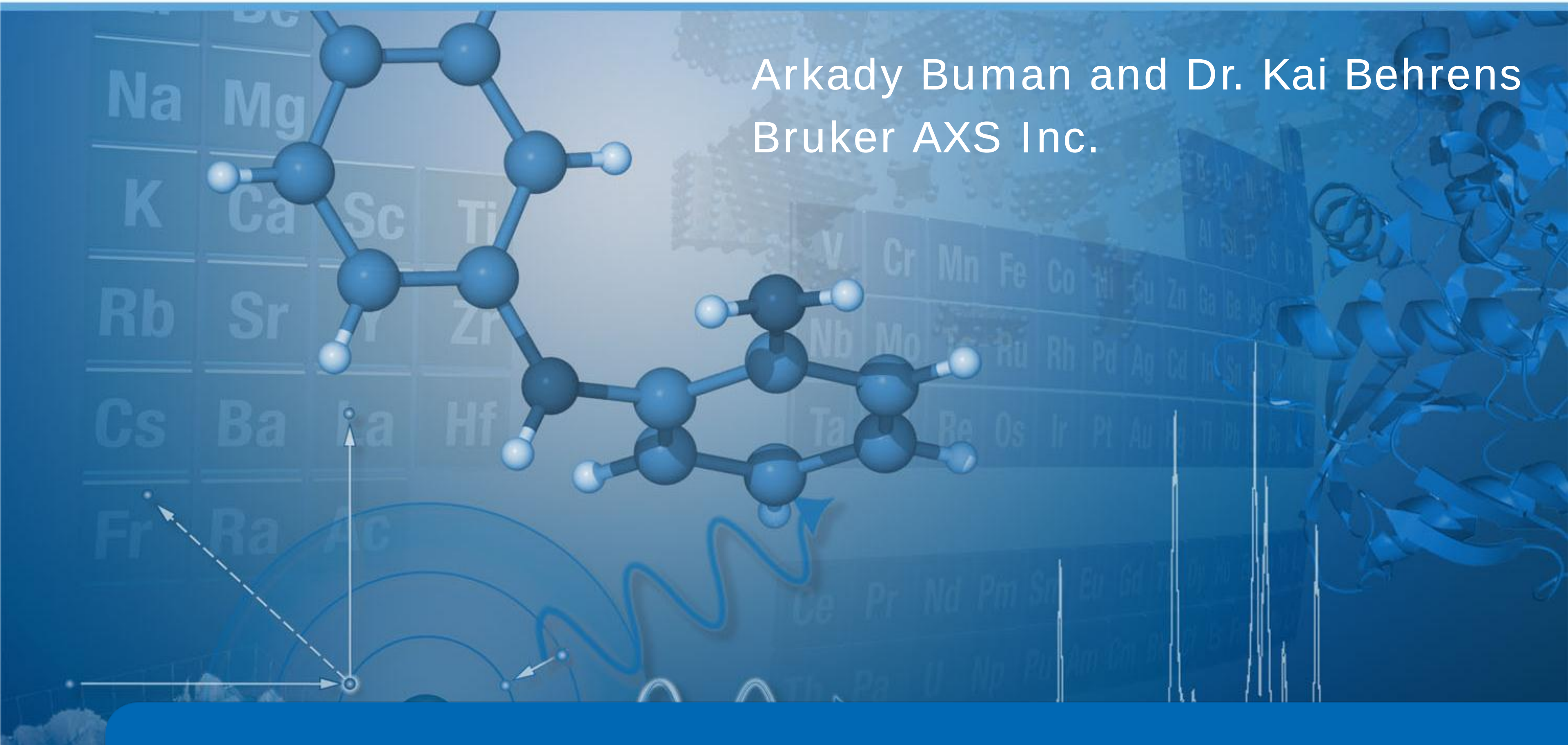
How to Increase Productivity in Mining with Automated Simultaneous XRF Analysis



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Today’s Speakers

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How to Increase Productivity in Mining with Automated Simultaneous XRF Analysis

- Mining economy
  - Outlook and trends
- The role of X-ray fluorescense (XRF) in mining operations
  - Basics in instrumentation and benefits for mining operations
  - Optimal instrumental setup for mining laboratories
- Process and quality control in mining operations
  - XRF applications in a mining lab
- Automation setup for optimal results
- Summary
- Q & A

Mining
Economic Trends Outlook

- The increasing urbanization in Asia (China, India) and Africa leads to higher demands for
  - base metals (iron, copper)
  - precious metals
  - rare earth elements
  - coal
- Global demands for copper and other valuable resources will increase
- Supply will not match worldwide need
  - Less high-grade resources
  - Lower grade, but more complex mineralogy in existing mines
  - Only small numbers of new mines

The Modern Mining Laboratory
Changing Requirements

- Resources with lower grades and more complex mineralogy will require
  - Closer monitoring of the mining process
    - Increasing number of samples from exploration
    - Monitoring of production drilling to steer exploitation direction
  - More analytical work to be done to increase mine efficiency

- Higher demand for evaluation of new mining sites

The Modern Mining Laboratory
Changing Requirements

Expected changes in the workforce:

- Smaller number of experienced staff available
- More mines in remote areas: availability of trained laboratory workers limited

- At the same time the demand for analytical quality results is increasing under challenging conditions

- The logical trend:
  - Increased use of XRF
  - Higher demand for automated laboratories

Why “Elements” Matter

Wide range of XRF applications for

- Geological surveys
- Mining: exploration and exploitation
- Industrial minerals
- Raw materials for
    - Cement
    - Ceramics, refractories, glass
    - Catalysts, chemicals, fodder
    - Metals

Analysis of major and minor elements as oxides

- for grade control and product quality (purity) based on fused beads

Analysis of traces

- for purity control, geological and environmental mapping based on pressed pellets

X-Ray Fluorescence Analysis (XRF)

Basic principles and applications

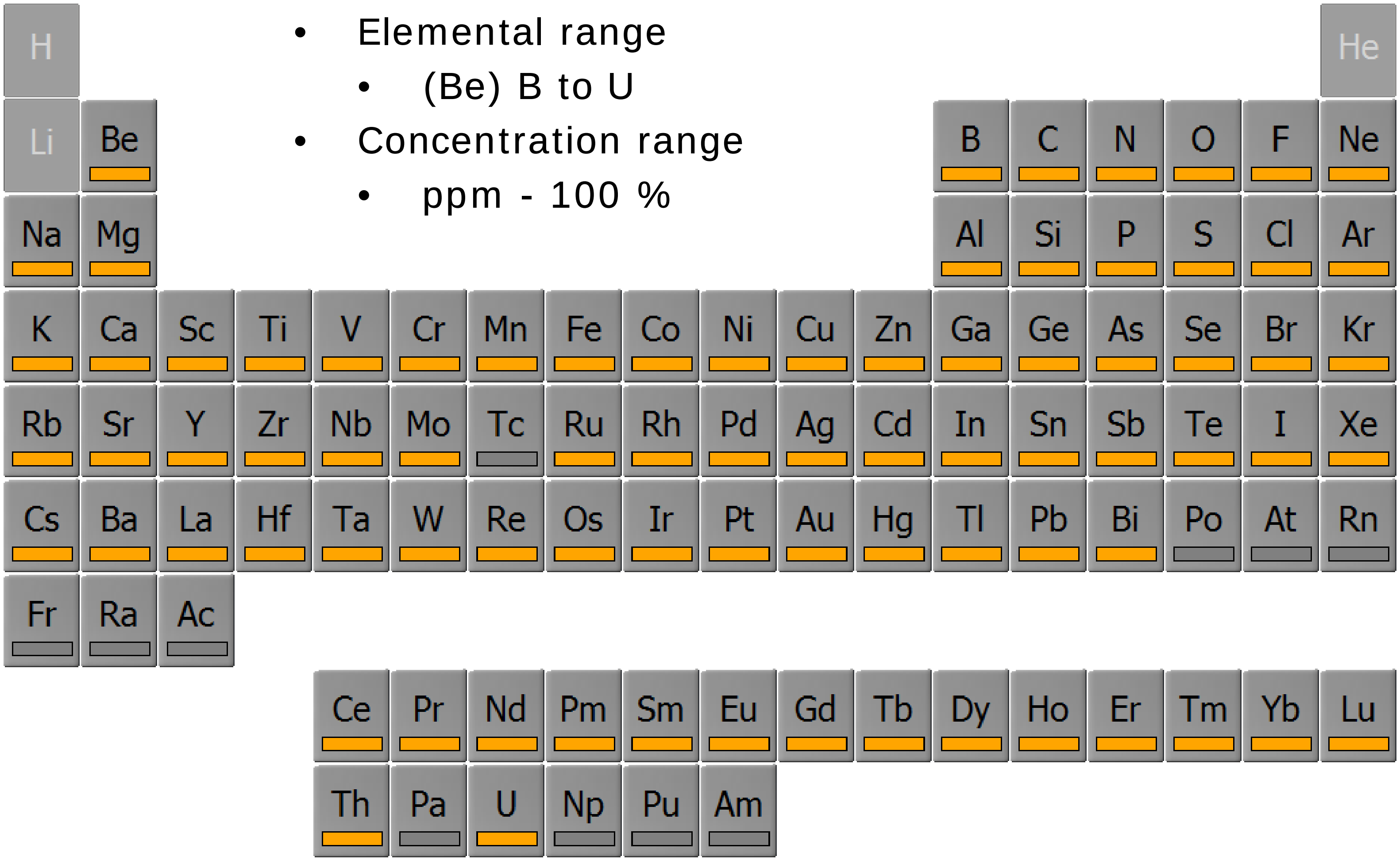
X-ray fluorescence analysis (XRF)
Definition

X-ray fluorescence analysis (XRF) or X-ray spectrometry

- A method for qualitative and quantitative analysis of the elemental composition by excitation of atoms and detection of their characteristic X-rays

X-ray fluorescence analysis (XRF)
Definition

- Elemental range
  - (Be) B to U
- Concentration range
  - ppm - 100 %



XRF X-ray Fluorescence Analysis
X-ray Spectrometry

- ... is the method for qualitative and quantitative analysis of elemental
- composition by excitation of atoms and detection of their characteristic
- X-rays: one form of "Electromagnetic Radiation"

Energy [keV]	Wavelength	Description
< 10-7	cm to km	Radio-waves
< 10-3	µm to cm	Microwaves
< 10-3	µm to mm	Infrared
0.0017 - 0.0033	380 to 750 nm	Visible light
0.0033 - 0.1	10 to 380 nm	Ultraviolet light
0.1 - 100	0.01 to 10 nm	X-rays
10 - 5000	0.0002 to 0.12 nm	Gamma radiation

X-ray fluorescence analysis (XRF) Capabilities

- Qualitative Analysis
  - Identification of elements
  - ”What’s inside?”

