# How to bring WDS capability to your SEM using the latest EDS technology?

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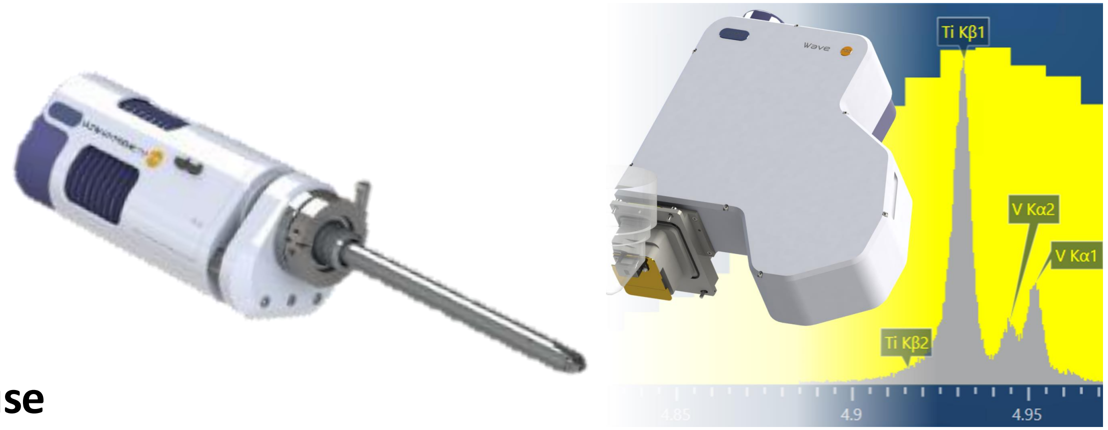
# Overview

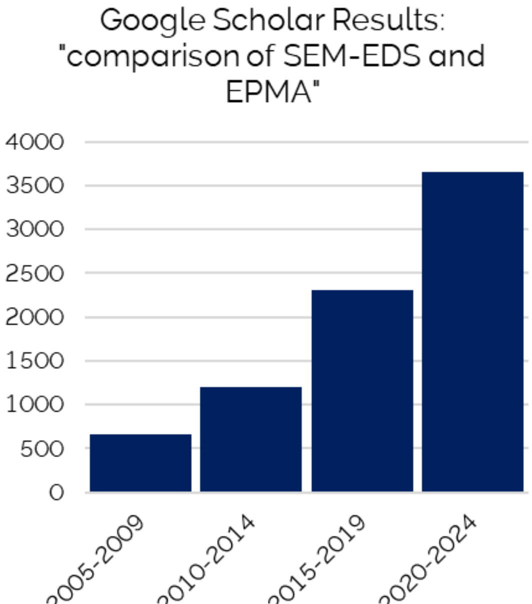
- **The growth in capability of EDS detector and data processing** has significantly reduced the advantage of a WDS system on the SEM.
- Applications where SEM-WDS analysis was traditionally the only means of producing reliable data are now increasingly rare
- EDS can more often provide an equivalent result using more familiar workflows and methods
- Modern EDS detectors supported by **Tru-Q IQ spectrum processing (new Ultim Max Infinity detector)** support applications where WDS has traditionally been preferred.
-
  - **Peak overlaps**
  - **Light element analysis**
  - **Minor and trace element constituents**
  - **Quantitative analysis**

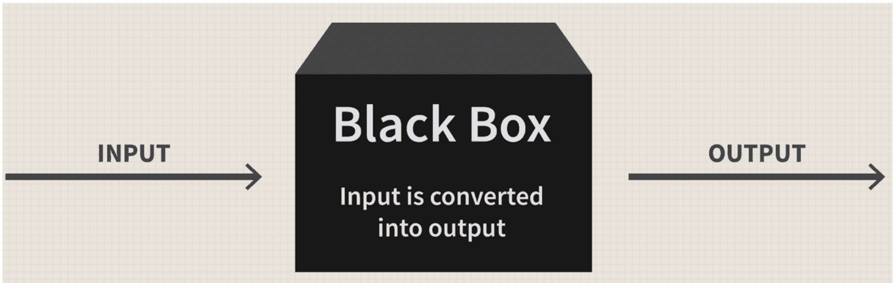


- EDS advantages:
- **Significant improvements in speed, versatility, and ease-of-use**
- **Combination** with the first of its kind **BEX** (backscattered electron and X-ray imaging) detector

# Why EDS is not used more for quant

- EPMA started with WDS
- Microprobe are instrument designed for quant, SEM are more general purpose
- Misconception that EDS does not have enough spectral resolution for quant
- Vendors offer one button click quant. Good for users that know what they are doing, bad for users that don’t.


Google Scholar Results: "comparison of SEM-EDS and EPMA"


INPUT

**Black Box**

Input is converted into output

OUTPUT

Duration: 60 mins

On Demand

Practical guide to high count rate EDS analysis

This webinar covers high count rate capabilities of EDS systems, consequences of working at high rate and main limitations. We also demonstrate how to perform this type of analysis.

Read more >

Duration: 60 mins

On Demand

Modern SEM-EDS Systems Explained - What is cutting edge technology today?

Cutting edge SEM-EDS systems can achieve extraordinary results achieving accuracy, speed and sensitivity that may surprise you. This educational session unlocks the key requirements.

Read more >

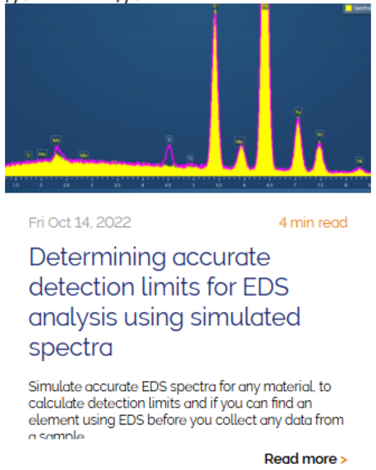
Bioimaging & Life Science | Batteries & Fuel Cells

Duration: 1 Hour

On Demand

Quantitative EDS Explained: How to achieve great results with or without using your own standards

Read more >


Fri Oct 14, 2022

4 min read

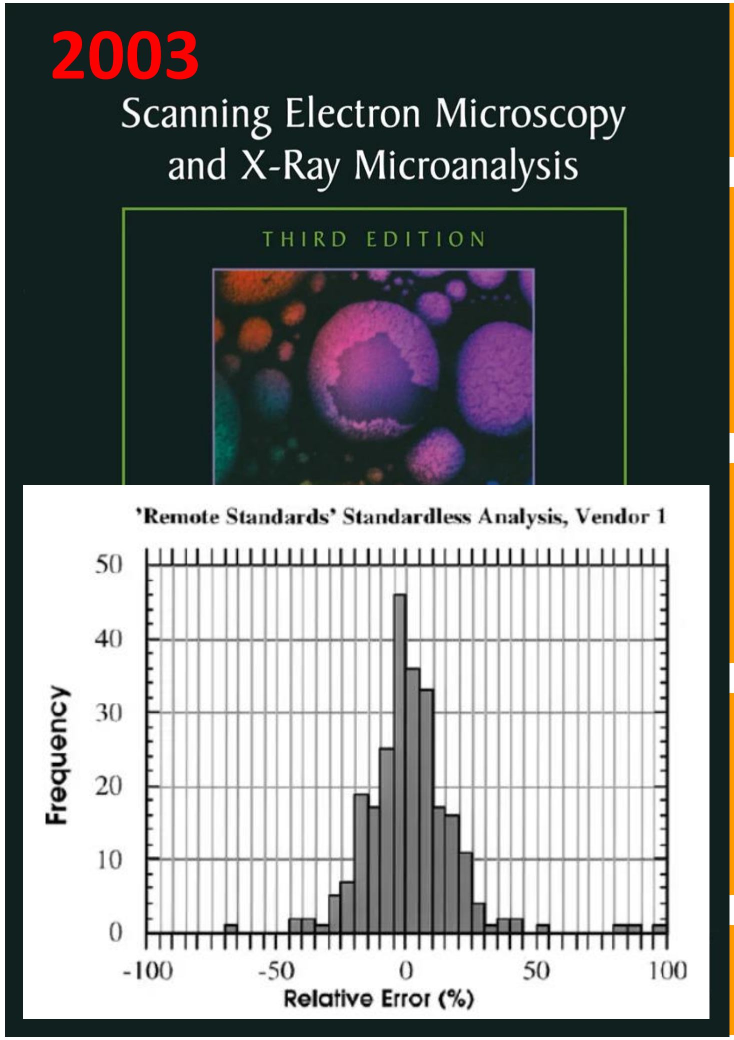
Determining accurate detection limits for EDS analysis using simulated spectra

Simulate accurate EDS spectra for any material, to calculate detection limits and if you can find an element using EDS before you collect any data from a sample

Read more >

# Rationale- Pushing for Greater EDS Acceptance

## What Does the Literature Say?



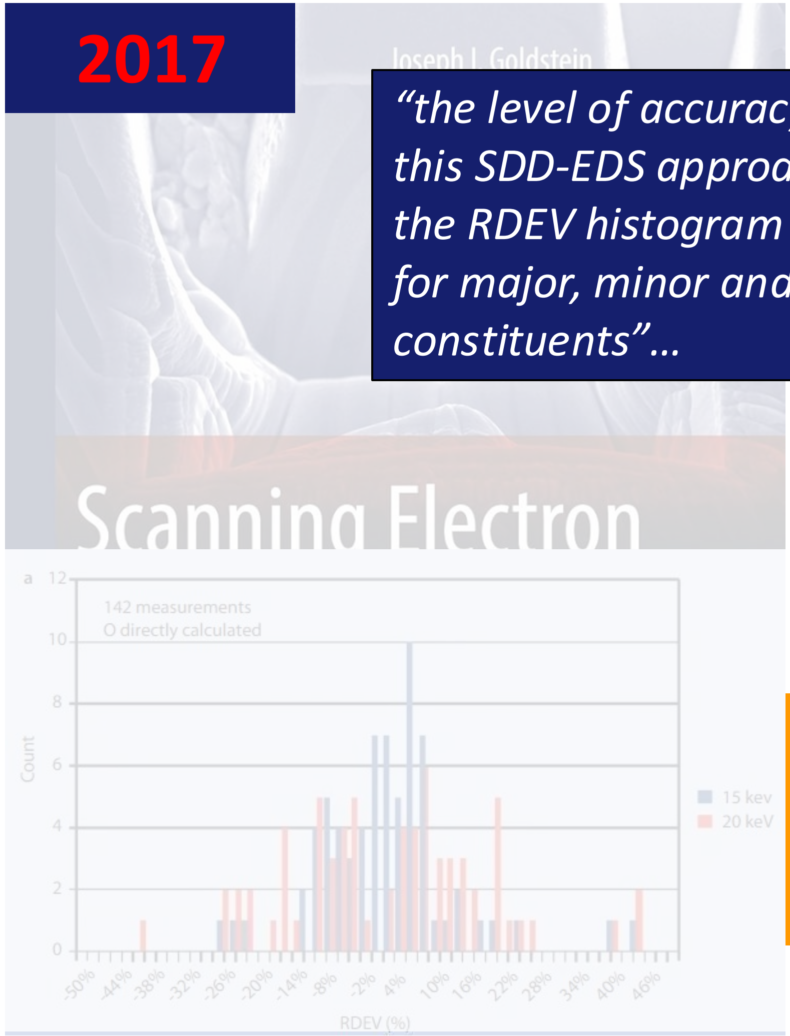
*"EDS spectrum processing is a "black box"*

*"Overlaps are so severe analysis of an element of interest cannot be carried out"*

*"Very difficult to detect the presence of a minor element"*

*"Standardless analysis produces "marked loss in accuracy"*

*"Standardless always normalised"*

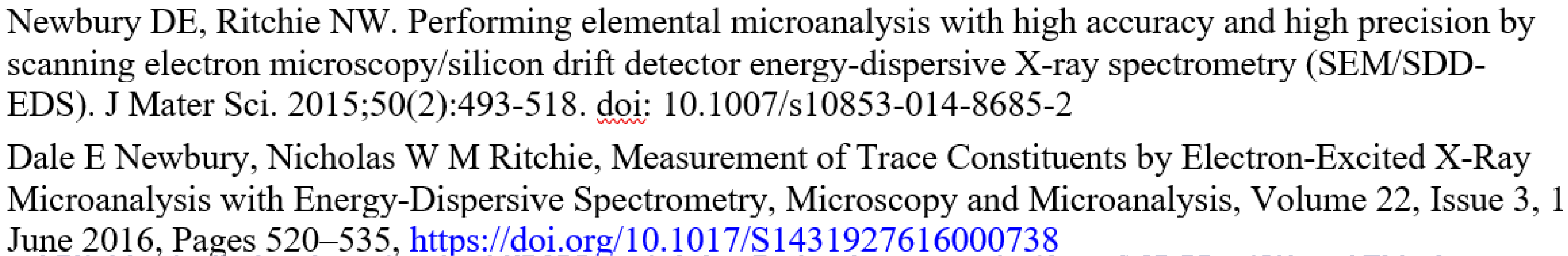


*"the level of accuracy achieved with this SDD-EDS approach fits within the RDEV histogram of EPMA-WDS for major, minor and trace constituents"…*

*Calibration should be performed regularly*

# Comparison between EDS and WDS data in journals

- Despite lower P/B ratios and worse spectral resolution, EDS has been shown to achieve high accuracy equivalent to that of WDS even in the cases of minor and trace elements with severe interference from a major element



Newbury DE, Ritchie NW. Performing elemental microanalysis with high accuracy and high precision by scanning electron microscopy/silicon drift detector energy-dispersive X-ray spectrometry (SEM/SDD-EDS). J Mater Sci. 2015;50(2):493-518. doi: 10.1007/s10853-014-8685-2

Dale E Newbury, Nicholas W M Ritchie, Measurement of Trace Constituents by Electron-Excited X-Ray Microanalysis with Energy-Dispersive Spectrometry, Microscopy and Microanalysis, Volume 22, Issue 3, 1 June 2016, Pages 520–535, https://doi.org/10.1017/S1431927616000738

- Newbury and Ritchie studied a glass standard K2496 containing Ba (major concentration of 42.99 wt%) and Ti (minor concentration of 1.8 wt%) and obtained a relative error of 2.4% for Ti; for BaTiO3 the deconvolution of Ba L and Ti K X-ray peaks is required for the accurate quantification.

The small relative error demonstrates the deconvolution of EDS X-ray peaks is possible even if one of the deconvolved X-ray series has a much lower intensity than the other.

- By acquiring spectra with 120 million counts, they detected the presence of 0.41 wt% Ta (overlapping with 11.5 wt% Si) and 0.41 wt% Ce (overlapping with 21.94 wt% Ba) in the K873 glass standard but obtained a worse quantification accuracy (~25%) than with major and minor element interferences
- The authors operated their EDS detectors with a time constant yielding a Mn Kα resolution of 128.5 eV and a dead time of ~10%, resulting in a low output count rate and long measurement time

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# What were the most common applications of SEM-WDS?

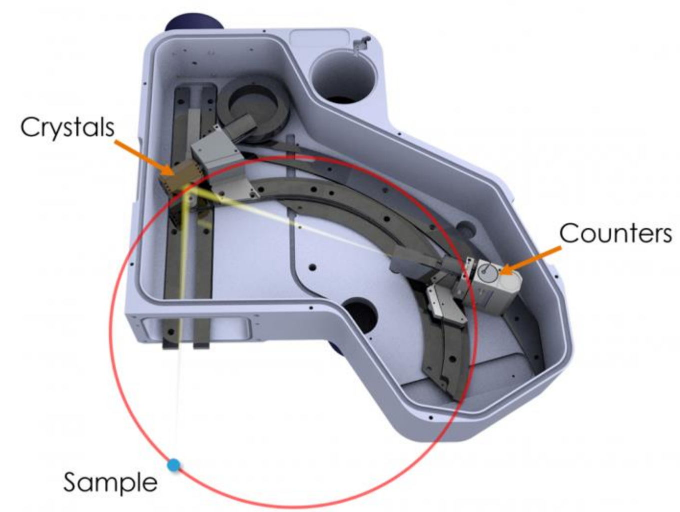
Setting the goal for EDS data quality

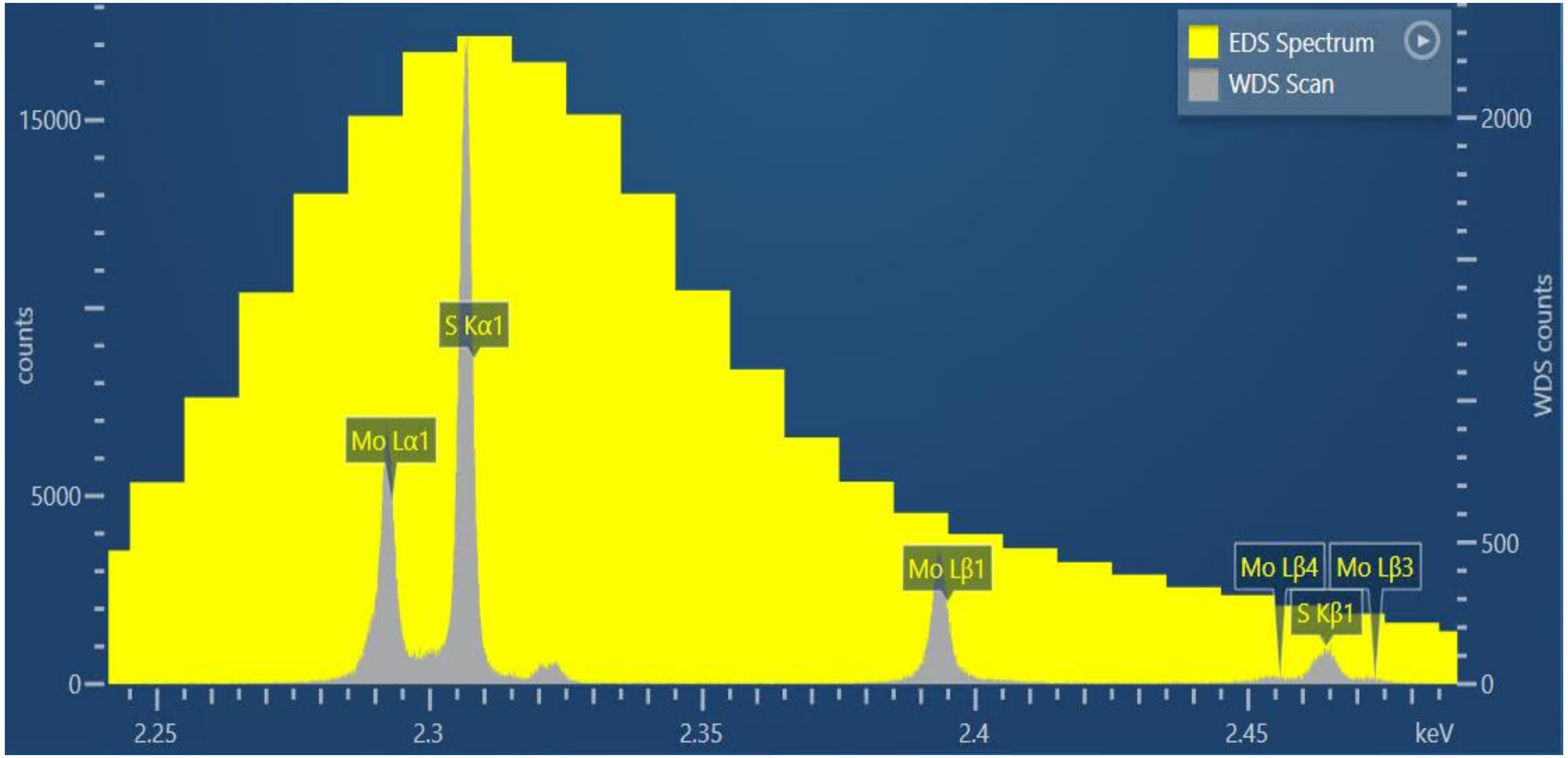
# SEM-WDS Applications: Peak Overlapps

Problematic overlaps:
Mo/S, Ba/Ti, Ti/V, Cr/Mn,
Fe/Co, REE, PGM, Re/W/Hf

**Wave had the highest spectral resolution achievable on an SEM**

- Diffracts select X-ray energies using Rowland circle geometry
- Bent and ground crystals and a motorised counter entrance slit further optimised resolution for every X-ray line
- Si Kα = <2 eV
- Fe Kα = <25 eV

