Update on Energy Dispersive X-ray Spectrometry with the Silicon Drift Detector (SDD) and Microstructural Characterization with NIST Lispix

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Advanced Instrumental Techniques and Software Algorithms in EPMA Workshop

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At the 2006 M&M Conference in Chicago, I gave the following paper:

50 years of x-ray mapping: a tribute to Peter Duncumb

X-ray Mapping in the Spectrum Image Mode at Output Count Rates above 100 kHz with the Silicon Drift Detector (SDD)

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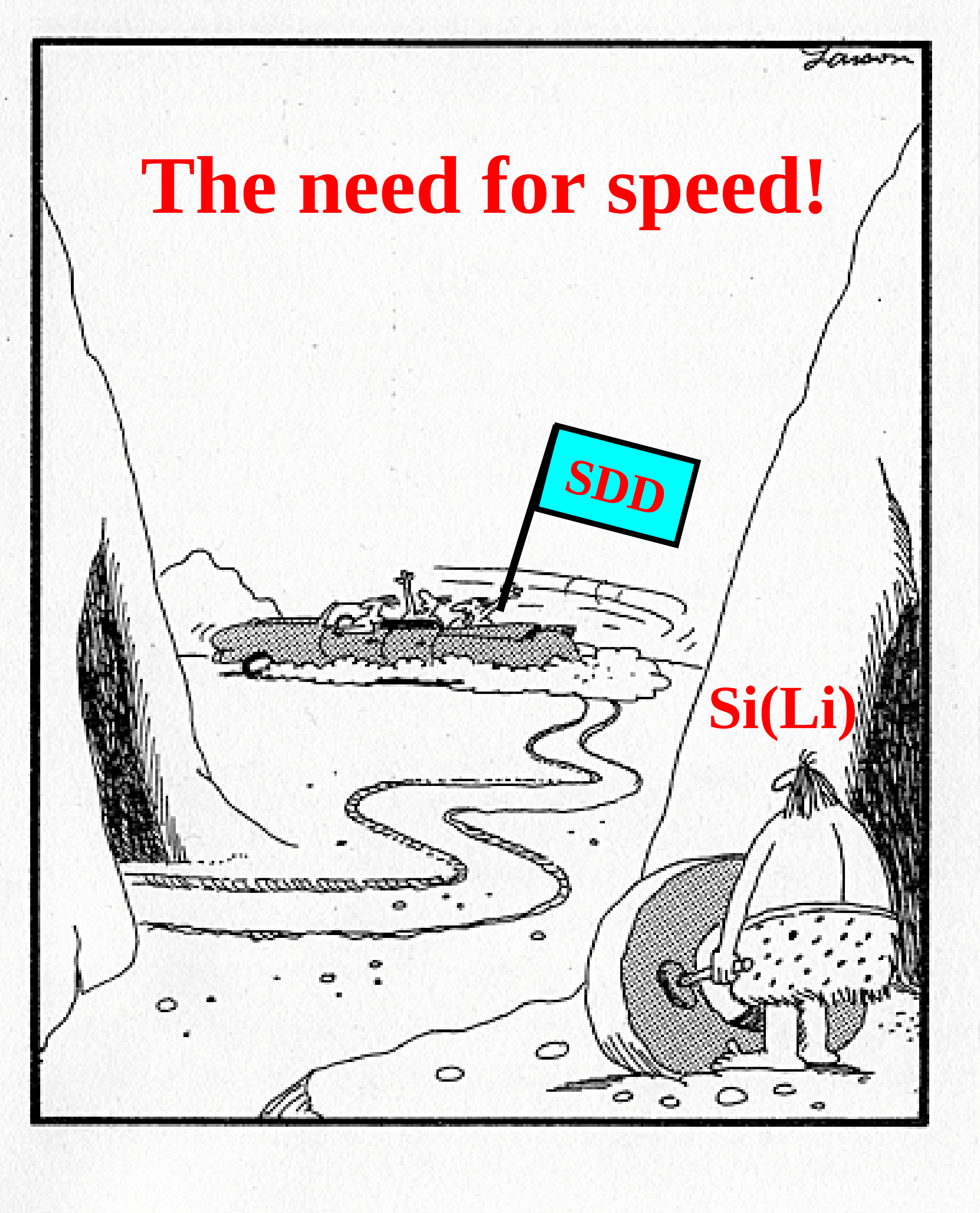
The new EDS: Silicon Drift Detector (SDD)

This paper examined the performance of the SDD for various parameters but especially for high speed X-ray Spectrum Imaging (XSI).

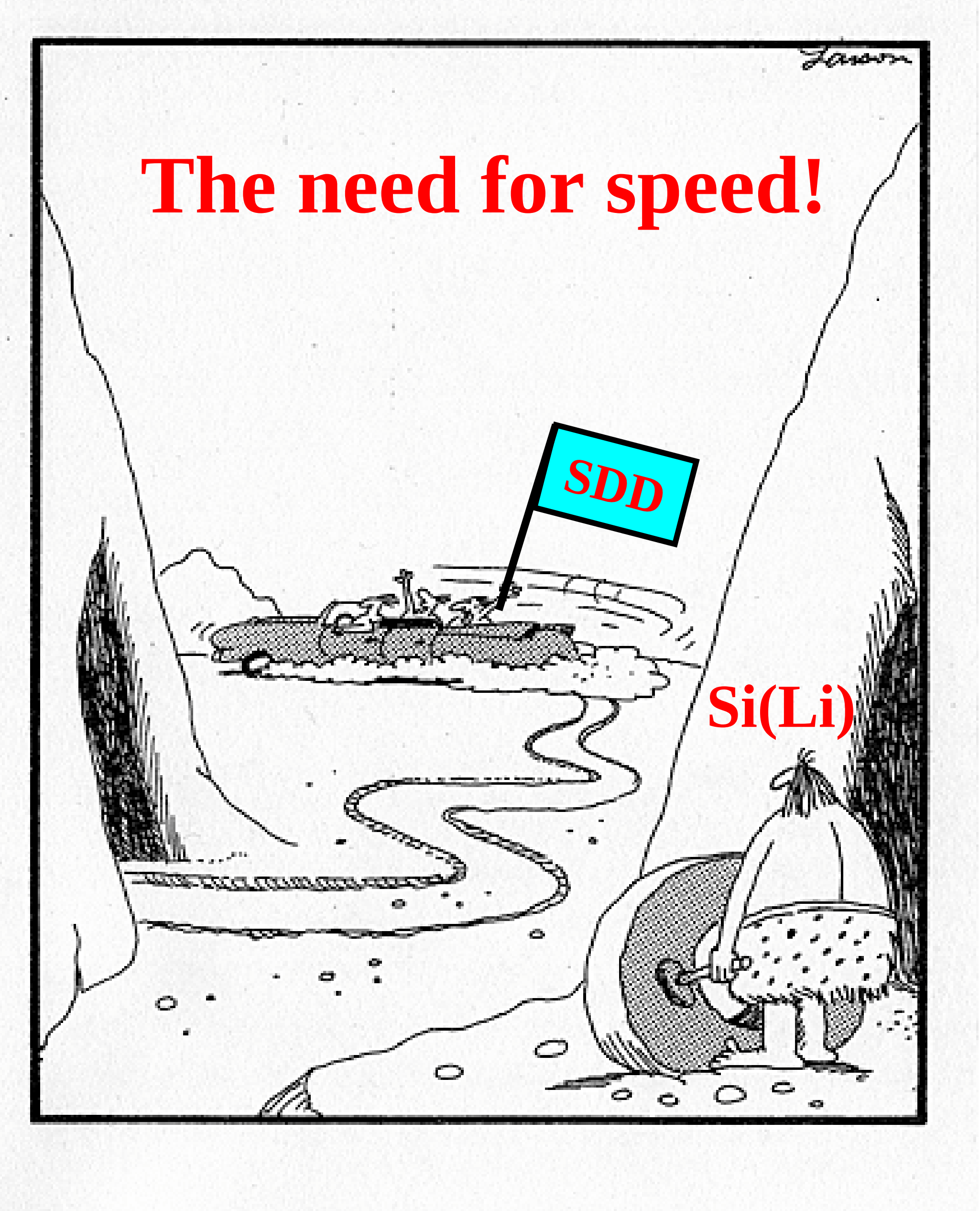


The new EDS: Silicon Drift Detector (SDD)

This paper examined the performance of the SDD for various parameters but especially for high speed X-ray Spectrum Imaging (XSI).

But as I gave the paper, I was already aware (thanks to July ‘06 communication from Joe Michael at Sandia) that my results were stale: 100 kHz OCR was already way behind the limiting performance envelope for SDD technology.

What had happened?



The Si(Li) Energy Dispersive X-ray Spectrometer

Fitzgerald, R, Keil, K. and Heinrich, K. Science v 159 (1968) 528

“Solid-State Energy-Dispersion Spectrometer for Electron-Microprobe X-ray Analysis”

EDS Mapping

Advantages:

1. Detects entire x-ray spectrum enabling mapping of all elements simultaneously

2. The unexpected can be discovered!

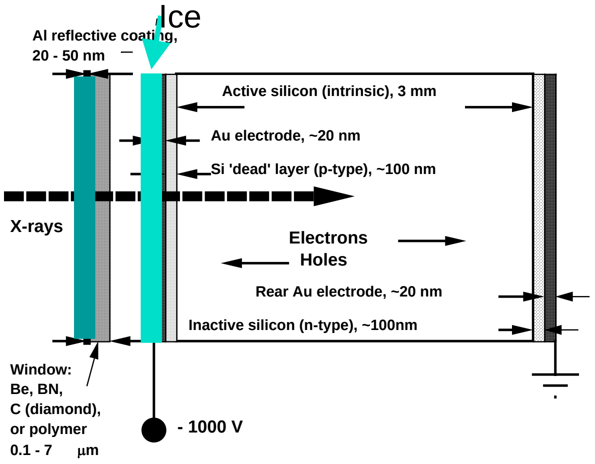
3. No defocusing

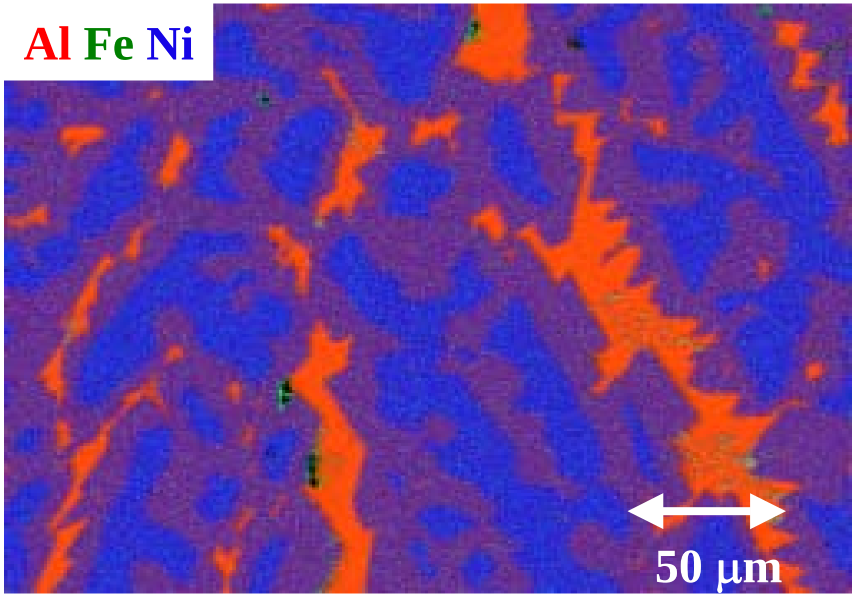
Disadvantages

1. Poor resolution: interferences

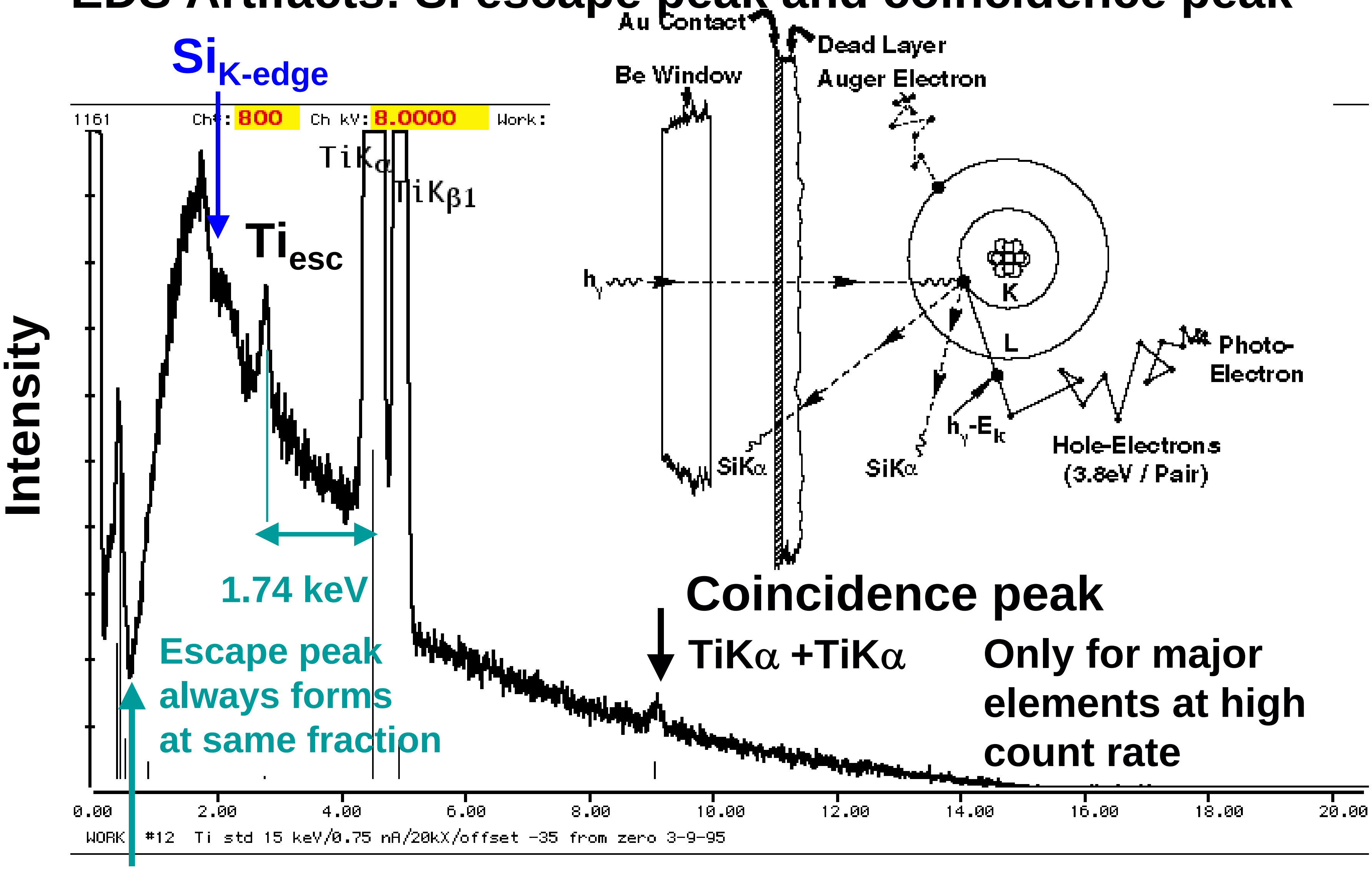
2. Lower P/B; more susceptible to continuum effects; poorer limits of detection

3. OCR: 2 kHz to 20 kHz





EDS Artifacts: Si escape peak and coincidence peak



$Si_{K\text{-edge}}$

Ti_{esc}

1.74 keV

Escape peak always forms at same fraction

Intensity

Coincidence peak

TiKα +TiKα

Only for major elements at high count rate

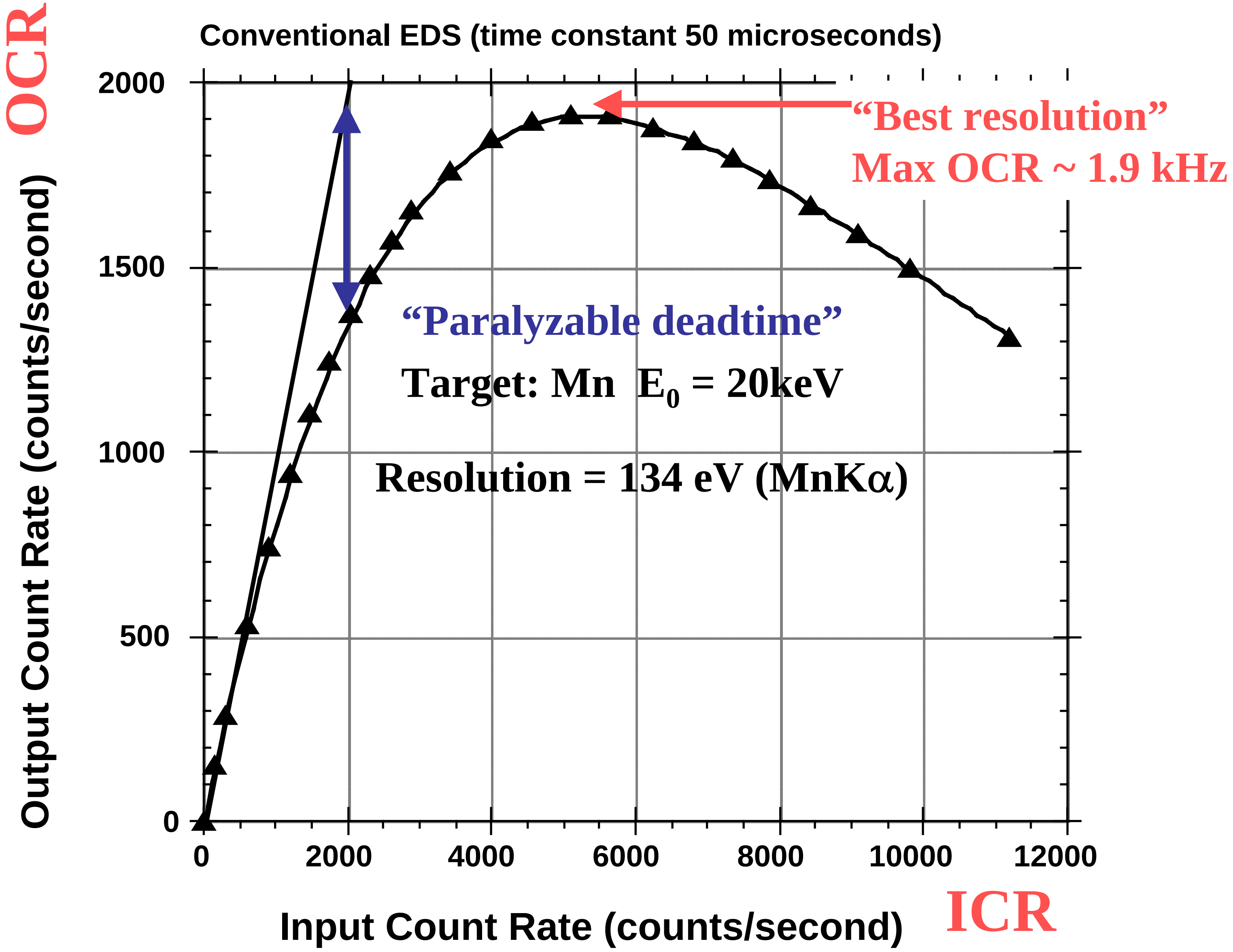
Drastic decrease in detector efficiency

Photon Energy (keV)

Spectrum appears to be continuously measured at all energies, but it is actually one photon at a time: deadtime

X-ray detection

Current State-of-the Art Si(Li)-EDS (30 mm^2)



Conventional EDS (time constant 50 microseconds)

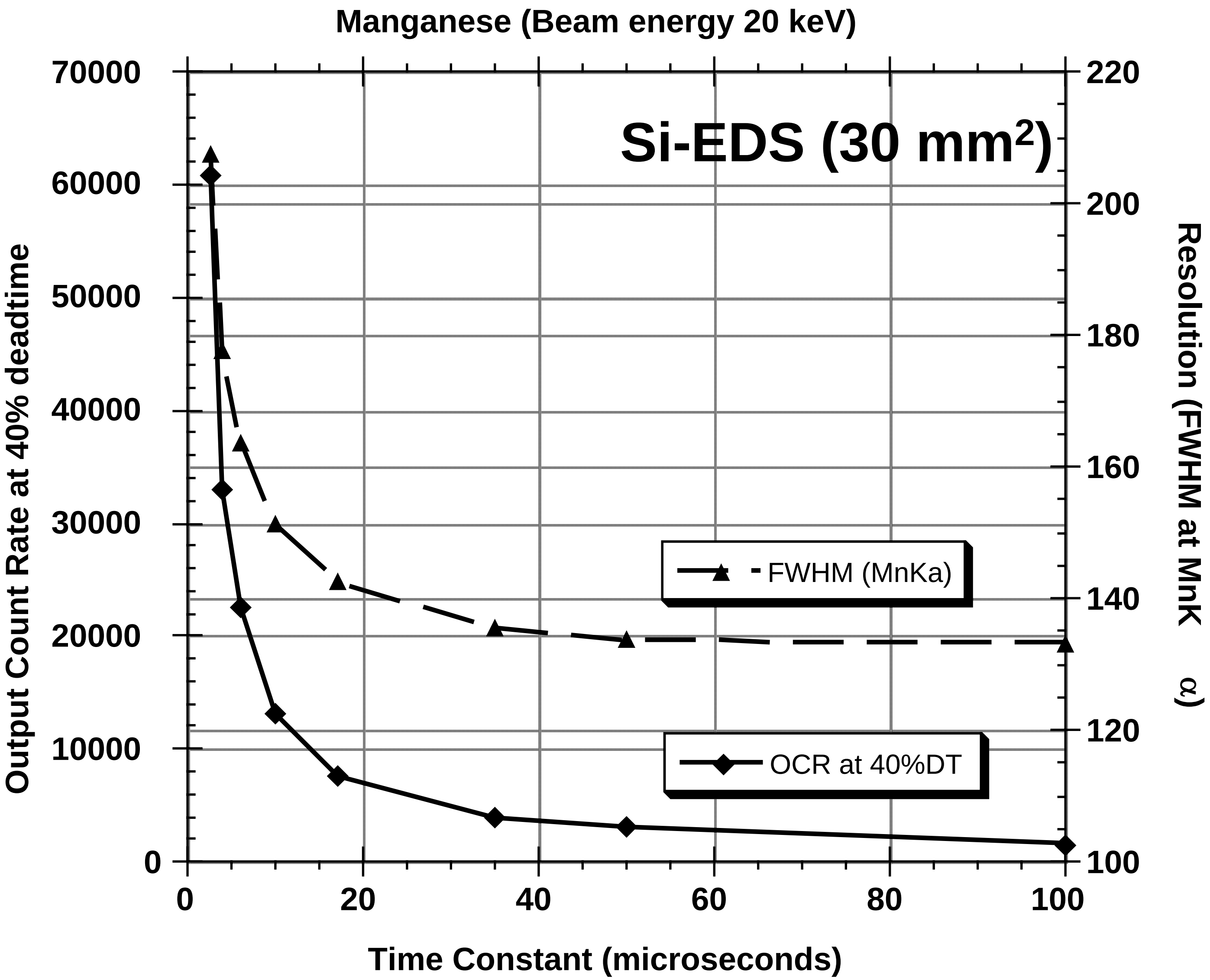
“Best resolution”
Max OCR ~ 1.9 kHz

“Paralyzable deadtime”

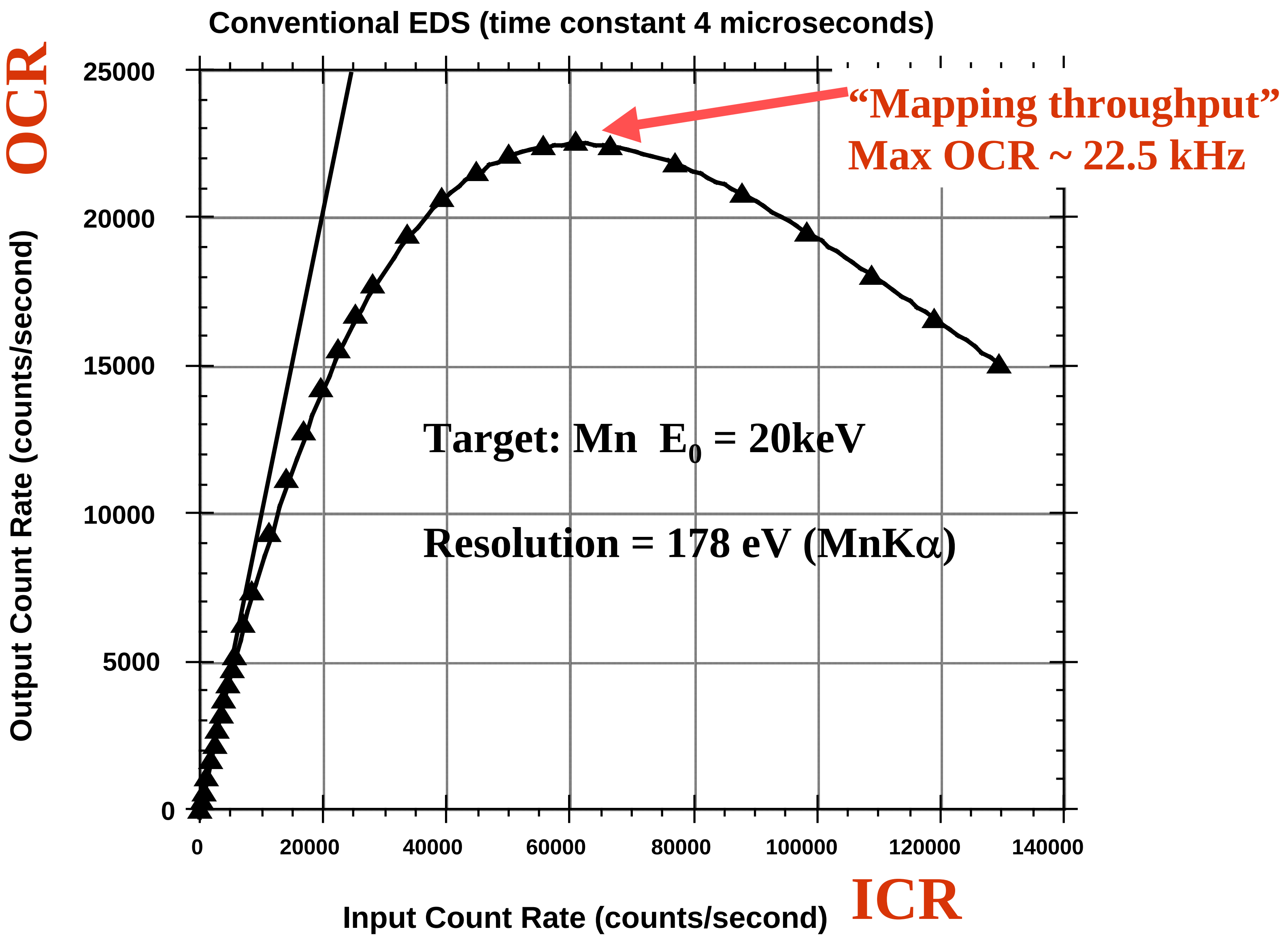
Target: Mn E_0 = 20keV

Resolution = 134 eV (MnKα)

Current digital signal processing with conventional Si-EDS enables count rates > 30 kHz (40% DT) with FWHM of ~180 eV (MnKα)



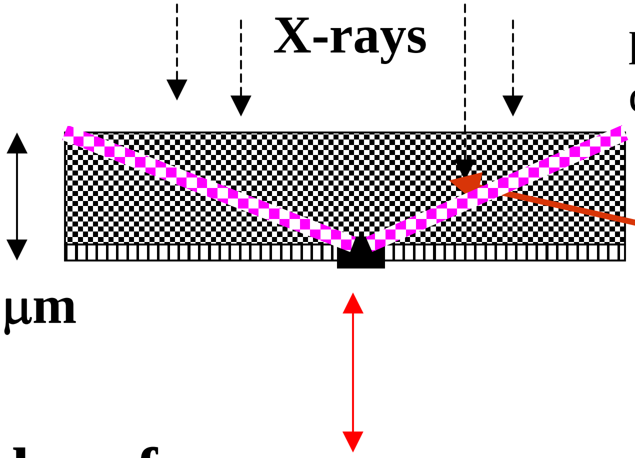
Current State-of-the Art Si(Li)-EDS (30 mm^2)



Target: Mn E_0 = 20keV

Resolution = 178 eV (MnKα)

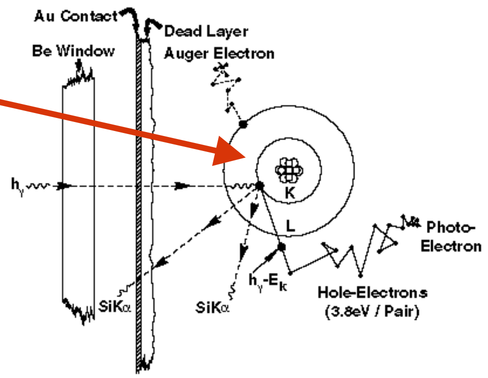
Silicon Drift Detector: Same detection physics as Si(Li)-EDS, but a radically different design



SDDs are thin!

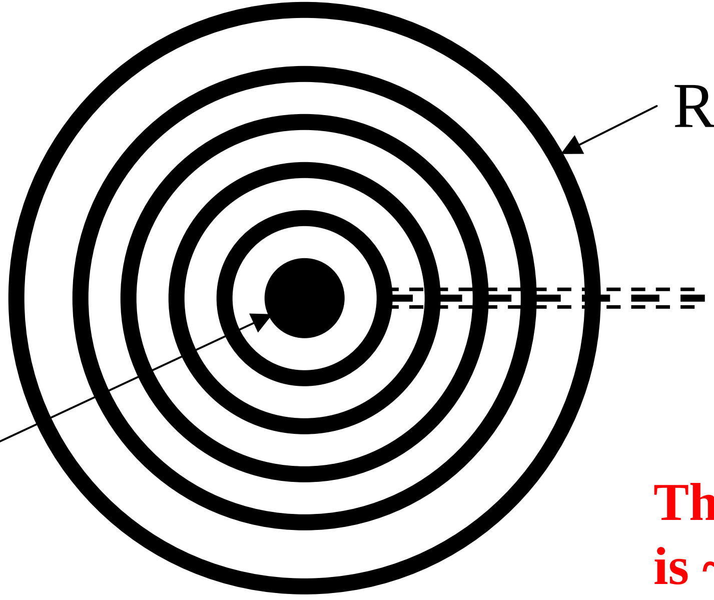
300 µm

X-ray absorption and ejection of photoelectron; inelastic scattering creates electron-hole pairs.



SDD Backsurface

SDDs have a complex back surface electrode structure. Applied potential creates internal collection channel



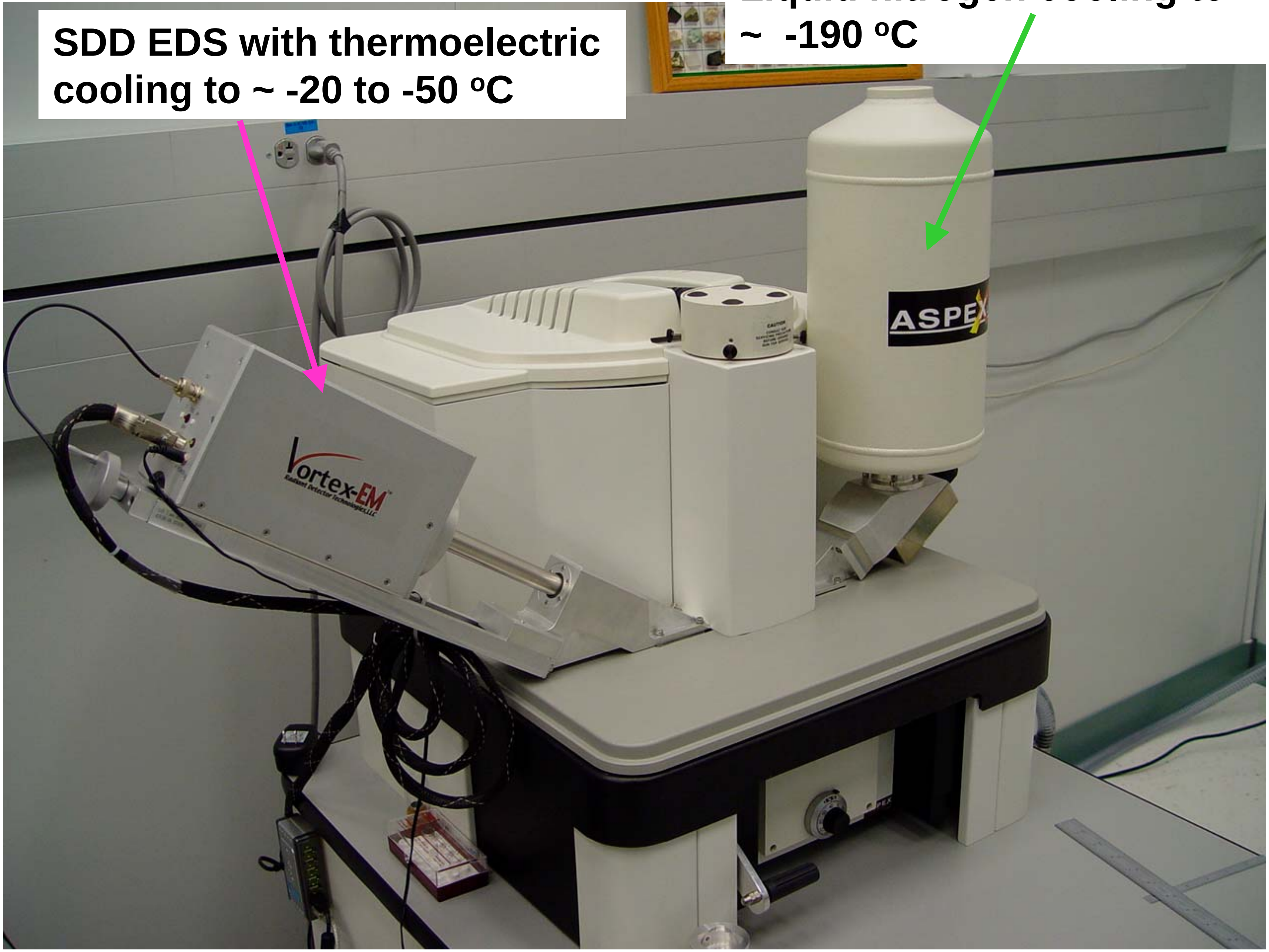
Ring electrodes

Resistor bridge

Central anode,
80 µm diameter

Active area 5 mm² to 100 mm²

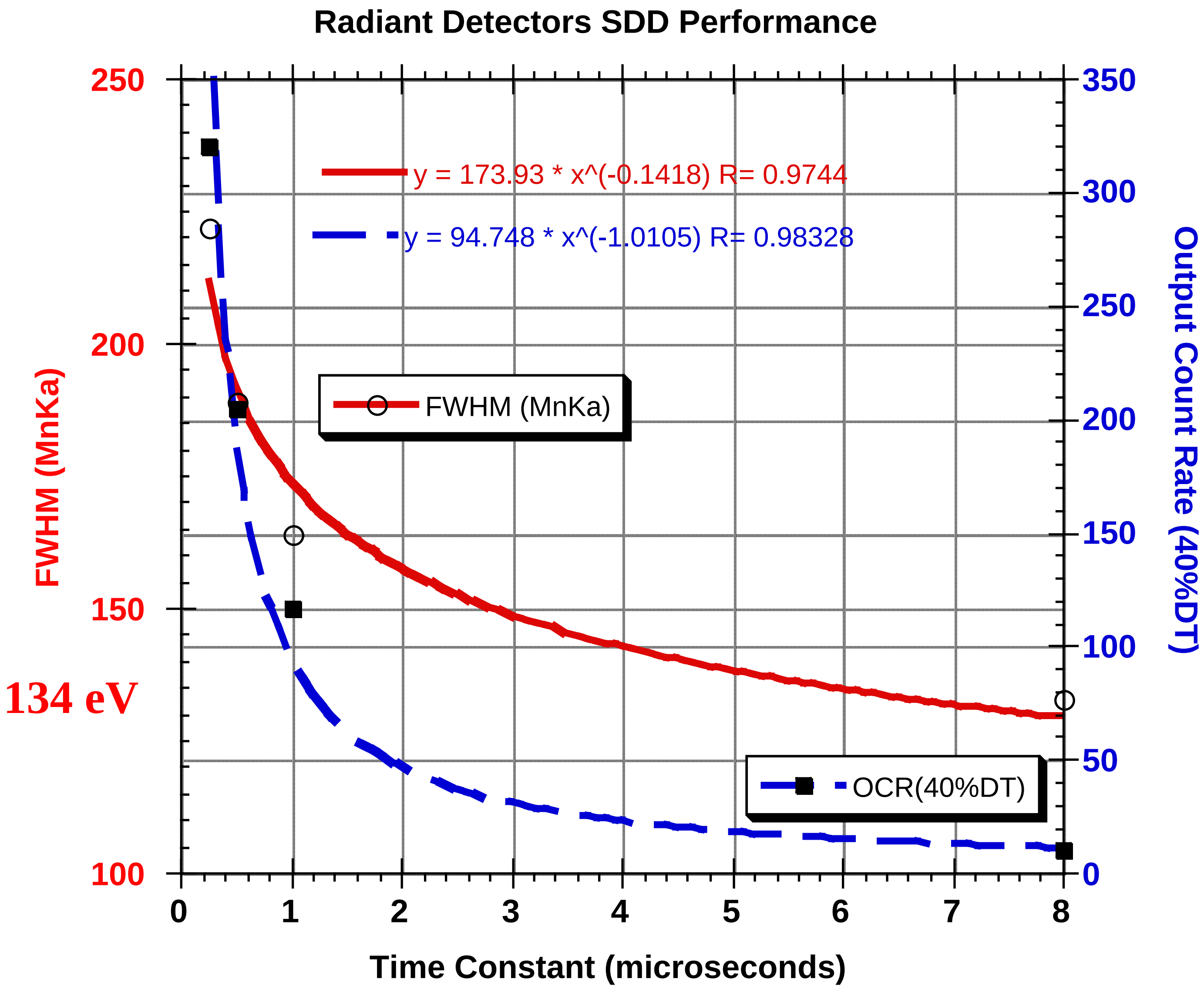
The anode of an SDD is ~ 0.005 mm² for a 50 mm² detector, about 1/10,000 the area of EDS