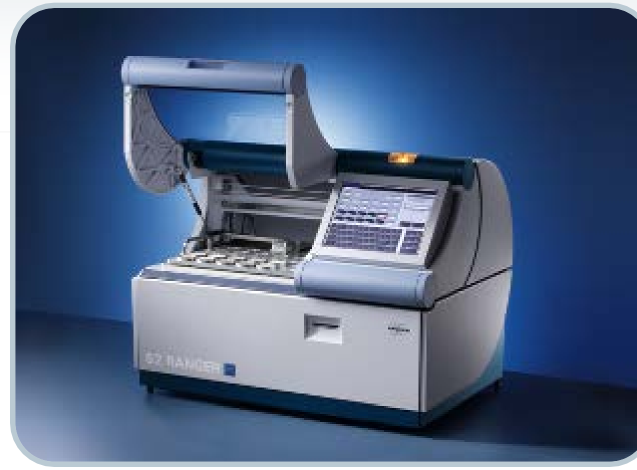
- Quantitative Analysis
  - Determination of concentrations
  - ”How much is inside?”

- Semi-Quantitative Analysis
  - Estimation of concentration
  - ”About how much?”

X-ray fluorescence analysis (XRF)
Bulk-XRF Product Line

S8 LION
S8 DRAGON

S8 TIGER



S2 RANGER

Wavelength-Dispersive
(WDXRF)

Energy-Dispersive
(EDXRF)

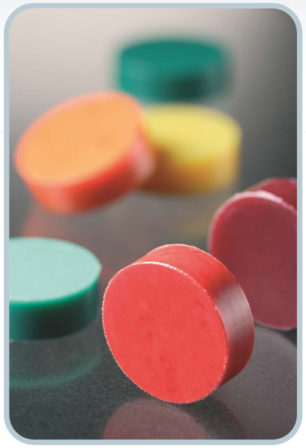
Simultaneous

Sequential

X-ray fluorescence analysis (XRF) Capabilities

Samples measured as

- Liquids
  - Directly
- Powders
  - Directly
  - As pressed pellets
  - As fused beads
- Bulks
  - Directly, after fitting into sample cups

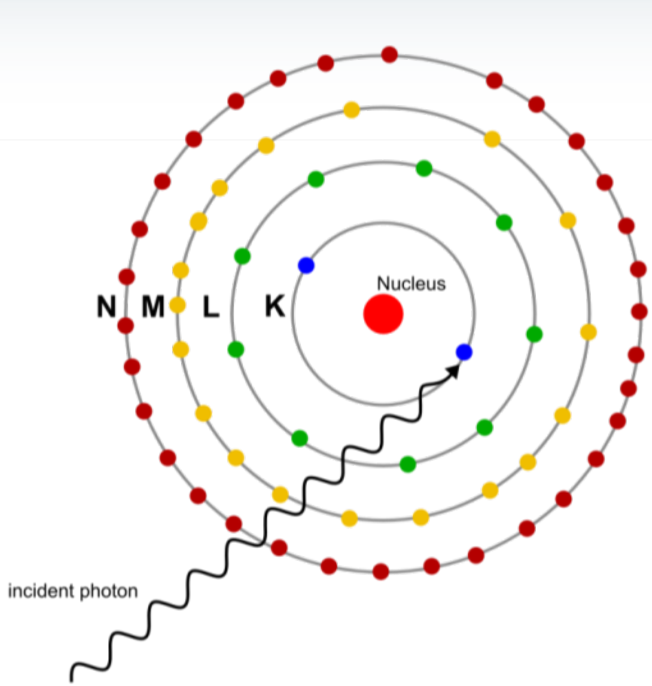



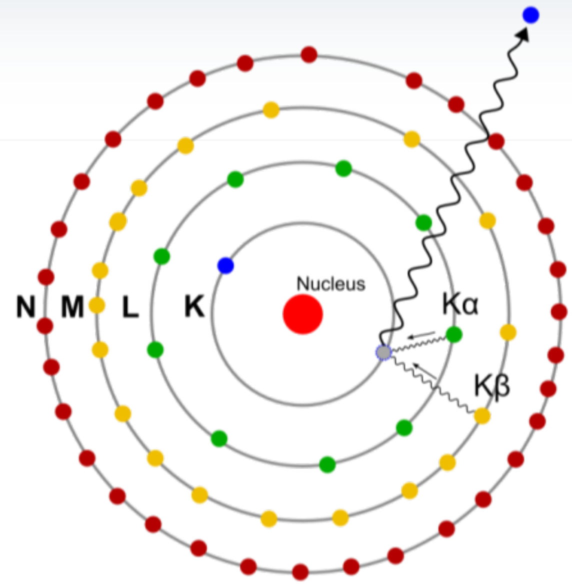


X-ray fluorescence analysis (XRF)
Advantages of XRF

- Solid and liquid samples can be analyzed directly: large range of applications
- Little or no sample preparation required
- Analysis is non-destructive (for the sample)
- Sampling-analysis result time is relatively short
- Quantitative and qualitative analyses are possible
- Accuracy and long term stability
- Elemental range: (Be) Na to U
- Linearity from ppm to 100%

X-ray fluorescence analysis (XRF)
Principle – Photoelectric effect

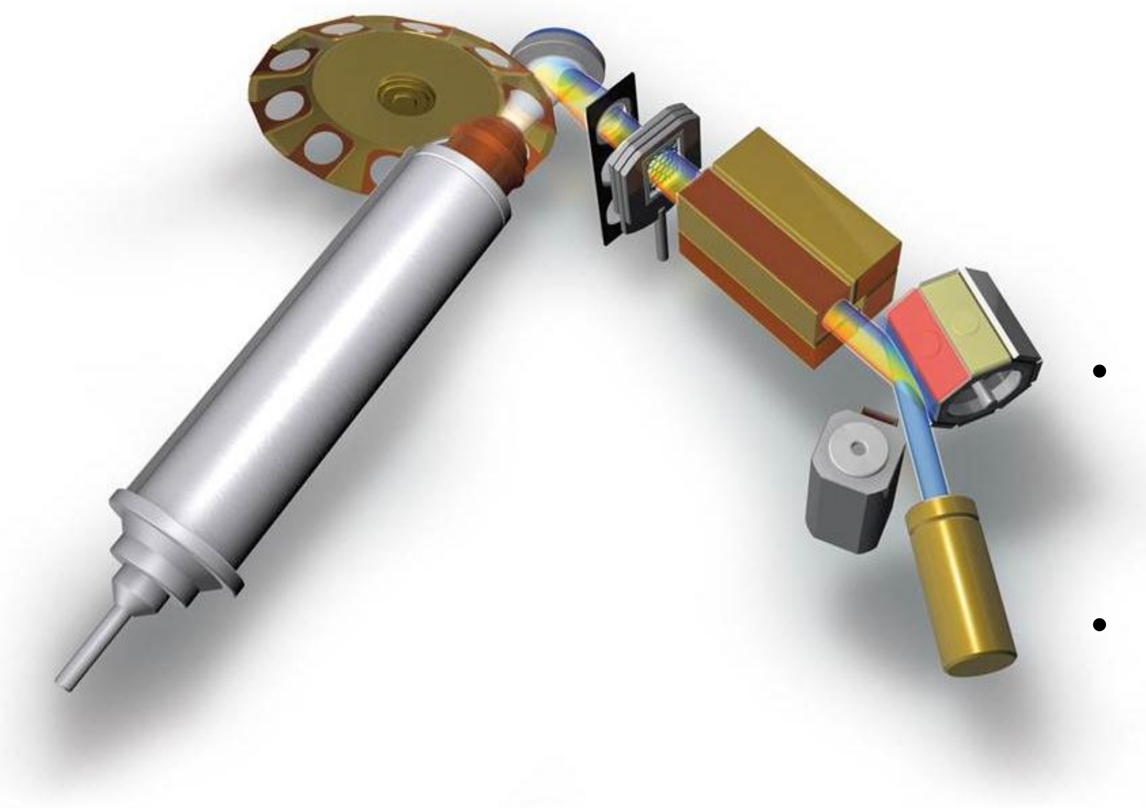




- Sample excited with a x-ray beam causing fluorescence
- Electron ejected from an inner shell of its atom
- Electron from a shell farther out falls into the vacancy
- Energy difference is emitted as X-ray photon

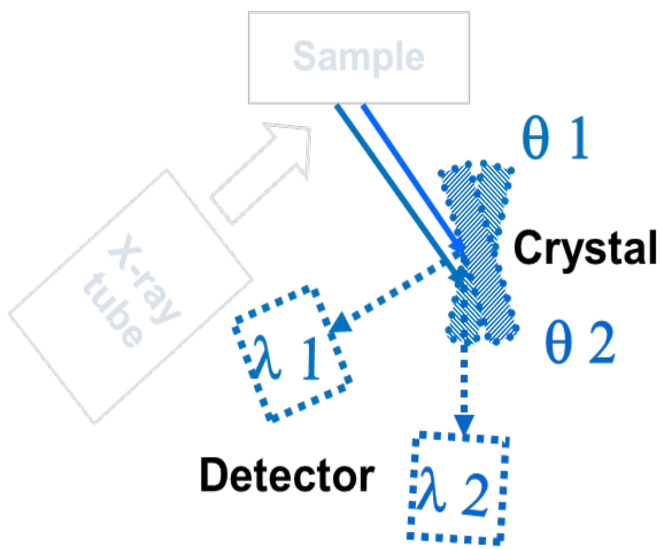
- Discrete energy or wavelength is characteristic for the emitting element
- Intensity of characteristic radiation is proportional to concentration of the element in the sample

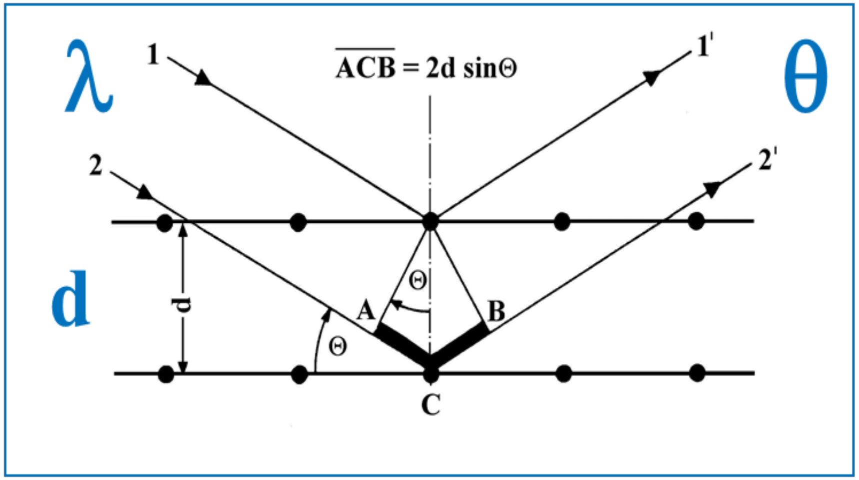
X-ray fluorescence analysis (XRF)
WDXRF – sequential



- X-rays produced by a tube are directed to the sample
  - Causes sample to produce X-rays that are characteristic of the atoms (elements) present
- Analyzer crystals are used to separate the X-rays into their individual components
- Detectors are used to convert the X-ray energy into an electrical pulse that is counted

