Complete Automated Analysis of Slag and Steel Samples by XRF and OES

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Welcome

Topics

- Why automation?
- Complete solutions for metals analysis
- Automated laboratory layout and sample flow
- Automation components
- OES: Q8 CORONADO and Q8 MAGELLAN
- XRF: S8 TIGER and S8 DRAGON
- Video: Fast Furnace Control
- Q&A

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Why Automation?
Limitations of Manual Labs

- Variable and unguaranteed response time
- Operator dependance
  - Mistakes
  - Motivation
- Human factor risks
  - Injuries
  - Difficult to handle complex tasks
  - Task repetition
- Costs associated with manpower: operators and supervisors
- Cost of sample rejection

Reasons to Automate

- Robotics are designed to handle repetitive tasks
- Tasks remain identical over time
- Sample preparation and instruments are reliable
- Automation is reliable
  - Strict application of SOPs
  - Strict application of all conditions
  - Conditions given by sample ID
  - No errors
- Better traceability
- Rejection percentage dramatically reduced with QC procedures
- Sample handling always done in the same way

Reasons to Automate

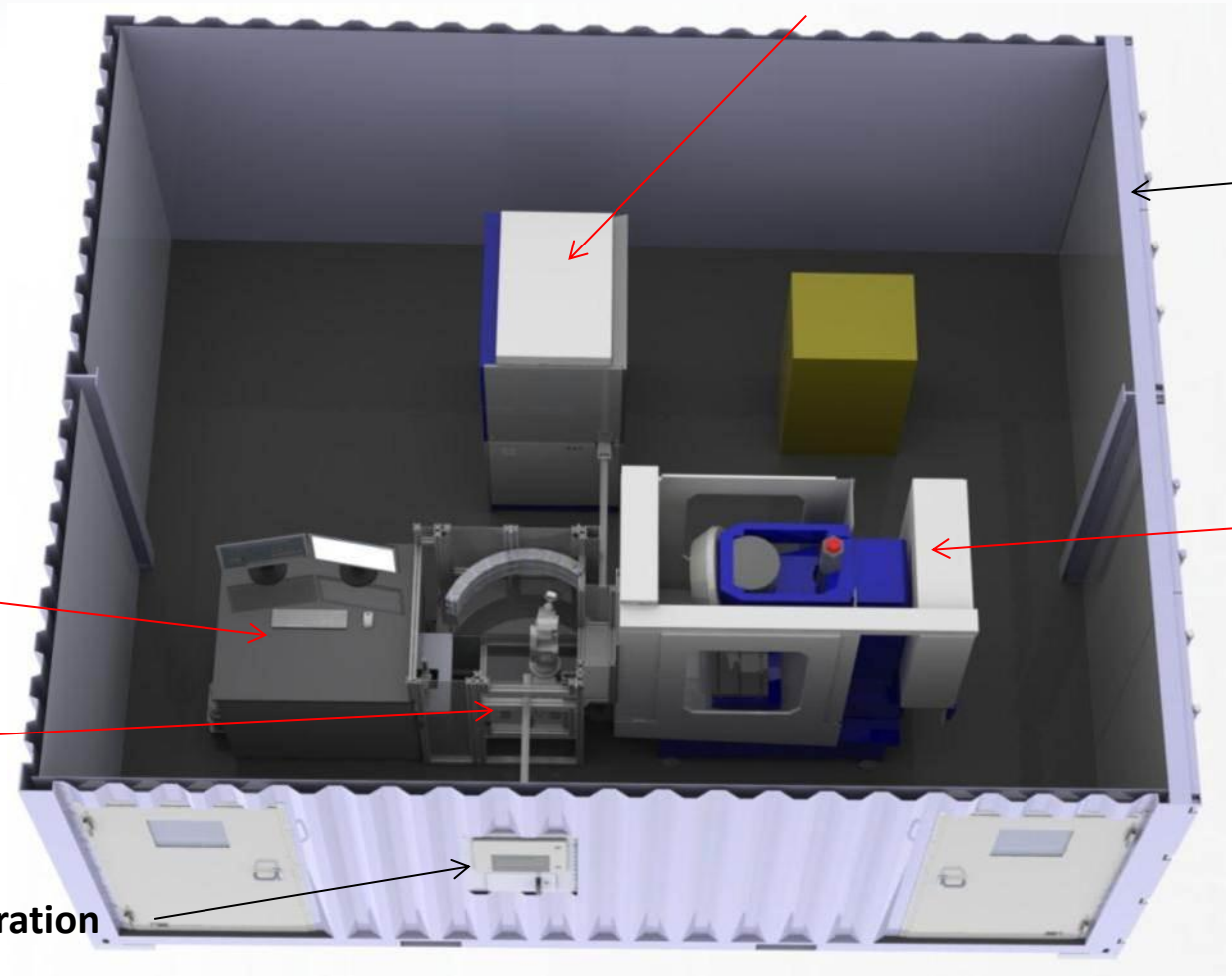
★ **Obtain results faster**

- Operator does not have to interpret results
- Less supervision required
- Results are transmitted automatically and immediately, to the right people
- Speed of analysis
- Higher throughput
- Dead time management
- Elimination of re-sampling
- No need to call supervisor to interpret results

