlet - Unsuitable sample weight - Not constant work flow (Recommendation: Try to assure a sample analysis every 5 minutes). Alternatively try to increase analysis, stabilizing and analysis power
Error message Open circuit	- Check O Ring at lower electrode (clean and grease it) - Check condition of lower electrode (Replace it)
Gas flow not constant	- Search for leakage (chemical tubes, furnace) - Check pressure of gas supply (2 – 4 bar)

Illustration of “Bubbling”

3 INDUCTION FURNACE ANALYZER FOR “C/S” ANALYSIS

ETRA combustion analysis with induction furnace are well suited for the reliable measurement of carbon and sulfur in predominantly inorganic samples like steel, iron and ores or building materials.

Topic	Description
Suitable analyzers	ELEMENTRAC CS-i
Outdated analyzer	CS 800
Typical sample	Iron, steel, cast iron, copper, other metals, ceramics, building materials, ores
Not supported	Fuels (coal, coke, oil), food, pharmaceuticals
Typical sample weights	60 – 1000 mg
Typical analysis time	30 – 80 seconds (only peaks)
Typical cycle time	60 – 180 seconds (total analysis time)

ELEMENTRAC CS-I with optional 36 position autoloader

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General usage

Illustration of a C/S analysis with the ELEMENTRAC CS-i

The sample is applied to a ceramic crucible and logged in with its sample weight in the software. After adding a suitable accelerator (e.g. tungsten) the crucible is placed on the pedestal. With starting the analysis the combustion zone is flushed with the carrier gas oxygen, followed by the application of power by the induction furnace. This causes a combustion of the sample and a release of the embedded carbon and sulfur as CO_2, and SO_2. These gases are measured in up two 4 IR cells.

3.1 Typical consumable and accessories

For C/S analyzers with induction furnace different consumables are available:

Part number	Name	Intended use
90145	Tongs for ceramic crucibles	Transporting of crucibles
88400-0229	Tweezer 160 mm, acuate ending	Application of solid samples, lids
23110 / 23111	Spatula 6 or Spatula 8	Application of samples
23113	Spoon	Dosing accelerator
90149	Ceramic crucibles, foil wrapped, 1000 pcs.	Analysis of steel samples
90148	Ceramic crucibles, bagged, 1000 pcs.	Analysis of ceramics, limestone (higher Carbon Blank value)
88600-0014	Ceramic lids (10 mm whole) for crucibles	Required for samples with very dusty combustion. Utilization can influence sulfur measurement.
88600-0017	Ceramic lids (4 mm whole) for crucibles	
90220	Tungsten accelerator	Standard accelerator for analysis of steel and other metallic samples (1.7 g)
90260	Iron accelerator	Additionally required (0.7 g) for analysis of not metallic samples or cast iron. High purity accelerator has lower C/S Blank values.
88600-0013	Iron accelerator high purity	
88600-0010	Eltracell accelerator	Analysis of refractories
90280	Tin accelerator	Can be used instead of iron (max. 0.5 g)
90240	Copper accelerator	Suitable for analysis of carbides (SiC; WC) **Note**: can supress sulfur measurements

3.2 Typical settings and required tools for standard applications

For the most common applications the following accelerators, weights, CRMS and crucibles are recommended:

Sample	Accelerator	Typical Sample Weight	Crucibles	CRMS for calibration
Steel/Iron	1.7 g Tungsten	250-1000 mg	90149	Steel CRMS 92400 -30xx 92400- 40x 92500- 100x
Ti,Zr,Cr,Mo	1.7 g Tungsten 0.7 g Iron ---------- Alternative 1.5 – 2 g Eltracell	Max 250 mg	90149	Steel CRMS Titanium CRM 91305-100x
Cast iron	1.7 g Tungsten 0.7 g Iron	200-500 mg	90149 or 90148	Cast iron CRM 92400 (-3090 and higher)
Copper	1 – 2 g Copper	500-1000 mg	90149	Steel CRM Copper CRM 91000-1001
Limestone, ores, soils, dusts, ferroalloys and comparable	1.7 g Tungsten 0.7 g Iron	50-250 mg	90149 or 90148	Limestone 90812-300x Soil 90817-300x Ore 91900-100x 91900-2001
Carbides (WC,SiC)	1 g Copper 0.7 g Iron ---------- Alternatively 1.7 g Tungsten 0.7 g Iron	Max 250 mg	90149 or 90148	$CaCO_3$ 90810 WC 90816-3001

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3.3 Utilization of accelerators

The accelerator is very important for a reliable C/S analysis with induction furnace. First it covers the sample and avoids swirling of the sample when oxygen is applied. Second the accelerator assures the reliable combustions of the sample. ELTRA recommends to apply the sample first, followed by the application of iron (when required) and tungsten. A constant application of accelerator (e.g. 1.7 ± 0.1 g tungsten) could improve the repeatability of measurements.

