Elemental Analysis for the Iron, Steel, and Metal Industry by OES, XRF and Combustion/Fusion

Bruker Webinar
July 09, 2020

Elemental Analysis for Iron, Steel and Metal Industry

Topics

- Introduction to steel and steel making process
  Iron & steel treated as representatives for all other metals
    - What is steel?
    - Workflow in an integrated steel plant
    - Individual strengths of the 3 complementary elemental analysis techniques: OES, XRF, Combustion/Fusion
- Full elemental analysis of metals by Optical Emission Spectrometry (OES)
    - Sample taking & preparation
    - OES principles
    - Examples with Q8 MAGELLAN
    - Automation with OES – Q8 MAGELLAN *online*
- Material and slag analysis by XRF
    - Principles & differentiation (ED vs WD)
    - Analysis of Raw-, Intermediate and final materials, online XRF
    - Optimized process control by slag analysis
    - Final material inspection

Elemental Analysis for Iron, Steel and Metal Industry

Topics

- Raw material- and slag-analysis by XRF
  - Principles & benefits
  - Raw materials analysis
  - Optimized process control by slag analysis
- Fast and precise analysis of C, S, O, N, H by combustion / fusion
  - Principles of carbon and sulfur analysis by combustion
  - Principles of oxygen, nitrogen and hydrogen analysis by fusion
  - Applications in the steel making process
  - Examples
  - Analysis of finished materials (e.g. welding rods) for diffusible hydrogen

Meet your speakers

Optical Emission Spectrometry
OES

Peter Paplewski
Product Manager OES, CS/ONH
Karlsruhe, Germany

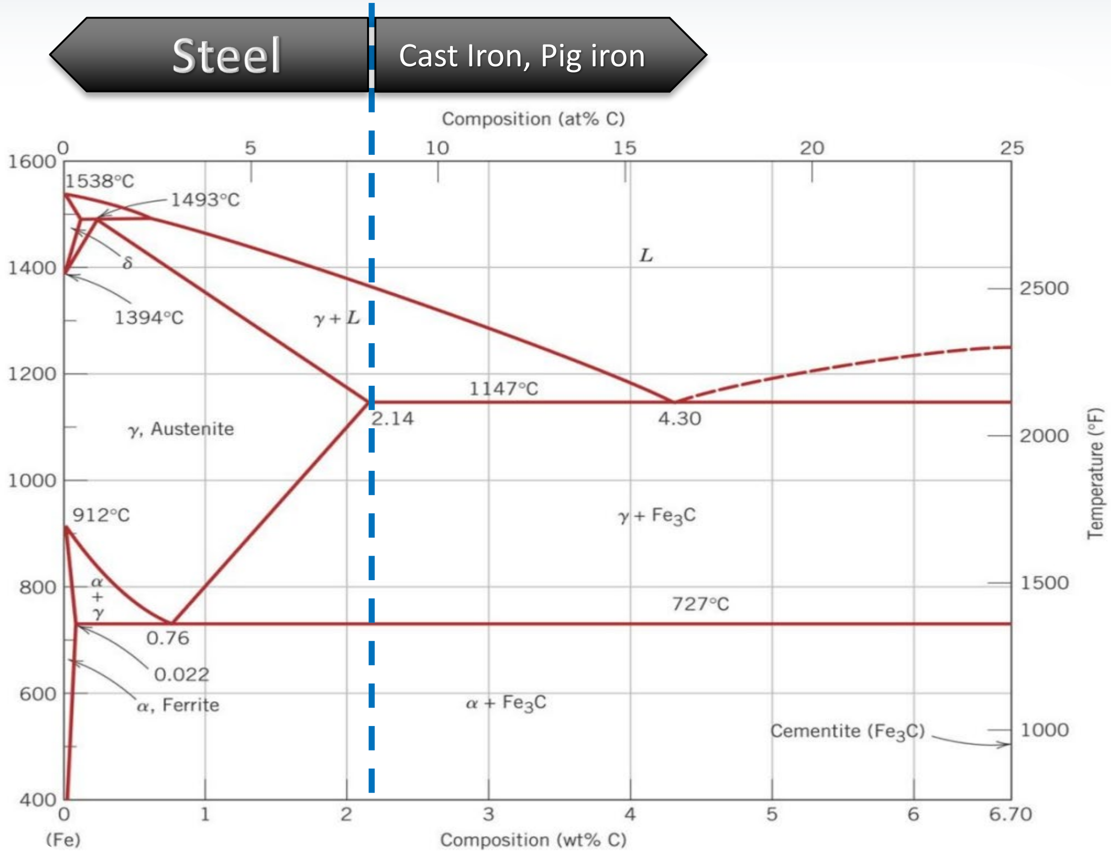
X-ray Fluorescence Spectrometry
XRF

Adrian Fiege
Product Manager XRF
Karlsruhe, Germany

Combustion/Fusion Analysis
CS/ONH

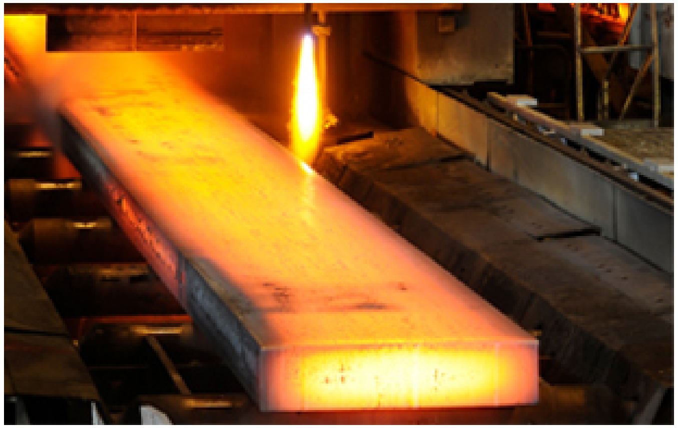
Kristin Odegaard
Technical Sales Specialist
CS/ONH, OES
Madison, WI, USA

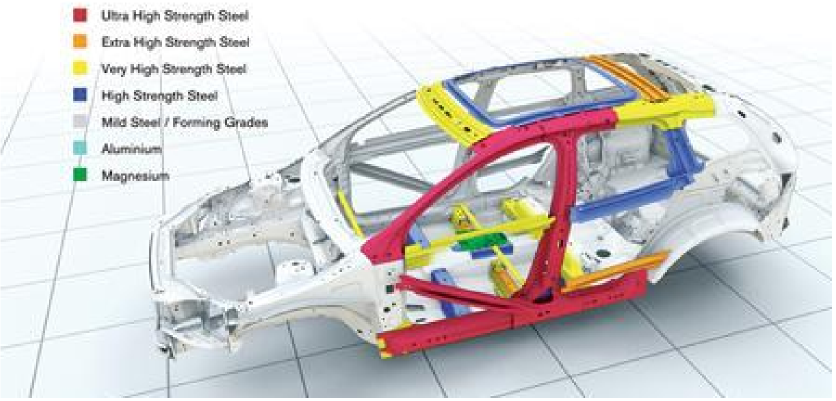
Introduction to steel and steel making
What is steel?



- **Steel**
  General name given to a large family of **alloys of iron with carbon**, containing less than 2.1% carbon and other elements
- Steel is such a versatile material because we can adjust its **composition** and internal **structure** to tailor its properties for certain applications
- More than 3.500 different grades of steel, of which 60% are younger than 4 years!
- If the Eiffel Tower were to be rebuilt today: only one-third of the steel originally used would be needed

Introduction to steel and steel making
Alloys of iron with carbon

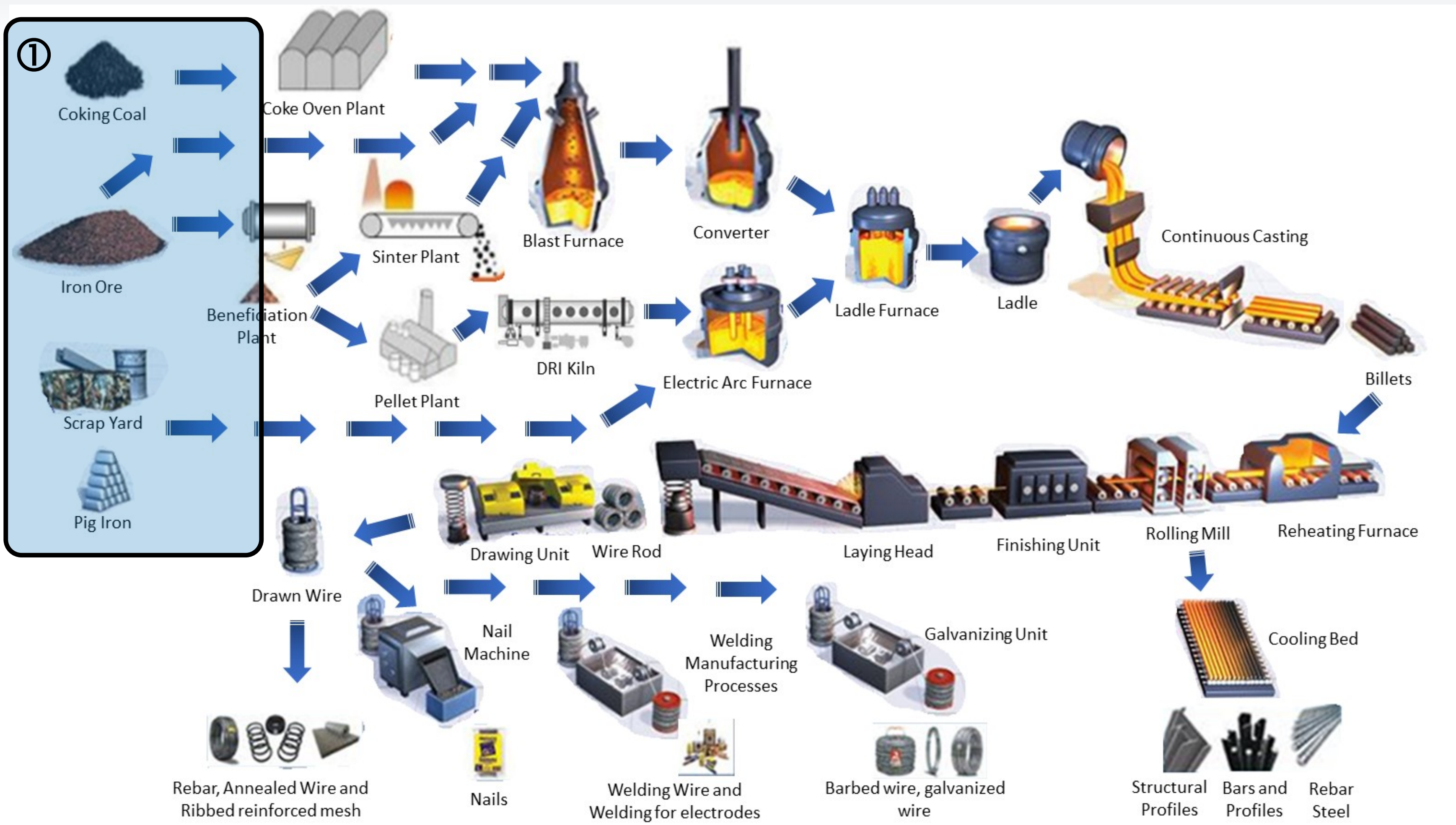




The final properties are influenced by

- Alloying: Adding other elements such as: **Si, Mn, C, P, S, Al, Ti, Mo, Cu, Cr, Ni, etc**. during furnace tapping (BOF, AOD, EAF) and/or in secondary metallurgy (Ladle furnace, RH Degasser, VD/VOD-Plant, CAS-OB , etc.)
- By rolling process (incl. heat treatments)

Introduction to steel and steel making
Workflow in an integrated steel plant



Workflow in an integrated steel plant
Raw materials

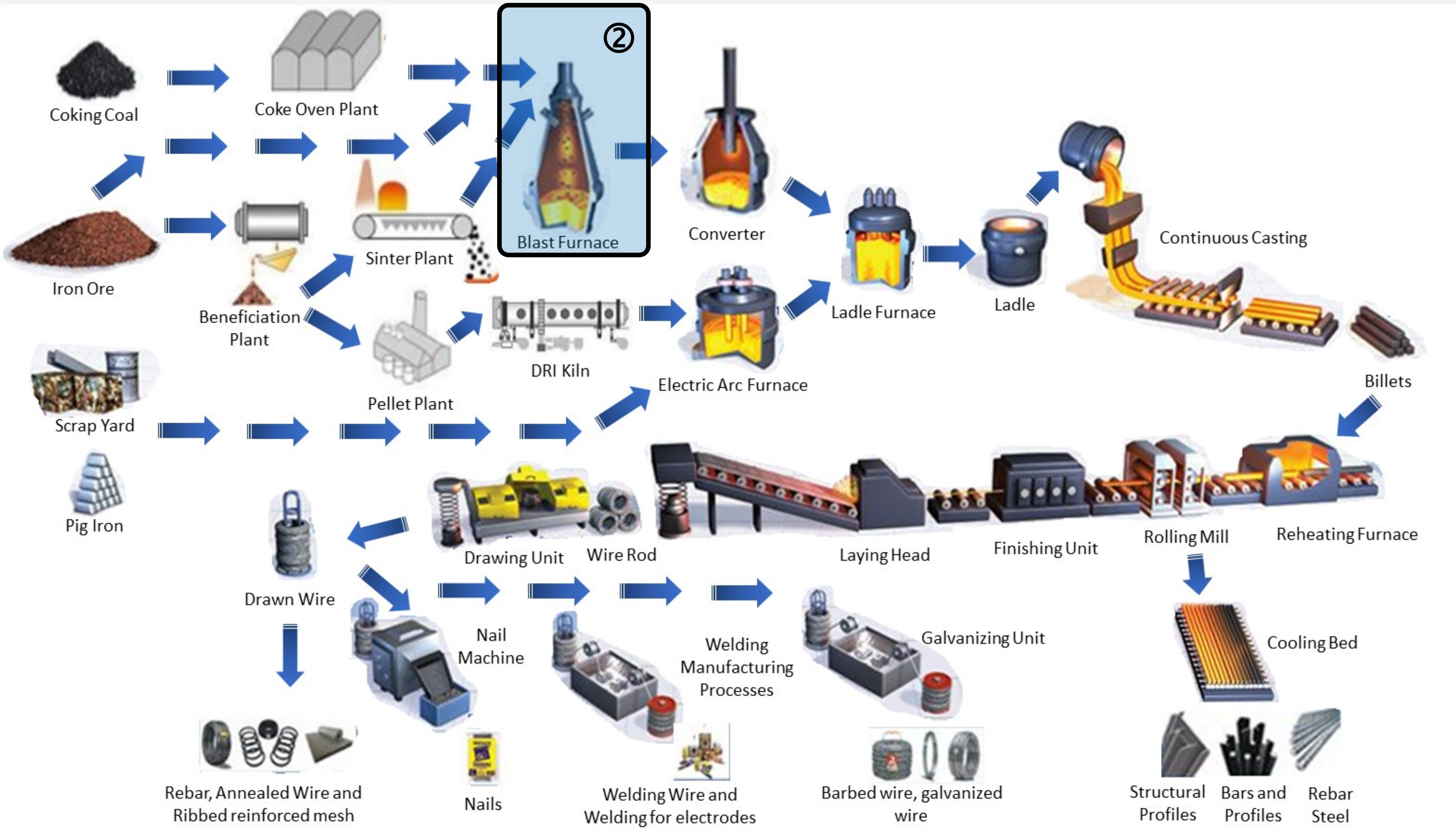
1. Raw materials

- Deposited in massive piles, ready to be processed
- Iron ore is analyzed immediately as it arrives to ensure it is the grade and quality ordered (and paid for)
- All input materials are analyzed for quality and purity
- Coke and associated waste are analyzed for quality and discharge purposes
- Sinter is analyzed prior going into the Blast Furnace
- Scrap is analyzed before going into Electric Arc Furnace

Iron Ore (Fe_xO_y)

