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

August 27, 2013

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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

S8 DRAGON

Container Cabin

Milling Machine

Q8 MAGELLAN

Robot

Sample registration
Laboratory Automation
Transport Tube Systems

28.08.2013
Transfer to OES or XRF Instrument

- Commissioning of the samples
- Turning of sample direction
- Control of sample alignment via optical sensor
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
• Best costs
• Less tubing, only one station in laboratory
• Frequency 55 samples per hour
Sample Flow - Pig Iron Analysis

Sample Entry & Identification
  ➤ Manual Entry Panel
  ➤ Bar Code Reader
  ➤ Download for Level II Computer

Sample Preparation
  Via Automatic Belt Grinder

Visions System Sample Inspection (Optional)

Q8 MAGELLAN Analysis by OES

Analysis Transmission

Sample Storage

Sample Marking

Reject Samples
Sample Flow – Slag Analysis

1. Slag sample is brought to container lab

2. Sample ID entry at XRF station

3. Sample loading face down into sample holder

4. Deposit sample holder on conveyor belt

5. Sample holder delivered to S8 Tiger

6. Instrument grabber positioning sample holder in autoloader

7. Loading of sample holder into S8 Tiger for analysis

8. Positioning of sample holder on discharge conveyor belt

9. Return of sample holders to loading station (1x day)

10. Disposal of slag samples out of sample rings

11. Storage of Sample holders at discharge station
Sample Flow - Steel & Slag Samples

- Casting of sample and pin removal
- Sample cooling to max. of 160°F
- Carrier Loading with Steel or Slag Sample
- Input of sample ID at touch screen of sending station -> system ready for sending
- Loading of carrier into one of the two open launch tubes
- Sample transport at speed of 23 fps (avg.)
- Carrier arrival receipt in lab in air-brake
- Automatic carrier unloading and onto conveyor belt
- Sample transfer and alignment to specific transfer point
- Sample transport to OE 1 / OE2 or automatic slag preparation (depending on sample ID)
- Transfer of Sample ID number to OE 1 / OE 2 or automatic slag preparation

Receipt of empty carriers possible at all times

Return of empty carrier to sending station

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Sample Flow - Steel & Slag Samples (Continued)

1. Sample feed by robot from belt to milling machine
2. Sample preparation via milling machine
3. Vision System: sample inspection
4. Q8 MAGELLAN Analysis by OES
5. Analysis Transmission
6. Sample Marking
7. Sample Storage
8. Radiation Detection (Optional)
9. Samples for gas analysis (Chute)
10. Accepted Samples (Bin)
11. Rejected Samples (Bin)

Automatic (Bruker)
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 (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

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

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## Steel Cleanliness by OES-PDA

### Application Report

#### Steel Cleanliness Analysis by OES-PDA

Rapid Quality Assurance Check with Q8 Magellan and MCI-Software

<table>
<thead>
<tr>
<th>Sample</th>
<th>Analyze No</th>
<th>O-total (ppm)</th>
<th>A-total (ppm)</th>
<th>A-insoluble (ppm)</th>
<th>app. K1 value (EN 10247)</th>
<th>PDA-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>72 - BBS</td>
<td>1</td>
<td>5.9</td>
<td>88</td>
<td>5.5</td>
<td>1.9</td>
<td>35100</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>6.4</td>
<td>88</td>
<td>6.1</td>
<td>2.0</td>
<td>37527</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>5.4</td>
<td>86</td>
<td>5.1</td>
<td>1.7</td>
<td>31491</td>
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<tr>
<td></td>
<td>4</td>
<td>6.3</td>
<td>87</td>
<td>6.1</td>
<td>1.9</td>
<td>36122</td>
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<tr>
<td></td>
<td>5</td>
<td>7.9</td>
<td>89</td>
<td>7.8</td>
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<tr>
<td>Mean value</td>
<td>6.4</td>
<td>88</td>
<td>6.1</td>
<td>2.0</td>
<td>37161</td>
<td></td>
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<tr>
<td>SD or RSD (PDA)</td>
<td>0.9</td>
<td>1.1</td>
<td>0.9</td>
<td>0.3</td>
<td>14</td>
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<table>
<thead>
<tr>
<th>Sample</th>
<th>Analyze No</th>
<th>O-total (ppm)</th>
<th>A-total (ppm)</th>
<th>A-insoluble (ppm)</th>
<th>app. K1 value (EN 10247)</th>
<th>PDA-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH4 - BBS</td>
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<td>13.1</td>
<td>54</td>
<td>13.3</td>
<td>4.2</td>
<td>77534</td>
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<td></td>
<td>2</td>
<td>13.7</td>
<td>53</td>
<td>13.8</td>
<td>4.2</td>
<td>78943</td>
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<td></td>
<td>3</td>
<td>10.3</td>
<td>50</td>
<td>10.2</td>
<td>3.2</td>
<td>68312</td>
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<td>4</td>
<td>12.9</td>
<td>55</td>
<td>12.8</td>
<td>4.0</td>
<td>73774</td>
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<td>13.4</td>
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<tr>
<td>Mean value</td>
<td>12.7</td>
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<td>12.7</td>
<td>4.0</td>
<td>73527</td>
<td></td>
</tr>
<tr>
<td>SD or RSD (PDA)</td>
<td>1.4</td>
<td>2.6</td>
<td>1.4</td>
<td>0.4</td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>
9000 Series printer offer the best balance between performance and flexibility in their 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
- 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