Application of tungsten with spoon (part number 23113)

Accelerator	1 spoon means	Comments
Tungsten	1.7 g	Standard accelerator for steel and iron samples. Can be combined with iron accelerator for the measurement of cast iron and not metal samples like cement, soil.
Iron (90260)	0.7 g	Recommended for analysis of ores, building materials and comparable samples. Must be used in combination with tungsten, (This iron accelerator has a higher blank value and deviation than the high purity iron accelerator)
Iron High purity	0.7 g	Recommended for precise carbon analysis of carbides, refractories (together with tungsten)
Eltracell	0.7 – 1 g	Suitable for analysis of refractories, cast iron and comparable samples Provides high combustion temperatures, but produces a lot of dust during combustion
Copper	1 g	Special accelerator for analysis of carbides and copper. Can be used purely or together with iron / tungsten. Advantage: Assures a smooth and reliable combustion Disadvantage: Can supress sulfur measurements

3.4 Application of correct sample weight

Typically a sample weight between 60 and 1000 mg is applied to the crucible and is covered with accelerator. The following list provides some basic tips and recommendations regarding the applicable sample weight for selected samples.

General recommendations

Application of homogeneous weights (e.g. 0.5 ± 0.05 g) improves the repeatability of measurements.

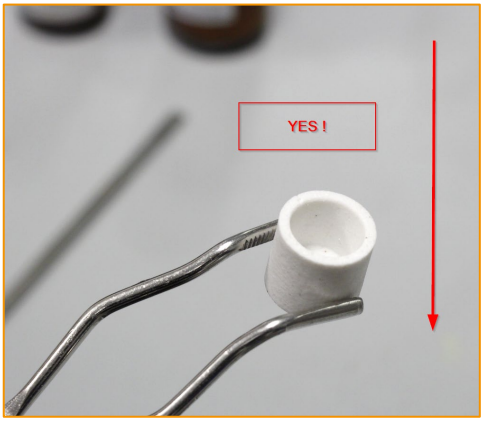
Steel samples: With high concentrations of other elements (Cr, Mn, Ti..) the application of pure tungsten may not be sufficient to release all the embedded carbon and sulfur. In this case increase the applied tungsten amount to 2 g, or apply 0.7 g Iron + 1.7 g tungsten or use 1.6 – 2 g ELTRACELL or use 1 – 2 g of copper (only valid for C measurement).

Type of sample	Shape	Recommendations regarding sample weight
Steel	Solid piece	A solid piece should be in the range between 0.3 and 1 g. When the application of 1.7 g tungsten is not sufficient follow the recommendations above.
Steel	Granule, chipped sample	Applied sample weights below 250 mg can show a worse repeatability for C/S. Application of high sample weights like 1000 mg can show an improved repeatability in the lower ppm range.
Cast iron	Solid, granule, powder	A sample weight of maximum 500 mg is recommended. The application of higher sample weights usually show worse repeatability.
Precious metals, Refractories	NN	These samples usually are limited to a sample amount of approx. 250 mg. Even with utilization of other accelerators the release of C/S could not be improved for higher sample weights
Limestone, cement, ores	Powder	These powders can show a high dust production or minor determination of C/S when too much sample is applied. Use 60 – 120 (150) mg
Carbides (WC)	Powder	For this sample the customer usually requests a high repeatability. The application of a constant weight of 250 +- 2 mg is urgently required.

3.5 Usage of crucibles

Please consider the following recommendations for handling of crucibles

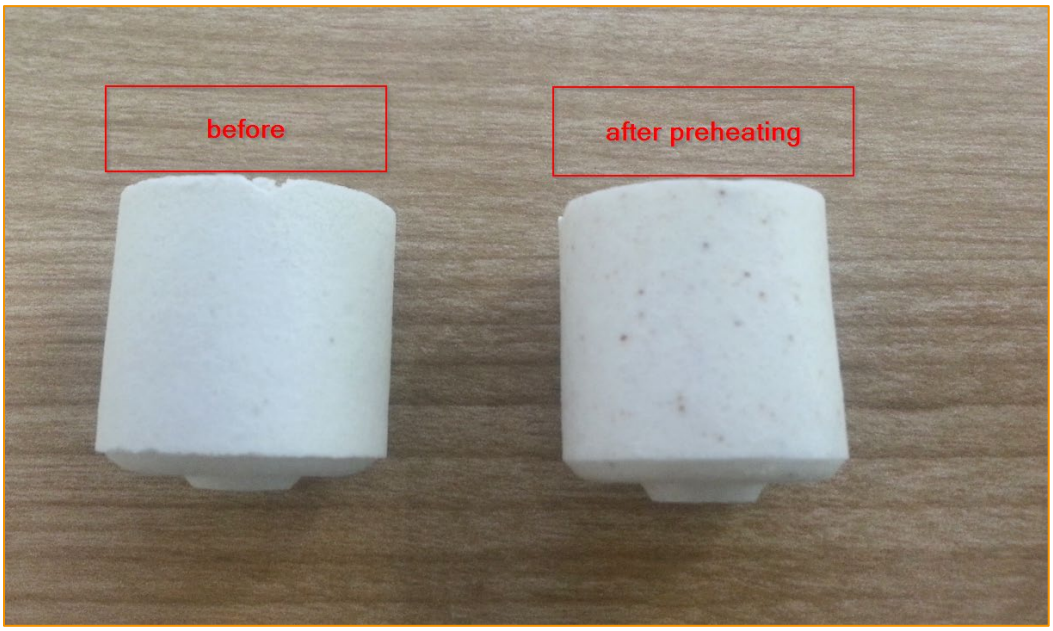
- Avoid touching the crucibles with your hands. Carbon blank value could be increased.