Tata Scunthorpe, UK

Introduction to steel and steel making
Workflow in an integrated steel plant



Workflow in an integrated steel plant
Blast Furnace

2. Blast Furnace

- Aim: Reduce iron oxides to metallic iron
- The liquid iron is called "hot metal" ("pig iron" when solidified)
- Hot metal temperature: 1340 – 1380 deg C
- Hot metal transported by torpedo ladles to the BOF converter
- Important elements in hot metal are:

Element	Concentration Level
Carbon, **C**	4.2 - 4.5 %
Silicon, **Si**	0.2 - 1.2 %
Manganese, **Mn**	0.2 - 0.5%
Sulfur, **S**	0.01 - 0.0.7%
Phosphorus, **P**	0.05 – 0.13 %

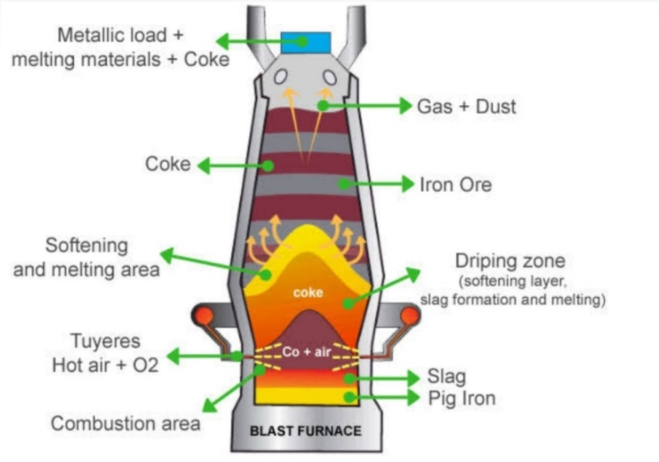
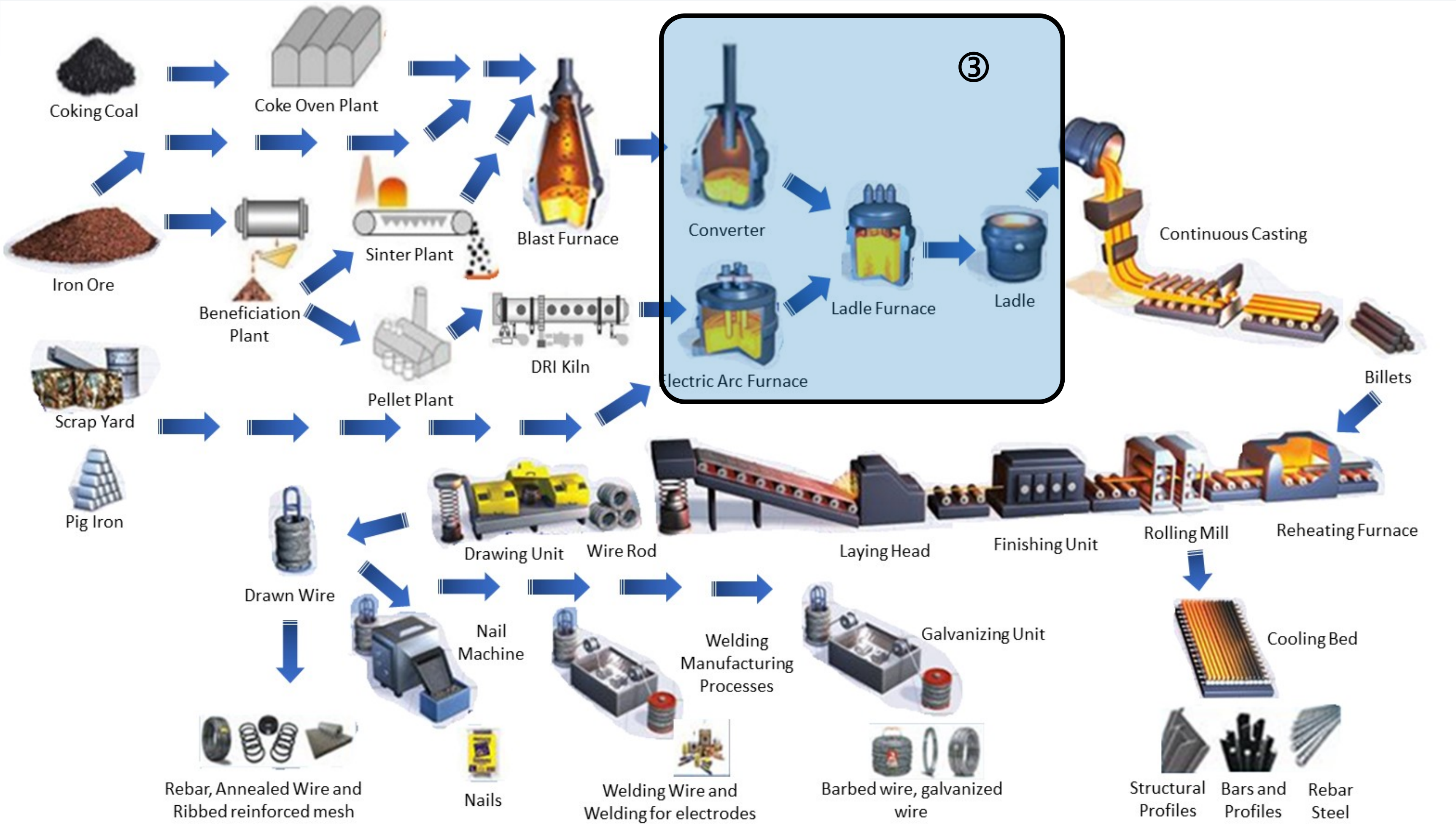


Figure 1 - Blast Furnace Operation

https://www.metalfurnace.ooo/2018/09/blast-furnace.html

Introduction to steel and steel making
Workflow in an integrated steel plant

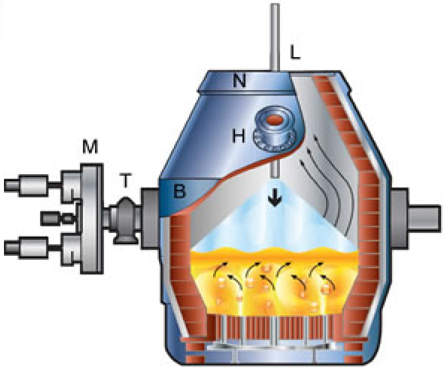


Workflow in an integrated steel plant
Primary & Secondary Metallurgy

3. Primary & Secondary Metallurgy

- Carbon reduction and impurity removal:
  - BOF converter: loaded with scrap & hot metal, addition of fluxes to form slags, a lance blows oxygen to lower concentrations of: **C, Si, P, Mn**
  - Electric Arc Furnace (EAF): loaded with scrap & direct reduced iron, addition of fluxes
- Fluxes (burnt-lime, dolomite, etc.)
  - Remove impurities by formation of slags, floating on top of the melt, slag reduces energy consumption and protects furnace lining refractories
  - Slag and steel are separated during tapping
- **Secondary metallurgy (ladle furnace refining)**
  - Goal: Deliver liquid steel to the caster…
  - On time, with right temperature, meeting total chemical specifications

O_2 Lance



Workflow in an integrated steel plant
Secondary Metallurgy

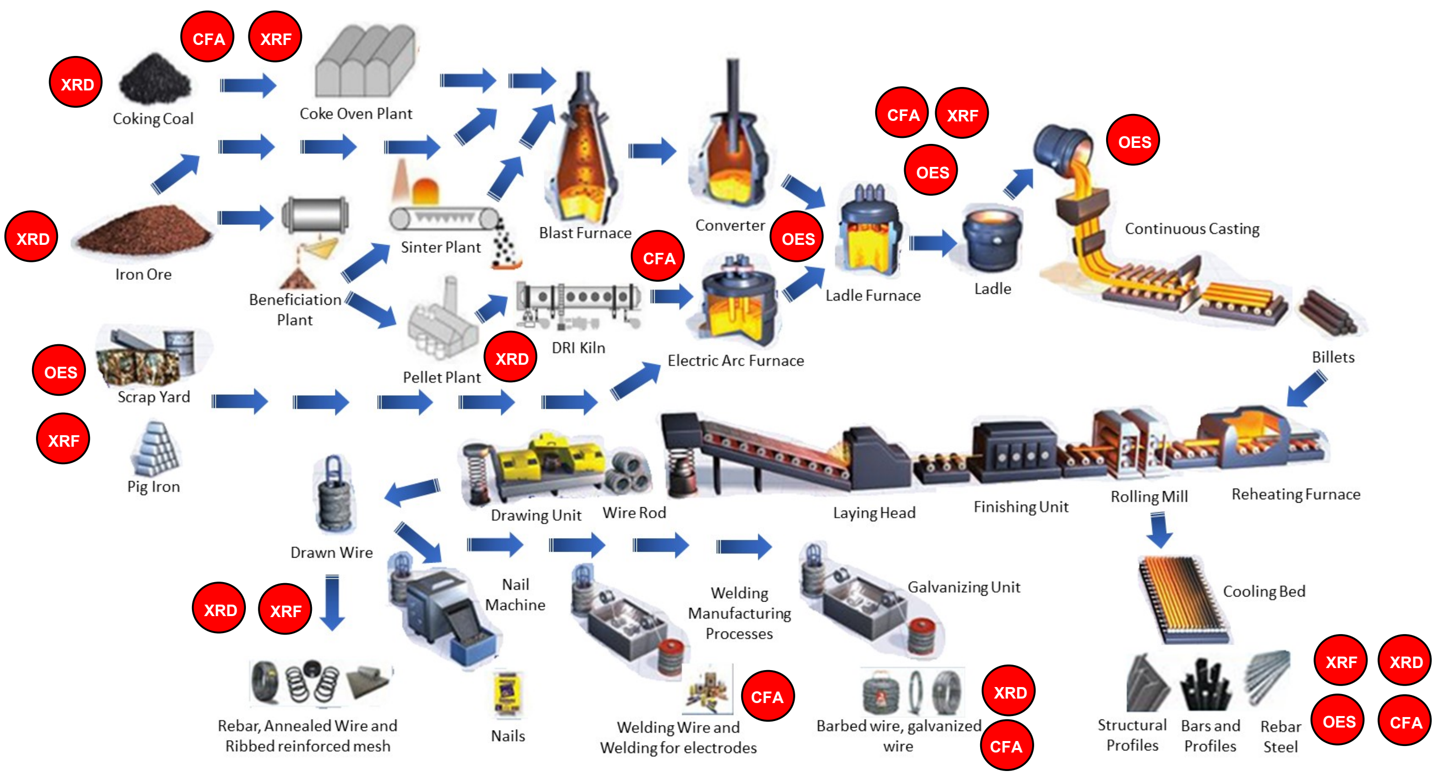
3. Secondary Metallurgy – Ladle Furnace

- Deoxidation of steel and slags (add Si, Al)
- Addition of alloying elements (Cr, Ni, Mo, V, …)
- Removal of impurities: refining (removal of S)
- Homogenization (temperature and chem. composition)
- Control of (non-metallic) inclusions
- Determines timing & productivity
- *Slag-Metal-Refractories Interactions*
- *Mass balance calculations*

Introduction to steel and steel making Analytical Techniques

- Integrated Plants (BOF), larger producers:
  typical minimum requirements: 1 XRD, 2 XRF, 3 OES, 2 CFA
- Mini-mills Plants (EAF), medium & small producers (incl. stainless steel*)
  typical minimum analytical requirements: 1 XRF, 2 OES, 2 CFA, 1 XRD*

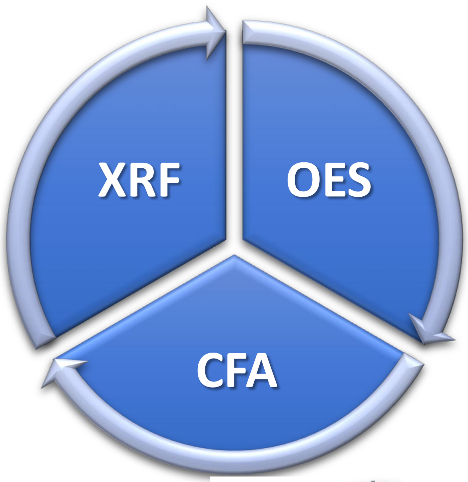
(CFA: combustion/fusion analysis)



3 Complementary Techniques

The techniques are complementary and complete each other

Metal analysis for high alloys, slag analysis



Fast and reliable, complete chemical composition in seconds

C, S, O, N, H

Bruker has a complete portfolio of techniques required by the steel and metals industry to provide a complete solution in elemental analysis

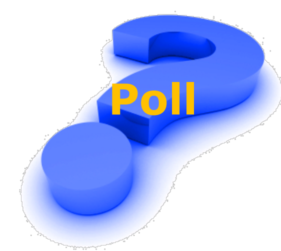
Characteristics of each technique

	OES	XRF	CFA (CS/ONH)
Element range	Wide element range	Wide element range	Limited (but some elements are not accessible by other techniques)
Concentration range	Perfect for metals: sub-ppm to high % (difficult @ high concentrations)	Very good: Sub-ppm to 100 %	Full: Sub-ppm to 100 %
Speed	Very fast (~30 s)	Medium to slow (depends on class and technique)	Fast (< 1 min)
Sample Types	Solid pieces, conductive material ⇒ metals	Any liquid or solid (no gases)	Inorganic Solids (no organics, no liquids)
Sample Forms	Solid pieces	any	any
Sample Prep. Requirements	Low - medium	Medium - high (depends on application, low compared to ICP)	Low - medium
Suitable for trace / impurities	yes / yes	yes / (yes) (depends on application and class)	Yes / yes
Statistical Representation	Medium (low ablation depth) Calibration & method matrix dependent	Low (low penetration depth in metals) Calibration matrix dependent	Complete (entire sample) Calibration matrix independent
Operation / Acquisition costs	Low / low - medium	Low / medium - high	High / low - medium

Audience Poll

Which of the following complementary analytical techniques for metals were you aware that Bruker offers? (Check all that apply.)

- ❑ Optical Emission Spectrometry (OES)
- ❑ X-ray Fluorescence (XRF)
- ❑ Combustion & Fusion Analysis for CS/ONH
- ❑ Handheld XRF (“guns”)
- ❑ X-ray Diffractometry (XRD)
- ❑ Other (e.g. NMR, FTIR/NIR, …)



Elemental Analysis for the Iron, Steel & Metal Industry

Optical Emission Spectrometry (OES)

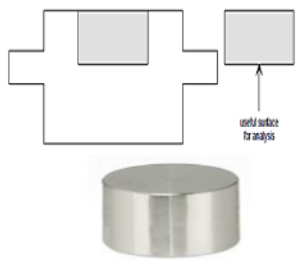
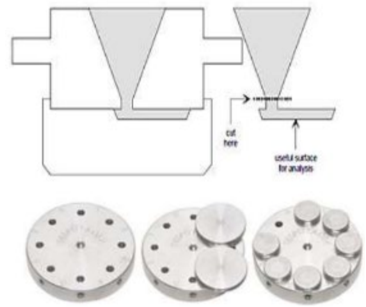
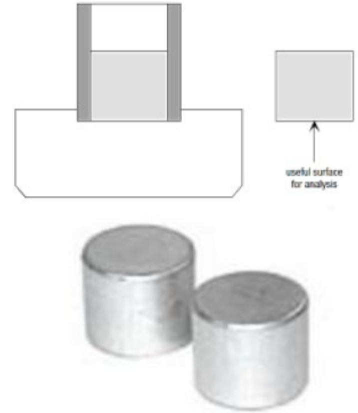
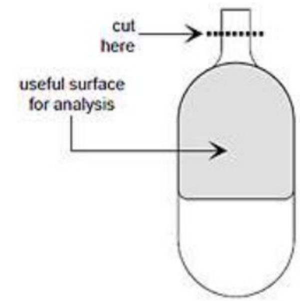
Producing samples for elemental analysis

Sample Taking → Sample Preparation → Chemical Analysis

Sample taking & sample preparation are both essential steps in the analytical process!