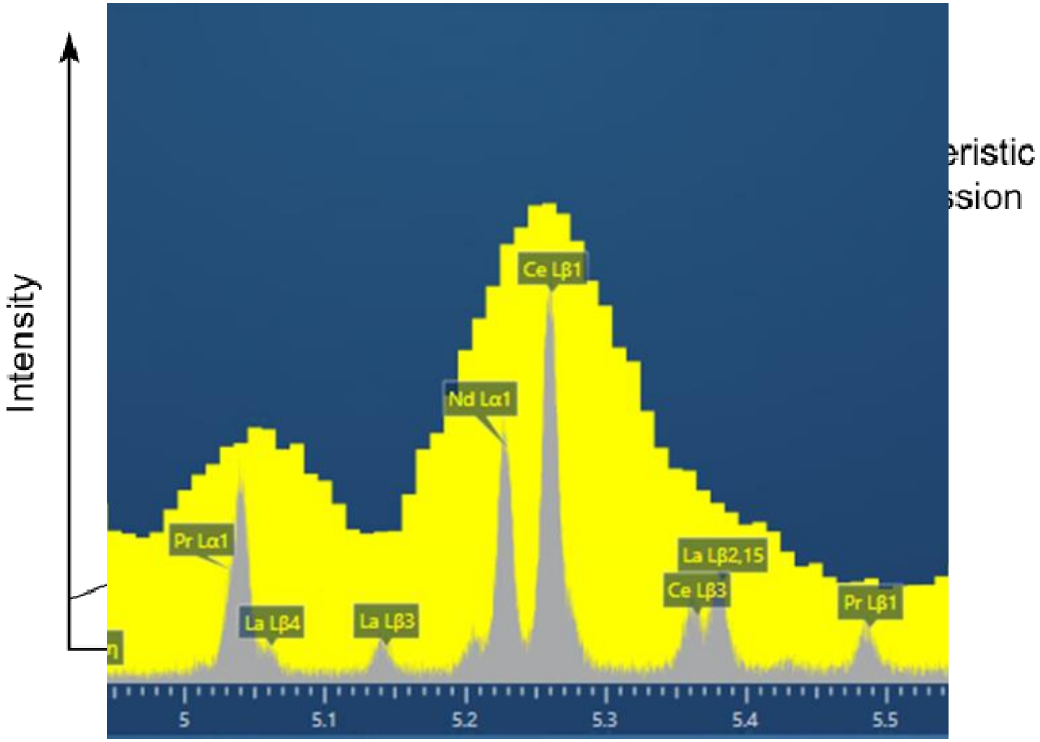


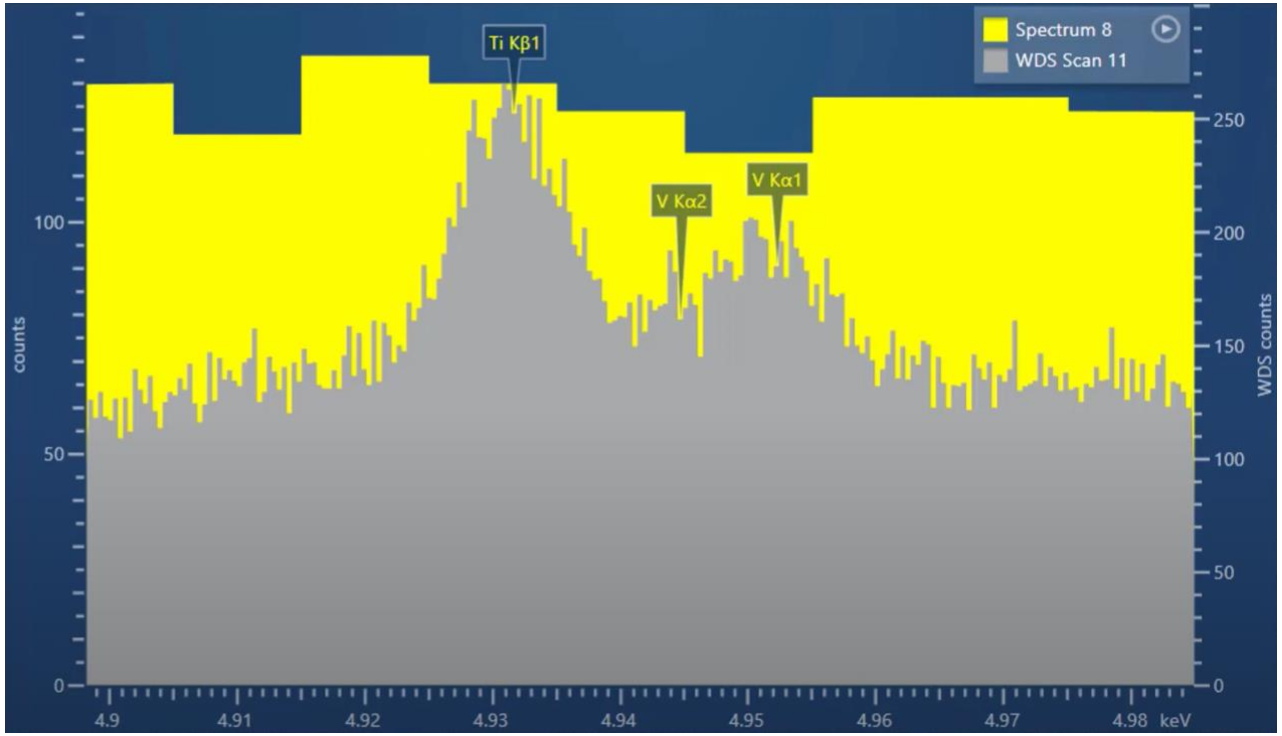


# SEM-WDS Applications: Minor and Trace Elements

**WDS is a powerful and well accepted technique quantification at several wt.% - 10’s ppm**

- High peak to background signal- EDS is impacted more by Bremsstrahlung
- Detection limits frequently less than 100 ppm, Si = 9 ppm, Fe = 15 ppm
- Spectral resolution is important where traces are similar eV to major elements

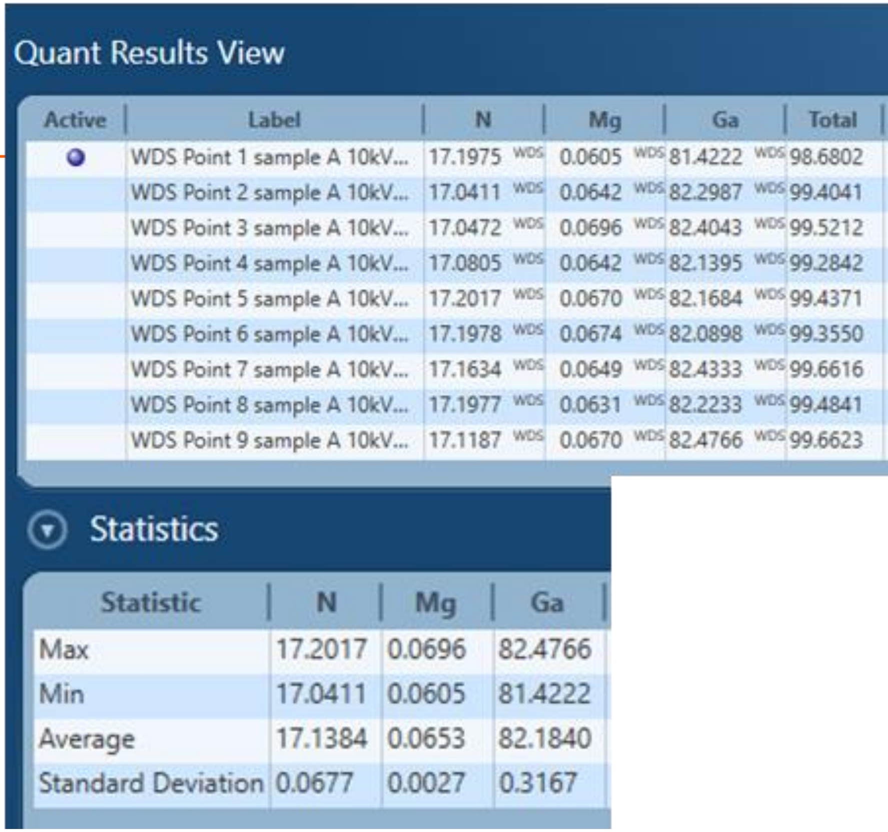




# SEM-WDS Applications: Quantitative Analysis

**WDS is a longstanding and well accepted method for quantitative analysis:**

- High accuracy- totals 100 ± 2 wt.%, σ < 2%
- Standardisation is imperative and performed regularly
- Improved counting statistics on minor-trace elements



Quant Results View

| Active | Label | N | Mg | Ga | Total |
|---|---|---|---|---|---|
| ● | WDS Point 1 sample A 10kV... | 17.1975 WDS | 0.0605 WDS | 81.4222 WDS | 98.6802 |
| | WDS Point 2 sample A 10kV... | 17.0411 WDS | 0.0642 WDS | 82.2987 WDS | 99.4041 |
| | WDS Point 3 sample A 10kV... | 17.0472 WDS | 0.0696 WDS | 82.4043 WDS | 99.5212 |
| | WDS Point 4 sample A 10kV... | 17.0805 WDS | 0.0642 WDS | 82.1395 WDS | 99.2842 |
| | WDS Point 5 sample A 10kV... | 17.2017 WDS | 0.0670 WDS | 82.1684 WDS | 99.4371 |
| | WDS Point 6 sample A 10kV... | 17.1978 WDS | 0.0674 WDS | 82.0898 WDS | 99.3550 |
| | WDS Point 7 sample A 10kV... | 17.1634 WDS | 0.0649 WDS | 82.4333 WDS | 99.6616 |
| | WDS Point 8 sample A 10kV... | 17.1977 WDS | 0.0631 WDS | 82.2233 WDS | 99.4841 |
| | WDS Point 9 sample A 10kV... | 17.1187 WDS | 0.0670 WDS | 82.4766 WDS | 99.6623 |

Statistics

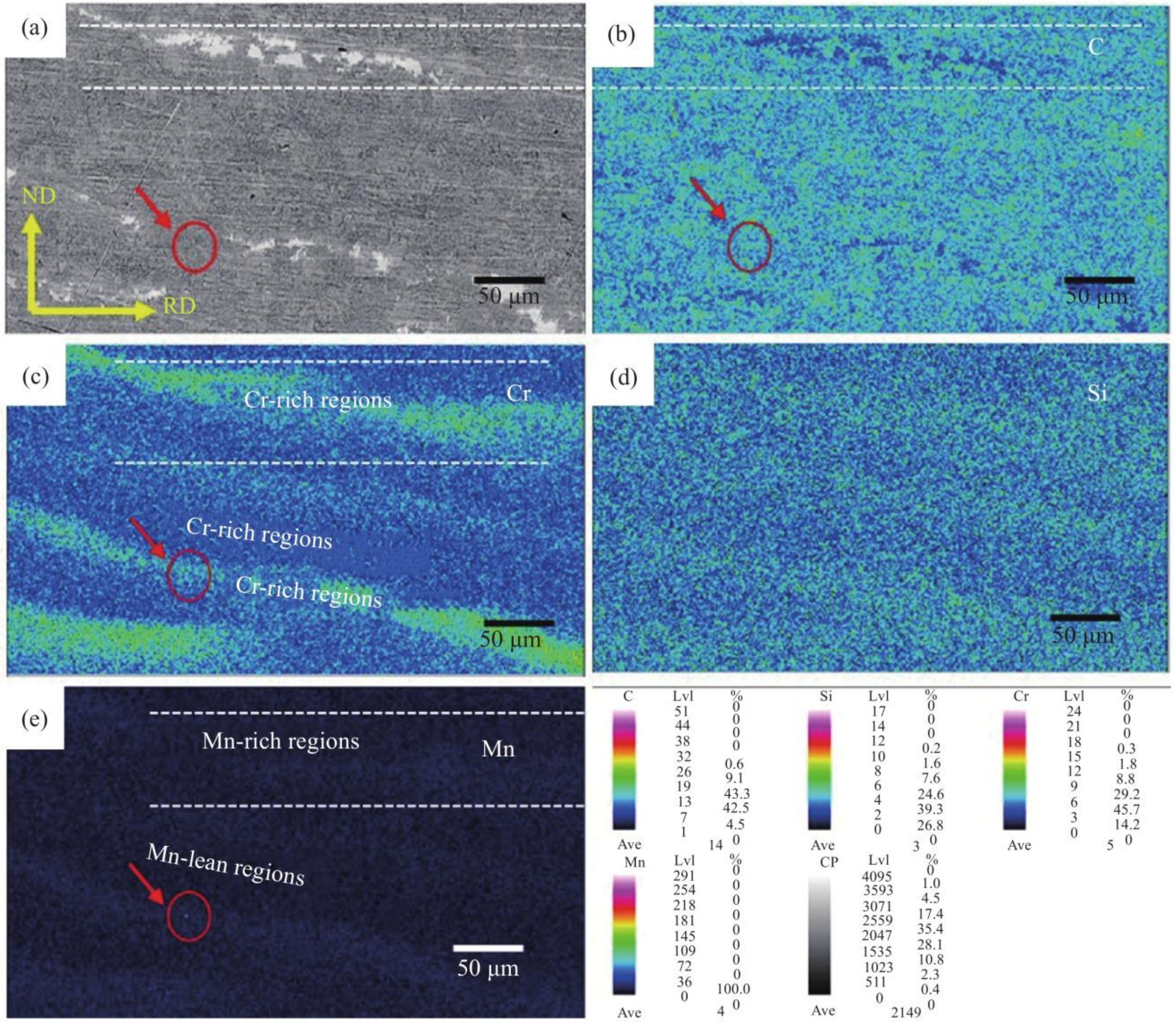
| Statistic | N | Mg | Ga |
|---|---|---|---|
| Max | 17.2017 | 0.0696 | 82.4766 |
| Min | 17.0411 | 0.0605 | 81.4222 |
| Average | 17.1384 | 0.0653 | 82.1840 |
| Standard Deviation | 0.0677 | 0.0027 | 0.3167 |

| | **Olivine analysis** | | **Element wt.%** | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | **Si** | **Fe** | **Ni** | **Mn** | **Mg** | **Ca** | **O** | **Total** |
| EPMA | **Electron microprobe** | Average | 16.87 | 25.90 | 0.03 | 0.42 | 18.25 | 0.02 | 38.79 | 100.28 |
| | | *1σ* | *0.085* | *0.396* | *0.011* | *0.021* | *0.211* | *0.008* | *0.172* | *0.474* |
| **AZtecWave** | **SEM WDS-only** | Average | 16.79 | 25.39 | 0.04 | 0.43 | 18.56 | 0.01 | 38.75 | 99.97 |
| | | *1σ* | *0.327* | *0.309* | *0.014* | *0.027* | *0.310* | *0.004* | *0.268* | *0.487* |
| | **SEM EDS+WDS** | Average | 17.02 | 25.24 | 0.04 | 0.43 | 18.14 | 0.01 | 38.69 | 99.57 |
| | | *1σ* | *0.059* | *0.320* | *0.014* | *0.027* | *0.204* | *0.004* | *0.118* | *0.201* |

# SEM-WDS Applications: Mapping

**WDS was used to perform 2D mapping of minor and trace element constituents:**

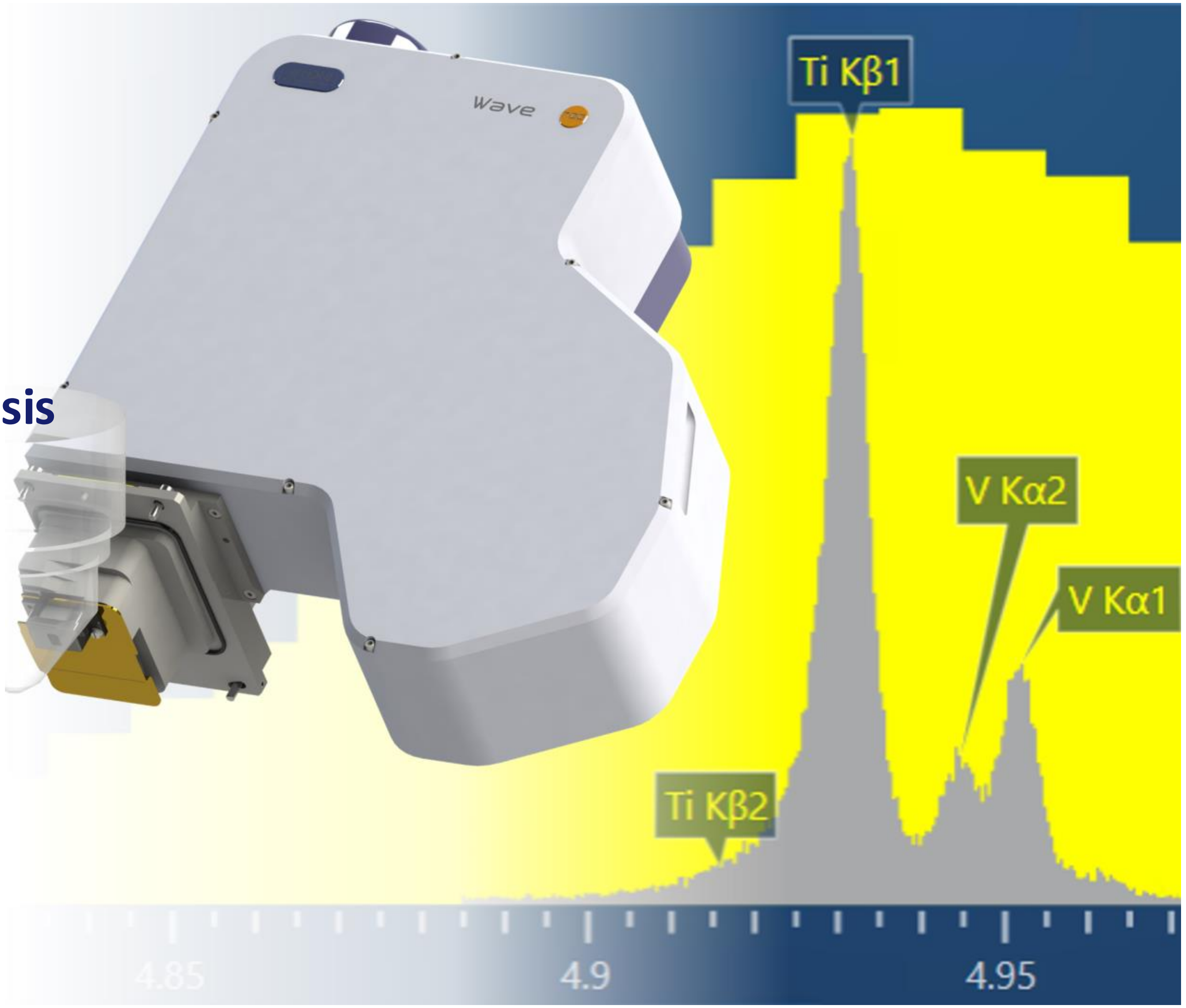
- Minor-trace element mapping
- High precision resolves subtle variation
- Eg diffusion in steel/variation in minerals
- High speed EDS produces spectral artefacts



# Summary SEM-WDS Applications

## WDS major application areas

- Analysis of difficult spectral overlaps
- Characterisation of minor and trace elements
- High precision and high accuracy quantitative analysis
- Minor-trace element mapping



# How EDS Caught up with WDS

## Technological advances in SEM-EDS

# Leaps in EDS Technology: Digital Spectrum Processing (1995)

**Early EDS systems relied on analogue electronics for signal processing, but digital electronics quickly became the dominant technology.**

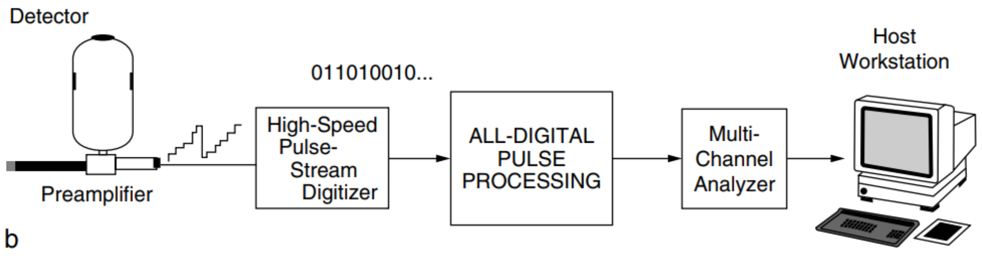
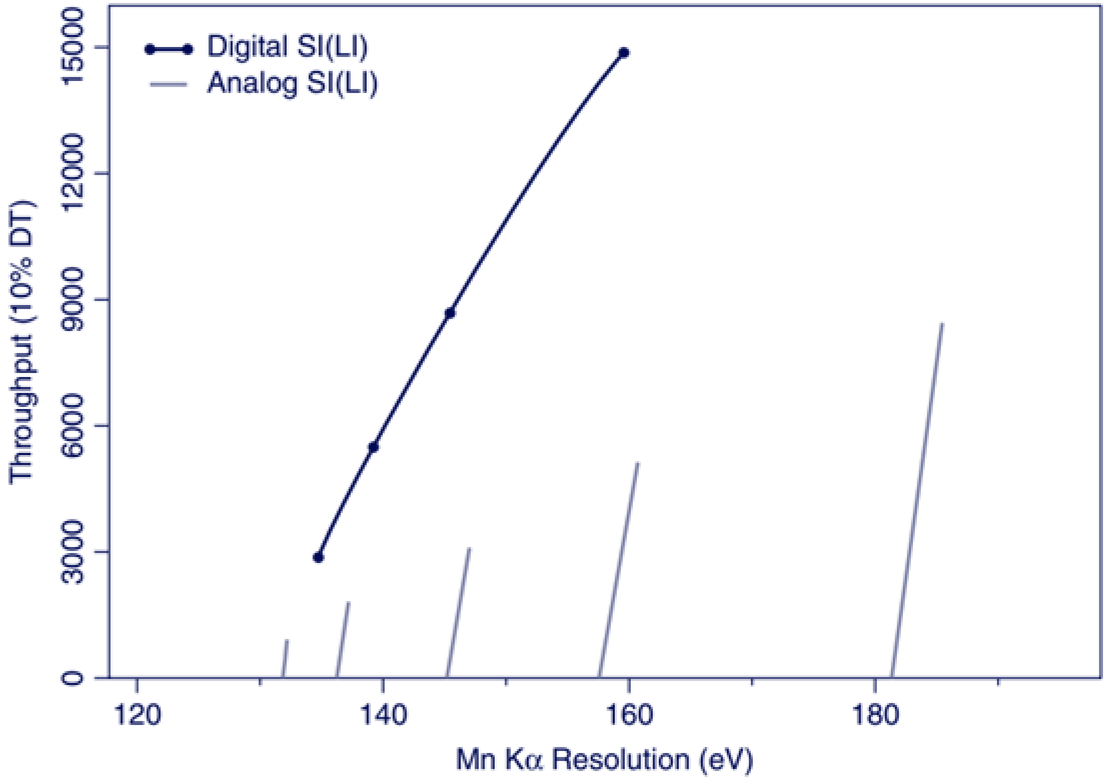


***Figure 7.12.*** Comparison of digital and analog pulse processing. (From Mott and Friel, 1995; courtesy of Princeton Gamma Tech.)