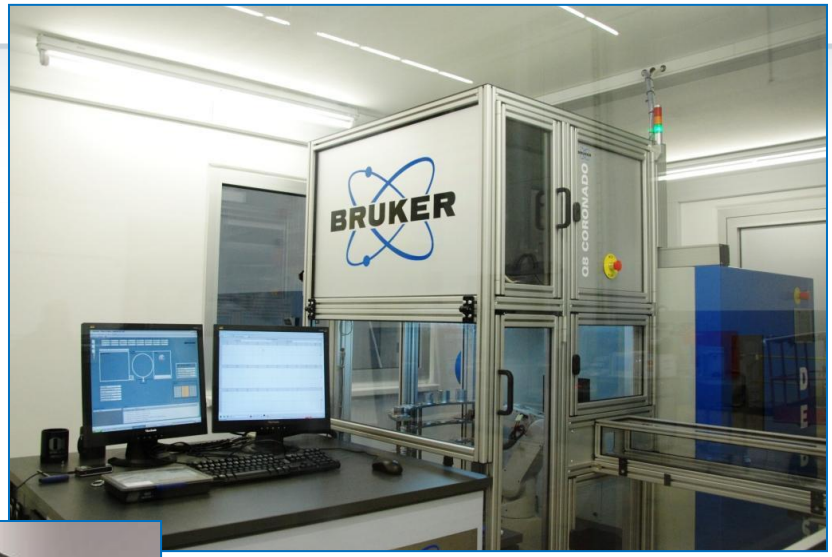
Q8 CORONADO Container Lab

Twin Container Lab
XRF & OES

Complete Solution for Metal Analysis

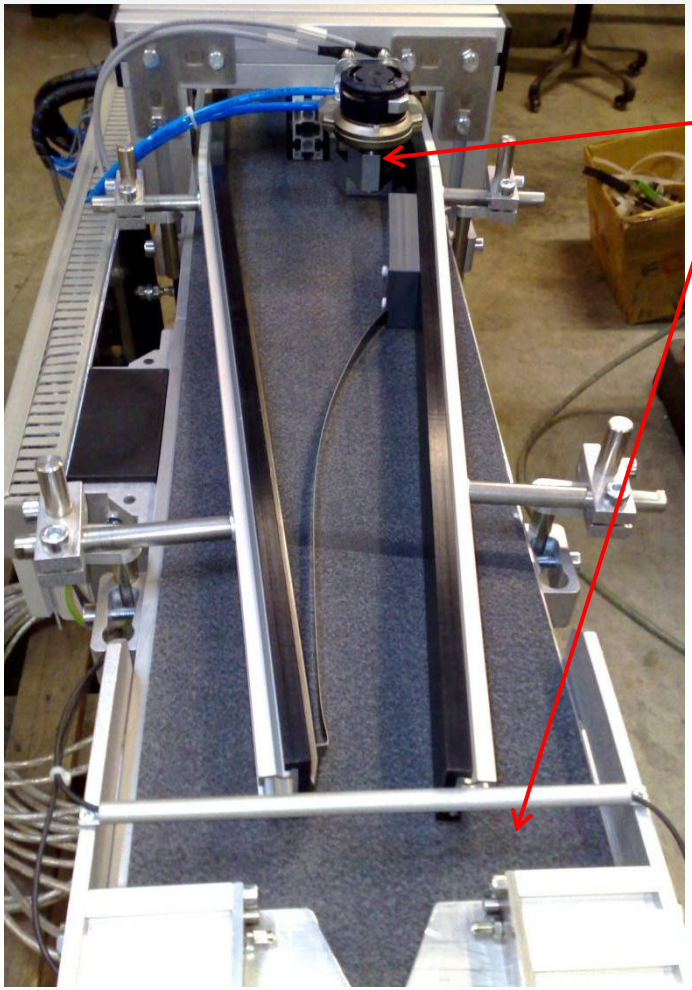


Laboratory Automation



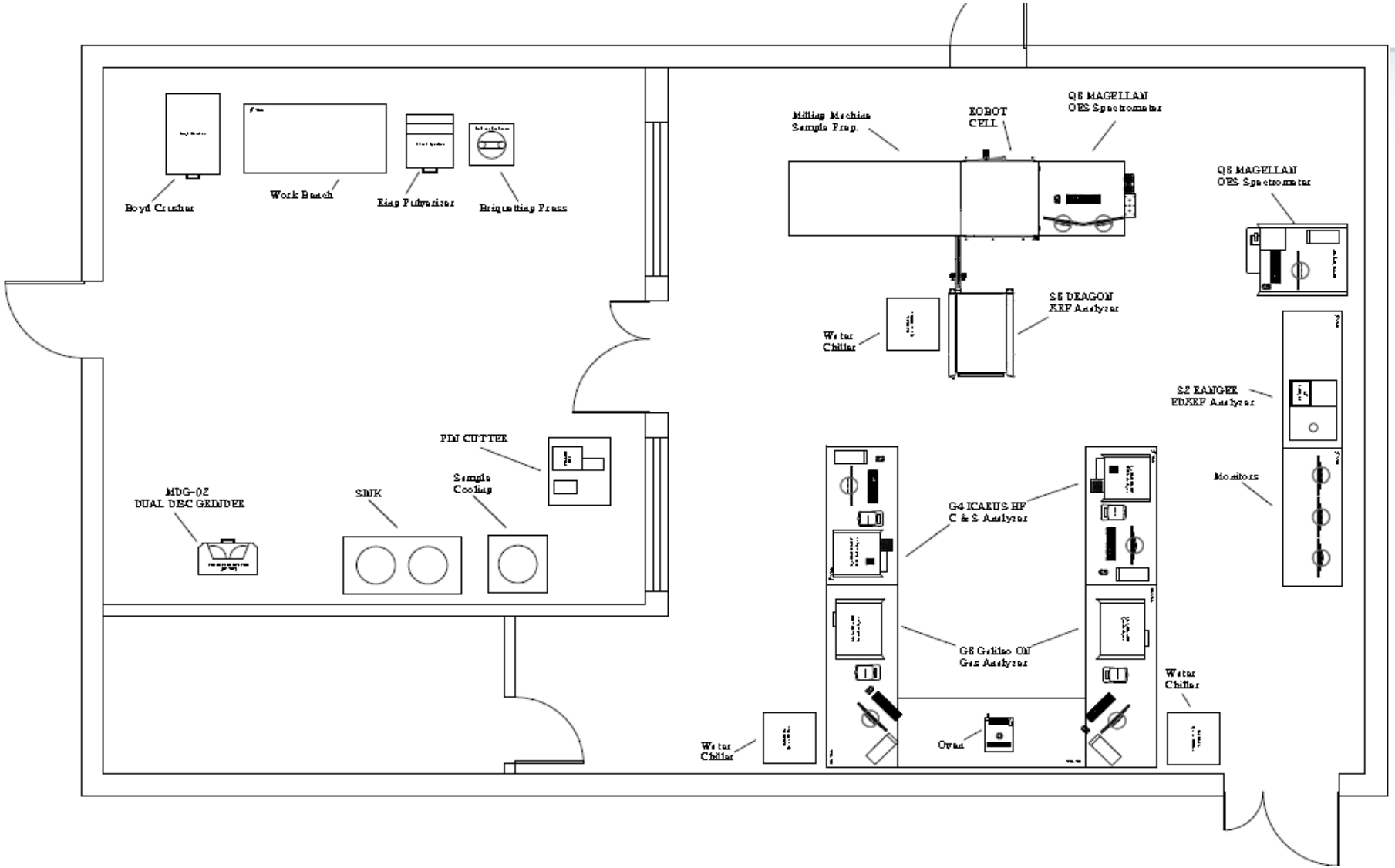
Transport Tube Systems

Transfer to OES or XRF Instrument



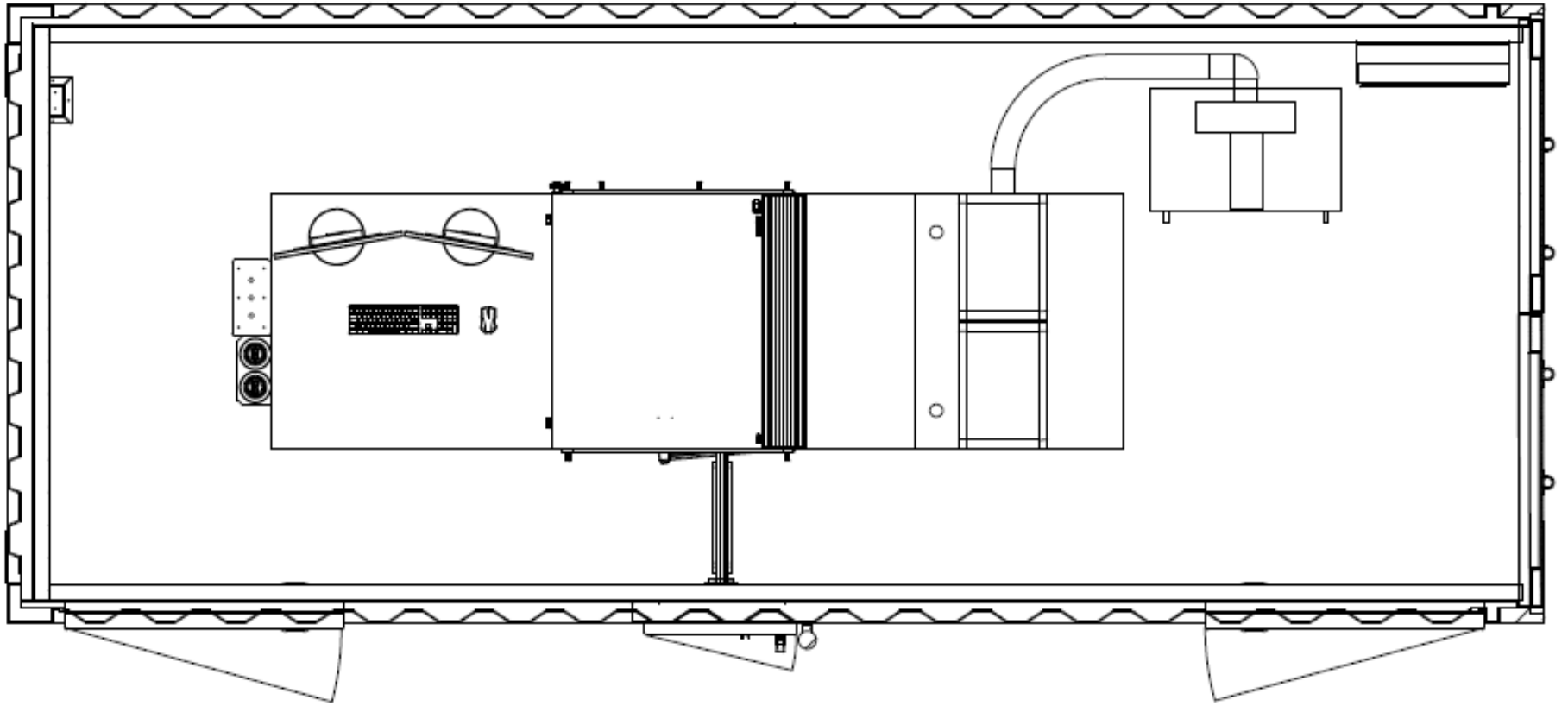
- Commissioning of the samples
- Turning of sample direction
- Control of sample alignment via optical sensor

Automated Laboratory Layout



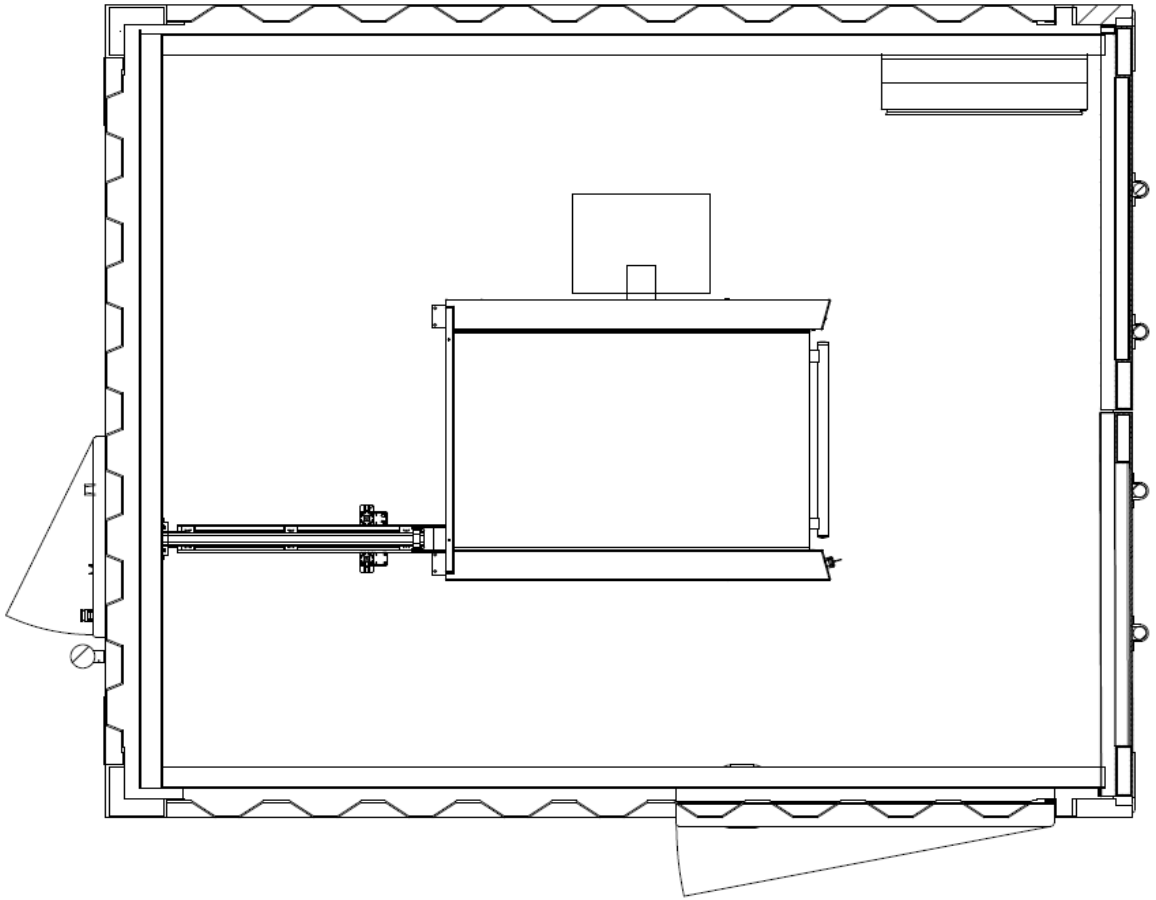
Layout Concept –
Q8 CORONADO Container Lab