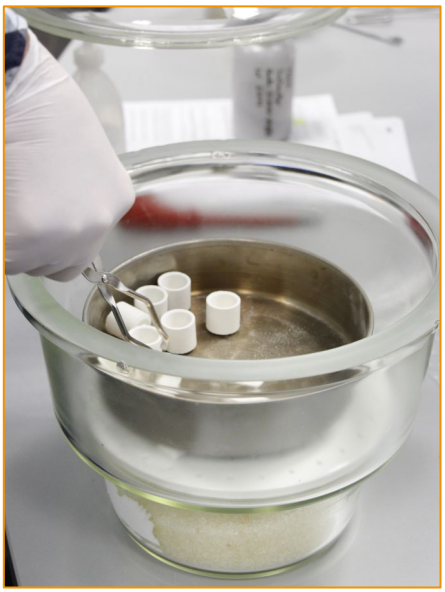
- Depending on the used crucibles and storage of crucibles the Blank value for carbon is different. The following table illustrates the effect.

CRM	Certified content (1 Sigma deviation)	Results with pre heated crucibles	Results with 90149 foil wrapped crucibles	Results with 90148 foil bagged crucibles	Results with 90148 foil bagged crucibles (longer air contact)
92400-3020#312A	C: 29 ± 5 ppm S: 43 ± 3 ppm	C: 29 ± 0.71 ppm S: 43. ± 0.36 ppm	C: 49 ± 7 ppm S: 40 ± 3 ppm	C: 55 ± 7 ppm S: 43 ± 1 ppm	C: 88 ± 11 ppm S: 44 ± 1 ppm
92400-3030#320L	C: 220 ± 10 ppm S: 320 ± 30 ppm	C: 220.02 ± 2.33 ppm S: 320.00 ± 0.95 ppm	C: 235 ± 2 ppm S: 321 ± 2 ppm	C: 237 ± 5 ppm S: 319 ± 1 ppm	C: 274 ± 7 ppm S: 326 ± 1 ppm
92400-3050#814F	C: 0.1820 ± 0.002 % S: 0.0370 ± 0.002 %	C: 0.1820 ± 0.0009 % S: 0.0370 ± 0.0001 %	C: 0.1820 ± 0.0021 % S: 0.0370 ± 0.0002 %	C: 0.1837 ± 0.0019 % S: 0.0367 ± 0.0002 %	C: 0.184 ± 0.0006 % S: 0.037 ± 0.0001 %
Medium Offset to the certified carbon values (based on 500 mg sample weight)	-	-	0-20 ppm	17-25 ppm	20-55 ppm

- **Note**:
  The best repeatability of carbon measurement could be obtained by the usage of calcinated (pre heated) crucibles. With higher carbon concentrations of the sample the carbon blank value of the crucible is negligible.
- Preheating (calcinating) of crucibles in a muffle furnace for in minimum 1hour at 1000°C could improve repeatability for carbon measurements in general.
- Some crucibles could change the colour when they are heated for a long time. This different colour does not affect the carbon blank value:



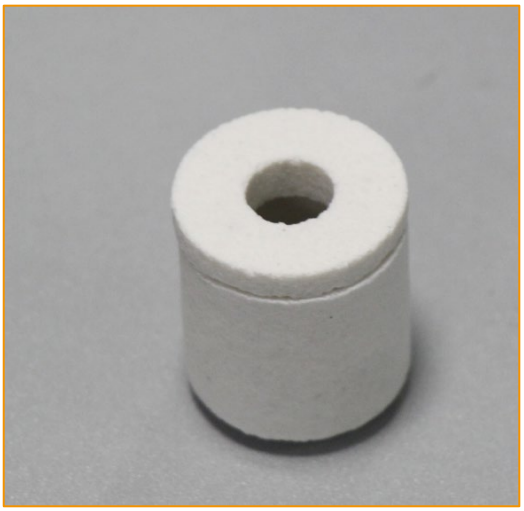
- Preheated (calcinated) crucibles should be stored in a desiccator.



3.6 Usage of lids

Some samples (e.g tin, ferromanganese) produce a lot of dust during combustion. To reduce the dust production and to improve the standard deviation of carbon measurement the application of a crucible lid is recommended.

It is just placed on top of the crucible.



Note:
The application of lids could influence the repeatability and recovery of sulfur in a negative way. Usually, a high flow of oxygen assures a high combustion temperature in the crucible and assures the complete release of sulfur as SO_2. When the oxygen flow is reduced in the crucible due to the application of a lid the resulting temperature may be not high enough to release all the embedded sulfur. This effect is sample depending.