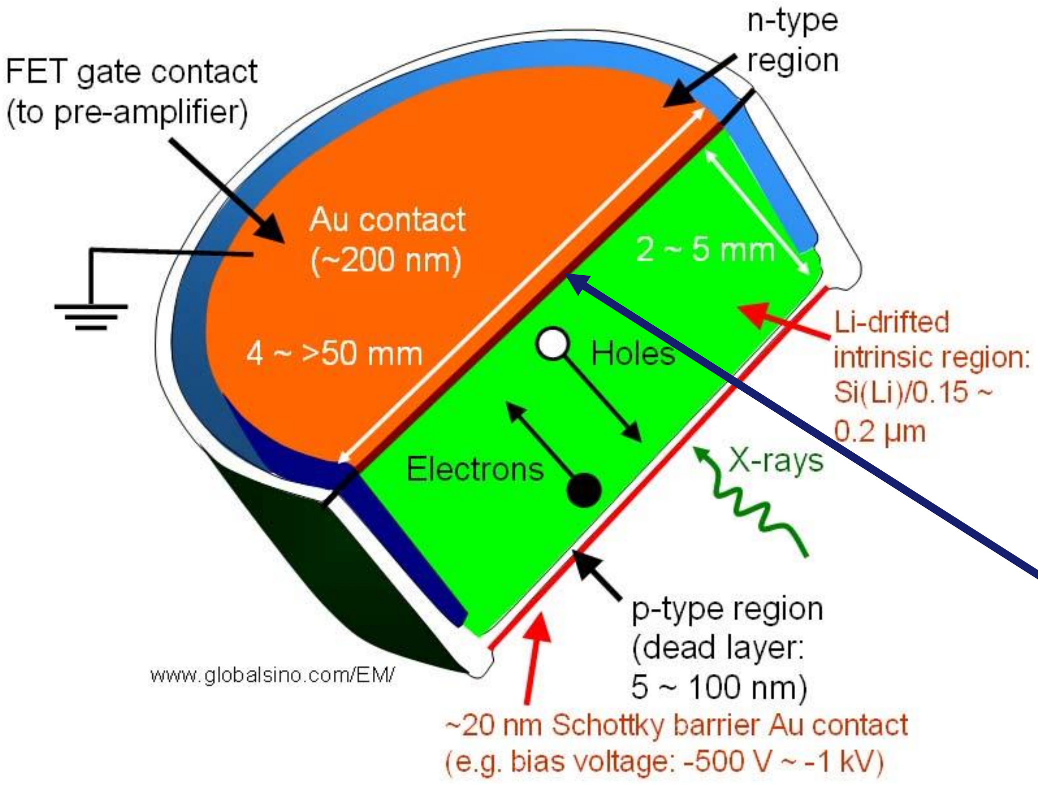


- Speed
- Resolution
- **Day-to-day stability**

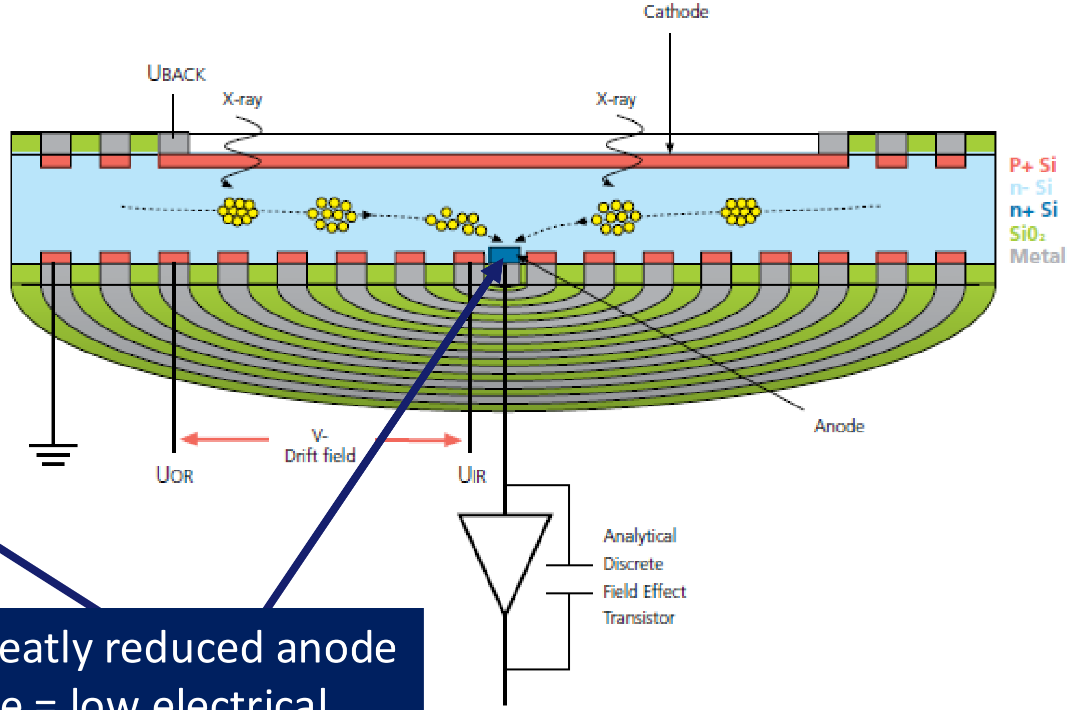
# Leaps in EDS Technology: Transition from SiLi to SDD (2000’s)

**Silicon drift detectors (SDD) created an order of magnitude leap in EDS speed and resolution**

1. Liquid nitrogen free
2. Superior resolution
3. Greatly increased count rate



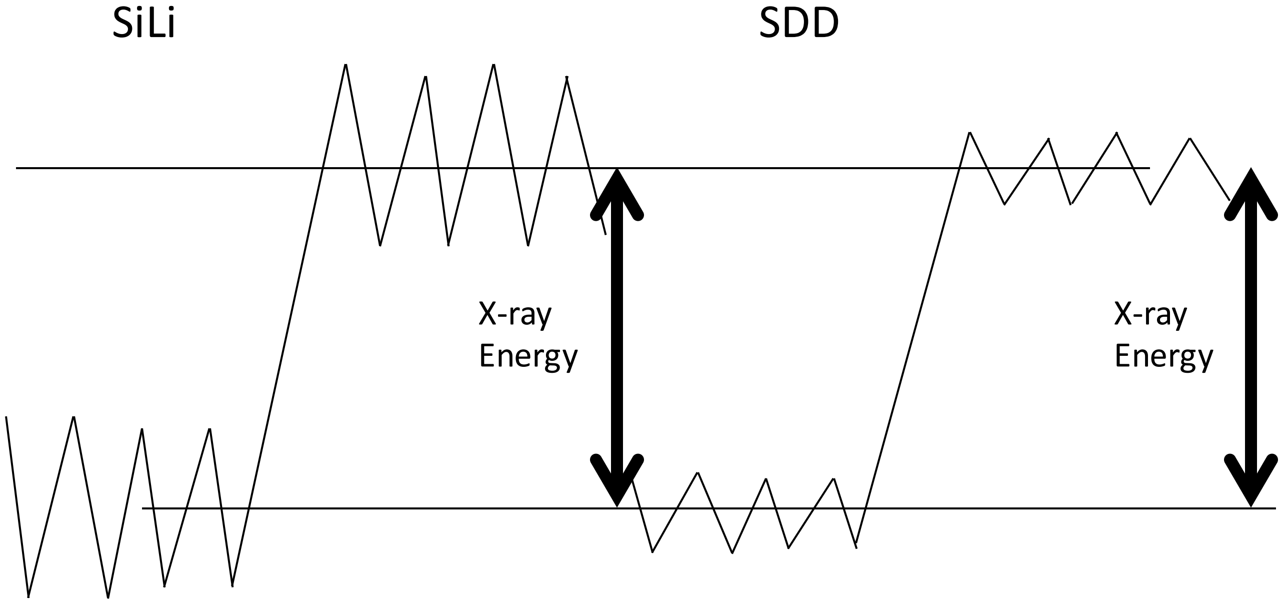
www.globalsino.com/EM/



Greatly reduced anode size = low electrical noise

# Leaps in EDS Technology: Transition from SiLi to SDD (2000's)

**The speed and Resolution benefits of low noise SSD's**



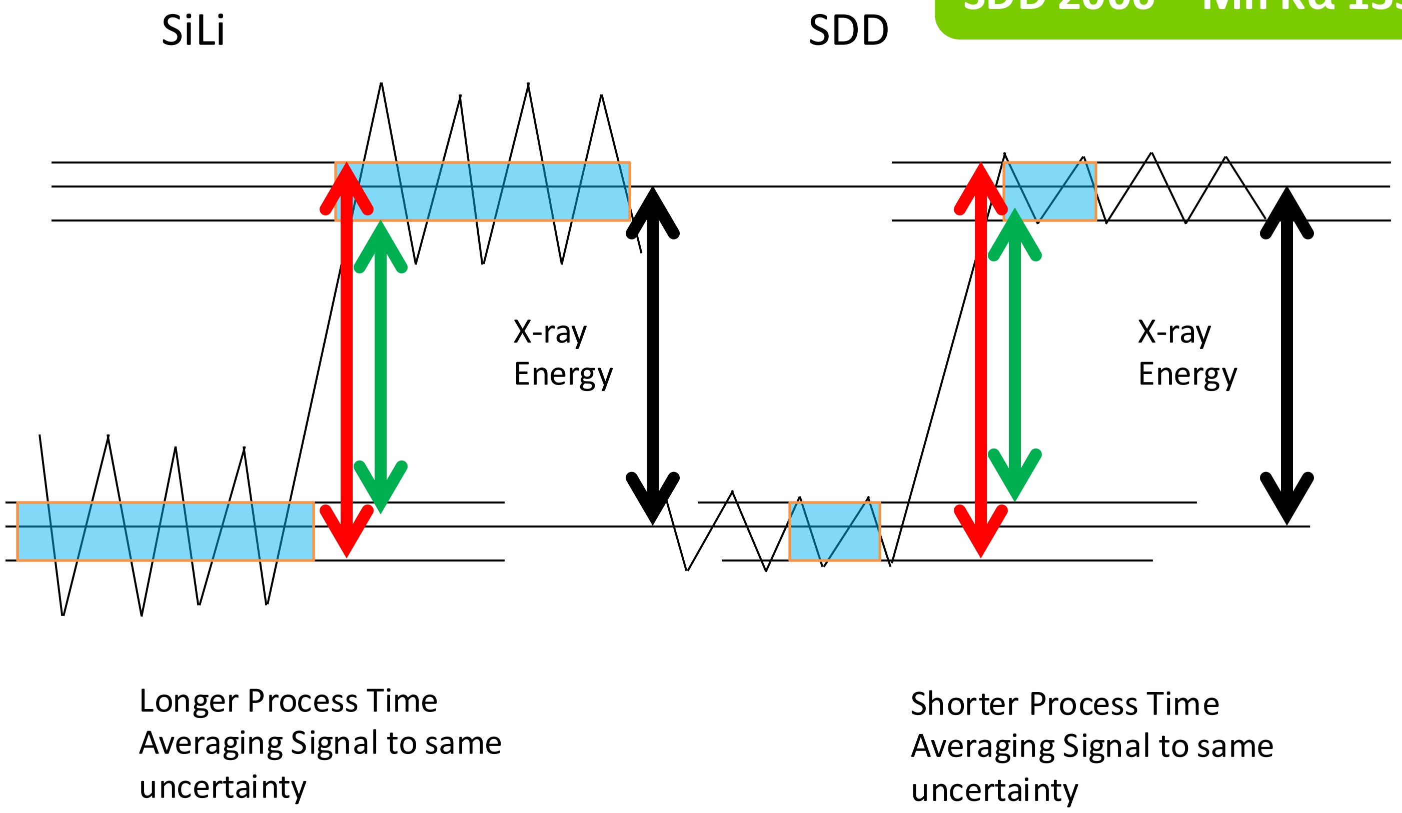
Voltage ramps

# Leaps in EDS Technology: Transition from SiLi to SDD (2000's)

**The speed and Resolution benefits of low noise SSD's**

**Si(Li) 2002 – Mn Kα 133 eV @ 2500 cps**
**SDD 2006 – Mn Kα 133 eV @ 20,000 cps**

**8 X count rate improvement**



Longer Process Time
Averaging Signal to same uncertainty

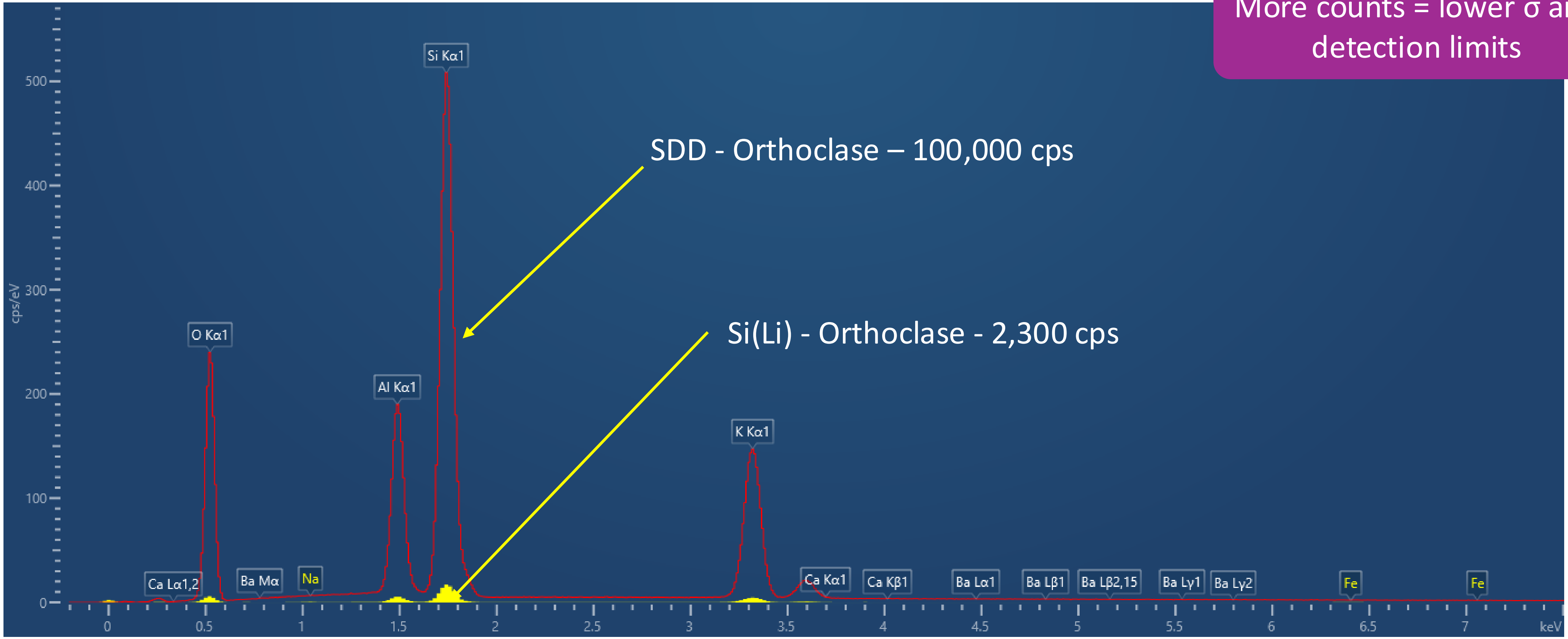
Shorter Process Time
Averaging Signal to same uncertainty

Lower noise-less time needed to process the signal

# Leaps in EDS Technology: Transition from SiLi to SDD (2000's)

**The speed and Resolution benefits of low noise SSD's**

More counts = lower σ and detection limits



SDD - Orthoclase – 100,000 cps

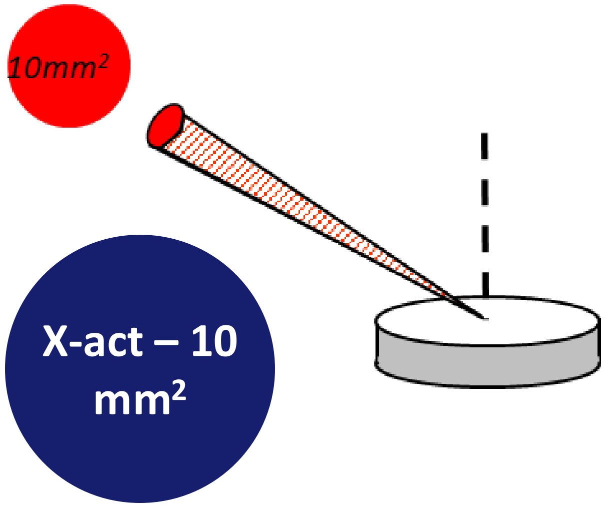
Si(Li) - Orthoclase - 2,300 cps

Si(Li) detector would take 40 sec to achieve 100,000 counts

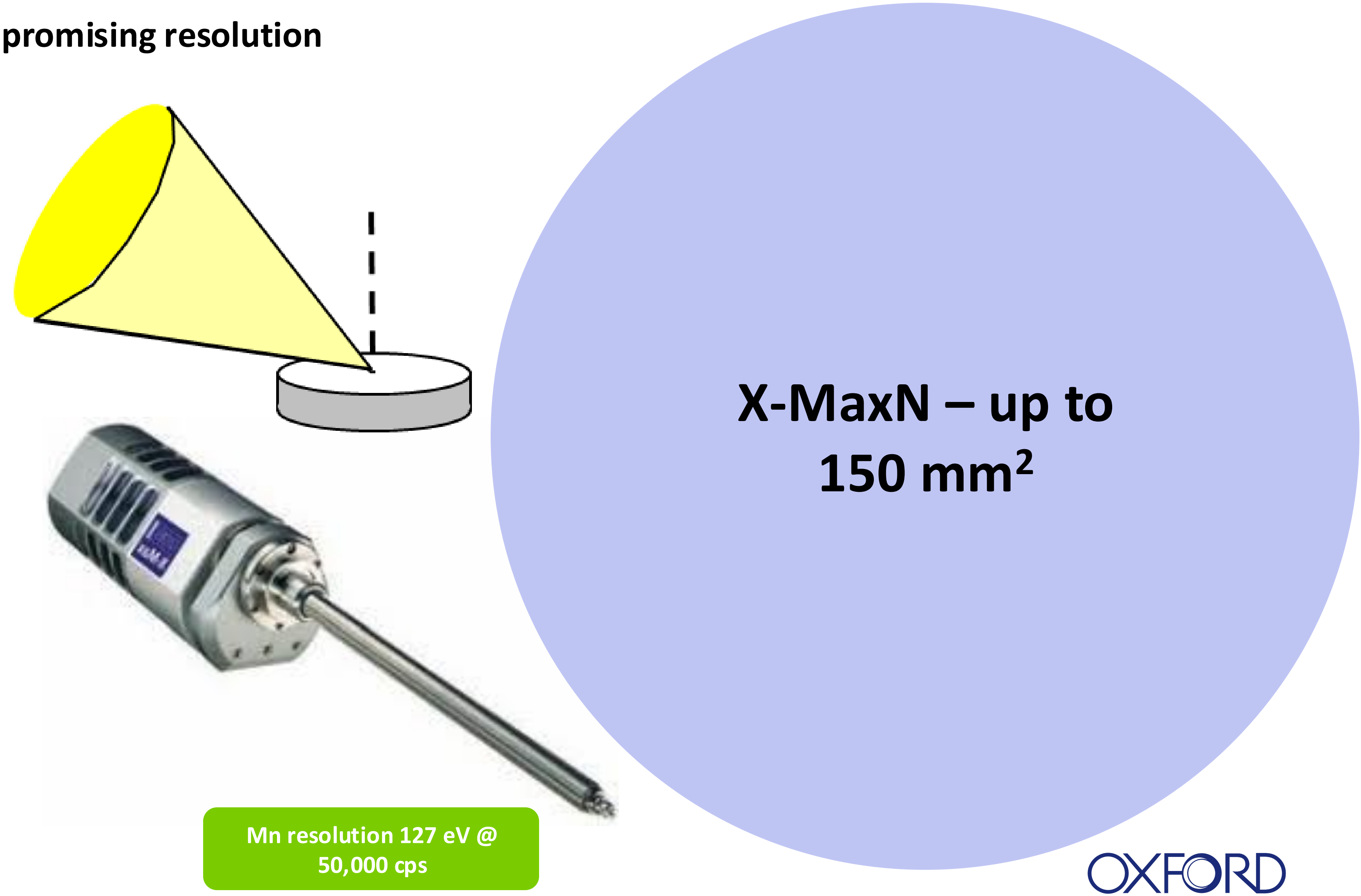
# Leaps in EDS Technology: Large Area SDD's (2012)

**Sensor sizes got larger without compromising resolution**

- Low beam current
- Low accelerating voltage
- More counts!