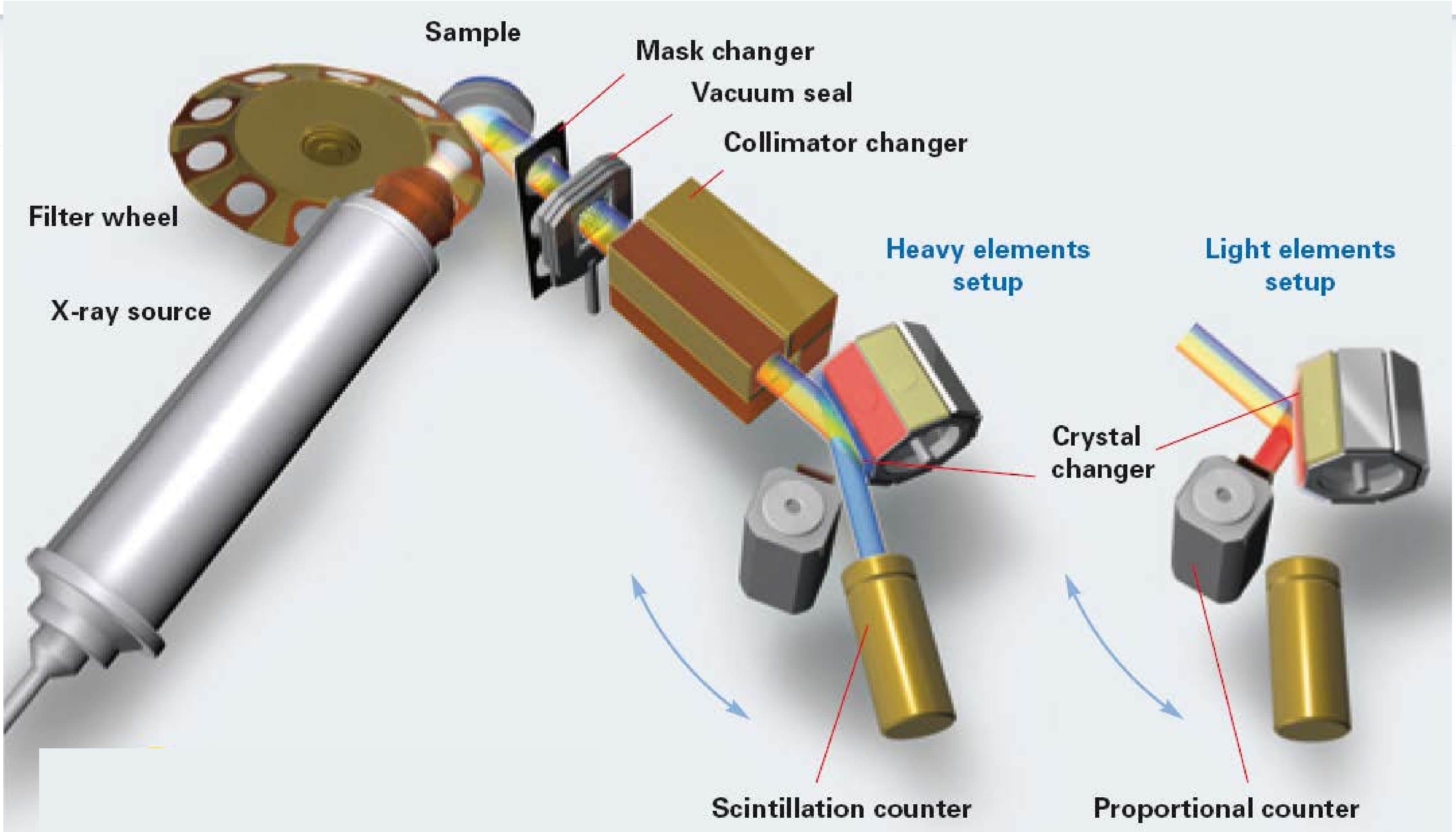
Wavelength-dispersive XRF (WDXRF)
Analyzer Crystal and Bragg´s Equation



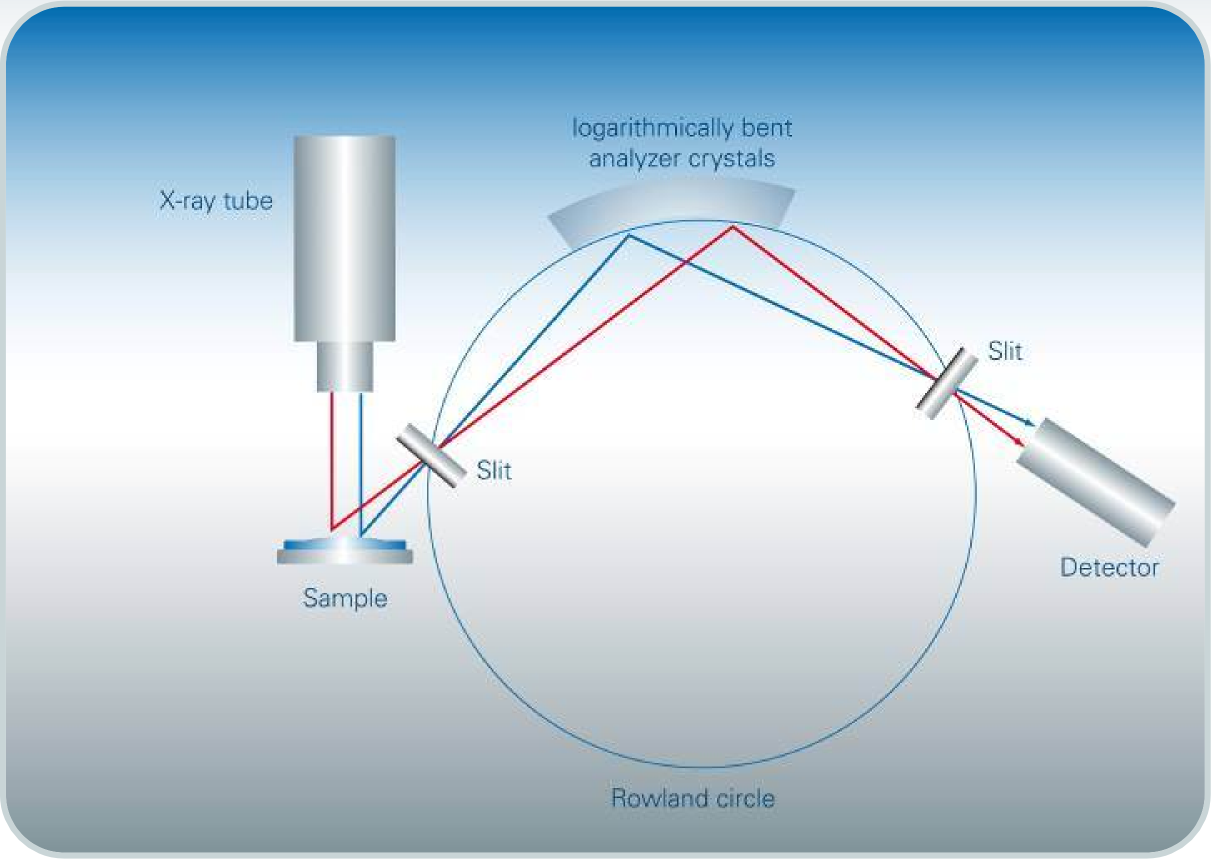


$$n\lambda = 2d\sin\Theta$$

WDXRF Sequential Spectrometer
Beam Path



S8 DRAGON
Rowland Circle Geometry



S8 DRAGON: Double slit beam path for high resolution with curved crystals

S8 DRAGON

- Designed for
  - Best analytical precision
  - Lowest detection limits
  - High analyzing speed
  - Fast response
  - High sample throughput

  - Unique combination for analytical flexibility and analytical data safety with the Multielement Channel

S8 DRAGON SampleCare™

Maximum uptime and lowest maintenance costs due to safe sample handling

Tube-above-sample geometry:

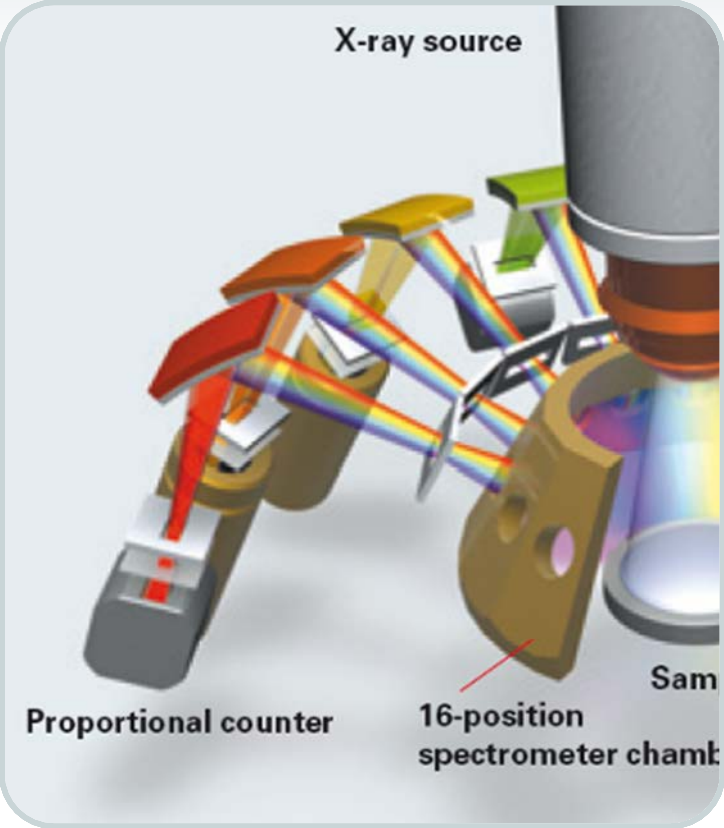
- No contamination and damage of the X-ray tube window
- No damages of flow counter foils

The S8 DRAGON is not affected by:

- Sample failures
- Dust from pressed powder samples

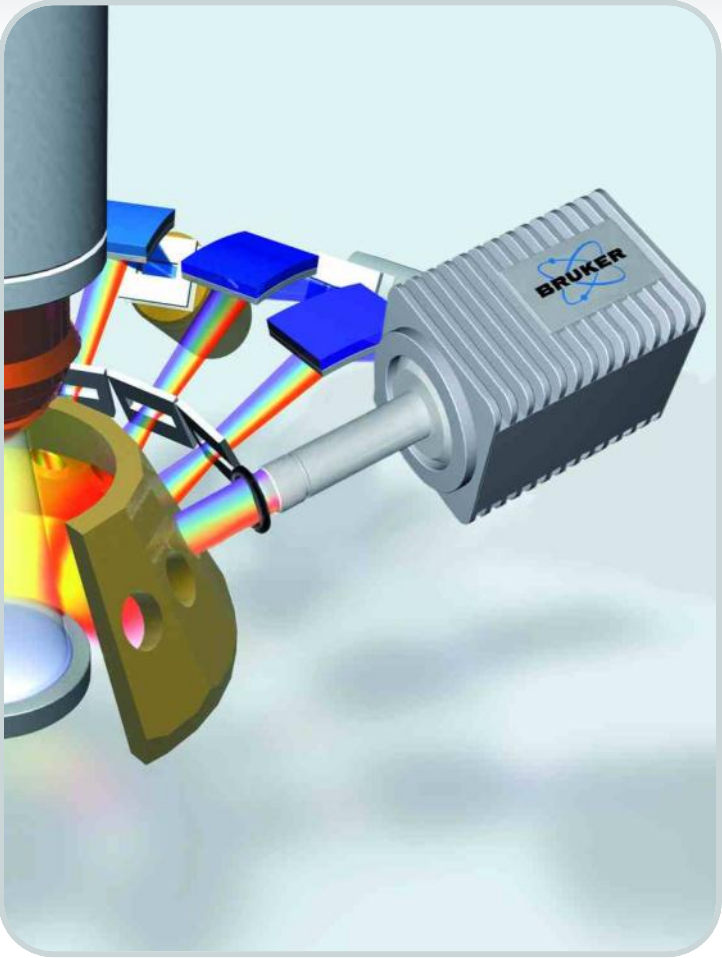
- Reliability by design

S8 DRAGON
The optimal solution - Less Is More



- Best analytical performance with max 15 channels plus Multielement Channel
- Highest intensity due to most compact beam path (close coupling of tube-sample-detector)
- Stable vacuum due to small volume of sample and spectrometer chamber with pre-evacuation step
- Less channels offer more performance

S8 DRAGON
Multielement Channel



- Essential benefits of the unique Multielement Channel ™

- Elemental fingerprinting
  - Identification and analysis of all elements from Na upwards
- Analytical flexibility
  - Contaminations can be traced
  - Analysis of non-routine samples
  - Upgrading of analytical methods with additional elements in minutes – no further installation of new hardware
- Dual-mode data acquisition
  - Internal backup for data safety with a second internal source

S8 DRAGON
Multielement Channel

XFlash technology

- 4th generation Silicon Drift Detector (SDD)

High-transmission window

- Unique energy resolution
- 129 eV FWHM
- @ Mn $K\alpha$
- @ 100,000 cps

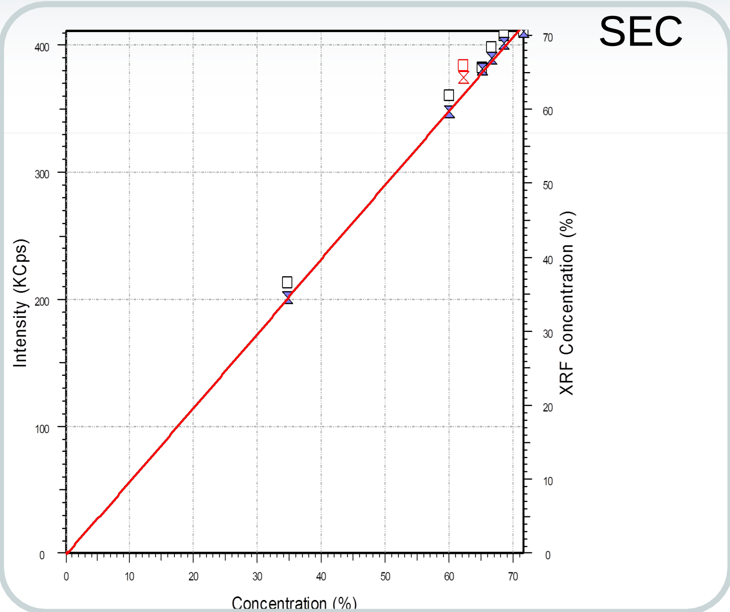
Unmatched count rates

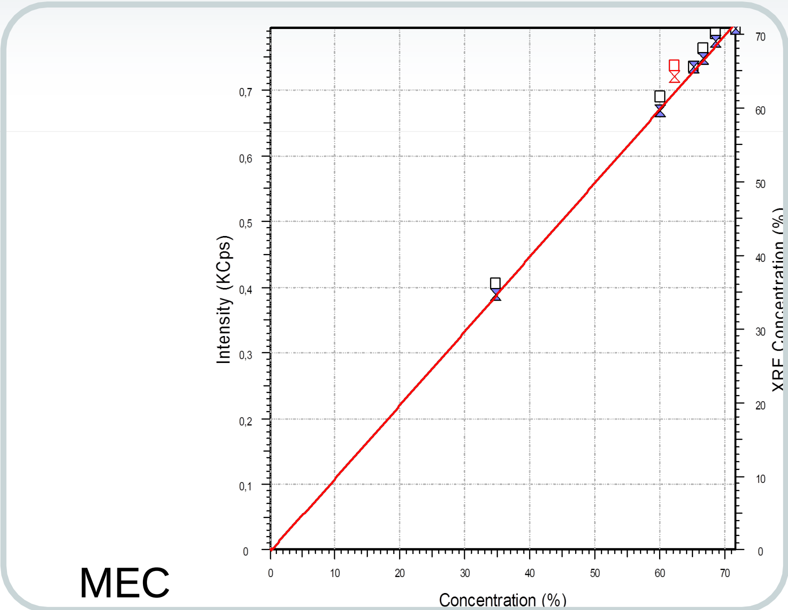
- up to 300,000 cps input count rate
- up to 100,000 cps output count rate
- without resolution degradation

Peltier cooled

- Maintenance free

More information on the Multielement Channel MEC





Calibration Details for Fe: Range 34.67 -71.50 %

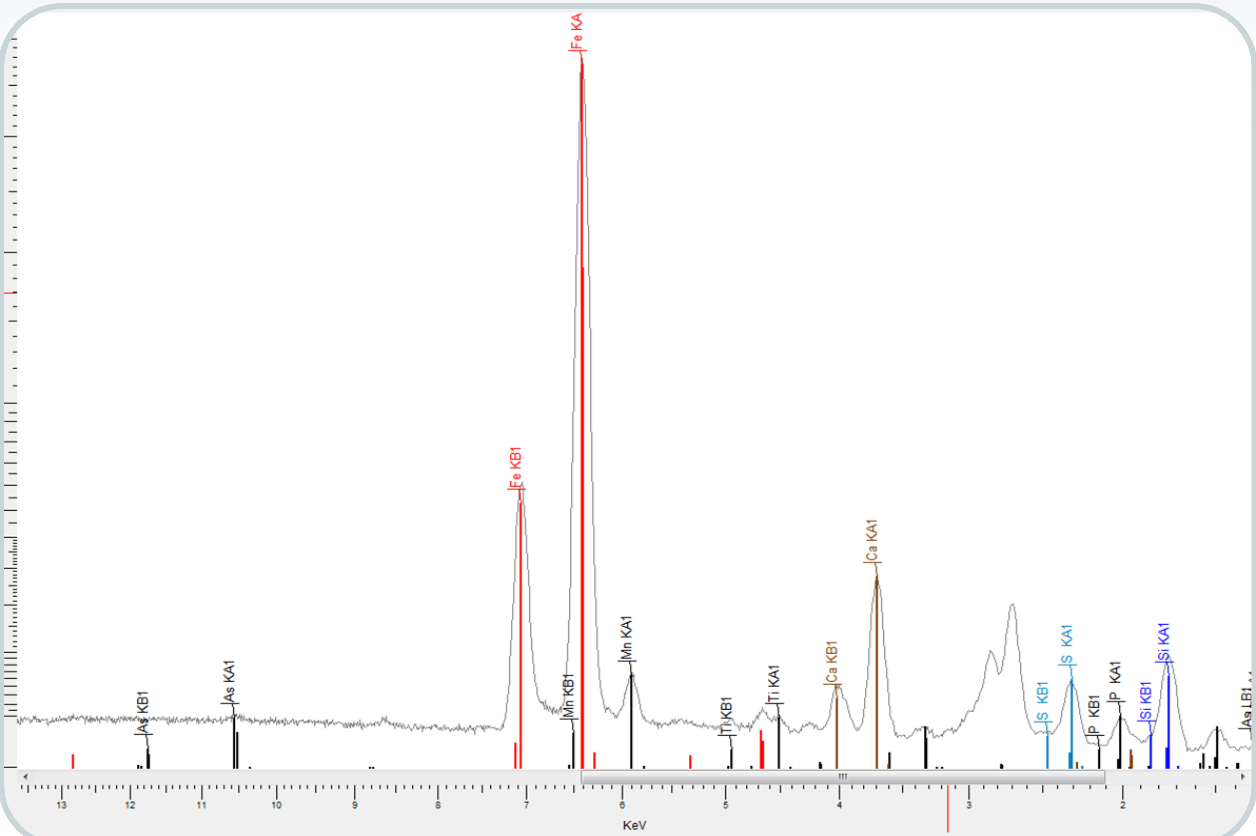
Fe (Single Element Channel) – LiF 200:

Calibration Std. Dev.: 0.563 %

Fe (Multielement Channel) – SDD:

Calibration Std. Dev.: 0.503 %

Spectrum Iron Ore
Dual Mode Data Acquisition



Parallel measurement of SEC (WDX) and Multielement Channel
Identification of trace elements, internal backup (second information source)

Sample preparation for XRF analysis

Preparation of Powders

Grind material to suitably small uniform particle size

- Aids binding of material when pressing pellets
- Aids dissolution when fusing samples
- Allows several particle layers to be measured

Prepare ground material as

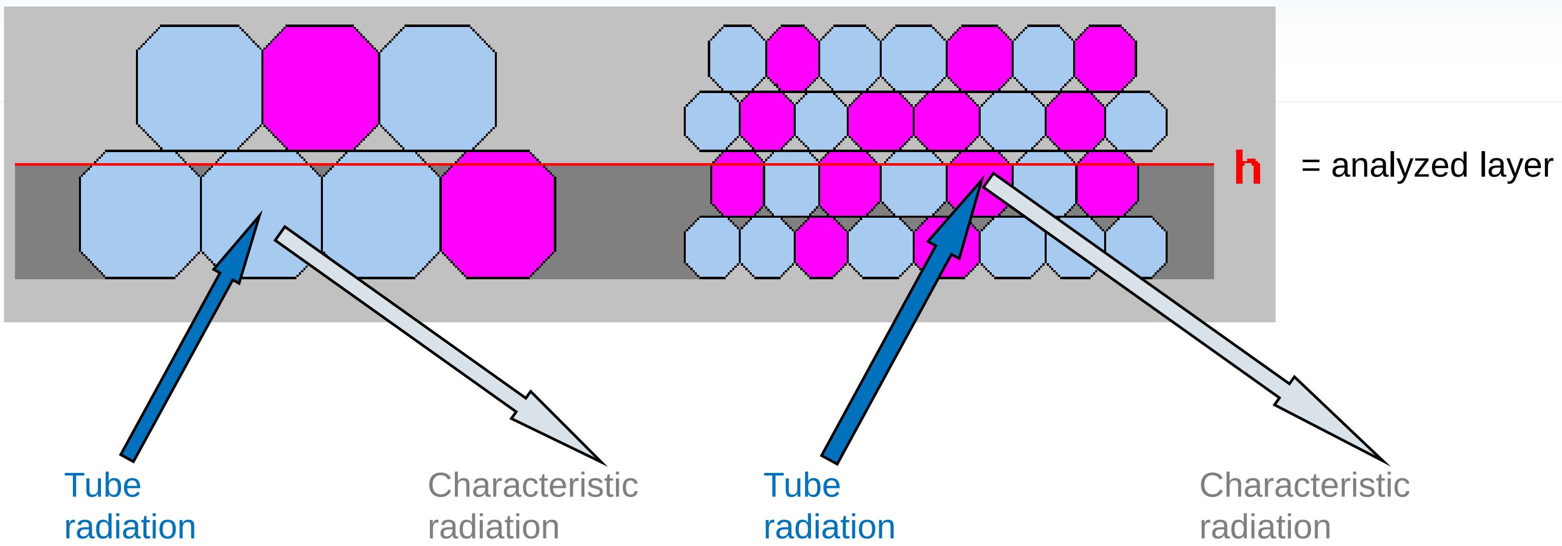
- Loose powder
- Pressed pellet
- Fused disc