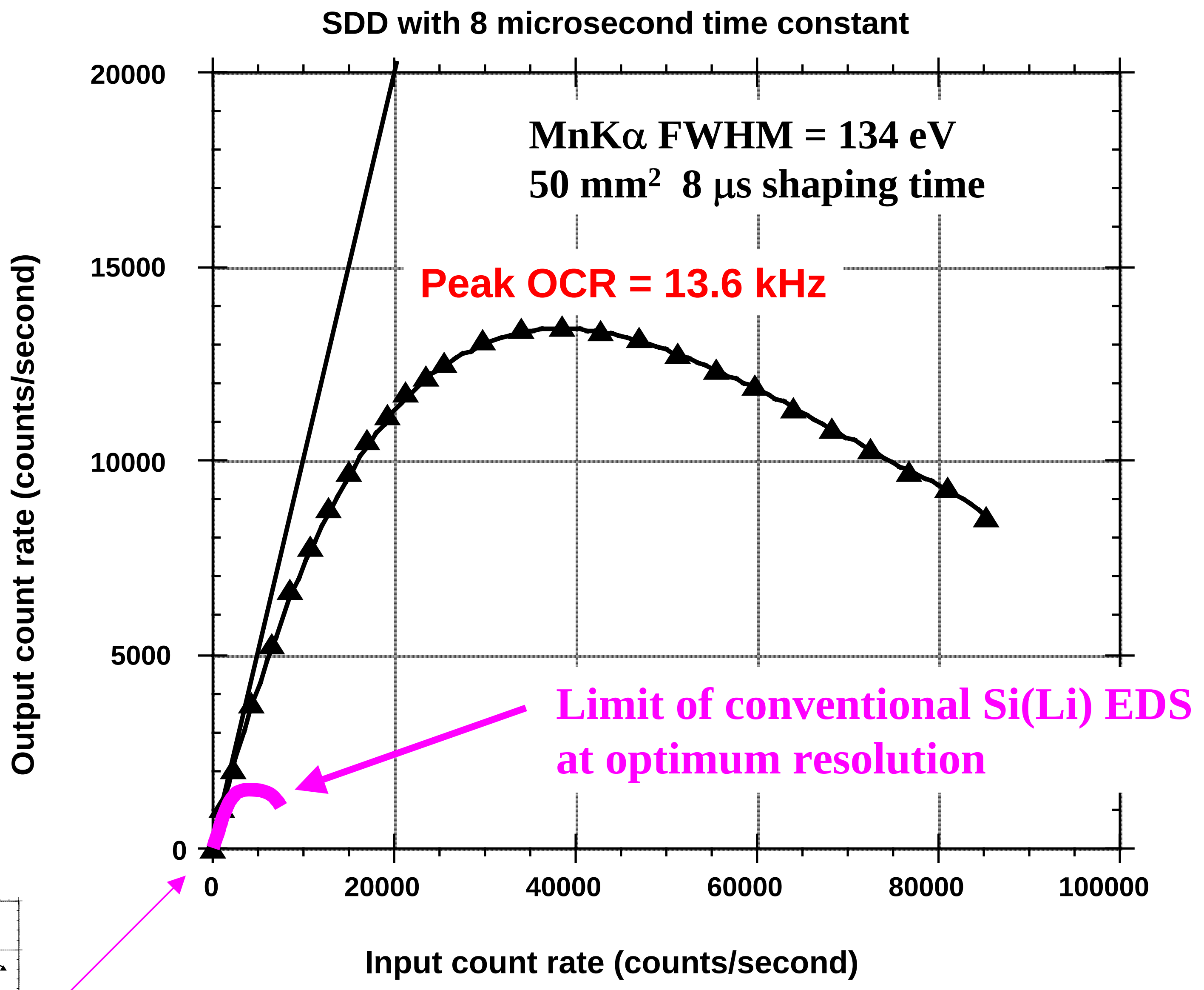


SDD EDS with thermoelectric cooling to ~ -20 to -50 °C

Conventional Si(Li) EDS with Liquid nitrogen cooling to ~ -190 °C

3rd generation Radiant “Vortex” 50 mm^2 SDD

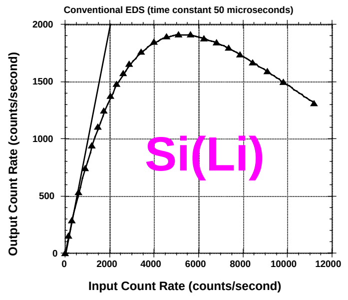




MnKα FWHM = 134 eV
50 mm^2 8 μs shaping time

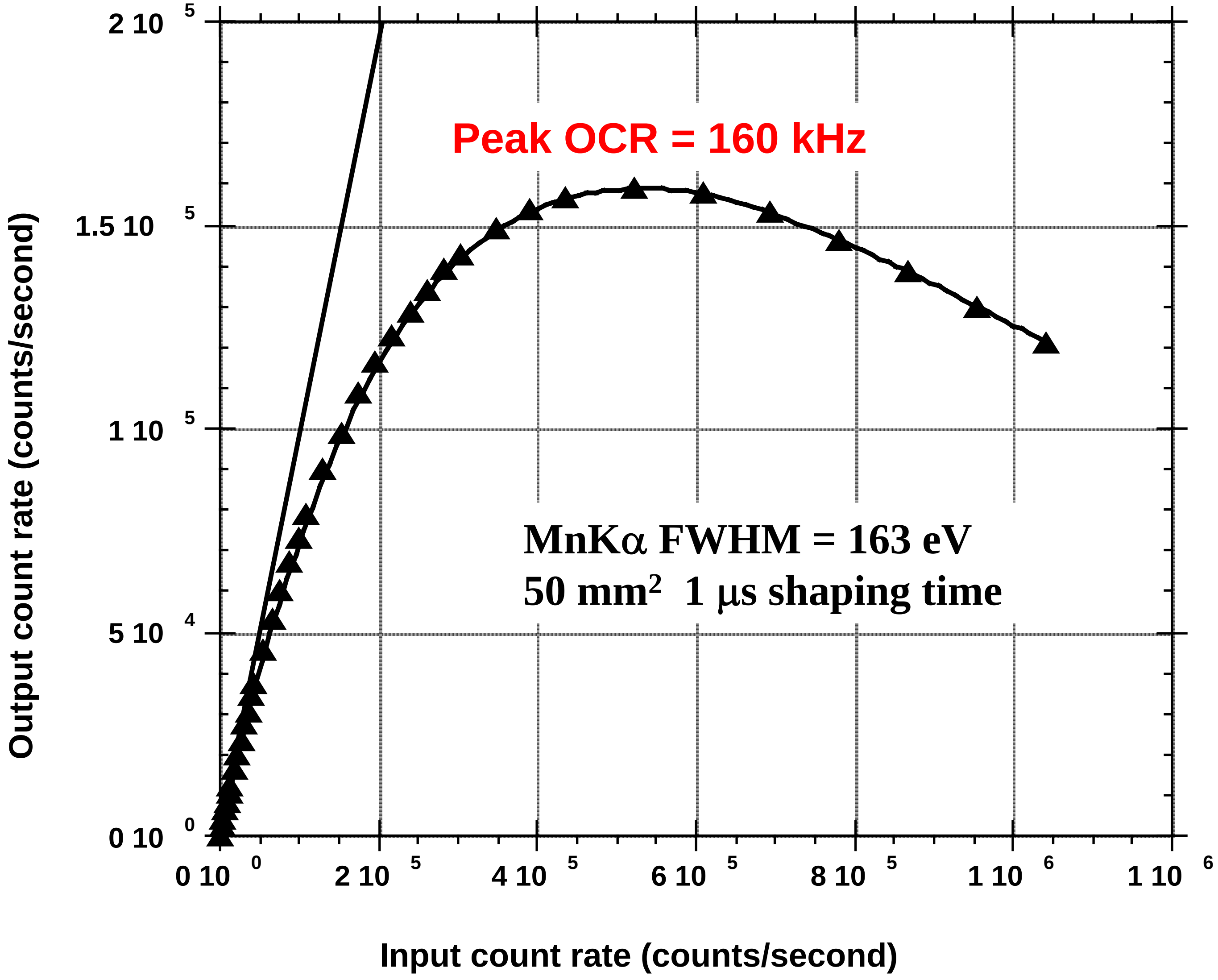
Peak OCR = 13.6 kHz

Limit of conventional Si(Li) EDS
at optimum resolution



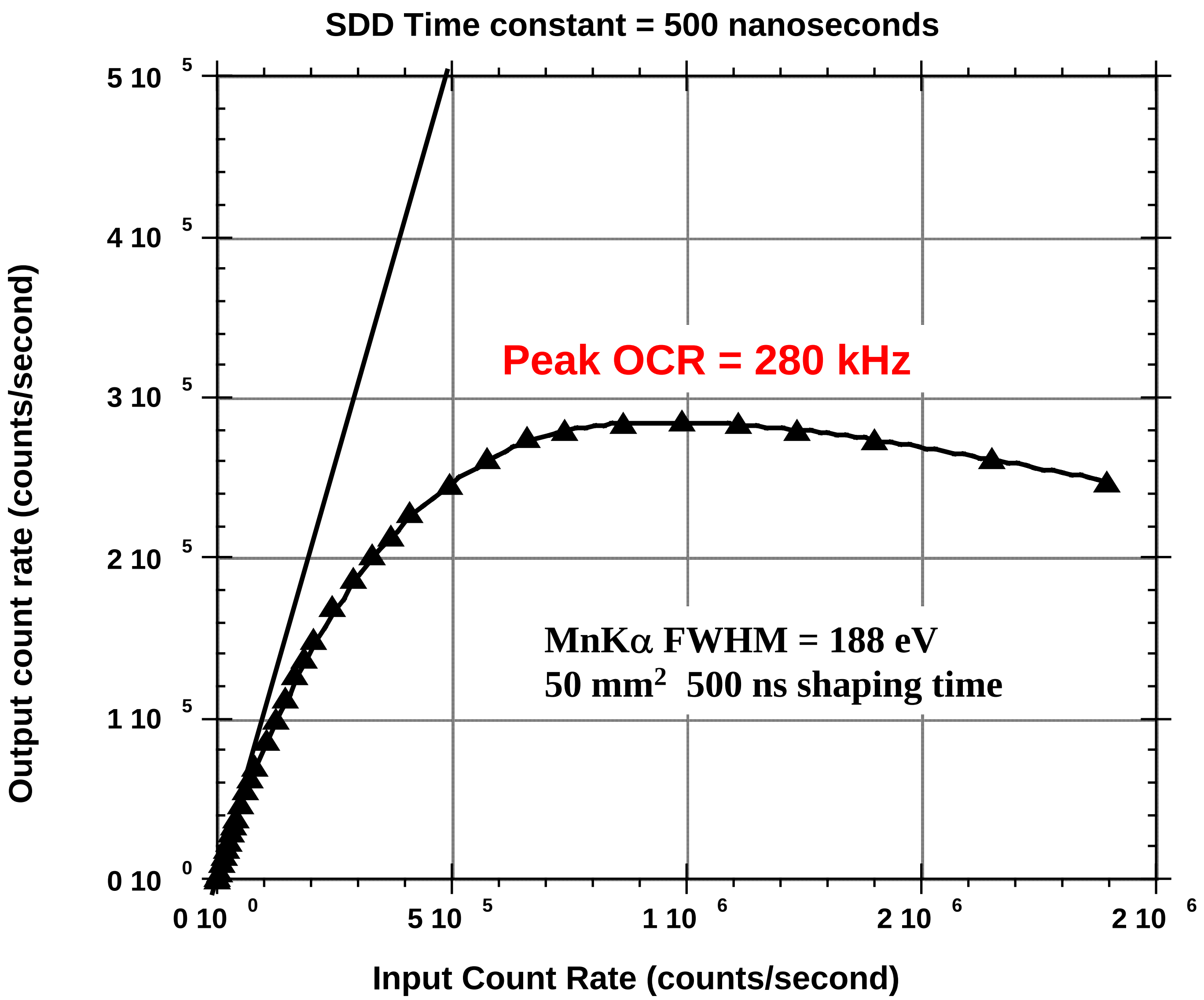
Si(Li)

SDD 1 microsecond time constant



Peak OCR = 160 kHz

MnKα FWHM = 163 eV
50 mm^2 1 μs shaping time

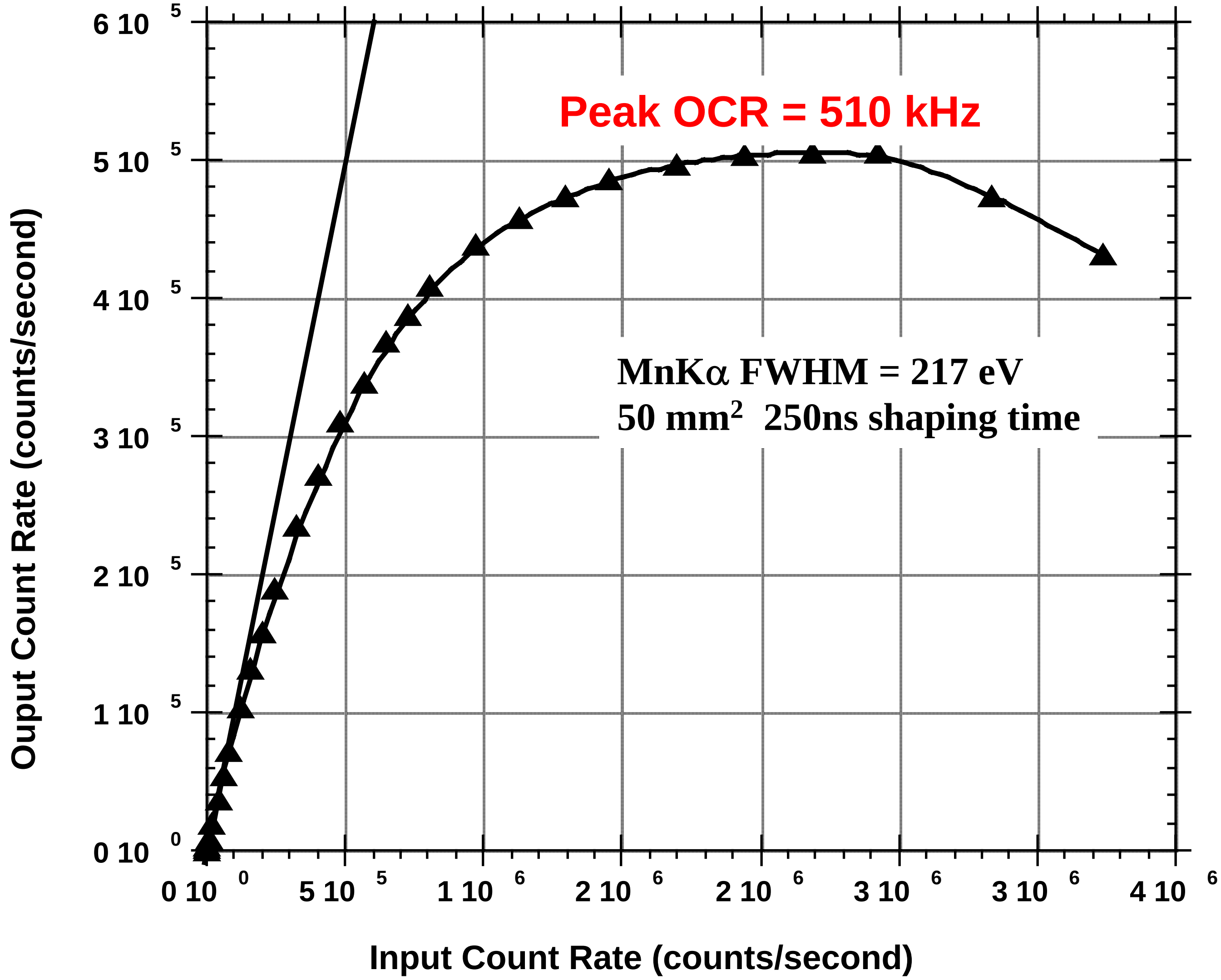


SDD Time constant = 500 nanoseconds

Peak OCR = 280 kHz

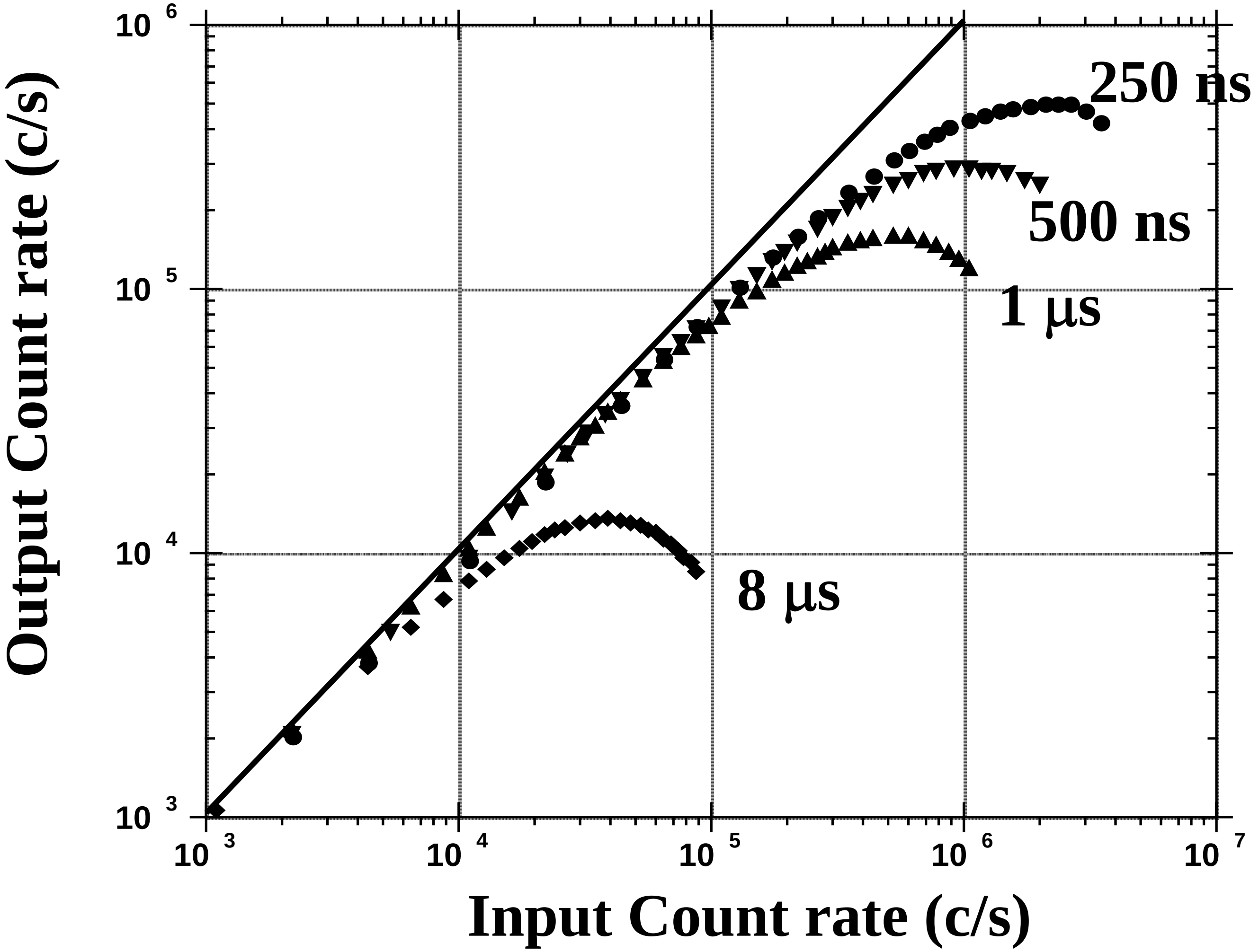
MnKα FWHM = 188 eV
50 mm^2 500 ns shaping time

SDD Time constant = 250 nanoseconds



Detector solid angle plus processing speed revolutionizes x-ray mapping!

Radiant Detectors 50 mm^2 VORTEX SDD



The Real Promise of the SDD:
High Speed Spectrum Image Mapping

“Three-minute egg
x-ray spectrum image”

Radiant SDD/SAMx spectrum imaging software

128x128x10ms (1.3 ms overhead) = 185 s total

50 mm^2 detector; 500 ns time constant; Res = 188 eV (MnKα)

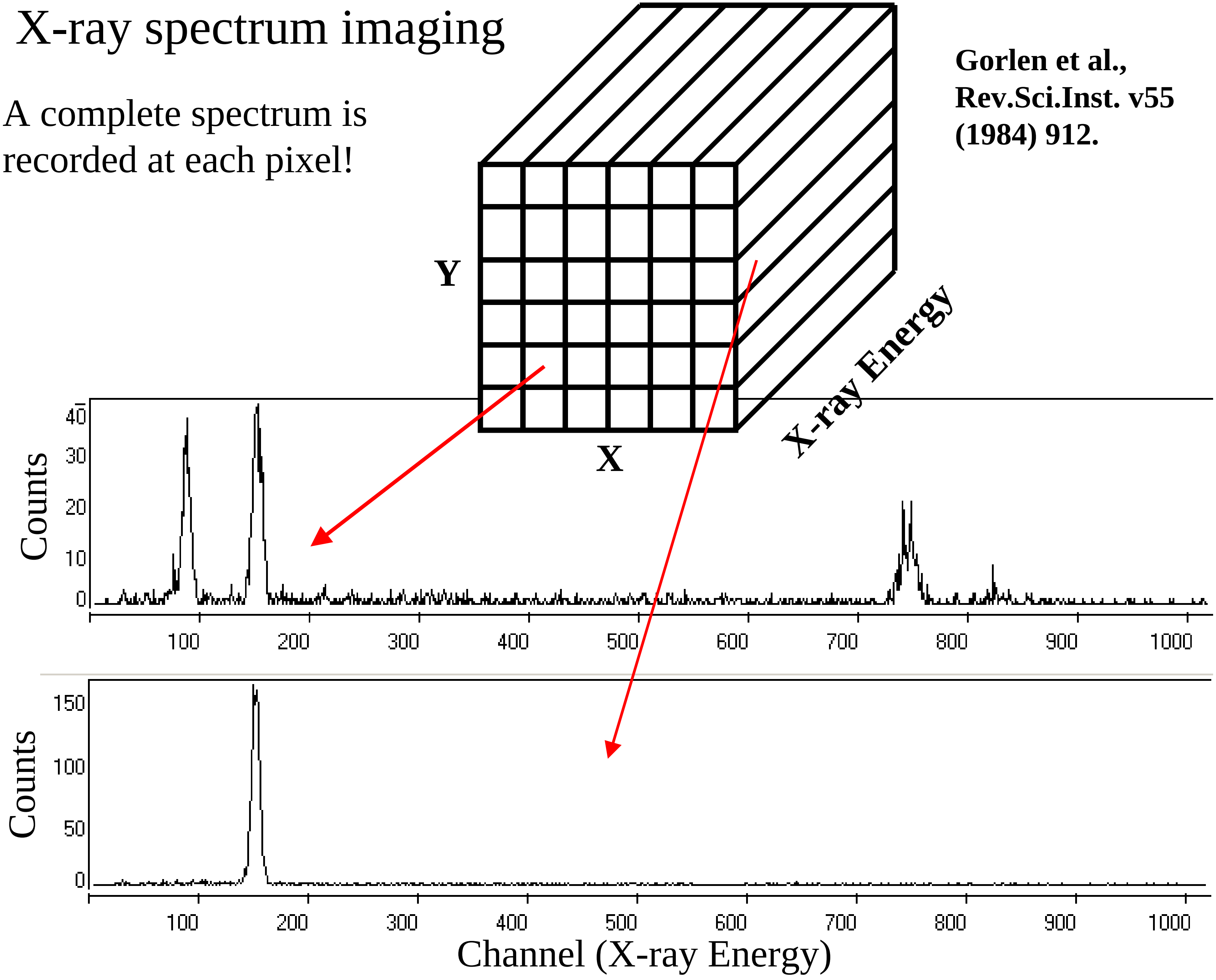
E_0 = 20 keV   i = 10nA

ICR: 320 kHz   OCR: 220 kHz   ~40%DT

X-ray spectrum imaging

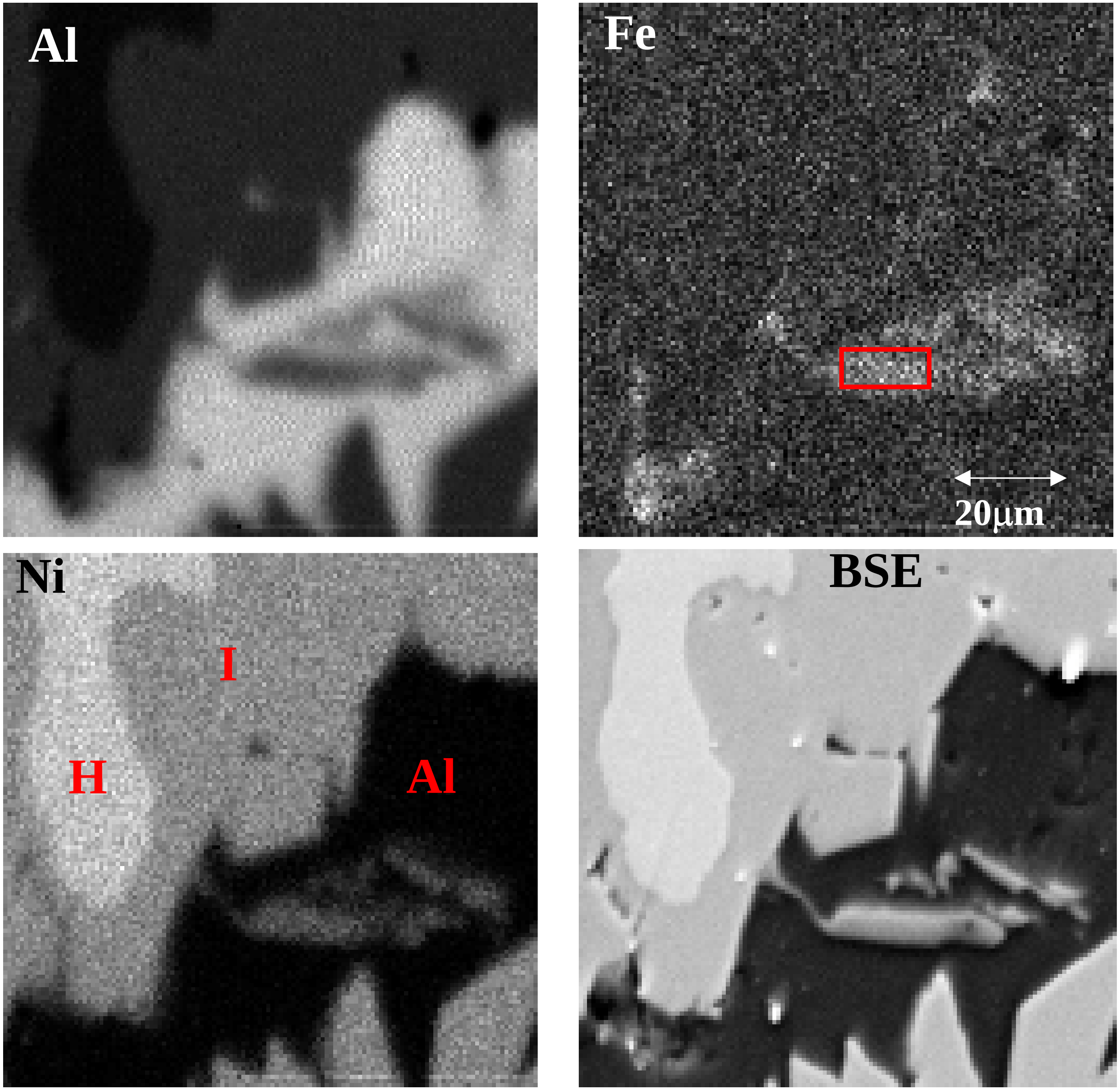
A complete spectrum is recorded at each pixel!

Gorlen et al., Rev.Sci.Inst. v55 (1984) 912.



Raney Nickel Alloy
E_0 = 20 keV 10 nA
TC = 500 ns (188 eV at MnKα)
128x128
10 ms per pixel
Mapping 185 sec

Phases
Al 99.5 Ni 0.5
Al 71.2 Ni 24.6 Fe 4.2
Al 60 Ni 40 "I"
Al 46.5 Ni 53.5 "H"



Update on Silicon Drift Detector (SDD) Performance

- Processing speed

Joe Michael’s news: he had obtained a quad detector with this performance!

Bruker SDD Performance

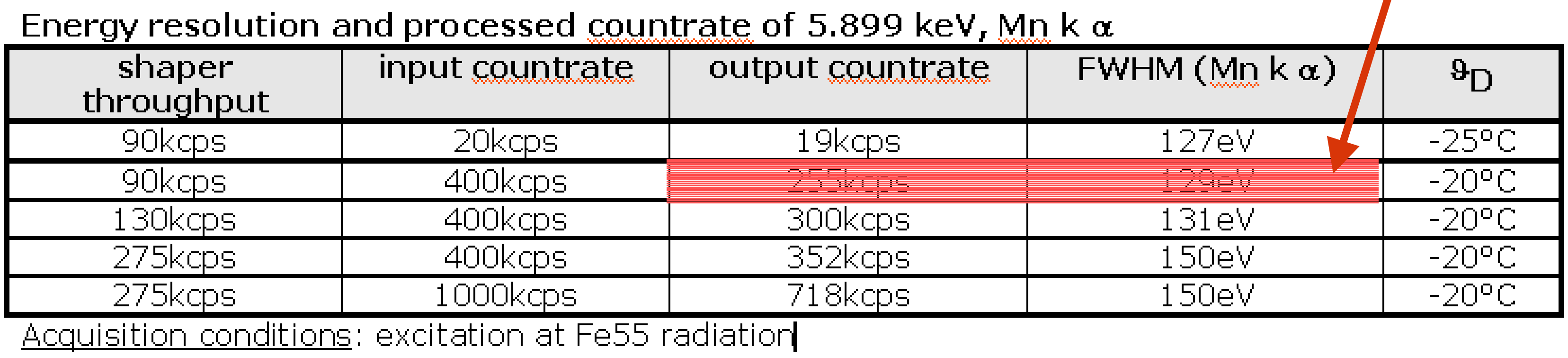
Detector rise time	<100ns
Optimum shaping time	700 ns (90 kcps max. throughput)
Maximum cooling	-25°C (at Iopt=2,5A, ta = 23°C, still air)
Thermostat set point	-20°C

Values for sum spectrum over all 4 detectors (measured at MnKa)

Peak shift (20 ... 800 kcps)	<5eV
Peak drift (24h)	<10eV
Peak/Background	2200:1

Energy resolution and processed countrate of 5.899 keV, Mn k

100x faster than Si(Li)!



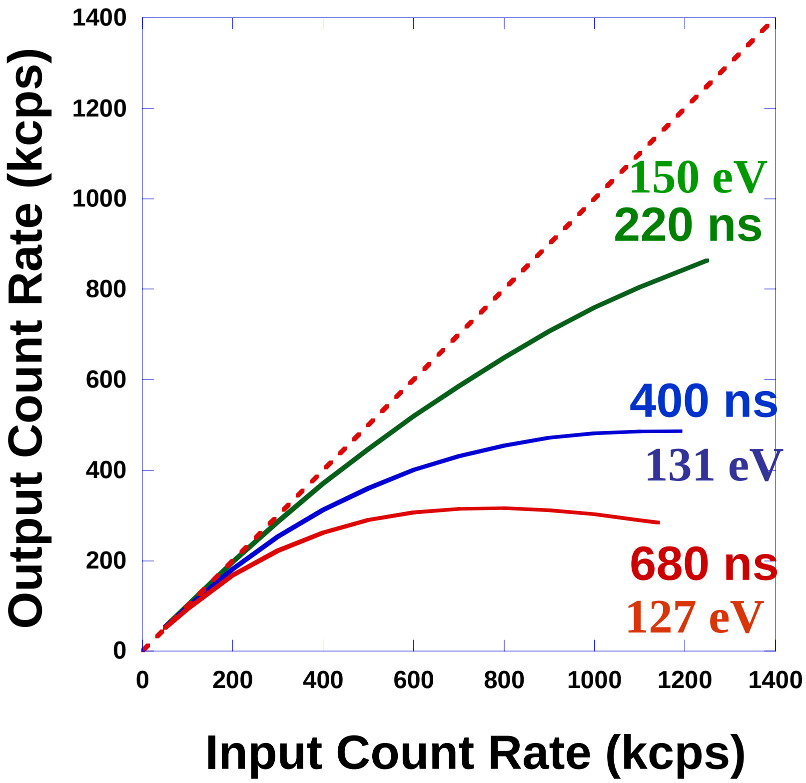
Energy resolution and processed countrate of 5.899 keV, Mn k α

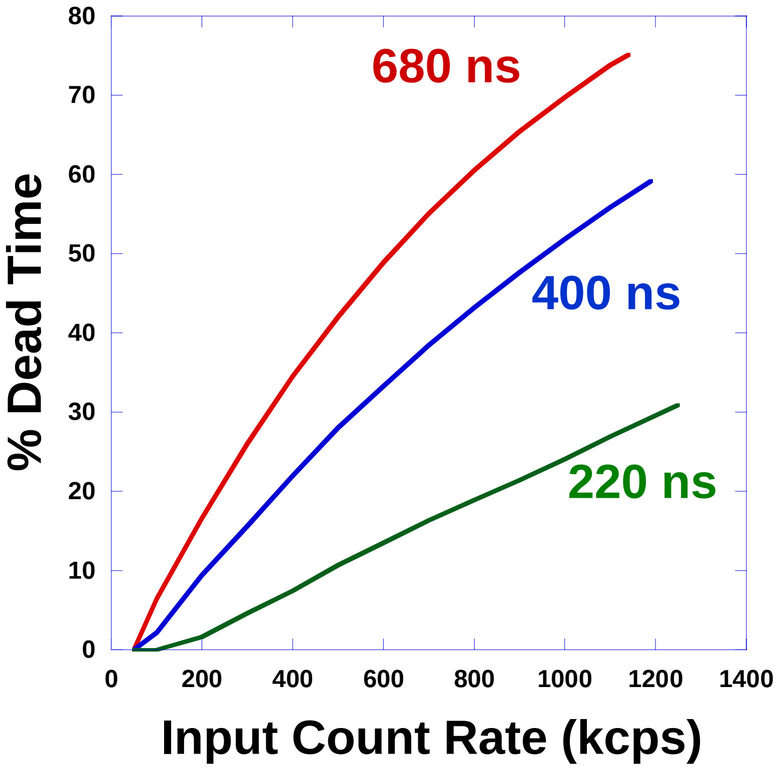
shaper throughput	input countrate	output countrate	FWHM (Mn k α)	ϑ_D
90kcps	20kcps	19kcps	127eV	-25°C
90kcps	400kcps	255kcps	129eV	-20°C
130kcps	400kcps	300kcps	131eV	-20°C
275kcps	400kcps	352kcps	150eV	-20°C
275kcps	1000kcps	718kcps	150eV	-20°C

Acquisition conditions: excitation at Fe55 radiation

Source: Bruker

The latest SDD (Bruker 4th generation)





Bruker: single SDD performance

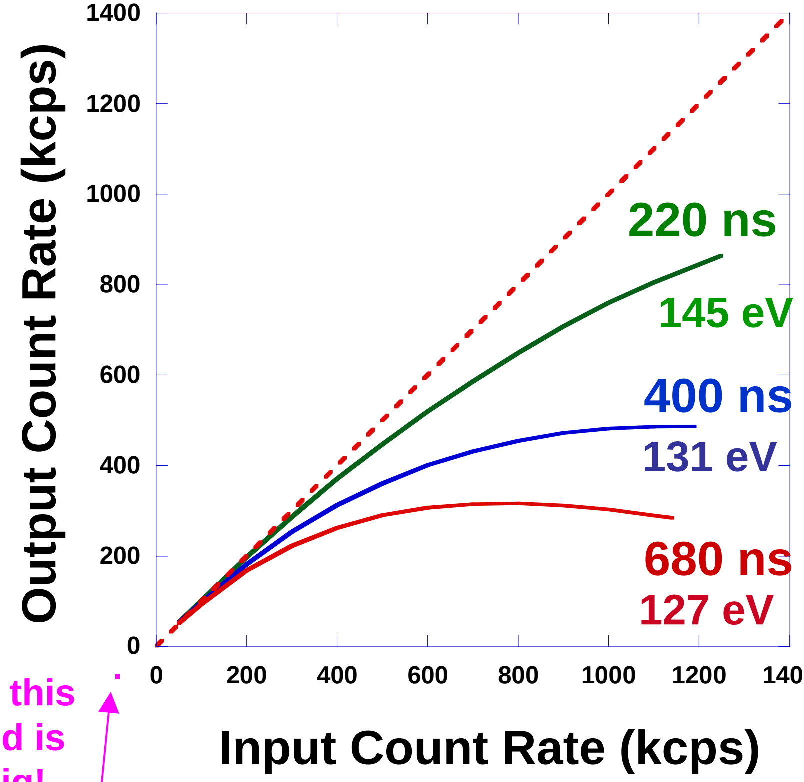
Four 10 mm^2 SDDs with multiplexing gives OCR > 1 MHz with resolution < 140 eV.