for Pig Iron, 20’ L x 8’ D



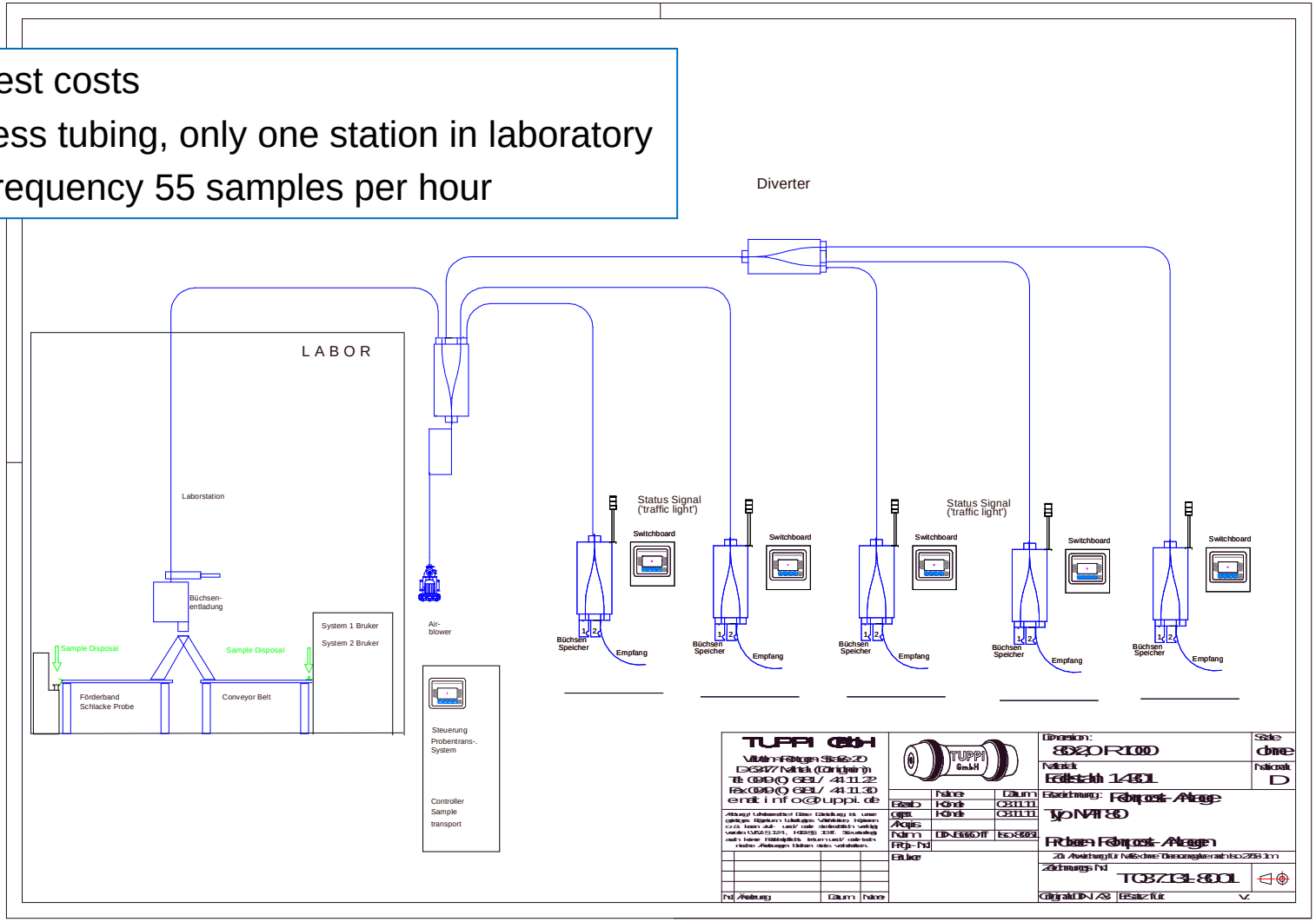
Layout Concept –
S8 TIGER Container Lab

for Slag, 10’ L x 8’ D

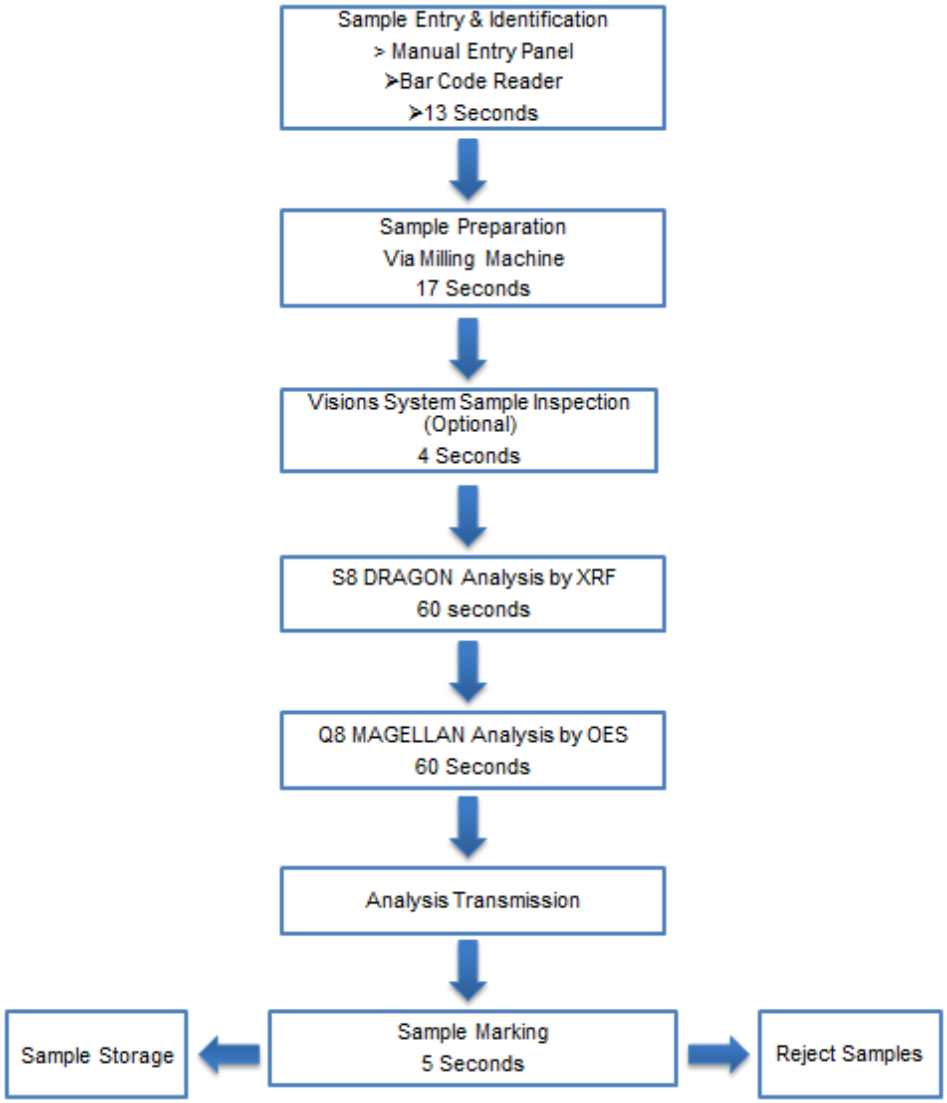


Layout Concept - Tuppi Transport Tube System

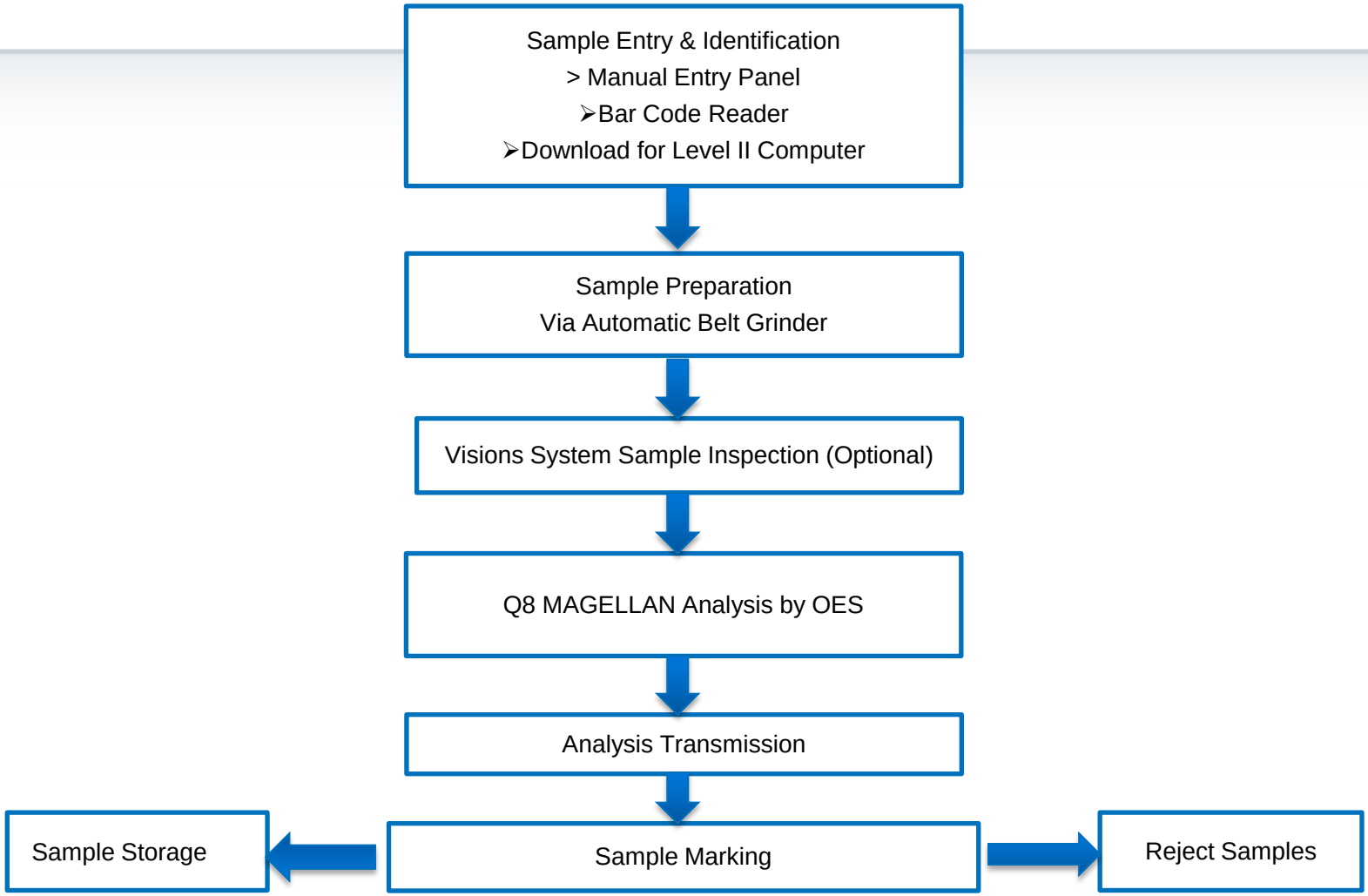
- Best costs
- Less tubing, only one station in laboratory
- Frequency 55 samples per hour