If samples are being pressed into pellets. it may be necessary to add a binder to the material to make it ”stick“ together

Typical binders are:

- Cellulose
- Wax

Preparation of Powders - Grinding

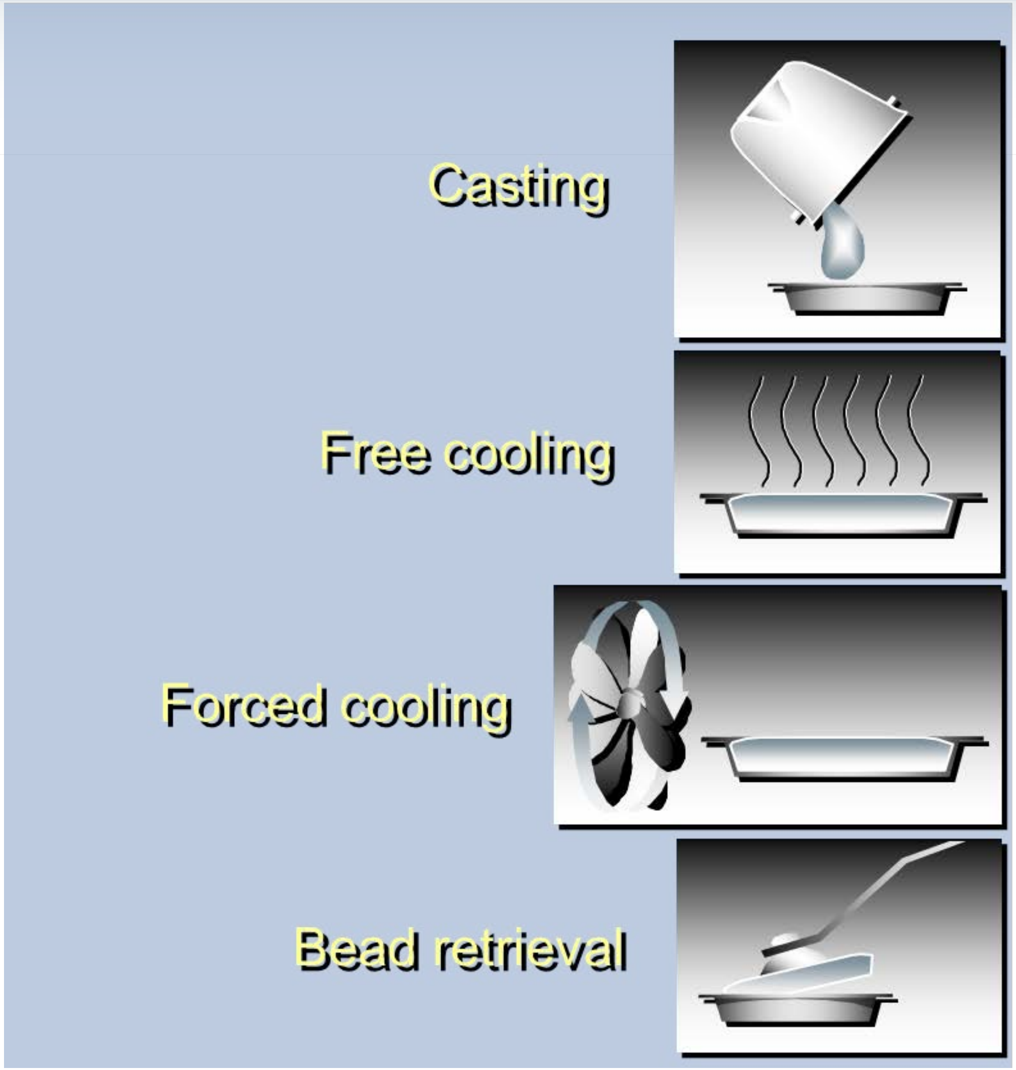


- Sample should be ground to uniform particle size
- Ideally the particle size should be much smaller than the analyzed layer depth

The Other Way…

- What if there are no matrix-matched standards available or no standards available at all?
- What if there are no mineralogical-matched samples?
- Bad sample homogeneity in pellets
- Higher accuracy needed
- Larger “calibration ranges” needed for materials and flexibility
- Traceable analysis to certified reference materials

Higher Accuracy
No grain size effects - Fusion



CLAISSE

Procedure
Repeatability of Sample Preparation

- Regardless of what method you use, it has to be repeatable
- Before the standards are prepared, use your own material and prepare seven samples of the same material

Procedure
Repeatability of Sample Preparation

- Regardless of what method you use, it has to be repeatable.
- Before the standards are prepared, use your own material and prepare seven samples of the same material.

Prepare seven samples of the same material (your own iron ore, for example).
Analyze them on the instrument in the following order:

- 1st sample 2nd sample
- 1st sample 3rd sample
- 1st sample 4th sample
- 1st sample 5th sample
- 1st sample 6th sample
- 1st sample 7th sample

Procedure
Repeatability of Sample Preparation

- Do statistical analysis of measurements of the seven different samples of the same material. The first column, where we measure the same sample will give you the instrument repeatability. The second column gives you the instrument and sample preparation repeatability combined. The difference between them is your sample preparation error.
- It is relatively easy now to see how repeatable your sample preparation is and it is even with the best methods 6-8 times higher than the instrumentation error, usually higher than that.

Procedure
Repeatability of Sample Preparation

All previous examples are for manual sample preparation and it is clear that sample prep is the major contributing factor to the variation of the results.

- Now with this data available it is time to make the decision:
    1. Accept the limitations of your manual sample prep method
    2. Go to automation sample preparation

Iron Ore
Rapid grade control

- Rapid control of element concentration
- Quality check
  - at the mining site
  - at shipping stations

Narrow concentration range and similar matrix allows pressed pellet preparation

- Quickest sample preparation:
- Crushing, milling, pressing with cellulose as binder
  (15 g sample plus 2 g binder)

- Maximum 60 s measurement time

- Simple automation path, high sample throughput, easy operation, fast feedback

S8 DRAGON Configuration

- Fe Kα, LiF200, scintillation counter, 95 % attenuator
- Mn Kα, LiF200, scintillation counter
- Si Kα, XS-CEM, flow counter
- P Kα, Ge, flow counter
- Al Kα, XS-CEM, flow counter
- Ca Kα, LiF200, flow counter
- Mg Kα, XS55, flow counter
- Ti Kα, LiF200, flow counter
- K Kα, LiF200, flow counter
- Co Kα, LiF200, scintillation counter
- Ni Kα, LiF200, scintillation counter
- Pb Lβ, LiF200, scintillation counter

- Multielement Channel

S8 DRAGON
Iron Ore – Pressed Pellets

Measurement:

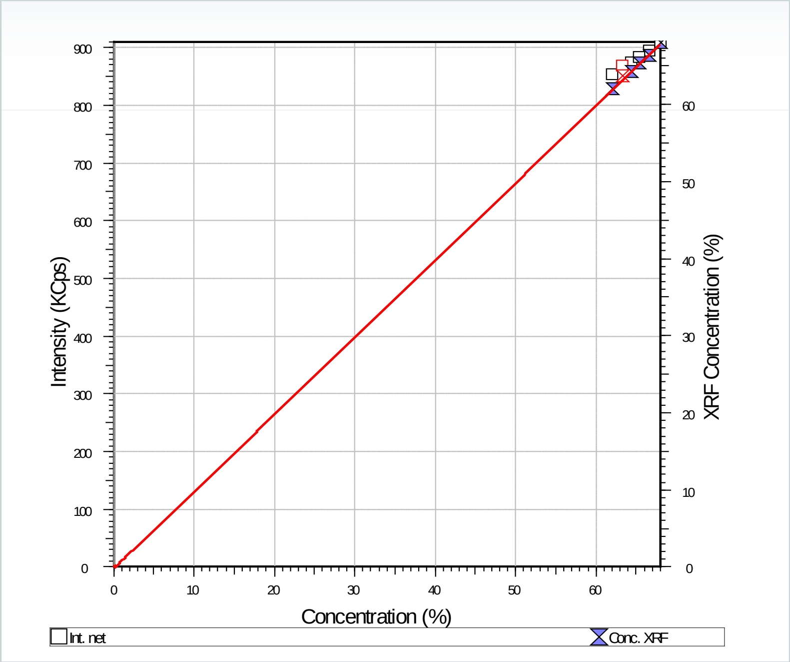
- 40 kV, 100 mA – Full 4 kW excitation

Configuration:

- Single Element Channels plus Multielement Channel

Elements

Fe, MgO, Al_2O_3, SiO_2, P, K_2O, CaO, TiO_2, Mn

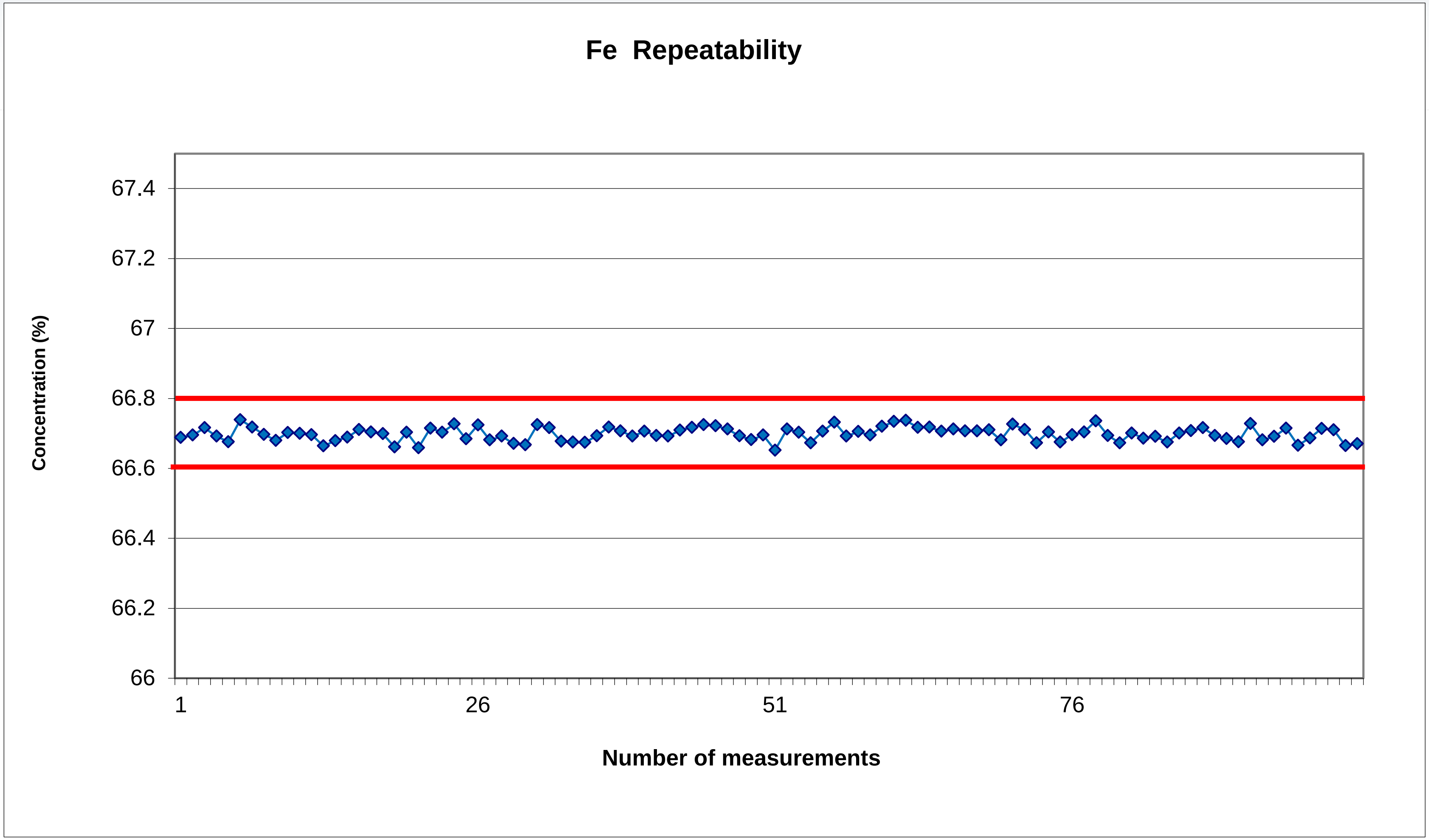


Fe calibration curve

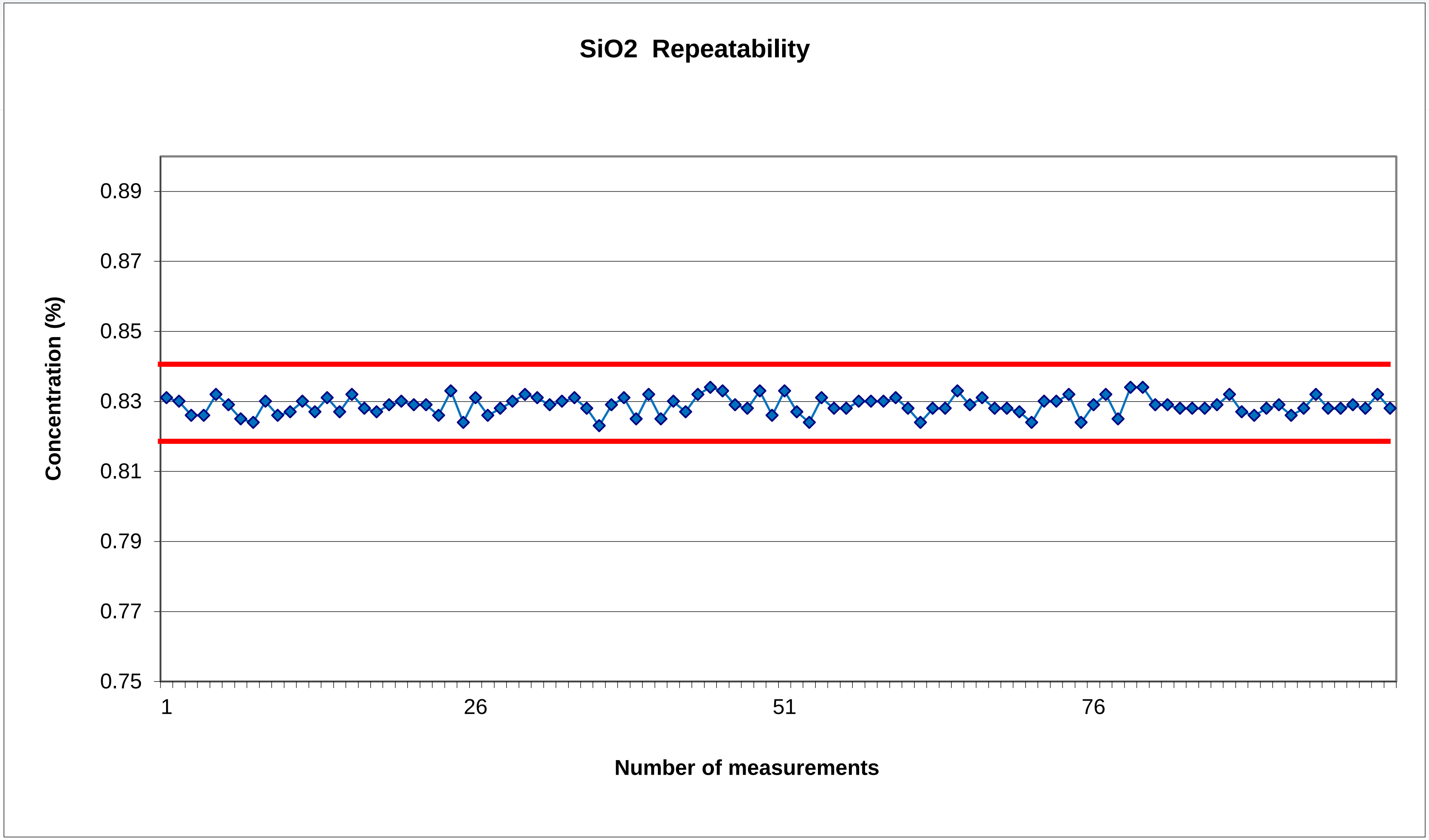
Calibration Details:

	Min [%]	Max [%]	Abs. Cal. Std. Dev. [%]	LOD [3 s. ppm]
Fe	62.0400	67.9400	0.0931	87
SiO2	0.5500	5.0000	0.0560	9
Al2O3	0.5400	1.2816	0.0317	22
P	0.0285	0.1960	0.0014	0.3
Mn	0.1000	1.3150	0.0155	100

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



Precision Test on SiO_2 in one week
200 measurements: 0.829 +/- 0.002



Performance Test
200 measurements - Summary

Sample	Average (%)	Abs. Std. Dev. (%)	Rel. Std. Dev. (%)
Fe (%)	66.698	0.020	0.029
SiO2 (%)	0.829	0.003	0.312
P (%)	0.040	0.000	0.368
Al2O3 (%)	0.902	0.004	0.422
Mn (%)	0.673	0.014	2.026
CaO (%)	0.013	0.001	4.003
MgO (%)	0.032	0.000	1.351
TiO2 (%)	0.053	0.001	2.319
K2O (%)	0.017	0.000	0.833

Iron Ore
Pressed Pellet

Quick grade control at mining labs:

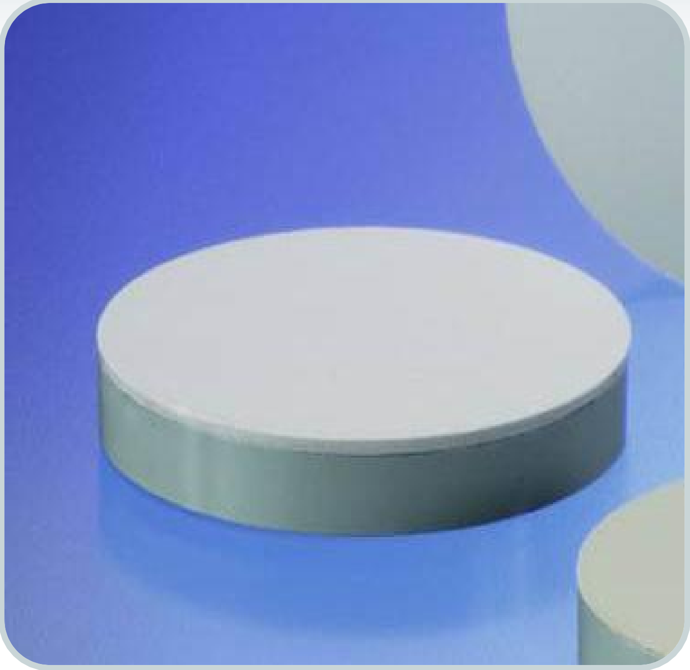
- Simple and fast sample preparation
- for narrow element ranges

- Maximum measurement time:
  - 60 s

- Typical measurement time:
  - 40 s

- Analytical precision:
  - Fe: **66.698 +/- 0.020 %**
    Rel. Std. Dev. 0.029

- Detection limit for traces:
  Si at 9 ppm
  P at 0.3 ppm



Iron Ore
Shipping Control

- Iron ore grade control in accreditied commercial service labs / customs / central labs
- Wide concentration ranges with huge variety of different matrices
- Matrix correction according ISO 9516
  - Loss corrected alphas
  - Variable alpha model
- Fused bead preparation
  - Handling of grain size effects
  - Sample dilution
  - Best accuracy

S8 DRAGON
Iron Ore – Fused beads

Measurement:

- 40 kV, 100 mA – Full 4 kW excitation

Configuration:

- Single Element Channels plus
  Multielement Channel

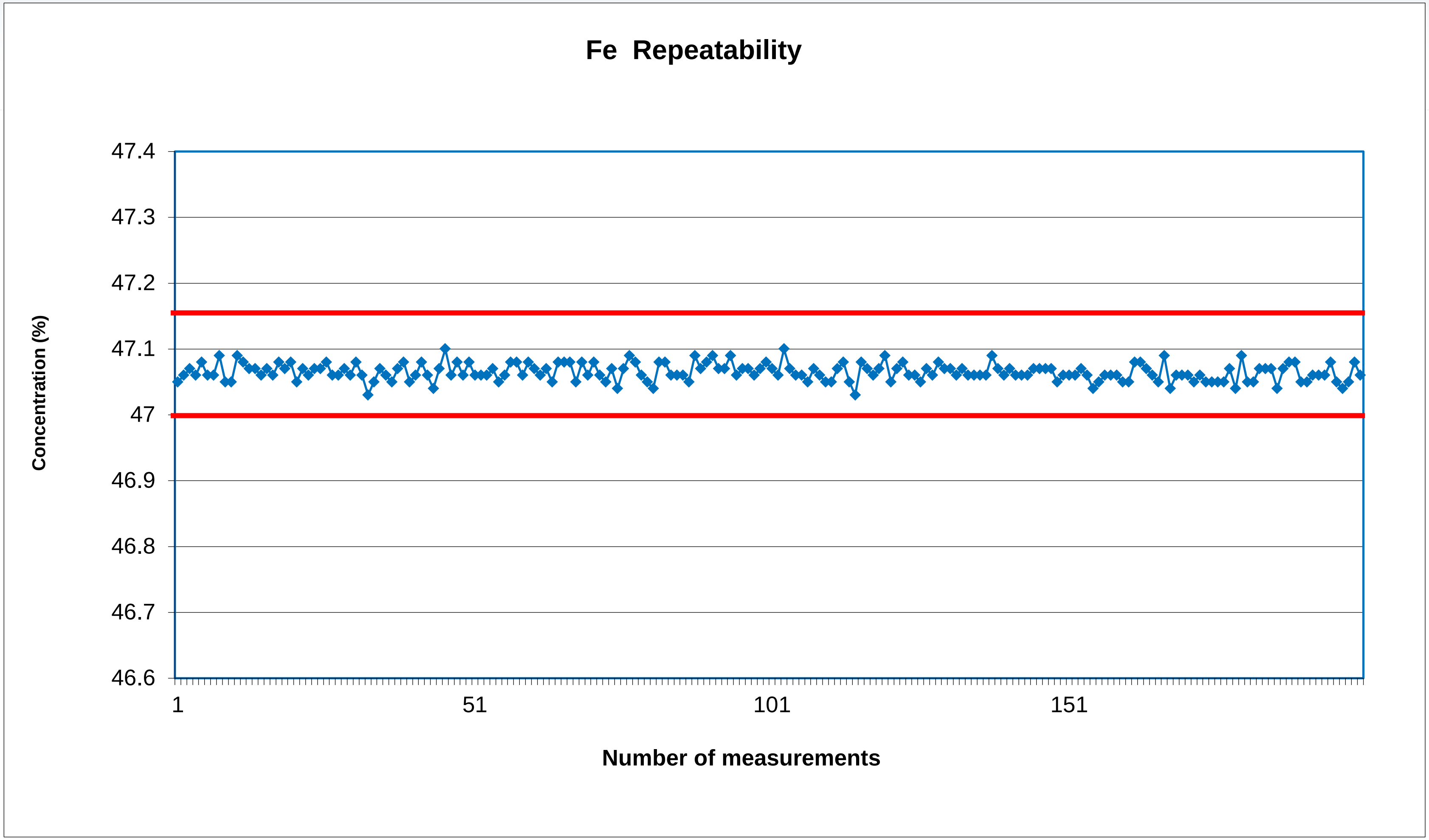
Elements

- Al, As, Ba, Ca, Cr, Co, Cu, Fe, K, Mg, Mn, Mo, Nb, Ni, P, Pb, S, Si, Sn, Ti, V, Zn

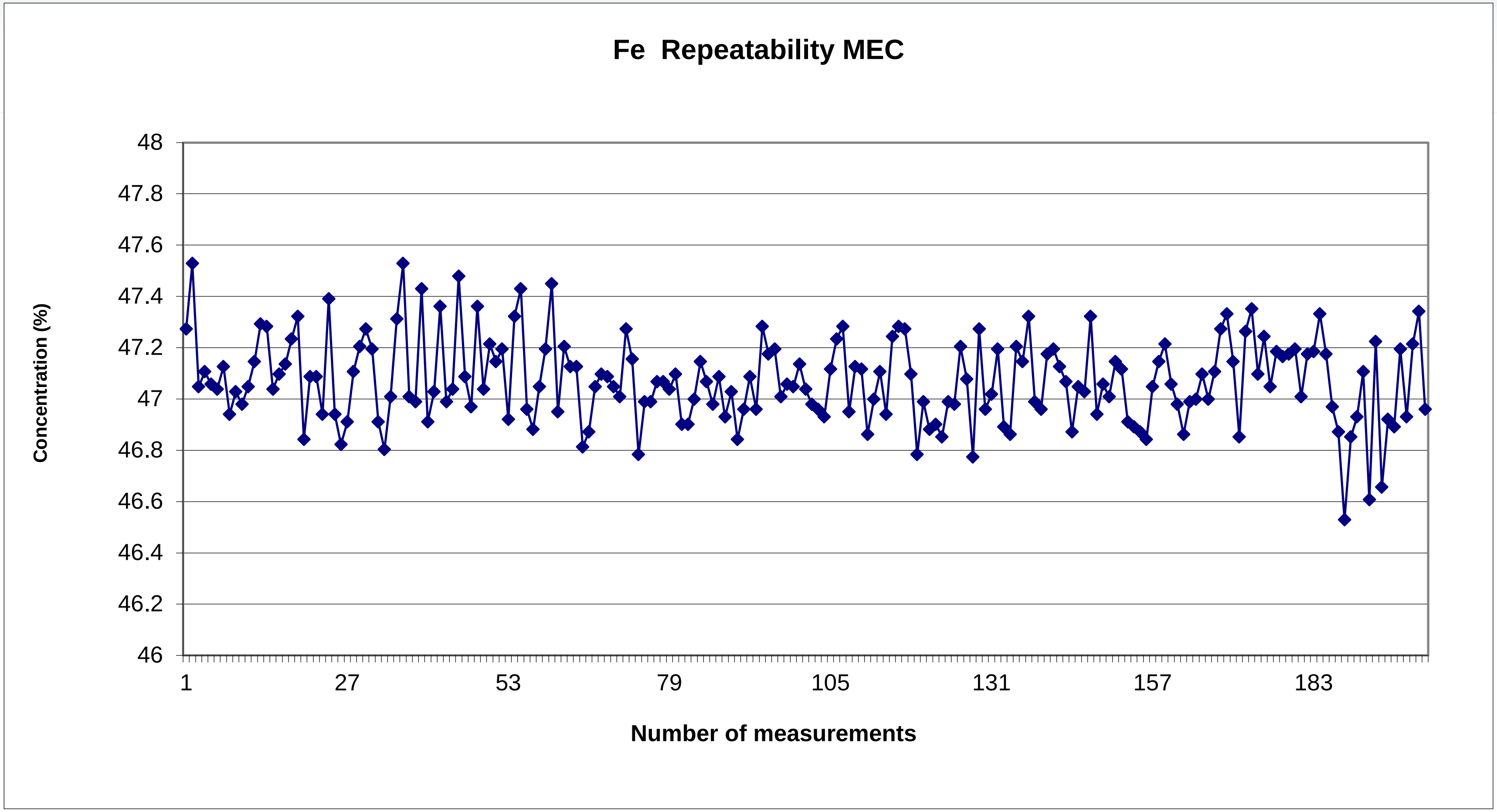
Calibration Details:

	Min [%]	Max [%]	Abs. Cal. Std. Dev. [%]	LOD [3 s. ppm]
Fe	34.6700	71.5000	0.563	45
SiO2	0.1610	38.5800	0.036	46
Al2O3	0.2080	3.3000	0.0324	85
P	0.0056	0.0432	0.0007	0.3
Mn	0.0130	4.3200	0.0045	1.6

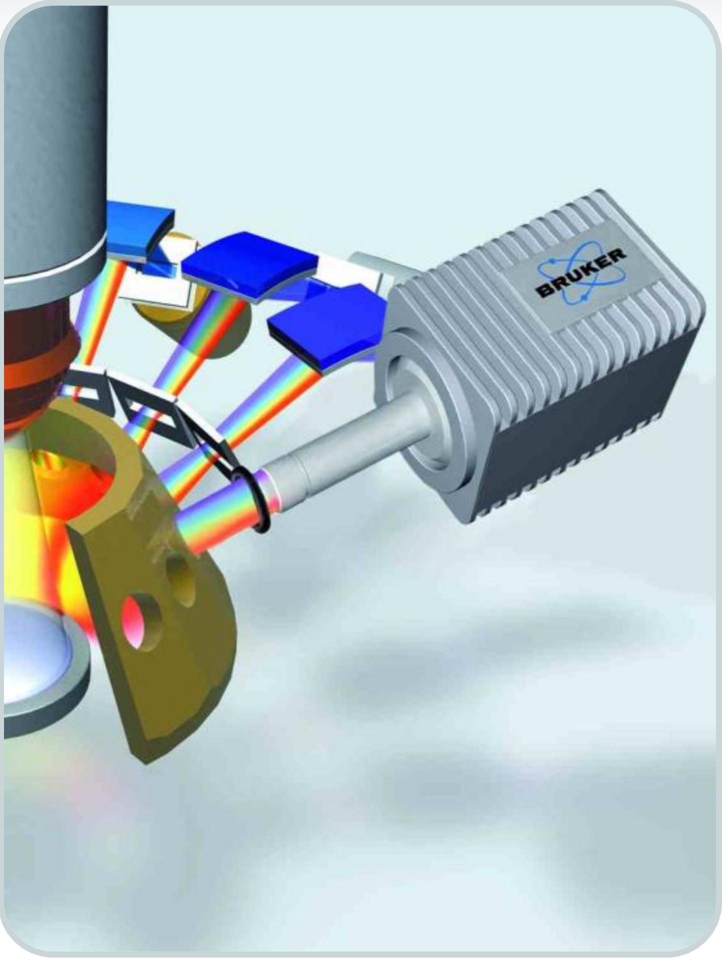
SEC: Precision Test on Fe in one week
200 measurements: 47.065 +/- 0.013