**Mn resolution 133 eV @ 20,000 cps**



**Mn resolution 127 eV @ 50,000 cps**

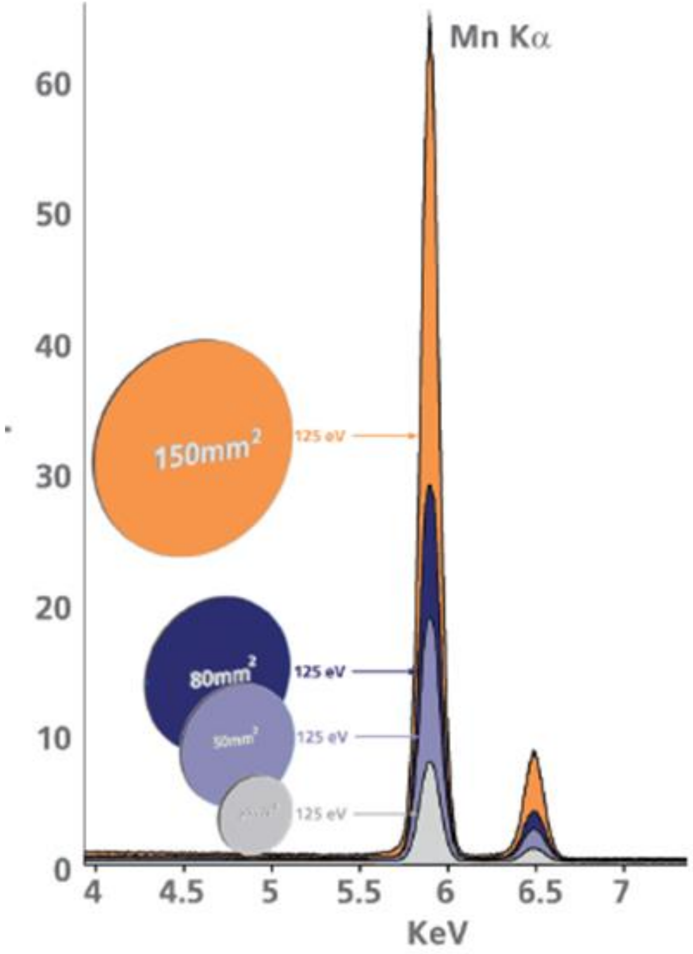
# Leaps in EDS Technology: Large Area SDD's (2012)

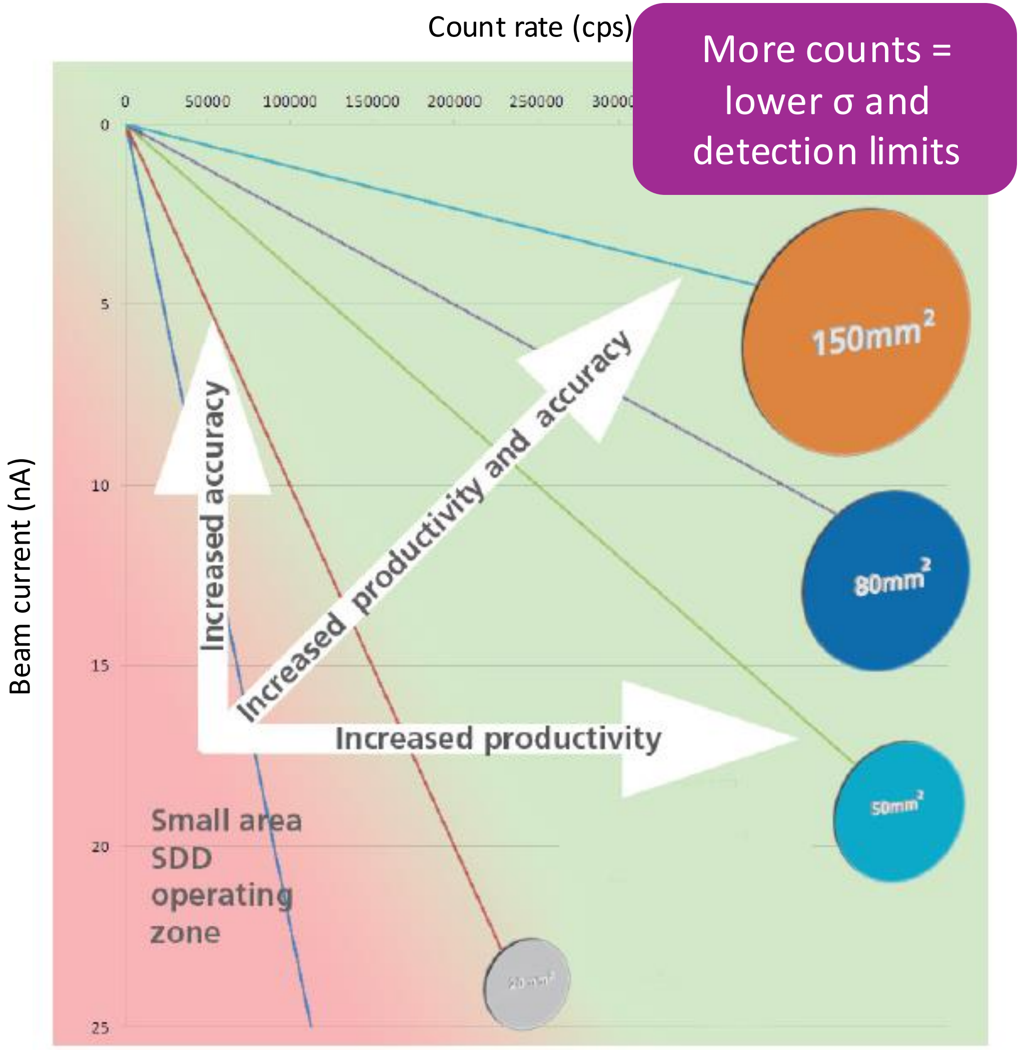
**SDD's got larger without compromising resolution**

A 150 mm² detector requires around 1 nA to generate 100,000 counts per second. In contrast, a 10 mm² detector requires >20 nA *

High speed analysis with:

- Low beam current
- Low accelerating voltage



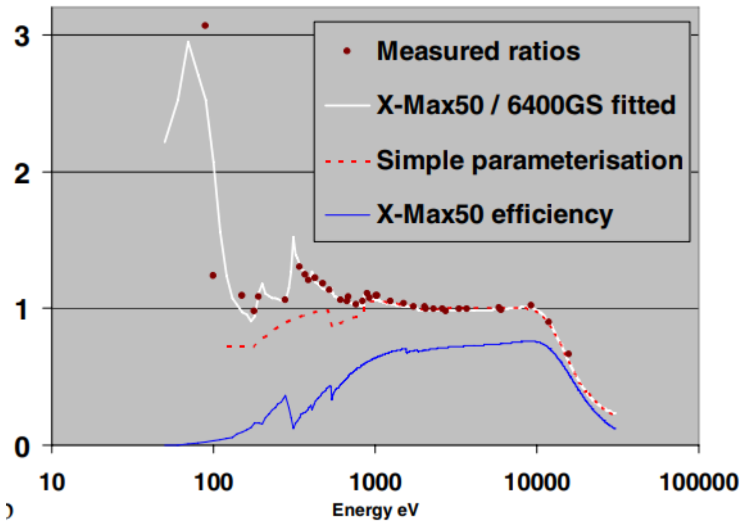


More counts = lower σ and detection limits

# Leaps in EDS Technology: Spectrum Processing (2010's)

**Sources of error in 'standardless' quantitative analysis have been drastically reduced**

**Detector efficiency**



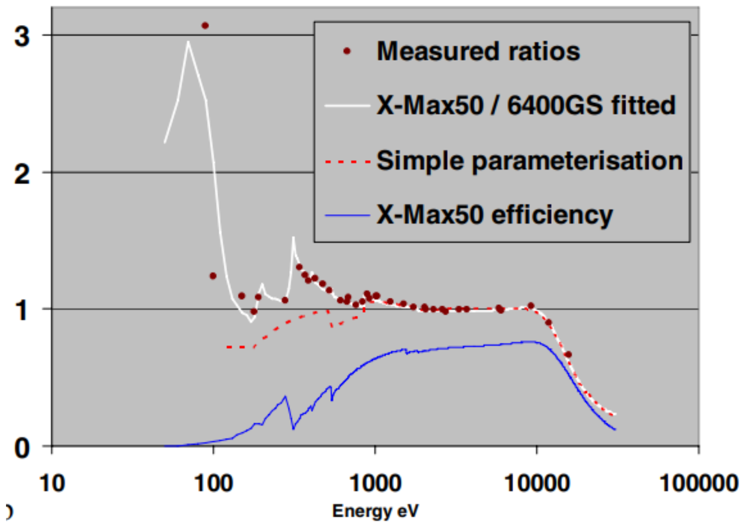
Improved low energy X-ray and light element quantification

**Statham, 2010, Statham 2009, Statham 2002, Newbury and Ritchie 2016, Eggert et al., 2012, Statham 2016, Pinard et al., 2020**

# Leaps in EDS Technology: Spectrum Processing (2010’s)

**Sources of error in ‘standardless’ quantitative analysis have been drastically reduced**

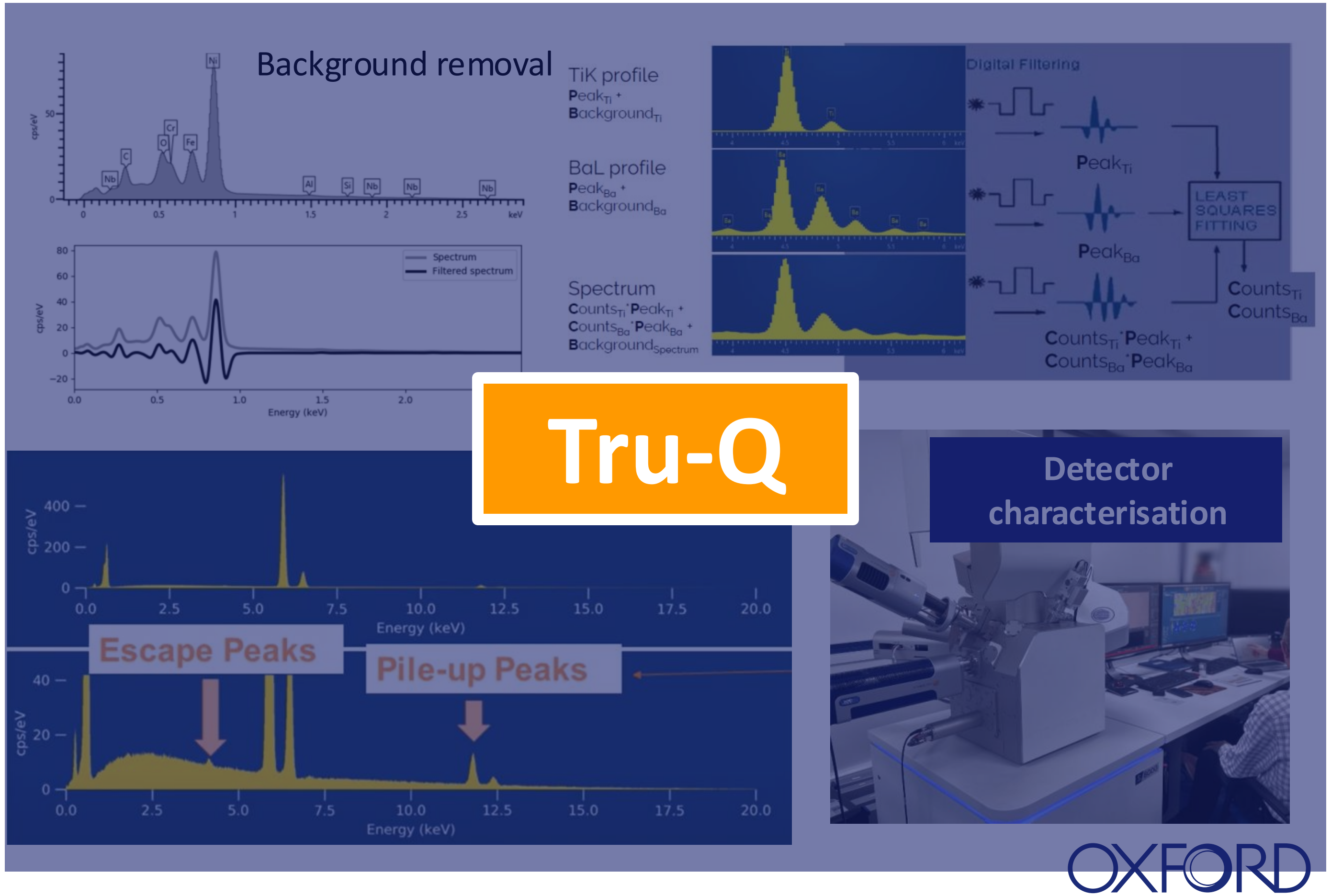
## Detector efficiency



Improved low energy X-ray and light element quantification

**Statham, 2010, Statham 2009, Statham 2002, Newbury and Ritchie 2016, Eggert et al., 2012, Statham 2016, Pinard et al., 2020**

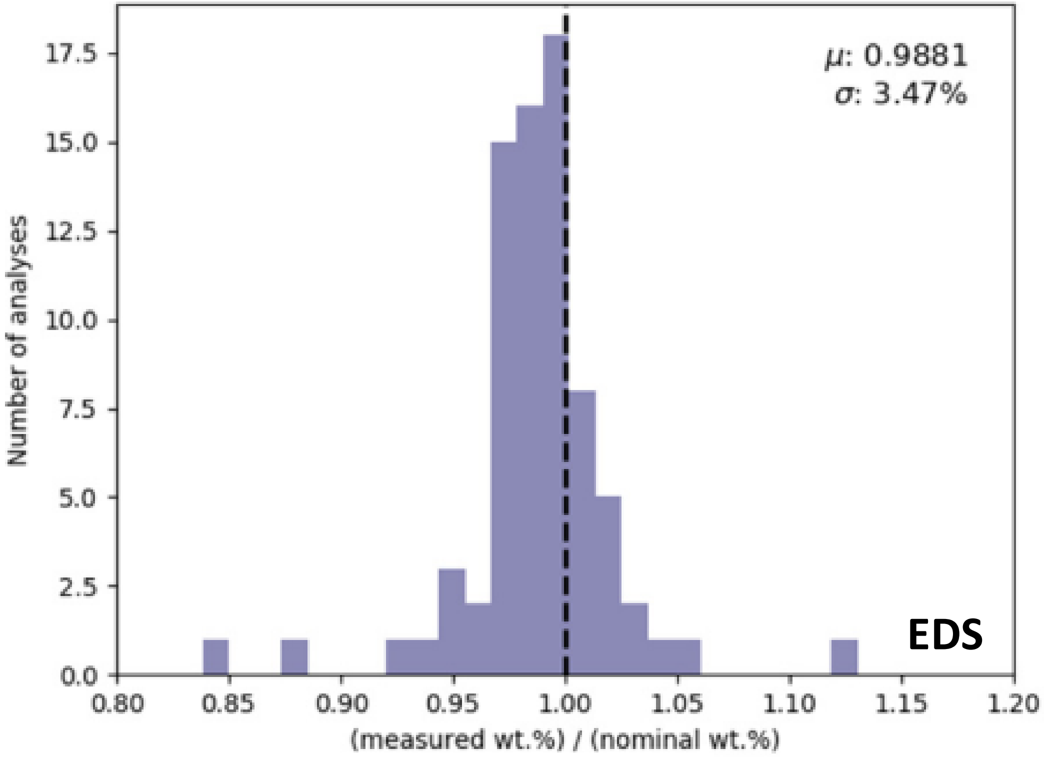
## Extracting Accurate X-ray Intensity



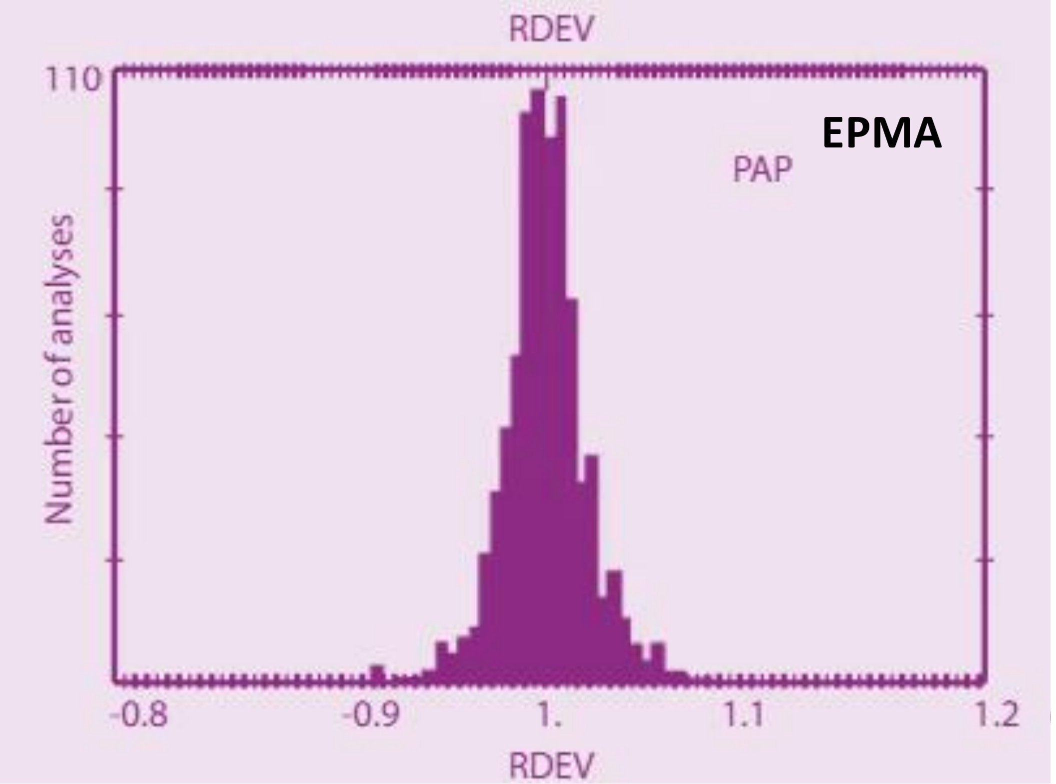
# Leaps in EDS Technology: Spectrum Processing (2010's)

**Sources of error in 'standardless' unnormalized quantitative analysis have been drastically reduced**

20 kV, 50 kcps



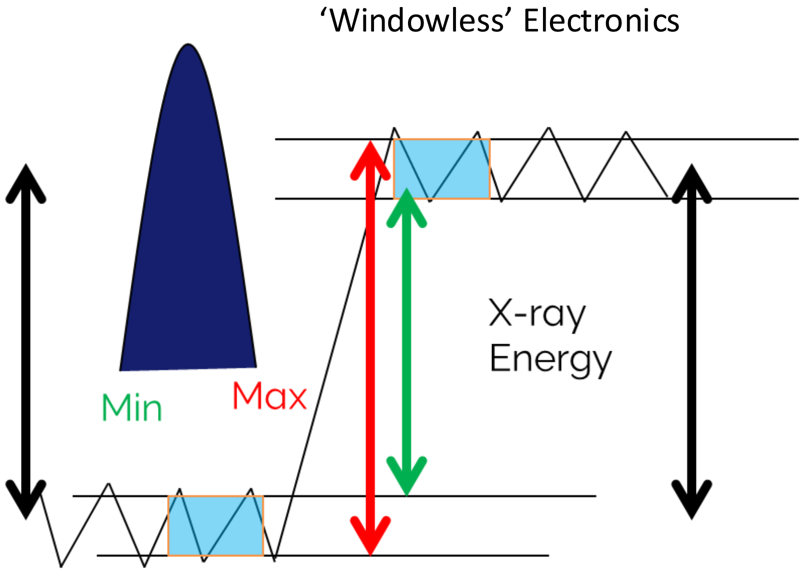
**Pinard et al., 2020**



**Pouchou and Pichoir, 1991**

# Leaps in EDS Technology: Ultim Max Series EDS (2018)

**Ultim Max brought windowless 'low-noise' electronics to the masses, increasing speed**



| Mn Res. | F Res. | C Res. | @ cps |
|---|---|---|---|
| 127eV | 64eV | 56eV | 130,000 |

**Same resolution as X-MaxN with 260% the count rate**

**Increased spee**

**More counts**

**lower σ and detection limits**

Tackle more challenging tasks with improved counting statistics:

- Minor and trace elements
- Quantification
- Low wt.% variation

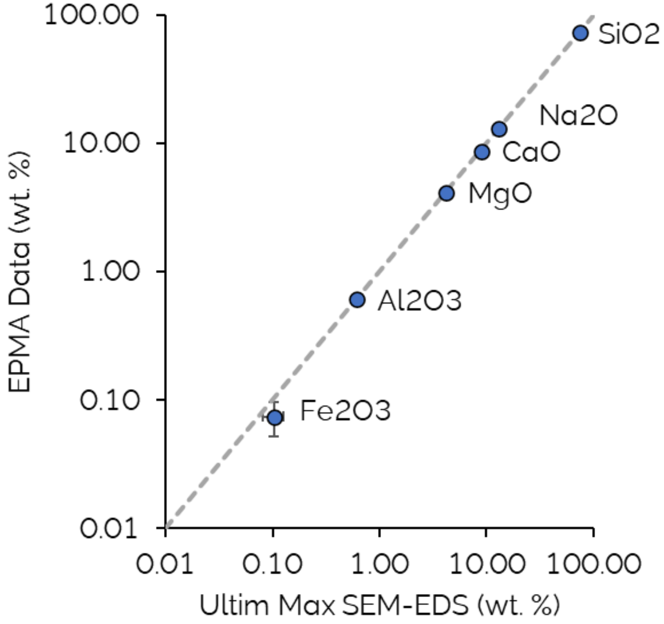
# Leaps in EDS Technology: Ultim Max Series EDS (2018)

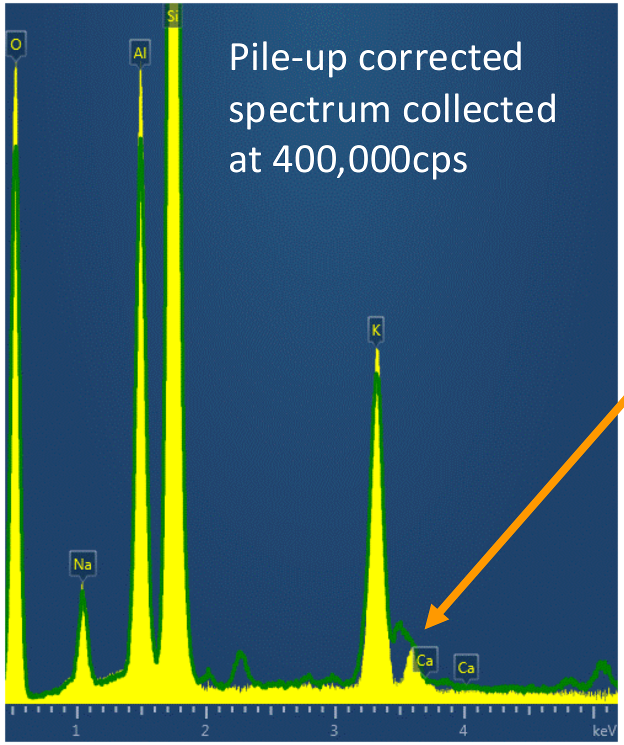
**Light, trace & minor elements with speed and reliability**