3.7 Utilization of a carrier gas purification furnace

The repeatability of carbon measurement in the lower ppm range (C < 0.005 %) could be improved by several tasks:

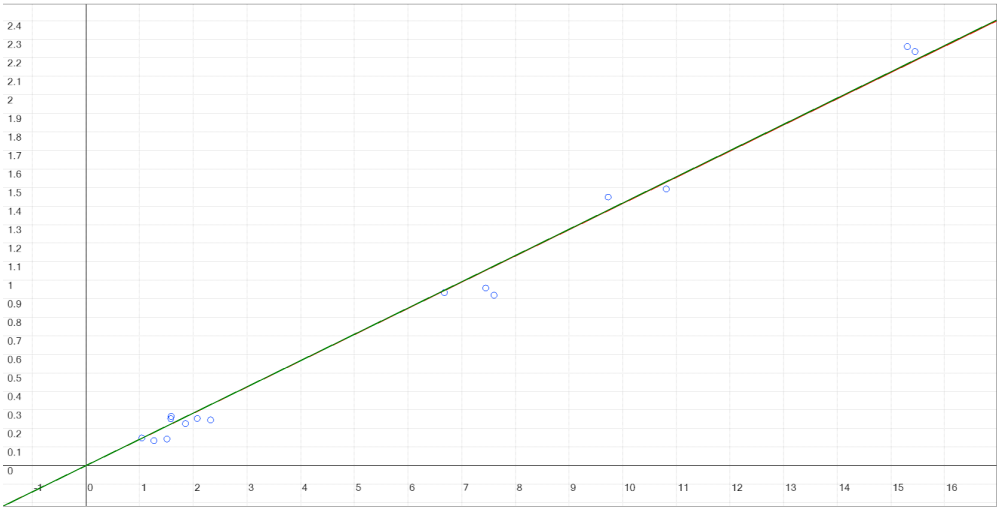
- Application of high sample weights (e.g. steel: 1000 mg),
- Preheating (calcinating) of crucibles
- Utilization of a carrier gas purification furnace (GRO 18).
  The carrier gas purification furnace oxidizes traces of hydrocarbons which may be present in the common carrier gas purity of 99.5 %. This reduces the carbon blank value. More details are given in the application note number 1080.

Typical measurements of steel samples with ultralow carbon concentrations can show results like this:

CRM Name	Certified carbon content (ppm)	Measured carbon content (ppm) (N=3) without carrier gas purification furnace	Measured carbon content (ppm) (N=3) with carrier gas purification furnace
502-401	2.6 ± 0.5	8.1 ± 0.2	3.2 ± 0.6
JSS 003-8	4.0 ± 0.6	11.5 ± 1.0	4.9 ± 04
502-704	5.0 ± 1.0	9.3 ± 0.6	4.4 ± 0.2
ECRM 285-2	18.0 ± 2.0	14.5 ± 1.0	19.5 ± 1.5
ECRM 284-3	25.0 ± 3.0	26.0 ± 1.9	24.7 ± 1.0
502-402	39.1 ± 0.9	34.6 ± 1.6	38.0 ± 0.5

A carrier gas purification furnace is connected between the applied carrier gas from the gas supply and the gas income of the CS analyzer. As illustrated in the table the carrier gas purification furnace has several positive effects on the carbon measurement in the lower ppm range

- Less carbon offset between certified and measured value
- Improved repeatability
- Linear calibration curve in the trace carbon range



Carbon calibration curve with 502-401, JSS 003-8, 502-704, ECRM 285-2, ECRM 284-3, 502-402

3.8 Typical errors of C/S measurements

Error	Possible reason (in order of relevance)
Deviating C/S results	- Application of too much sample - Contamination of heat shield (old sample falls into the crucible) - Dust trap filter has to be cleaned - Leakage - See upper furnace: O rings not sealed - Broken or dirty combustion tube - Blocked tube between furnace and dust trap - Replace Balston filter - Flatline in the measuring curves: Exchange HF tube
Minor determination of sulfur	- Replace anhydrone below heated dust trap (do not use glass wool on top) - Check temperature of dust trap (must be more than “hand worm”) - Check if sample contains copper. If yes clean furnace and apply a smooth combustion (e.g. by setting the gas flow to: chamber only: 25 seconds) - Replace Balston filter
Minor determination of C/S in the lower ppm range (< 50 ppm)	- Analyse a sufficient sample weight (1000 mg) - Apply a suitable calibration (CRMS between 0.0001-0.005 % C/S) - Perform leakage check, Maintenance of furnace and all chemicals - Use a suitable minimum analysis time (20-30 sec) and fitting maximum time (30 – 40 sec)
Deviating C results for low concentrations (< 0.005 %C)	- Use calcinated (preheated crucibles) - Apply a suitable sample weight (1000 mg) - Utilize a carrier gas purification furnace