- Must be homogeneous and representative
- Should be free from inclusions, cavities, fissures, burrs and ridges
- If taken from melt, cooling process should be controlled to obtain samples with defined structures; rapid cooling is usually advantageous

Sample shapes in metal industry

Non-Ferrous Metals	***Metals – Fe, Ni, Co Bases***	***Cast Irons***	***Traces***	***Steel***
				
The mushroom form sample taking is the most used for non-ferrous metals.	Very simple mold, more used for alloys of Fe, Ni and Co bases.	The mold is specific adapted for general casting irons, and pig iron in particular. Very high cooling speed.	The ring mold type is recommended for casting pure metals (trace analysis) or metals that have low tendency to segregation. The sample is homogeneous on its surface, but only on some few millimeters in depth.	The sample taking can be greatly simplified with a *SPEMIS* probe that can take the liquid metal directly from the casting.

Sample shapes for steel

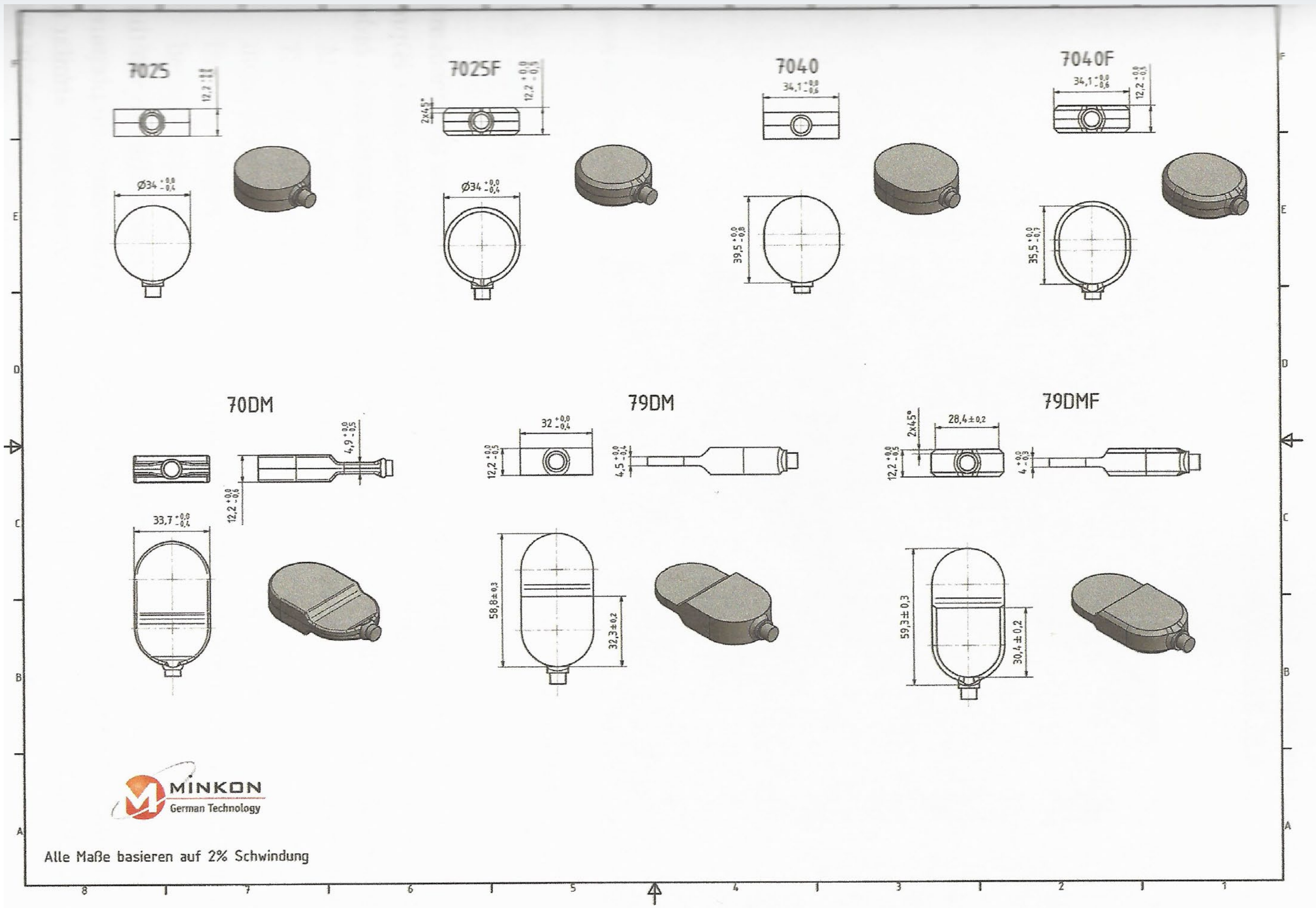


Figure 4.4: Different sample shapes (printed with friendly permission of the company MINKON GmbH, Heinrich-Hertz-Str. 30–32, D-40699 Erkrath, Germany).

Sample preparation

Requirements

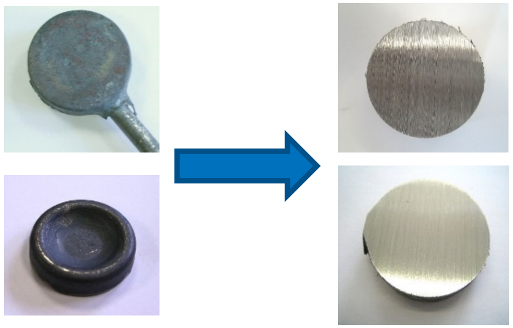
- Sample as homogenous as possible
- Clean & flat surface for reliable and reproducible measurements

Grinding (disc or belt grinder)

- For Fe, Co, Ni, Ti, Cu alloys
- Grid 40-80 of
  - corundum paper (most common & economic)
  - silicon carbide (beware of Si, C content)
  - zirconium oxide (long lifetime, more expensive)

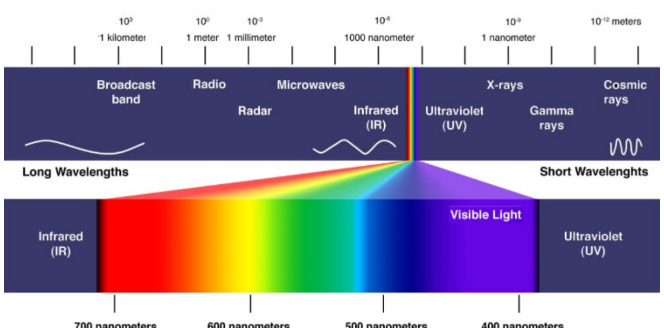
Milling

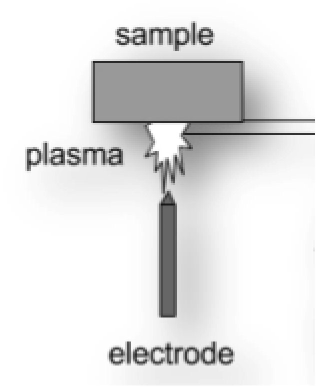
- For Al, Mg, Pb, Sn, Zn, Cu & Fe alloys
- Milling machine, clamping unit, cutting plates must suit the application (e.g. hard skin layer on cast iron)

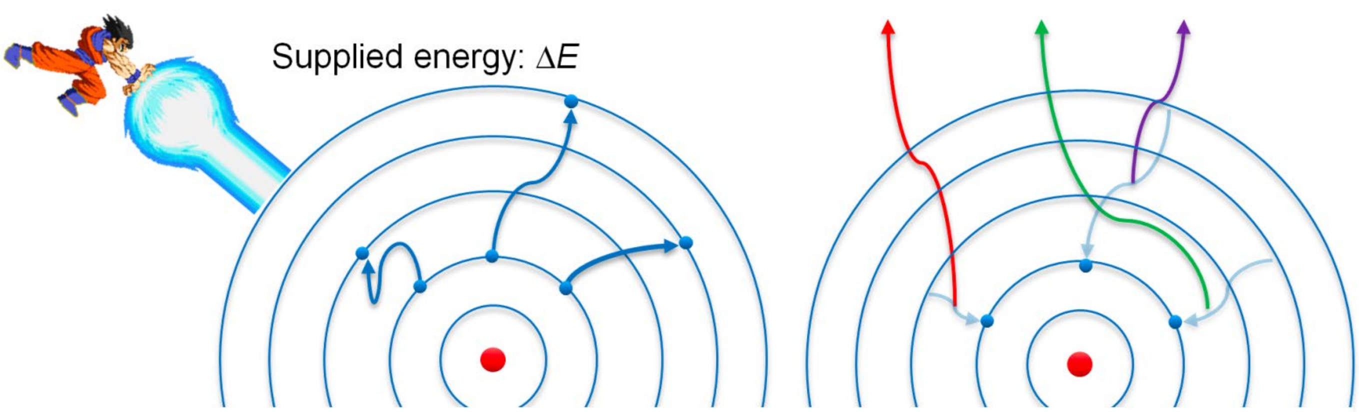


Full Alloy Analysis by OES
Physical Principles

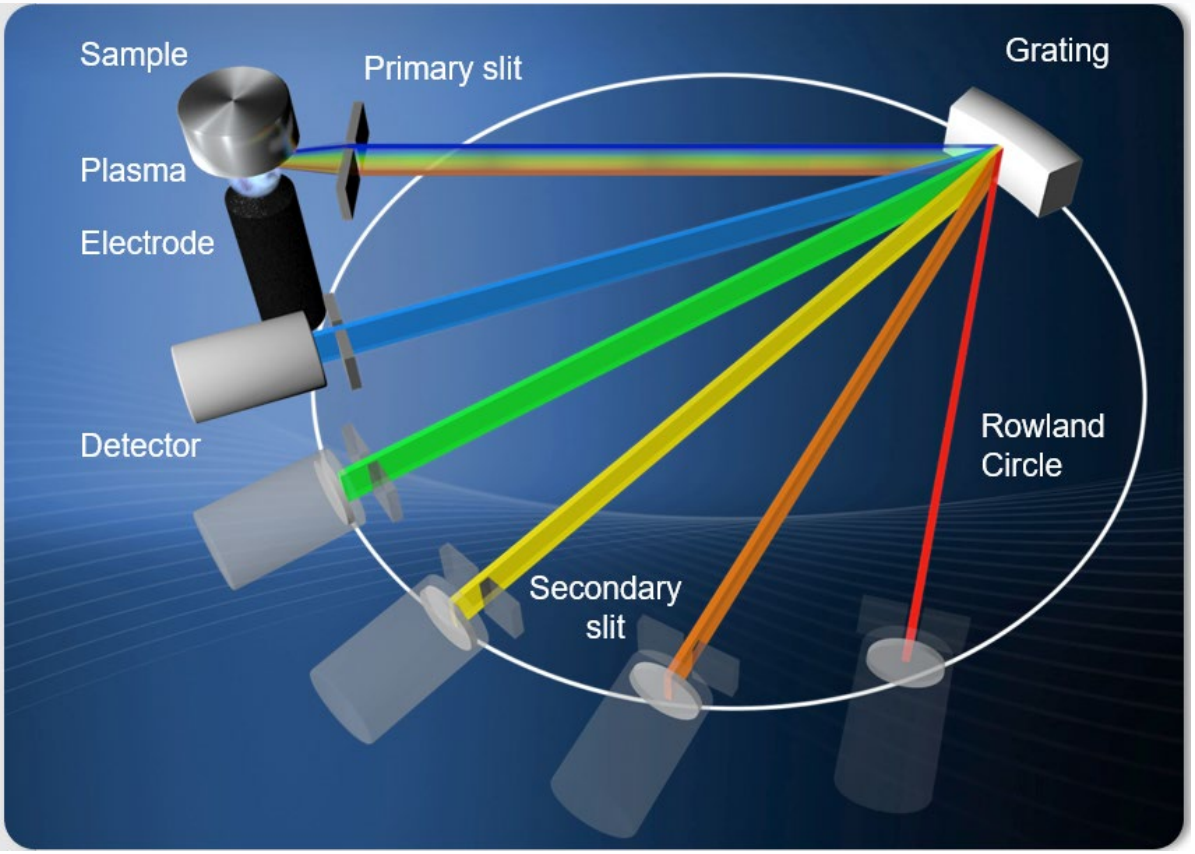
- A metallic (conductive) sample is placed above an electrode;
  a high frequency (AC), high voltage is applied between electrode and sample
  ⇒ high energetic sparks are discharged onto the sample
- The sparking happens in a pure atmosphere of argon (Ar), Ar gets ionized and a stable Ar plasma is formed
- The spark hitting the sample surface ablates/atomizes parts of the sample
- These generated atoms are not in the electronic ground state but in an excited state
- When an electron "falls" back to the ground state, a photon of specific energy (wavelength) is emitted







Full Alloy Analysis by OES
Physical Principles



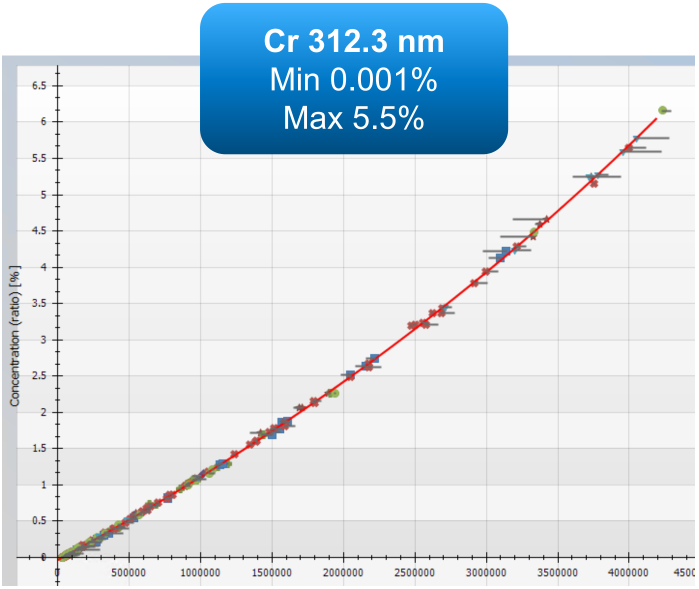
- Emitted light is guided into the optical system and dispersed by a diffraction grating.
- Detectors on specific positions on the Rowland circle are quantifying emitted light.

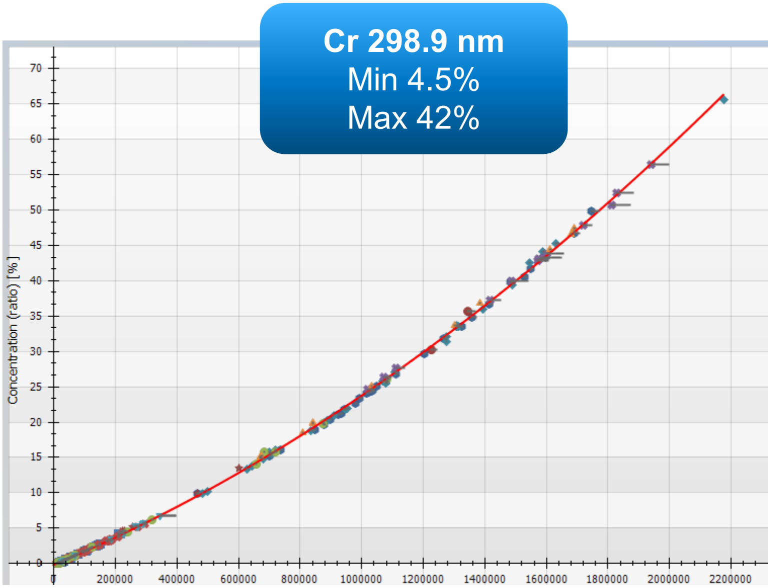
Useful wavelength in OES: 120 – 800 nm

- The intensity of a specific line correlates directly to the abundance (concentration) of the element in the sample.