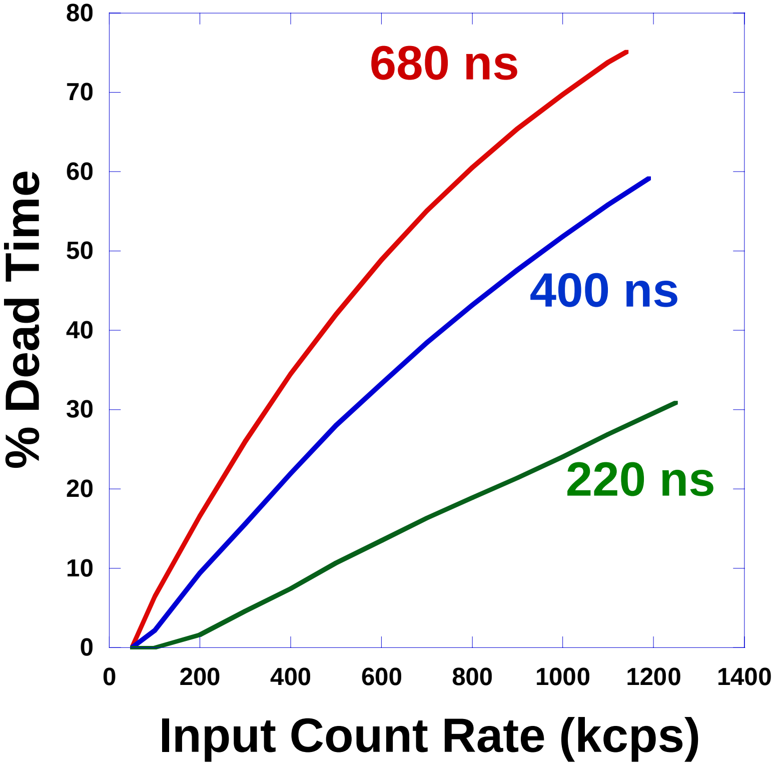
Bruker Quad SDD
Data: Joe Michael,
Sandia National Lab
Albuquerque, NM

But wait! The latest SDD (Bruker 4th generation)

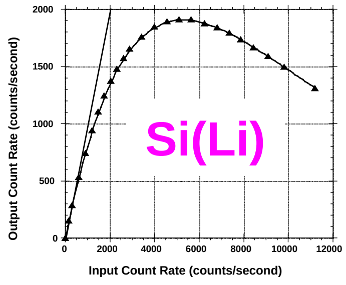
Data: Joe Michael,
Sandia National Lab
Albuquerque, NM

Bruker 10 mm^2 single SDD (from P-N Sensors)





Even this
period is
too big!



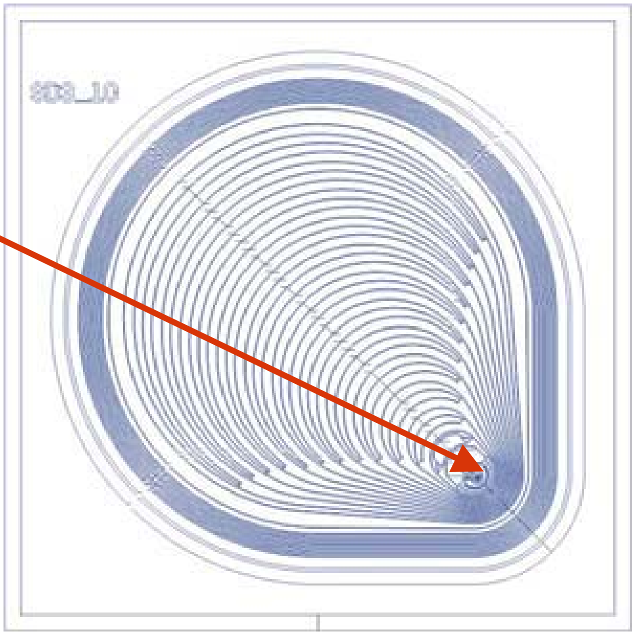
Si(Li) EDS "best resolution"
OCR vs ICR performance
disappears on this plot!!
(covering only 1% of the
0 – 200 unit spacing)

Four 10 mm^2 SDDs with multiplexing gives OCR > 1 MHz with resolution < 140 eV.

Why is this SDD so much faster?

Bruker “Tear–drop” Shaped SDD

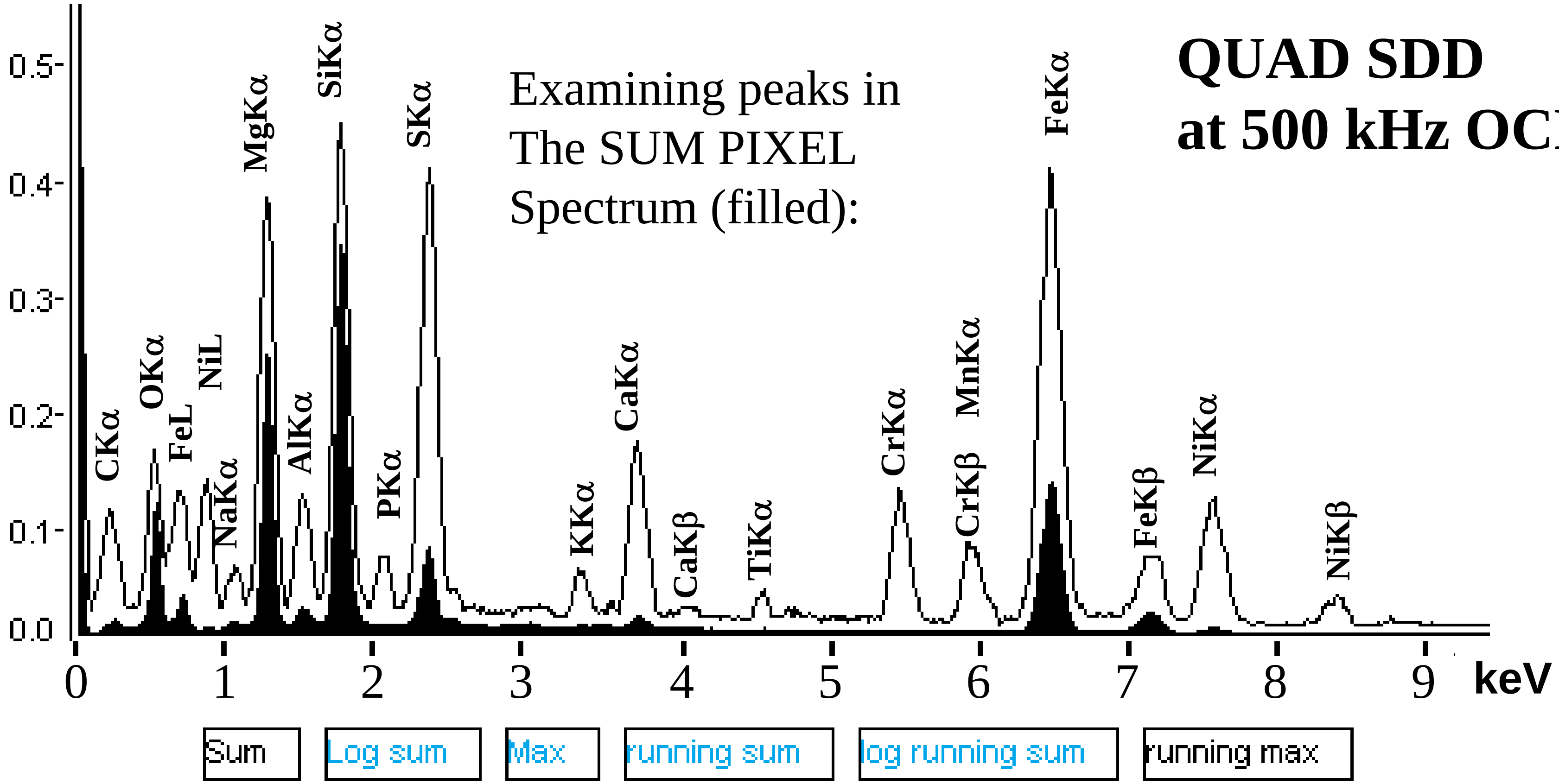
OCR speed: On-chip integrated charge amplifier eliminates microphonics; off-center location protects amplifier from x-rays



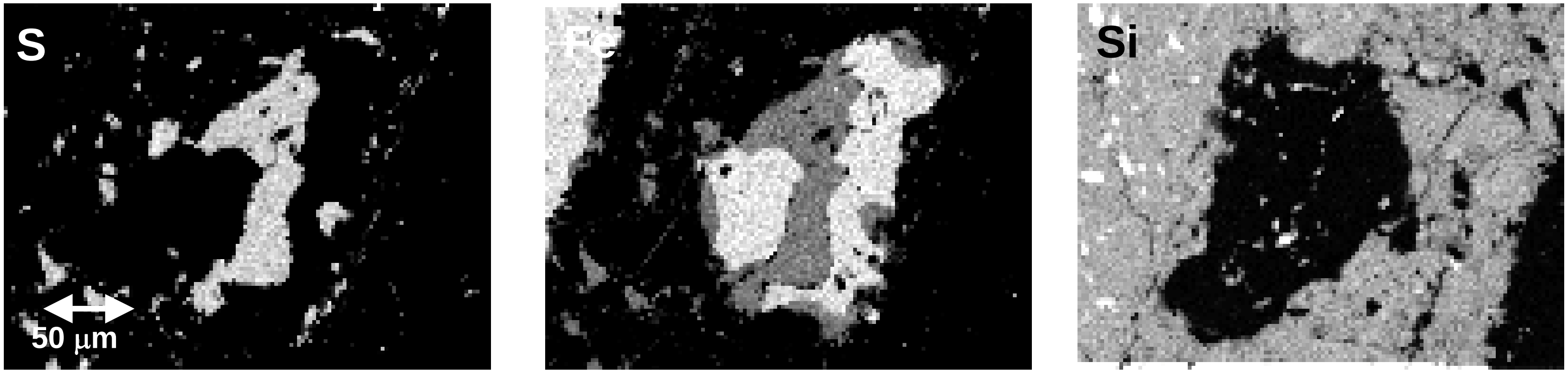
M&M2005_Optimized Readout Methods of Silicon Drift Detectors for High Resolution Spectroscopy in Micro-Beam Analysis

H.Soltau*, P.Lechner*, A. Niculae*, G. Lutz**, L. Strüder**, C. Fiorini*** and A. Longoni ***
R. Eckhard,* G. Schaller,** and F. Schopper**

QUAD SDD
at 500 kHz OCR



Examining peaks in
The SUM PIXEL
Spectrum (filled):



This is a 12 second x-ray spectrum image (128x96 pixels)!

Update on Silicon Drift Detector (SDD) Performance

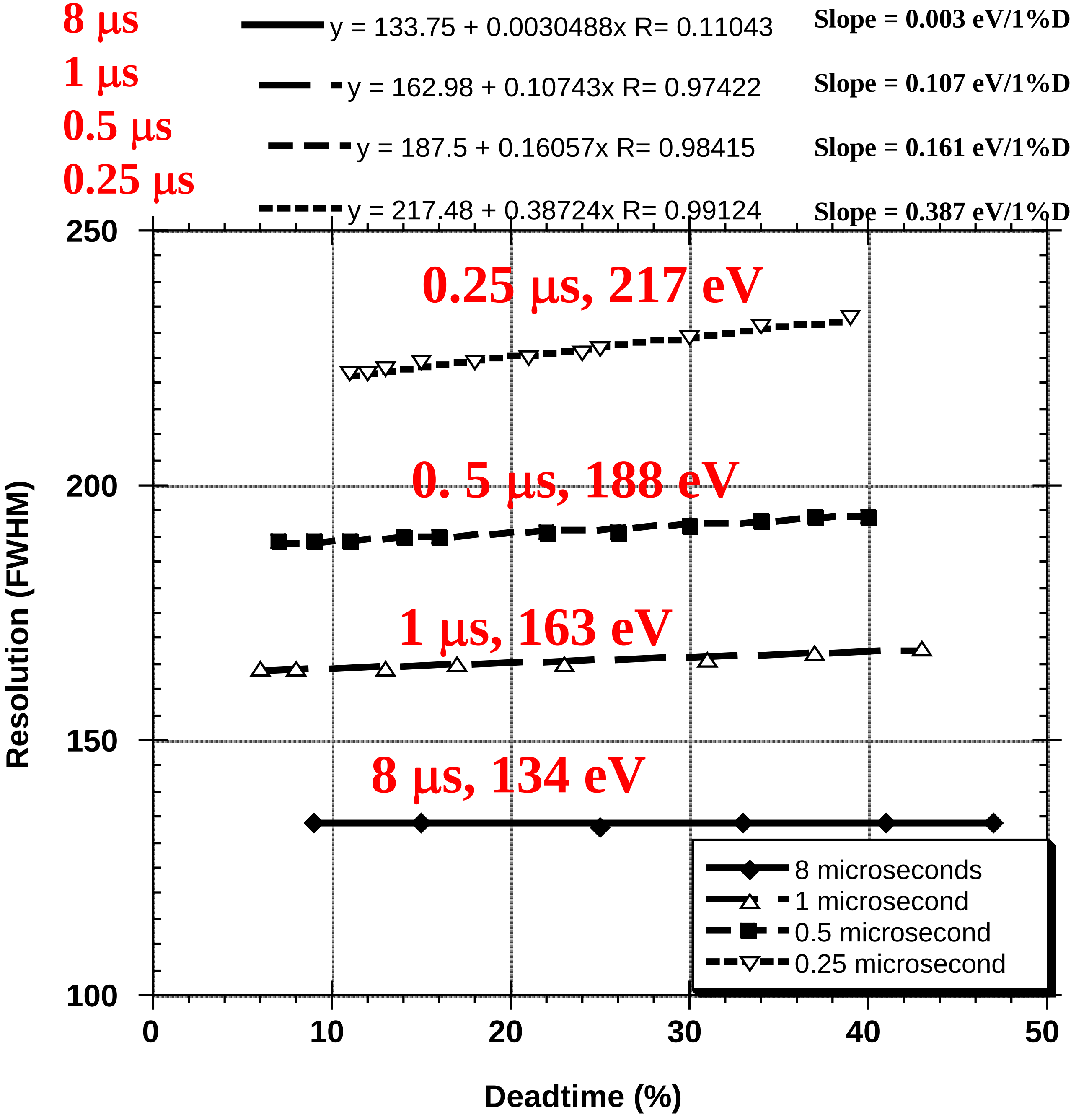
- Processing speed
- Spectral quality
  - Resolution and peak position stability

3rd generation Radiant 50 mm^2 SDD

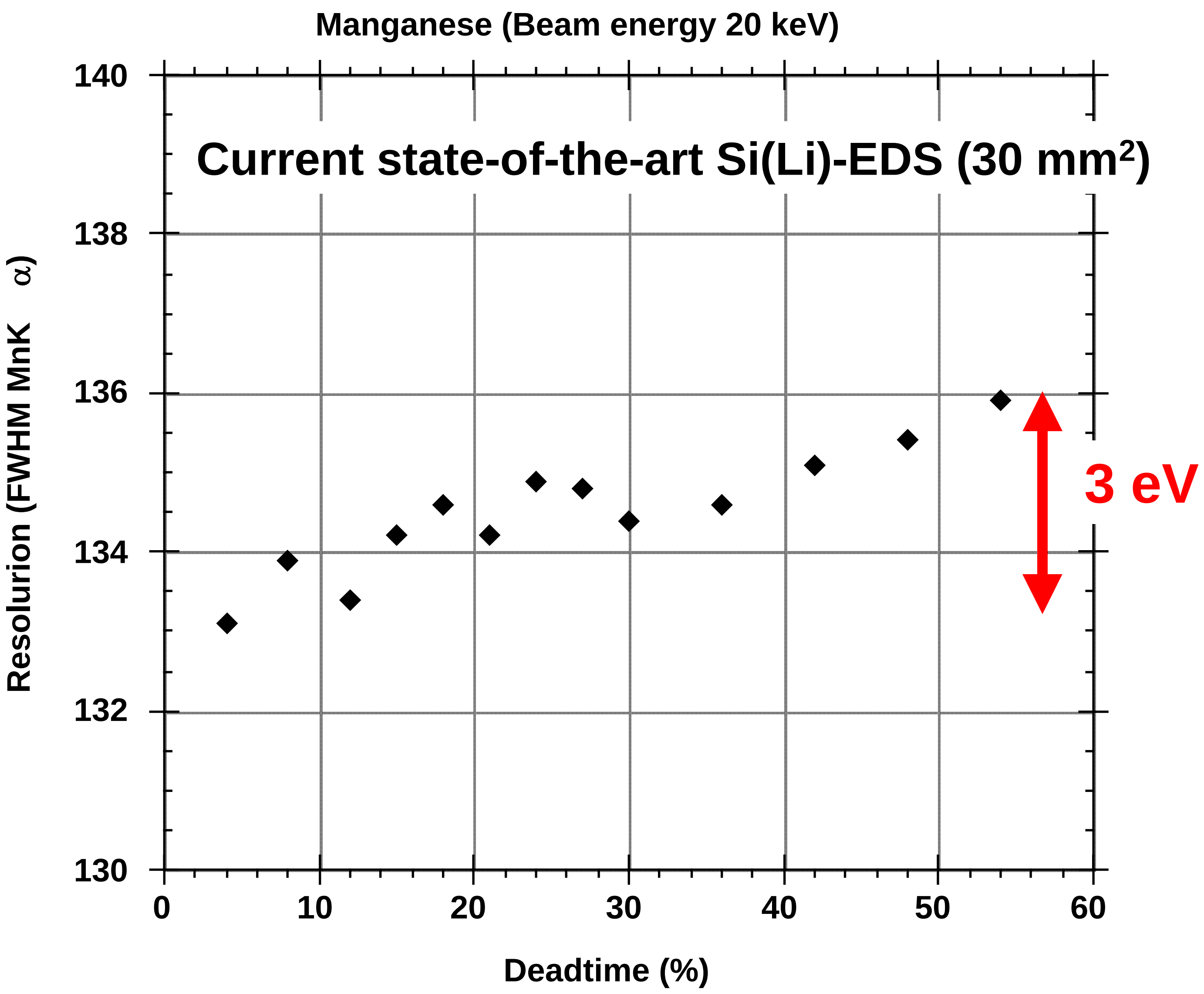
Note constancy of peak shape with input count rate! This is vital for spectral studies, including spectrum imaging.

8 µs
1 µs
0.5 µs
0.25 µs

Slope = 0.003 eV/1%DT
Slope = 0.107 eV/1%DT
Slope = 0.161 eV/1%DT
Slope = 0.387 eV/1%DT

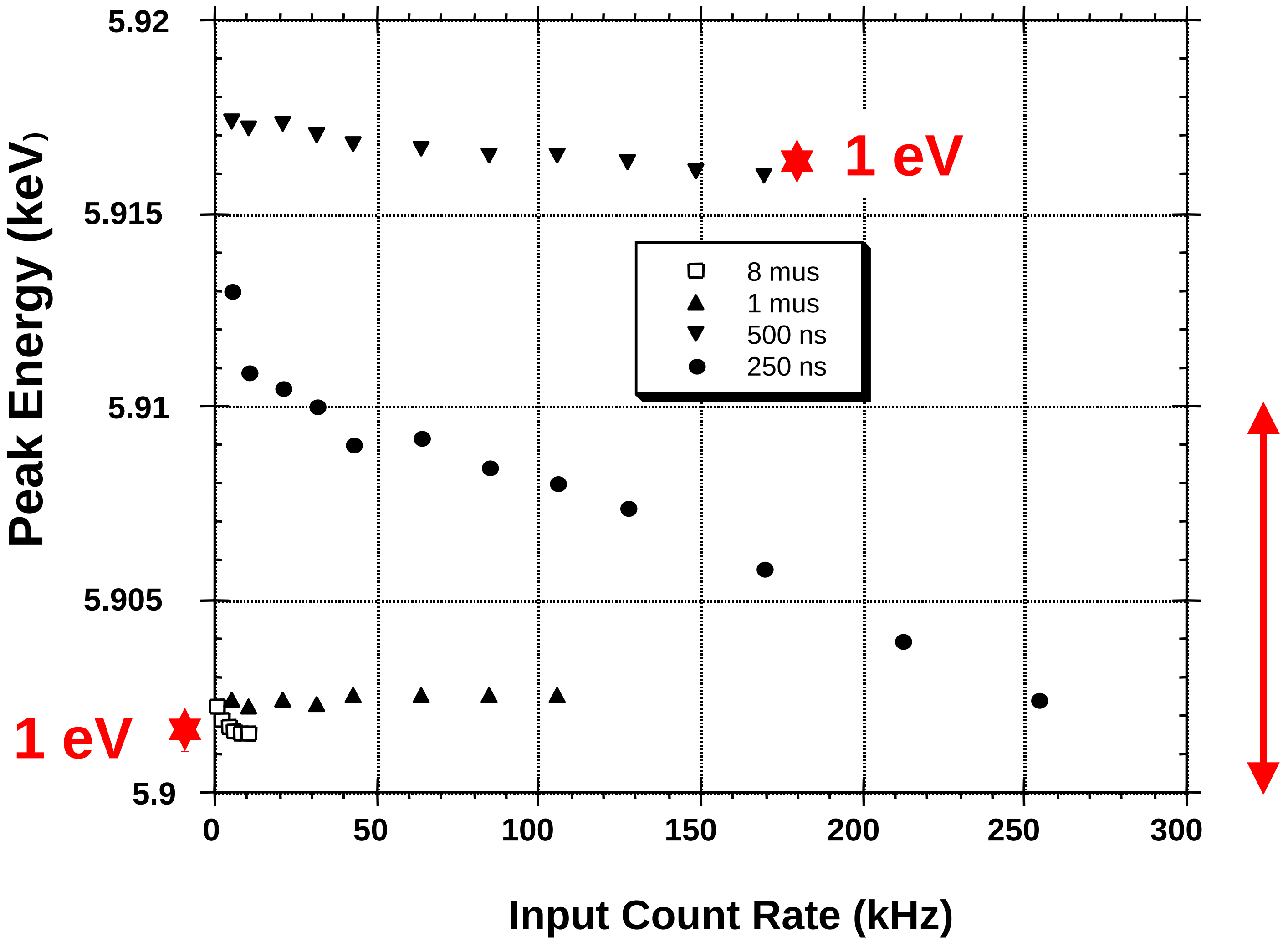


For a given choice of time constant, the resolution should remain constant with deadtime.



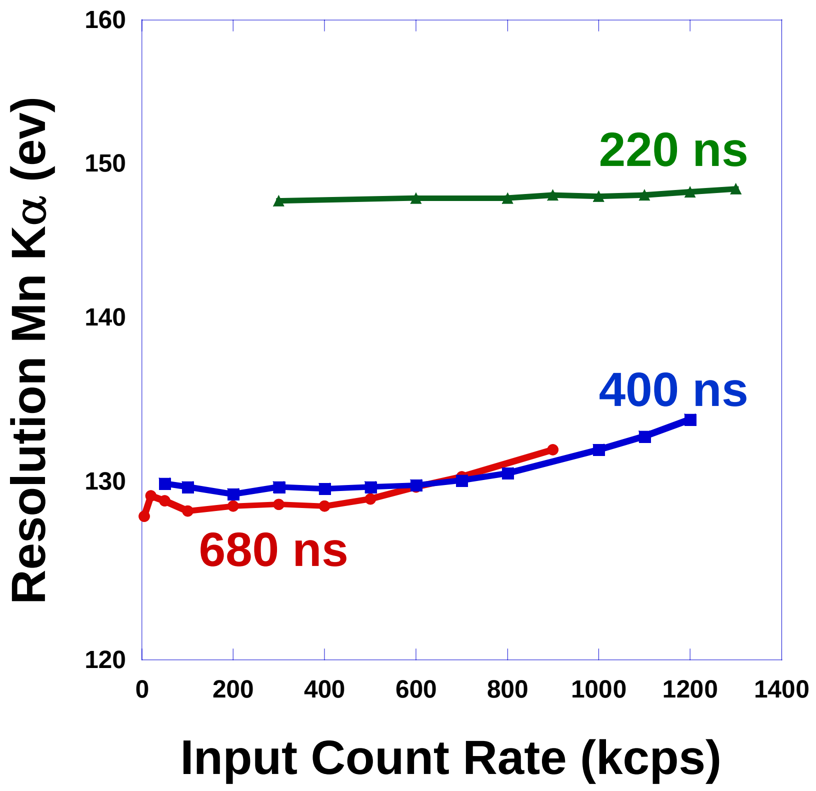
3rd generation
Radiant
50 mm^2 SDD

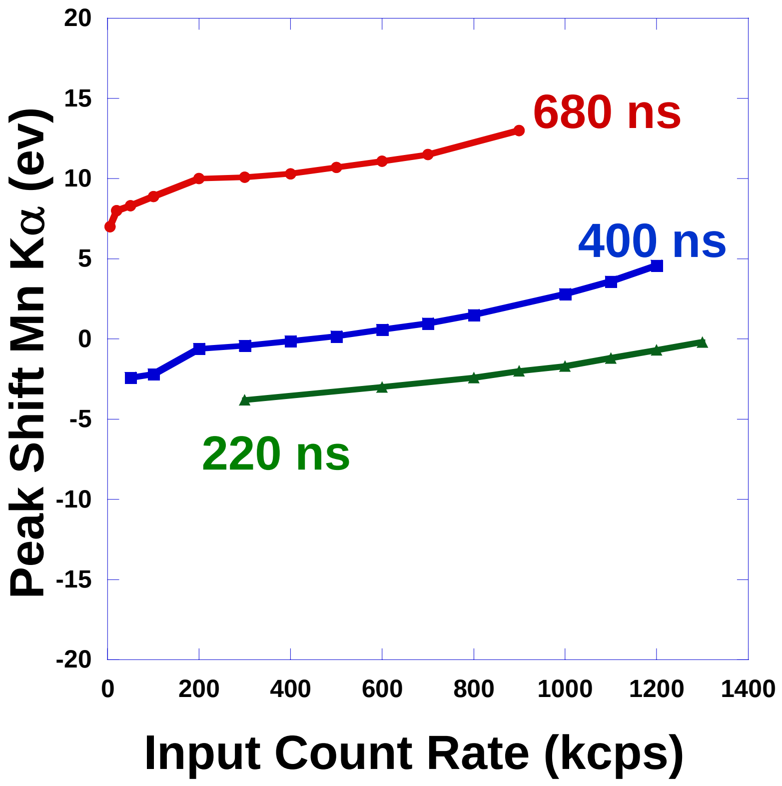
Peak Channel Stability



One
10-eV
channel

Stability of Resolution and Peak Position

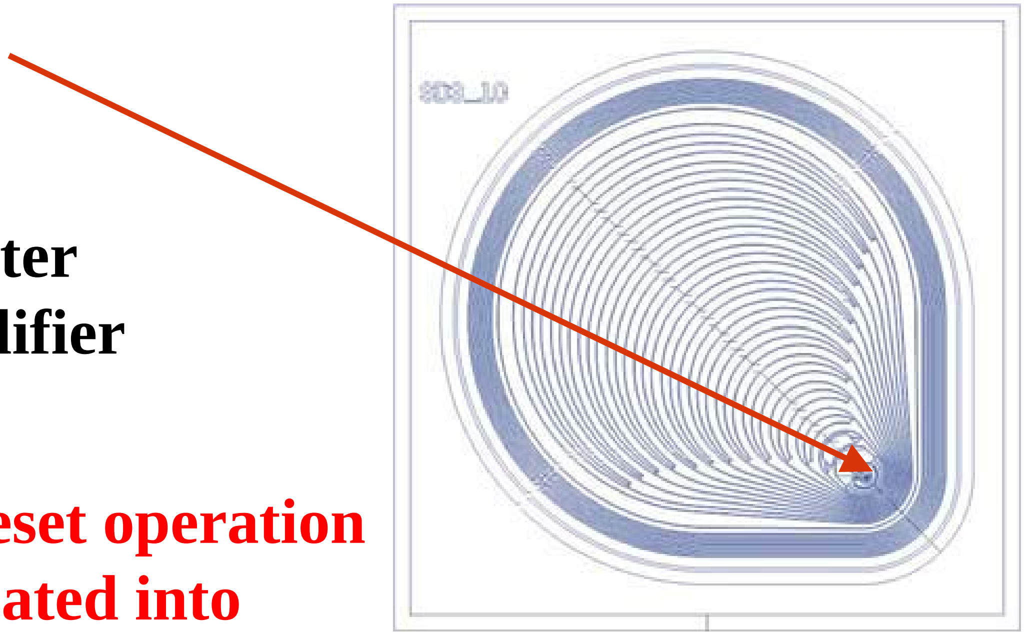




Bruker Quad SDD
Data: Joe Michael,
Sandia National Lab
Albuquerque, NM

Why is this SDD so much faster?

Bruker “Tear–drop” Shaped SDD



OCR speed: On-chip integrated charge amplifier eliminates microphonics; off-center location protects amplifier from x-rays

Peak stability: pulse reset operation with reset diode integrated into the readout anode on the chip.

M&M2005_Optimized Readout Methods of Silicon Drift Detectors for High Resolution Spectroscopy in Micro-Beam Analysis
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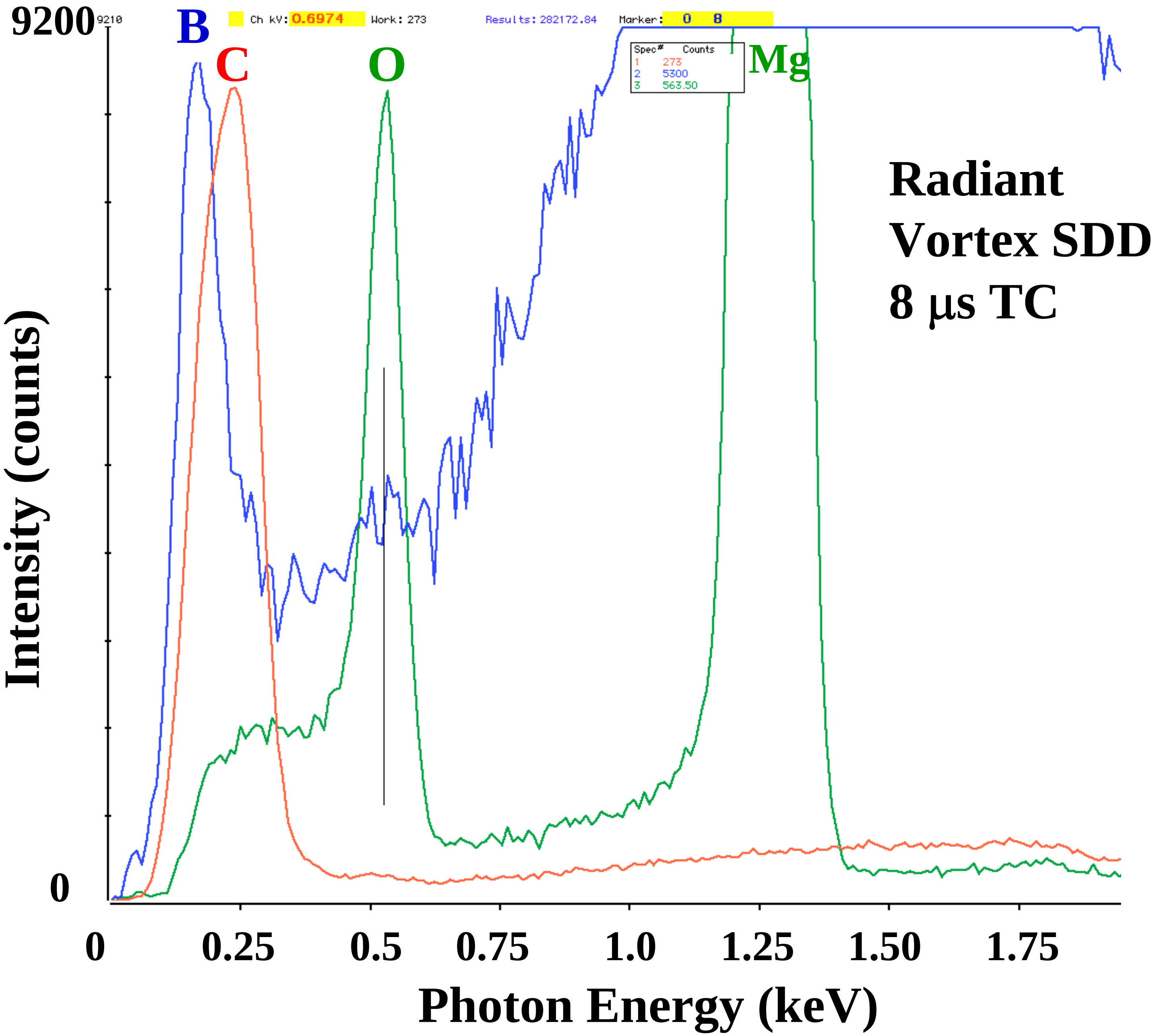
M&M2006_Optimization of the Peak-to-Background Ratio and the Low Energy Response of Silicon Drift Detectors for High Resolution X-ray Spectroscopy
A. Niculae*, H.Soltau*, P.Lechner*, A.Liebl*, G. Lutz**, L. Strüder**, A. Longoni***
R. Eckhard*, G. Schaller** and F. Schopper**

Update on Silicon Drift Detector (SDD) Performance

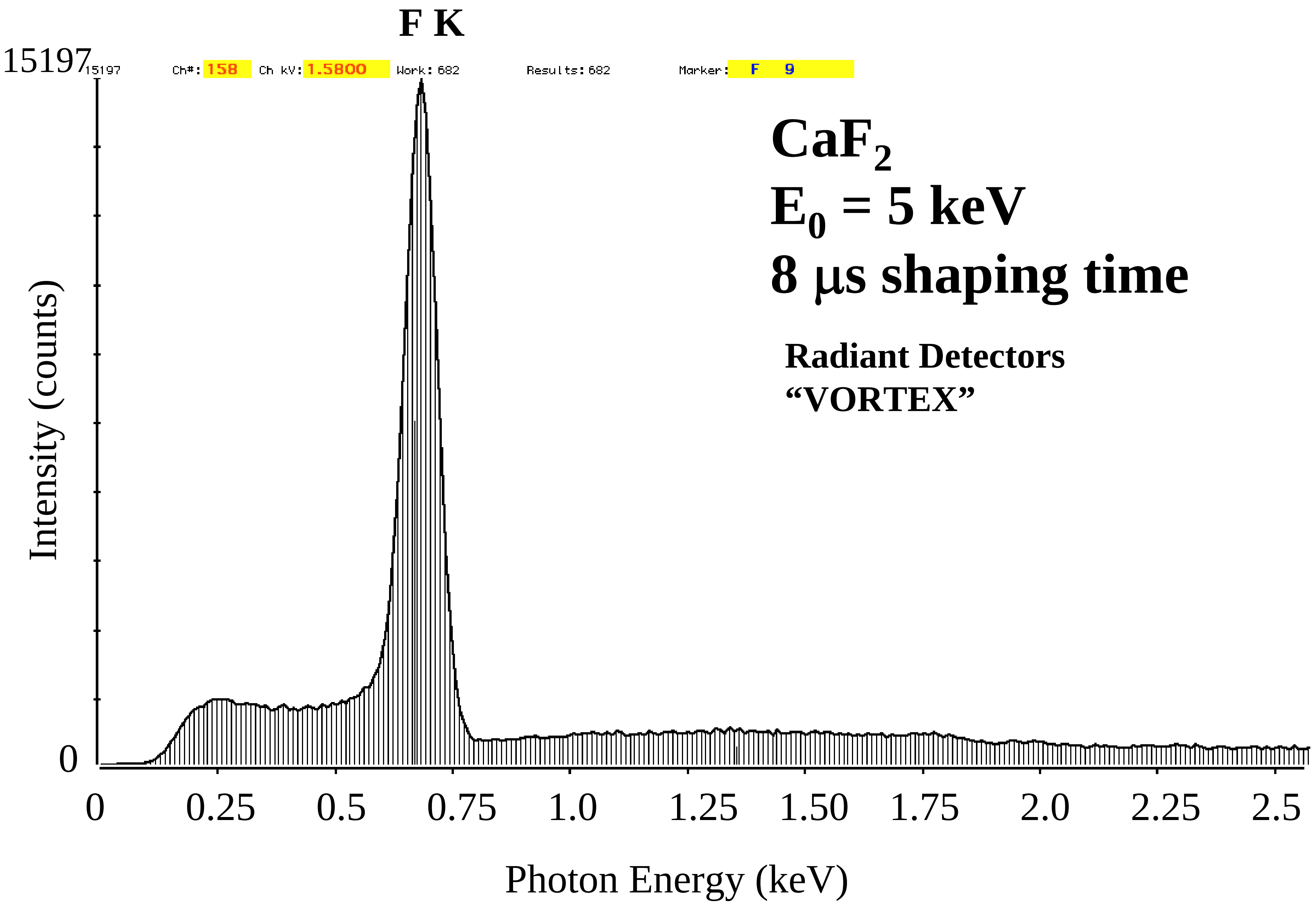
- Processing speed
- Spectral quality
  - Resolution and peak position stability
  - Low photon energy peaks