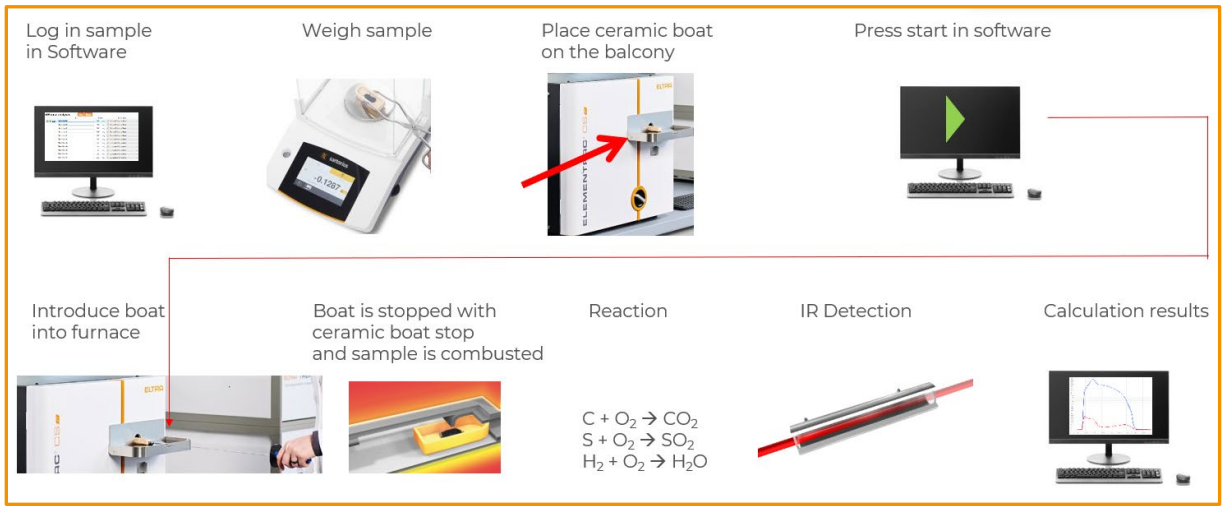
4 RESISTANCE FURNACE ANALYZER FOR “C(H)S” ANALYSIS

ELTRA combustion analyzers with resistance furnace are well suited for the reliable measurement of organic and selected inorganic samples.

Topic	Description
Current analyzer	ELEMENTRAC CS-r / CHS-r
Outdated analyzer	CS 580 / CHS 580
Typical samples	Coal, coke, oil, soil, ores, limestone
Not supported samples	Metals like steel, iron, copper
Typical sample weights	60 – 350 mg
Typical analysis time	30 – 240 seconds (only peaks)
Typical cycle time	50 – 300 sec (total analysis time)

ELEMENTRAC CS-r (with optional touch screen) and CHS-r

General usage



Usage of ELEMENTRAC C(H)S-r

Resistance furnace analyzer can be configured as C; S; C/S or C/H/S analyzer. In any configuration a sample has to be weight in a ceramic boat and the sample weight as well as its ID has to be logged in the software. With start of analysis the sample has to be introduced into the hot furnace for combustion. The released H_2O, CO_2 and SO_2 amounts are measured in up to 4 IR cells.

4.1 Typical consumable and accessories

For combustion analysis with resistance furnace the following consumables are common:

Part number	Name	Intended Use
90145	Tongs	Transportation of boats
23110 /23111	Spatula 6 or Spatula 8	Application of sample
90153	Re-Usable ceramic boats, 500 pieces	Analysis of fuels and other fast burning samples
90160	Disposable porcelain boat, 1000 pieces	Analysis of all common samples
88600-0011	Re-Usable ceramic boats, 95x13x10 mm, 500 pieces	Ideal suited for precise C/H/S measurements
90840	Quartz sand	For analysis of liquid samples and covering of fast burning samples (e.g. EDTA)
88600-0008	Combsolid	Recommended for analysis of sulfur in rocks, ores, cement
88400-0517	Transfer pipette	Application of liquid samples

88600-0008: Combsolid (tungsten trioxide): oxidizes sulfur in cement, rocks

4.2 Typical settings and required tools for standard applications

The following table provides common settings for the most analyzed samples:

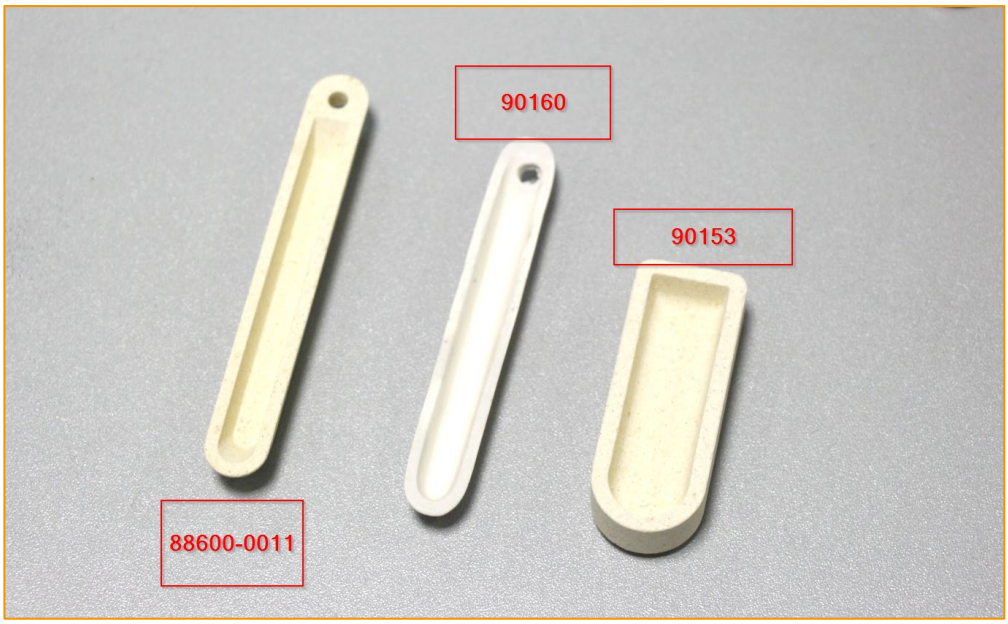
Sample	Recommended Boat; Other tools	Sample weight [mg]	Combustion Temperature [°C]	Suitable CRMs
Coal/Coke	90153 or 90160 or 88600-0011	150 – 350	1350	Coal 92511-30xx Coal prem 92550-30xx Coke prem 92560-3010 Pet coke 92570-30xx
Ores	90160 maybe 88600-0008	50 – 350	1450	Ores 91900-100x
Building materials	90160 and 88600-0008	80 – 250	1500	Limestone 90812-30xx
Oils	90153 or 88600-011 and 90840	30 – 150	1350 (1200)	Oil 92530