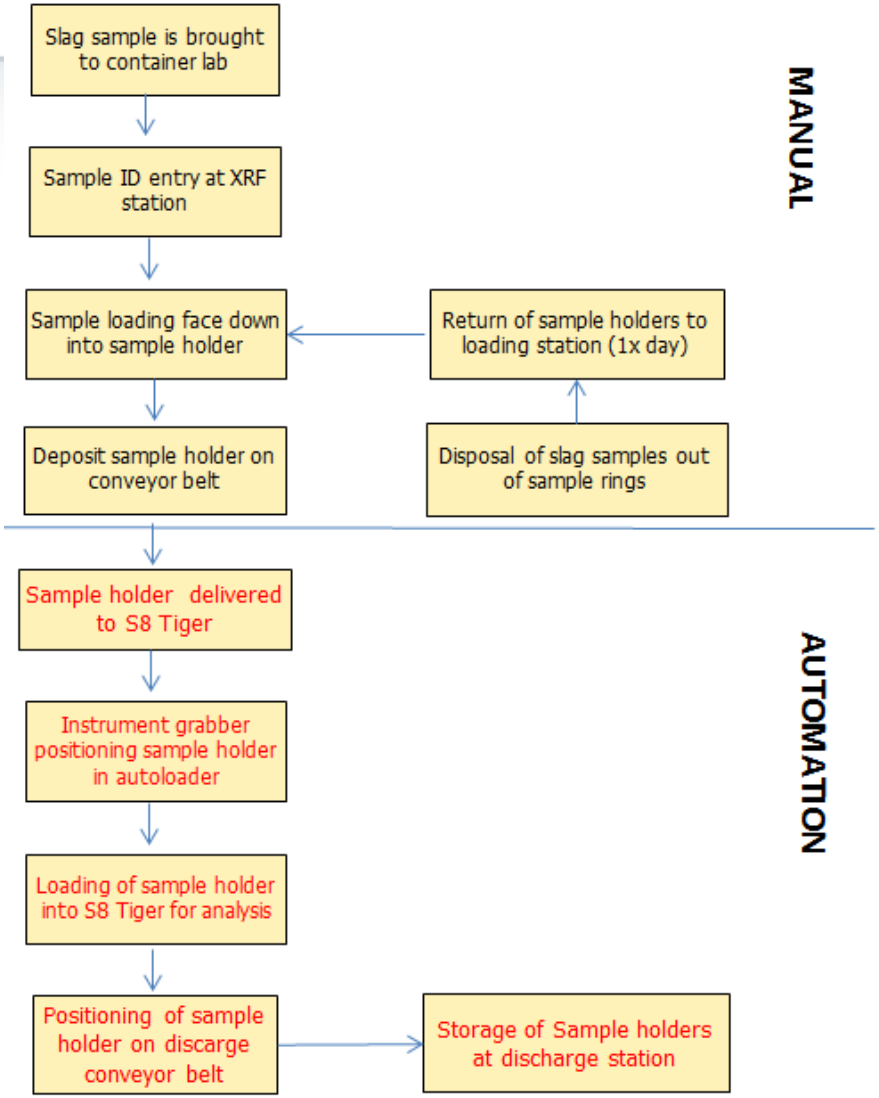
Production Sample Workflow



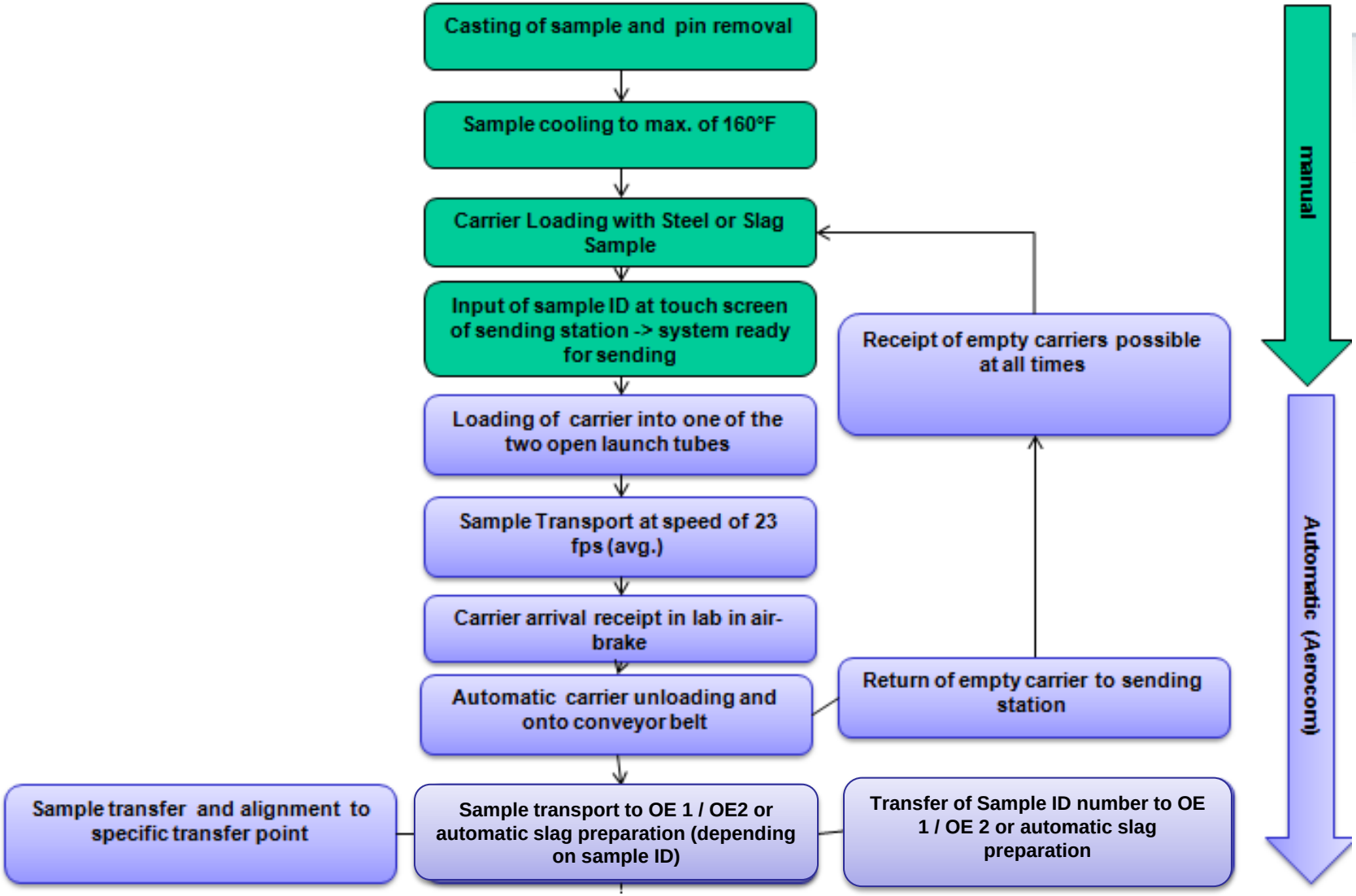
Sample Flow - Pig Iron Analysis



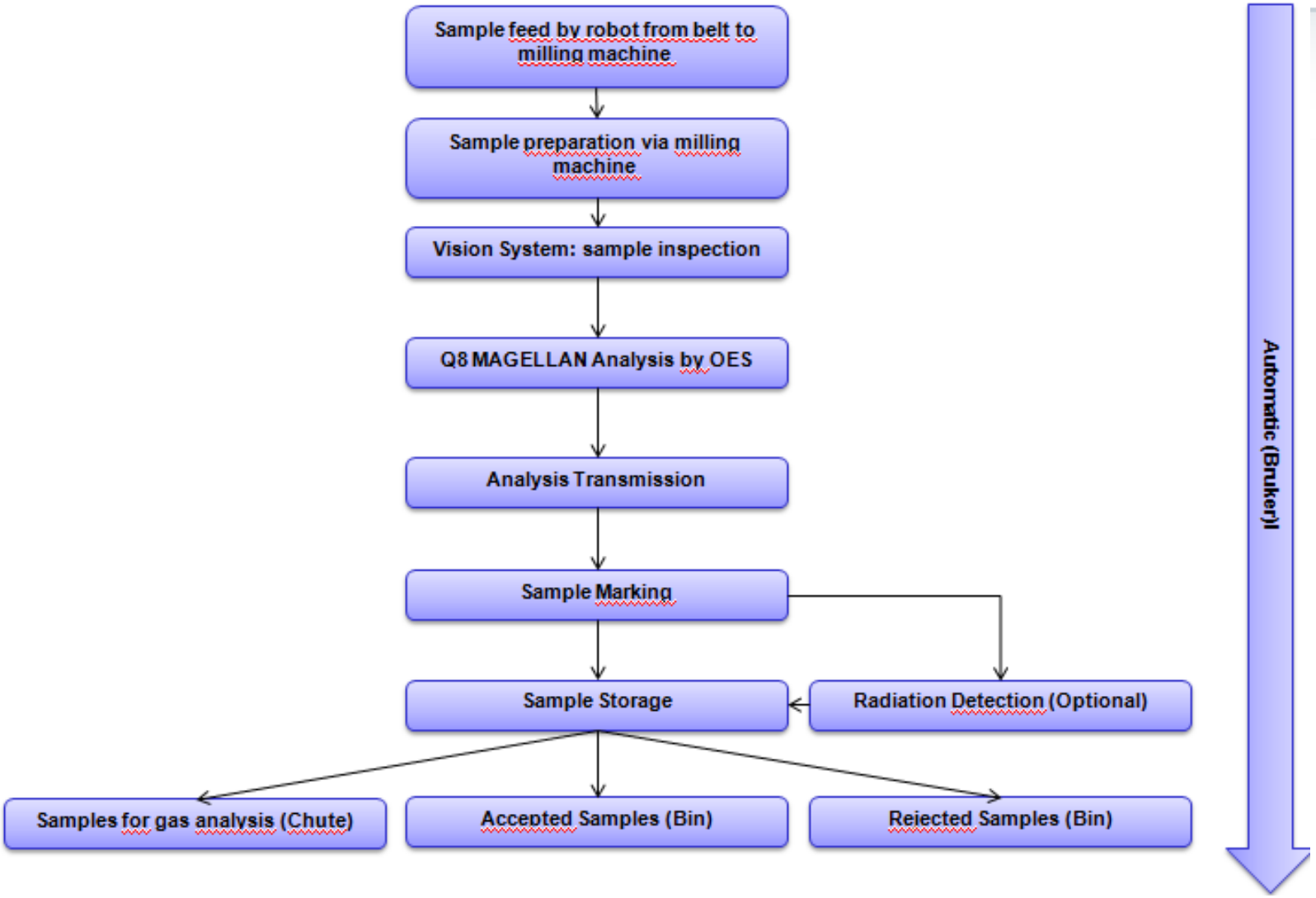
Sample Flow – Slag Analysis



Sample Flow - Steel & Slag Samples



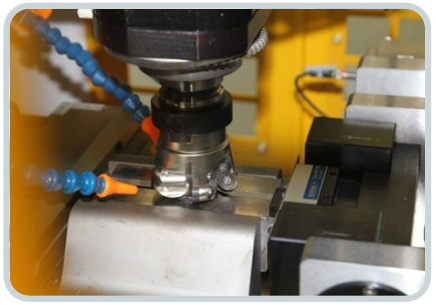
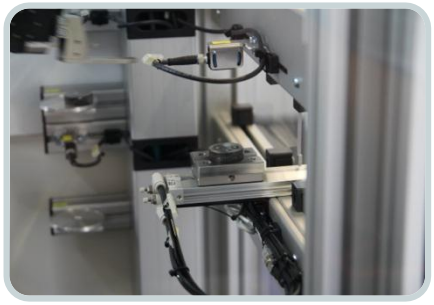
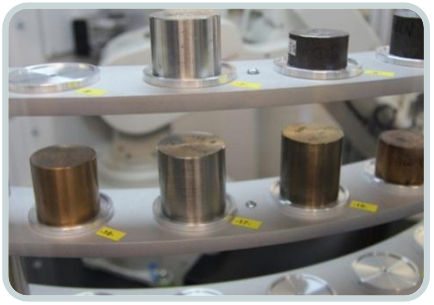
Sample Flow - Steel & Slag Samples (Continued)



Automation Components





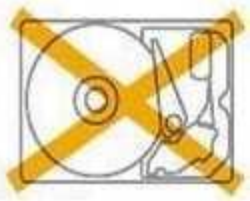






Sample Entry Terminal

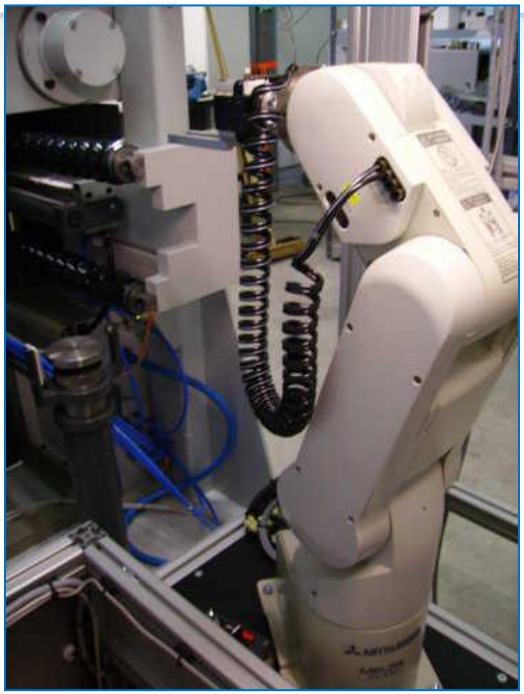
Sample-ID Terminal with Touch-Screen

Robust Touch Panel IPC
12,1 Inch TFT 800x600
Analog resistive touch
IP 65 front-side
Windows CE 5.0
Replaceable display protection film

No rotating parts (e.g. hard disk)



Q8 CORONADO System Components - Robot



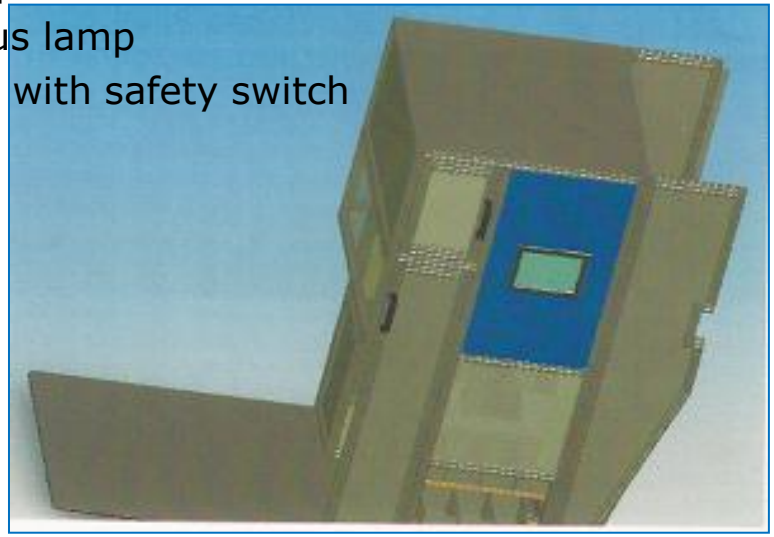
6-axis articulated arm robot with pneumatic hand:

- Payload max. 2 kg
- Reach 410 mm
- Repeatability +/- 0.02mm
- Max. speed 2.2 m/s

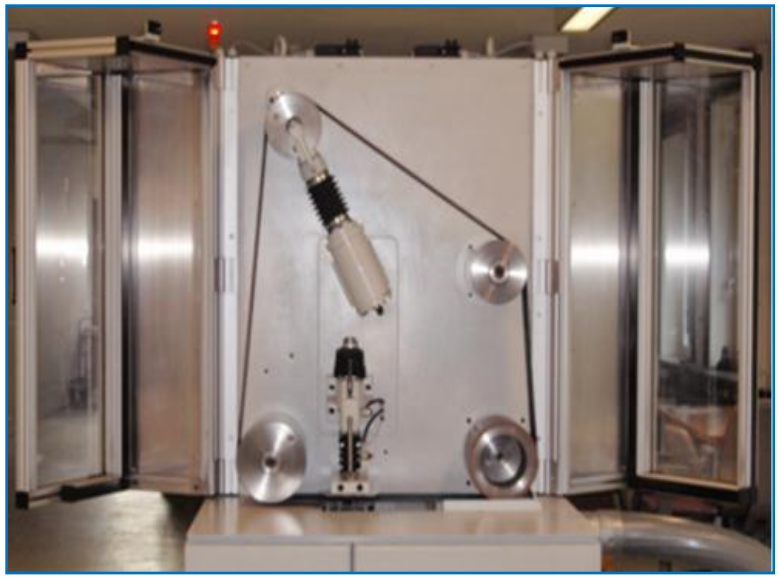
Robot Enclosure

Safety enclosure between spectrometer and sample preparation machine containing:

- Robot, control, drive unit
- Sample entry station / linear transport
- Sample-ID terminal
- Sample rack for system samples
- Archive boxes / drop-off positions
- Electric switch cabinet
- Computer with USB and Ethernet hub
- Status lamp
- Door with safety switch



Automation with Grinding Machine



- Short-time grinding of even hard materials (iron)
- No cooling of sample with additional cooling medium required
- Robust construction
- Easy belt changing due to upper belt guide
- Little system downtime
- Cast iron application: preparation time of 15-18 s including sample handling
- Belt lifetime of approx. 350 samples with 0.3 mm removal each
- Sample temperature < 65°C

Automation with Milling Machine for Preparation of Steel Samples

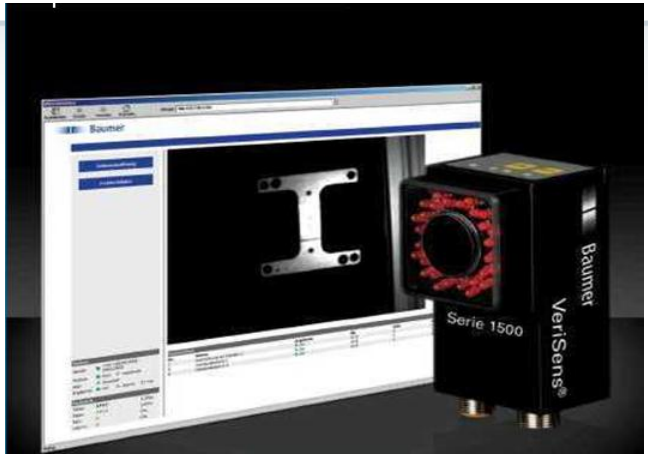


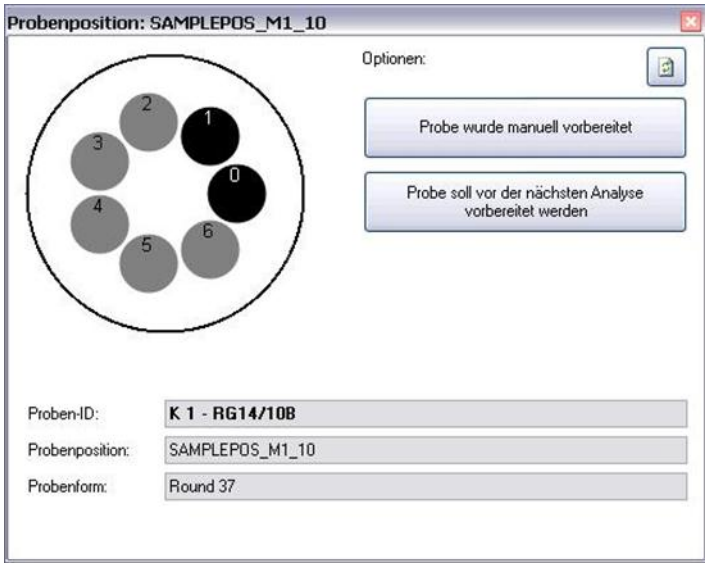





- Fully Automatic Milling Machine for the preparation of ferrous and non-ferrous metals for OES and XRF
- Meets requirements of the sample surface for these type of analysis procedures
- Sample clamping able to handle different pre-defined sample shapes
- Sample clamp integrated into a special handling system
- Depending on sample type, various milling heads can be used
- Milling heads are provided with exchangeable cutting knives
- Different types of cutting knives are available, such as tungsten carbide or CerMets
- Sample feed speed as well as feed motion adjusted in accordance with respective sample and type of material
- Special milling parameters used for control and standardization samples to optimize sample consumption

Visual Inspection System





Visual Inspection System (Option)

A Visual Inspection System can be installed within the robot cell for checking sample surfaces after milling and prior to transfer to the analyzers. The vision camera and illumination system provide live images via standard web browser of the sample surface with images been transferred via Ethernet and TCP/IP to the system pc.