Tackle More Challenging Tasks with Greater Counting Statistics:

- Minor and trace elements
- Quantification

Glass analysis – 200 kcps





Pile-up corrected spectrum collected at 400,000cps

Minor Ca in Orthoclase, large interference from pile-up

Low degradation in resolution with increased count rate

| | O | Na | Al | Si | K | Ca |
|---|---|---|---|---|---|---|
| **4,000cps** | 46.68 | 2.55 | 9.92 | 30.56 | 10.04 | 0.25 |
| **400,000cps** | 46.71 | 2.55 | 9.89 | 30.62 | 9.97 | 0.27 |

# Leaps in EDS Technology: Ultim ∞ series & Tru-Q IQ (2024)

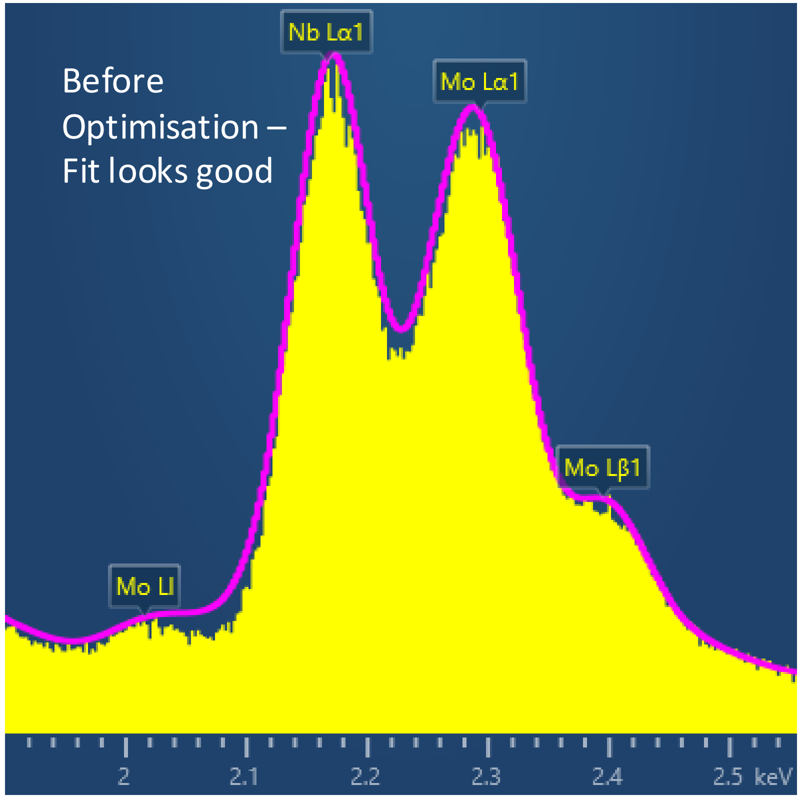
- **New Tru-Q® IQ where every detector produces the same high quality, reliable results** solving the most difficult overlaps and finding the most hidden elements
- **Optimisation of the data processing engine to fit the unique performance of every detector**
- **Peak shapes / positions at all energies that fit the collected data,** so the smallest signals are revealed

Mn resolution ≤ 127 eV @ 200,000 cps

**Same resolution as Ultim with 150% count rate**

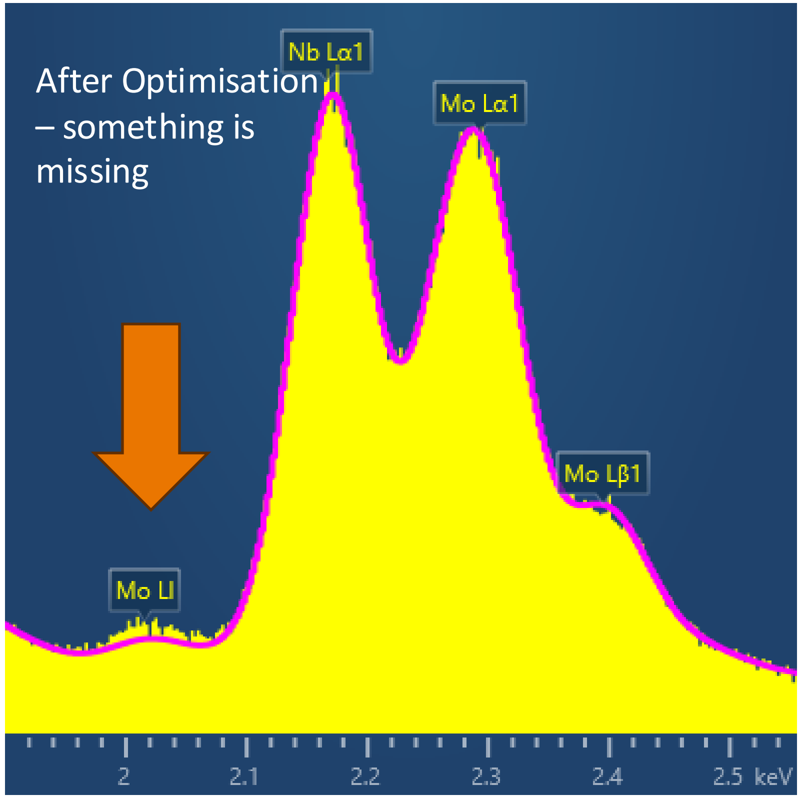
# Leaps in EDS Technology: Ultim ∞ series & Tru-Q IQ (2024)

## Improved spectrum processing ability with individual characterisation



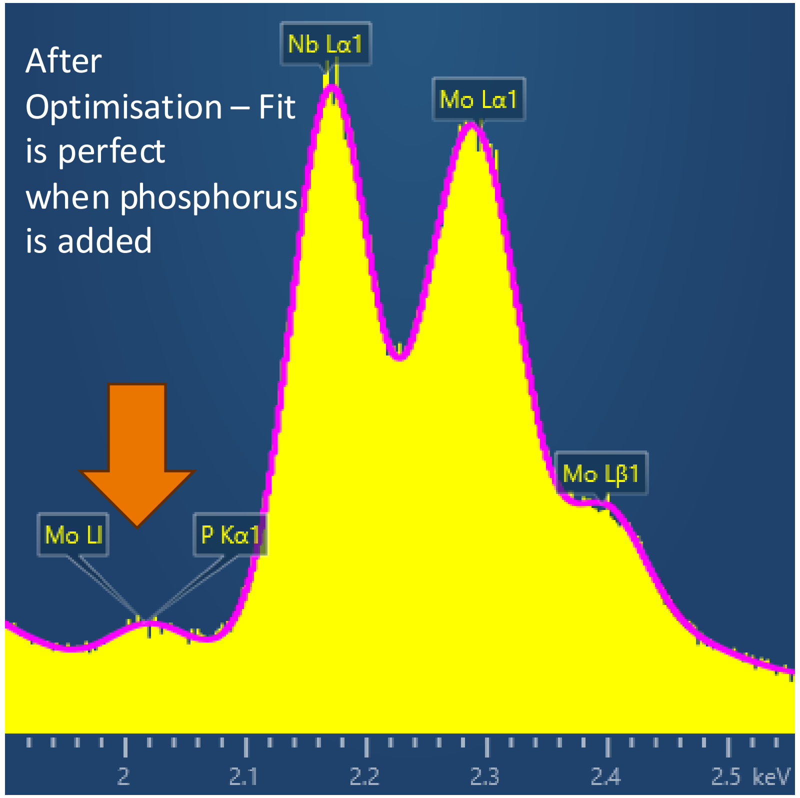
Before Optimisation – Fit looks good

**With Tru-Q IQ** every Infinity detector will be able to detect the most hidden element signals.



After Optimisation – something is missing

Processing optimised for unique performance of each detector.

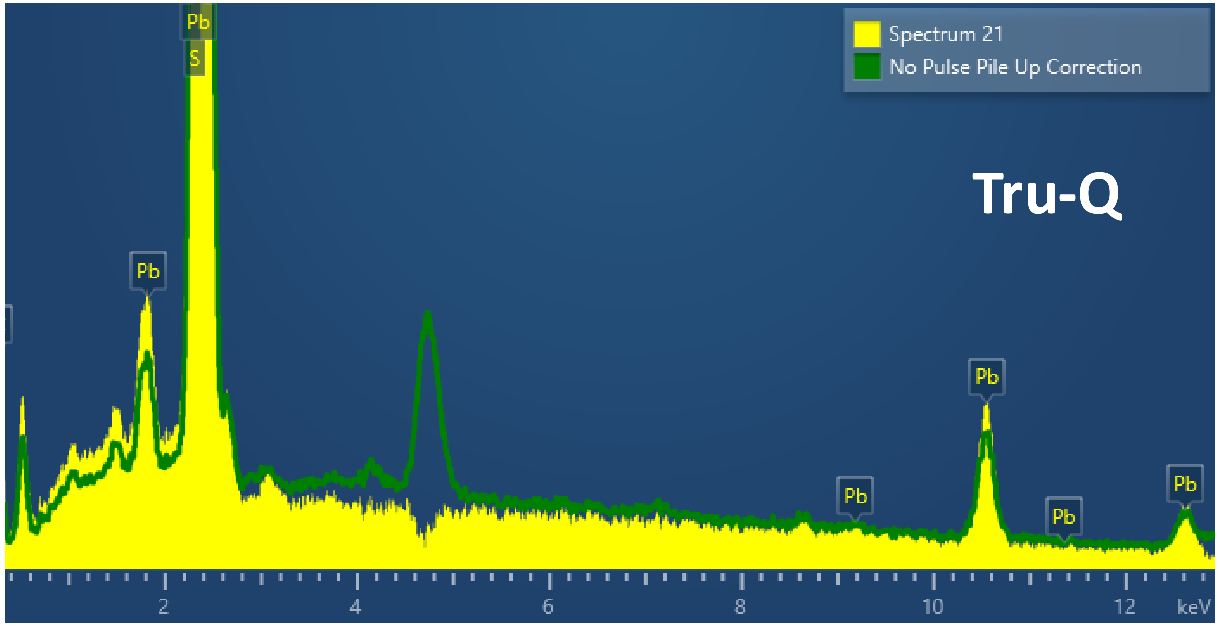


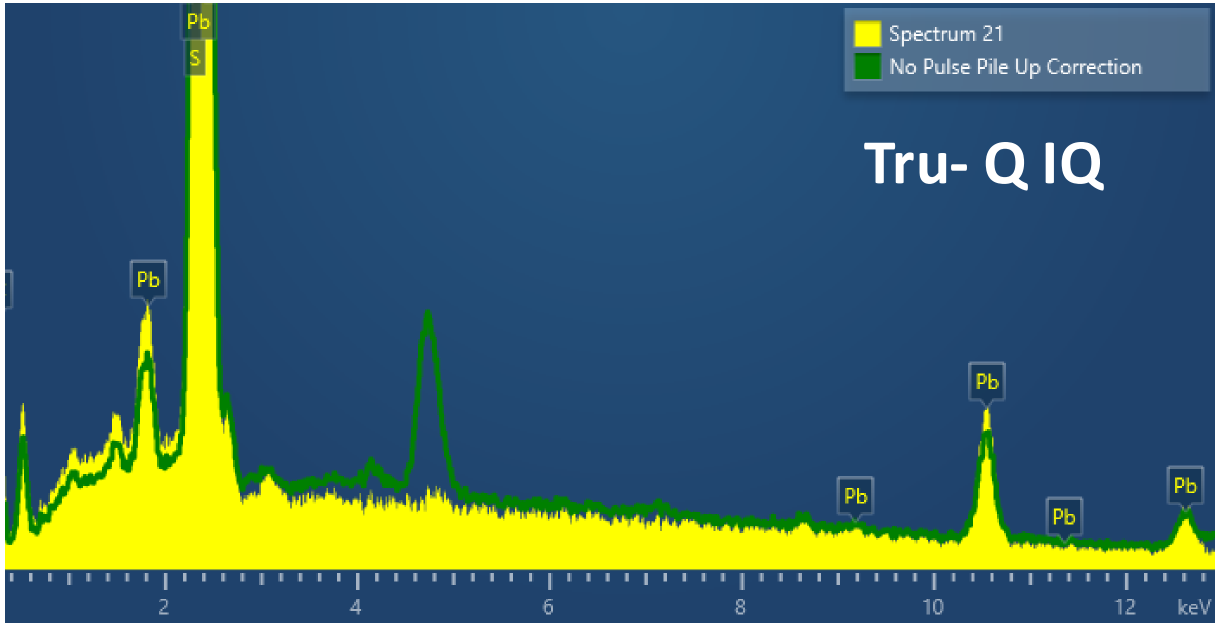
After Optimisation – Fit is perfect when phosphorus is added

- Maintained accuracy at high speeds
- Practical trace and minor element characterisation

# Tru-Q IQ (pile up)

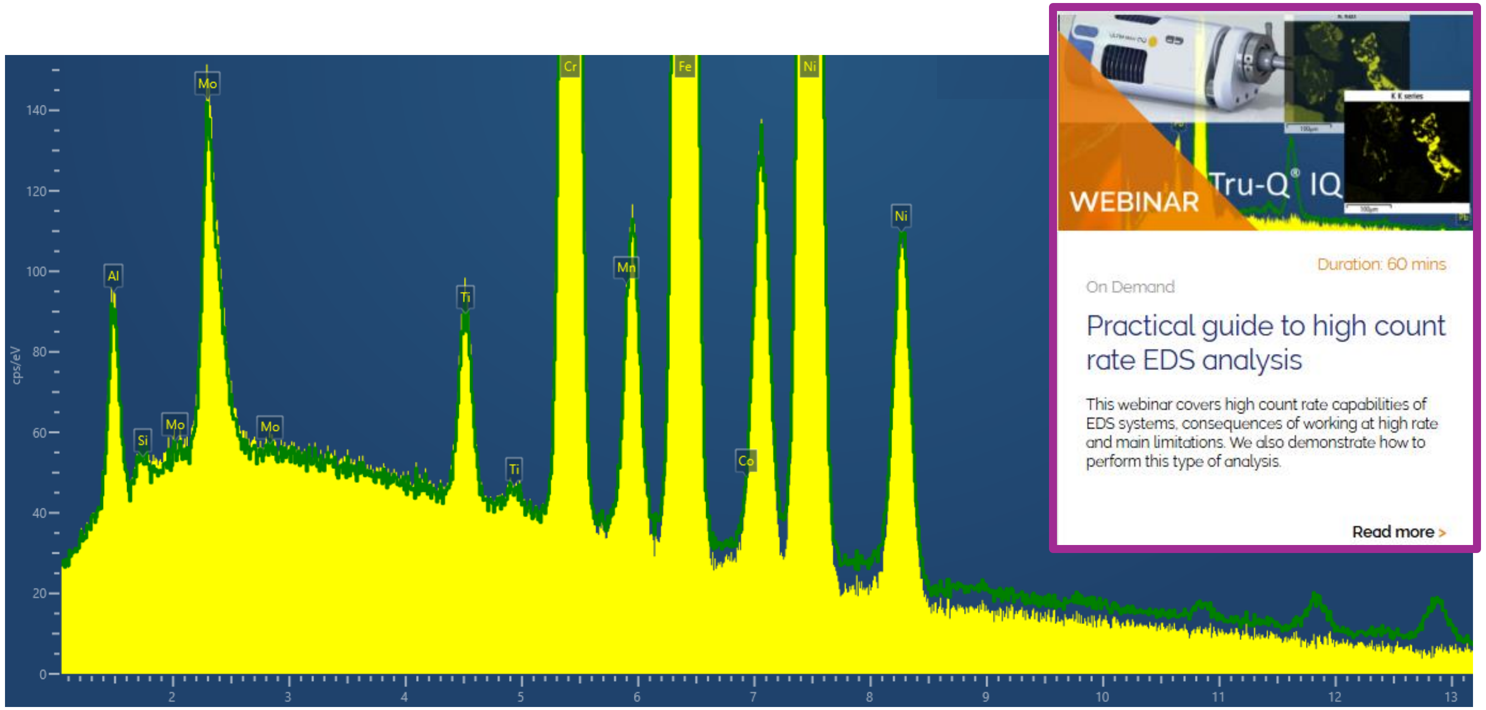
- Example of Pile-up coefficient optimisation (PUC)





# Leaps in EDS Technology: Ultim ∞ series & Tru-Q IQ (2024)

## Optimal pile up correction gives accurate quant even at high count rate



Duration: 60 mins

On Demand

Practical guide to high count rate EDS analysis

This webinar covers high count rate capabilities of EDS systems, consequences of working at high rate and main limitations. We also demonstrate how to perform this type of analysis.

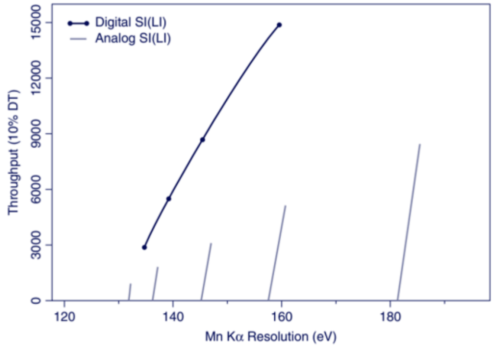
Read more >

| Element | wt% with PPC | wt% without PPC | Diff. (%) |
|---|---|---|---|
| Al | 1.3552 | 1.2381 | 8.6 |
| Si | 0.13 | 0.14 | -10.0 |
| Ti | 1.20 | 1.10 | 7.9 |
| Cr | 17.92 | 16.54 | 7.7 |
| Mn | 0.03 | 0.00 | 93.1 |
| Fe | 34.32 | 31.52 | 8.2 |
| Co | 0.10 | 0.09 | 6.5 |
| Ni | 42.65 | 39.16 | 8.2 |
| Mo | 3.44 | 3.18 | 7.5 |
| Total | 101.14 | 92.97 | |

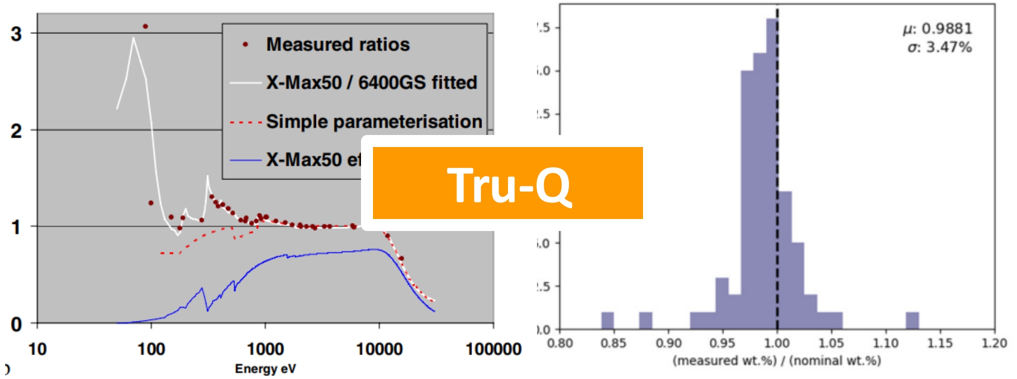
- Un-normalised data
- low and high-count rate data (115 cps vs 840 cps)
- Same quality of the results with different settings