Full alloy analysis by OES Calibration

- The calibration process involves determining the exact mathematical relationship (function) between the intensity (I) of the light emitted at the certain wavelength (λ) and the concentration (c) of the element producing the light.
- In fact, OES uses intensity ratios (elemental / matrix reference) to improve calibration and long term stability.
- Different emission lines with different sensitivities might be used for trace vs. bulk analysis.
- Factory calibration is built with certified reference material, but possible to include e.g. your own standards





Full Alloy Analysis by OES
Product Overview

Q2 ION

Ultra compact metals analyzer

- Patented optics with AAC for instant operation readiness
- Incoming Material Inspection, PMI and Quality Assurance
- Ultra compact, portable, affordable

Q4 TASMAN

Advanced multi-optics benchtop OES

- 3 models cover every QA/QC task
- Outstanding analytical performance with low cot of ownership
- Pneumatic sample clamp for productivity and ease-of-use

Q8 MAGELLAN

High-end PMT-based vacuum spectrometer

- Best LODs and stability
- One-button operation (automatic clamp)
- Q8 MAGELLAN *online* ready for full automation

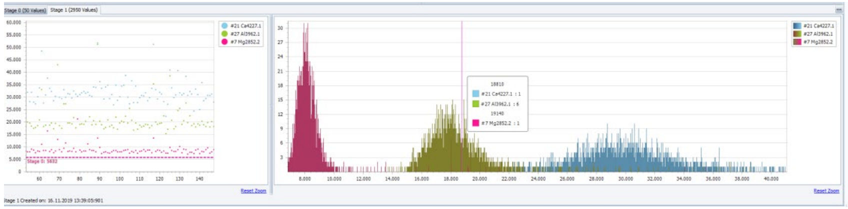
Full Alloy Analysis by OES
At a glance: Q8 MAGELLAN

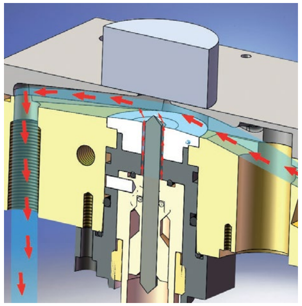
Designed for demanding applications

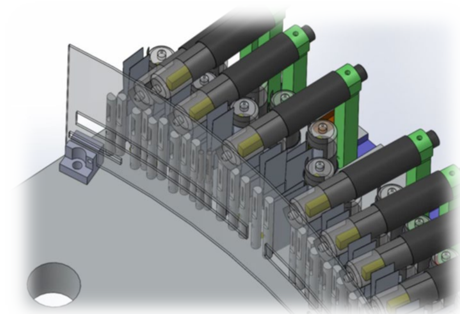
- Paschen-Runge mount, 750 mm focal lengths
- Photomultiplier (PMT) technology, up to 80 channels
- High vacuum optic (no purge)
- Single-Spark reading and evaluation (SSE)

Figures for Fe-base

- Analysis time: ~25 s
- Burn-spot size: ~4 mm (spark-stand plate opening: 12 mm)
- Penetration depth: ~18 µm
- Ablation rate: ~70 ng/spark (average)
- Total sparks for 1 "burn": 4800-6000
- Total sample mass: ~0.4 mg / burn







Full Alloy Analysis by OES
Fe 110 (low alloy steel)

ELEMENTAL.ANALYSIS - User: AppDev - Instrument: Q8S_B

File View Analysis Help

METHODS EDITOR | ANALYSIS

Flush

Sample: St 52 / 1.0580 /

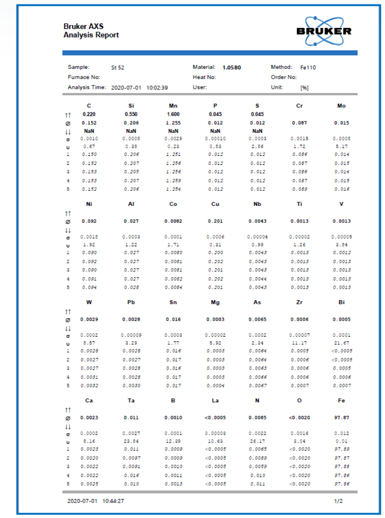
…	C [%]	Si [%]	Mn [%]	P [%]	S [%]	Cr [%]	Mo [%]	Ni [%]	Al [%]	Co [%]
2	0.152	0.207	1.256	0.012	0.012	0.087	0.015	0.092	0.027	0.0081
3	0.153	0.205	1.256	0.012	0.012	0.086	0.014	0.090	0.027	0.0081
4	0.153	0.207	1.259	0.012	0.012	0.087	0.015	0.091	0.027	0.0082
5	0.152	0.206	1.254	0.012	0.012	0.089	0.016	0.094	0.028	0.0084
↑↑	0.22	0.55	1.6	0.045	0.045					
ø	**0.152**	**0.206**	**1.255**	**0.012**	**0.012**	**0.087**	**0.015**	**0.092**	**0.027**	**0.0082**
↓↓										
σ	0.0010	0.0008	0.0029	0.00010	0.0003	0.0015	0.0008	0.0018	0.0003	0.0001
υ	0.67	0.38	0.23	0.83	2.86	1.72	5.17	1.92	1.22	1.71

…	Cu [%]	Nb [%]	Ti [%]	V [%]	W [%]	Pb [%]	Sn [%]	Mg [%]	As [%]	Zr [%]
2	0.202	0.0043	0.0013	0.0013	0.0027	0.0027	0.017	0.0003	0.0064	0.0006
3	0.201	0.0043	0.0013	0.0013	0.0027	0.0028	0.016	0.0003	0.0063	0.0006
4	0.202	0.0044	0.0013	0.0013	0.0031	0.0028	0.017	0.0003	0.0066	0.0006
5	0.201	0.0043	0.0013	0.0013	0.0032	0.0030	0.017	0.0004	0.0067	0.0007
↑↑										
ø	**0.201**	**0.0043**	**0.0013**	**0.0013**	**0.0029**	**0.0028**	**0.016**	**0.0003**	**0.0065**	**0.0006**
↓↓										
σ	0.0006	0.00004	0.00002	0.00005	0.0002	0.00009	0.0003	0.00002	0.0002	0.00007
υ	0.31	0.99	1.26	3.84	8.57	3.29	1.77	5.92	2.34	11.17

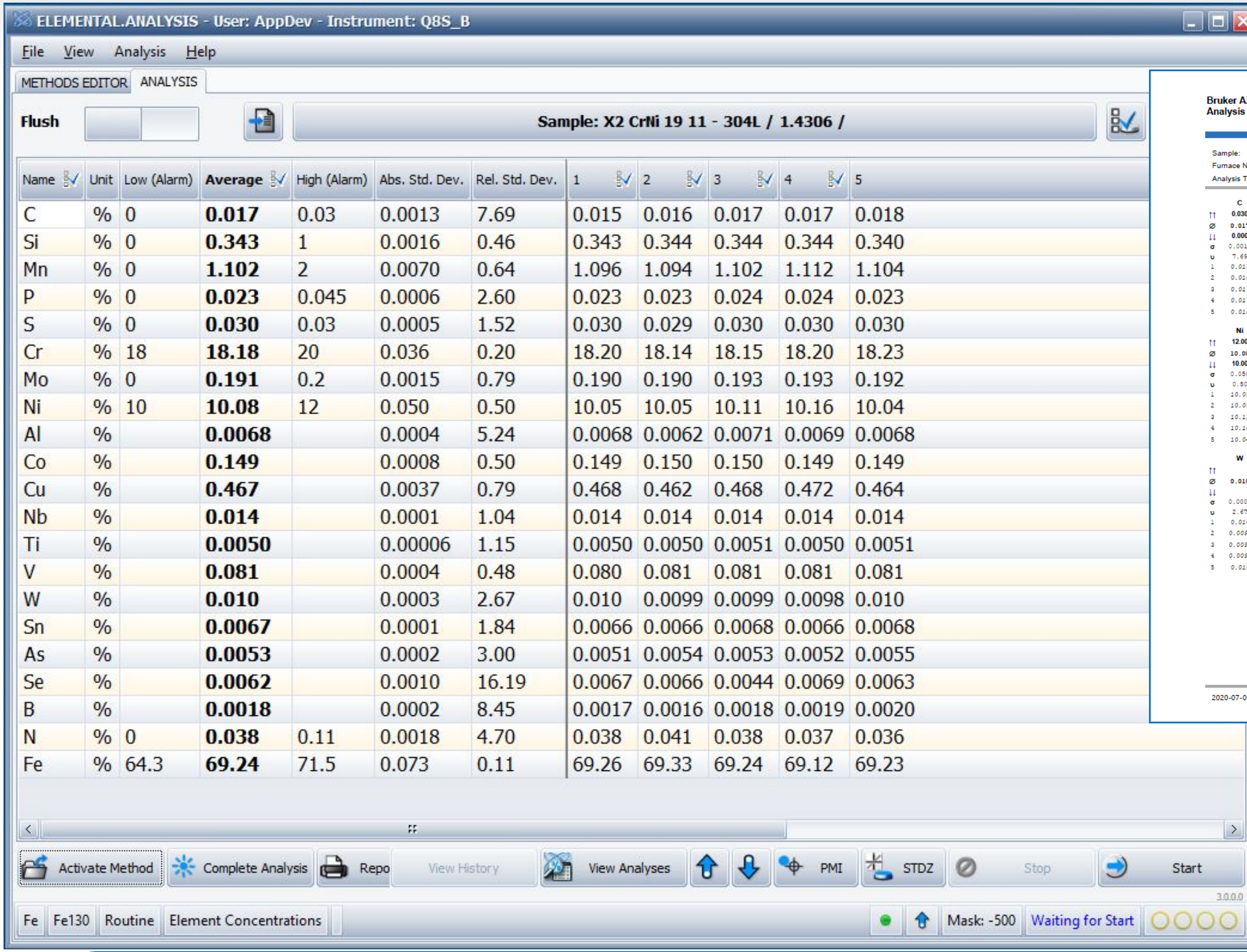
…	Bi [%]	Ca [%]	Ta [%]	B [%]	La [%]	N [%]	O [%]	Fe [%]	Ceq [%]
2	<0.0005	0.0020	0.0097	0.0009	<0.0005	0.0089	<0.0020	97.87	0.540
3	0.0005	0.0022	0.0091	0.0010	<0.0005	0.0059	<0.0020	97.88	0.538
4	0.0006	0.0022	0.016	0.0011	<0.0005	0.010	<0.0020	97.86	0.541
5	0.0007	0.0025	0.010	0.0013	<0.0005	0.011	<0.0020	97.86	0.545
↑↑									
ø	**0.0005**	**0.0023**	**0.011**	**0.0010**	**<0.0005**	**0.0085**	**<0.0020**	**97.87**	**0.540**
↓↓									
σ	0.0001	0.0002	0.0027	0.0001	0.00003	0.0022	0.0016	0.012	0.0039
υ	21.67	8.16	23.84	12.39	10.63	26.17	3.04	0.01	0.72

Activate Method | Complete Analysis | Report Analysis | Reject Analysis | View History | View Analyses | PMI | STDZ | Stop | Start

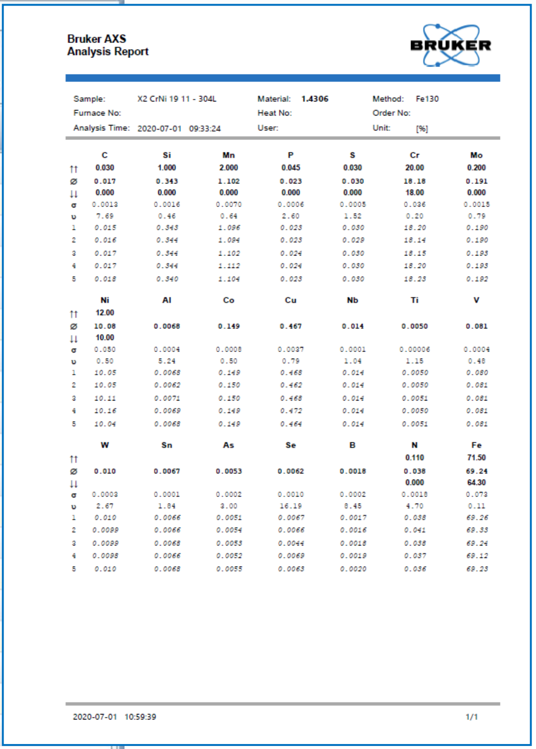
Fe | Fe110 | Routine | Element Concentrations | Mask: -500 | Waiting for Start



Full Alloy Analysis by OES
Fe 130 (Cr/CrNi)



Name	Unit	Low (Alarm)	Average	High (Alarm)	Abs. Std. Dev.	Rel. Std. Dev.	1	2	3	4	5
C	%	0	**0.017**	0.03	0.0013	7.69	0.015	0.016	0.017	0.017	0.018
Si	%	0	**0.343**	1	0.0016	0.46	0.343	0.344	0.344	0.344	0.340
Mn	%	0	**1.102**	2	0.0070	0.64	1.096	1.094	1.102	1.112	1.104
P	%	0	**0.023**	0.045	0.0006	2.60	0.023	0.023	0.024	0.024	0.023
S	%	0	**0.030**	0.03	0.0005	1.52	0.030	0.029	0.030	0.030	0.030
Cr	%	18	**18.18**	20	0.036	0.20	18.20	18.14	18.15	18.20	18.23
Mo	%	0	**0.191**	0.2	0.0015	0.79	0.190	0.190	0.193	0.193	0.192
Ni	%	10	**10.08**	12	0.050	0.50	10.05	10.05	10.11	10.16	10.04
Al	%		**0.0068**		0.0004	5.24	0.0068	0.0062	0.0071	0.0069	0.0068
Co	%		**0.149**		0.0008	0.50	0.149	0.150	0.150	0.149	0.149
Cu	%		**0.467**		0.0037	0.79	0.468	0.462	0.468	0.472	0.464
Nb	%		**0.014**		0.0001	1.04	0.014	0.014	0.014	0.014	0.014
Ti	%		**0.0050**		0.00006	1.15	0.0050	0.0050	0.0051	0.0050	0.0051
V	%		**0.081**		0.0004	0.48	0.080	0.081	0.081	0.081	0.081
W	%		**0.010**		0.0003	2.67	0.010	0.0099	0.0099	0.0098	0.010
Sn	%		**0.0067**		0.0001	1.84	0.0066	0.0066	0.0068	0.0066	0.0068
As	%		**0.0053**		0.0002	3.00	0.0051	0.0054	0.0053	0.0052	0.0055
Se	%		**0.0062**		0.0010	16.19	0.0067	0.0066	0.0044	0.0069	0.0063
B	%		**0.0018**		0.0002	8.45	0.0017	0.0016	0.0018	0.0019	0.0020
N	%	0	**0.038**	0.11	0.0018	4.70	0.038	0.041	0.038	0.037	0.036
Fe	%	64.3	**69.24**	71.5	0.073	0.11	69.26	69.33	69.24	69.12	69.23



Bruker AXS
Analysis Report

Sample: X2 CrNi 19 11 - 304L Material: **1.4306** Method: Fe130
Furnace No: Heat No: Order No:
Analysis Time: 2020-07-01 09:33:24 User: Unit: [%]

	C	Si	Mn	P	S	Cr	Mo
↑↑	0.030	1.000	2.000	0.045	0.030	20.00	0.200
⌀	0.017	0.343	1.102	0.023	0.030	18.18	0.191
↓↓	0.000	0.000	0.000	0.000	0.000	18.00	0.000
σ	0.0013	0.0016	0.0070	0.0006	0.0005	0.036	0.0015
υ	7.69	0.46	0.64	2.60	1.52	0.20	0.79
1	0.015	0.343	1.096	0.023	0.030	18.20	0.190
2	0.016	0.344	1.094	0.023	0.029	18.14	0.190
3	0.017	0.344	1.102	0.024	0.030	18.15	0.193
4	0.017	0.344	1.112	0.024	0.030	18.20	0.193
5	0.018	0.340	1.104	0.023	0.030	18.23	0.192