The system PC software performs analysis of the recorded image and decides if the sample surface is suitable for analysis by XRF and OES. The illumination system highlights surface defects (such as cracks, holes, etc). These portions are marked internally to eliminate them from available testing zones.

Depending on the pre-determined testing zones the robot positions the samples in the appropriate spot on the spark stand of the OES. A sample with marginal surface quality can still be analyzed by XRF with results been flagged "XRF – For Information Only"

Q8 MAGELLAN Spark Spectrometer

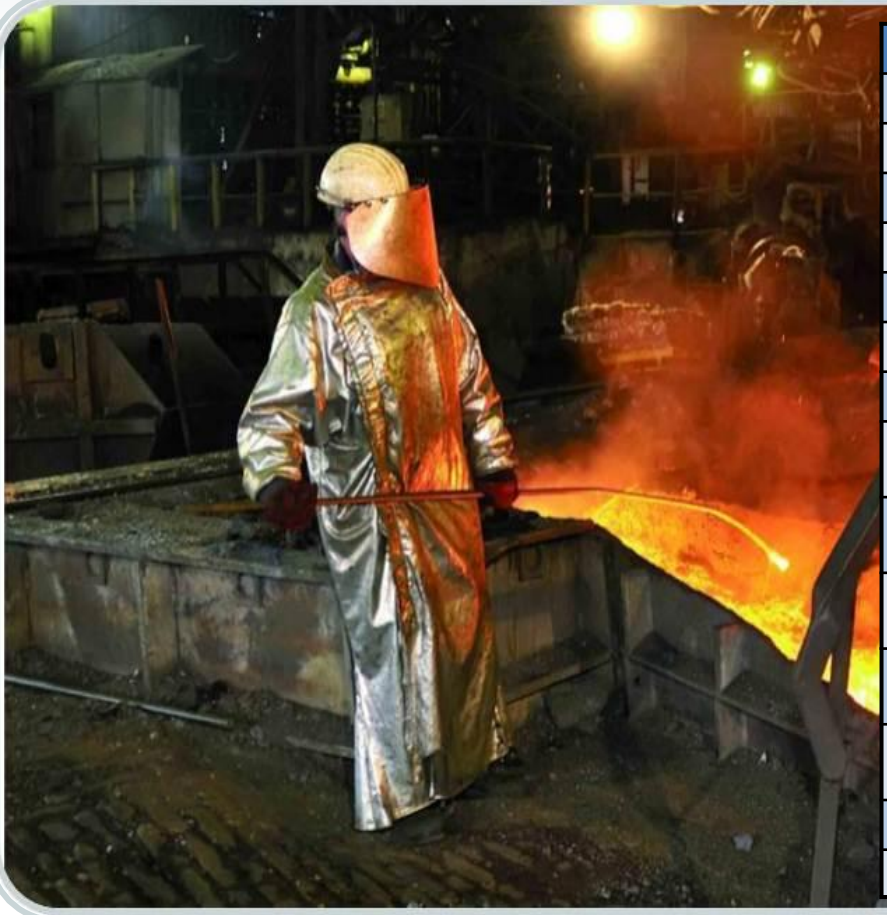
Q8 MAGELLAN Spectrometer with high resolution
750 mm optical system
Channel Photomultiplier
Time resolved single spark evaluation for all analytical channels

QMatrix Software Package with extensions:
QAI – Automation Interface
Automatic averaging
Argon monitoring
Vacuum monitoring

Spark Stand Automation

- Cleaning the electrode
- Cleaning the sparkstand plate
- Holding down the sample during sparking
- Sealing the sparkstand hole against contamination with air

Typical Nitrogen Performance



Sample	Sample 1	Sample 2	RN 14/36
1	0.0039	0.0072	0.031
2	0.0039	0.0067	0.0303
3	0.0039	0.007	0.0303
4	0.0042	0.0068	0.0297
5	0.0043	0.0077	0.0308
6	0.0038	0.007	0.0306
7	0.004	0.0068	0.0306
8	0.004	0.0069	
9	0.0035	0.0071	
10	0.004	0.0069	
11	0.0038	0.0068	
Average	0.0039	0.007	0.0305
SD	0.0002	0.0003	0.0004

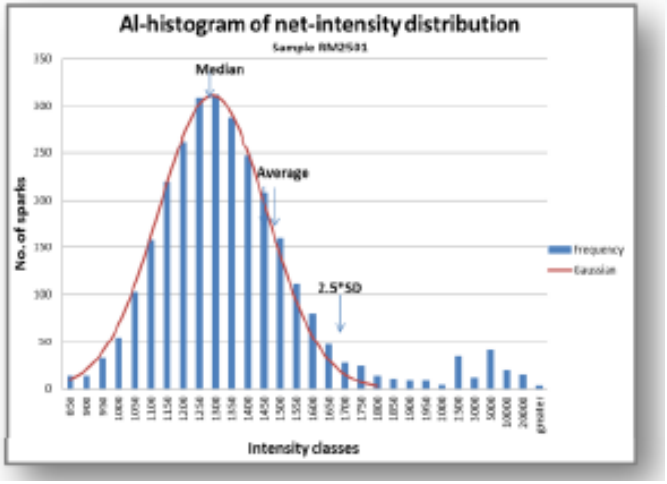
Steel Cleanliness by OES-PDA

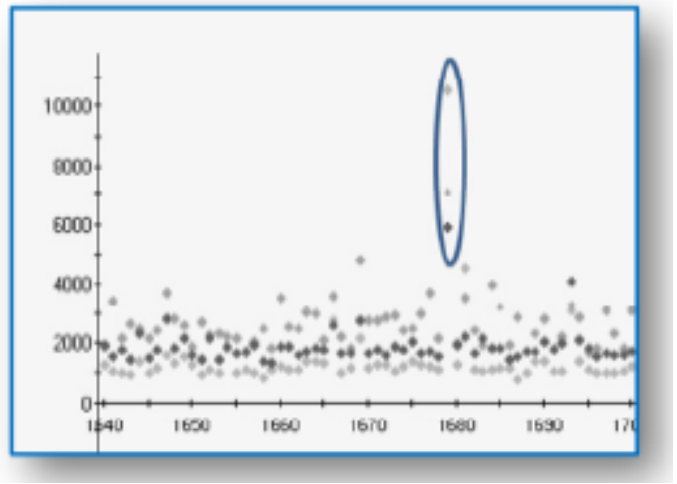
Application Report

Steel Cleanliness Analysis by OES-PDA

Rapid Quality Assurance Check with
Q8 Magellan and MCI-Software

Sample	Analyse No	O-total (ppm)	Al-total (ppm)	Al-insoluble (ppm)	app. K1 value (EN 10247)	PDA-value
72 - BBS O_{REF} = 7 ppm (gas analysis)	1	5,9	88	5,5	1,9	35100
	2	6,4	88	6,1	2,0	37527
	3	5,4	86	5,1	1,7	31491
	4	6,3	87	6,1	1,9	36122
	5	7,9	89	7,6	2,5	45566
	Mean value	6,4	88	6,1	2,0	37161
	SD or RSD (PDA)	0,9	1,1	0,9	0,3	14
NH 4 - BBS O_{REF} = 12 ppm (gas analysis)	1	13,1	54	13,3	4,2	77534
	2	13,7	53	13,8	4,2	78343
	3	10,3	50	10,2	3,2	60312
	4	12,9	55	12,8	4,0	73774
	5	13,5	57	13,4	4,2	77671
	Mean value	12,7	54	12,7	4,0	73527
	SD or RSD (PDA)	1,4	2,6	1,4	0,4	10





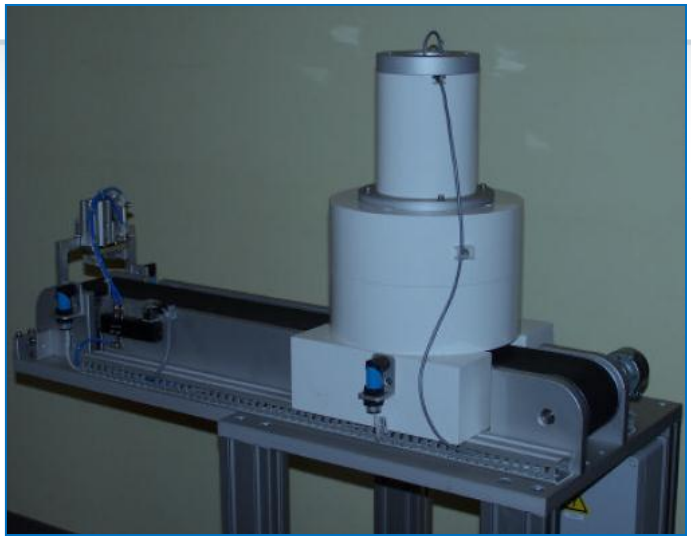
Sample Marking Systems

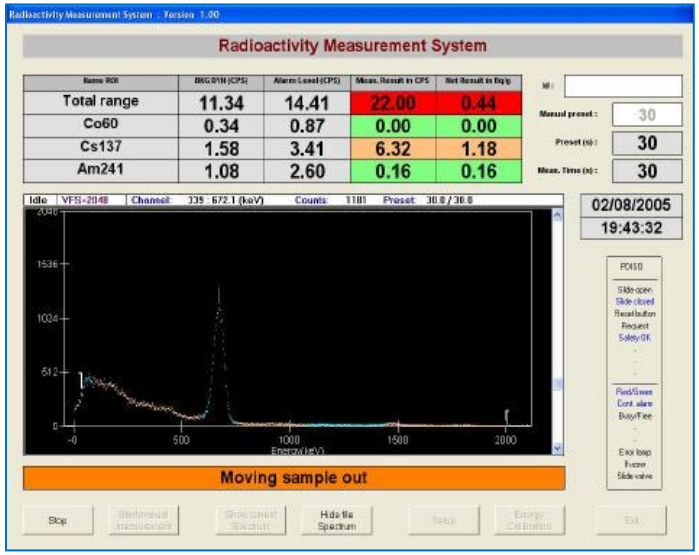
9000 Series printer offer the best balance between performance and flexibility in there segment for an easy integration in all Manufacturing environments. Their state-of-the-art design requires minimal attention and provides high quality coding.The continuous inkjet technology prints the best before dates,Logos, alphanumeric text and 1D and 2D barcodes for different Industries

E8 i52 - Dot peen marking machine designed to be integrated into production line, user-friendly software enables easy creation of texts and graphic symbols and entering marking parameters such as: XY axis positioning, impact force, height and width of characters, resolution between dots etc.

This printer has the following features:
The print head block can be opened providing smooth loading of media and ribbon.
Various kinds of media can be used as the media sensors can be moved from the centre to the left edge of the media. When the optional interface board is installed, Web functions such as remote maintenance and other advanced network features are available.