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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

<table>
<thead>
<tr>
<th>Feature</th>
<th>S8 TIGER</th>
<th>S8 DRAGON</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of Analysis for Metals and Slags</td>
<td>5 – 10 minutes</td>
<td>&lt; 1 minute</td>
</tr>
<tr>
<td>Elemental Range</td>
<td>B - U</td>
<td>B - U 14 elements + MEC</td>
</tr>
<tr>
<td>Measure Background</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Analysis</td>
<td>Slag</td>
<td>Metals</td>
</tr>
<tr>
<td>Backup Analysis</td>
<td>Metals</td>
<td></td>
</tr>
<tr>
<td>Can be Automated</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Date: 28.08.2013
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
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
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

Fe Repeatability

Concentration (%) vs. Number of measurements
**Precision Test for Steel**

40 s measurement time - Dual Mode

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (%)</th>
<th>RSD (%)</th>
<th>LLD (PPM)</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si Kα1</td>
<td>0 – 4.1</td>
<td>0.056</td>
<td>350</td>
<td>Multielement Channel</td>
</tr>
<tr>
<td>P Kα1</td>
<td>0 – 0.1</td>
<td>0.004</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Ti Kα1</td>
<td>0 – 2.2</td>
<td>0.08</td>
<td>3</td>
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<tr>
<td>V Kα1</td>
<td>0 – 9.7</td>
<td>0.15</td>
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<tr>
<td>Cr Kα1</td>
<td>0 – 30</td>
<td>0.07</td>
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<td>Mn Kα1</td>
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<td>0 – 20</td>
<td>0.07</td>
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<td></td>
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<tr>
<td>Fe Kα1</td>
<td>50 – 100</td>
<td>0.8</td>
<td></td>
<td>Multielement Channel</td>
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<tr>
<td>Co Kα1</td>
<td>0 – 2</td>
<td>0.02</td>
<td>2</td>
<td>Two ranges</td>
</tr>
<tr>
<td></td>
<td>0 -10</td>
<td>0.02</td>
<td></td>
<td></td>
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<tr>
<td>Ni Kα1</td>
<td>0 – 6.2</td>
<td>0.008</td>
<td>2</td>
<td>Two ranges</td>
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<td></td>
<td>0 – 35</td>
<td>0.06</td>
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28.08.2013
## Precision Test for Steel
### 40 s measurement time - Dual Mode

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (%)</th>
<th>RSD (%)</th>
<th>LLD (PPM)</th>
<th>Comment</th>
</tr>
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<tbody>
<tr>
<td>Cu Kα1</td>
<td>0 – 3.3</td>
<td>0.02</td>
<td>2</td>
<td></td>
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<tr>
<td>Zn Kα1</td>
<td>0 – 0.1</td>
<td>0.004</td>
<td>16</td>
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<tr>
<td>As Kα1</td>
<td>0 – 0.1</td>
<td>0.002</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>Zr Kα1</td>
<td>0 – 0.1</td>
<td>0.007</td>
<td>10</td>
<td></td>
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<tr>
<td>Nb Kα1</td>
<td>0 – 1.1</td>
<td>0.01</td>
<td>75</td>
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<td>Mo Kα1</td>
<td>0 – 0.3</td>
<td>0.003</td>
<td>1</td>
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<tr>
<td></td>
<td>0 – 3.2</td>
<td>0.013</td>
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<td>Sn Kα1</td>
<td>0 – 0.2</td>
<td>0.002</td>
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<tr>
<td>Ta Lβ1</td>
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<td>0.008</td>
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<tr>
<td>W Lα1</td>
<td>0 – 11</td>
<td>0.06</td>
<td>6</td>
<td></td>
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<tr>
<td></td>
<td>0 – 20</td>
<td>0.10</td>
<td></td>
<td></td>
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<tr>
<td>Pb Lα1</td>
<td>0 – 1.1</td>
<td>0.009</td>
<td>3</td>
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</tr>
</tbody>
</table>

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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

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!
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XRF and OES

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In this issue: Nano-materials research with NS HORIZON, wine analysis with D2 PHASER, X-ray structures with X8 PROSPECTOR, fast protein sizing with INCROPIX, and automation for installs quality control.

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**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 “tun” of 360 days each. Their calendar was based on the positions of the sun, moon and stars, and told the Maya 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 filled with good things to come. Just as you’re probably planning for new research, writing papers, tightening quality controls, and looking all over 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 aiming to bring you innovations that will help you in your work.

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**Bruker/MIT Symposium**

February 12-16, 2012
Cambridge, MA, USA

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**At Pittcon** on March 17-21 in Philadelphia, our suite, booths and displays 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-pleasing, interactive multimedia on iPads and Apple TVs. All to inform you about Bruker products and services. Be sure to stop by booth #2005 and take the tour.

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Join us, too, as we celebrate the Pittcon Heritage Award.
Your Partner for Steel Works Solutions