Low photon energy performance



Radiant
Vortex SDD
8 µs TC

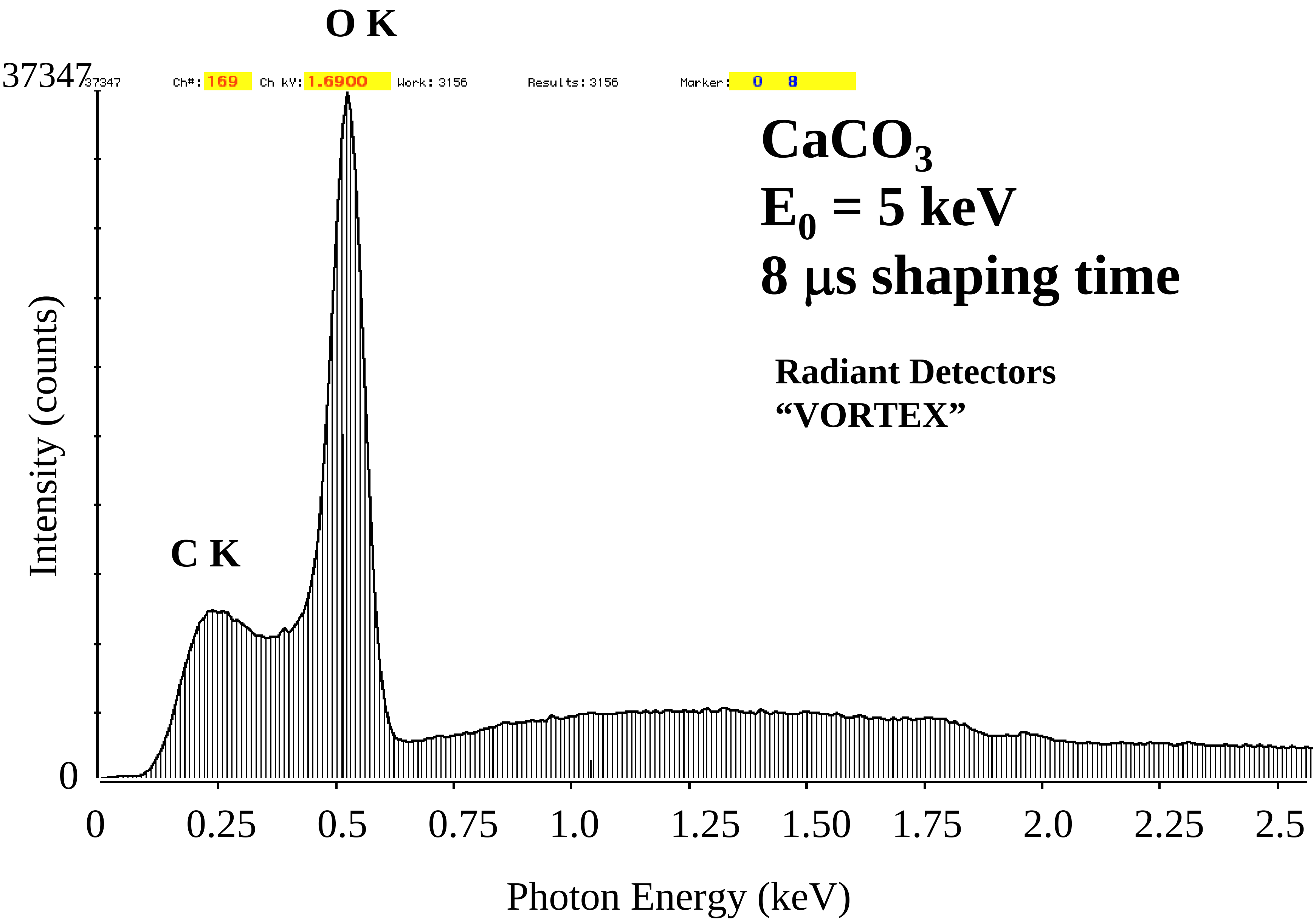
Low photon energy performance



CaF_2
E_0 = 5 keV
8 µs shaping time

Radiant Detectors
“VORTEX”

Low photon energy performance



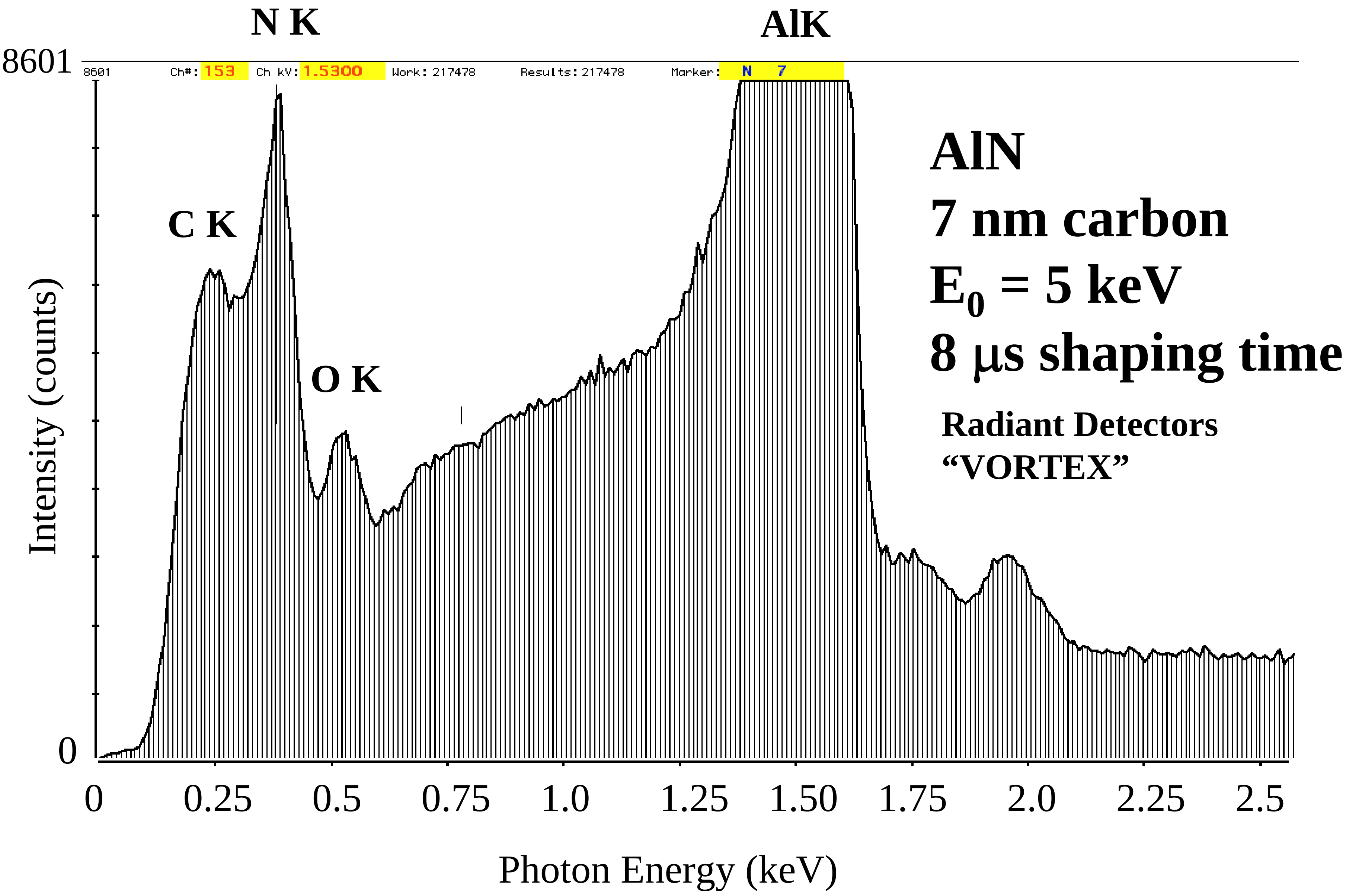
$CaCO_3$

E_0 = 5 keV

8 µs shaping time

Radiant Detectors
“VORTEX”

Low photon energy performance



AlN

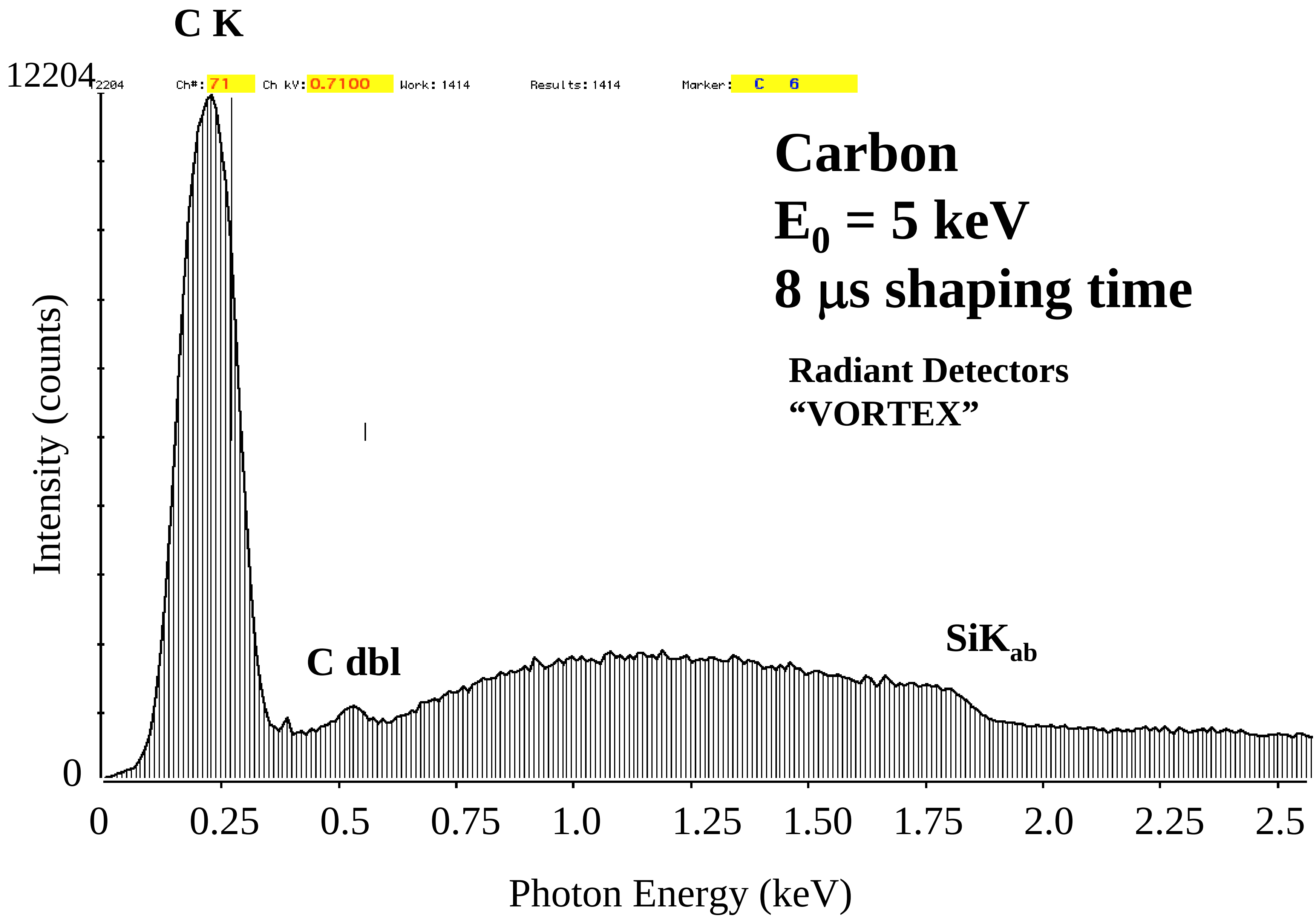
7 nm carbon

E_0 = 5 keV

8 µs shaping time

Radiant Detectors
“VORTEX”

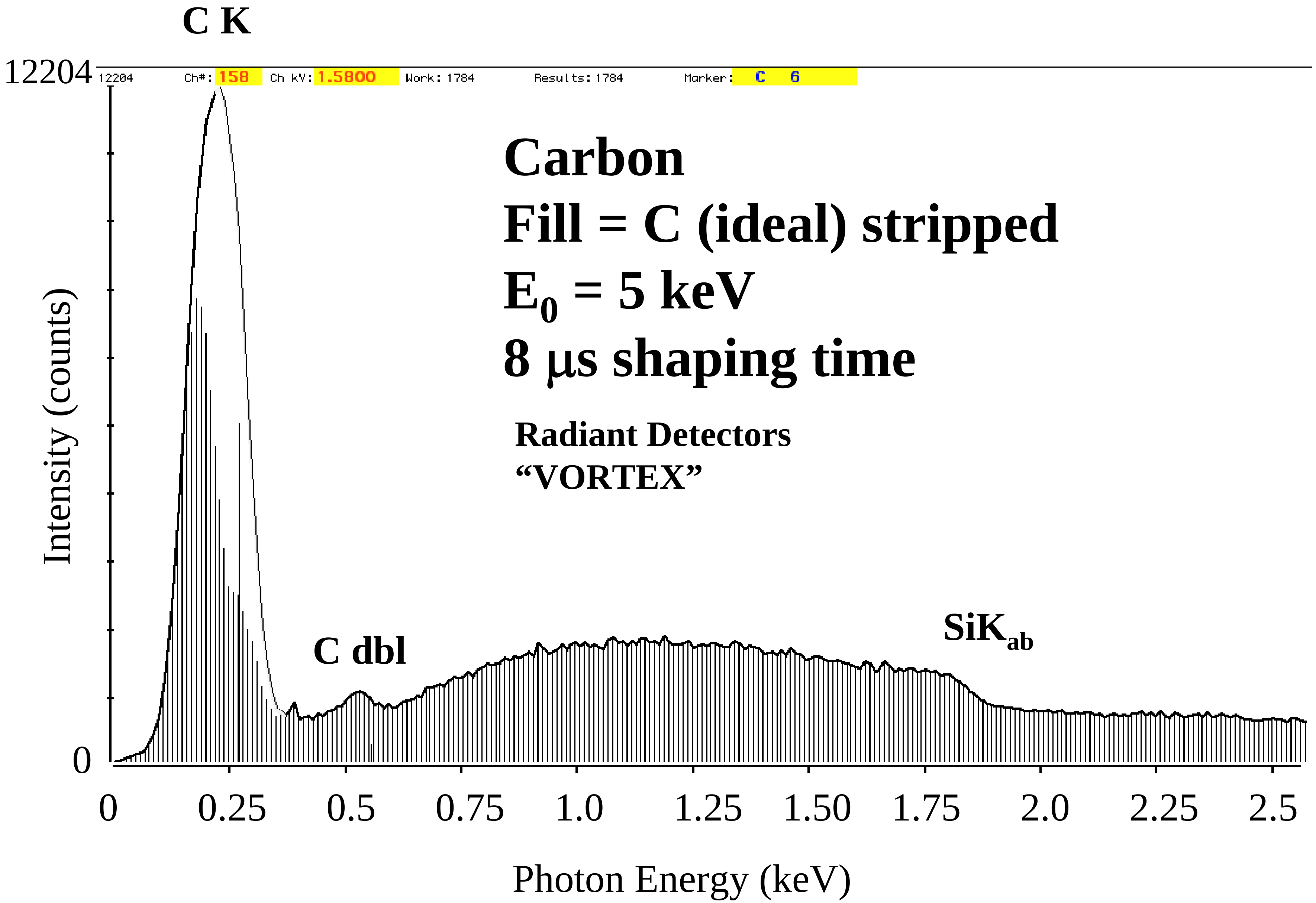
Low photon energy performance



Carbon
E_0 = 5 keV
8 μs shaping time

Radiant Detectors
"VORTEX"

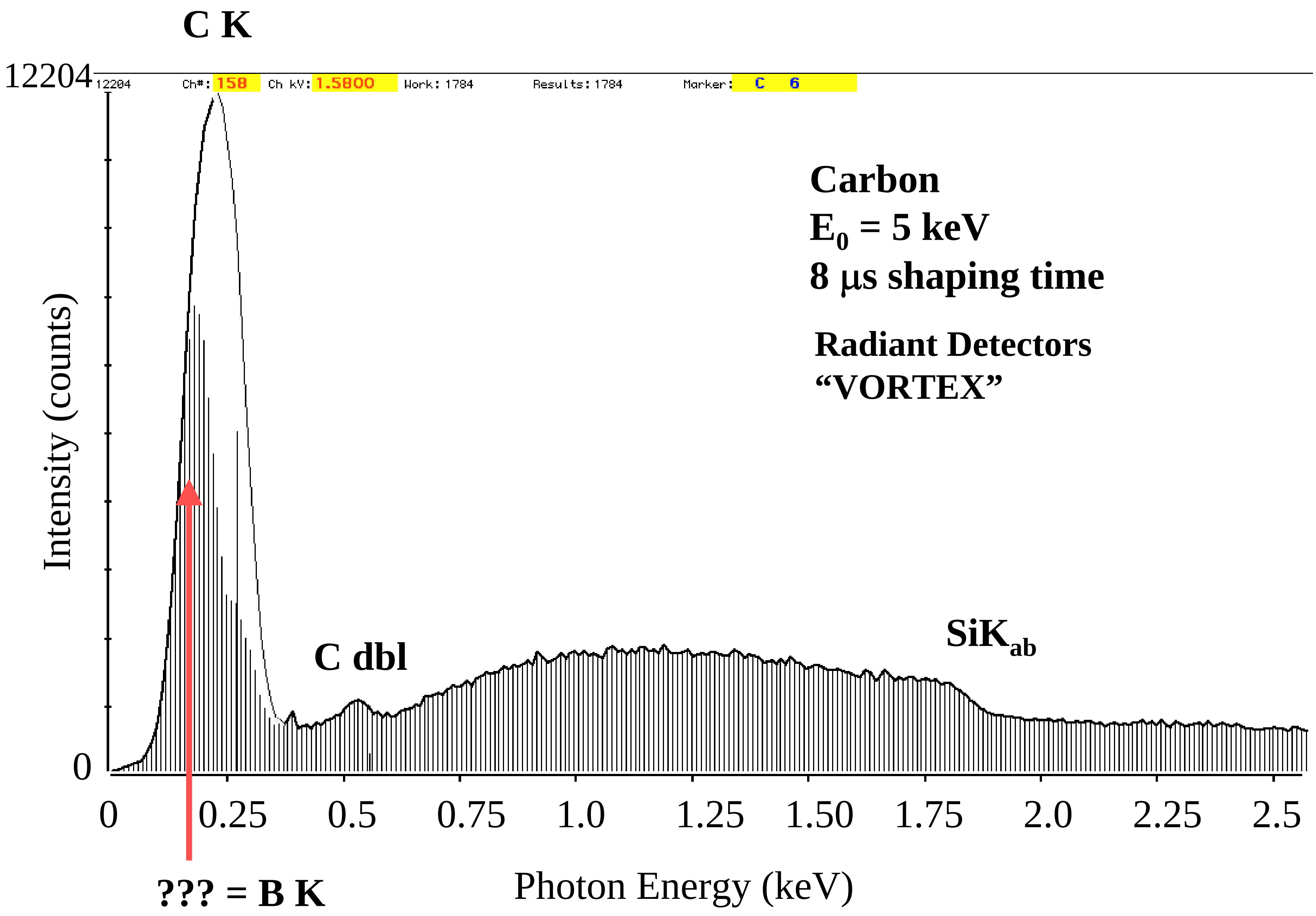
Low photon energy performance



Carbon
Fill = C (ideal) stripped
E_0 = 5 keV
8 µs shaping time

Radiant Detectors
“VORTEX”

Low photon energy performance



Carbon
E_0 = 5 keV
8 μs shaping time

Radiant Detectors
“VORTEX”

??? = B K

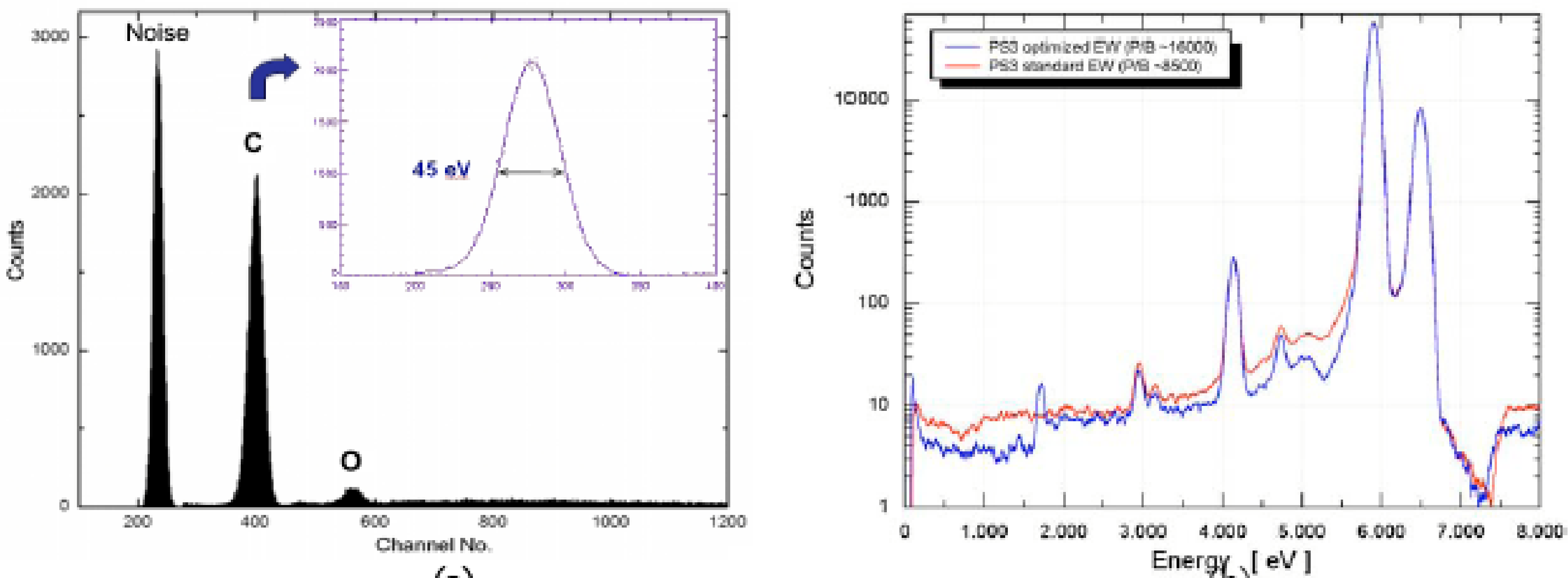
1430 CD
DOI: 10.1017/S1431927607078117

Microsc Microanal 13(Suppl 2), 2007

Optimum Performance for Microanalysis with Silicon Drift Detectors with Integrated FET

A. Niculae*, H.Soltau*, P.Lechner*, A.Liebl*, G. Lutz*, L. Strüder**, R. Eckhard*,
G. Schaller** and F. Schopper**

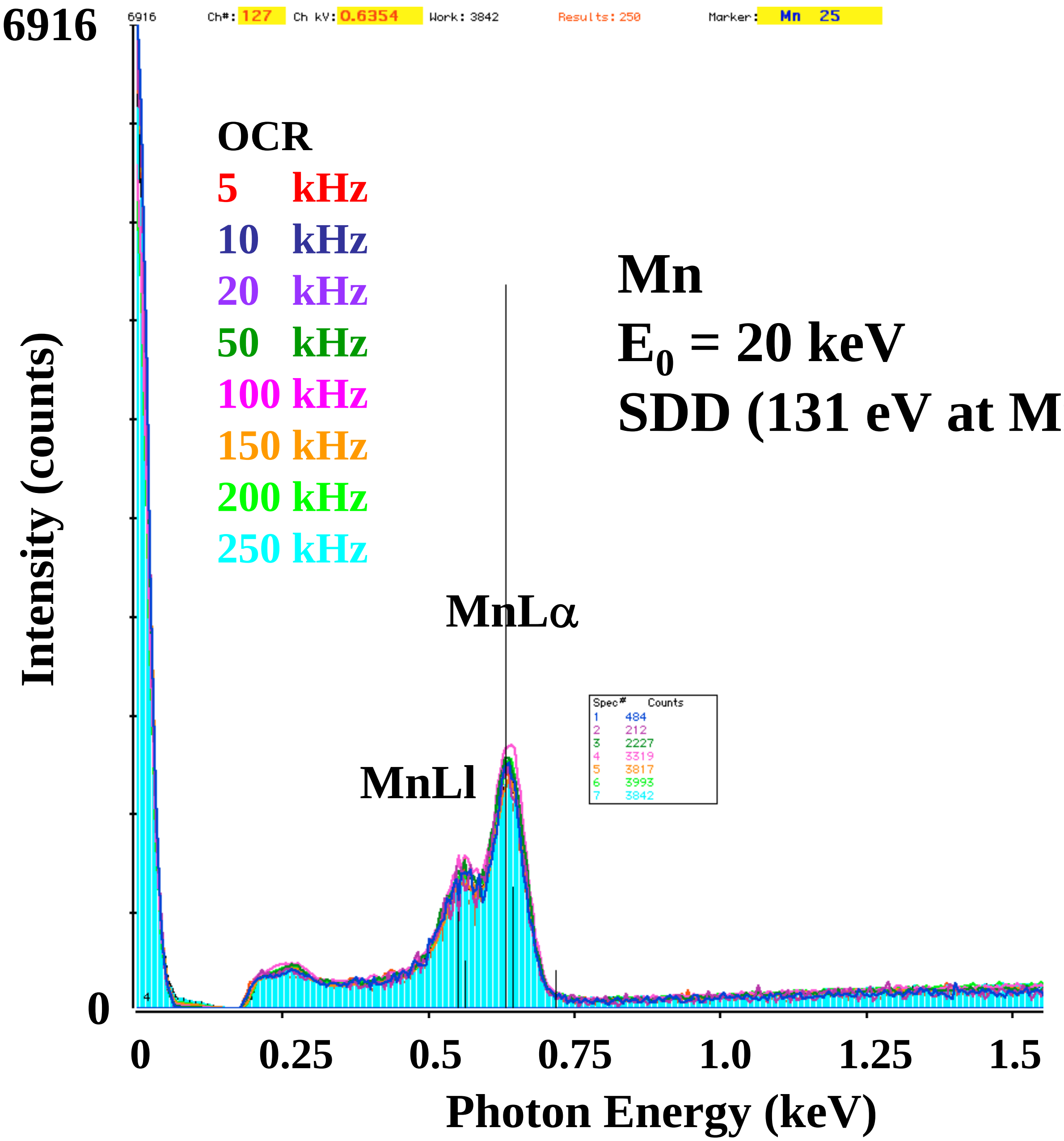
*PNSensor GmbH, Römerstr. 28, D-80803 München, Germany
**MPI Halbleiterlabor, Otto-Hahn-Ring 6, D-81739 München, Germany



(a) (b)

Fig. 2 a) Carbon spectrum measured with a 10 mm^2 SD^3 detector with optimized EW (pnWindow); b) Spectrum of ^{55}Fe source measured with 10 mm^2 SD^3 detectors with standard and optimized EW.

Spectral stability in low photon energy range



Mn
E_0 = 20 keV
SDD (131 eV at MnKα)

DOI: 10.1017/S1431927607071607

Microsc Microanal 13(Suppl 2), 2007

Fast X-ray Mapping with Excellent Light Element Performance from an SDD

Del Redfern, Alan Sandborg and Bob Anderhalt

EDAX Inc, 91 McKee Drive, Mahwah, NJ 07430

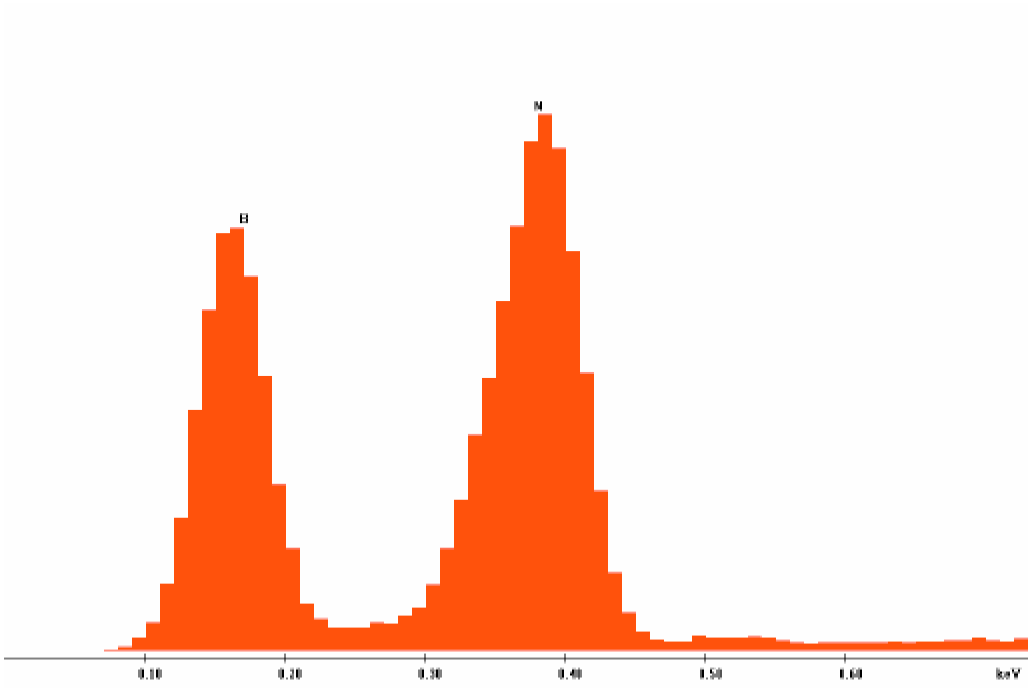
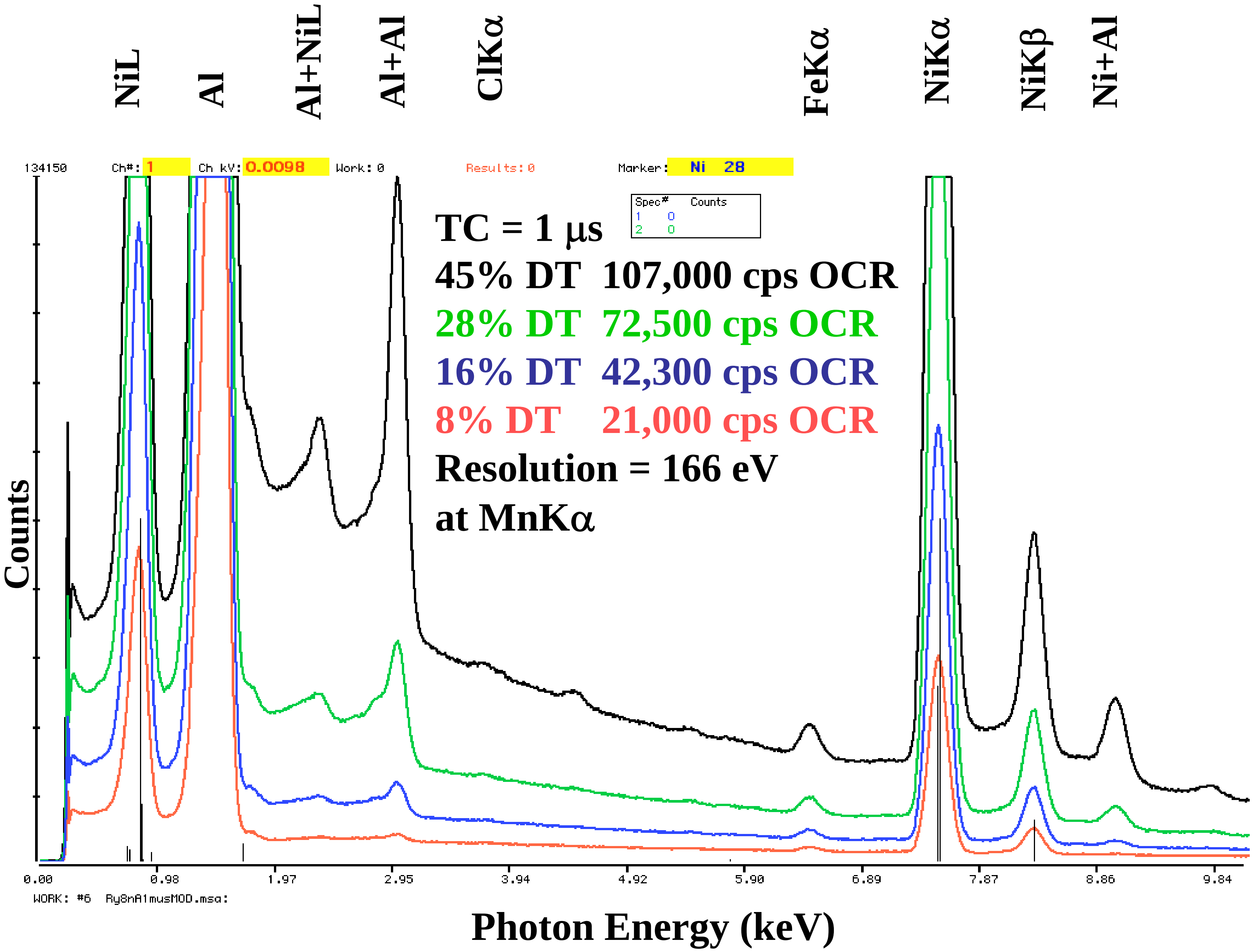


Figure 1: Light element spectra from the Apollo 40 SDD

Update on Silicon Drift Detector (SDD) Performance

- Processing speed
- Spectral quality
  - Resolution and peak position stability
  - Low photon energy peaks
  - Coincidence peaks



TC = 1 µs

45% DT 107,000 cps OCR

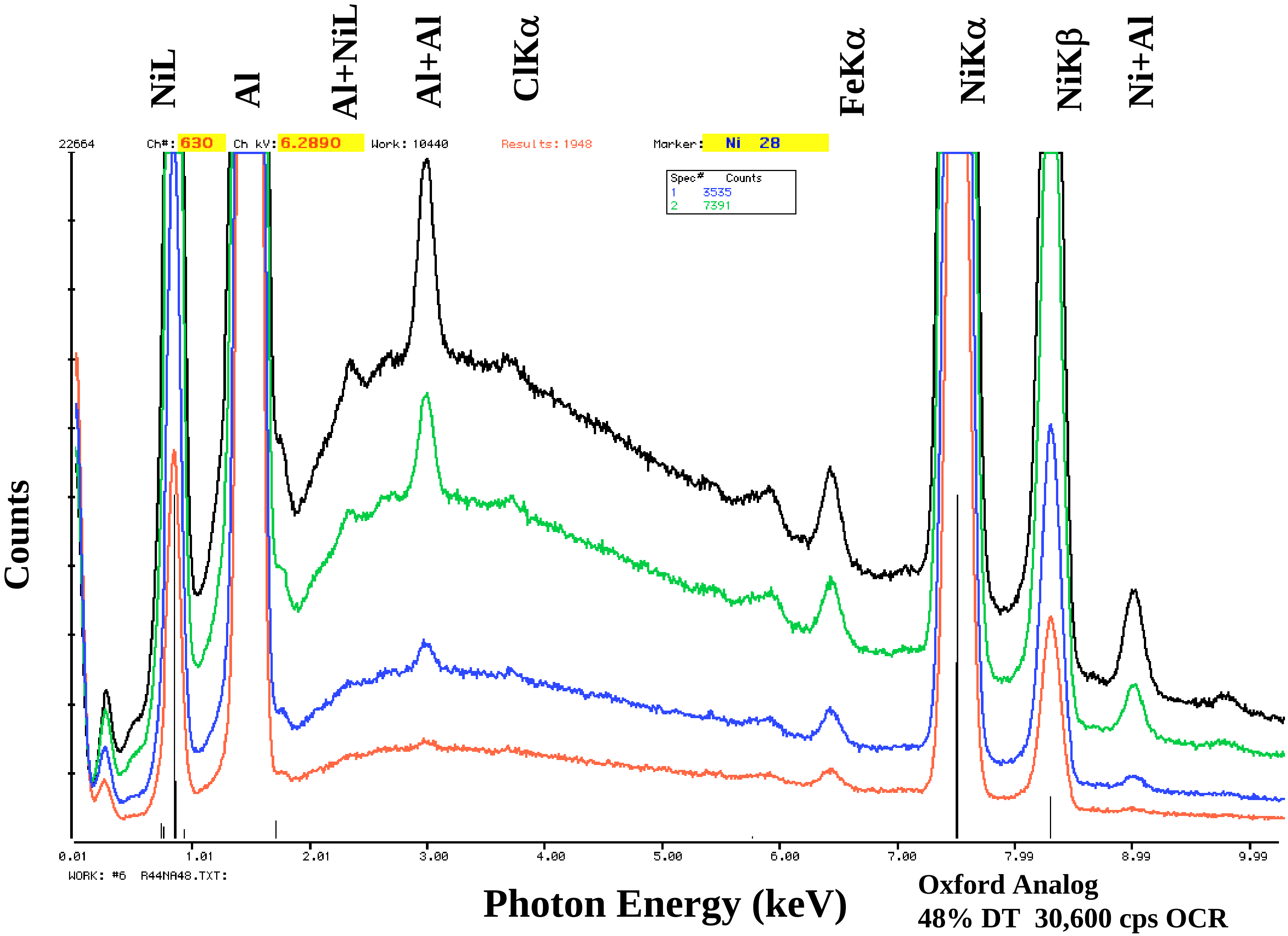
28% DT 72,500 cps OCR

16% DT 42,300 cps OCR

8% DT 21,000 cps OCR

Resolution = 166 eV at MnKα

3rd Generation SDD (Radiant Detectors LLC)