Radiation Detection System

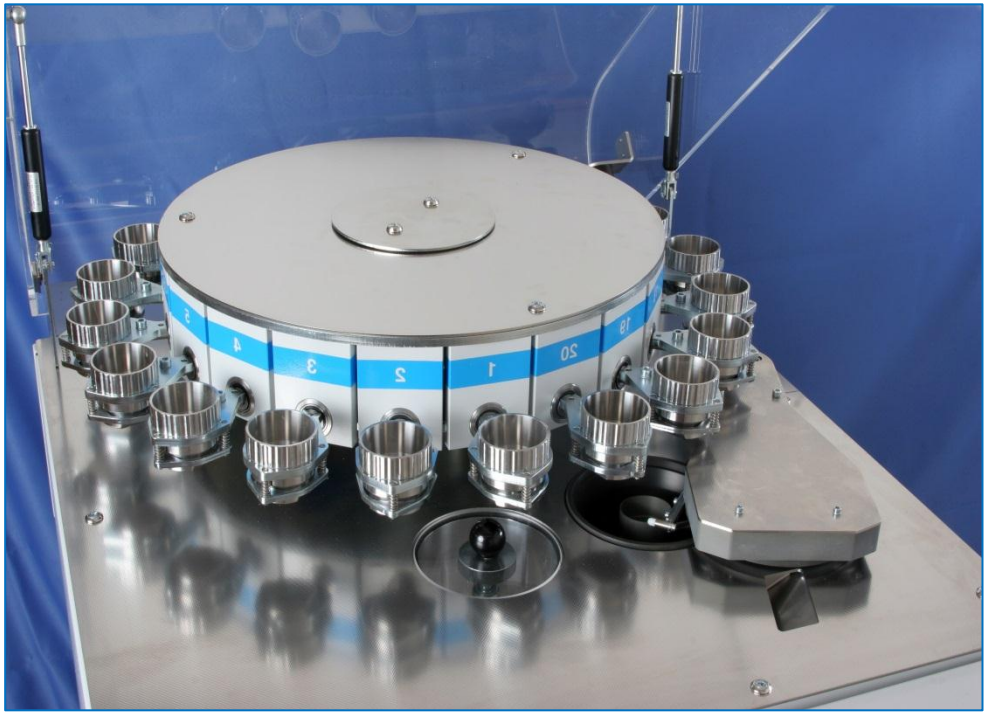




Features

- a robust floor standing aluminum structure with adjustable working height
- NaI(Tl) scintillation detector + Multi Channel Analyzer (uniSpec)
- 7 cm lead shielding with extra shielding over the uniSpec to improve detection limits
- electrically driven conveyor belt with double slide to sort Contaminated/Non Contaminated samples
  OR
  pneumatic drawer for sample input/output
- mechanical design adjustable according to customer's specifications
- human interface for alarm visualization and resetting
- PC based version with spectrum visualization and isotope recognition
- all measurement parameters and results are stored in a MS Access compatible database
- serial interface for communicating sample ID and measurements results
- dynamic background subtraction
- detection limit better than 0,1 Bq/g Co60-equivalent for a 100g sample and a measurement time of 60 seconds
- a Co-60 source included to check the system at regular times

Automatic Slag Preparation



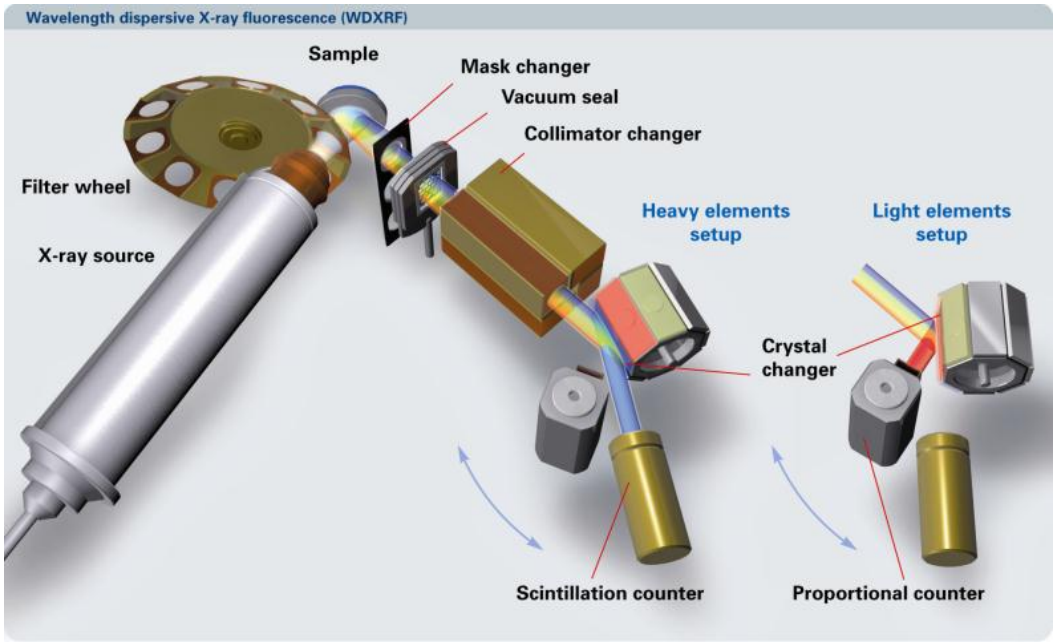
Lab Automation - APMplus
NEW Preparation Unit from Polysius

- Designed for routine operation
  - High throughput
- Automatic operation
  - Material in, pellet out
  - Belt conveyor for automatic transport to X-ray analyzer
  - Excellent reproducibility
- Compact and silent
- Easy to install

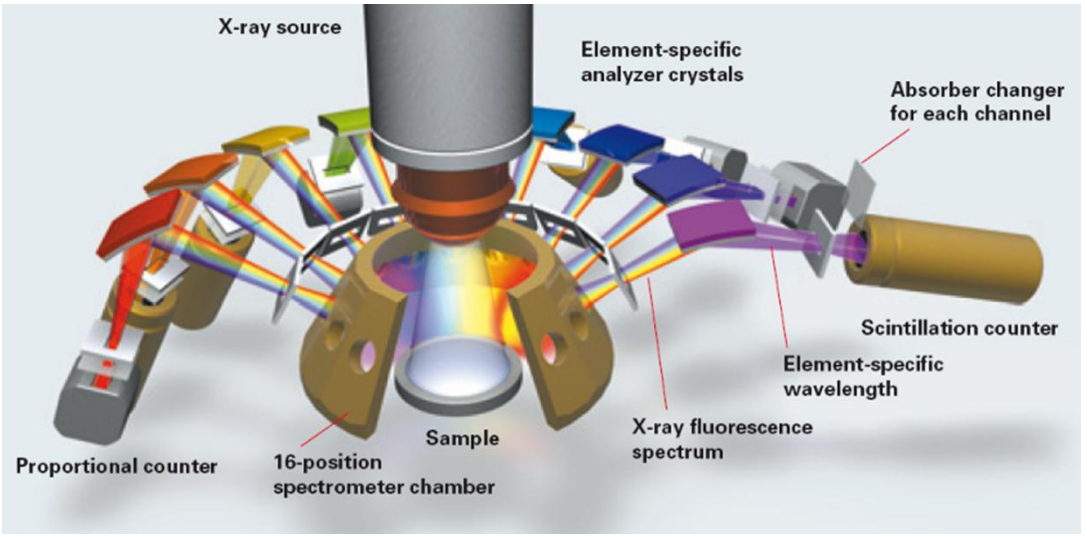
Lab Automation - APMplus
NEW Preparation Unit from Polysius

- Manual batch sample input
  20-position turntable magazine
- Fine grinding mill
- Grinding vessel cleaning
- Grinding aid dosing
- Tablet pressing
- Tablet cleaning
- Steel ring magazine
- Steel ring cleaning
- Steel ring output via conveyor belt

Wavelength Dispersive XRF Sequential – S8 TIGER



Wavelength Dispersive XRF Simultaneous – S8 DRAGON



Comparison
S8 TIGER vs S8 DRAGON

	S8 TIGER	S8 DRAGON
Time of Analysis for Metals and Slags	5 – 10 minutes	< 1 minute
Elemental Range	B - U	B - U 14 elements + MEC
Measure Background	Yes	No
Analysis	Slag	Metals
Backup Analysis	Metals	
Can be Automated	Yes	Yes

S8 DRAGON - EasyLoad

Sample Handling:

Sample cups
- 8 positions stand-alone

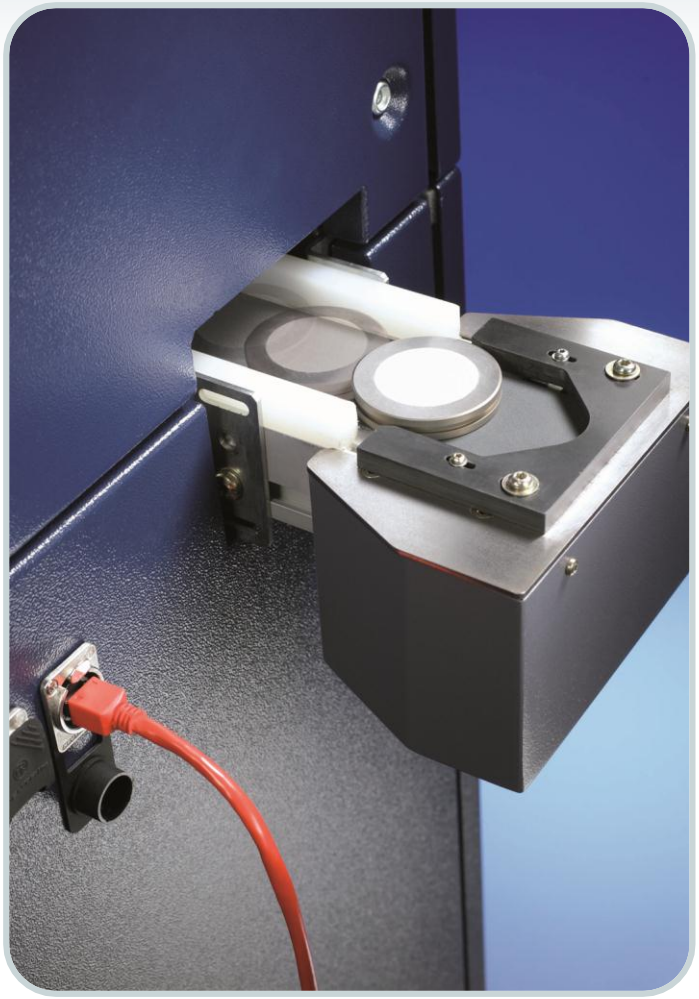
40-mm steel rings
- 12 positions stand-alone
- 9 positions - ONLINE

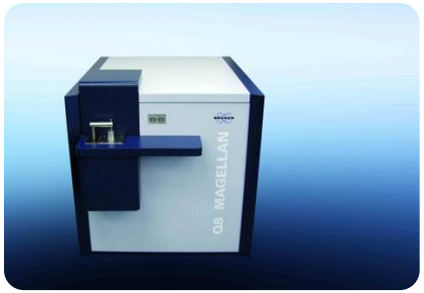
51.5-mm steel rings
- 10 positions stand-alone
- 8 positions – ONLINE

Bare samples
- Diameter: e.g. 33 mm (cylindric)
- Thickness: max. 30 mm
- Weight: max. 500 g

S8 DRAGON
Teamwork to Perfection

- Automated sample transport and pickup at the back of the unit
- Interface for belt connection or robot
- Sample Magazine accessible for non-routine samples from the front
- Easy and simple integration path to automated sample preparation:
  - AXSCOM interface
  - TCP/IP connection
  - Link to Bruker OES





S8 DRAGON - Steel

Low alloy steel with one smart combination of SEC and multielement channel (MEC):

- 19 elements in 40 s measurement time
- Excellent precision
- Best accuracy

- Additional analysis of iron ore and slags

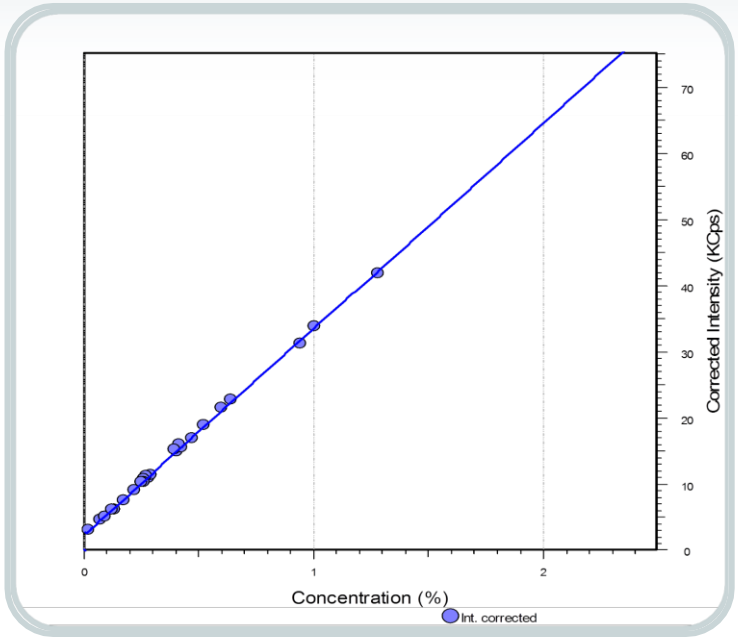
- Monitoring of process materials to track contaminations