# Summary: How EDS Closed the Gap to WDS

Digital Spectrum Processing



**Speed –resolution - stability**



**Tru-Q**

**Accurate & repeatable spectrum processing for major, minor and traces**

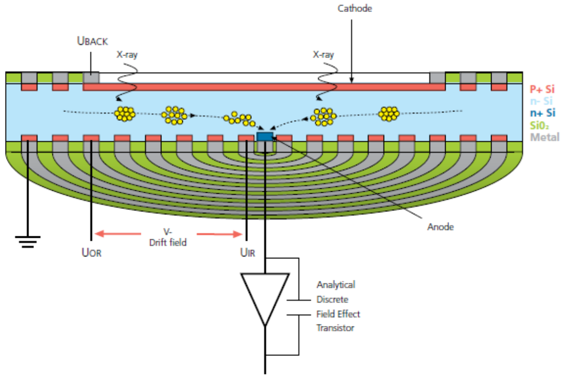
**Tru-Q IQ**

**Further noise reduction, advanced pile-up correction and individual optimization**

**1990**

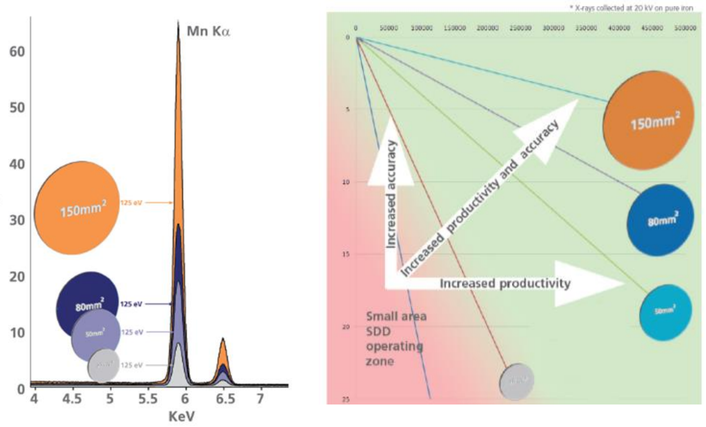
**2025**

SDD Sensors



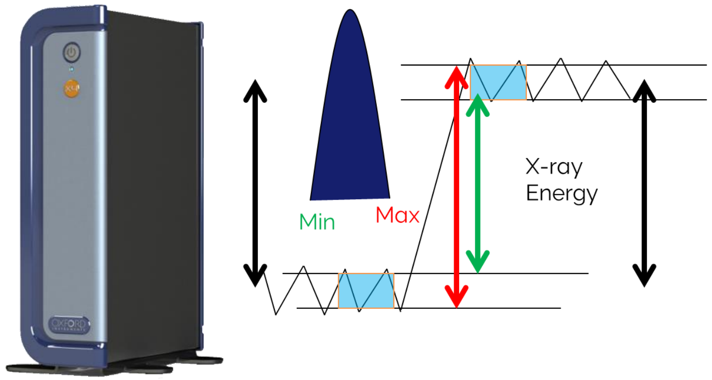
**Reduced noise – higher count rates**

Large-Area SDD Sensors



**More counts – same resolution – lower currents**

Ultim Max

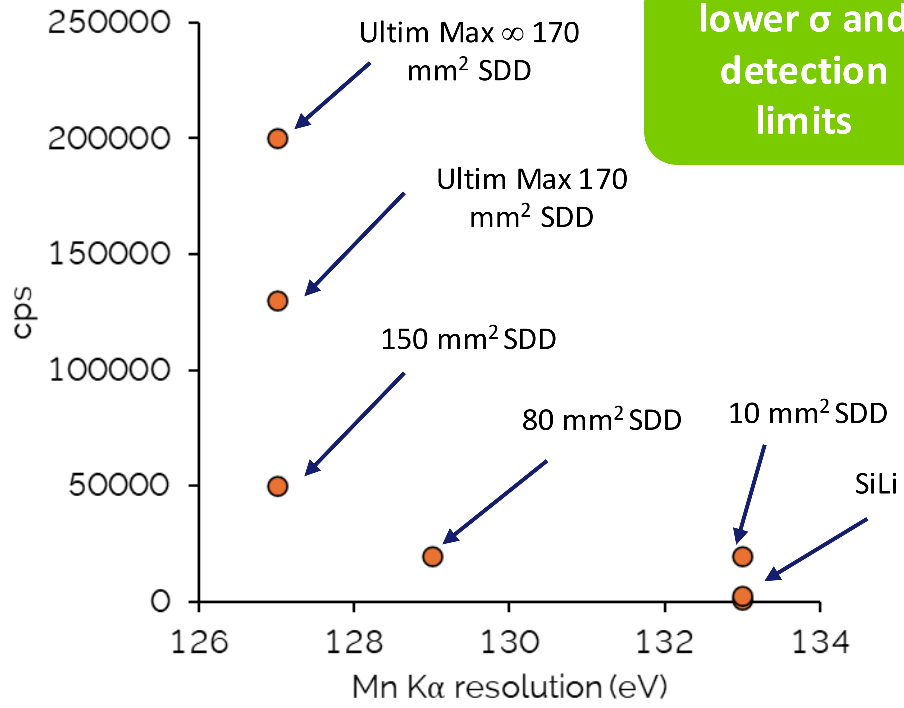


**Reduced noise - higher count rates**

# Summary: How EDS Closed the Gap to WDS

## Spectral Resolution and Speed

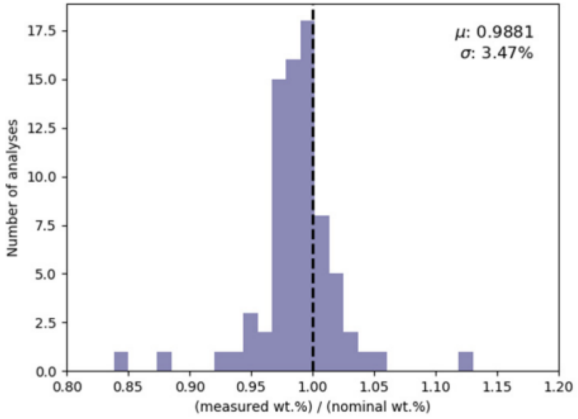
**Generate lots of counts in far less time!!**



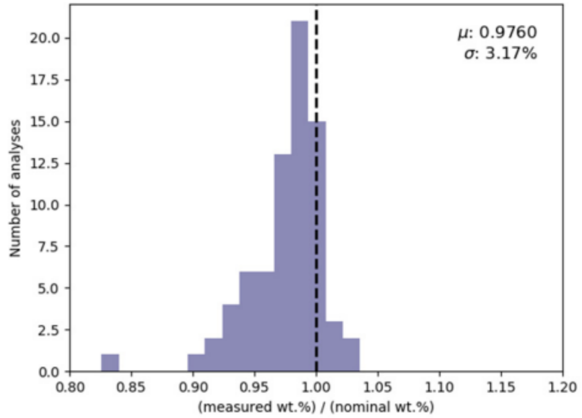
**lower σ and detection limits**

## Accuracy and Precision

**20 kV, 50 kcps**



**20 kV, 200 kcps**



## Detection limits

In the 100's of ppm

- Estimated using 3σ and synthesised spectrum
- Dependent on the matrix and kV

Detection Limit in Steel – 20 kV – 100 M counts

| Element | Wt. % | Wt. % σ | Detection limit (3 X wt.% σ) |
|---|---|---|---|
| Ti | 1 | 0.004 | 0.012 |

**Bulk (wt.%), Ni = 8, Fe = 70, Mn = 2, Cr = 19, Ti = 1**

# How to Achieve These Results

Realising WDS data quality thanks to modern EDS technology (in practice…)

# Best practice workflow

## Recommended Workflow for Quantitative Analysis

OXFORD INSTRUMENTS

Contact us:

**Introduction**

- Within AZtec "standardless" method is the default method for quantitative analysis, and if used in conjunction with the Oxford Instruments Recommended Workflow, it will give fast, accurate results for all but the most challenging and complex samples.
- This recommended workflow describes several considerations that are important for ensuring that quantitative analysis gives the correct results.

1

**Sample preparation and stage setup**

- Align any samples/standards so that they are at the same height on the stage
- Samples should be: flat, polished, conductive (e.g. C coating)
- Load a pure metal (e.g. Cu, Co, Mn, Si) for beam measurements

2

**SEM and EDS setup**

- Check and set the recommended WD for your system
- Set the accelerating voltage (20 kV recommended for standardless microanalysis )
- Remove lens hysteresis
- Use the stage Z position to bring the sample into focus

3

**SEM analytical conditions**

- Adjust beam current to achieve:
  - Input count rate between 50 and 200 kcps
  - Set process time (PT) to between 3-6 and ensure dead time is ~50% or below

4

**Beam measurement - for un-normalised quant**

- Perform a beam measurement on the pure metal
- Check focus (adjust stage Z)
- Set magnification to >10,000x
- Use same accelerating voltage and beam current for the beam measurement and the unknown sample
- Check beam currently stability during your session

5

**Measuring the sample**

- Enter appropriate coating information
- High magnification (e.g. >10,000x)
- EDS Acquire Spectrum settings:
  - Set count limit to 1,000,000 counts
  - Ensure Pulse Pile Up Correction is on

6

**During/post acquisition**

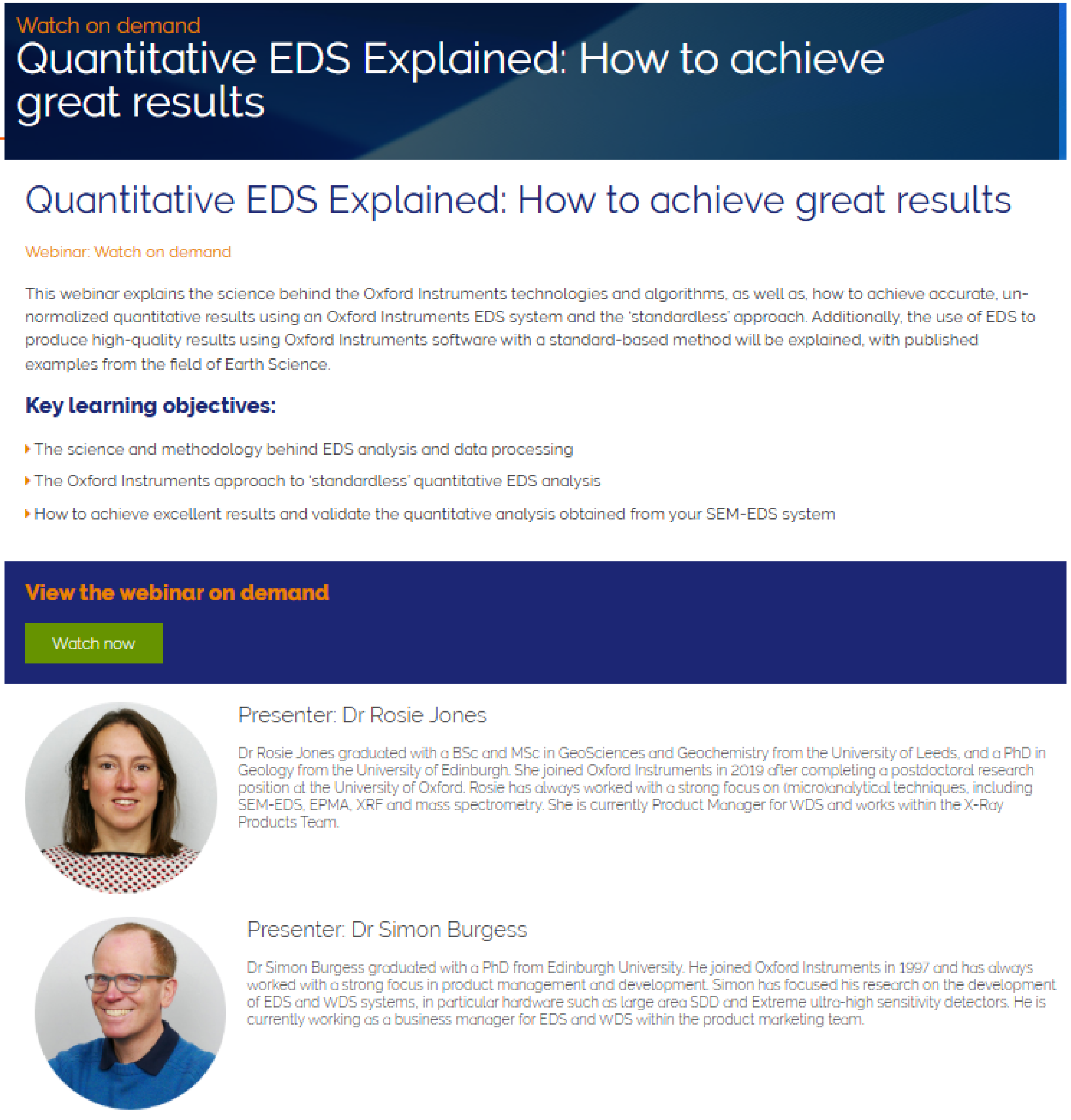
- Verify element AutoID is correct
- This can be done using:
- Fitted spectrum (pink)
  - Analytical total, normally 100% ± 2% (unnormalised analysis)
  - Load a pure metal (e.g. Cu, Co, Mn, Si) for beam measurements

7

**During/post acquisition**

- Use 'Calculate Composition' to view and interrogate the result
- Check the quant settings
- Processing options
  - Normalised vs. Un-normalised
  - Select the appropriate quant standardisations database – Factory or User

Tip: 'Quant standardisations' for 10-30 kV 'Quant standardisations (5 kV set)' for 5-10 kV



Watch on demand

## Quantitative EDS Explained: How to achieve great results

## Quantitative EDS Explained: How to achieve great results

Webinar: Watch on demand

This webinar explains the science behind the Oxford Instruments technologies and algorithms, as well as, how to achieve accurate, un-normalized quantitative results using an Oxford Instruments EDS system and the 'standardless' approach. Additionally, the use of EDS to produce high-quality results using Oxford Instruments software with a standard-based method will be explained, with published examples from the field of Earth Science.

### Key learning objectives:

- The science and methodology behind EDS analysis and data processing
- The Oxford Instruments approach to 'standardless' quantitative EDS analysis
- How to achieve excellent results and validate the quantitative analysis obtained from your SEM-EDS system

**View the webinar on demand**

Watch now

Presenter: Dr Rosie Jones

Dr Rosie Jones graduated with a BSc and MSc in GeoSciences and Geochemistry from the University of Leeds, and a PhD in Geology from the University of Edinburgh. She joined Oxford Instruments in 2019 after completing a postdoctoral research position at the University of Oxford. Rosie has always worked with a strong focus on (micro)analytical techniques, including SEM-EDS, EPMA, XRF and mass spectrometry. She is currently Product Manager for WDS and works within the X-Ray Products Team.

Presenter: Dr Simon Burgess

Dr Simon Burgess graduated with a PhD from Edinburgh University. He joined Oxford Instruments in 1997 and has always worked with a strong focus in product management and development. Simon has focused his research on the development of EDS and WDS systems, in particular hardware such as large area SDD and Extreme ultra-high sensitivity detectors. He is currently working as a business manager for EDS and WDS within the product marketing team.

**WEBINAR**

OXFORD INSTRUMENTS

# Application examples

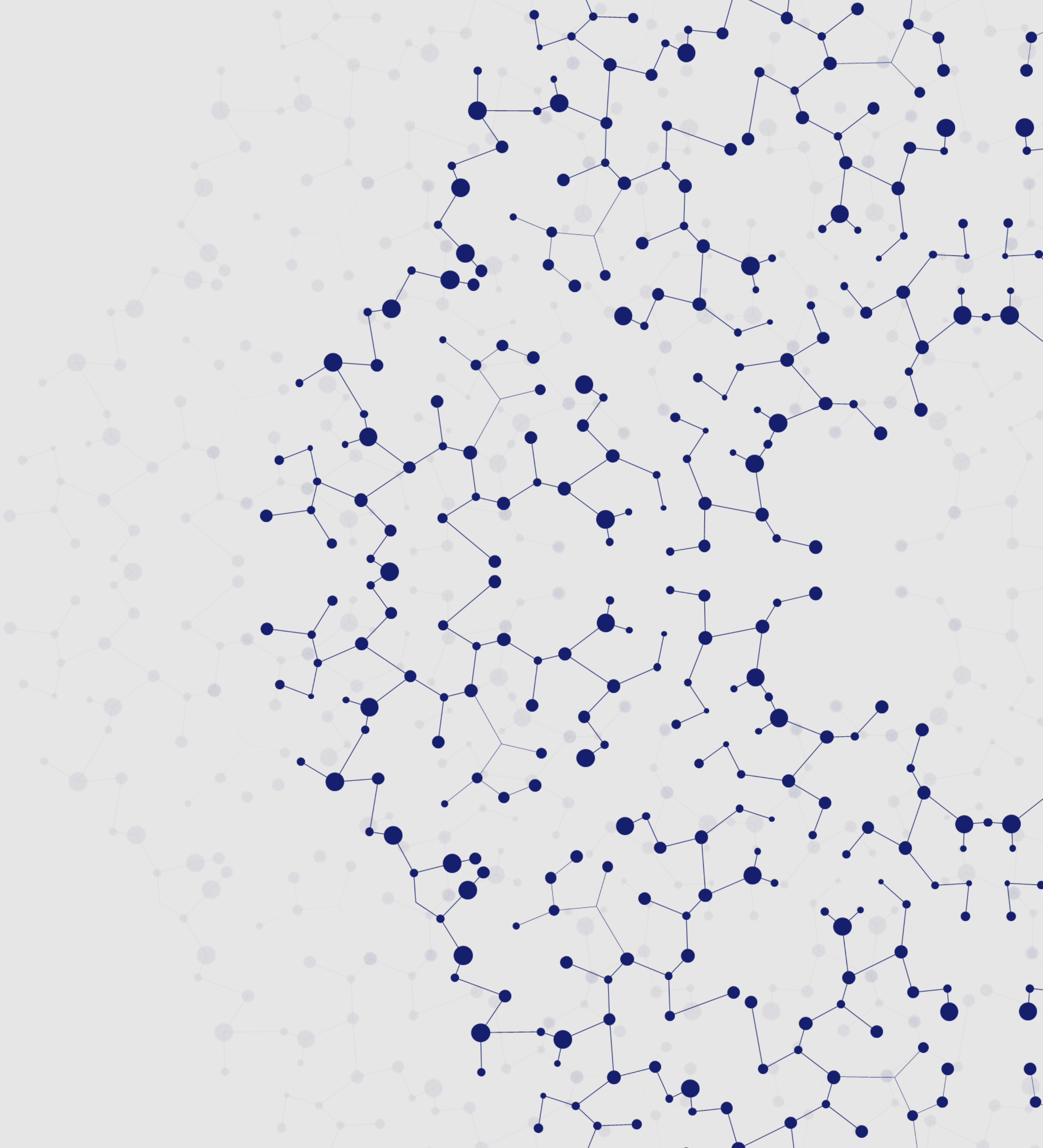
# Summary SEM-WDS Applications

## WDS major application areas

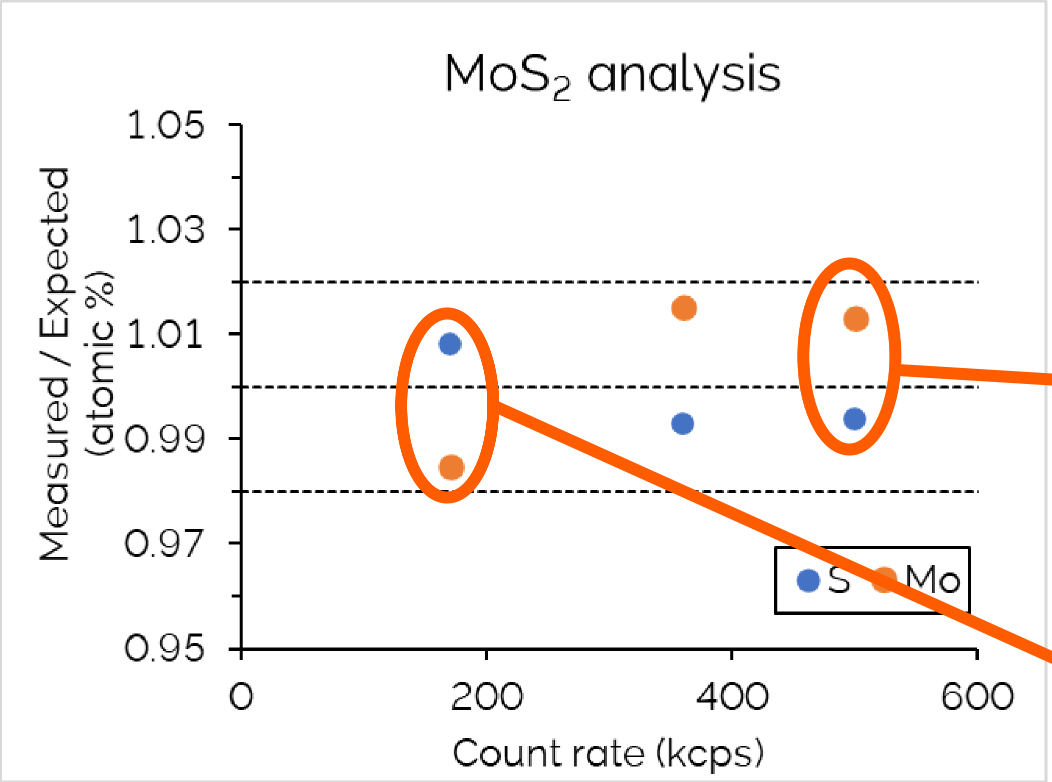
- Analysis of difficult spectral overlaps
- Characterisation of minor and trace elements
- High precision and high accuracy quantitative analysis
- Minor-trace element mapping

# Solving Peak Overlaps

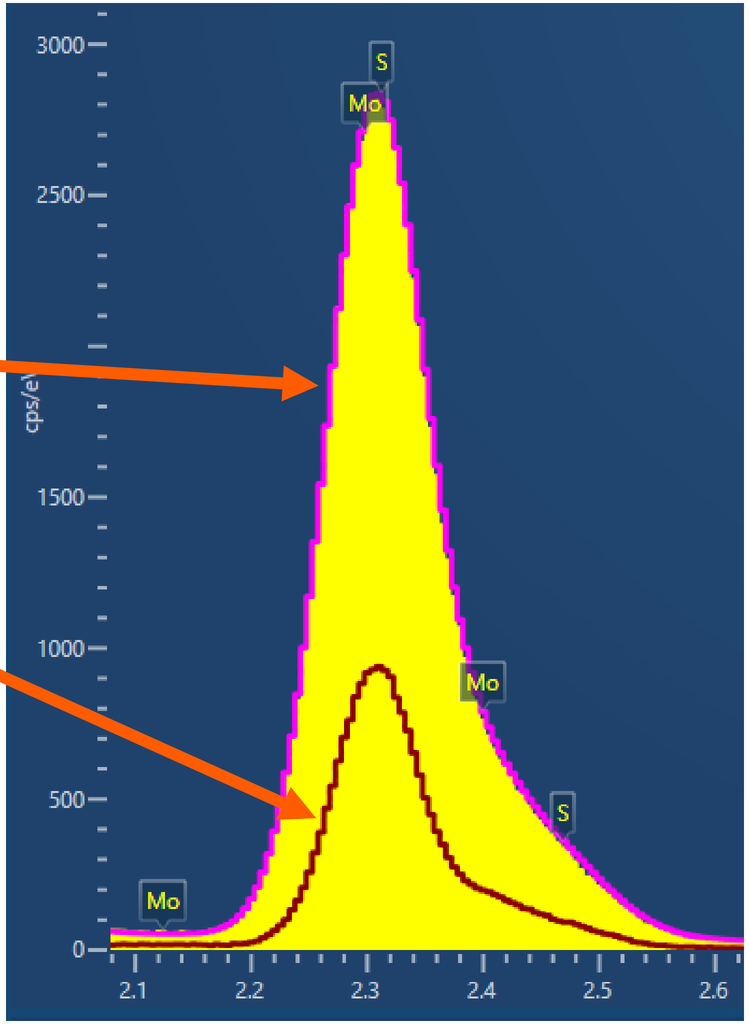
## Deconvoluting Mo and S in $MoS_2$



# Solving Peak Overlaps: $MoS_2$



| Atomic % | | |
|---|---|---|
| Count rate (kcps) | S | Mo |
| 170 | 67.21 | 32.79 |
| 380 | 66.19 | 33.81 |
| 500 | 66.26 | 33.74 |