Oxford Analog

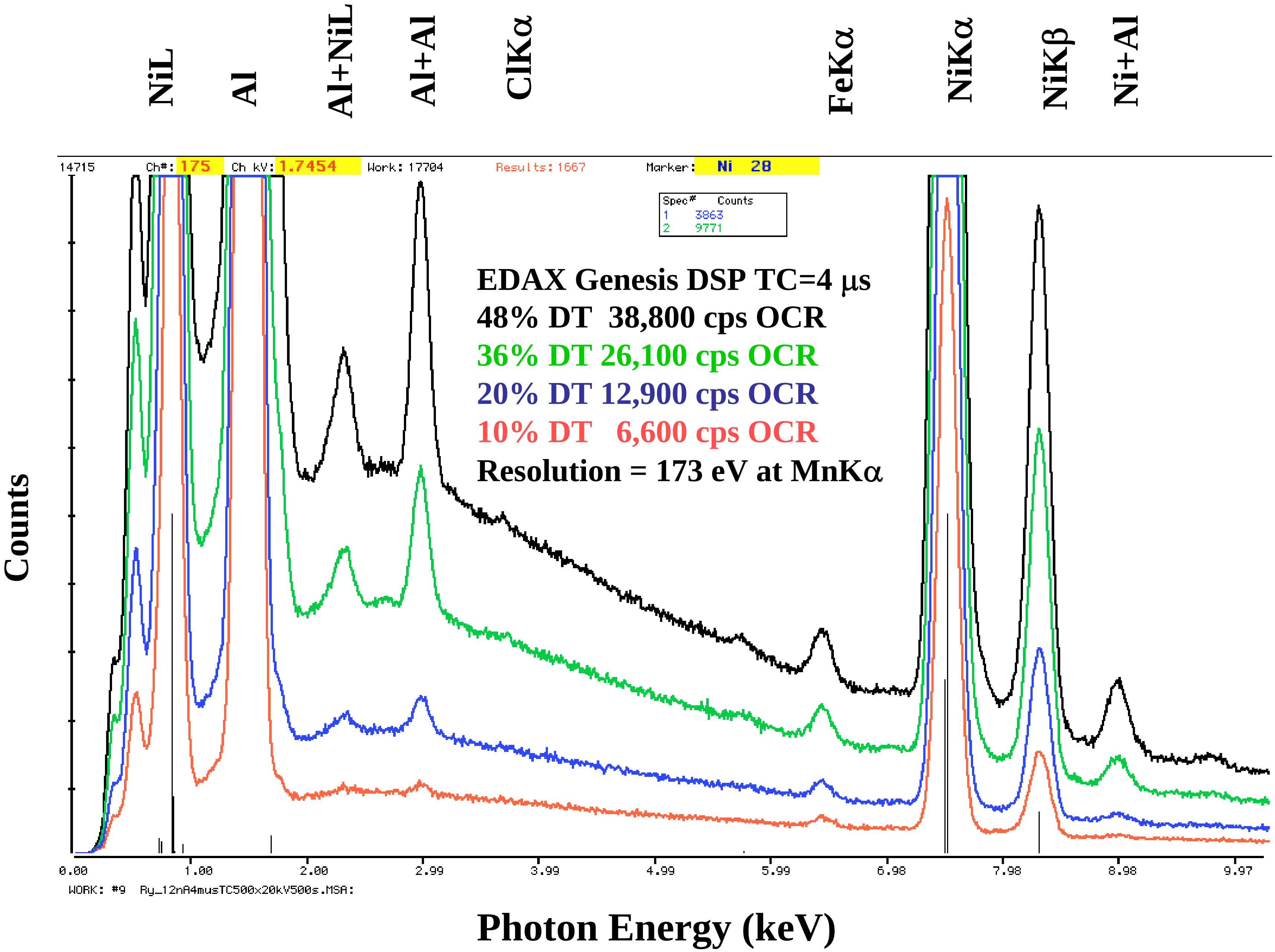
48% DT 30,600 cps OCR

38% DT 26,200 cps OCR

22% DT 17,200 cps OCR

8% DT 9,900 cps OCR

Resolution = 166 eV at MnKα



EDAX Genesis DSP TC=4 μs

48% DT 38,800 cps OCR

36% DT 26,100 cps OCR

20% DT 12,900 cps OCR

10% DT 6,600 cps OCR

Resolution = 173 eV at MnKα

1434 CD
DOI: 10.1017/S1431927607078312

Microsc Microanal 13(Suppl 2), 2007

Improved EDS Pileup Rejection for Low Energies at High Count Rates

R. B. Mott

PulseTor LLC, 328 Rileyville Road, Ringoes, NJ 08551-1501

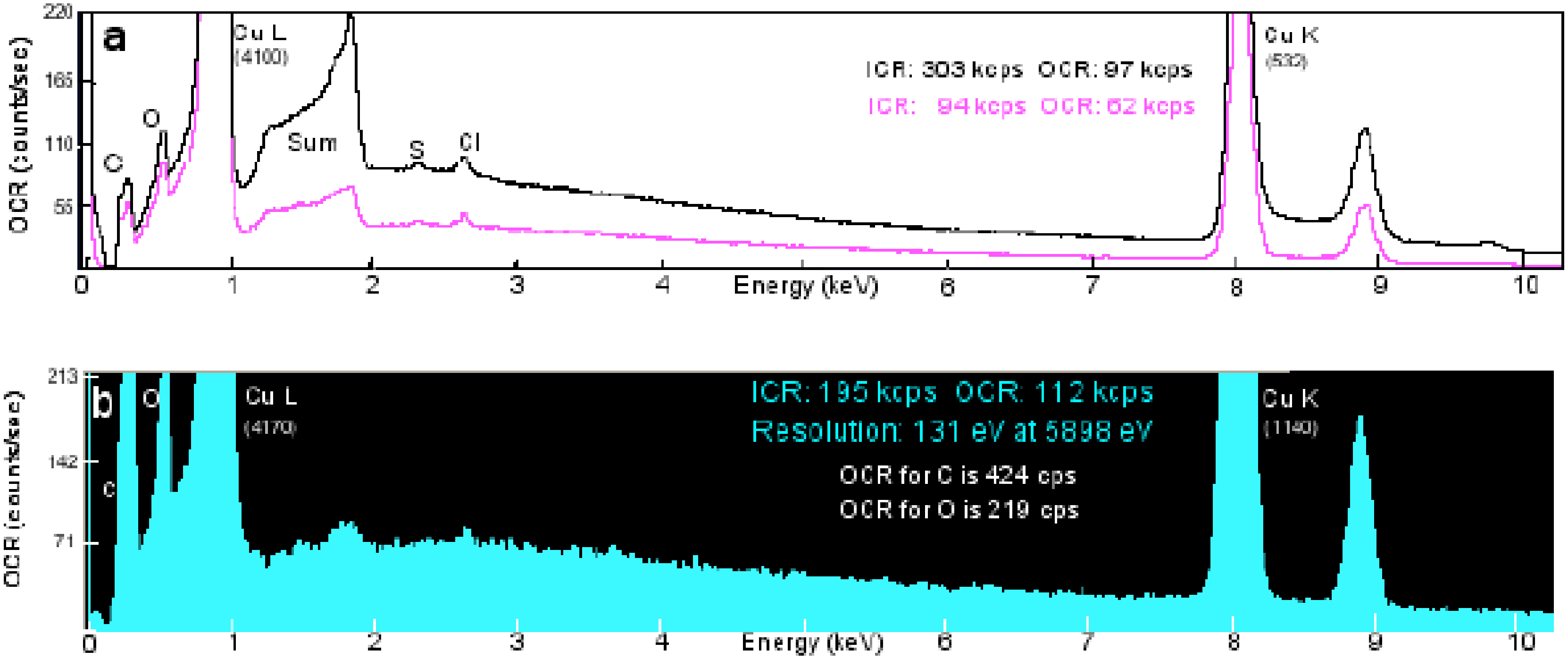


FIG. 2. Sample is contaminated Cu: (a) Bruker XFlash™ detector with Bruker pulse processing; (b) Bruker XFlash™ detector with PulseTor pulse processing.

1428 CD
DOI: 10.1017/S1431927607073242

Microsc Microanal 13(Suppl 2), 20

Digital pulse processing and pile up correction for accurate interpretation of high rate SDD spectrum images

P.J.Statham

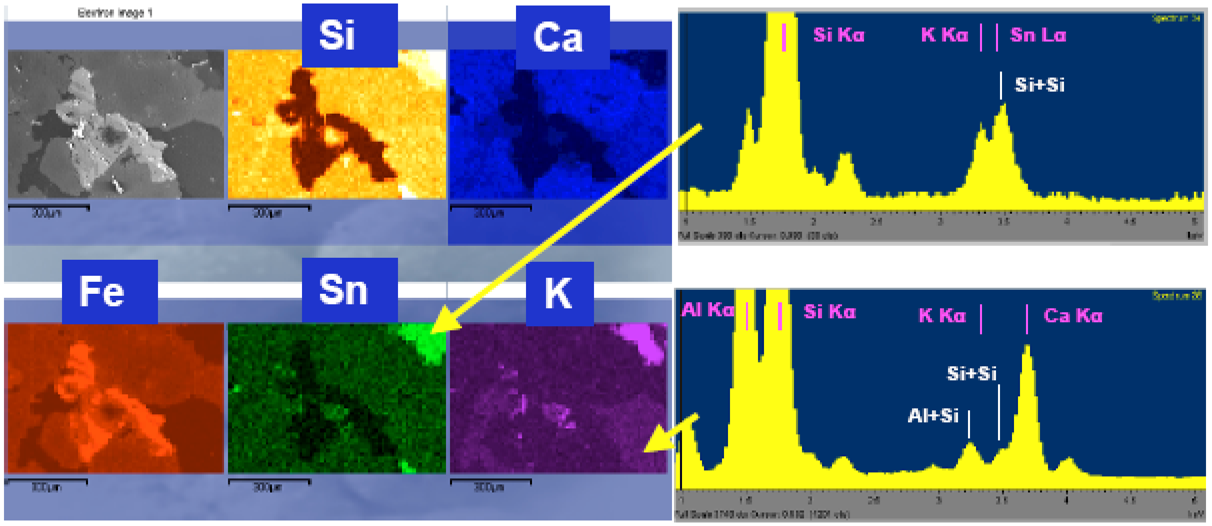


Fig.1 : X-ray data obtained at 250kcps. Conventional x-ray maps show integrated intensity for energy windows spanning each elemental peak Top spectrum is average over bright region in Sn area bottom right of K map.

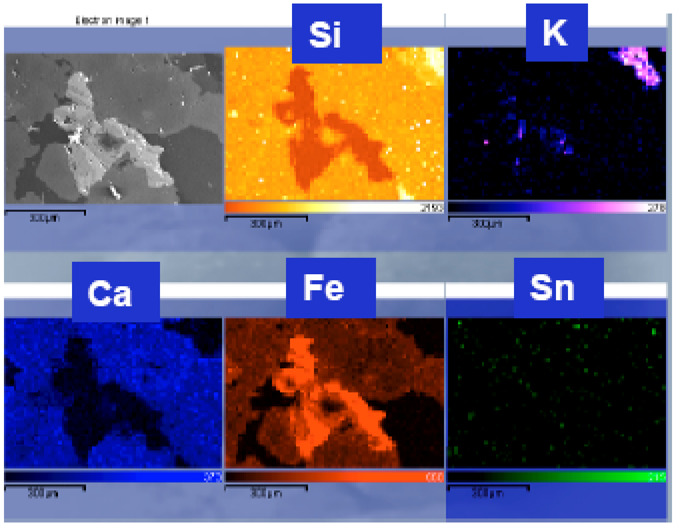


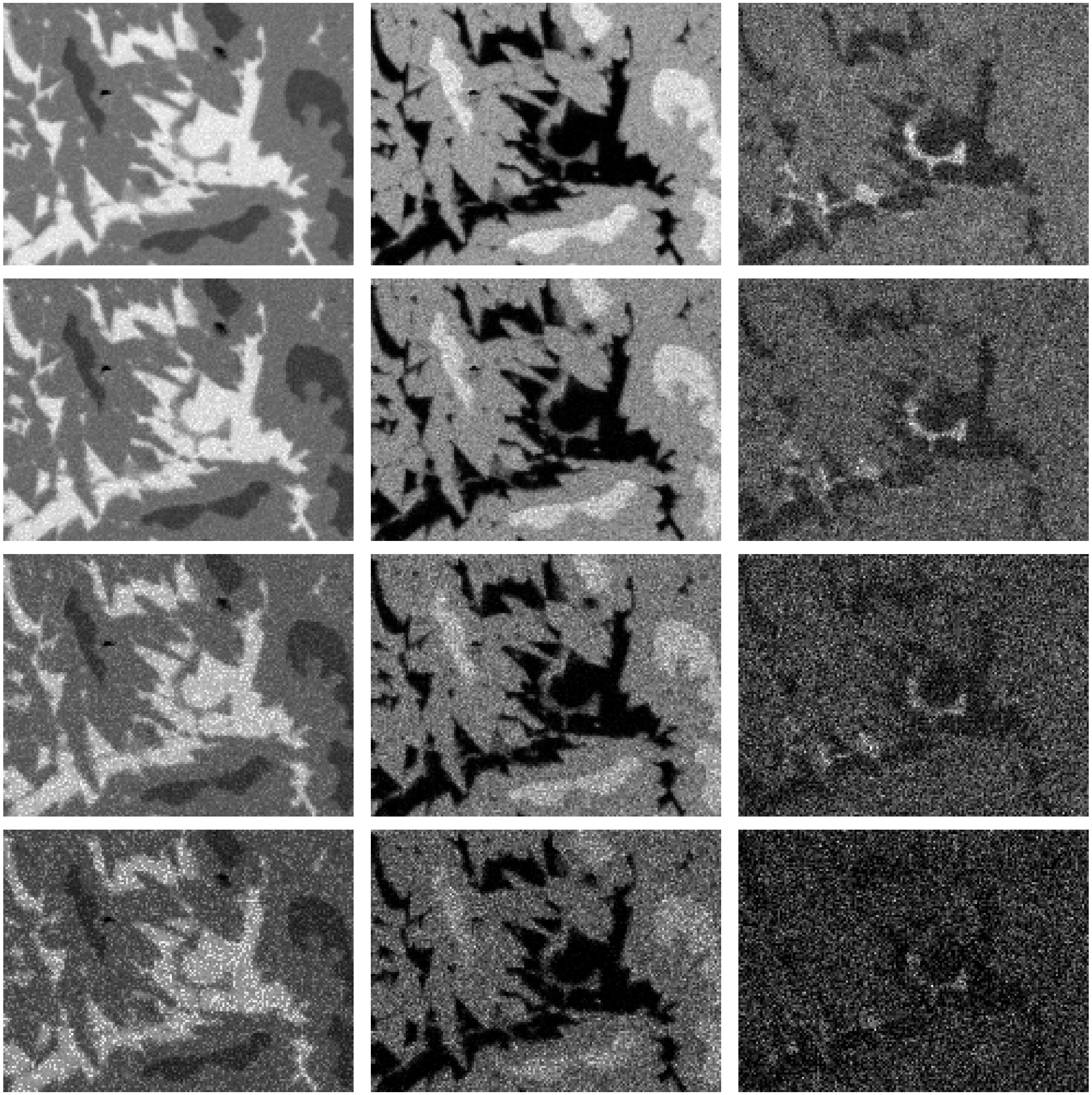
Fig.2 : Same spectral image data as for fig.1. Quantitative element maps obtained after applying pile-up correction algorithm to spectra at individual pixels, then using digital filter and least squares fitting of peak profiles to subtract background and resolve peak overlaps.

SDD can provide the OCR speed

- To exploit this OCR speed for mapping (e.g., by x-ray spectrum imaging), we need:
- Suitably intense x-ray production, that is, an SEM that can deliver high beam currents (10 – 1000 nA) and a specimen that can withstand high dose without degradation.

SDD can provide the OCR speed

- To exploit this OCR speed for mapping (e.g., by x-ray spectrum imaging), we need:
- Suitably intense x-ray production, that is, an SEM that can deliver high beam currents (10 – 1000 nA) and a specimen that can withstand high dose without degradation.
- Low (ideally “no”) overhead x-ray event processing (try to avoid wasting time sorting and storing “no information” channels)



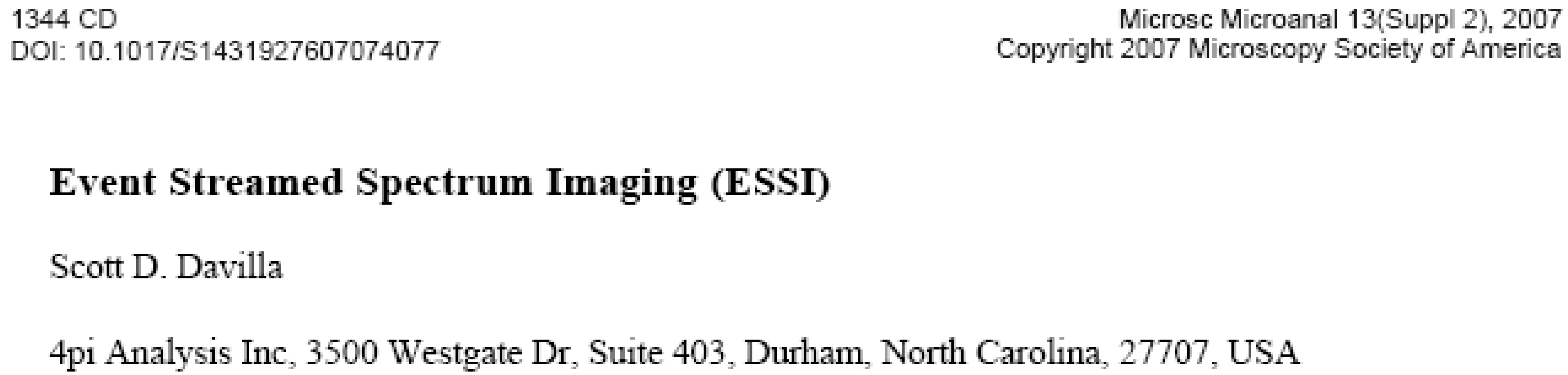
160x120 pixels +1.3 ms overhead	How fast?
10 ms	217 s 192 s 12%
5 ms	121 s 96 s 21%
2 ms	63 s 38 s 40%
1 ms	44 s 19 s 57%

↔ **100 µm**

SDD can provide the OCR speed

- To exploit this OCR speed for mapping (e.g., by x-ray spectrum imaging), we need:
- Suitably intense x-ray production, that is, an SEM that can deliver high beam currents (10 – 1000 nA) and a specimen that can withstand high dose without degradation.

2. Low (ideally “no”) overhead x-ray event processing: use “position tagged spectrometry” implemented as “event streaming in hardware”, e.g., see:



1344 CD
DOI: 10.1017/S1431927607074077

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Event Streamed Spectrum Imaging (ESSI)

Scott D. Davilla

4pi Analysis Inc, 3500 Westgate Dr, Suite 403, Durham, North Carolina, 27707, USA

Efficient handling of data stream from SDD: Event Streaming (No overhead; move units of information)

- X-ray events are outputted directly from the x-ray pulse processor’s auxiliary interface bus into the x-y scan generator. The events are then interpreted and combined with the x-ray position information into pixel events. The pixel events are packetized and streamed to a host computer where they are buffered and stored. The whole process takes place at the hardware level with an extremely high speed and is thus highly efficient. **(Pixel overhead μs rather than ms)**

High Speed Spectrum Imaging of Raney Nickel Alloy Using a Large Area Silicon Multi-Cathode Detector (Vortex-EMTM) and Event Streaming Technique M&M 2006

L. Feng*, V. D. Saveliev*, S. Barkan*, C. R. Tull*, M. Takahashi*, N. Matsumori*, S. D. Davilla**, J. S. Iwanczyk***, D. E. Newbury**** and J. A. Small****

SDD can provide the OCR speed

- To exploit this OCR speed for mapping (e.g., by x-ray spectrum imaging), we need:
- Suitably intense x-ray production, that is, an SEM that can deliver high beam currents (10 – 1000 nA) and a specimen that can withstand high dose without degradation.

2. Low (ideally “no”) overhead x-ray event processing: use “position tagged spectrometry” implemented as “event streaming in hardware”,

With these conditions reasonably satisfied:

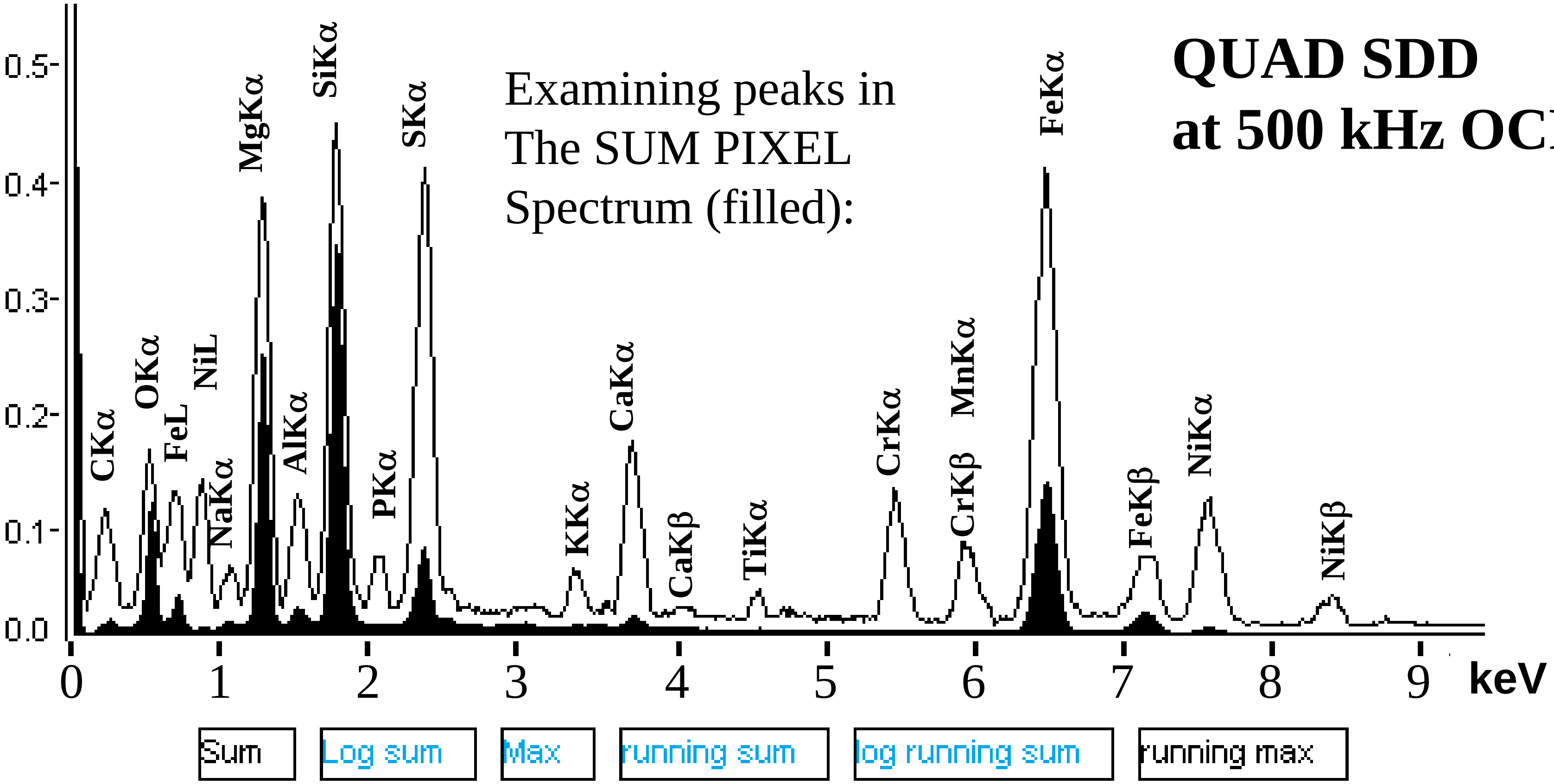
Specimen: Portland St. Meteorite

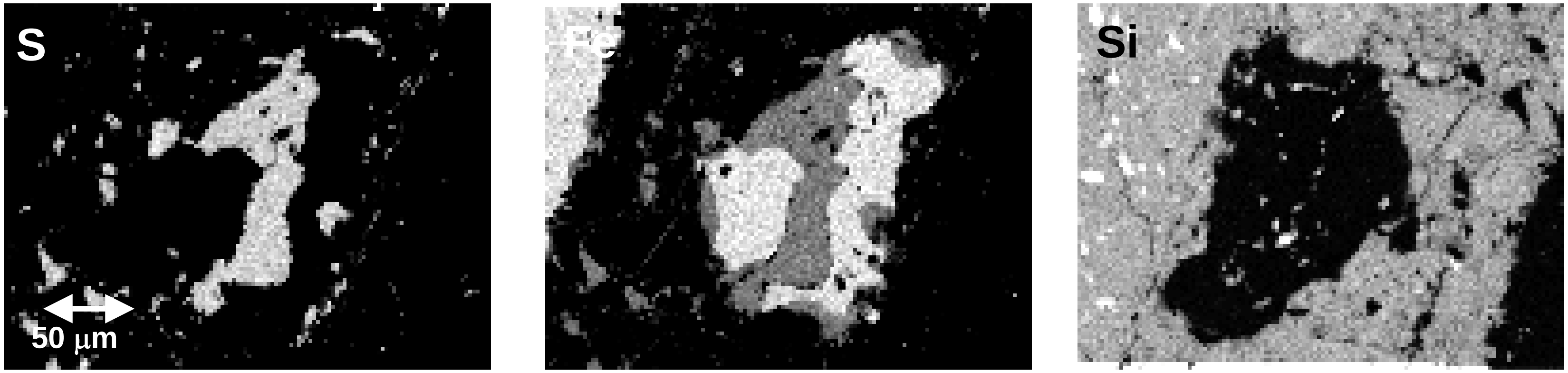
OCR = 550kHz Quad SDD, resolution MnKα < 140 eV

128 x 96 pixels; 1 ms dwell per pixel = 12.3 seconds

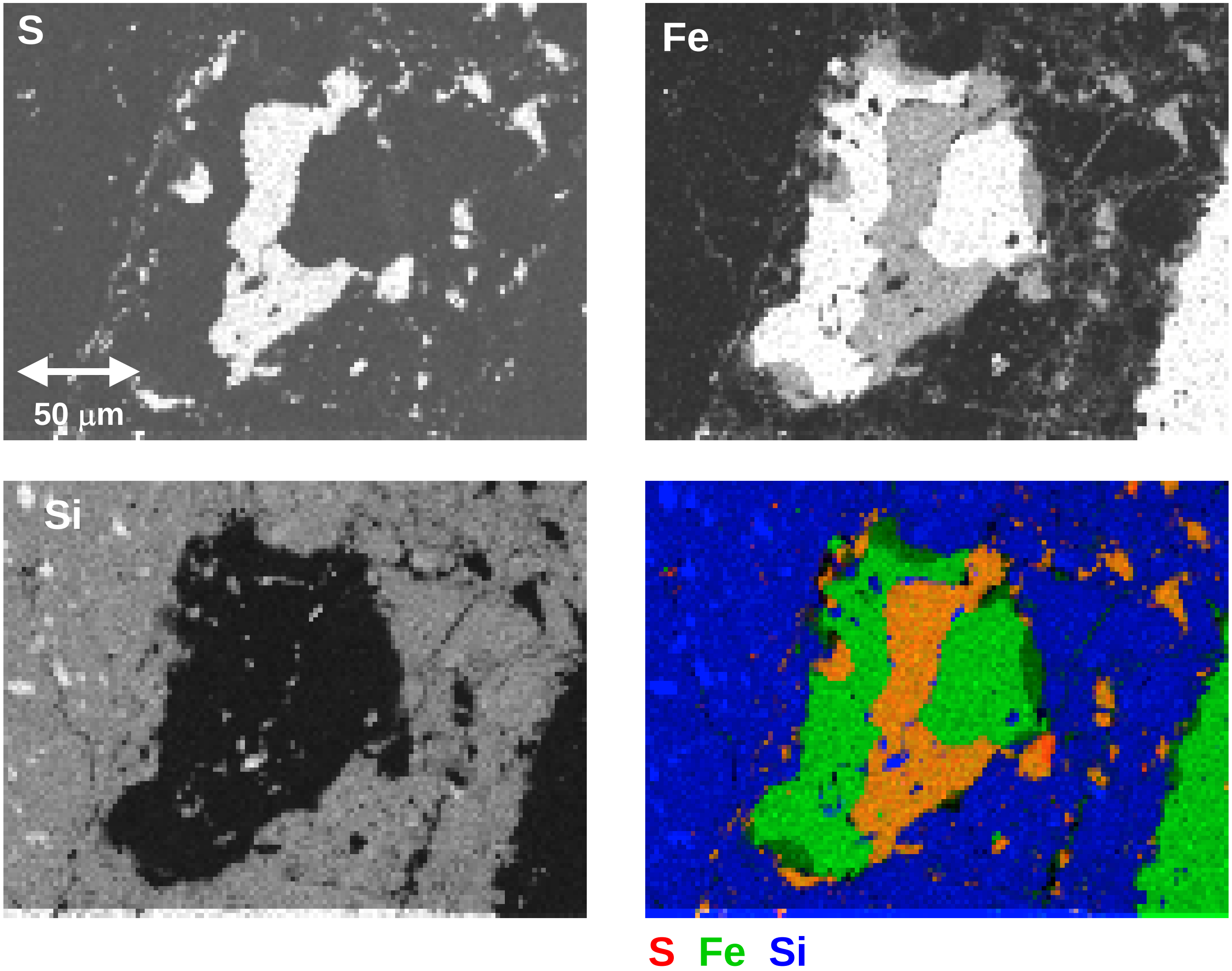
QUAD SDD
at 500 kHz OCR

Examining peaks in
The SUM PIXEL
Spectrum (filled):





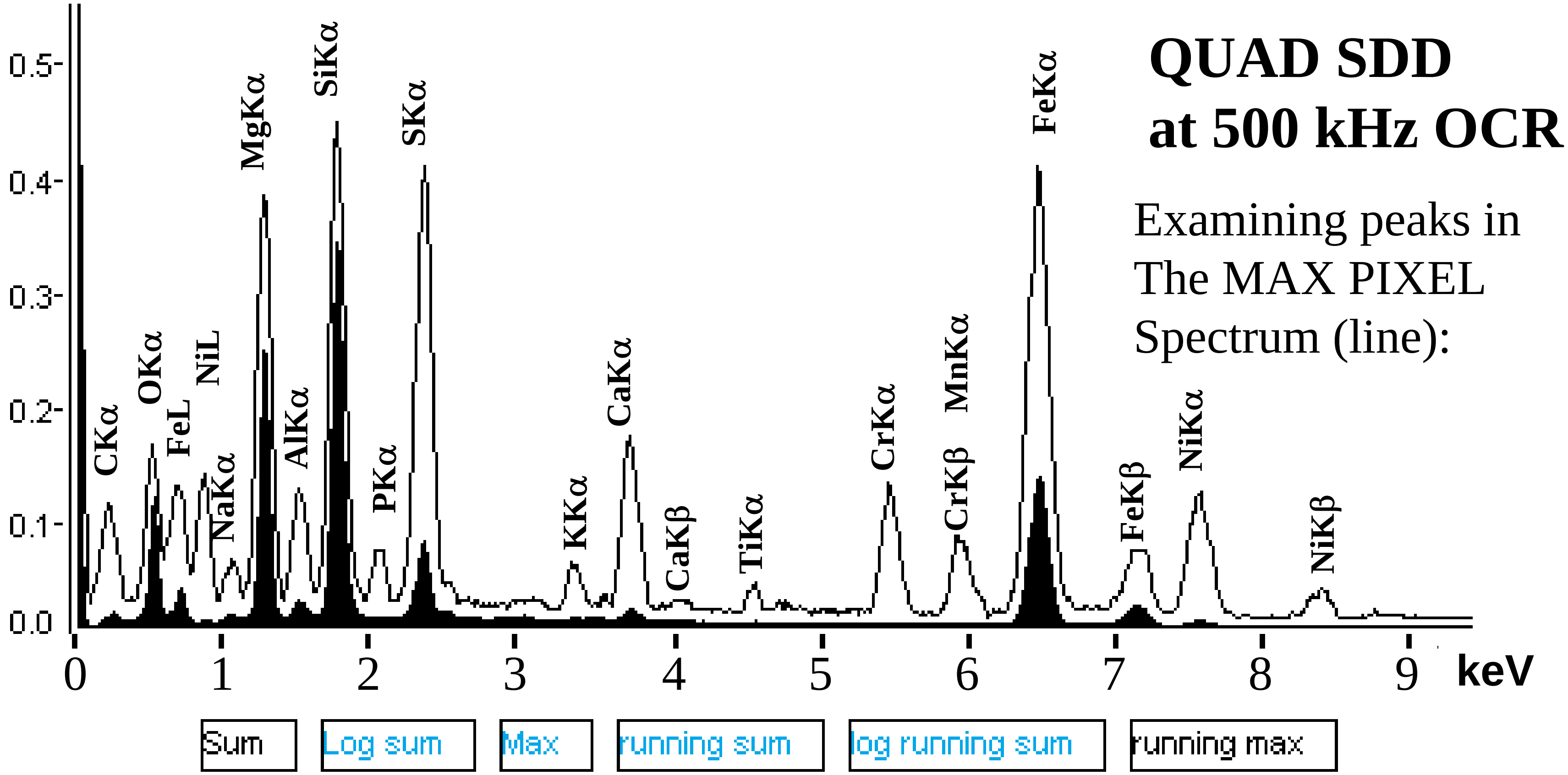
This is a 12 second x-ray spectrum image (128x96 pixels)!

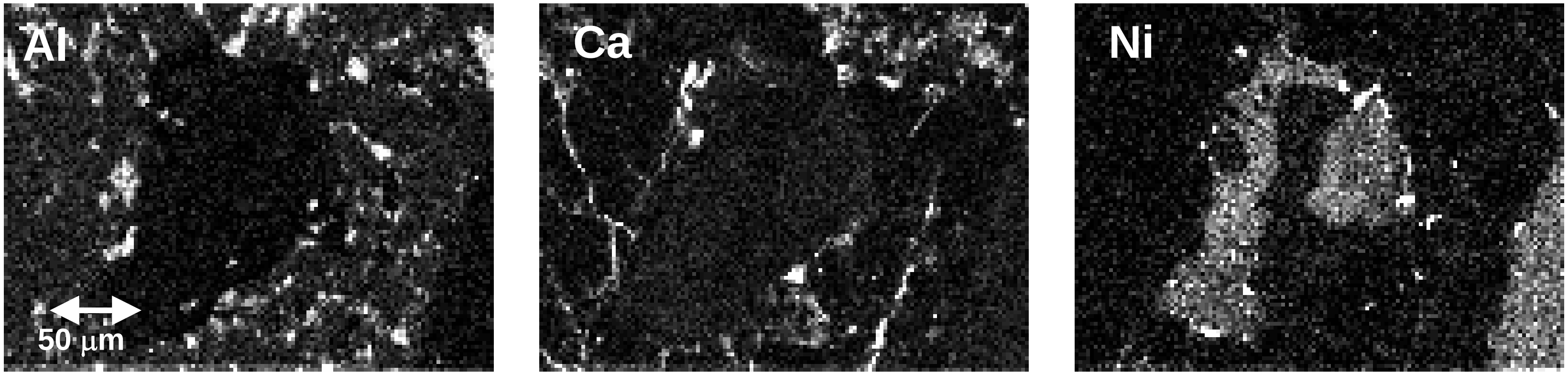


This is a 12 second x-ray spectrum image (128x96 pixels)!

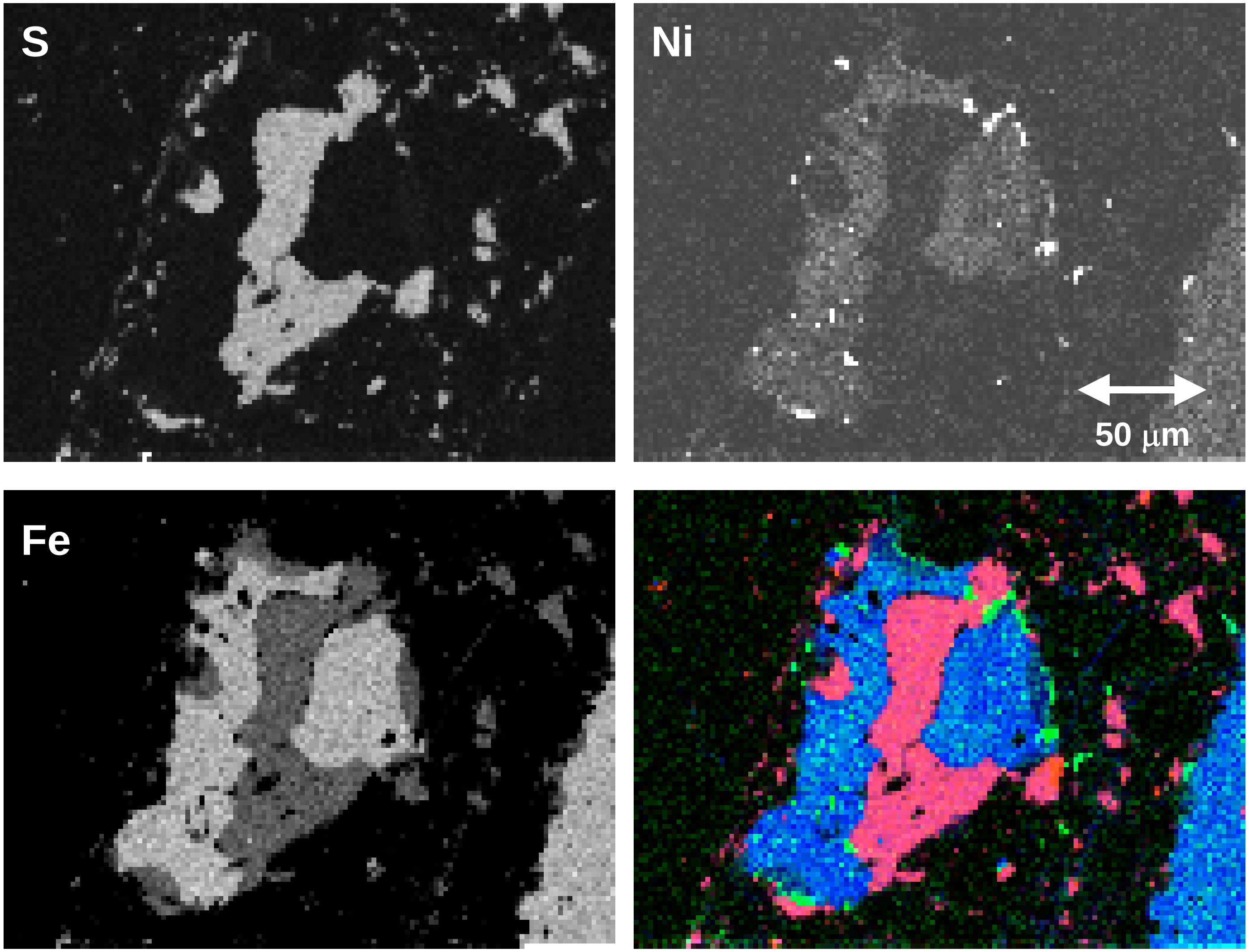
QUAD SDD
at 500 kHz OCR

Examining peaks in
The MAX PIXEL
Spectrum (line):

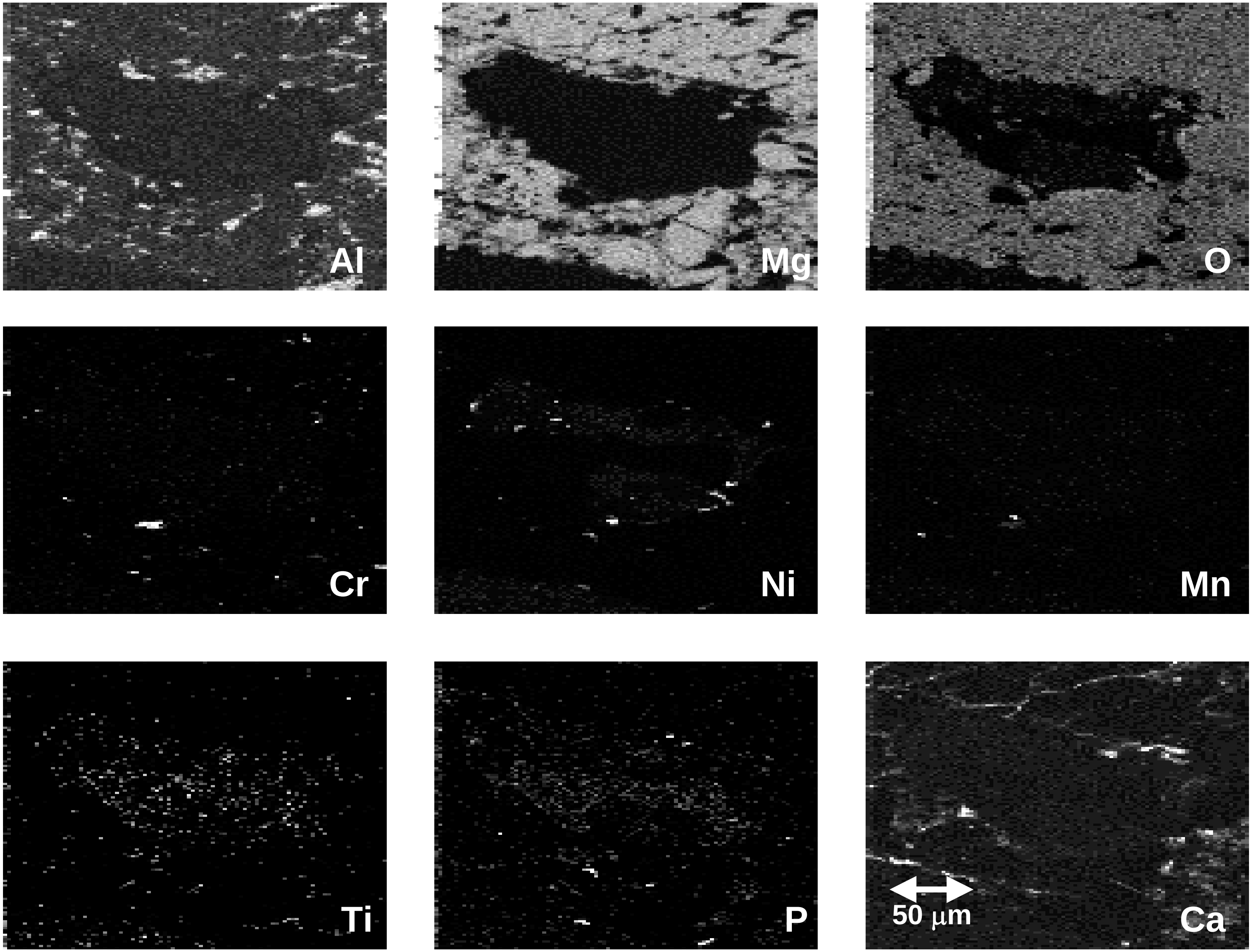




This is a 12 second x-ray spectrum image (128x96 pixels)!