S8 DRAGON - Steel

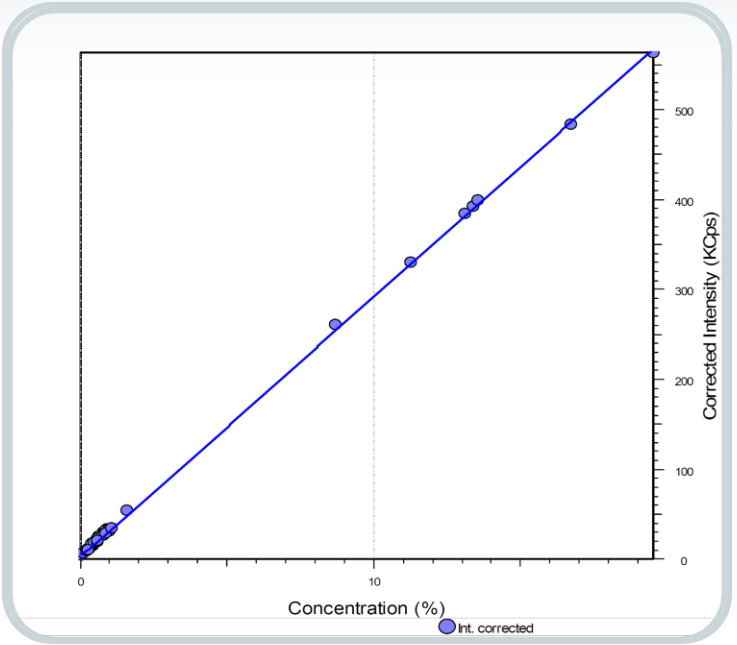
Elements to be analyzed (19): Si, P, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Zr, Nb, Mo, Sn, Ta, W, Pb

- Measurement time = 40 s, plus handling time
- Dual Mode acquisition with Multielement Channel and 14 SEC

S8 DRAGON - Steel Calibration for Mn



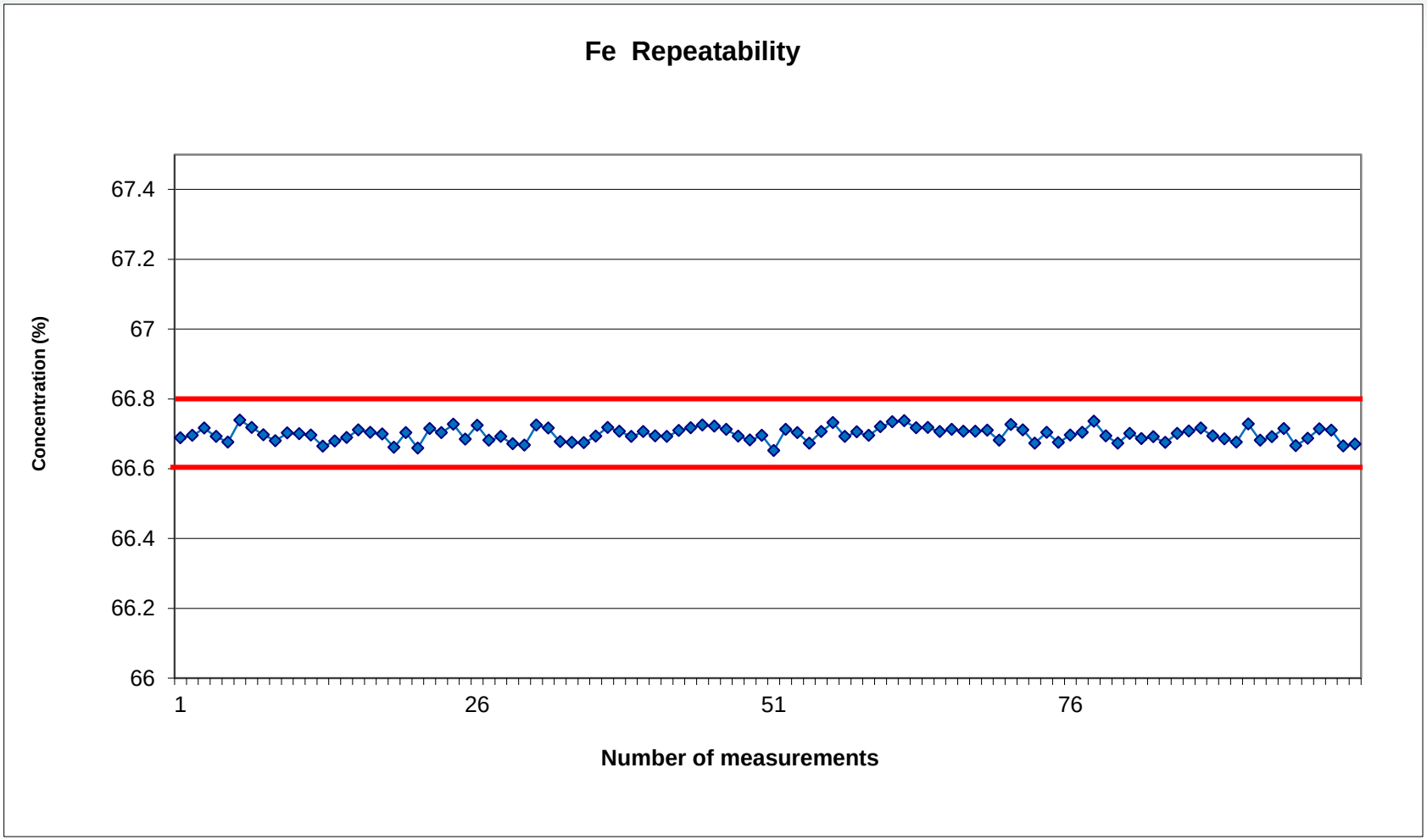
Mn Kα1 Minor
Range: 0 – 1.2 %
RMD = 0.01%



Mn Kα1 Major
Range: 0 – 19.5 %
RMD = 0.07%

- Two ranges with automatic switch depending on intensity

Precision Test on Fe in one week
200 measurements: 66.698 +/- 0.020



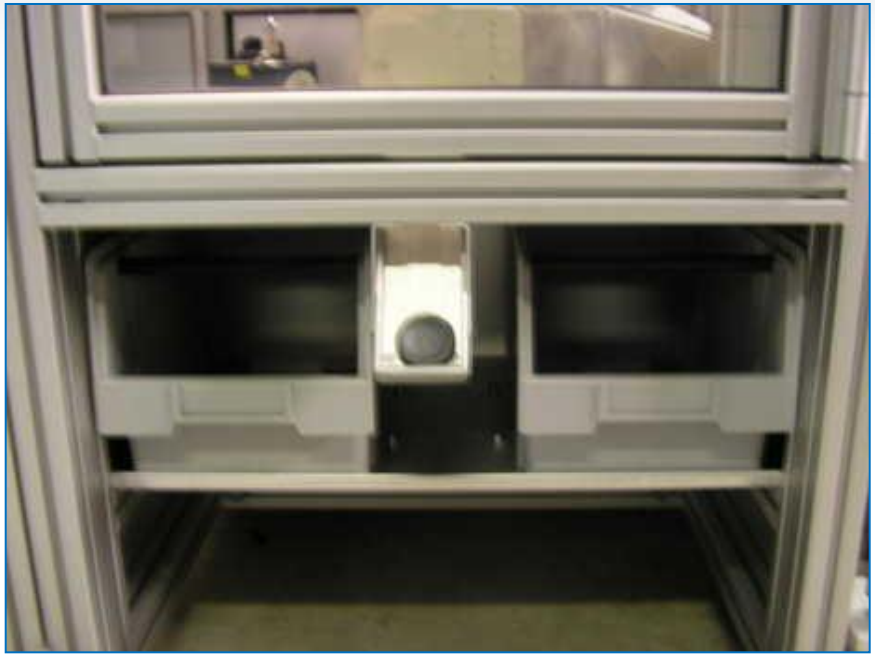
Precision Test for Steel
40 s measurement time - Dual Mode

	Concentration (%)	RSD (%)	LLD (PPM)	Comment
Si Kα1	0 – 4.1	0.056	350	Multielement Channel
P Kα1	0 – 0.1	0.004	2	
Ti Kα1	0 – 2.2	0.08	3	
V Kα1	0 – 9.7	0.15	3	
Cr Kα1	0 – 30	0.07	2	
Mn Kα1	0 – 2	0.01	3	Two ranges
	0 – 20	0.07		
Fe Kα1	50 – 100	0.8		Multielement Channel
Co Kα1	0 – 2	0.02	2	Two ranges
	0 -10	0.02		
Ni Kα1	0 – 6.2	0.008	2	Two ranges
	0 – 35	0.06		

Precision Test for Steel
40 s measurement time - Dual Mode

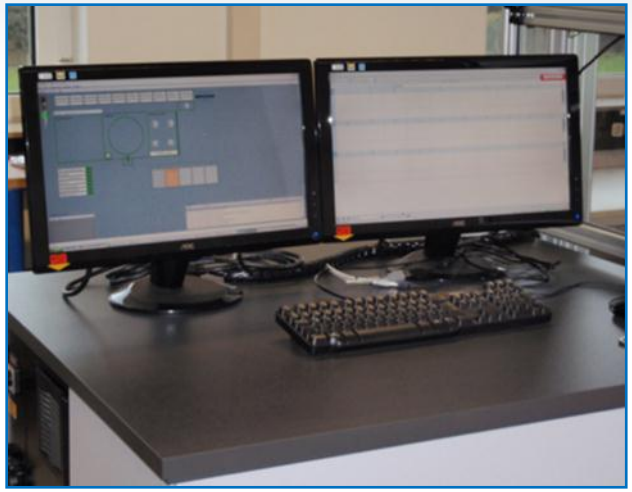
	Concentration (%)	RSD (%)	LLD (PPM)	Comment
Cu Kα1	0 – 3.3	0.02	2	
Zn Kα1	0 – 0.1	0.004	16	Multielement Channel
As Kα1	0 – 0.1	0.002	6	
Zr Kα1	0 – 0.1	0.007	10	
Nb Kα1	0 – 1.1	0.01	75	Multielement Channel
Mo Kα1	0 – 0.3	0.003	1	Two ranges
	0 – 3.2	0.013		
Sn Kα1	0 – 0.2	0.002	4	
Ta Lβ1	0 – 0.3	0.008	12	
W Lα1	0 – 11	0,06	6	Two ranges
	0 – 20	0.10		
Pb Lα1	0 – 1.1	0.009	3	

Sample Archive



- Selective output of production samples in box or on sequential slide
- Boxes are removable
- Optional sample output to external transport system

System Computer



- Two displays
- Standard PC System Windows 7™
- UPS protects the PC against power failure and line surge, avoids loss of data
- Ethernet Switch
- USB 2.0 Hub
- Adjustable overpressure in PC box for cooling and protection against contamination

Safety Concept

- Access control with security doors equipped with electrical sensors
- Door switches directly affect robot controller
- Emergency-Stop security relays with Categorie 4
- Opening the grinding belt housing does not lead to downtime of the rest of the system
- Maintenance-accessibility to OES spectrometer has been considered

Monitoring Analytical Performance

- Automatic measurement of control samples to detect instrument drift in OES or XRF Spectrometer
- Automatic measurement of standardization samples to standardize (recalibrate) the instrument
- Storage of standardization analysis results in SQL database
- Optimized sample preparation to save expensive reference sample material

Q8 CORONADO Video

http://youtu.be/3ep-A0pKw4U

OR

http://www.bruker.com/products/x-ray-diffraction-and-elemental-analysis/optical-emission-spectrometry/q8-coronado/overview.html