	Ni	Al	Co	Cu	Nb	Ti	V
↑↑	12.00						
⌀	10.08	0.0068	0.149	0.467	0.014	0.0050	0.081
↓↓	10.00						
σ	0.050	0.0004	0.0008	0.0037	0.0001	0.00006	0.0004
υ	0.50	5.24	0.50	0.79	1.04	1.15	0.48
1	10.05	0.0068	0.149	0.468	0.014	0.0050	0.080
2	10.05	0.0062	0.150	0.462	0.014	0.0050	0.081
3	10.11	0.0071	0.150	0.468	0.014	0.0051	0.081
4	10.16	0.0069	0.149	0.472	0.014	0.0050	0.081
5	10.04	0.0068	0.149	0.464	0.014	0.0051	0.081

	W	Sn	As	Se	B	N	Fe
↑↑						0.110	71.50
⌀	0.010	0.0067	0.0053	0.0062	0.0018	0.038	69.24
↓↓						0.000	64.30
σ	0.0003	0.0001	0.0002	0.0010	0.0002	0.0018	0.073
υ	2.67	1.84	3.00	16.19	8.45	4.70	0.11
1	0.010	0.0066	0.0051	0.0067	0.0017	0.038	69.26
2	0.0099	0.0066	0.0054	0.0066	0.0016	0.041	69.33
3	0.0099	0.0068	0.0053	0.0044	0.0018	0.038	69.24
4	0.0098	0.0066	0.0052	0.0069	0.0019	0.037	69.12
5	0.010	0.0068	0.0055	0.0063	0.0020	0.036	69.23

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Elemental Analysis for the Iron, Steel & Metal Industry

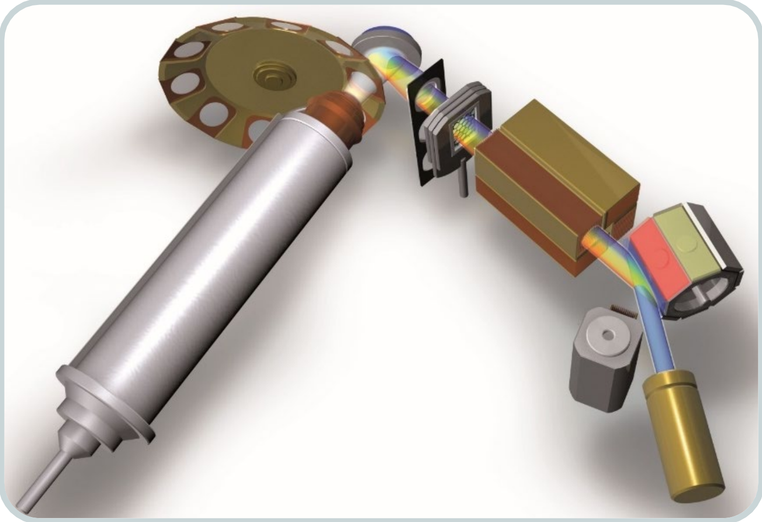
X-ray Fluorescence (XRF)

Audience Poll

What are your main goals with elemental analysis – What are you trying to achieve? (Check all that apply.)

- ❑ Check quality of incoming material
- ❑ Increase percentage of recycled material in production
- ❑ Optimize production costs
- ❑ Increase throughput
- ❑ Optimize final product quality
- ❑ Other (specify)

EDXRF or WDXRF?
Depends on the requirements!



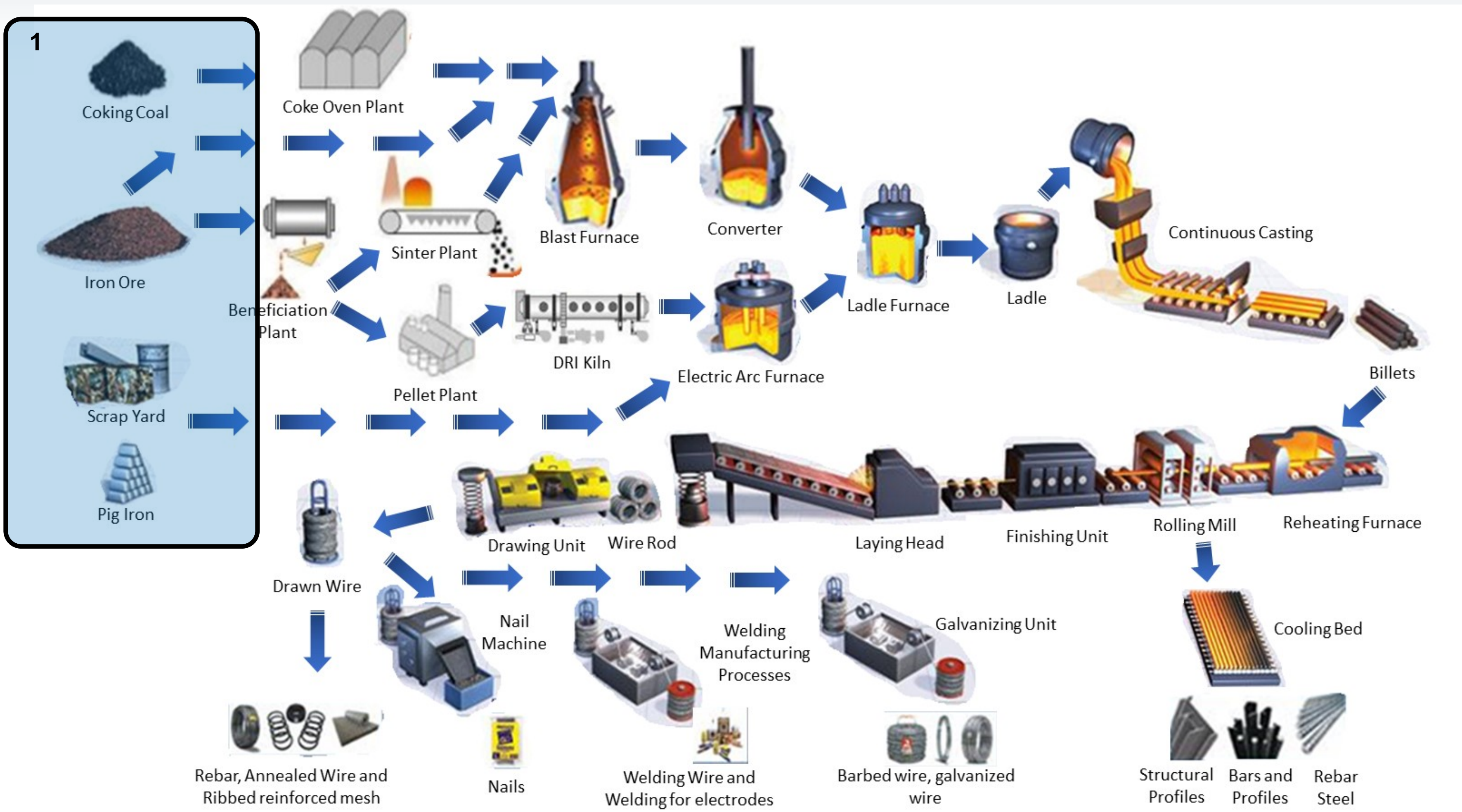
WDXRF

- Sophisticated setup
- Best resolution and sensitivity
- Detects intensity at a given wavelength

EDXRF

- Simple, robust, compact
- Detects energy and intensity

XRF for Raw Materials



S2 KODIAK – Online-Analysis of Scrap Sorting and Certification

- Steel producers aim toward **circular economy**
  - Increase the use of recycled steel to reduce CO_2 emission
- **Tramp elements** (Cu, Cr, Ni, Mo…) need to be monitored
  - The thresholds depend on the production process
    - Blast furnace / converter
    - Electric are furnace
- **Online elemental analysis** to
  - Enhance the sorting procedure
  - Determine the average composition of a truck load
- **Integration**: Sicon Germany/America

time	t/h	Fe %	Cu %	Mn %	Mo %	Ti %	Zn %
6 AM	72.20	97.14	0.406	0.301	0.004	0.004	0.045
7 AM	79.41	97.11	0.432	0.292	0.004	0.004	0.053
8 AM	62.45	97.06	0.499	0.295	0.004	0.004	0.049
9 AM	71.71	97.06	0.478	0.300	0.004	0.004	0.052
…	…	…	…	…	…	…	…
Sum	652.0	--	--	--	--	--	--
AVG	--	97.10	0.46	0.30	0.01	0.01	0.05

Exemplary report

S2 KODIAK
The perfect fit for online applications

Online EDXRF multi-element analyzer for real-time results for Mining, **Material Sorting / Recycling**, Coatings and more.

- Encapsulated, ruggedized design; *tested*:
  - Shock & Vibration
  - Temperature
  - Water spray and Condensing water

- Enhanced system cooling
- Automatic system startup and self alignment
- Autonomous operation 24/7
- Integrated UPS, camera, distance sensor
- TCP/IP data transfer to plant control SW
- Maintenance-free operation
- No radioactive sources

S8 TIGER for Incoming Material Inspection Iron Ore/Sinter

- According to ISO 9516 (WDXRF)
- Fused beads
  - At commercial and service labs
  - At central labs
  - At customs
- Maximum 60 s measurement time
- Excellent long-term precision for 200 measurements

Sample	Average (%)	Abs. Std. Dev. (%)	Rel. Std. Dev. (%)
Fe (%)	**47.065**	0.013	0.028
SiO_2 (%)	**0.673**	0.006	0.869
P (%)	**0.031**	0.001	0.746
Al_2O_3 (%)	**4.081**	0.014	0.334
Mn (%)	**15.348**	0.016	0.102
CaO (%)	**0.032**	0.001	1.867
MgO (%)	**0.032**	0.002	5.401
TiO_2 (%)	**0.023**	0.003	11.705
K_2O (%)	**0.160**	0.001	0.484

GEO-QUANT Iron Ore Solution

- **GEO-QUANT Iron Ore contains**
  - 16 certified reference materials (no synthetic standards)
  - Evaluation samples
  - User Manual
  - Preparation Manual

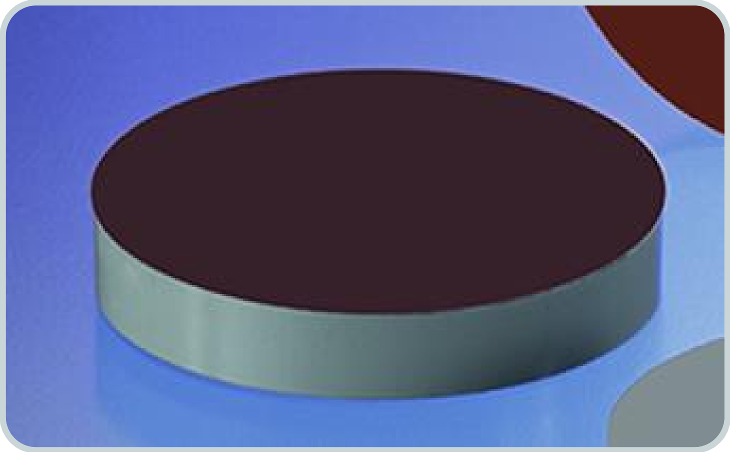
- **GEO-QUANT Iron Ore requires**
  - LIF 220 crystal
  - XS 100 crystal
    - Covers F to Cl
    - Increased sensitivity compared to the XS-CEM
    - Enhanced long term stability compared to the PET
    - Not temperature sensitive
    - **Shorter measurement time for elements from F to Cl**

S6 JAGUAR - Benchtop WDXRF
Coal and Coke

Coal is widely used in metal making (aluminum, steel) and power generation (electricity)

Analysis of coal, coke and carbon products is vital:

- Prevent contamination of metal products
- Inhibit (steel) corrosion (monitoring of Cl)
- Reduce environmental impact (reducing S content)

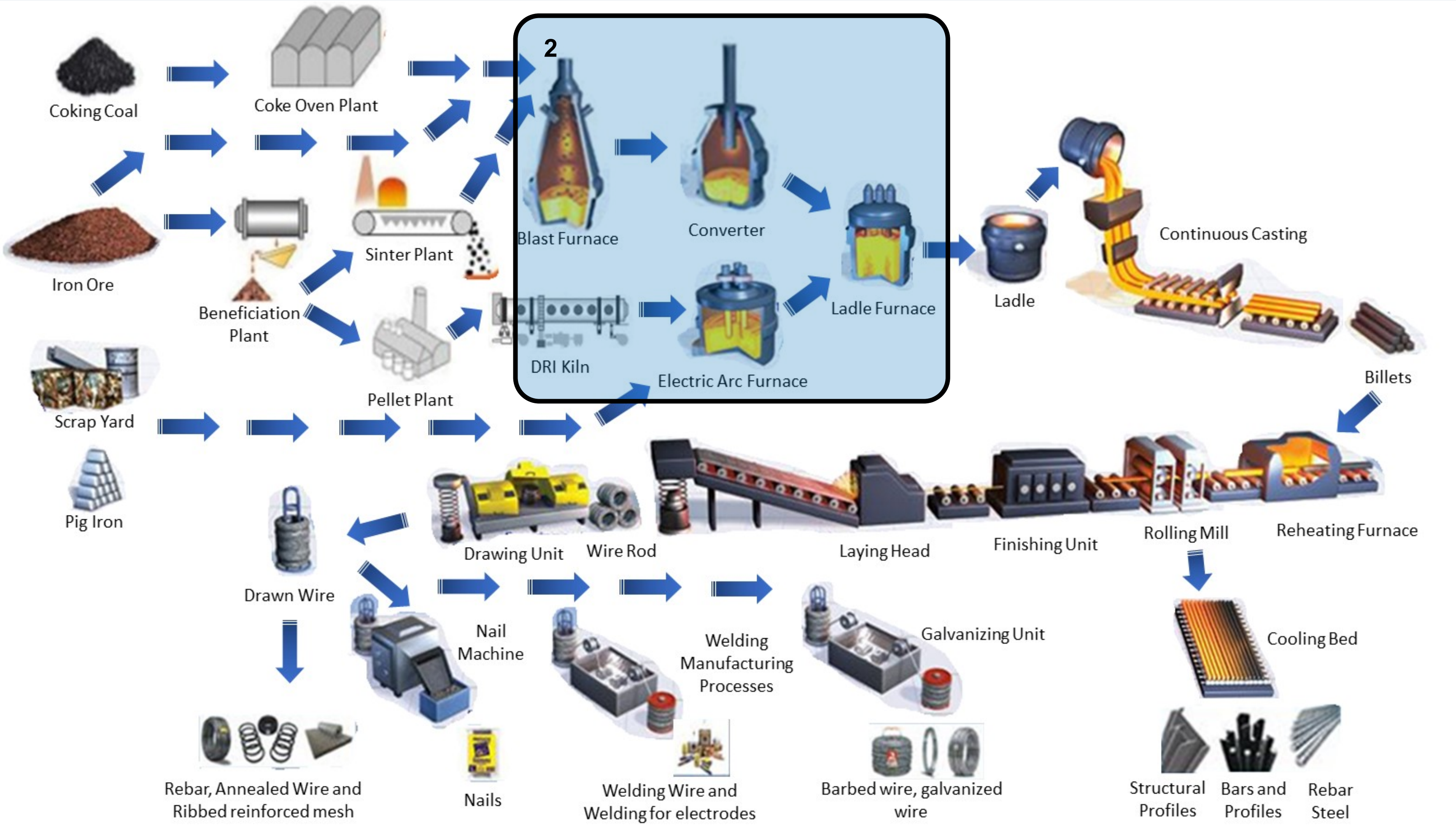


S6 JAGUAR - Benchtop WDXRF
Coal and Coke

Element	XRF conc.	Cert. Conc.	Abs Std Dev.	Rel Std Dev.
S {%]	3.40	3.30	0.02	0.72
Ni [ppm]	128.00	124.00	4.48	3.50
Si [ppm]	24.00	28.00	1.15	4.80
Fe [ppm]	276.00	266.00	2.65	0.96
Na [ppm]	623.00	645.00	43.61	7.00
Al [ppm]	153.00	150.00	5.66	3.70
Ca [ppm]	112.00	107.00	2.35	2.10
K [ppm]	17.00	17.00	1.84	10.80
Cl [ppm]	100.00	n.a.		
Ti [ppm]	5.00	4.00	0.31	6.10
Zn [ppm]	40.00	41.00	0.10	0.25
V [ppm]	302.00	300.00	2.33	0.77

Optimal accuracy for:
- Minor elements, such as S
- Traces, e.g. Si, Cl, Fe

XRF for Production Monitoring



S2 PUMA Series 2
Slag analysis made fast and reliable

Slag composition needs to be monitored to achieve e.g. optimal metal purity and least attack on refractory lining.