ELTRA CRM: Coal

ELTRA CRM: EDTA

4.3 General usage of combustion boats

For combustion analyser with resistance furnace several combustion boats are available:

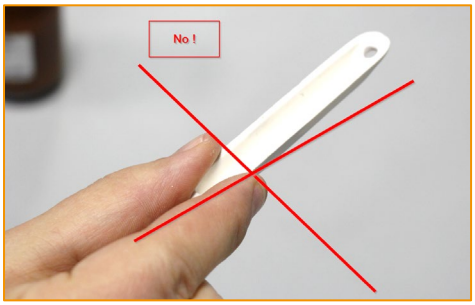


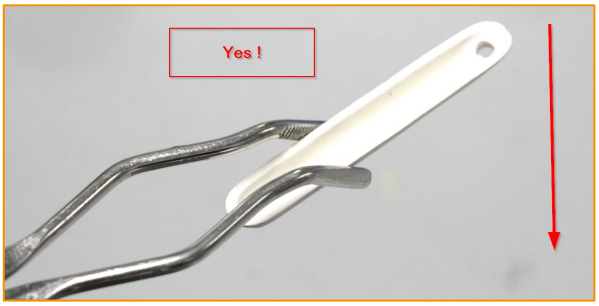
Combustion boats for C(H)S resistance furnace analyzer

Part number	Typical recommended samples
90153	Coal, coke, oil, plants, plastics
90160	Analysis of all common samples
88600-0011	Ideal suited for precise C/H/S measurements

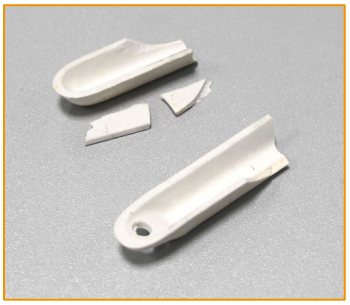
The following recommendations are valid for daily practice

- Do not touch the boats with hands, please use tongs to avoid carbon contamination



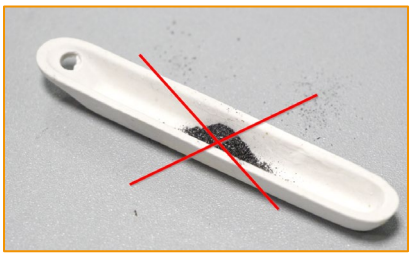


- When high temperature applications are applied (1400°C and more) the single use ceramic boats could break



- To avoid breaking the following steps are suitable:

a) Avoid to place intensive burning samples (like chemicals or fuels) on one spot, spread the sample on the whole boat



b) For temperatures above 1400°C the 88600-0011 boats should be the first choice for the analysis of carbon, hydrogen and sulfur. When disposable boats (90160) should be used an increased preheating (calcinating) time (6 hours at 1000°C) and storage in a desiccator is recommended.

4.4 Application of “accelerators”

Reliable C/H/S analysis of some samples could require the additional application of quartz sand or combsolid (tungsten oxide)

Tool	Recommended for analysis of	Description
90840: Quartz sand	Fast burning samples like oil, EDTA	Quartz sand absorbs liquids and assures a reliable combustion. Solid fast burning samples can be covered with quartz sand to reduce the intensity of combustion. This can improve the repeatability of measurement.
88600-0008: Combsolid	Slow burning samples like cement, limestone, ores, rocks, $BaSO_4$	Combsolid influences mainly the measurement (recovery) of sulfur. Combsolid oxidizes sulfur which maybe cannot be released by the applied temperature.

4.4.1 Usage of combsolid

Please use a small boat (90160 or 88600-0011), add the sample and cover the sample completely with combsolid. Afterwards it can be analysed in a resistance furnace.

Application of combsolid

4.4.2 Usage of quartz sand

Oil or other liquid samples should not be placed directly on the porcelain boot. Please add quartz sand first, afterwards the liquid sample, followed by additional quartz sand

Analysis procedure for analyzing liquid samples like oil

This procedure is also suitable to improve the repeatability of C/H/S measurement in fast burning samples (e.g. plant material).

For analysis of fast burning samples the utilization of bigger boats (90153) are recommended.