Bruker Solution for Fast Furnace Control

Complete automation with

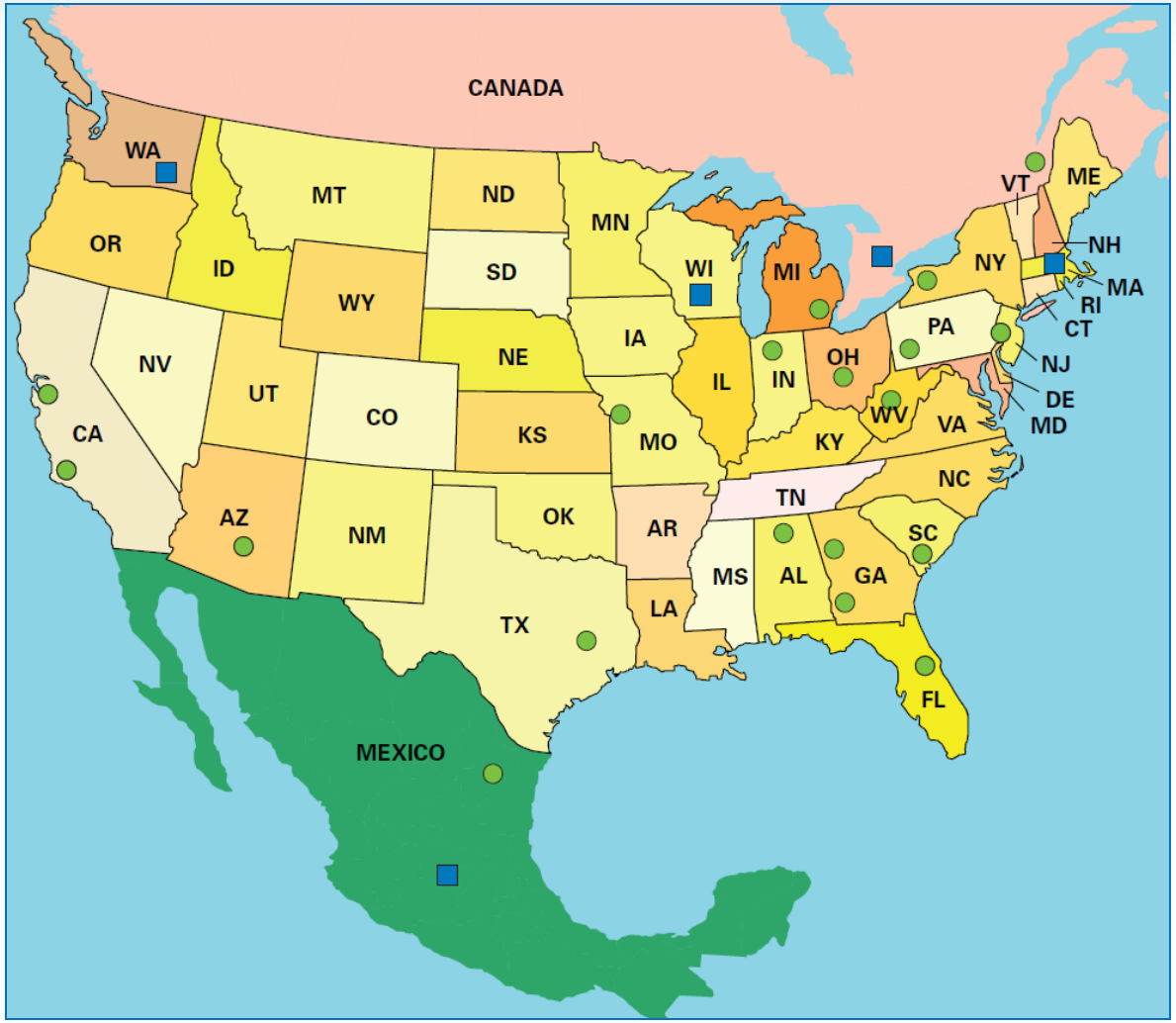
- Sample preparation
  - High speed milling
  - High speed grinding
- X-ray fluorescence analysis
  - Major and minor elements in less than 40 s
- Optical emission spectrometry
  - Traces and light elements

- For fast furnace control
  - High analytical speed
  - Best precision
  - Best accuracy
  - Enhanced trace element
  - Maximum uptime

Bruker Elemental Analysis

- Complete product portfolio for instrumentation in:
  - Optical Emission Spectrometry
  - Combustion Gas Analysis
  - X-ray Fluorescence Spectrometry
  - Sample Preparation
- For metals:
  - Steel Mills and Smelters
  - Foundries
  - Aerospace and Automotive
  - Metal Recycling Facilities
  - Commercial Service Laboratories
- The missing piece for large companies is now available:
  - Twin Automation

Bruker Elemental Support Centers



North America

- Support Headquarters
- Support Centers

Questions?

Any questions?

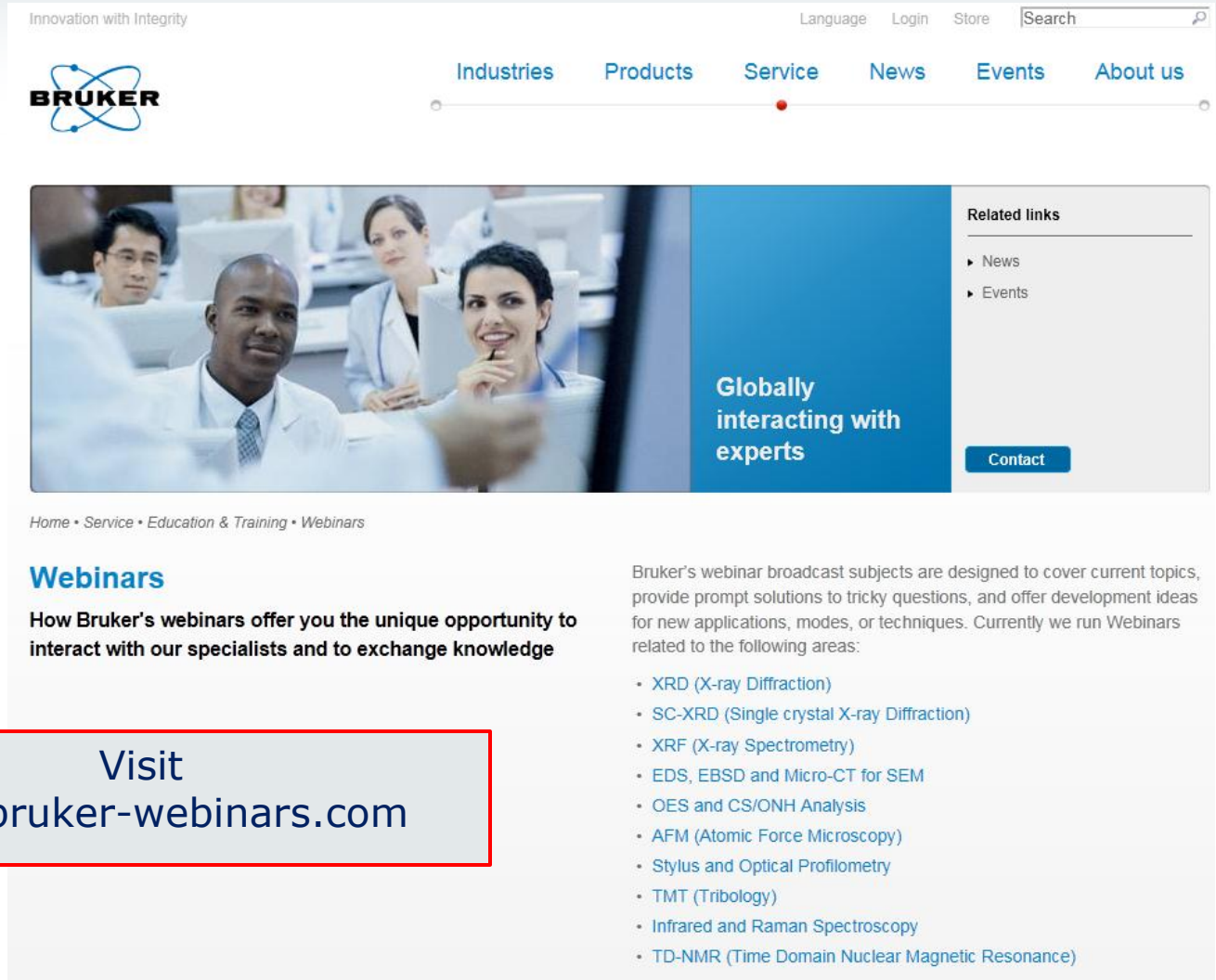
Please type any questions you may have for our speakers in the Q&A panel and click Send.

How did we do?

When you exit the webinar, please fill out our evaluation survey to let us know. We appreciate your feedback.

Thank you!

Past and Upcoming Webinars
XRF and OES



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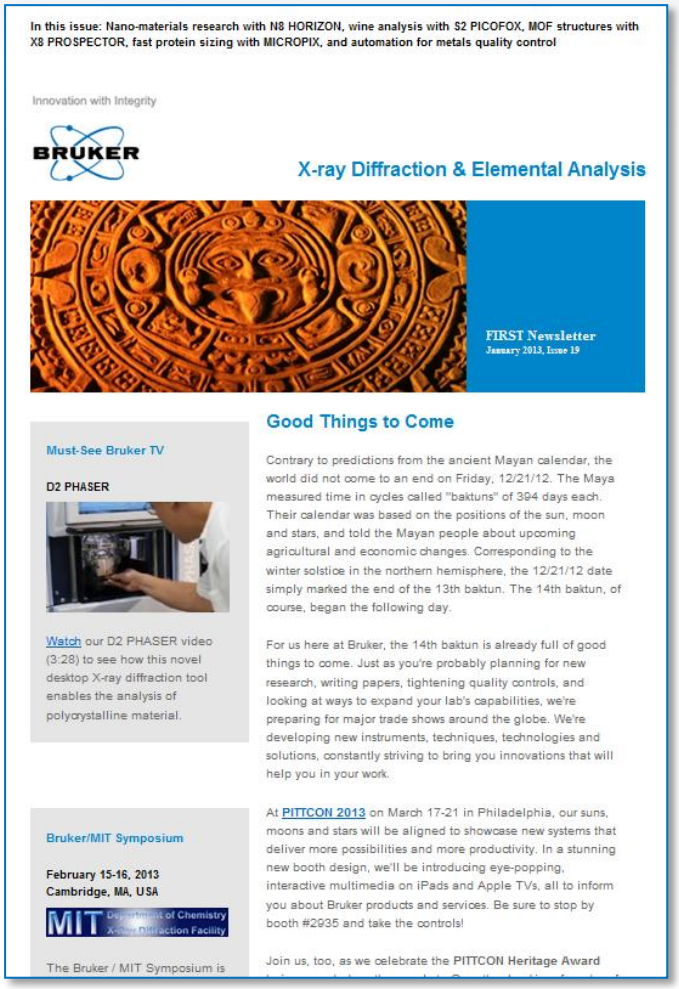

In this issue: Nano-materials research with N8 HORIZON, wine analysis with S2 PICOFOX, MOF structures with X8 PROSPECTOR, fast protein sizing with MICROPIX, and automation for metals quality control

Innovation with Integrity

BRUKER

X-ray Diffraction & Elemental Analysis

FIRST Newsletter
January 2013, Issue 19

Must-See Bruker TV

D2 PHASER

Watch our D2 PHASER video (3:28) to see how this novel desktop X-ray diffraction tool enables the analysis of polycrystalline material.

Good Things to Come

Contrary to predictions from the ancient Mayan calendar, the world did not come to an end on Friday, 12/21/12. The Maya measured time in cycles called "baktuns" of 394 days each. Their calendar was based on the positions of the sun, moon and stars, and told the Mayan people about upcoming agricultural and economic changes. Corresponding to the winter solstice in the northern hemisphere, the 12/21/12 date simply marked the end of the 13th baktun. The 14th baktun, of course, began the following day.

For us here at Bruker, the 14th baktun is already full of good things to come. Just as you're probably planning for new research, writing papers, tightening quality controls, and looking at ways to expand your lab's capabilities, we're preparing for major trade shows around the globe. We're developing new instruments, techniques, technologies and solutions, constantly striving to bring you innovations that will help you in your work.

Bruker/MIT Symposium

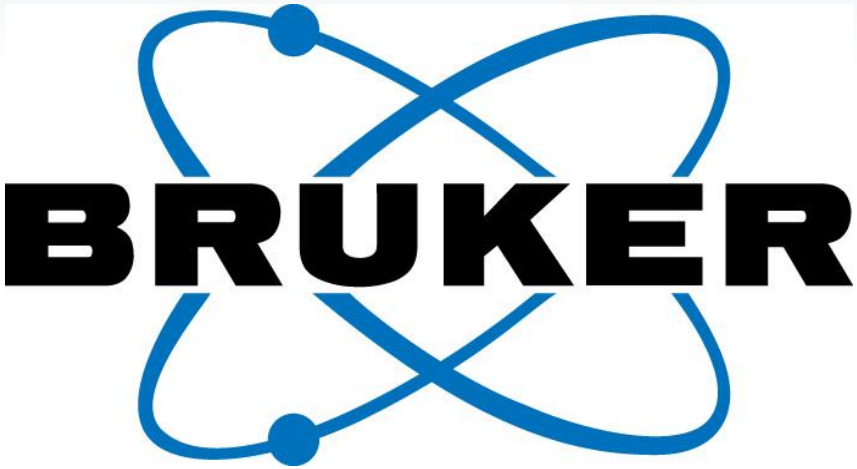
February 15-16, 2013
Cambridge, MA, USA

MIT Department of Chemistry X-Ray Diffraction Facility

The Bruker / MIT Symposium is

At PITTCON 2013 on March 17-21 in Philadelphia, our suns, moons and stars will be aligned to showcase new systems that deliver more possibilities and more productivity. In a stunning new booth design, we'll be introducing eye-popping, interactive multimedia on iPads and Apple TVs, all to inform you about Bruker products and services. Be sure to stop by booth #2935 and take the controls!

Join us, too, as we celebrate the PITTCON Heritage Award



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