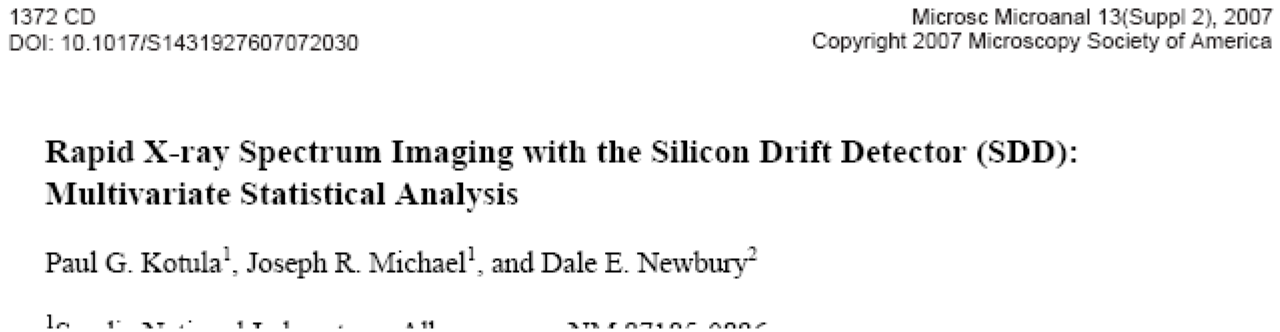
This is a 12 second x-ray spectrum image (128x96 pixels)!



This is a 12 second x-ray spectrum image!

With x-ray spectrum imaging now possible in 10 to 100 seconds with optimized SDD:

- 1. How can we recover information buried in these ~ 100 Mbyte – 1 Gbyte files?
  - Sophisticated statistical approach, see:



1372 CD
DOI: 10.1017/S1431927607072030

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Rapid X-ray Spectrum Imaging with the Silicon Drift Detector (SDD): Multivariate Statistical Analysis

Paul G. Kotula[1], Joseph R. Michael[1], and Dale E. Newbury[2]

- Simple minded, operator intensive approach:

NIST LISPIX (available for free at www.nist.gov; search “LISPIX”)

LISPIX is a comprehensive image manipulation software engine that includes many tools that are useful for the study of x-ray spectrum images and extraction of information.

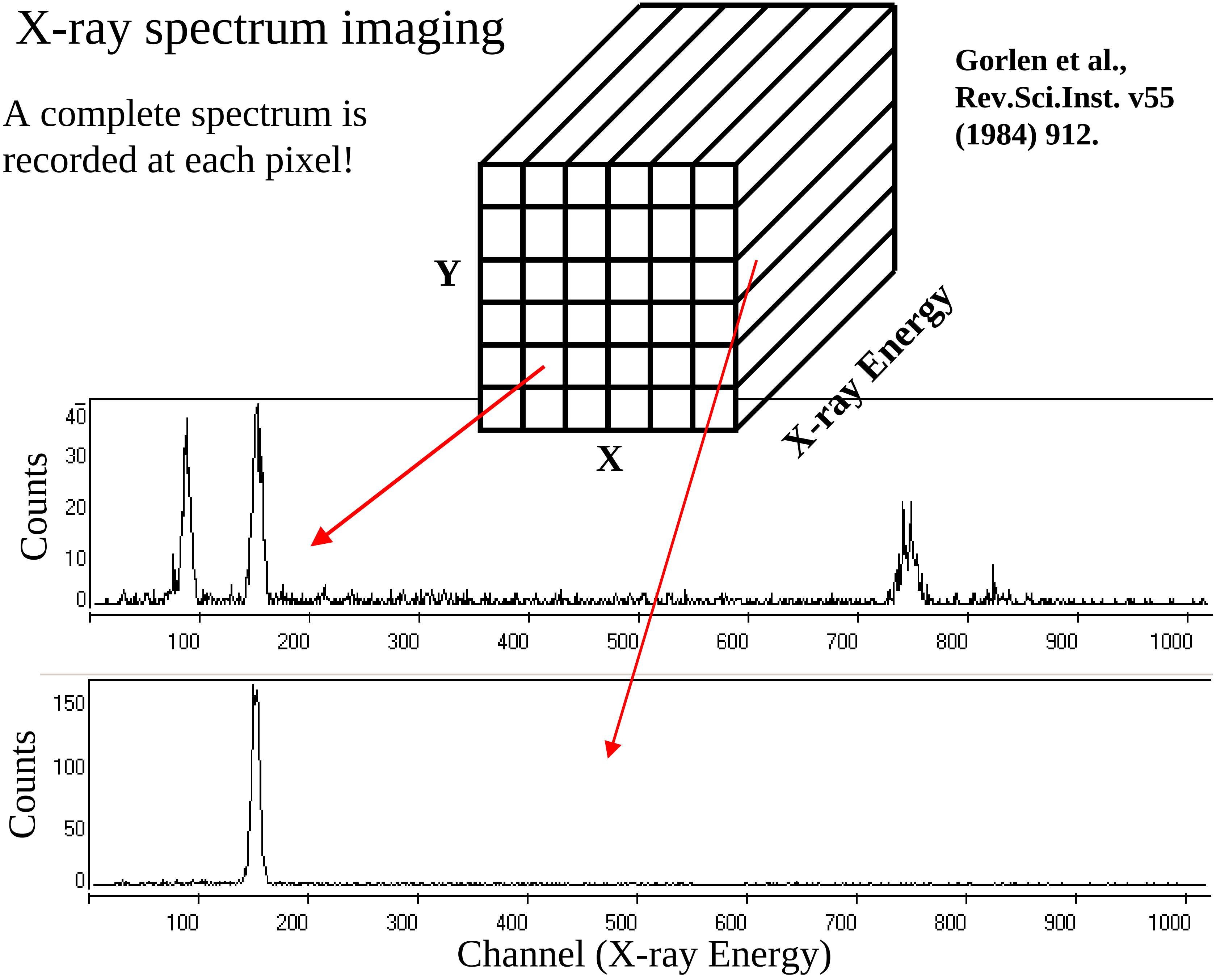
LISPIX "Derived Spectrum" Tools

- **Two views of the x-ray spectrum image (XSI)**
  - **As an array of true spectra, one at each pixel**

X-ray spectrum imaging

A complete spectrum is recorded at each pixel!

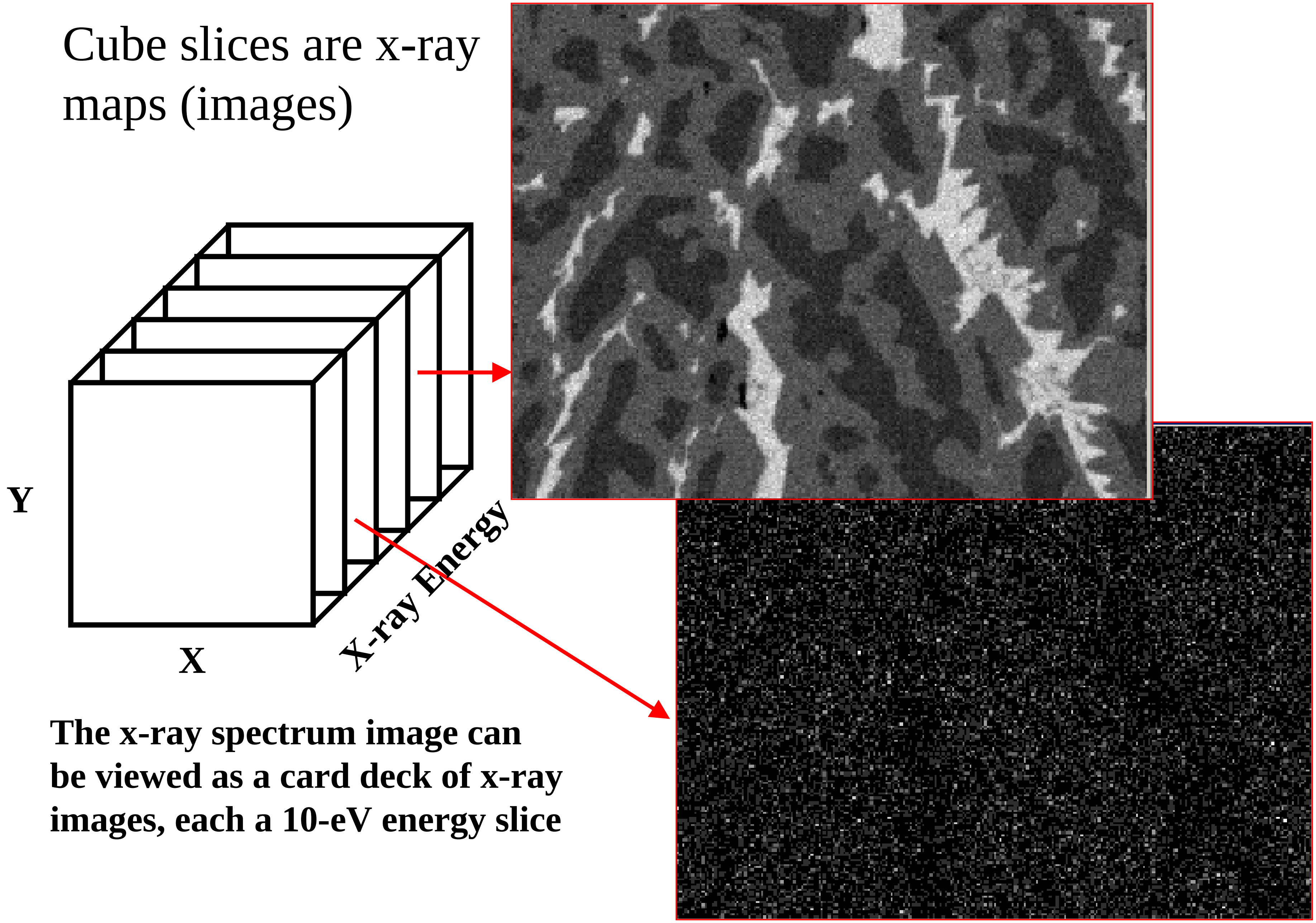
Gorlen et al., Rev.Sci.Inst. v55 (1984) 912.



LISPIX "Derived Spectrum" Tools

- **Two views of the x-ray spectrum image (XSI)**
  - **As an array of true spectra, one at each pixel**
  - **As a "card deck" of x-ray images, each 10-eV wide**

Cube slices are x-ray maps (images)



The x-ray spectrum image can be viewed as a card deck of x-ray images, each a 10-eV energy slice

LISPIX “Derived Spectrum” Tools

- **Two views of the x-ray spectrum image (XSI)**
  - **As an array of true spectra, one at each pixel**
  - **As a “card deck” of x-ray images, each 10-eV wide**
- **Basis of Lispix approach: “derived spectra” SUM Spectrum**
  - **Add all counts on a card to find intensity for that keV**

Derived x-ray spectra are calculated from measured spectra within the cube.

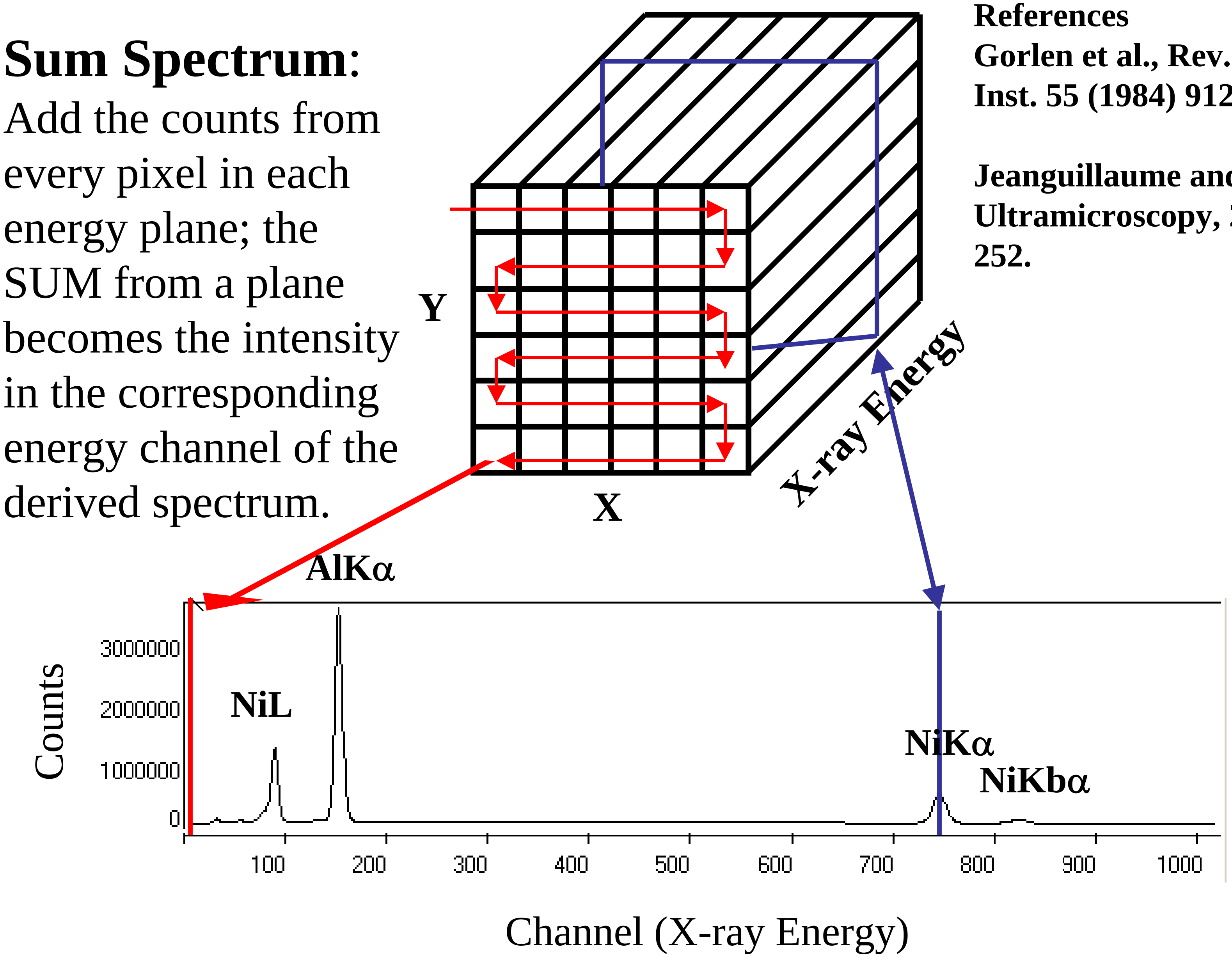
Sum Spectrum:

Add the counts from every pixel in each energy plane; the SUM from a plane becomes the intensity in the corresponding energy channel of the derived spectrum.

References

Gorlen et al., Rev.Sci. Inst. 55 (1984) 912.

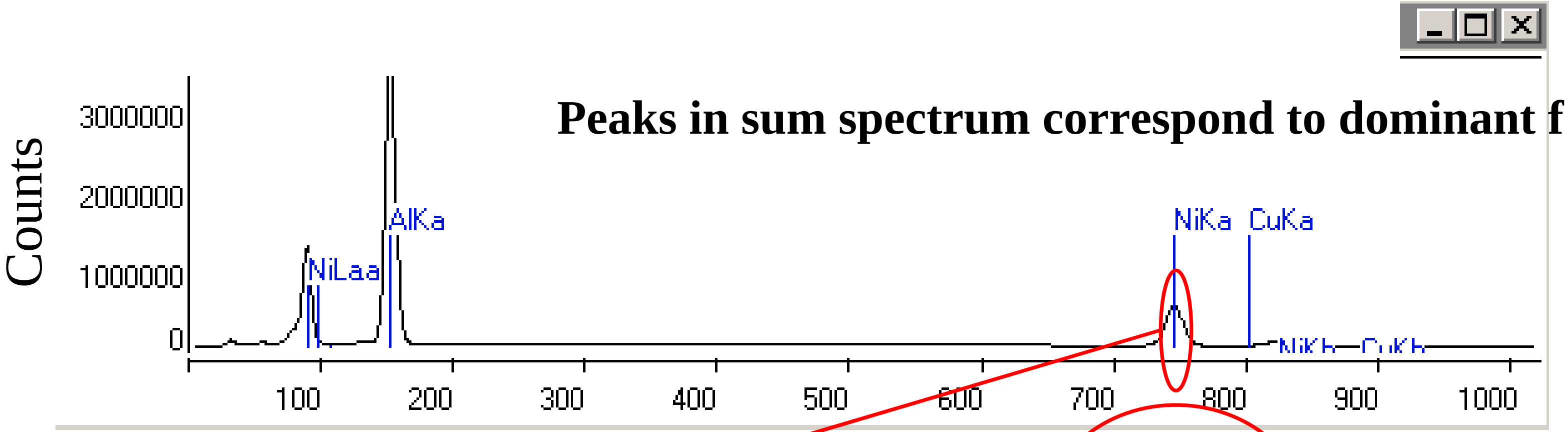
Jeanguillaume and Colliex, Ultramicroscopy, 28 (1989) 252.



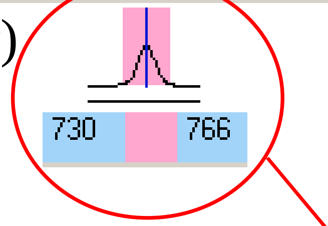
LISPIX “Derived Spectrum” Tools

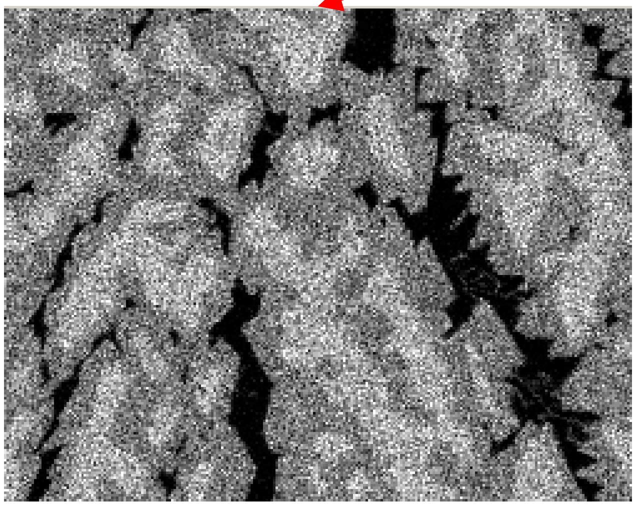
- Two views of the x-ray spectrum image (XSI)
  - As an array of true spectra, one at each pixel
  - As a “card deck” of x-ray images, each 10-eV wide
- **SUM Spectrum**
  - Add all counts on a card to find intensity for that keV
  - **Peaks identify high abundance features**

Peaks in the SUM spectrum correspond to x-ray lines of elements.

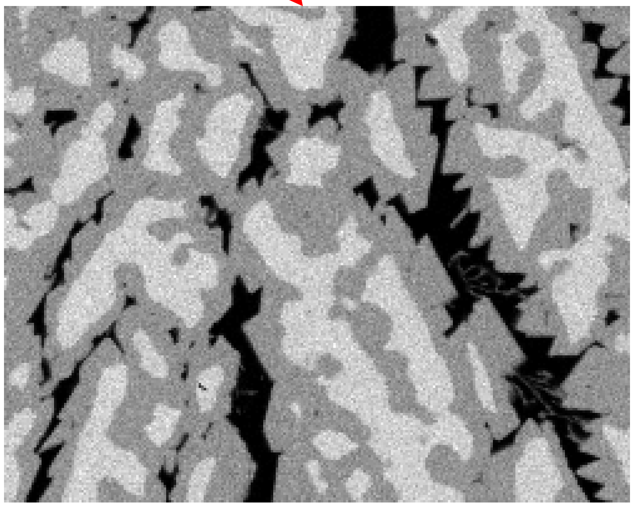


Peaks in sum spectrum correspond to dominant features.





Single ‘slice’ or x-ray channel



Multichannel image for Ni peak.

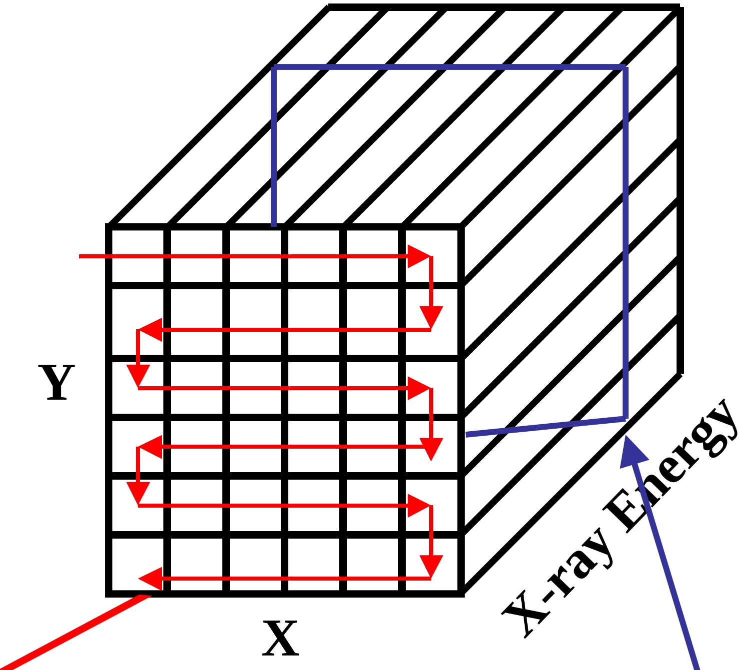
LISPIX constructs the image corresponding to an energy plane or group of energy planes.

Developing Derived Spectrum Tools

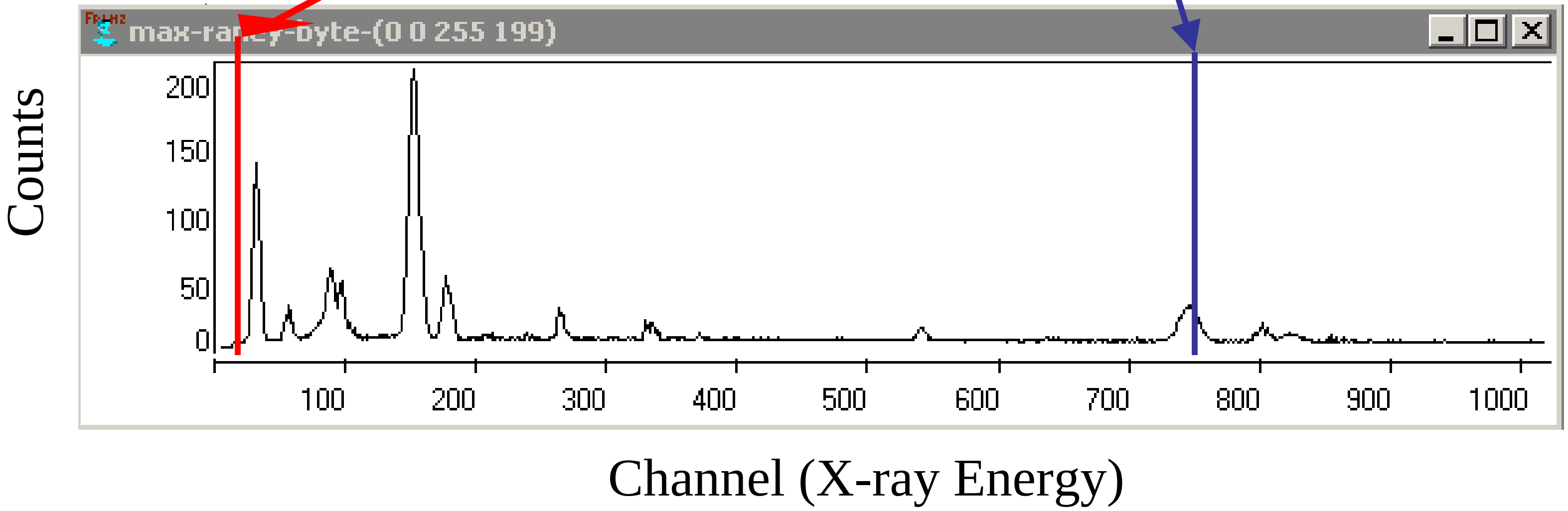
- **SUM spectrum quickly locates spectral features with high abundance**
- **Problem: How do we find rare, <u>unanticipated</u> features in the datacube (200 Mbytes):**
  - **Rare feature may occur only at one pixel. For a 256x200 scan, one pixel represents 1/51,200**
  - **Any element (except H, He, Li) may be of interest**

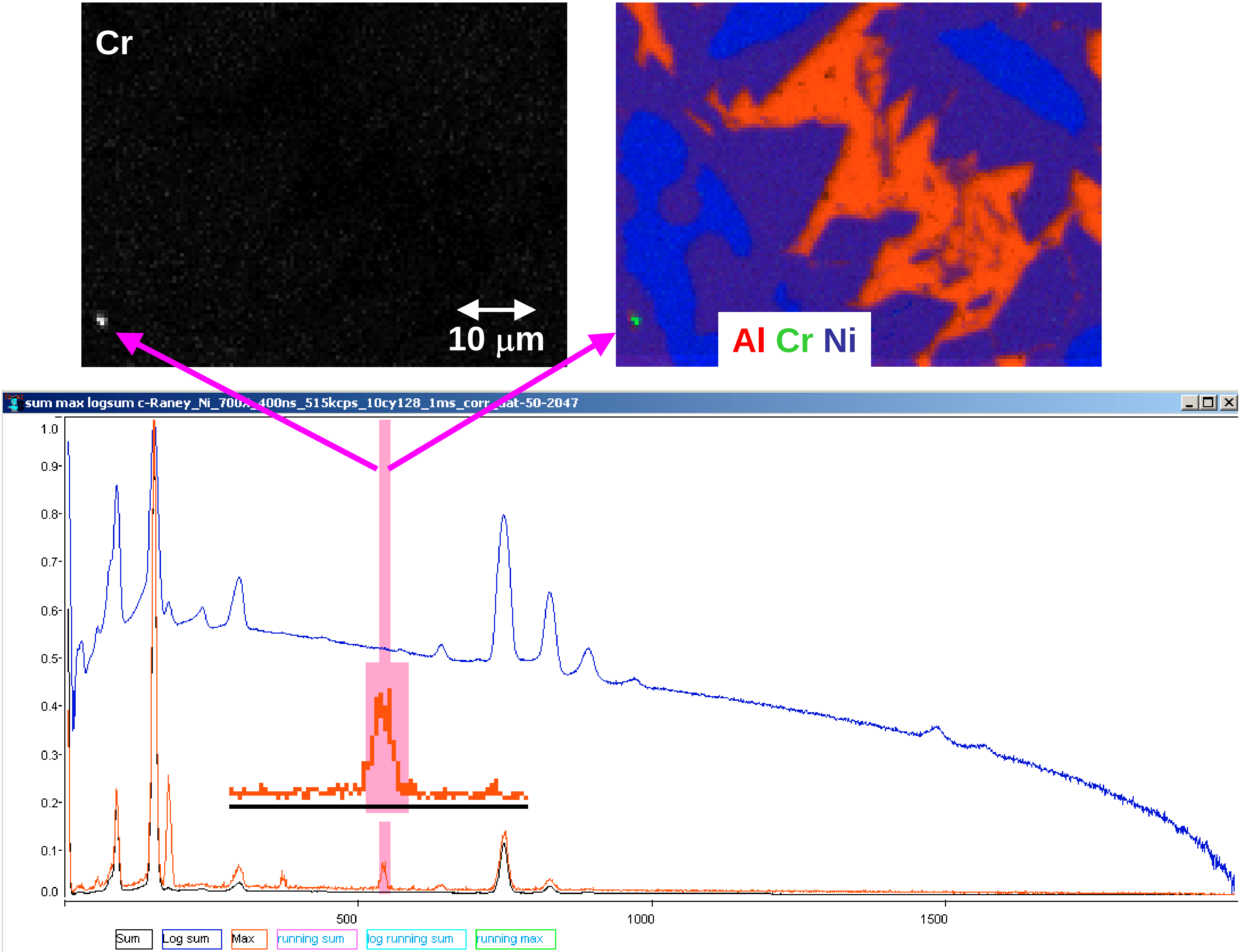
Maximum Pixel Spectrum

Bright and Newbury,
J. Microscopy,
216 (2004) 186



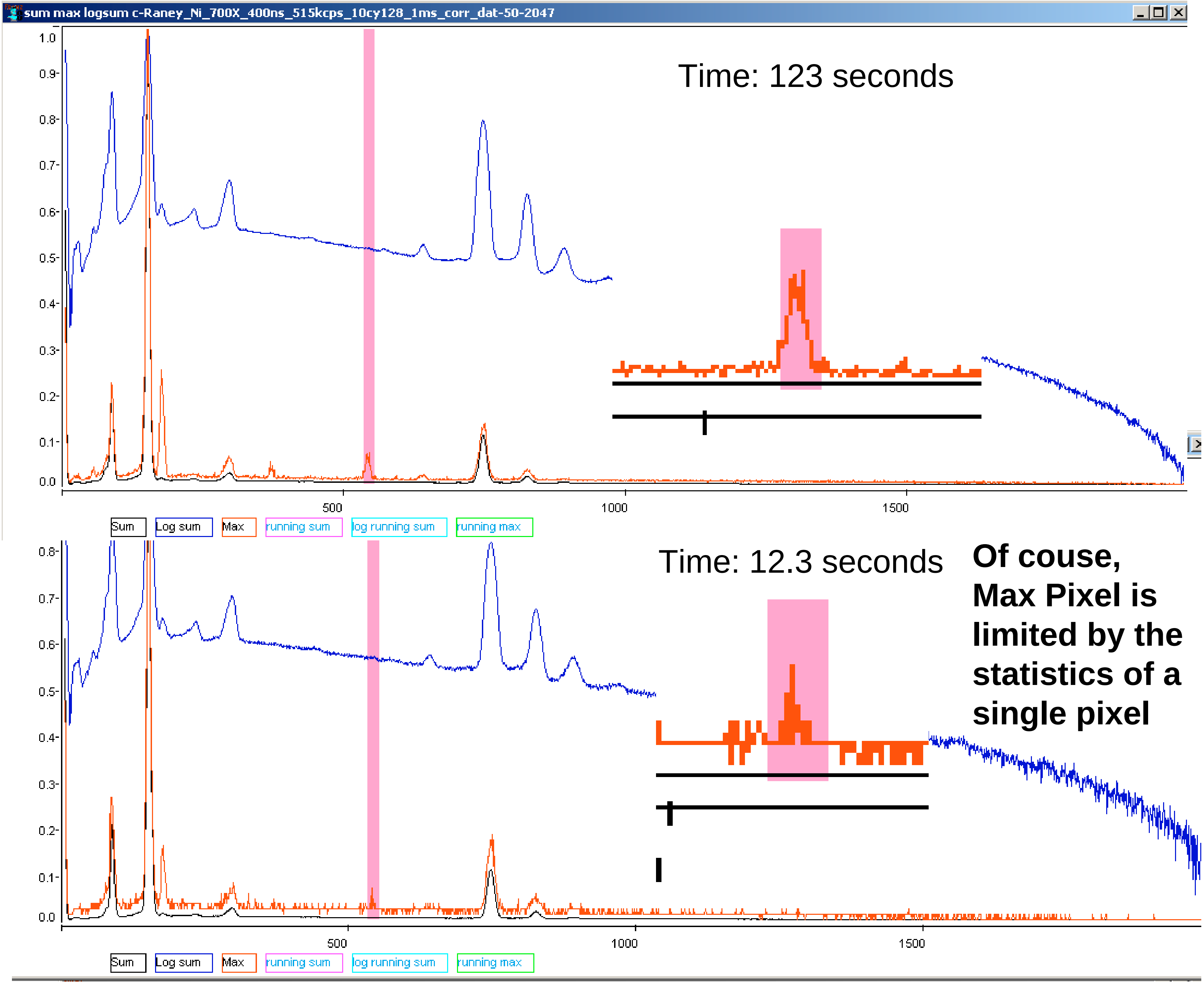
Test every pixel in an energy plane to find the MAXIMUM value. This maximum value becomes the intensity in the corresponding energy channel of the derived Maximum Pixel Spectrum.