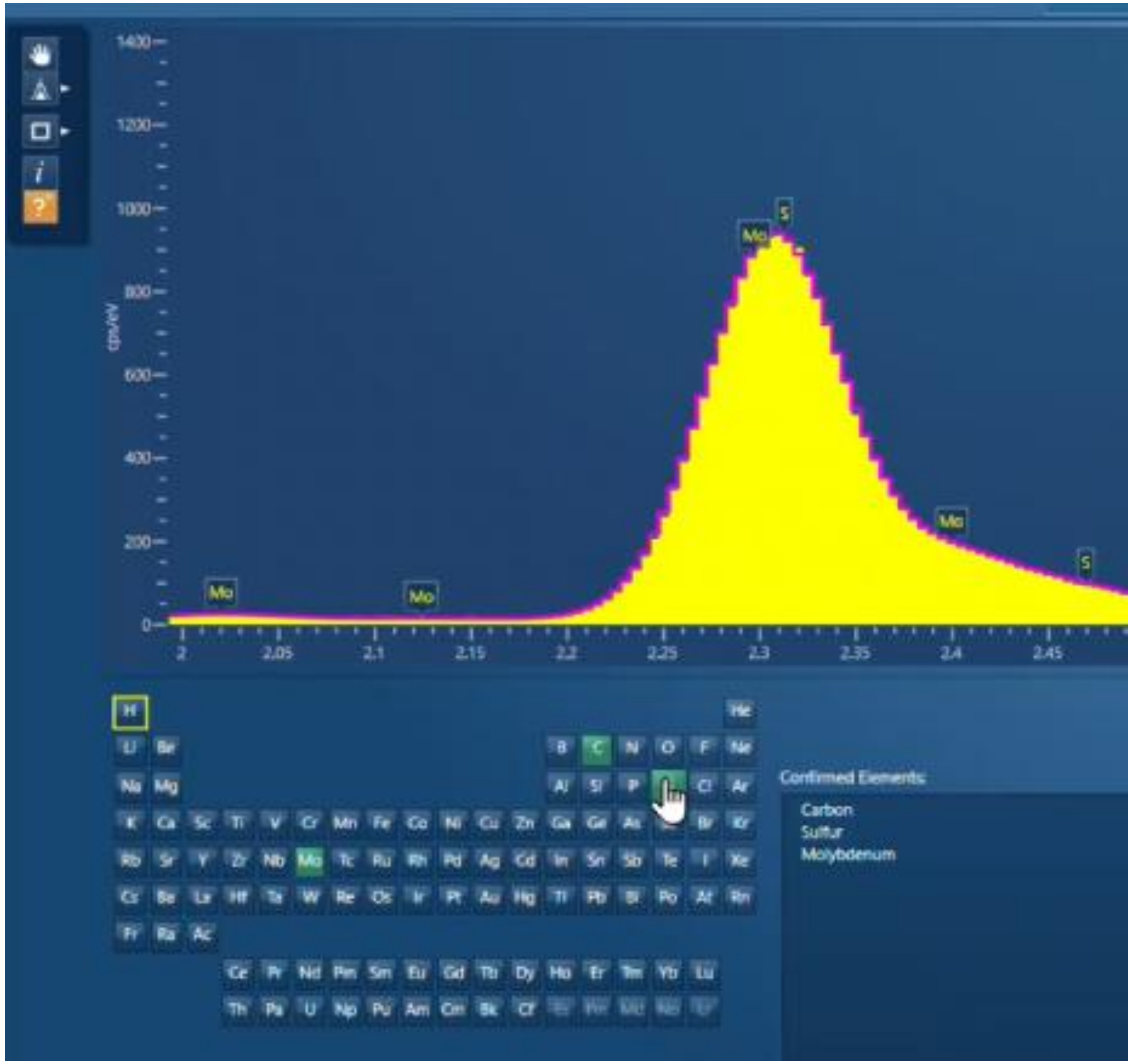


S Kα = 2308 eV
Mo Lα = 2293 eV

**15 eV offset**

< 2% deviation from expected in 2 seconds

# Solving Peak Overlaps: $MoS_2$



Confirmed Elements:
Carbon
Sulfur
Molybdenum

Confirmed Elements:
Carbon
Molybdenum

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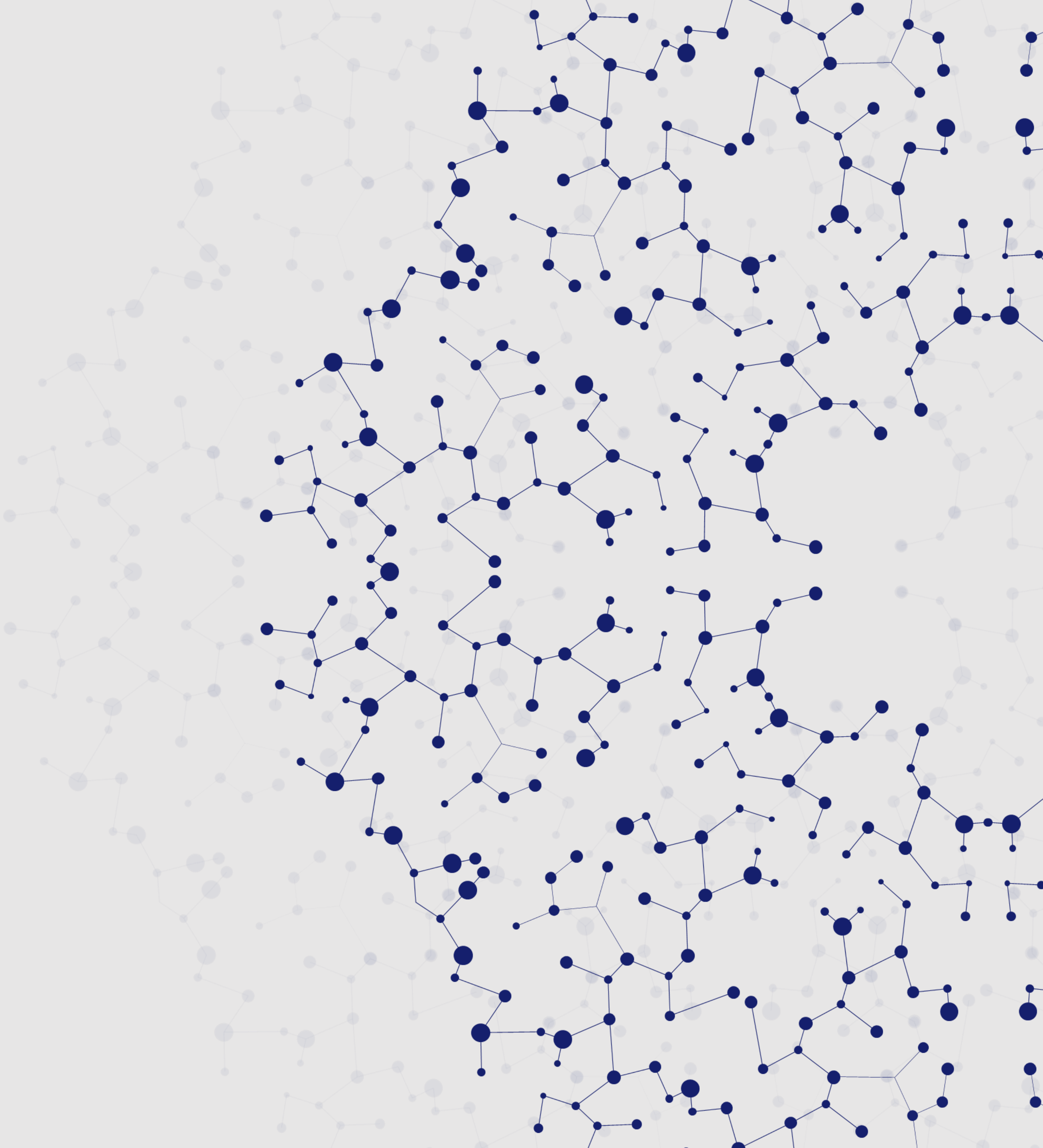
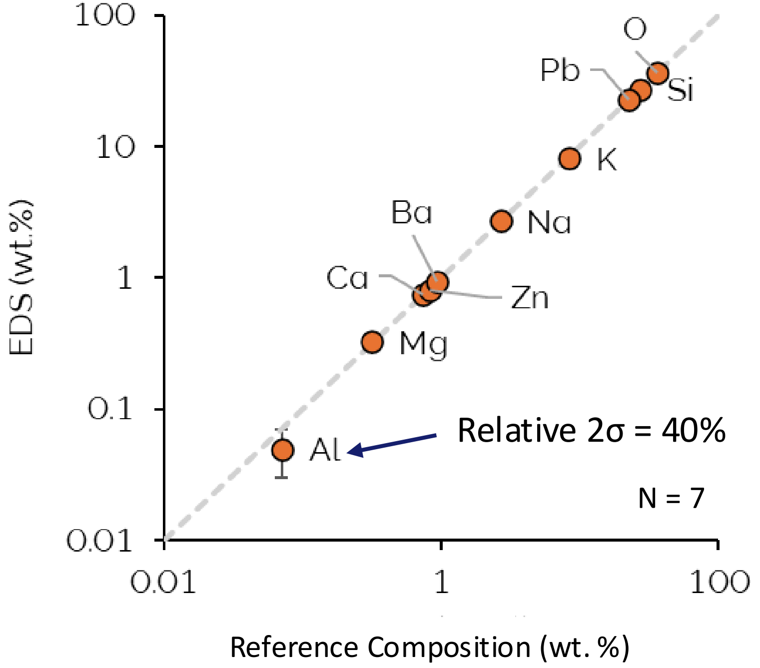


# Trace Element Quantification

Quantifying glass trace elements in the 100’s ppm

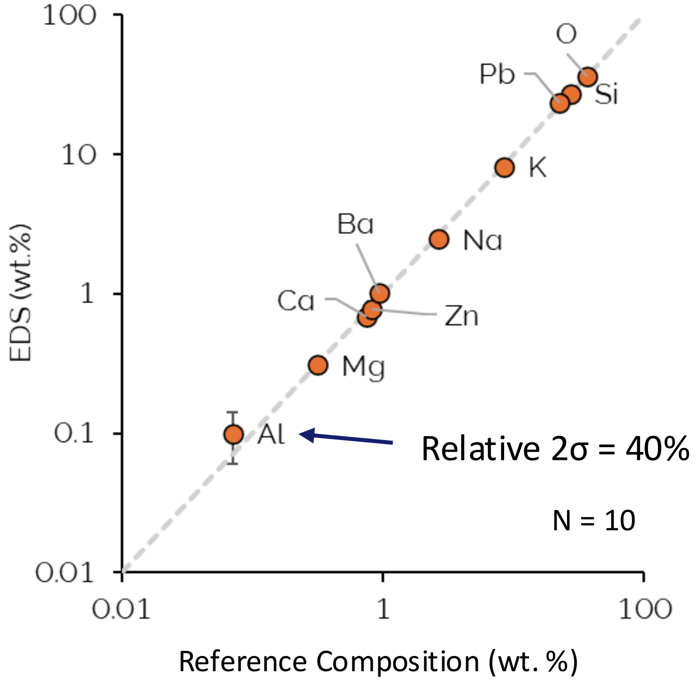
# Glass Standard Quantification: Majors, Minors and Traces



**BCR – 165 kcps – 2 M counts**

Relative 2σ = 40%

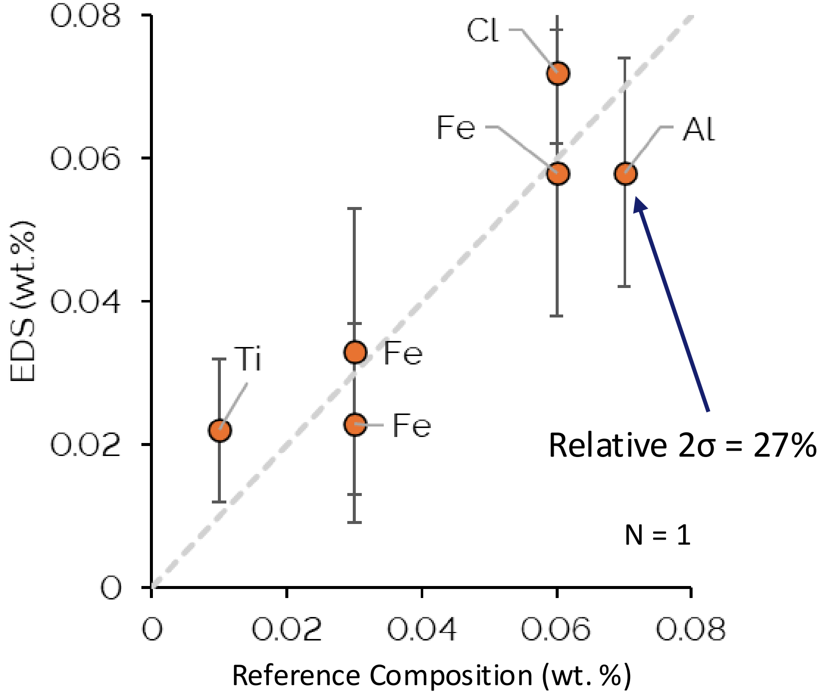
N = 7



**BCR – 670 kcps – 2 M counts**

Relative 2σ = 40%

N = 10



**Glass Trace Elements - 670 kcps – 10 M counts**

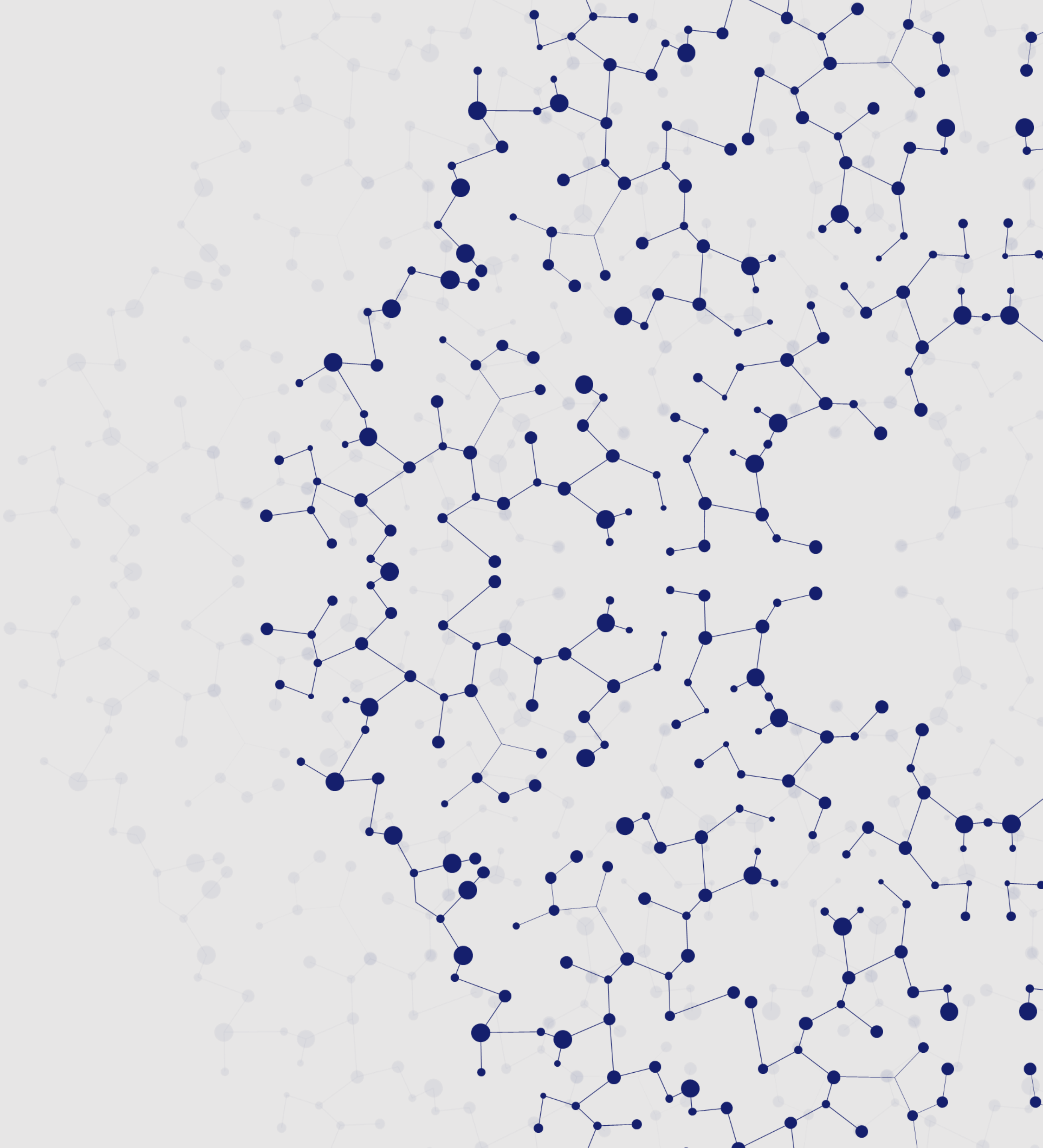
Relative 2σ = 27%

N = 1

**Reference composition determined by WDS**

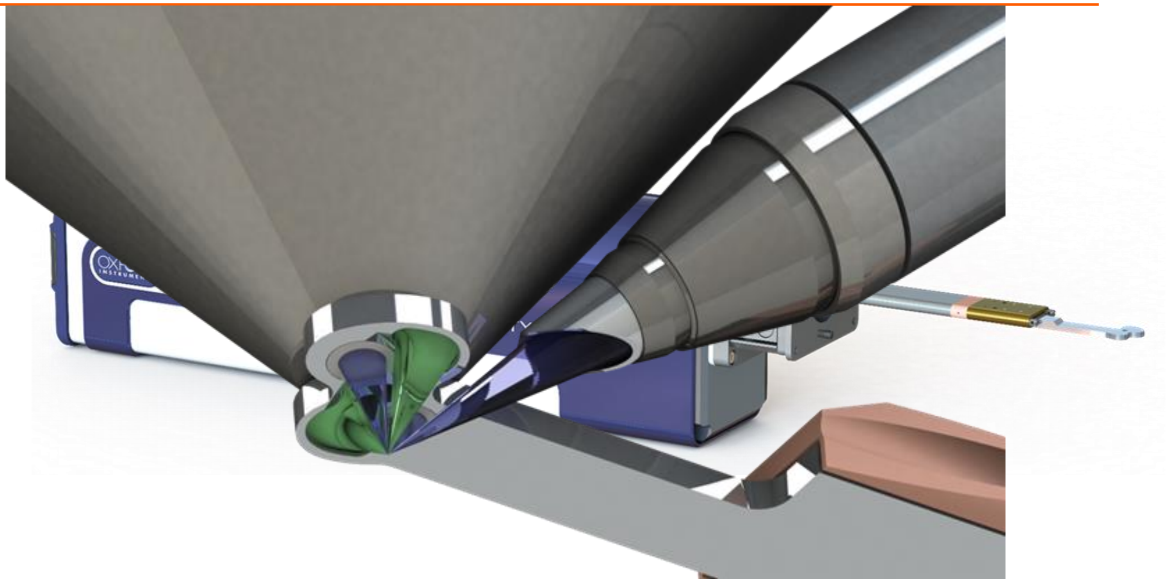
**Error bars = 2σ**

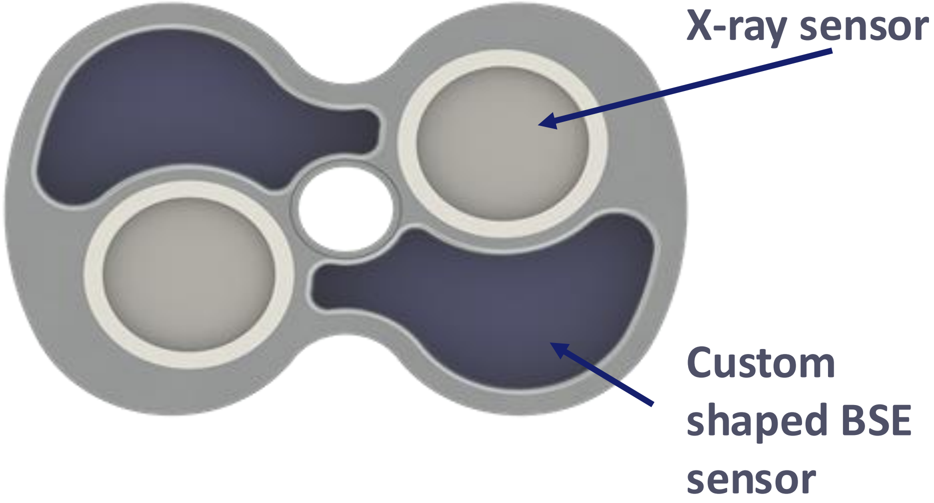
# Minor and trace elements mapping with BEX