There are different types for slags in steel production, e.g.:

- Electric Arc Furnace (EAF) Slag
- Ladle Metallurgy Furnace (LMF) Slag

The difference in steel production procedure results in different slag compositions and, thus, in different requirements for analytical solutions.

Reliable results are required within minutes!

S2 PUMA Series 2
Slag analysis made fast and reliable

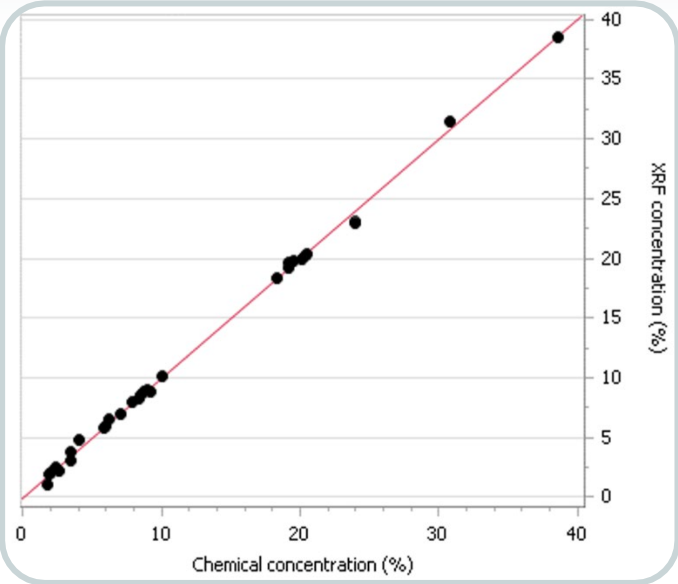
Optimal excitation of the sample is ensured by:

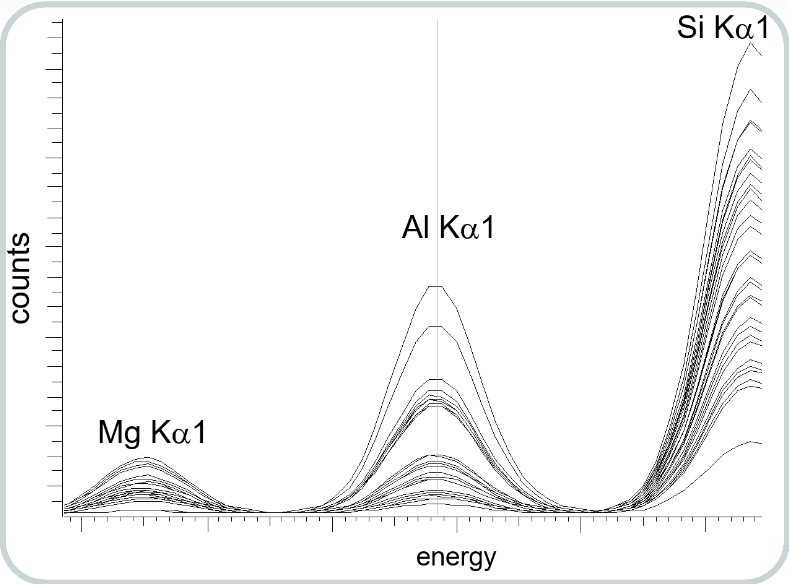
- High power 50-Watt X-ray tube
- Closely coupled optics
- 10-position primary beam filter
- The Next generation silicon drift detectors (SDD)

S2 PUMA Series 2 Single

- Convenient and fail-safe sample handling
  - Load the sample, touch the button and get results within minutes!
- Ease-of-use thanks to TouchControl™
- Sample Rotation for heterogeneous samples
- SampleCare™ protects vital system components
- Dedicated Light Element configuration (e.g. for F)
  - Optional vacuum mode for best light element performance and lowest cost of operation (no He)

S2 PUMA Series 2
Slag analysis made fast and reliable





- Calibration curve for MgO
- Calibration peaks for Mg Kα1, Al Kα1 and Si Kα1

S2 PUMA Series 2
Slag analysis made fast and reliable

- Equipped with the HighSense™ LE detector, the S2 PUMA Series 2 achieves outstanding performance – even for light elements and with a single analytical range – in just **2 minutes** counting time!

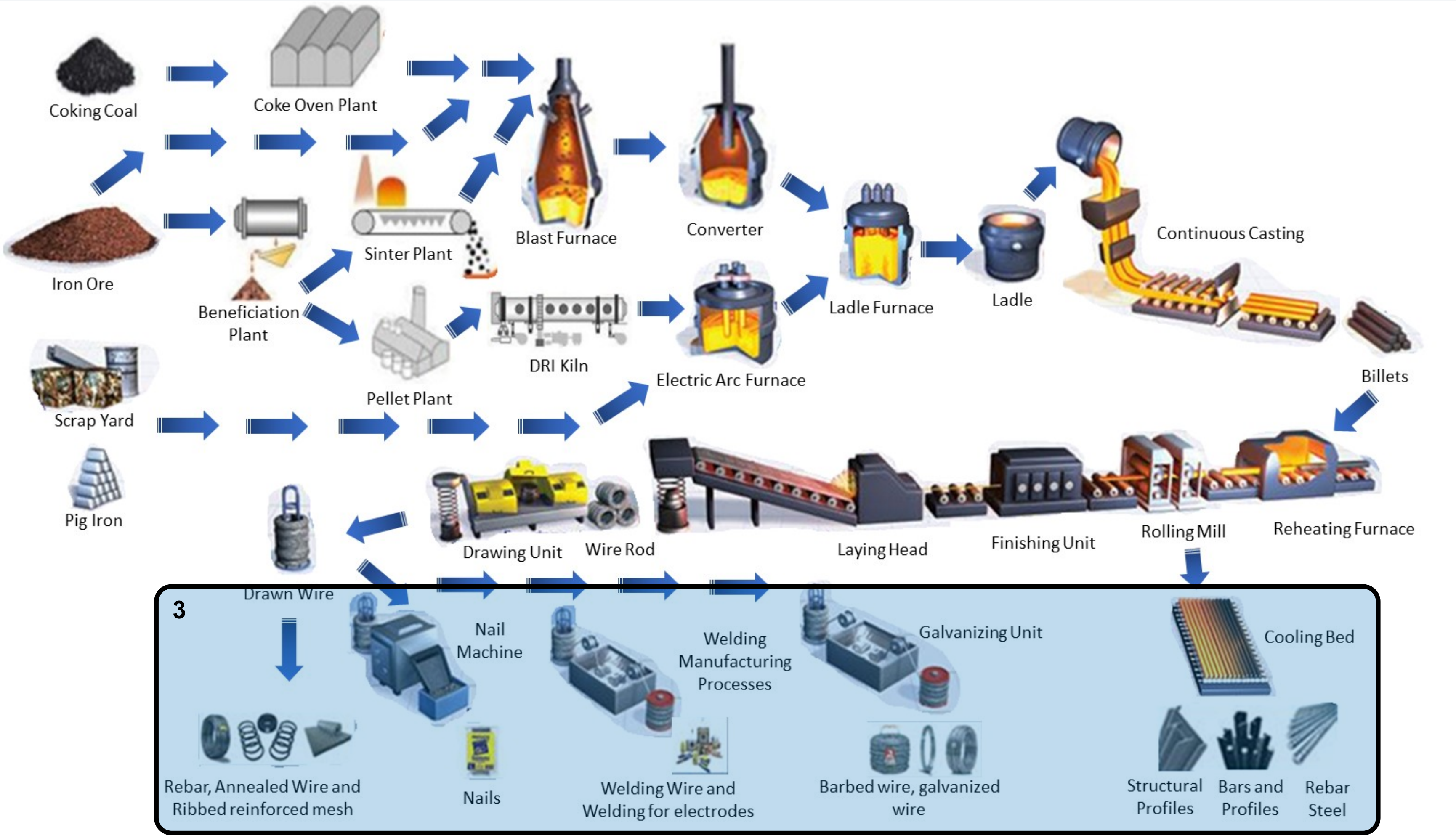
- Precision is typically < 1 % RSD

Compound	LMF Slags [wt%]
F	0.03 – 7.9
MgO	0.2 – 21.2
Al_2O_3	1.8 – 38.6
SiO_2	7.4 – 51.4
P_2O_5	0 – 1.6
S	0 – 1.2
CaO	0.6 – 60.4
TiO_2	0.01 – 2.2
MnO	0.06 – 14.9
FeO	0.1 – 17.2

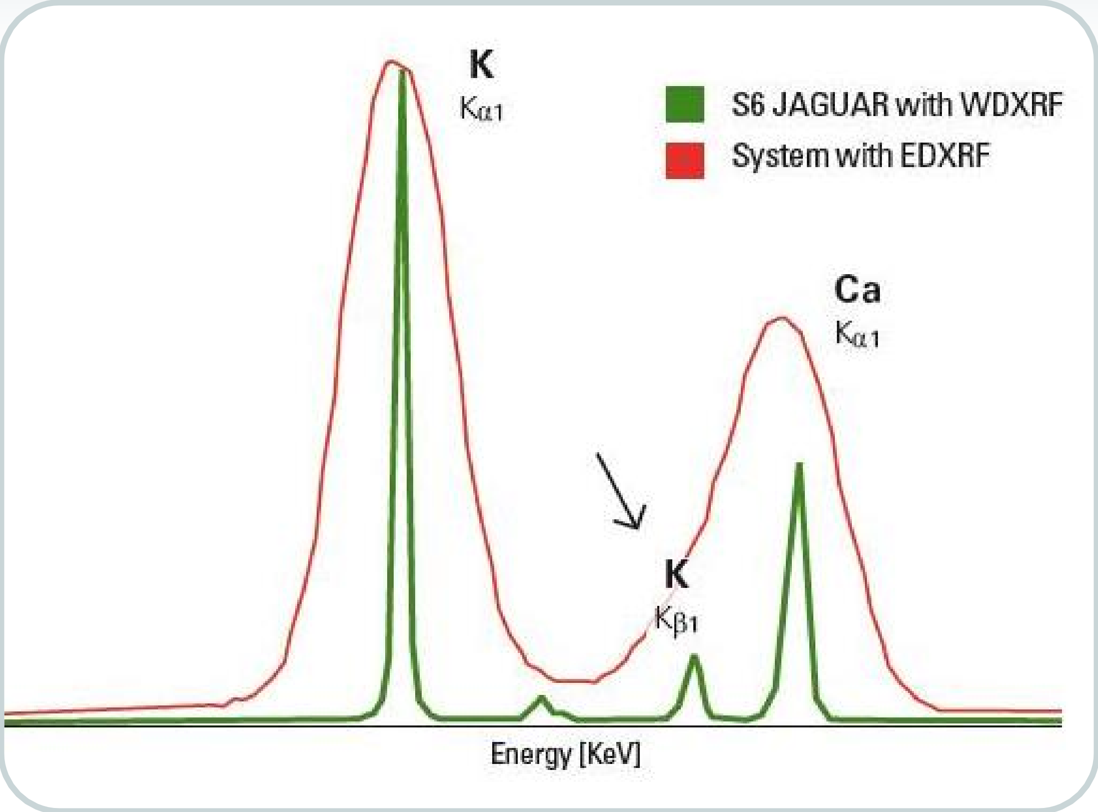
Elements	Voltage [kV]	Current [mA]	Measurement time [s]	Beam Filter	Mode
F, Mg, Al, Si, P, S, Ca, Ti, Mn, Fe	20	automatic*	120	none	Vacuum

*Current is maximized automatically for best count statistic.

XRF for Quality Control of Steel Products

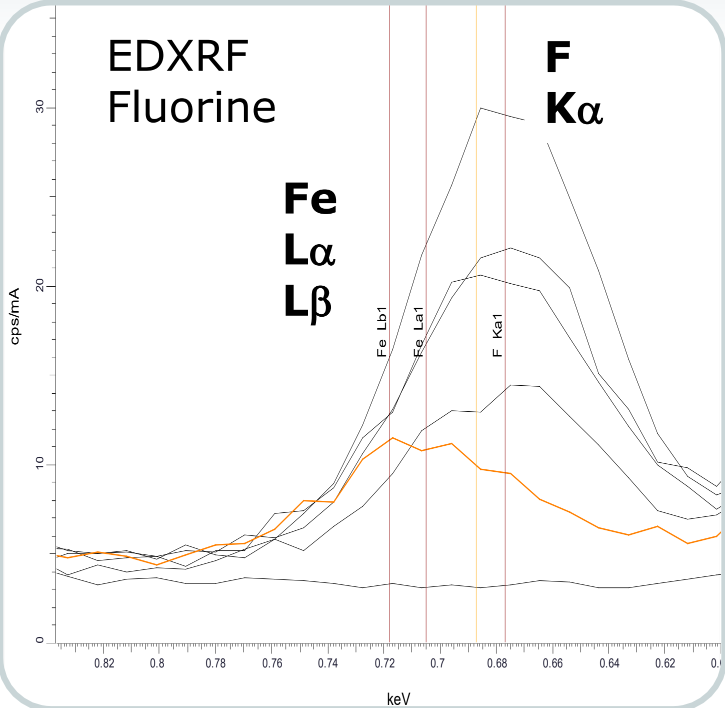


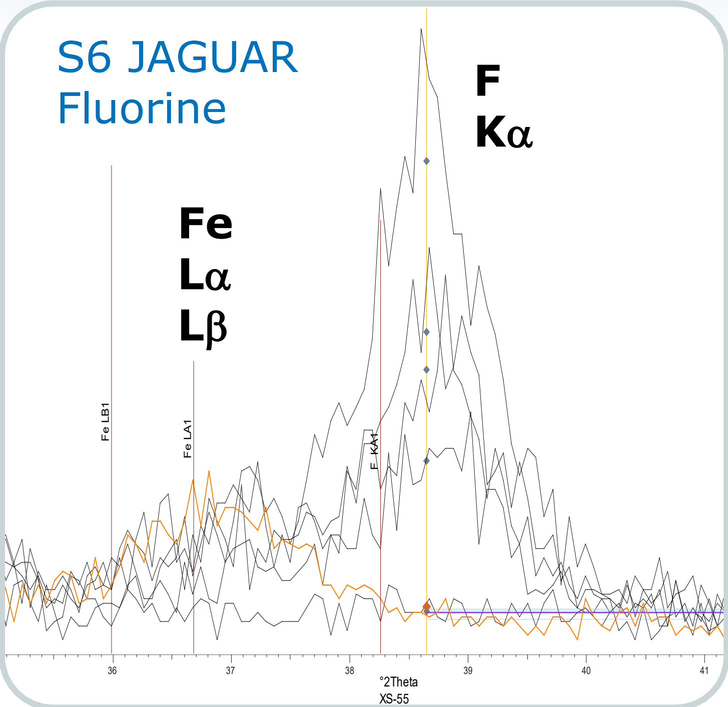
S6 JAGUAR
HighSense™ Goniometer: High Resolution



The S6 JAGUAR with WDXRF HighSense Goniometer exceeds EDXRF-based systems in resolution and analytical precision

S6 JAGUAR versus EDXRF
F in Foundry Products





Strong overlap of F Ka and Fe La with **EDXRF** leads to medium accuracy and precision: **Min 3.59 % –> 3.78 % <- Max 4.07 %**

S6 JAGUAR: Optimal resolution, clear separation of both lines, high sensitivity with 400 W power: **Min 3.97 % -> 4.03 % <- Max 4.07 %**

S6 JAGUAR
Low Alloy Steels

Quality Control of low alloy and mild steels:

Analysis of Fe, Ti, V, Cr, Mn, Co, Ni, Cu, Mo, W

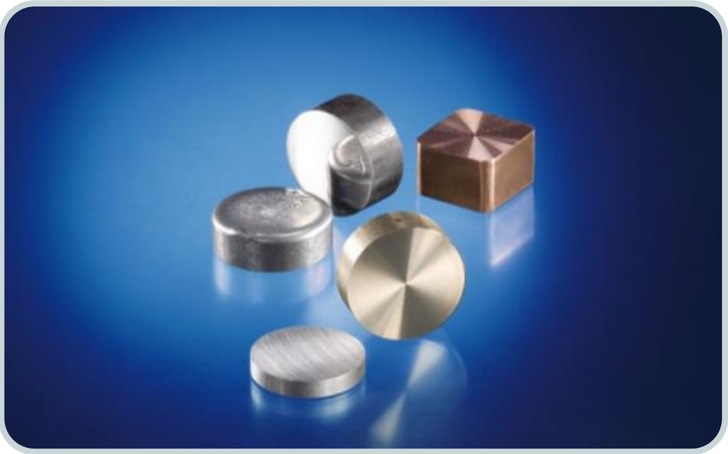
Impurities with negative impact on steel quality:

Al, Si, P, S, Cu, As, Sb, Pb

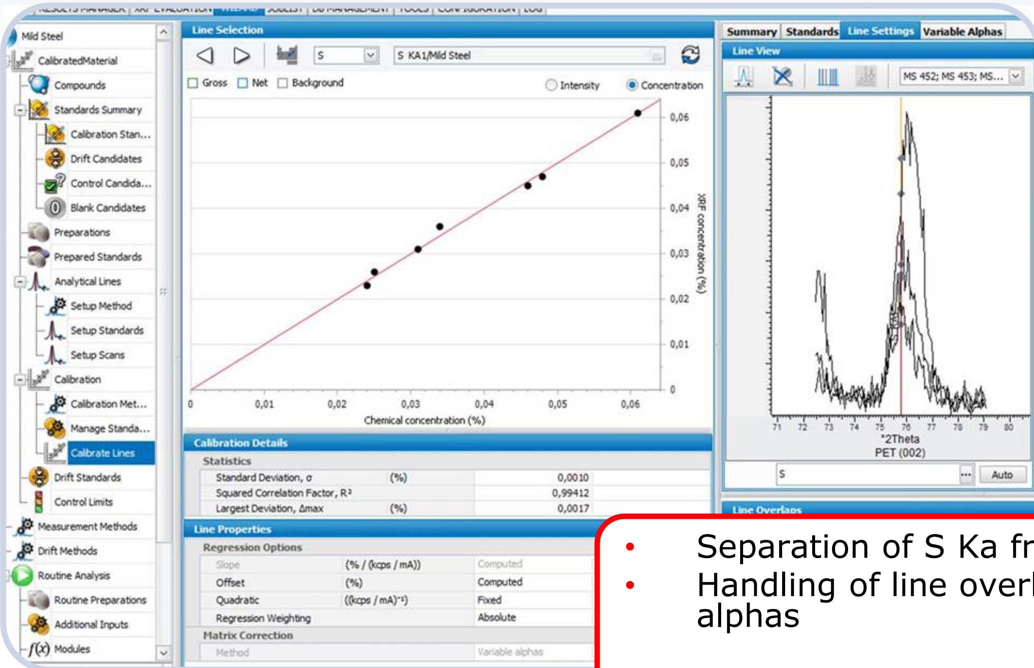
- Incoming inspection of raw material for manufacturing
- Specifying alloy types

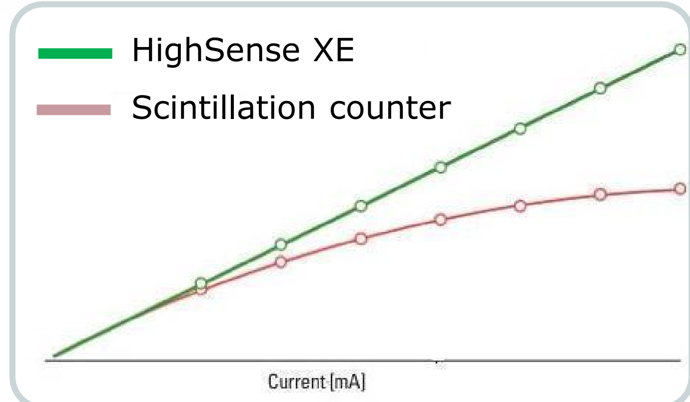
Current WDXRF spectrometers are suffering for major elements due to saturation of the scintillation counter

Sum, escape peaks and crystal artefacts will even worsen the situation (Pulse Height Distribution)



S6 JAGUAR
Low Alloy Steels





- Separation of S Ka from Mo La with high resolution WDXRF
- Handling of line overlaps and FP matrix correction with variable alphas

New solid state HighSense XE detector replaces conventional scintillation detector:

- State-of-the-art technology
- Medium and heavy element detection
- Linear range of 2 Mcps
- 2 times better energy resolution

S8 TIGER for highest throughput
Cast iron

Process Control (CAST IRON) with the S8 TIGER, 4 kW:

15 elements:

C, Si, P, S, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Sn, Pb

Time-to-result:

1 min 47 sec. per sample

Sample throughput:

30 samples/h

Fast, accurate and precise process and quality control of cast iron, including carbon analysis

HIGHEST SPEED ANALYSIS

S8 TIGER for highest throughput
Cast iron

Sample	Si (%)	Mn (%)	P (%)	S (%)	V (%)	Ti (%)	C (%)
cast	1.488	3.08	0.321	0.175	0.036	0.265	3.74
cast/1	1.488	3.08	0.320	0.175	0.035	0.264	3.78
cast/2	1.488	3.08	0.323	0.174	0.036	0.265	3.78
cast/3	1.488	3.08	0.323	0.175	0.035	0.266	3.78
cast/4	1.491	3.08	0.324	0.175	0.035	0.265	3.79
cast/5	1.483	3.08	0.323	0.175	0.035	0.265	3.80
cast/6	1.487	3.08	0.323	0.174	0.036	0.264	3.80
cast/7	1.490	3.08	0.323	0.175	0.036	0.266	3.80
cast/8	1.490	3.08	0.323	0.175	0.036	0.264	3.82
cast/9	1.490	3.08	0.323	0.175	0.036	0.264	3.83
cast/10	1.490	3.08	0.322	0.175	0.036	0.264	3.82
Mean	**1.488**	**3.08**	**0.323**	**0.175**	**0.036**	**0.265**	**3.79**
Std. Dev.	0.002	0.001	0.001	0.0002	0.001	0.001	0.026
RSD	0.16	0.04	0.36	0.24	1.45	0.31	0.69

Elemental Analysis for the Iron, Steel & Metal Industry

Combustion/Fusion Analysis

Audience Poll

Prior to this webinar, were you aware that Bruker offers C/S and O/N/H analyzers?

- Yes
- Somewhat
- Not really
- No

Topics

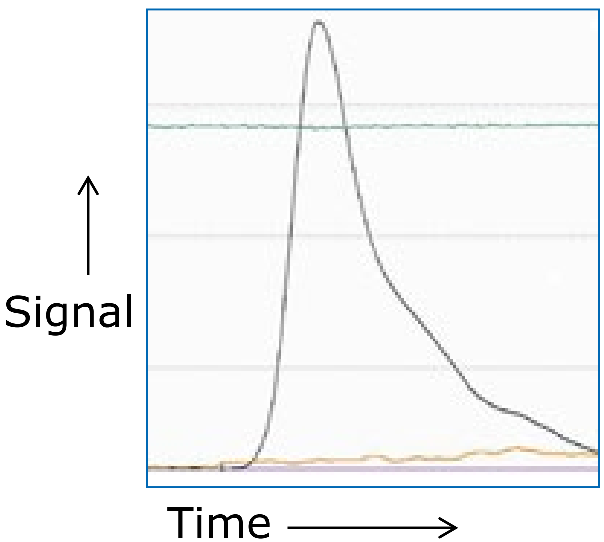
- Introduction to the term Elemental Analysis
  - C/S & O/N/H for *inorganic* materials
    - Basic Principals, Definitions, and Benefits
  - Overview of BAXS CGA Product Line
    - C/S (by HF-Induction, NDIR-Detection)

      ➡ G4 ICARUS HF
    - O/N/H (by Inert Gas Fusion)

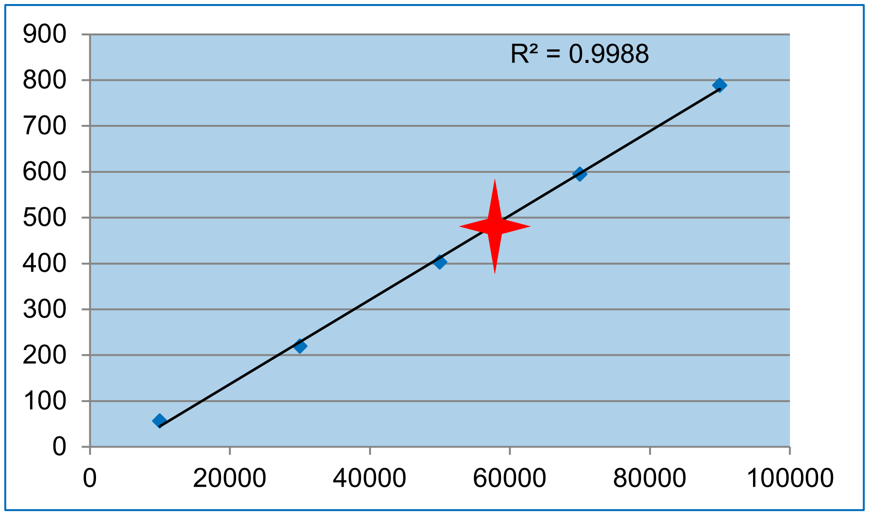
      ➡ G6 LEONARDO, G8 GALILEO

Elemental Analysis Definitions

Elemental Analysis: **Qualitative** & **Quantitative** determination of elemental (and isotopic) composition of a material (major, trace and ultra-trace). Not limited to any specific matrix, target analyte or method.



Calibration Curve



Qualitative: What elements are present?

Quantitative: How much of those elements are present?

Elemental Analysis
Definitions

- **Combustion Gas Analysis**: Uses oxygen as a carrier gas **(Carbon/Sulfur)** and a combustion occurs.

- **Fusion Gas Analysis**: Sample is fused in the absence of Oxygen (but using inert carrier gases like He, Ar, N2). No combustion occurs but decomposition/release of gaseous reaction products **(Oxygen, Hydrogen, Nitrogen)**.

The basic principle

Light element analysis in inorganic solids

- Weighing of dry solid sample

Turnings

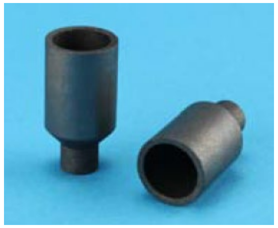
Billets

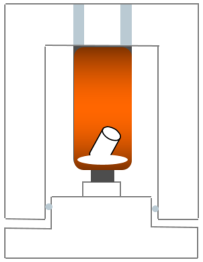
Bars

Powders

- The sample is placed in a crucible and then heated HF-Induction furnace (C/S) or Electrode furnace (O/N/H)

Combustion



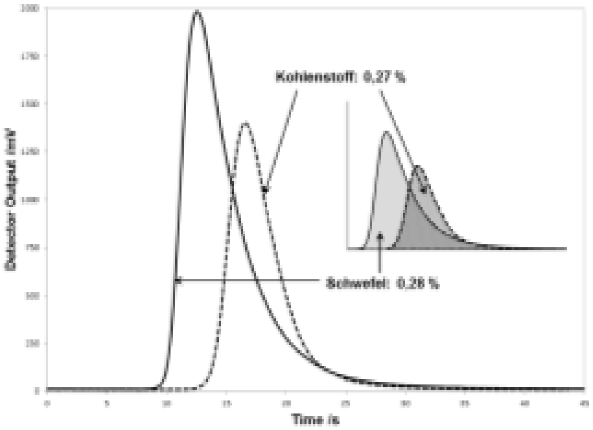


Fusion

Elemental Analysis of Light Elements (C,S / O,N,H)
Gas Release and Detection

- The elements: C, S, N, O, H are released from the sample in a stable gaseous form (= molecules) and transported by the carrier gas stream to detectors for quantification. Eventually disturbing by-product are removed upfront

- Detection Principles: IR-absorption (NDIR), Thermal Conductivity (TC) or Mass Spectrometry (MS)



ONH – why analyze them?

BRUKER

Oxygen	Nitrogen	Hydrogen
• Forms oxidic inclusion with Al, Si, Ca… in steel • Inclusions degrade mechanical properties (e.g. impact strength, brittleness) • Oxygen entering steel while forging causes red shortness	• Forms inclusions which reduce toughness, aging stability and increase risk due to segregation (> 120 ppm) • Alloying of N (e.g. 0.4%) for stabilizing of austenitic grades (CrNiMn steel) and improving the hardness of Mn steels	• Strong influence on mechanical properties of steel: forming pores and cracks • Can lead to most diverse form of damage: flake formation, pickling, blistering, hydrogen-induced embrittlement
O	N	H

The importance of C and S analysis

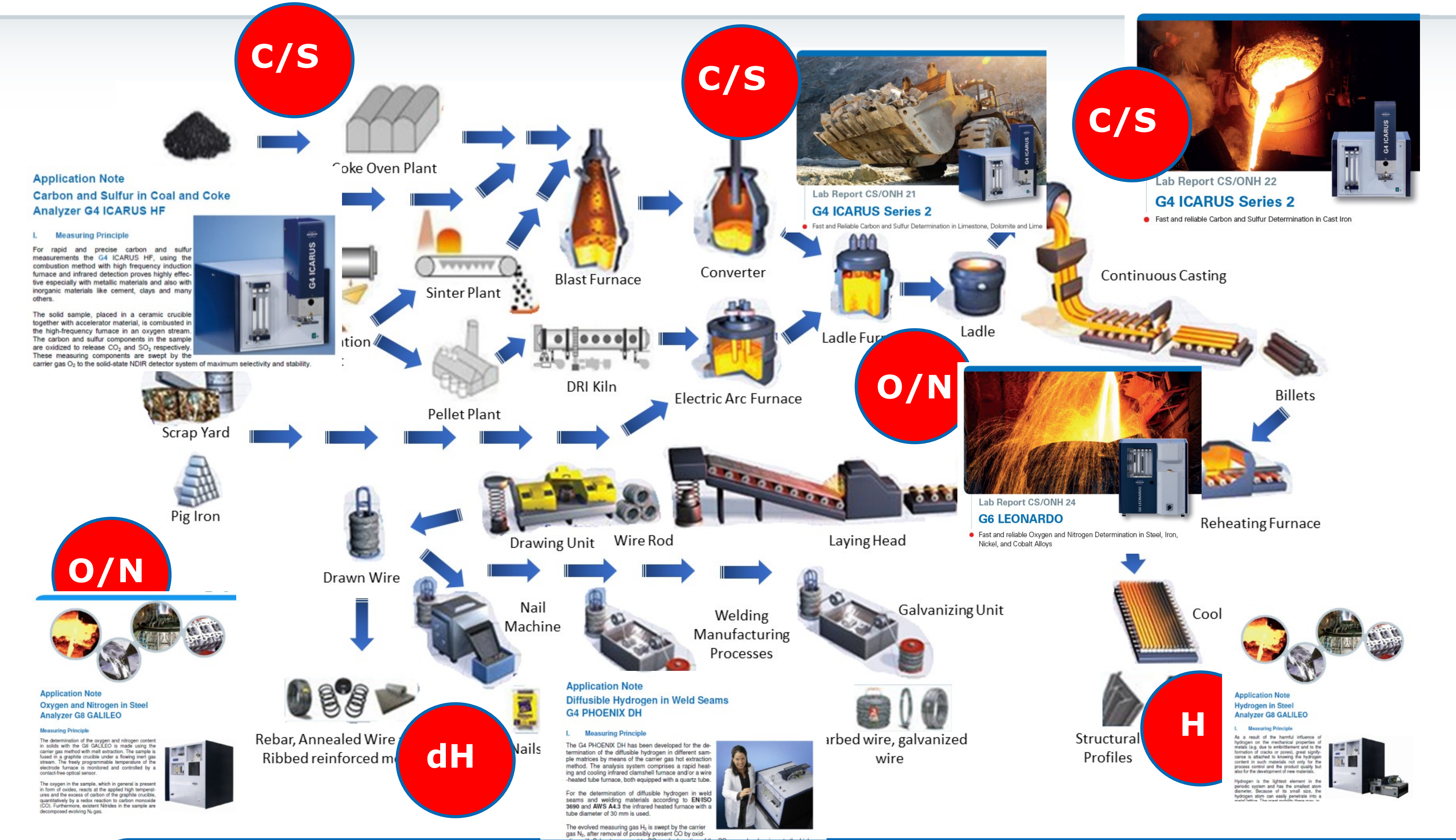
Carbon

- Most alloying element in steel
- Influences hardness, wear resistance, workability, etc.
- Typical concentrations:
  - Carbon steel: ~0.05-2% C
  - Cast iron: ~2-4% C
  - Tungsten carbide: ~6.1% C