4.5 Typical errors of C/H/S measurements

Error	Description
General Handling	- Introduction of the sample: After starting of the analysis the CS-r stabilizes its baselines. In these time period no sample can be applied to the furnace, - Wrong position of the ceramic boat The sample must be introduced into the hot zone of the furnace (until the boat stop) - Too fast introduction of the sample Depending on the filling level of the boat a fast introduction (within 1-2 seconds) can cause sample lost and miner determination of C/H/S. A slow introduction (5-10 seconds) is helpful
Minor determination of sulfur	- Check the applied temperature - Check the channel settings (sulfur in rocks or limestone could be released lately in the combustion process. Sometimes no signal is visible for 120 seconds - Check the anhydrone tube. Anhydrone should be dry and no glass wool is allowed on top
Deviating C results in the lower concentration range (< 0.1%)	- Consider the blank values of the ceramic boats. Preheating (Calcinating) like C/S crucibles is recommended. - Apply a suitable (constant) sample weight
Deviating Hydrogen measurement	- Dry samples at 105°C (1 h) - Preheat (calcinate) ceramic boats
Saturation of channels	- Depending on the channel configuration the channels could easily be saturated when a high concentration is present: Adjust the applied sample weight - When saturation is caused by an intensive combustion # Reduce Temperature (when allowed) # Reduce sample weight (when possible) # Use the bigger boats (90153) instead of the small boats # Cover sample with quartz sand # Introduce sample extremely slowly int the furnace (20 seconds)

5 ANNEX

This Annex provides additional information about available ELTRA application notes and compliant standards of ELTRA analyzers. When more information is required, please contact: www.eltra.com or Info@eltra.com

5.1 Available application notes for outdated analyzers

Number	Analysis of	Sample
		Analyzer CHS 580
1002	C/H/S	Wood
1017	C/H/S	Oil
		Analyzer CS 580
1000	C/S	Coal
1001	C/S	Coke
1003	C/S	Wood
		Analyzer CS 800
1008	C/S	Ore
1009	C/S	Pure Iron
1010	C/S	Fly ash
1016	C/S	Copper
1023	C/S	Steel rings
		Analyzer ONH 2000
1011	O/N	Steel: Single crucibles
1012	O/N	Steel: Inner and outer crucibles
1013	O/N	Titanium
1014	O	Copper
1027	O/N	CeO2

Analyzer ONH-p1 (rotating sample lock)		
Number	**Analysis of**	**Sample**
1019	H	Steel
1020	O/N	Steel
1021	O/N	Titanium
1022	H	Titanium
1024	O/N	Steel granulates
1025	O	Copper
1026	O/N	Ferrochrome
1046	O/N	Titanium: Reduced power
1047	N	Silicon nitride
1052	O/H	TiH2
1057	O/N	Steel granulates & Powders via Manual load

1058	O	Copper with carrier gas Argon

5.2 Application notes for current ELTRA analyzers

Number	Analysis of	Sample
	Analyzer CS 580A	
1098	C/S	Plant Material
	Analyzer TGA Thermostep	
1004	Moisture Ash	Fluor
1005	Moisture Ash	Milk
1006	Moisture Ash	Biscuits
1007	Moisture Ash	Sausages
1015	Moisture Ash/LOI	Limestone
1069	Moisture Ash Volatile	Coal/coke

ELEMENTRAC CS-i		
Number	**Analysis of**	**Sample**
1028	C/S	Ores
1029	C/S	Machining steel
1030	C/S	Case Hardening steel
1031	C/S	High alloyed steel
1032	C/S	Nb stabilized steel
1033	C	Tungsten Carbide
1034	C/S	High purity Nickel
1035	C/S	Limestone
1036	C/S	Cast iron
1037	C/S	Copper
1038	C/S	Soil
1039	S	Soda-Lime-Silica Glass
1040	C/S	Pure Iron
1041	C/S	Steel rings
1042	C	Silicon carbide
1043	C/S	Ferrochrome (highC)
1044	C/S	Ferrochrome (lowC)
1045	S	Zinc sulfide
1048	C/S	Blast furnace dust
1049	S	Coke
1050	C/S	Electrode furnace dust
1051	C/S	Furnace slag
1053	C	Titanium
1054	C/S	Nickel alloy
1055	C/S	Chromium
1056	C/S	Titanium
1077	C/S	Ultra-pure iron
1078	C/S	Inconel
1089	C/S	Ferro-Niobium
1090	C/S	Ferro-Manganese
1091	C/S	Nitrided Ferro Silicon
1097	C/S	C/S analysis in steel and cast iron by utilization of different accelerators
1100	C/S	Lead samples (battery production)

ELEMENTRAC CS-I and CS-d , CS-r; CHS-r application notes		
ELEMENTRAC CS-I with carrier gas purification furnace (GRO 18)		
Number	**Analysis of**	**Sample**
1080	C/S	Measurement of ultra-low carbon steel with utilization of the GRO 18.
ELEMENTRAC CSi + CSi as cement configuration		
1065	C/S	Cement was measured with a CS-I standard configuration and an enlarged anhydrone (cement) configuration
ELEMENTRAC CSi (cement configuration)		
1067	C/S	Measurement of ore and iron ore
ELEMENTRA CS-I / CS-d (with halogen trap)		
1068	C/S	Measurement of C/S in CaF2 sample with the CS-I and the halogen trap
ELEMENTRAC CS-d (resistance furnace)		
1059	C/S	Coal
1060	C/S	Coke and Pet coke
1061	C/S	Limestone
1062	C/S	Ore
1063	C/S	Soil
1064	S	Zinc sulfide
ELEMENTRAC CS-d (resistance + induction furnace)		
1066	S	Measurement of copper concentrates and other sulfides with resistance and induction furnace
1092	C	Measurement of coated aluminium oxide with resistance and induction furnace
ELEMENTRAC CS-r		
1081	C/S	Coal
1082	C/S	Coke and pet coke
1083	S	Coal (ultra-low sulfur)
1084	C/S	Soil
1085	C/S	Iron ore
1086	C/S	Copper ore
1087	C/S	Rock
ELEMENTRAC CHS-r		
1088	C/H/S	Coal