Cr
10 µm
Al Cr Ni
sum max logsum c-Raney_Ni_700x_400ns_515kcps_10cy128_1ms_corr_dat-50-2047
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
500
1000
1500
Sum
Log sum
Max
running sum
log running sum
running max

LISPIX “Derived Spectrum” Tools

- Two views of the x-ray spectrum image (XSI)
  - As an array of true spectra, one at each pixel
  - As a “card deck” of x-ray images, each 10-eV wide
- SUM Spectrum
  - Add all counts on a card to find intensity for that keV
  - Peaks identify high abundance features
- **MAX PIXEL Spectrum**
  - **Find maximum value on a card and plot for that keV**
  - **Locates rare, unanticipated features**


sum max logsum c-Raney_Ni_700X_400ns_515kcps_10cy128_1ms_corr_dat-50-2047

Time: 123 seconds

Time: 12.3 seconds

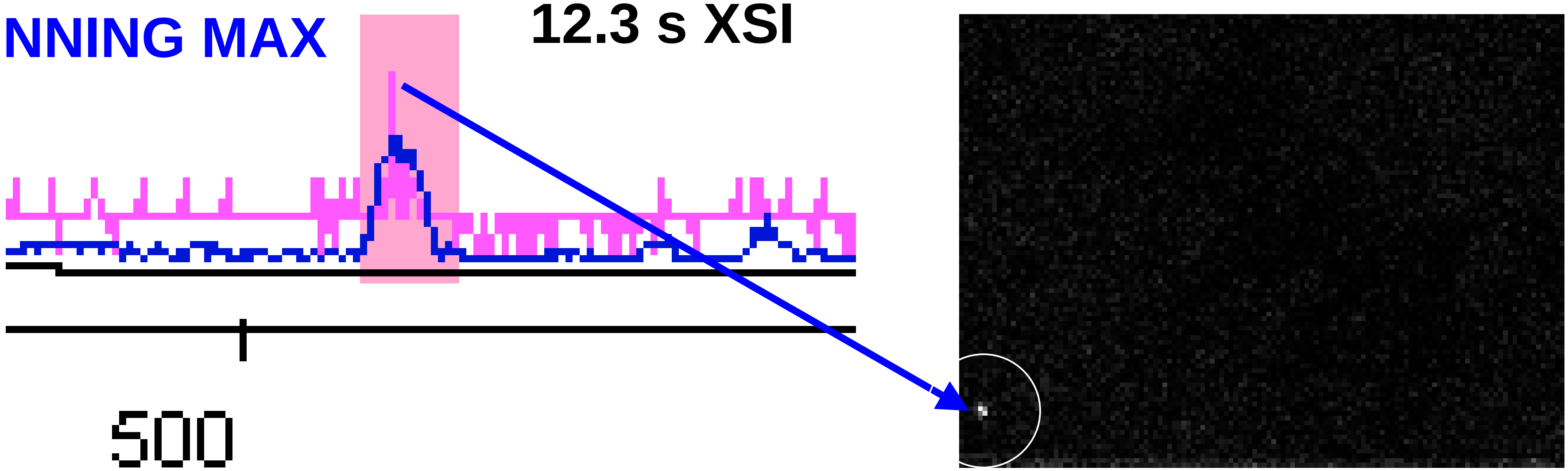
Of couse, Max Pixel is limited by the statistics of a single pixel

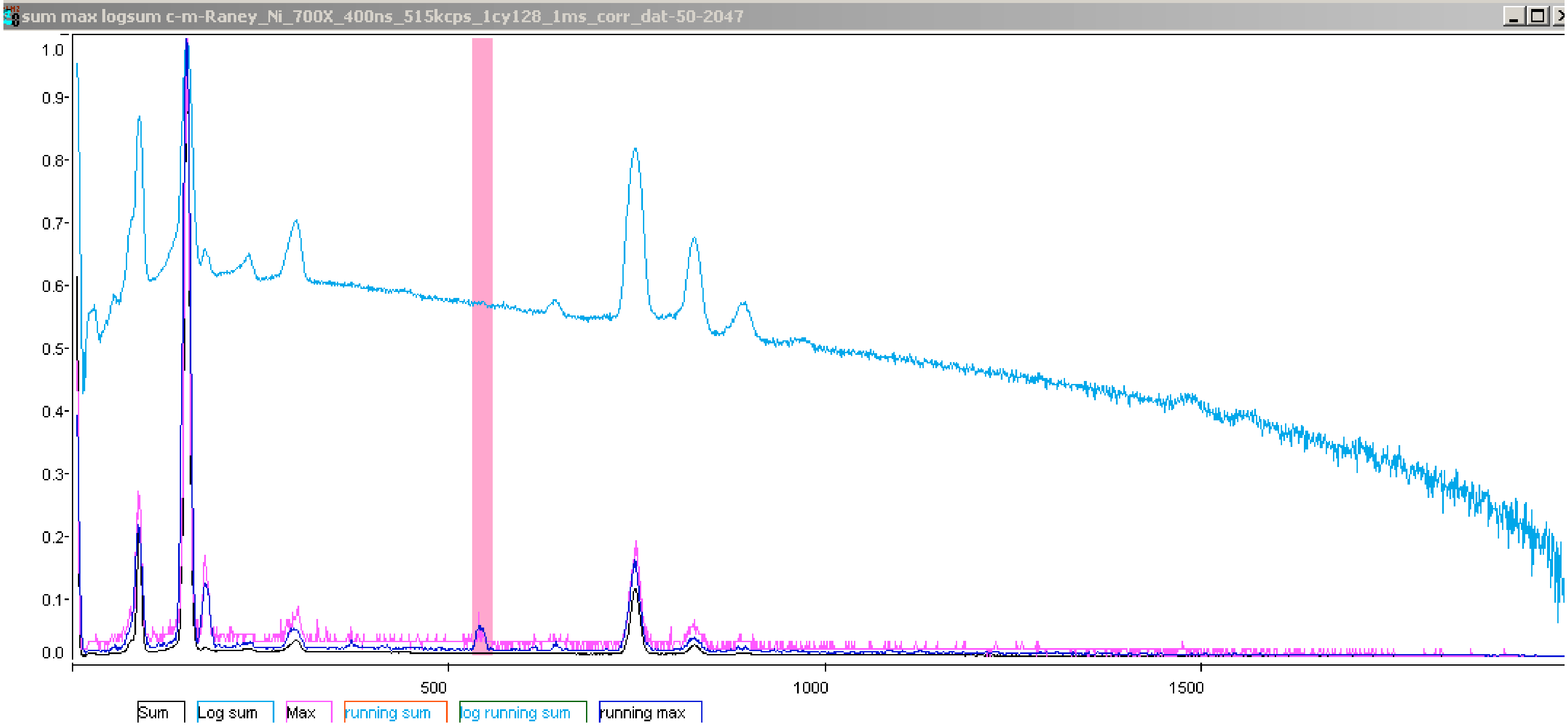
LISPIX “Derived Spectrum” Tools

- **Two views of the x-ray spectrum image (XSI)**
  - **As an array of true spectra, one at each pixel**
  - **As a “card deck” of x-ray images, each 10-eV wide**
- **SUM Spectrum**
  - **Add all counts on a card to find intensity for that keV**
  - **Peaks identify high abundance features**
- **MAX PIXEL Spectrum**
  - **Find maximum value on a card and plot for that keV**
  - **Locates rare, unanticipated features**
  - **May not be sensitive to dilute constituents**
- **RUNNING MAX Spectrum**
  - **First average over n-m to n+m cards (e.g., m = 3) to create new $n_{average}$ card**
  - **Then perform maximum value search on $n_{average}$ card**

RUNNING MAX

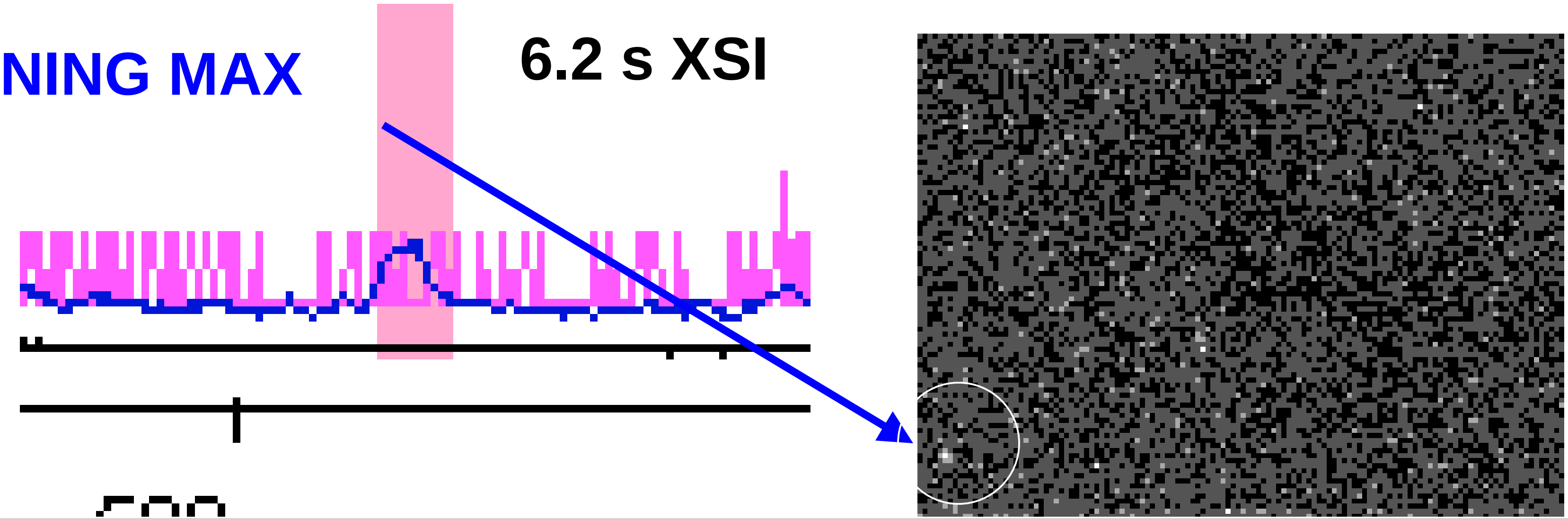
12.3 s XSI

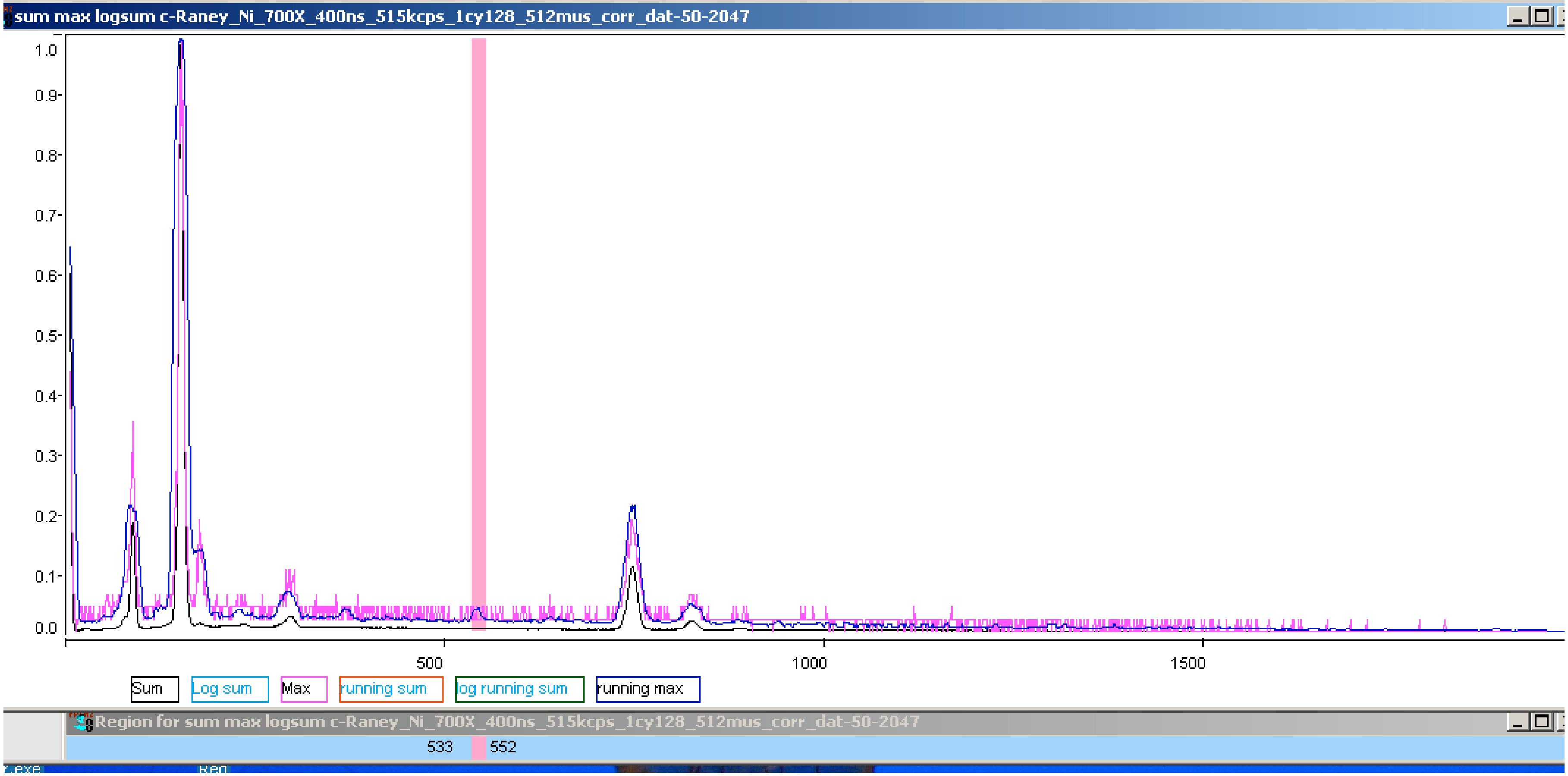




RUNNING MAX

6.2 s XSI





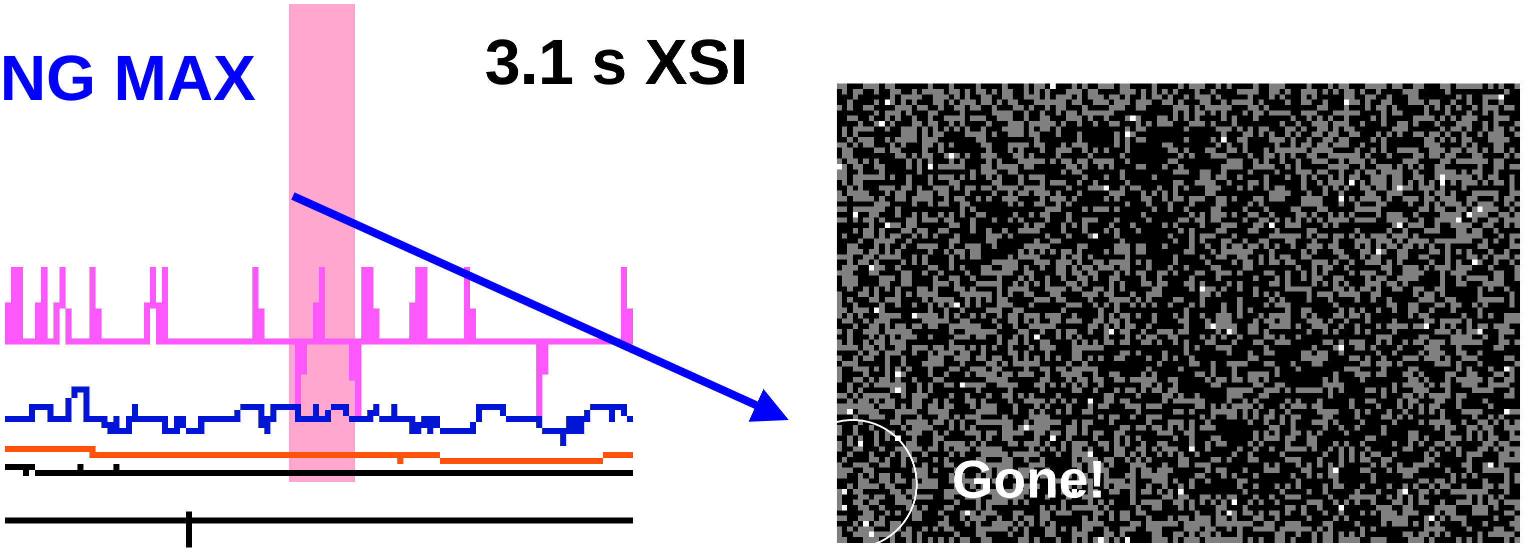
sum max logsum c-Raney_Ni_700X_400ns_515kcps_1cy128_512mus_corr_dat-50-2047

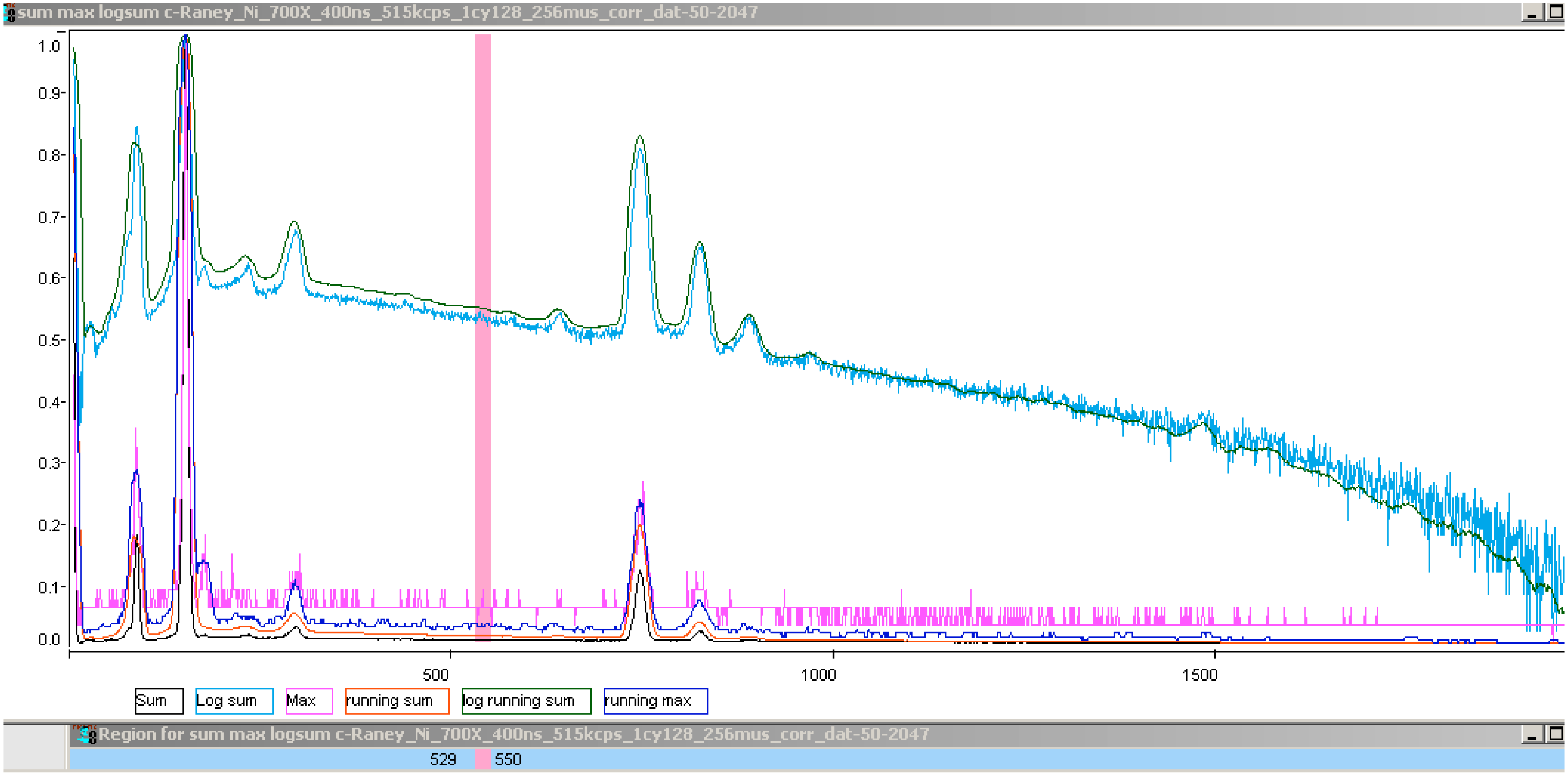
Region for sum max logsum c-Raney_Ni_700X_400ns_515kcps_1cy128_512mus_corr_dat-50-2047

533 552

RUNNING MAX

3.1 s XSI





LISPIX “Derived Spectrum” Tools

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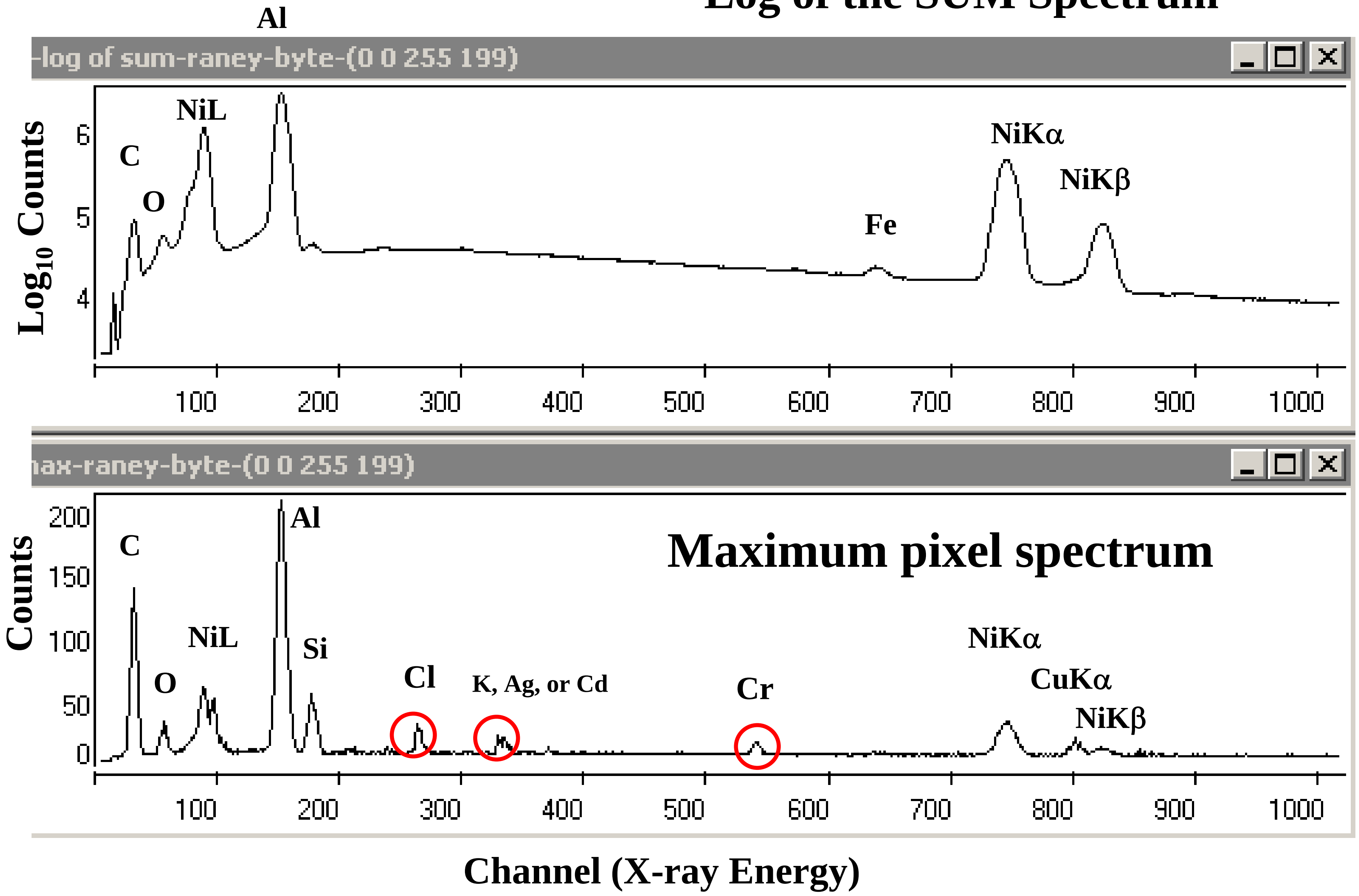
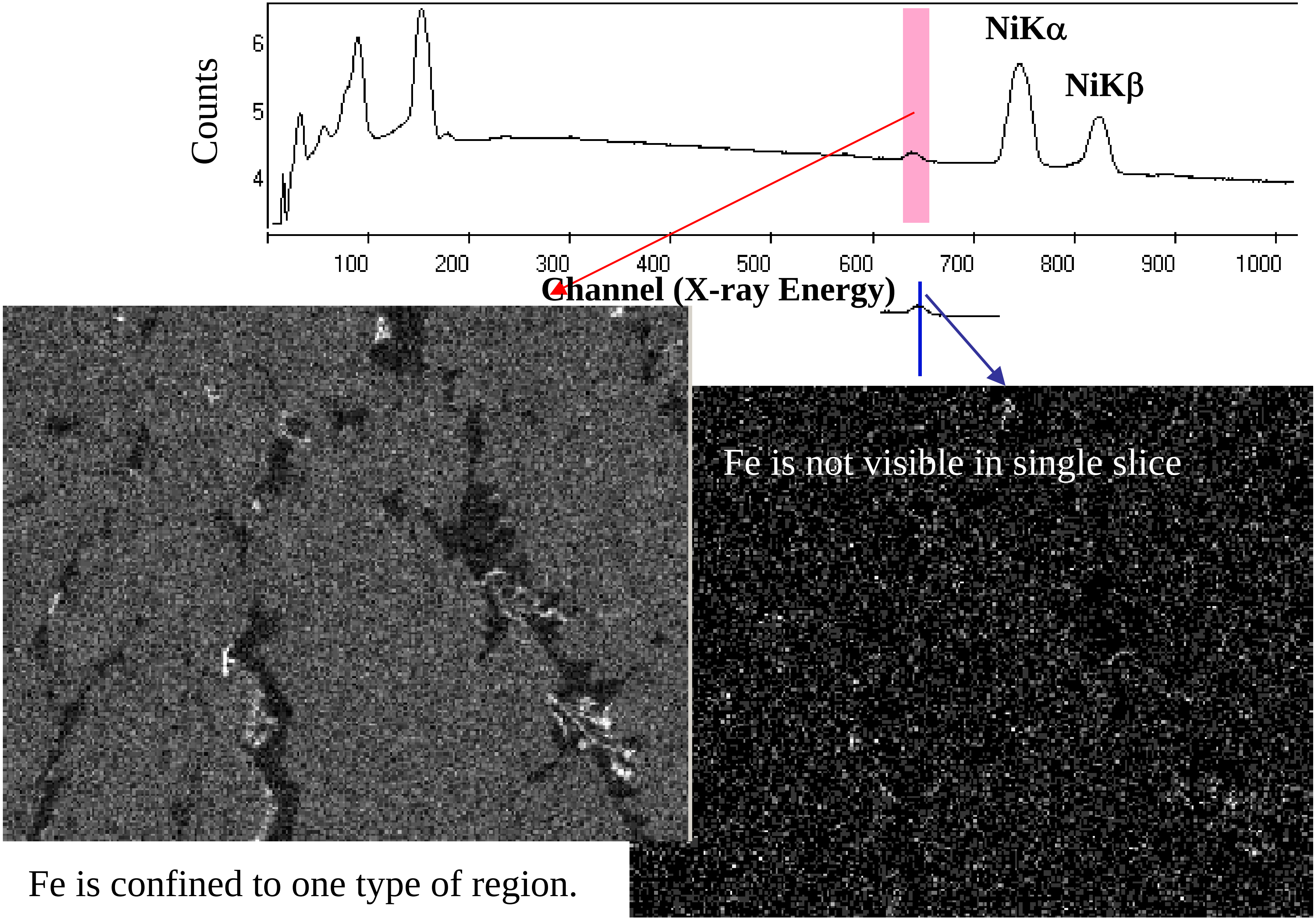
  - **As an array of true spectra, one at each pixel**
  - **As a “card deck” of x-ray images, each 10-eV wide**
- **SUM Spectrum**
  - **Add all counts on a card to find intensity for that keV**
  - **Peaks identify high abundance features**
- **MAX PIXEL Spectrum**
  - **Find maximum value on a card and plot for that keV**
  - **Locates rare, unanticipated features**
  - **May not be sensitive to dilute constituents**

Log of the SUM Spectrum



Finding dilute iron with the Log of the Sum Spectrum



Fe is not visible in single slice

Fe is confined to one type of region.

Developing Strategy for Rapid Mapping

- **How many counts are “enough” to establish visibility for an object in a compositional map?**

Developing Strategy for Rapid Mapping

- **How many counts are "enough" to establish visibility for an object in a compositional map?**
- **The answer depends on several factors:**

1. The concentration, C (mass fraction)

2. The characteristic x-ray energy

  a. Overvoltage, $U_0 = E_0/E_c$ $I_{ch} \sim (U_0 - 1)^n$

  b. Specimen self-absorption $I/I_0 = \exp[-(\mu/\rho)\,\rho\, s]$

  c. X-ray detector efficiency

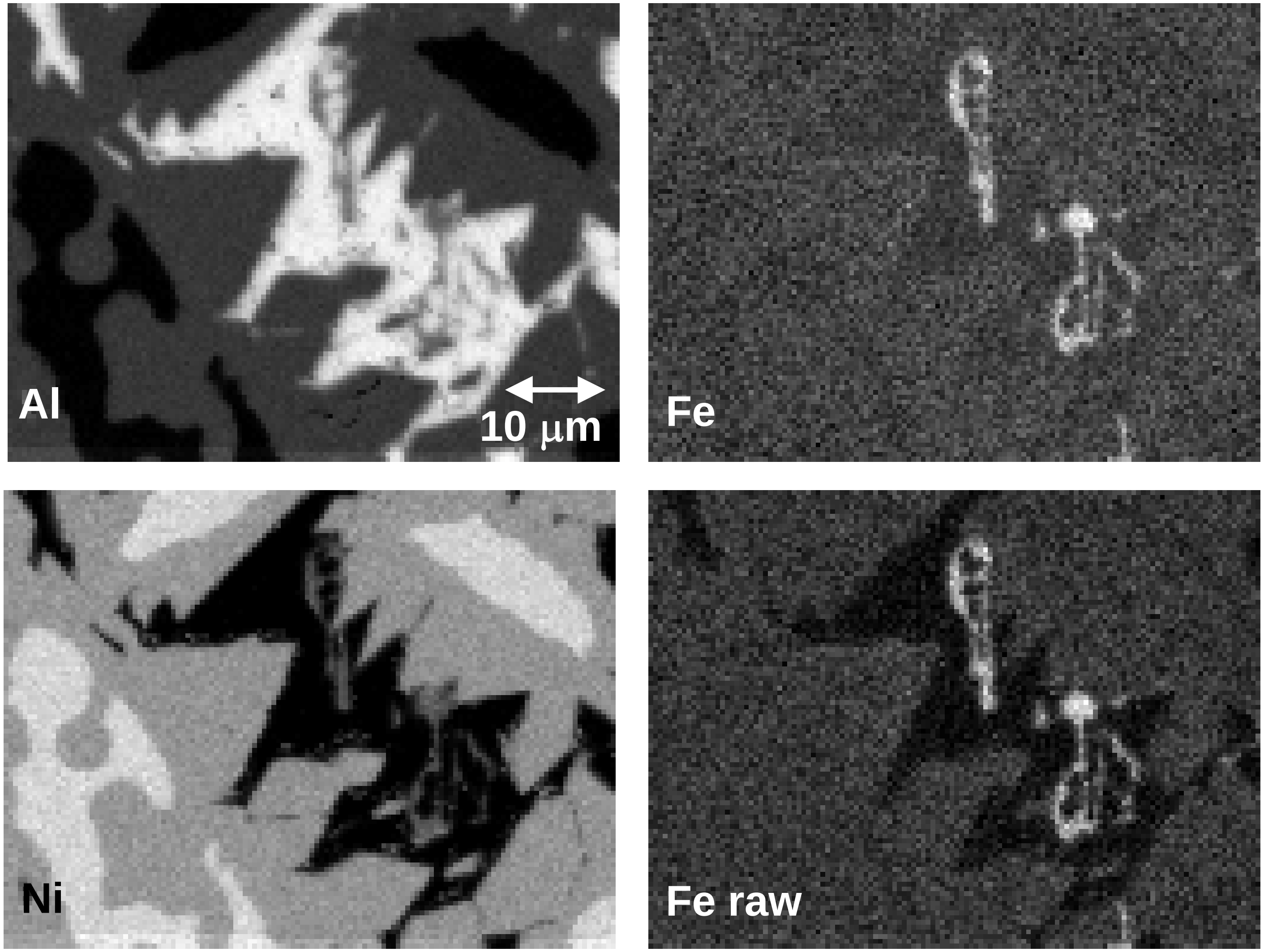
3. X-ray background (continuum) $I_{cm} \sim Z\,(U - 1)$

4. The level of compositional contrast, e.g. vs. Bkg or vs. a different concentration.

128x96 1 ms dwell, 10 frames 123 s total 550kHz OCR

Al_{max}=3846

Fe_{max}=93



Background
Corrected;
Fe Contrast
vs
background

Ni_{max}=662

Phases

Al 99.5 Ni 0.5

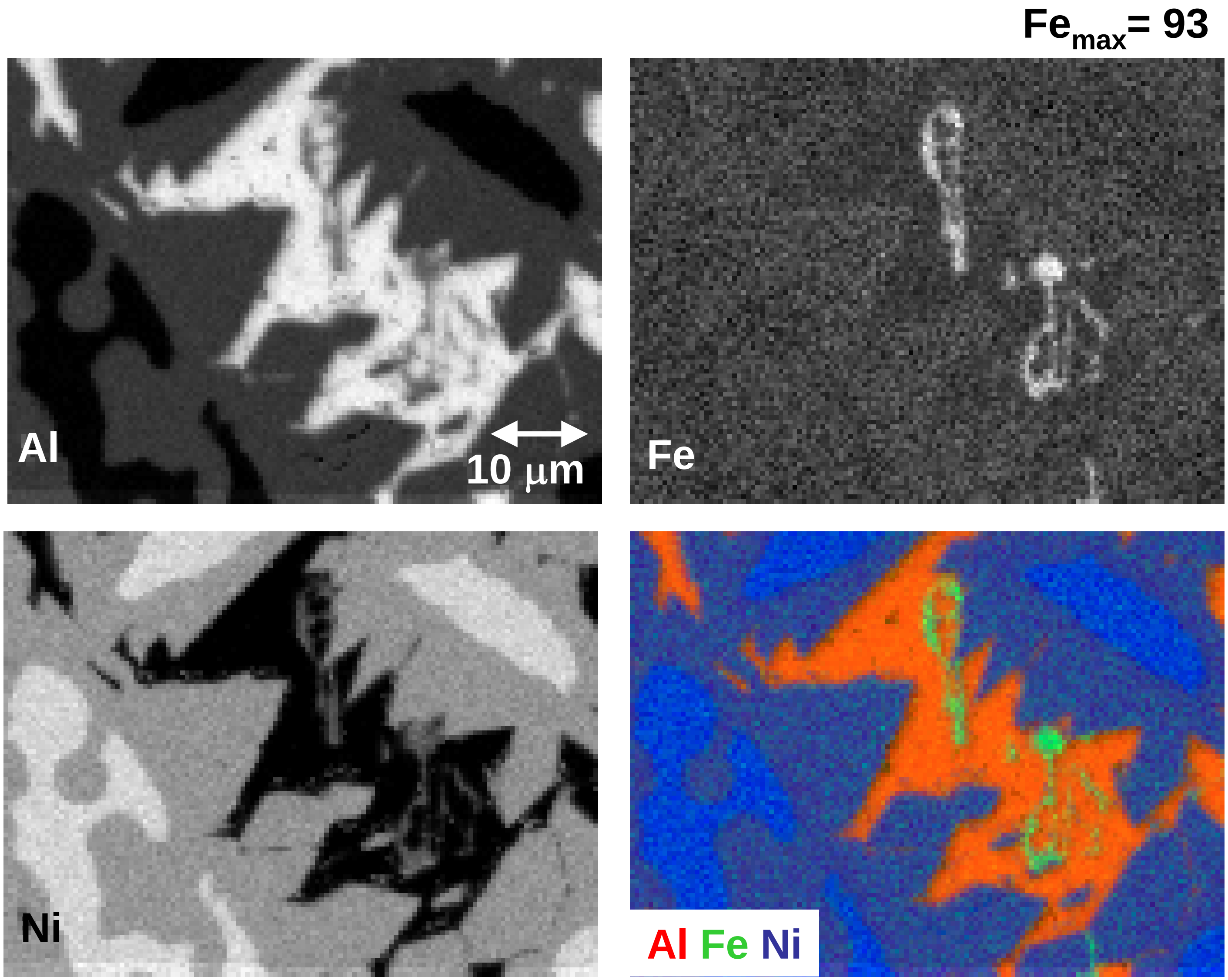
Al 71.2 Ni 24.6 Fe 4.2

Al 60 Ni 40

Al 46.5 Ni 53.5

This dose gives sufficient Fe counts to perform a background correction.