# What is Unity?

- **Unity is the world's first BEX detector**
- **Retractable below the pole piece BEX detector**
- **Unity sensors**
  - **Two X-ray sensors (SDD)** providing enhanced sensitivity ranging from 7 to 120 times higher than typical EDS detectors (e.g. 20 X more sensitive than EDS 65mm$^2$ detector)
  - **Two custom shaped solid state BSE sensors** high end BSE sensors
- **Optimised to collect maximum signal without obscuring EDS**
- **Optimized head thickness (3mm) and central hole (3mm) for daily use**
- **Unity Head is Peltier cooled for optimum X-ray performance**
- **Unity design and AZtecLive software features reduces the risk of collision. In addition, the Unity head is replaceable for extra piece of mind**

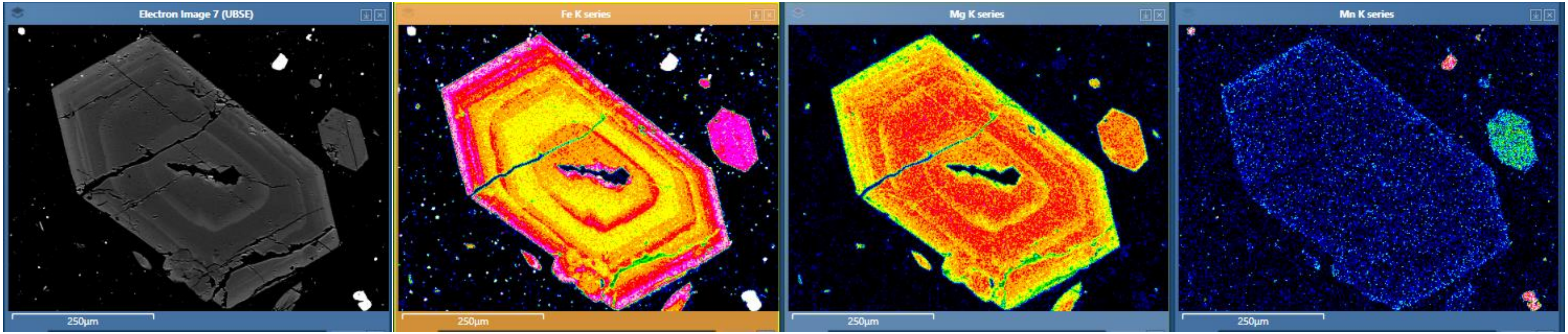




**PATENT GRANTED** GB2621003 **PATENT PENDING** US2023-0258587 EP4179301A1 JP2023-533021 CN116438622

# Visualising minor and trace elements with BEX

*Unity BEX & Ultim Max Infinity*



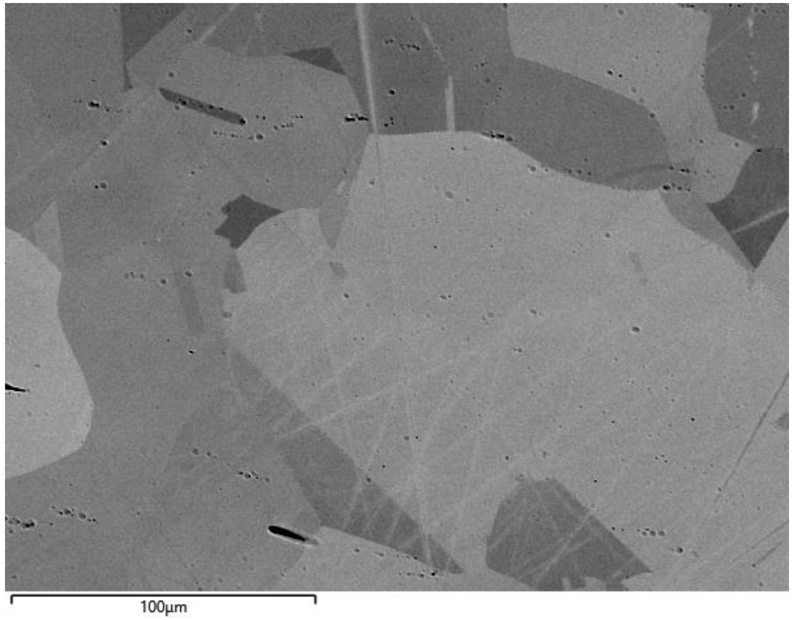
**Mn rich rim** <0.7 wt%

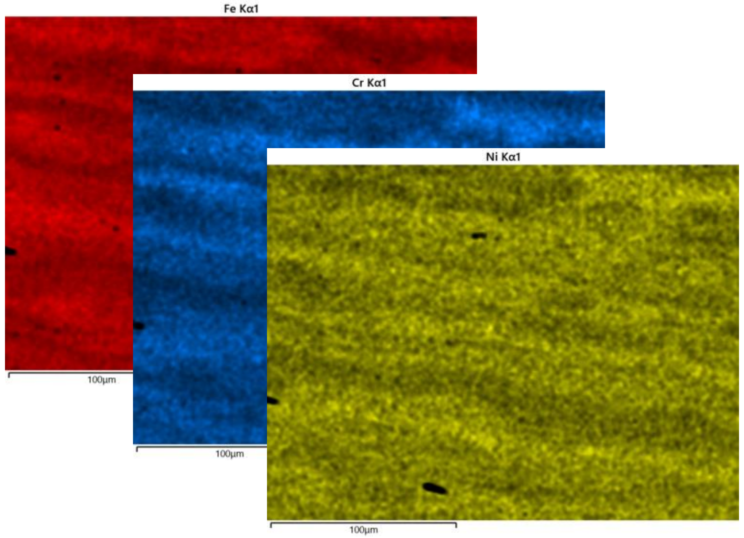
**Challenge:** Mapping elemental concentrations below 0.7 wt%

Unity captures incredibly high signal levels which allow us to visualise zonation of trace levels of Mn in this porphyritic dacite lava from Mt. Pinatubo, Philippines.  Data was acquired in a single frame BEX acquisition (<20min) where equivalent WDS data would take at least 1 hour.

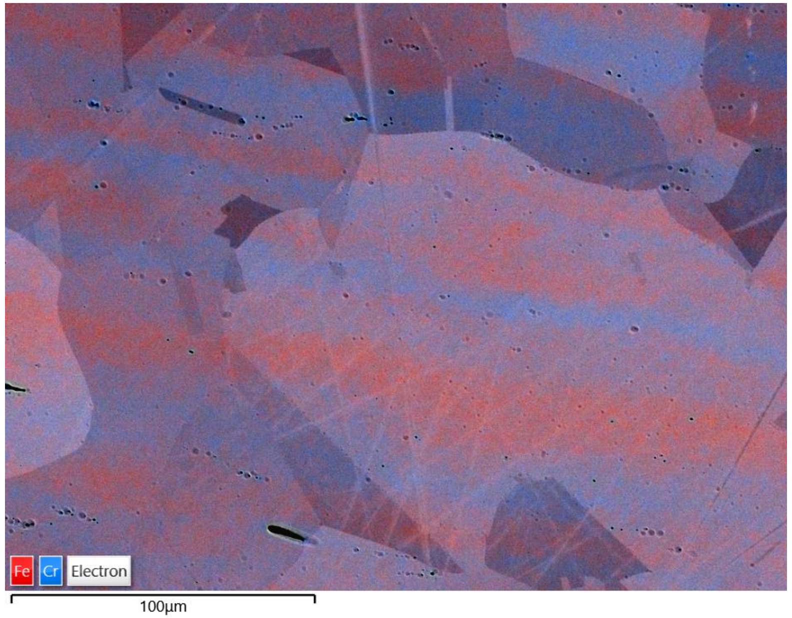
# Visualising small element variation with BEX

*Unity BEX, Ultim Infinity & AutoPhaseMap*





**Ni** 0.1wt% delta **Cr** 0.4 wt% delta



**Challenge:** Mapping small element concentrations variation below 1 wt%

Unity captures incredibly high signal levels which allow us to detect segregation of Cr & Ni in steel in maps collected in just a few minutes. With Unity, we can use Cartography to extend this analysis to mm-scale samples, where WDS would require days to cover a similar area.

# Most challenging EDS applications

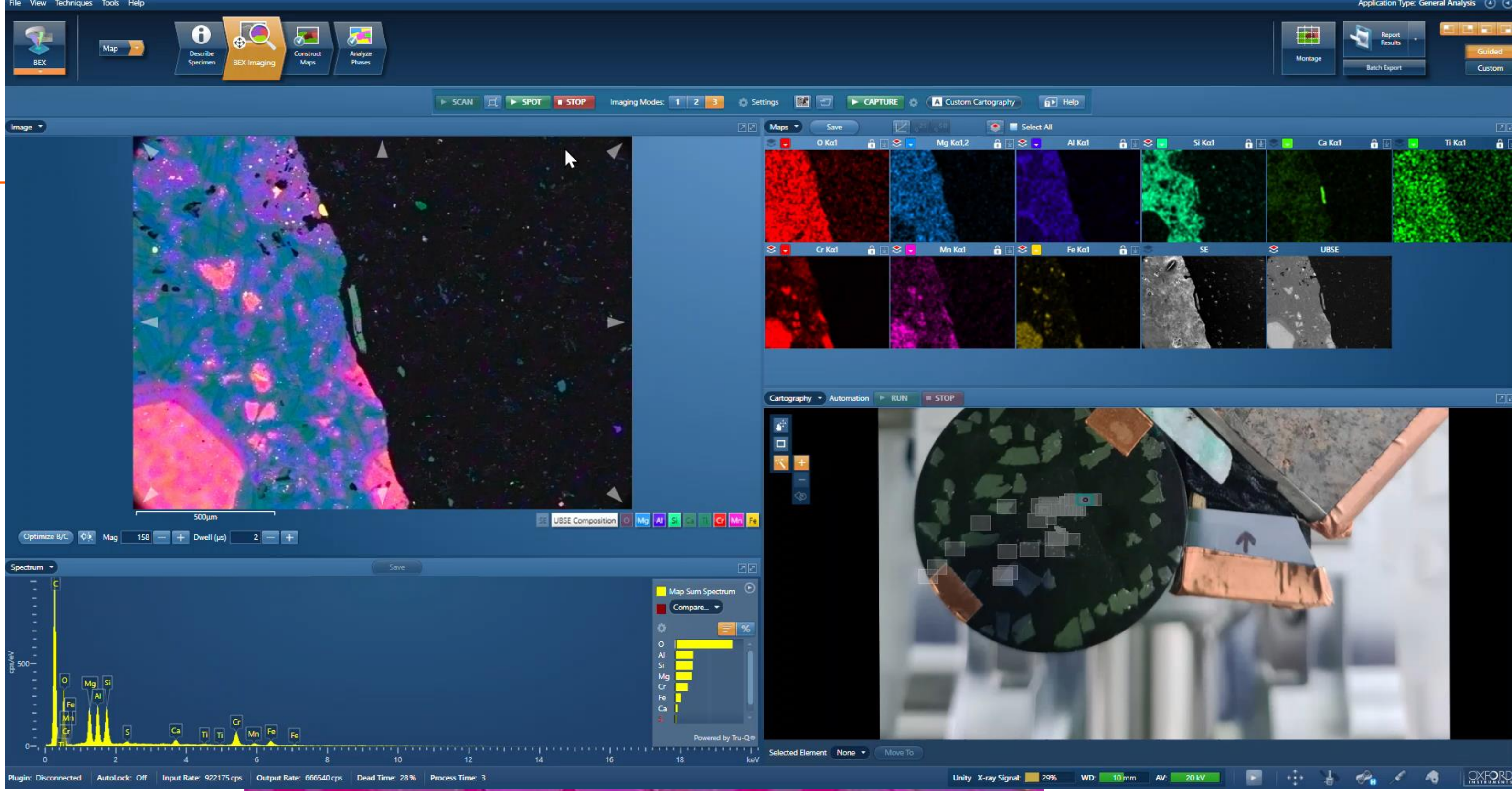
Overlapped X-ray peaks of major elements

Trace elements with concentrations below 1 wt%

EDS quantification at high count rates (> 200 kcps)

# Materials and methods

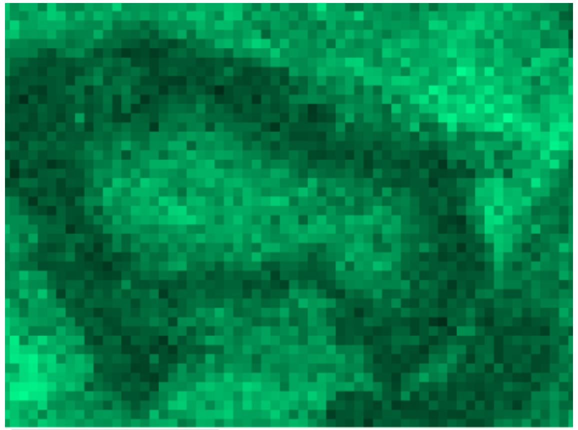
- **Challenge:**
- Well-defined standards containing minor and trace elements whose X-ray peak overlaps with other peaks
  - **Analysed samples: obtained from the experimental work in a steel production furnace** Mn (trace element) affected by overlap with Cr-Kβ (major element), 48 eV separation.
  - **Standard steel sample (XPE alloy)** similar peak overlap between Cr-Kβ and Mn Kα (17 wt% Cr, 0.05 wt% Mn)
  - **Experimental setup:** EDS (Ultim Max Infinity 65, Oxford Instruments) WDS (AZtec Wave, Oxford Instruments,) Hitachi SU-70SEM



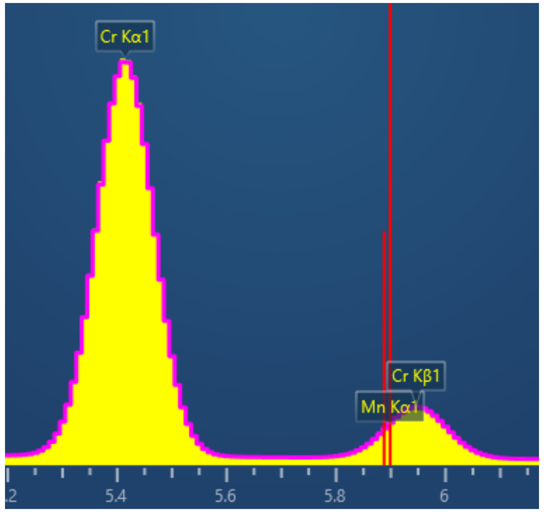
Heterogeneous microstructure contains various major elements and trace elements

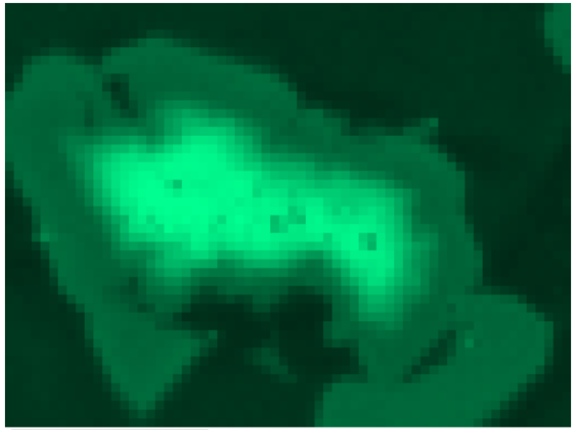
# Analysis of unknown slag sample

- **Mapping EDS and WDS, 20kV, 20nA using PT3 and an input count rate of 500 kcps (dead time of ~45%)**
- **WDS map reference, does not suffer from the strong overlap, and sum peaks**

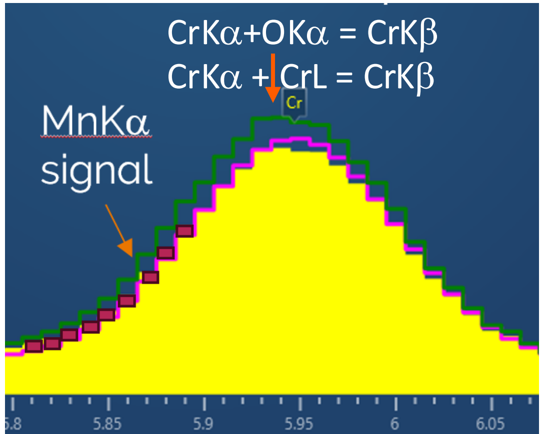


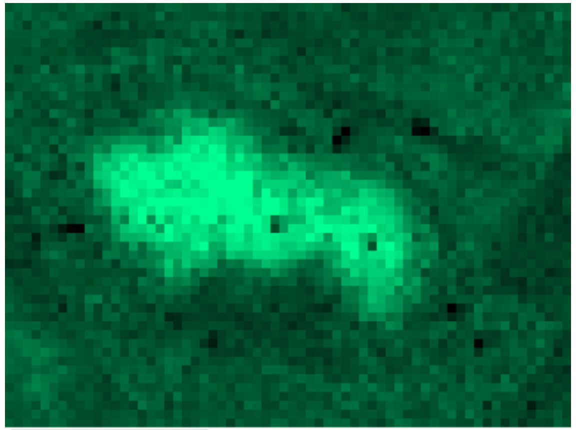
**WDS**



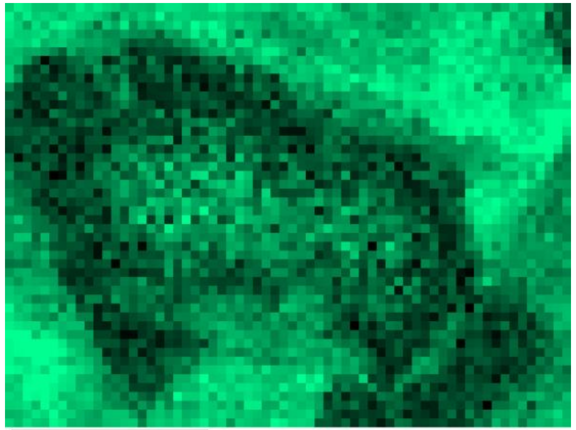


**Window integral**





**Peak deconvolution**



**Pulse pile-up correction and peak deconvolution**

- Even after performing spectrum processing at every pixel of the map to filter the continuum and deconvolve peaks WDS and EDS maps do not display the same information.
- EDS true distribution after pulse pile up correction at every pixel-Mn 0.2 wt%

# Summary: EDS has largely removed the advantage of SEM-WDS

**30 Years of EDS Technology Development:**

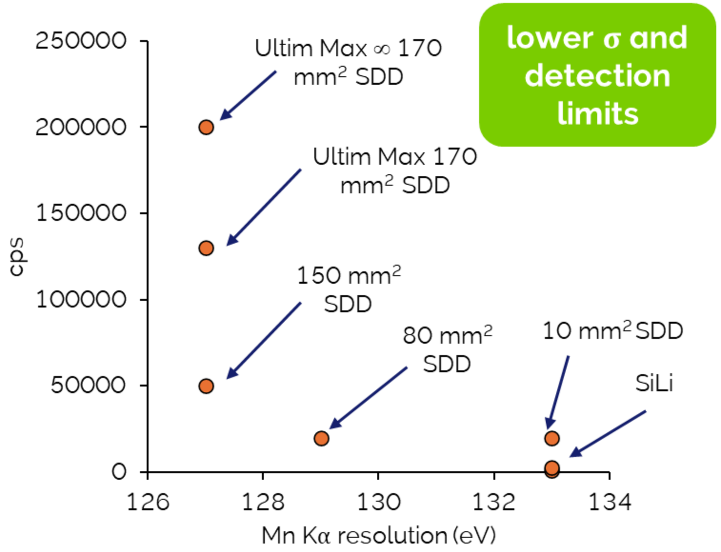
1. Digital Spectrum Processing
2. SDD and Large Area SDD
3. Spectrum Processing, Tru-Q and Tru-Q IQ
4. Low noise electronics
5. Pulse pile up correction

**EDS technology can now rival WDS data quality for the most complex tasks...**

1. Peak overlaps
2. Quantification: major, minor AND traces
3. Mapping: minor and trace elements

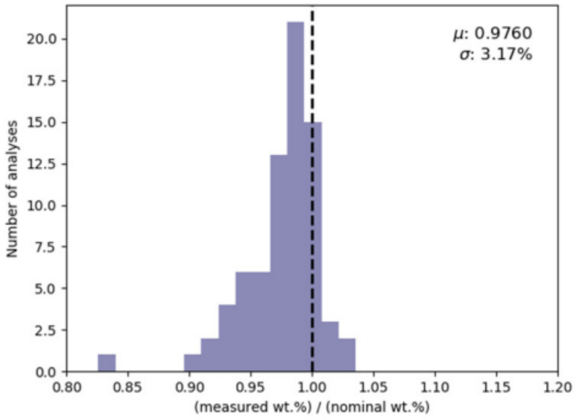
**User friendly, simultaneous acquisition, much faster**

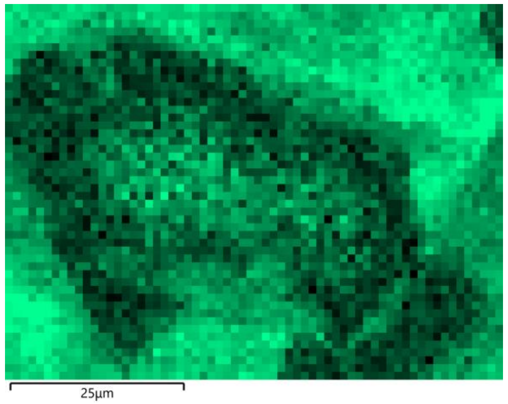
**Generate lots of counts in far less time!!**



**lower σ and detection limits**

**20 kV, 200 kcps**





Detection Limit in Steel – 20 kV – 100 M counts

| Element | Wt. % | Wt. % σ | Detection limit (3 X wt.% σ) |
|---|---|---|---|
| Ti | 1 | 0.004 | 0.012 |

**Bulk (wt.%), Ni = 8, Fe = 70, Mn = 2, Cr = 19, Ti = 1**

# WDS like data quality

# Thank you for your attention!

Dr. Lucia Spasevski
Product Manager
Lucia.Spasevski@oxinst.com

Time for your questions