MEC: Precision Test on Fe in one week
200 measurements: 47.072 +/- 0.166



S8 DRAGON
Multielement Channel

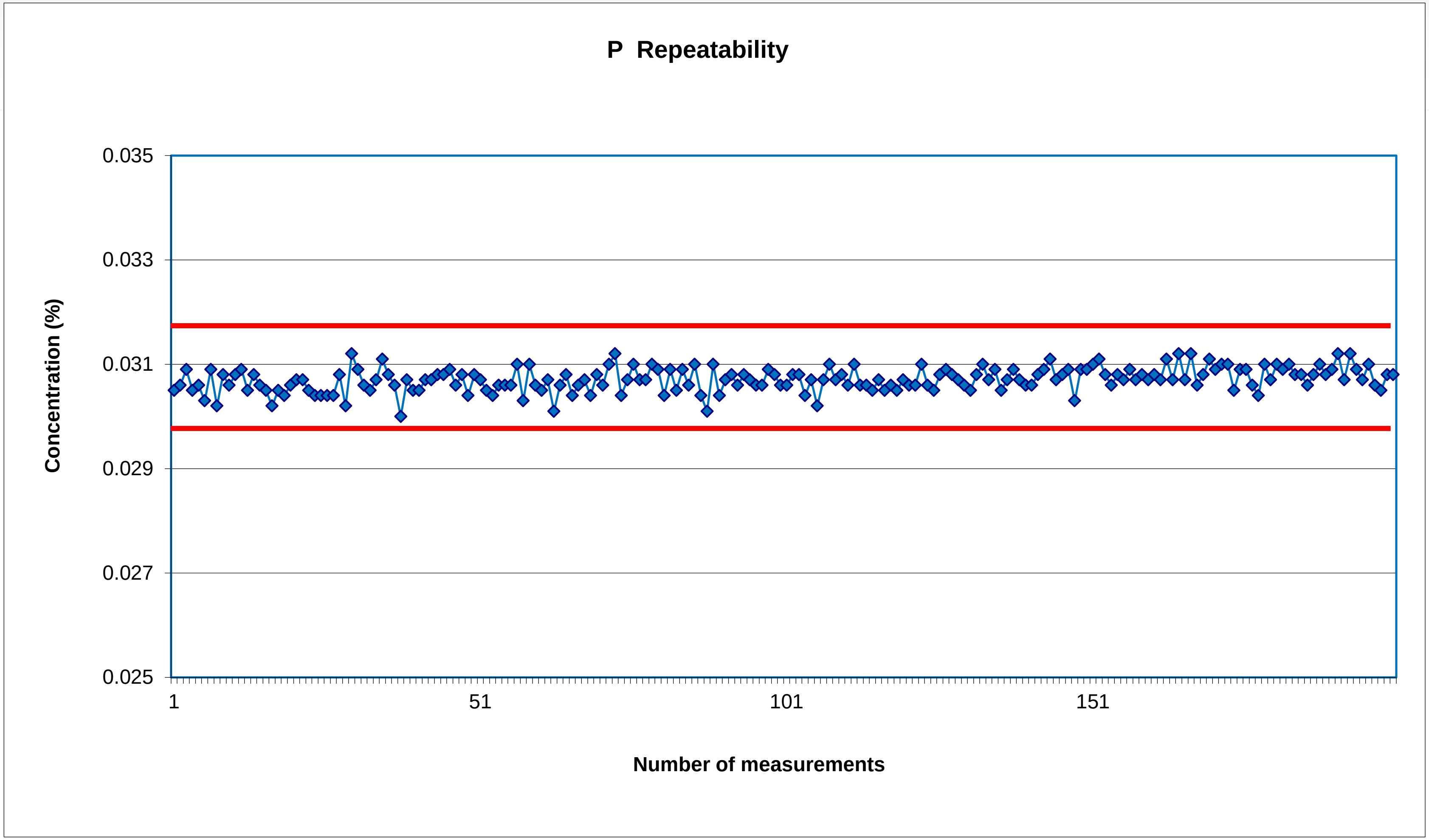


Comparison of SEC and MEC

- Accuracy at the same level between SEC and MEC
- Precision better for SEC
- Therefore, elements for best precision and lowest detection limits to be analyzed with SEC

- Screening and internal backup with MEC
- Every sample has the fingerprint spectra for later evaluation available
- Consistency check between SEC and MEC with every measurement
- New elements are added instantly

SEC: Precision Test on P in one week
200 measurements: 0.031 +/- 0.001



Performance Test in one week
200 measurements - Summary

Sample	Average (%)	Abs. Std. Dev. (%)	Rel. Std. Dev. (%)
Fe (%)	47.065	0.013	0.028
SiO2 (%)	0.673	0.006	0.869
P (%)	0.031	0.001	0.746
Al2O3 (%)	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
TiO2 (%)	0.023	0.003	11.705
K2O (%)	0.160	0.001	0.484

S8 DRAGON
Iron Ore Analysis

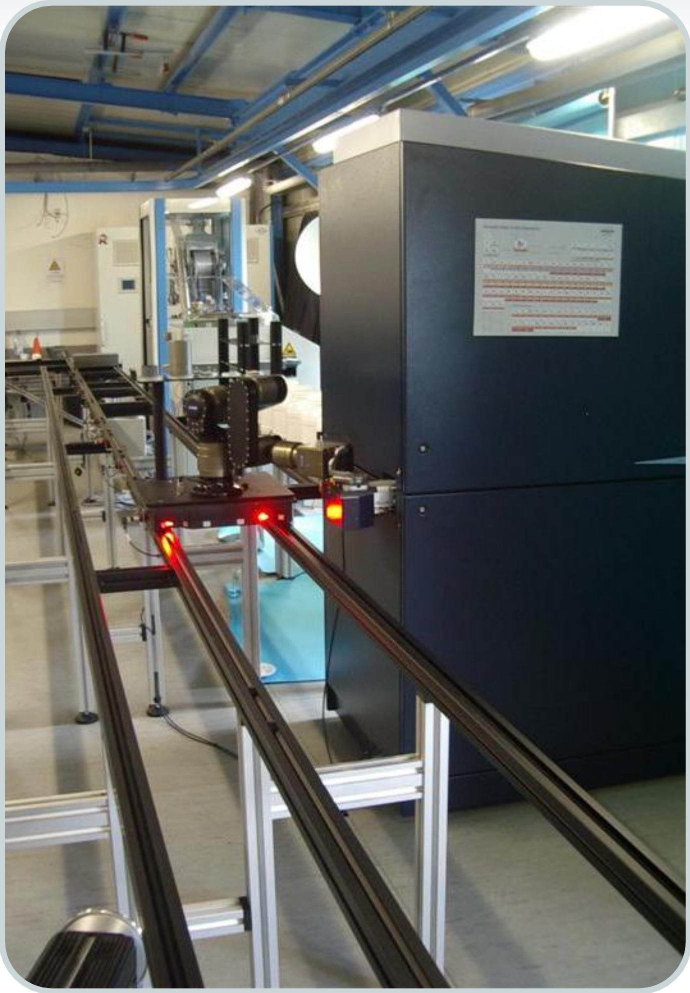
- Designed for
  - Best analytical precision
  - Lowest detection limits
  - High analyzing speed
  - Fast response
  - High sample throughput

  - Unique combination for analytical flexibility and analytical data safety with the Multielement Channel

Typical automation set up of XRF and XRD

Automation line-up
- Sample Preparation
- XRD
- XRF

The modern mining laboratory
Future trends



- High dregree of automation in laboratories
  - Automated sample preparation
    - Crushing
    - Milling
    - Pressing

      or
    - Fusing
  - Sample logistics
    - Specimen transport
    - Sample feed to instruments
  - XRF analysis
    - Unattended
    - Automatic system alignment and quality check(regularly)
  - Data transfer to LIMS

The modern mining laboratory
Reasons to automate

- Supply of better data quality
  - Less deviations due to no human errors
- Increased workplace safety
  - Less contacts with hazardous chemicals
  - Reduced staff leads to lower chances for accidents
  - Robots don‘t get tired. less performance fluctuations

- Higher investment and clever concept needed
  - Less flexibility
  - Start-up time longer
  - Regular maintenance needed

Automated Sample Preparation Concepts

- Sample taking and crushing as first step
- Sample transportation by tube post

- Grinding, dosing with weighing are done automated in a very precise way

- Preparation of pressed pellets or fused beads for accurate and fast analyis

- Typical turnaround times
  - Pressed pellets: 2-3 minutes
  - Fused beads: 15-20 minutes

- Instrument precision 0.05 % rel.
- Manual sample preparation factor of 10
- Automated sample preparation factor of only 5

The Automated Laboratory Sample Preparation

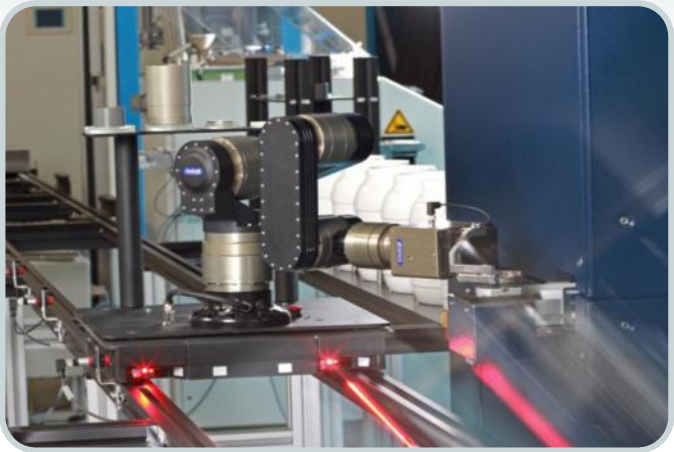
Automated mill and press systems are commercially available from

- ThyssenKrupp Polysius
- Herzog
- FLSmidth

- Providing the sample pressed in steel rings
- Passing it on conveyor belts or robots

- Offering automation interface to connect with plant control systems

The Automated Laboratory Sample Logistics



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- Use of robots in automation: providing higher flexibility and allowing more compact automation layout
- Conveyor belt systems typically less expensive and doesn‘t require teaching, running with lower maintenance costs
- Feeding of samples from the back of the system is beneficial
  - System still accessible for non routine samples
  - Backup operation by manual input when automation is out of service

Summary

- If high throughput of the samples is not required – manual sample preparation will do the job, but the user has to be aware of the precision limitations. Also sequential WDX instrument most likely will be sufficient.
- For process control where high precision and throughput are needed, automated sample preparation in combination with simultaneous WDX instrument will provide the best possible solution.

Conclusions
Automation lineup

- Automated sample preparation will improve
  - Better analytical precision by a factor of two compared to manual sample preparation
  - Releases workforce from heavy work with hazardous chemicals
  - Increases lab effciency especially on remote sides where no experienced work staff is available
  - Increases sample throughput
    - Up to 1400 samples per Sim-WDXRF instrument, such as the S8 DRAGON
    - Up to 600 samples per Seq-WDXRF instrument, such as the S8 TIGER

  - Requires continous and constant sample flow (similar types)
  - Higher investment in the beginning
  - Requires regular maintenance and available workforce for trouble-shooting

S8 DRAGON in automation for mining applications

- Sim-WDXRF instrument setup for maximum precision
- Robust configuration for high instrument uptime
- Sample loading in automation from the back
- Available sample loading manually from the front
- Enhanced analytical flexibility due to Multielementchannel
  - Single element channel backup
  - Screening tool for non-routine elements
  - Standardless analysis
  - Independent control of single element channels (consistency)
  - Automated system self diagnosis

Q&A

Any questions?

Please type any questions
you may have for our speakers
in the Q&A panel and
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Thank you!

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Webinar	Content
Nov 20, 2012 **TXRF for Trace Element Analysis of Air, Land and Water**	This one-hour live webinar demonstrates the capabilities of TXRF for trace element analysis of air (aerosol and filters), land (soils and sediments), and water (fresh and effluents). Learn about level of detection, ease-of-use, regulated analysis, and advantages of TXRF in various, everyday environmental applications. Register now

Webinar	Content
Dec 13, 2010 **Trace Element Analysis of Industrial Wastewater and Sewage with TXRF and ICP-MS**	Join Mike Beauchaine and Andrew Toms as they present the capabilities of TXRF and ICP-MS for trace element analysis of wastewater. Learn about the combined advantages of the two techniques for routine, industrial and mobile lab testing. View recording Download slides

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