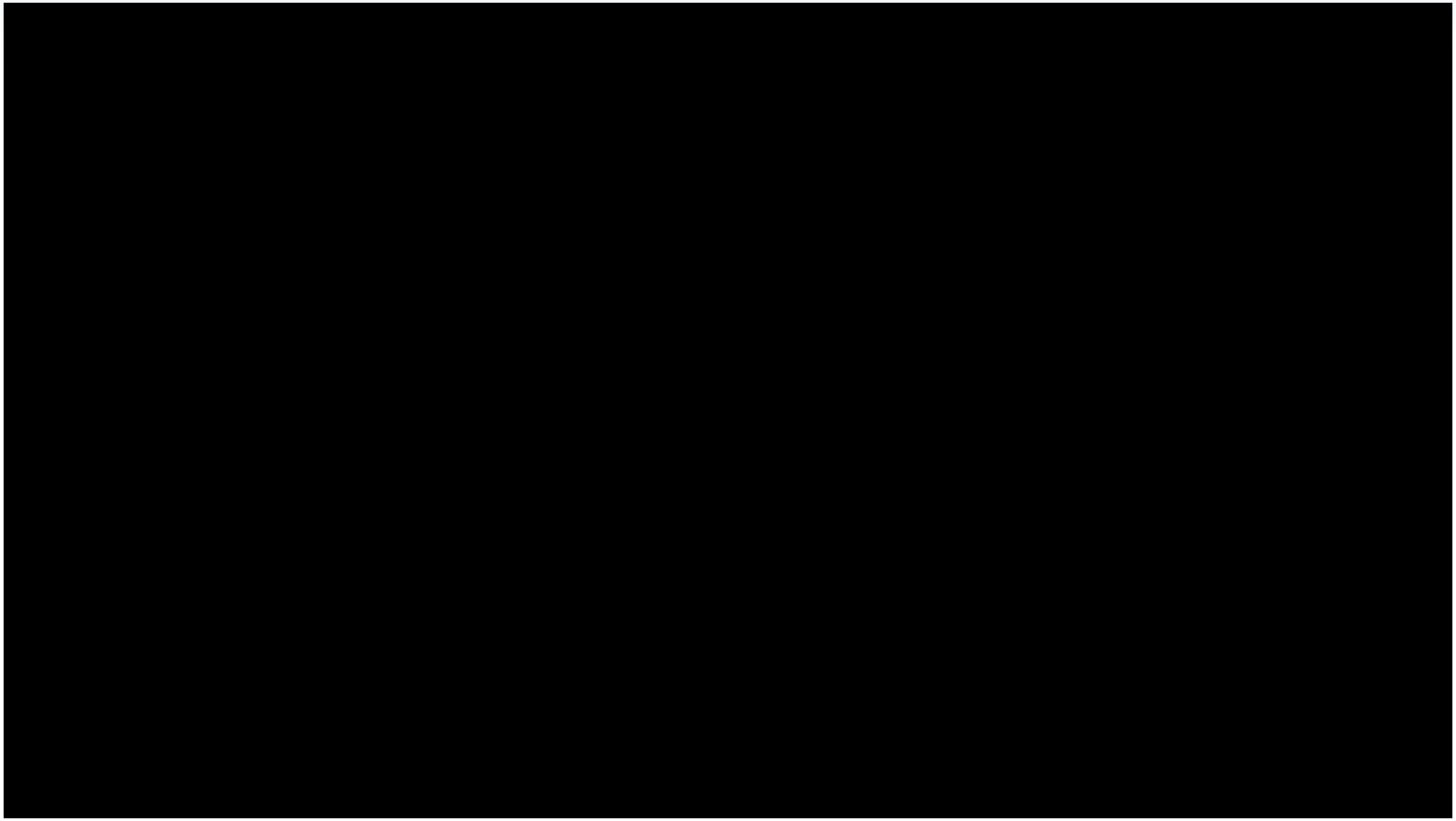
Sulfur

- Undesirable in steel
- Influences brittleness, conductivity, workability, formability, etc.
- Typical concentration in steel: ~100-200 ppm S

Typical Applications
C/S, O/N/H



G4 ICARUS Series 2
Carbon and Sulfur



Overview of Product Line
ON/H

G8 GALILEO
Most flexible system offered, able to quantify ON/H

G6 LEONARO
Most economic system offered, able to quantify O or N or H, ON or OH

G6 LEONARDO
Results O/N

Standard B.S. HON T

Oxygen 0.0044 Wt % (+/- 0.0004)   Nitrogen 0.0365 Wt % (+/- 0.0008)

BS-HON T (1g steel pin)		
certified values:	O: 0.0044 (± 0.0004) %	
	N: 0.0365 (± 0.0008) %	
Mass / g	Oxygen / %	Nitrogen / %
1.0188	0.0045	0.0364
1.0178	0.0045	0.0366
1.0193	0.0042	0.0359
1.0191	0.0044	0.0369
1.0196	0.0043	0.0366
Mean[3)]	**0.0044**	**0.0365**
STD[3)]	0.0001	0.0004

G6 LEONARDO
Results O/N

Standard YSB C 41340B-2011

Oxygen 0.0008 Wt % (+/- 0.00008) Nitrogen 0.0084 Wt % (+/- 0.0003)

YSB C 41340B-2011 (1g ball)		
certified values:	O: 0.0008 (± 0.00008)%	
	N: 0.0084 (± 0.0003)%	
Mass / g	Oxygen / %	Nitrogen / %
0.9543	0.00083	0.0088
0.9542	0.00095	0.0084
0.9535	0.00085	0.0080
0.9549	0.00087	0.0082
0.9539	0.00081	0.0086
Mean	**0.00086**	**0.0084**
STD	0.00005	0.0003

G4 PHOENIX
Diffusible Hydrogen for Welding

- Diffusible Hydrogen is responsible for material failure in steel and other metals:
  - Hydrogen diffuses through the material and is absorbed into so-called “traps” (voids, pores, grain boundaries, micro-cracks, substituted atoms)
  - Can cause embrittlement of the material
  - **Extremely important for automotive, welding and aerospace!**
- G4 PHOENIX and G8 GALILEO + IR07 can measure the amount of diffusible H in a sample using an IR furnace coupled to either TCD or MS

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Lab Reports Available

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Lab Report CS/ONH 23

G4 ICARUS Series 2

- Fast and reliable Carbon and Sulfur Determination in Ore Concentrates, Metal-Bearing Ores and related materials

Many metals occur naturally in form of sulfide minerals like Pyrite (FeS_2), Chalcocite (Cu_2S), Argentite (Ag_2S), Galena (PbS), Sphalerite (ZnS) and many more. Since the amount of economically important metal relative to the ore is small, a first step in the metal extraction process is the separation of target minerals from unwanted rock, gangue and waste minerals. The result, called ore concentrate, is a mixture of the target metal sulfide ores, other sulfide minerals and some gangue.

The sulfur level of the concentrate provides an estimation of the metal-in-concentrate and is used as a quality control measure, determining the efficiency of the concentration process.

In the metal recovery process, determining the carbon and sulfur level in metal-bearing ores is a necessary step for the mine operation to control the metallurgical process kinetics and to assess environmental aspects of the process waste. This is possible due to the fact that most metal-bearing

Elemental Analysis

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Lab Report CS/ONH 22

G4 ICARUS Series 2

- Fast and reliable Carbon and Sulfur Determination in Cast Iron

Introduction

The importance of the element carbon in iron and steel making is well known. Properties like hardness, hardenability, ductility and brittleness depend to a large extent on the content of carbon in the material. That makes carbon the main alloying element in cast iron with typical levels greater than 2%. In the iron matrix, carbon can be present in different forms, e.g. dissolved in the matrix, bound to other elements as carbides, or elementary as free graphite.

Sulfur in contrast is typically an unwanted, contaminant element in cast iron. It can e.g. prevent the graphite formation in cast iron and thus impact the mechanical properties and its workability. Quality- and Process-Control require the fast and accurate analysis of the carbon and sulfur content in cast iron and casted products. This Lab Report shows the simplicity, speed and reliability of carbon and sulfur determination by the G4 ICARUS Series 2 using a high-frequency (HF) induction furnace.

Elemental Analysis

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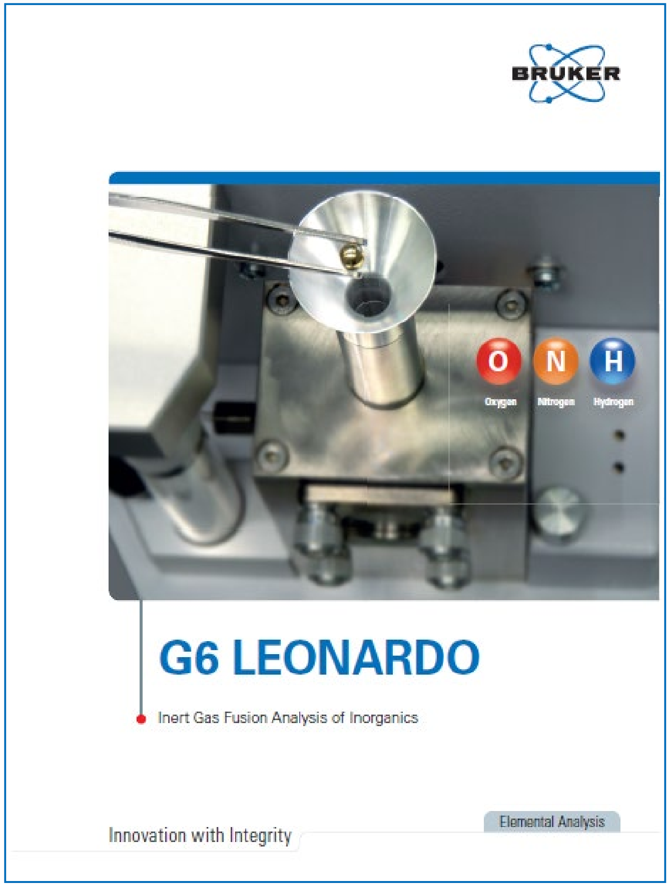
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O Oxygen N Nitrogen H Hydrogen

G6 LEONARDO

- Inert Gas Fusion Analysis of Inorganics

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Elemental Analysis


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Lab Report CS/ONH 24

G6 LEONARDO

- Fast and reliable Oxygen and Nitrogen Determination in Steel, Iron, Nickel, and Cobalt Alloys

Introduction

Oxygen is first used in the steel making process, which is a controlled oxidation process, to remove excess carbon and impurities such as P, Si, and Mn from the hot metal. The level of dissolved oxygen in liquid steel must be lowered because oxygen reacts with carbon, forming CO, which results in excessive porosity during solidification. After the refining, a deoxidation process for removal of residual oxides (inclusions) follows.
The nitrogen level present in the alloy has an influence on its mechanical and corrosion properties.

Increasing nitrogen levels can improve hardness and corrosion resistance of certain alloys but too high nitrogen levels can cause embrittlement, low ductility and inconsistent mechanical properties.

Thus, determining the amount of oxygen and nitrogen is critical to process and quality control when making steel but also nickel, and cobalt base alloys. The G6 LEONARDO provides a cost-effective, efficient and reliable means of determining oxygen and nitrogen in these materials.

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Elemental Analysis

Questions and Answers

Any questions?

Please type any questions
you may have for our speakers
in the Q&A panel and
click Send.

Thank you!

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Virtual Events for X-ray Diffraction and Elemental Analysis

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You've come to the right place. In these trying times of social distancing and working from home, Bruker wants to assure everyone that we are here for you and are committed to keeping you up to date on the latest in our solutions and applications. This webpage will serve as your resource for all of our X-ray Diffraction and Elemental Analysis digital events, including webinars, online trainings, and virtual meetings. We look forward to seeing you online soon!

Live Webinars

Jul 15 - *Introduction to Analysis of Light Elements (C,S - O,N,H) in Inorganic Materials*

The analysis of light elements can be particularly difficult, and with some analytical techniques, almost impossible. Join us for this 45-minute educational webinar to learn how combustion and fusion gas analysis can obtain accurate and precise results for inorganic materials. Whether you're in the steel, cement, mining, aerospace, or additive manufacturing industry, discover how Bruker AXS gas analyzers make your lab life a lot easier.

Register now >

Jul 9 - *Elemental Analysis for the Iron, Steel and Metal Industry by OES, XRF and Combustion/Fusion*

Working in the steel, copper, aluminum, or metal recycling industry? Join us for this 1-hour webinar as our specialists introduce 3 major techniques for elemental analysis:

- Optical Emission Spectrometry (OES)
- X-ray Fluorescence Spectrometry (XRF)
- Combustion/Fusion Analysis (CS/ONH)

The strengths and applications of each technique will also be covered.

Register now >

Jul 8 - *Ways to Improve the Productivity of Your Single Crystal Diffractometer*

In chemical crystallography or structural biology, the best X-ray diffractometers are those that are always in use, consistently producing publication-quality data. Whether you are running an older system or a state-of-the-art instrument, join us for this 45-minute webinar as we show you ways to upgrade to the latest technologies or add accessories that will enhance your diffractometer's versatility and attract more users to your facility.

Register now >

Upcoming CS/ONH Webinar


Webinar

Join us for an educational webinar:

Introduction to Analysis of Light Elements (C,S – O,N,H) in Inorganic Materials

The analysis of light elements can be particularly difficult, and with some analytical techniques, almost impossible. Join us for this educational webinar to learn about how combustion and fusion gas analysis can obtain accurate and precise results for inorganic materials.

Whether you're in the steel, cement, mining, additive manufacturing, or aerospace industry, our gas analyzer product line is sure to make your lab life a lot easier. With our dedicated Application Scientists, Service and Sales Engineers, Bruker is able to help your company meet their analytical needs in the most efficient and affordable way.

During this 40 minute complimentary webinar, specialists from Bruker will:

- **Introduce Theory of Elemental Analysis**
- **Instrumentation for Light Element Analysis**
- **Bruker's Product Line**

The webinar will conclude with an interactive Q&A session, where attendees may ask questions to the panelists.

Register now for the session that best fits your schedule.

Register for Session 1 (9 am CDT / 10 pm SGT)

Register for Session 2 (3 pm CDT / 10 pm CEST)

If you cannot attend, feel free to register anyway, and we will send you a link to the recording to watch at your convenience.

We look forward to seeing you there!

Wednesday, July 15, 2020

Session 1:
9:00 am CDT (Chicago)
4:00 pm CEST (Berlin)
10:00 pm SGT (Singapore)

Session 2:
3:00 pm CDT (Chicago)
10:00 pm CEST (Berlin)

Speakers:

Kristin Odegaard
Technical Sales Specialist, CGA & OES
Madison, Wisconsin, USA

Christian Zuehlke
Business Development Manager
Combustion/Fusion Analysis (CS/ONH)
Karlsruhe, Germany

Introduction to Analysis of Light Elements (C,S – O,N,H) in Inorganic Materials

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