ELEMENTRAC ONH-p2		
Number	**Analysis of**	**Sample**
1070	O	Copper
1071	O/N	Nickel powder
1072	O/N	Steel granulates& drillings
1073	O/N	Steel pins
1074	O/N	Titanium
1075	H	Titanium
1076	H	Steel
1079	O/H	Steel
1099	O/N	Silicon nitride
1101	O/N	Ferro alloys
ELEMENTRAC ON-p2 (carrier gas Argon)		
1093	O/N	Steel pins
1094	O/N	Steel granulates
1095	O	Copper
1096	O/N	Titanium
ELEMENTRAC ONH-p2 (Autoloader)		
1102	O/N/H	O/N/H analysis in steel, copper and titanium by utilization of the autoloader

5.3 ELTRA analyzers and compliant standards

ELEMENTRAC ONH-p1 / ONH-p2 and ONH 2000 series	
Standard	**Title**
ASTM E 1019-18	Nitrogen and oxygen in steel, iron, nickel and cobalt alloys
ASTM E 1409-13	Oxygen and nitrogen in titanium
ASTM E 1447-09	Hydrogen in titanium and titanium alloys
ASTM C 1494-13 (reapproved 2018)	Nitrogen, oxygen in silicon nitride powder
ASTM C 1457-18	Hydrogen in uranium oxide
ASTM C 1854-17	Hydrogen in mixed oxides ((U,Po)O2)
ASTM E 2575-19	Oxygen in copper
ASTM E 2792-13	Hydrogen in aluminium
DIN EN 3976: 2007	Titanium and titanium alloys – determination of hydrogen
DIN EN ISO 4491-4:2019	Metallic powders – determination of oxygen content
DIN EN ISO 10720:2007 DIN EN ISO 15351:2010	Steel and iron – determination of nitrogen content
DIN EN ISO 21068-3: 2008	Chemical analysis of silicon carbide – nitrogen, oxygen
ISO 17053:2005	Steel and iron- determination of oxygen
SO 22963:2008	Titanium and titanium alloys- determination of oxygen
DIN 54387-3:2016	Ceramic raw and basic materials – total oxygen and nitrogen in boron carbide, boron nitride

ELEMENTRAC CSi; CS800	
Standard	**Title**
ASTM E 1019-18	Carbon and Sulfur in steel, iron, nickel and cobalt alloys
ASTM C 1408-16	Carbon in uranium oxide powders
ASTM C 1494-13 (reapproved 2018)	Carbon in silicon nitride powder
ASTM E 1915-13	Metal bearing ores and related materials – carbon and sulfur analysis
ASTM E 1941-10	Carbon in refractory and reactive metals
DIN EN ISO 7526:2020	Ferronickel – determination of sulfur content
DIN EN ISO 15350:2010	Steel and iron- determination of total carbon and sulfur
DIN EN ISO 15349-2:2020	Unalloyed steel – determination of low carbon
DIN EN ISO 9556: 1989	Steel and iron – determination of total carbon
ISO/ TR 9686:2017	Direct reduced iron – determination of carbon and/or sulfur
ISO/TS 10719:2016	Cast irons – determination of non-combined carbon content
ISO 21614:2008	Determination of carbon content of UO2; (U,Gd)O2
ISO 9891:1994	Carbon content in uranium dioxide powder
ISO 11873: 2005	Determination of sulfur and carbon content in cobalt metal powders
ISO 4689-3:2017	Iron ores – determination of sulfur
DIN EN 24935: 1992 (identical with ISO 4935)	Steel and iron – Determination of sulfur content
ISO 13902:1997	Steel and iron – determination of high sulfur content
SO 7524: 2020	Nickel, ferronickel and nickel alloys – determination of carbon content
DIN EN 1744-1: 2013	Test for chemical properties of aggregates – sulfur
DIN 54387-3:2016	Ceramic raw and basic materials – total carbon in boron carbide, boron nitride
UOP Method 703-09	Carbon on catalysts by induction furnace combustion

CS-d (resistance furnace); C(H)S 580A ; C(H)S-r	
Standard	**Title**
ASTM D 1552-16	Sulfur in petroleum products
ASTM D 1619-16a	Test for carbon black – sulfur content
ASTM D 4239- 18	Sulfur in coal and coke
ASTM D 5016-16	Total sulfur in coal and coke combustion residues
ASTM D 6316-17	Total, combustible, and carbonate carbon in solid residues from coal and coke
ASTM D 7633-13 (reapproved 2018)	Test for carbon black – carbon content
ASTM D 7662-15	Carbon content in carbon black feedstock oils
ASTM D 7679-16	Sulfur content in carbon black feedstock oils
ISO 15178: 2000	Soil quality – determination of total sulfur
ISO 19579:2006	Solid mineral fuels – determination of sulfur
DIN EN 15936:2020 (also valid for TIC Module)	Sludge, treated biowaste, soil and waste – determination of total organic carbon (TOC)

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