128x96 1 ms dwell, 10 frames 123 s total 550kHz OCR



Phases

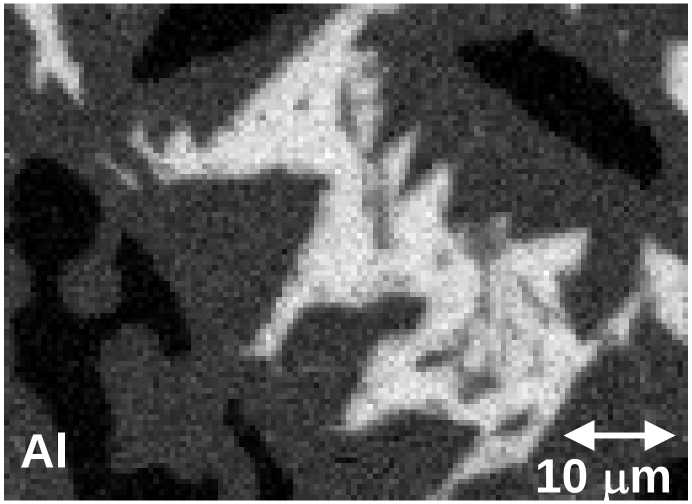
Al 99.5 Ni 0.5

Al 71.2 Ni 24.6 Fe 4.2

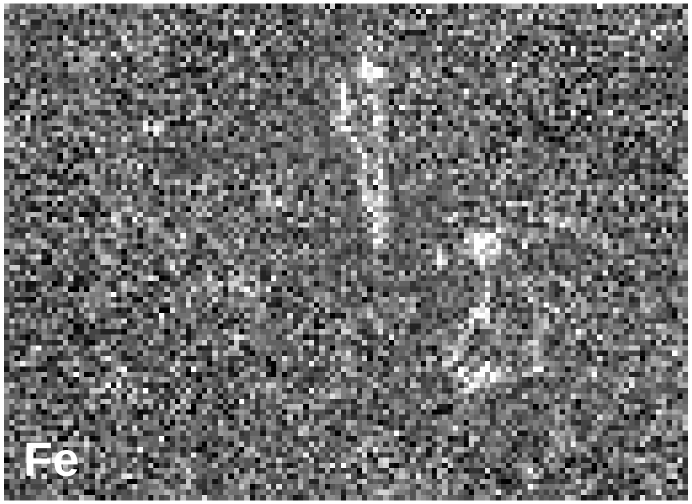
Al 60   Ni 40

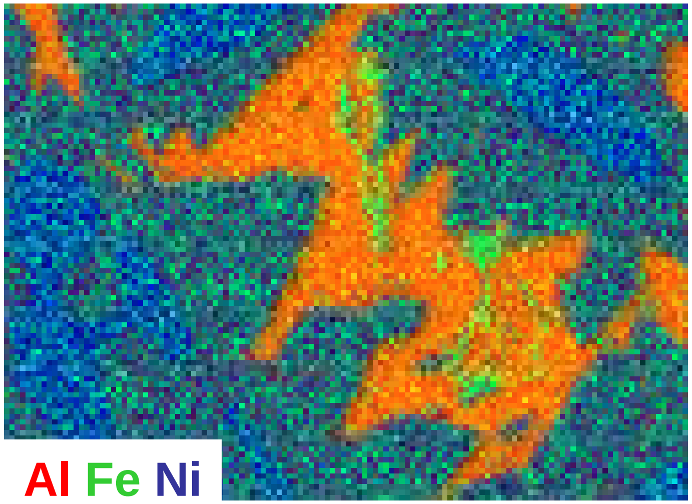
Al 46.5 Ni 53.5

Reduce dose by 10; 128x96 1 ms dwell, 1 frame 12.3 s total 550kHz OCR



$\mathbf{Fe_{max} = 9}$





Phases

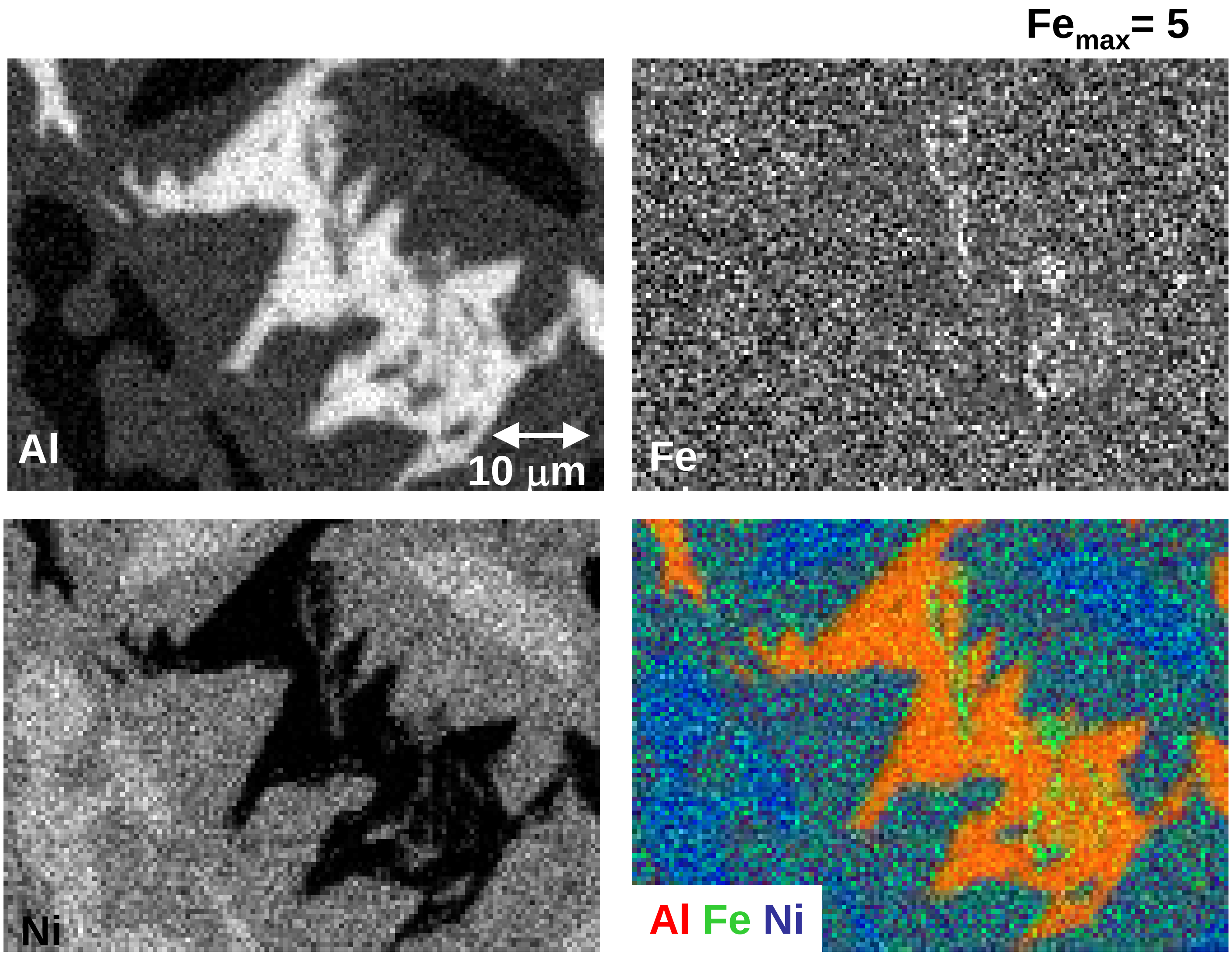
Al 99.5 Ni 0.5

Al 71.2 Ni 24.6 Fe 4.2

Al 60   Ni 40

Al 46.5 Ni 53.5

128x96 512 μs dwell, 1 frame 6.2 s total 550kHz OCR

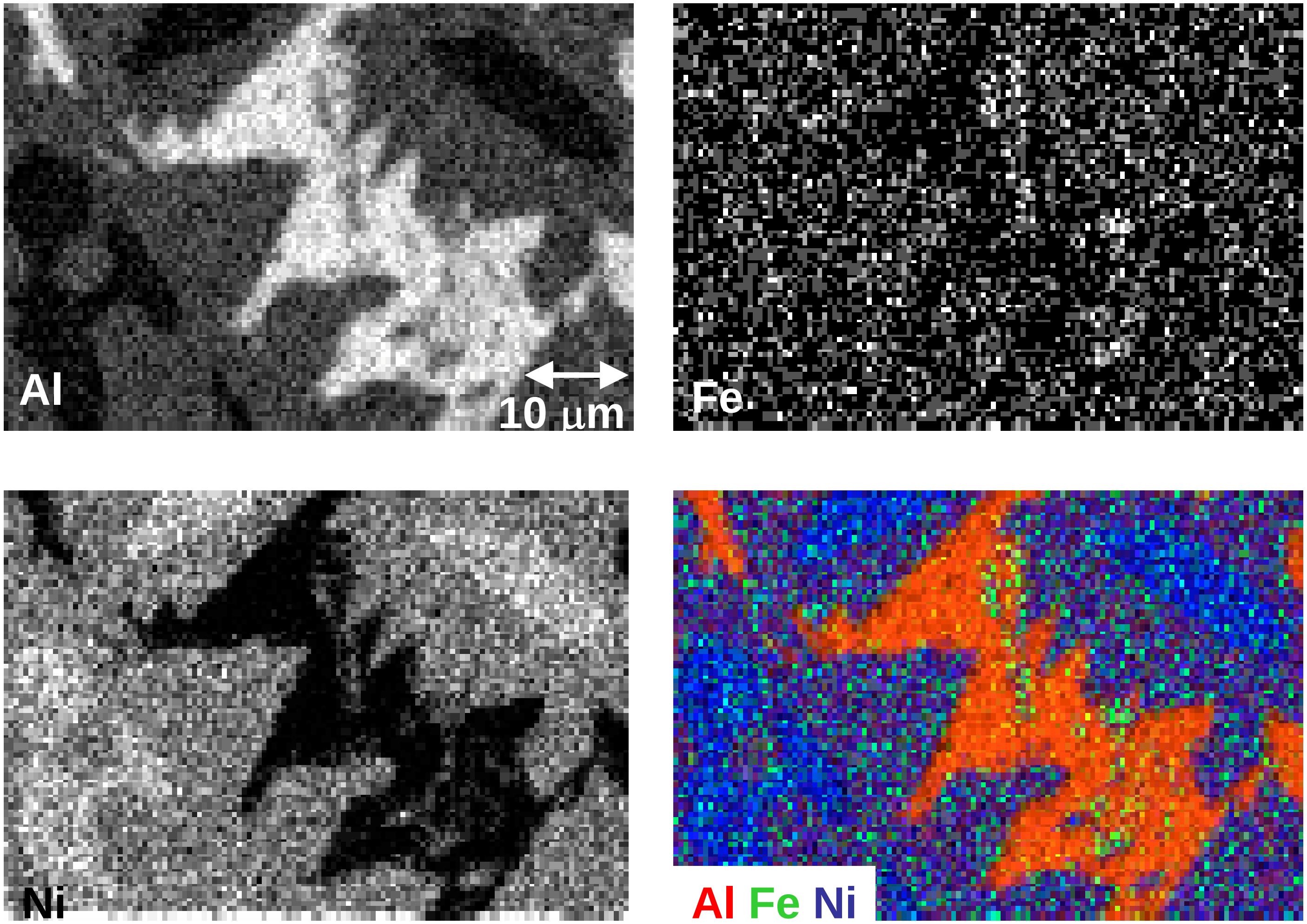


Phases

Al 99.5 Ni 0.5	**Al 60 Ni 40**
Al 71.2 Ni 24.6 Fe 4.2	**Al 46.5 Ni 53.5**

128x96 256 μs dwell, 1 frame 3.1 s total 550kHz OCR

$Fe_{max}= 3$



Phases

Al 99.5 Ni 0.5

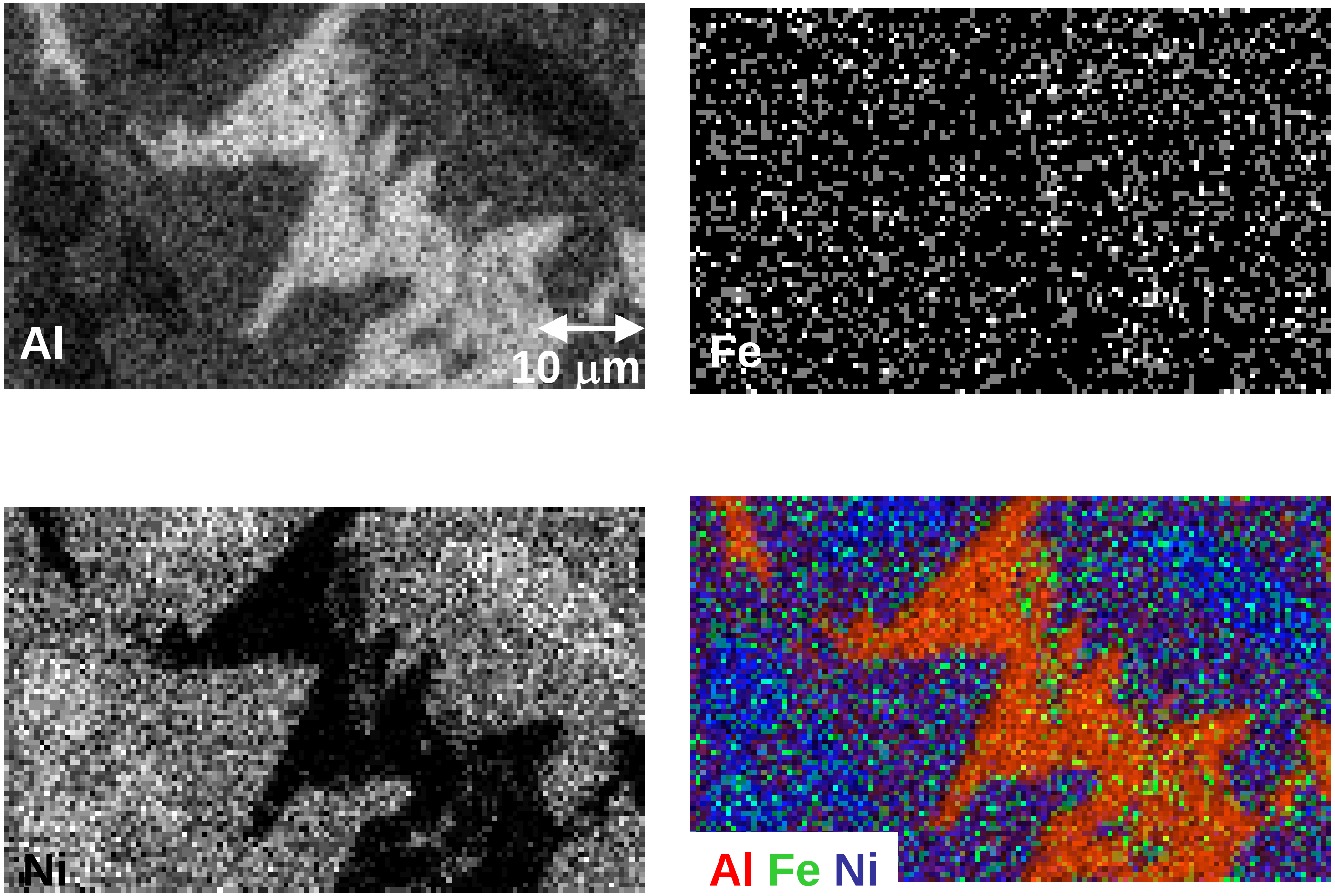
Al 71.2 Ni 24.6 Fe 4.2

Al 60 Ni 40

Al 46.5 Ni 53.5

128x96 128 μs dwell, 1 frame 1.6 s total 550kHz OCR

$\mathrm{Fe}_{\mathrm{max}}$= 2



Phases

Al 99.5 Ni 0.5	**Al 60 Ni 40**
Al 71.2 Ni 24.6 Fe 4.2	**Al 46.5 Ni 53.5**

Developing Strategy for Rapid Mapping

- **How many counts are "enough" to establish visibility for an object in a compositional map?**
- **This issue is closely related to the classic Rose (1948) criterion for contrast visibility threshold in scanned images. For an object relative to a featureless background, Rose requires the signal to exceed the noise by a factor ("Rose factor", RF) of at least 5:**

Signal = $(n_2 – n_1) > RF * \sigma_n$ (noise) = $RF\ n_1^{1/2}$

Consider the case of a compositional feature containing element A viewed against a featureless background that does not contain element A

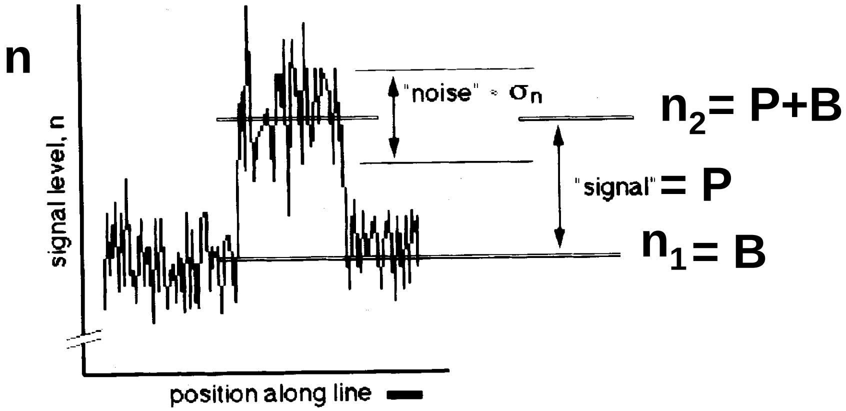
For x-rays, $n_1 = B$ (continuum) while $n_2 = P$ (characteristic) + B

Signal = $(n_2 – n_1) = (P+B – B) = P > RF\ B^{1/2}$

$P/B > RF\ B^{1/2}/B$

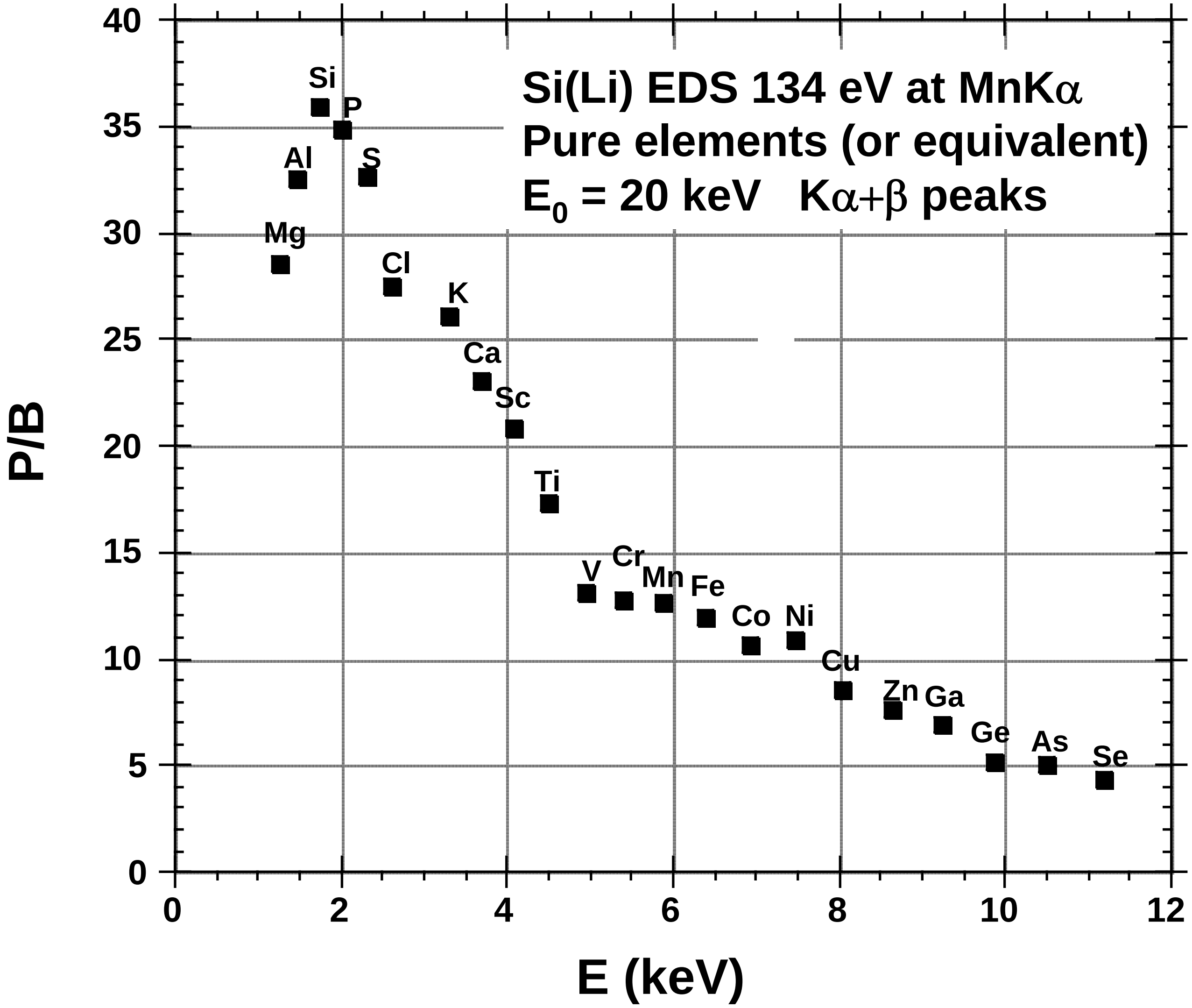
$P/B > RF/B^{1/2}$

$P/B > 5/B^{1/2}$

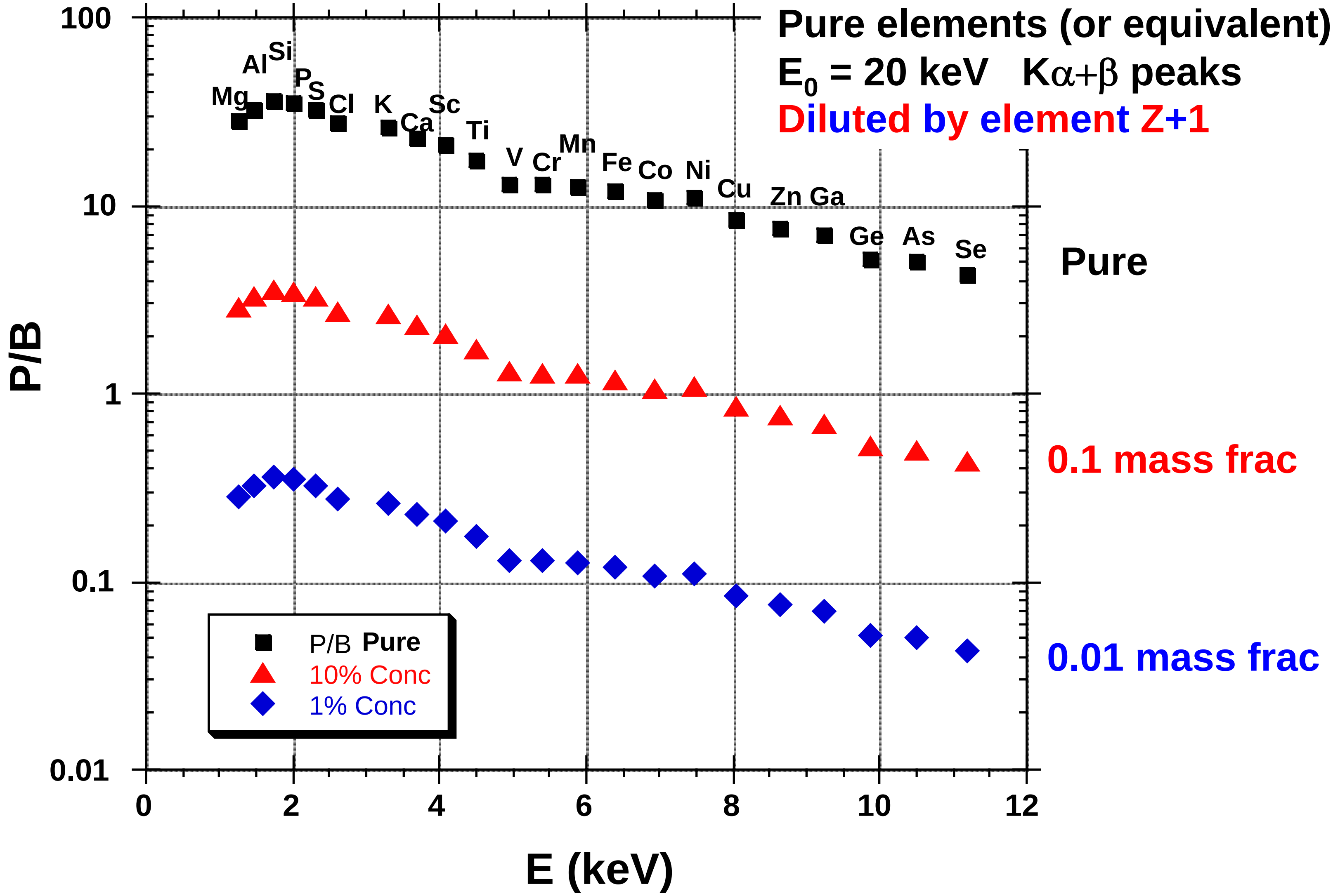


For an element localized in one phase

Measurements of Peak-to-Background (P/B)



Measured and Estimated of Peak-to-Background (P/B)

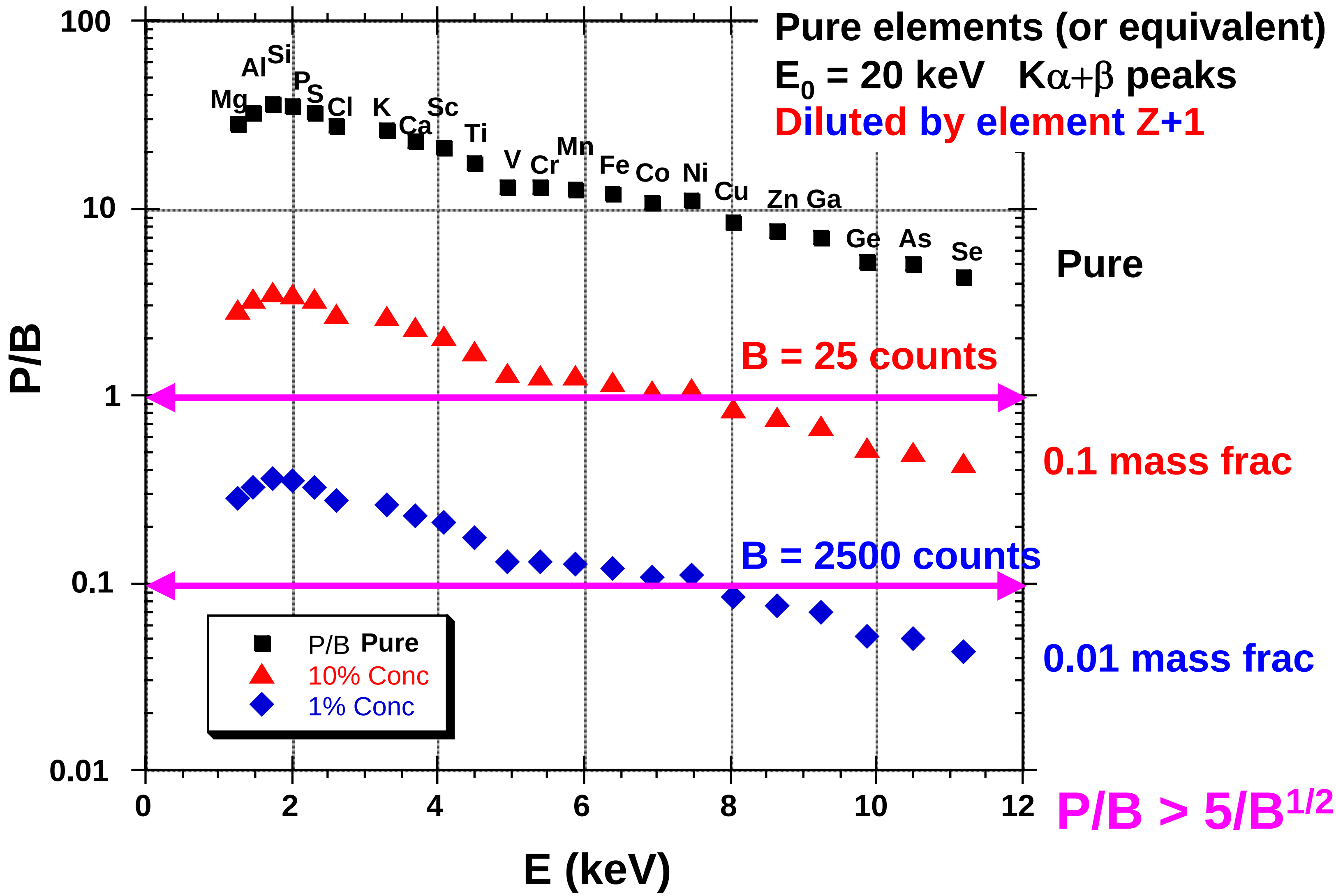


Pure elements (or equivalent)

E_0 = 20 keV Kα+β peaks

Diluted by element Z+1

Measured and Estimated of Peak-to-Background (P/B)



Pure elements (or equivalent)

E_0 = 20 keV Kα+β peaks

Diluted by element Z+1

$P/B > 5/B^{1/2}$

Developing Strategy for Rapid Mapping

- **How many counts are "enough" to establish visibility for an object in a compositional map?**
- **The answer depends on several factors:**

1. The concentration, C (mass fraction)

2. The characteristic x-ray energy

a. Overvoltage, $U_0 = E_0/E_c$ $I_{ch} \sim (U_0 - 1)^n$

b. Specimen self-absorption $I/I_0 = \exp[-(\mu/\rho)\,\rho\,s]$

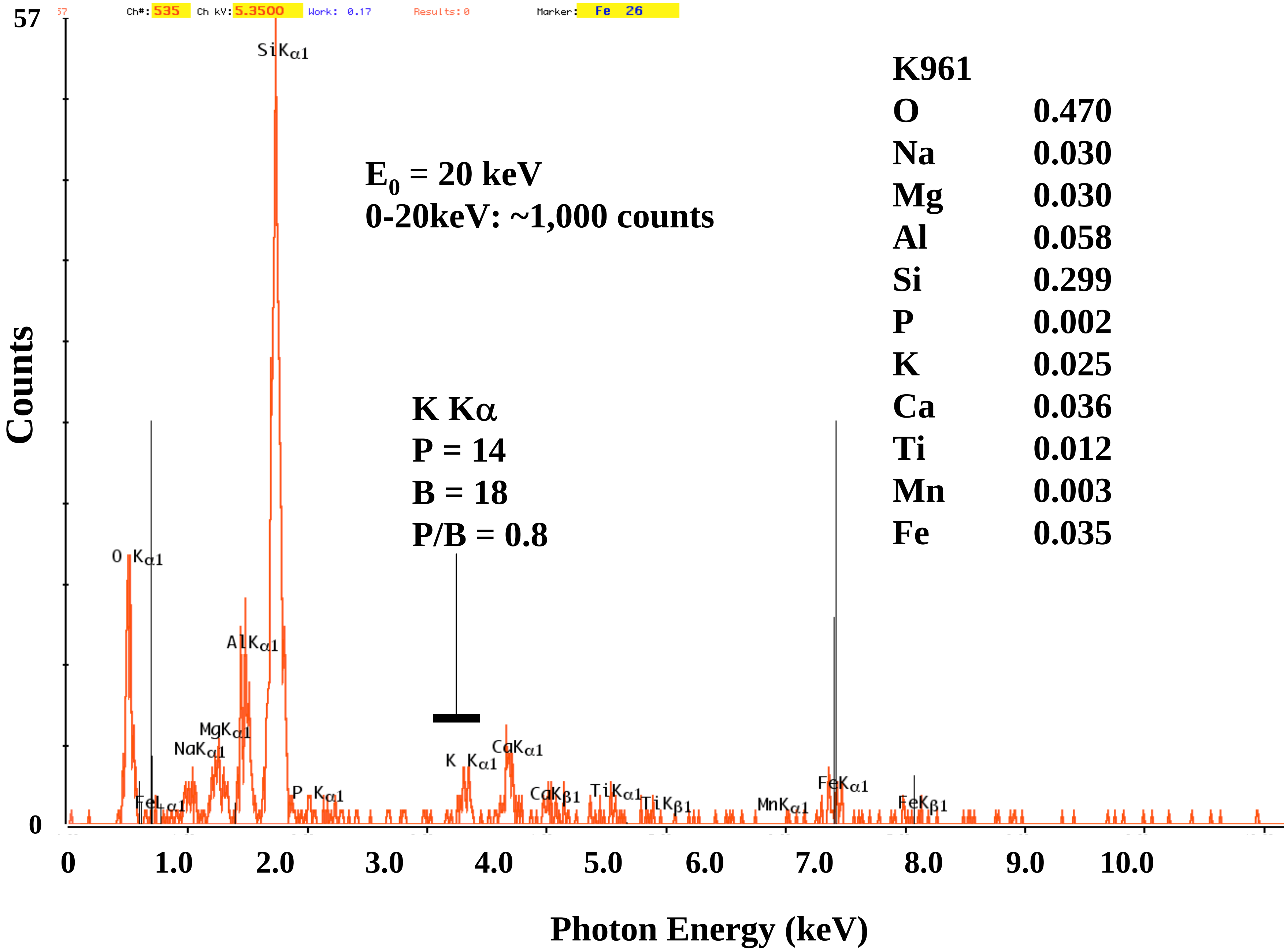
c. X-ray detector efficiency

3. X-ray background (continuum) $I_{cm} \sim Z\,(U - 1)$

4. The level of compositional contrast, e.g. vs. B or vs. a different concentration.

- **A compositional image simulator is needed:**
  - **DTSA for spectral details, such as P/B (soon NIST Monte)**
  - **LISPIX to synthesis and process the images for display**

Desktop Spectrum Analyzer (DTSA) Simulation



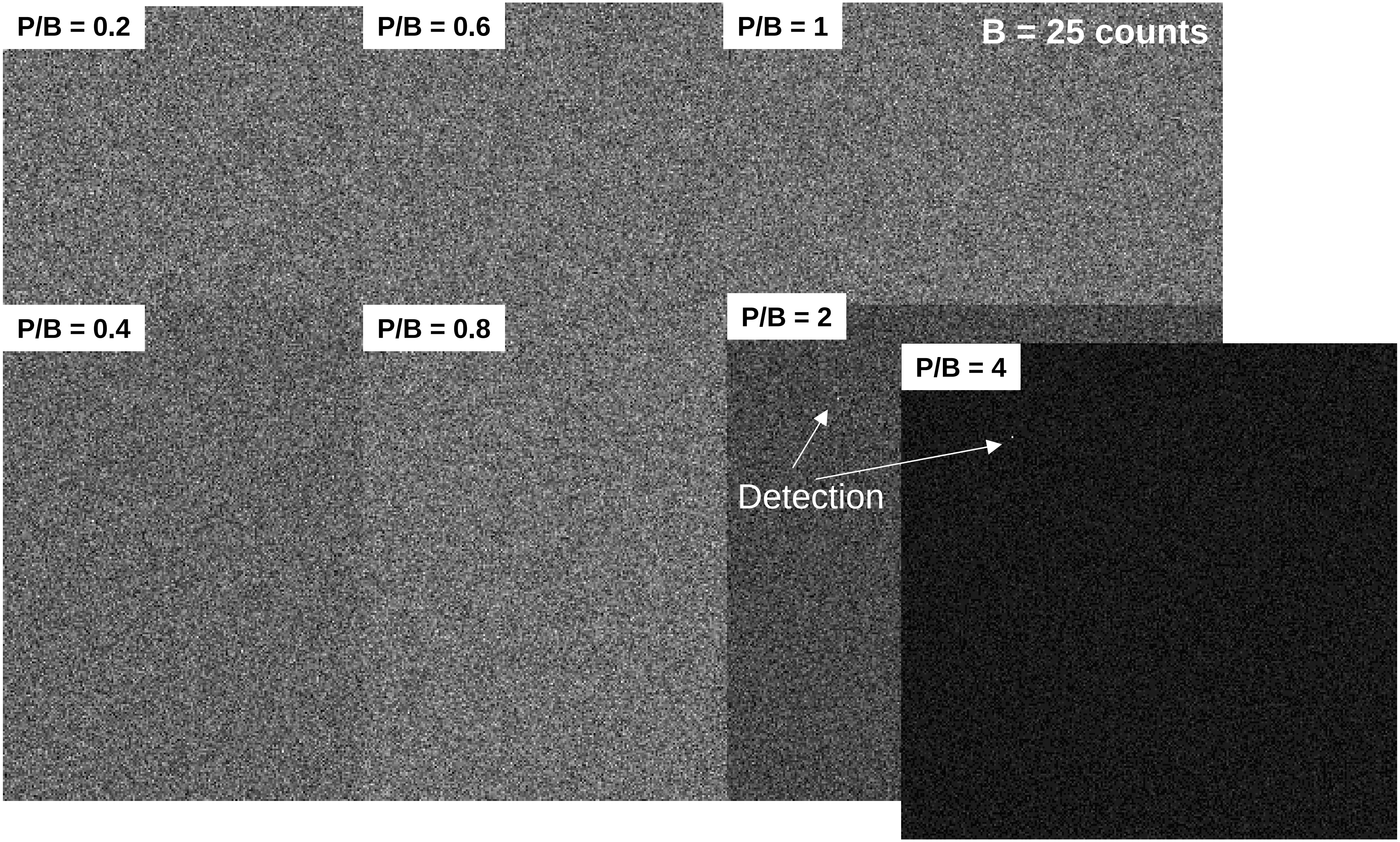
E_0 = 20 keV
0-20keV: ~1,000 counts

K Kα
P = 14
B = 18
P/B = 0.8

K961	
O	0.470
Na	0.030
Mg	0.030
Al	0.058
Si	0.299
P	0.002
K	0.025
Ca	0.036
Ti	0.012
Mn	0.003
Fe	0.035

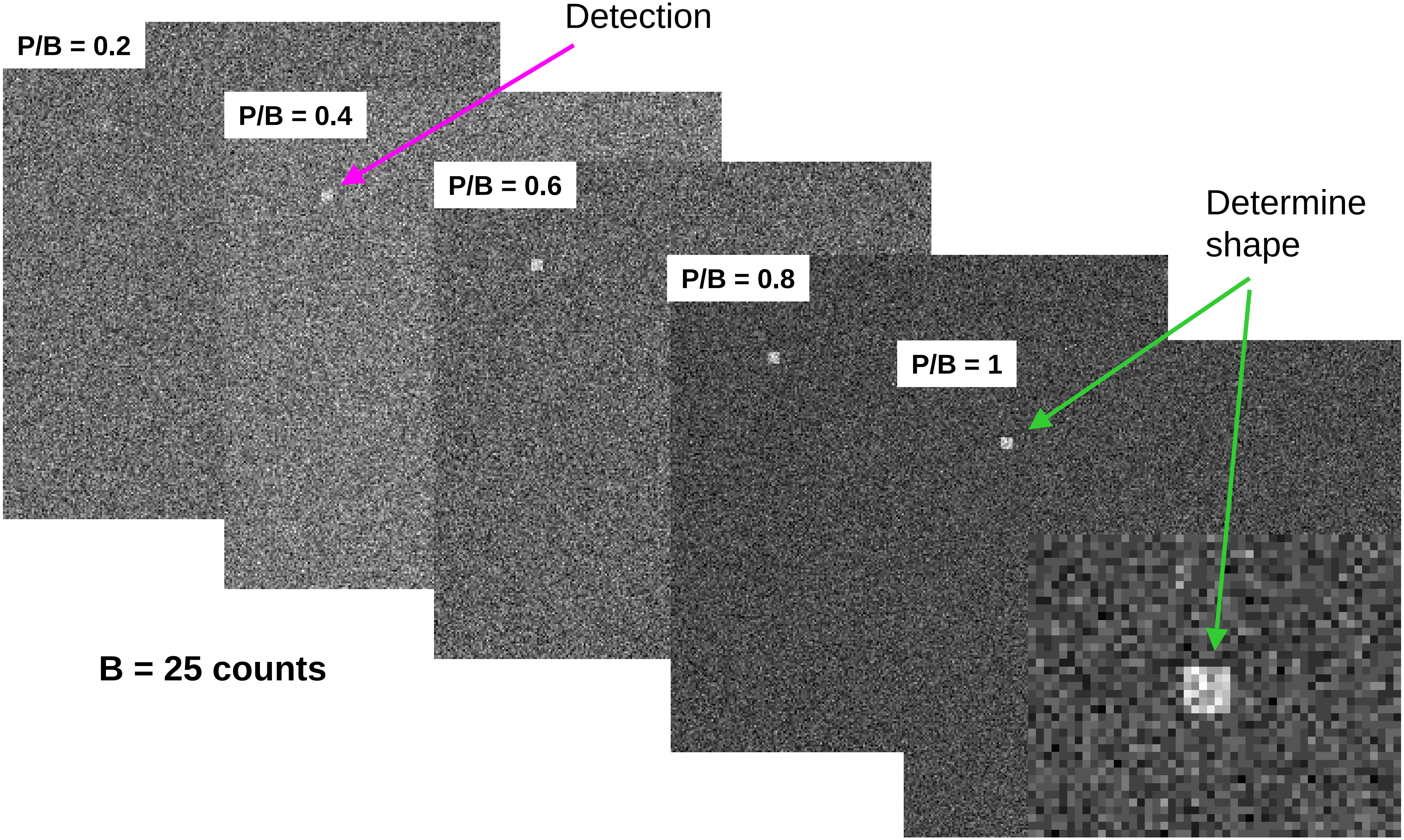
Size Matters! Effect of Feature Size on Visibility

Feature: 1 pixel (Rose criterion) = 0.4% of image width (256 pixels)



Size Matters! Effect of Feature Size on Visibility

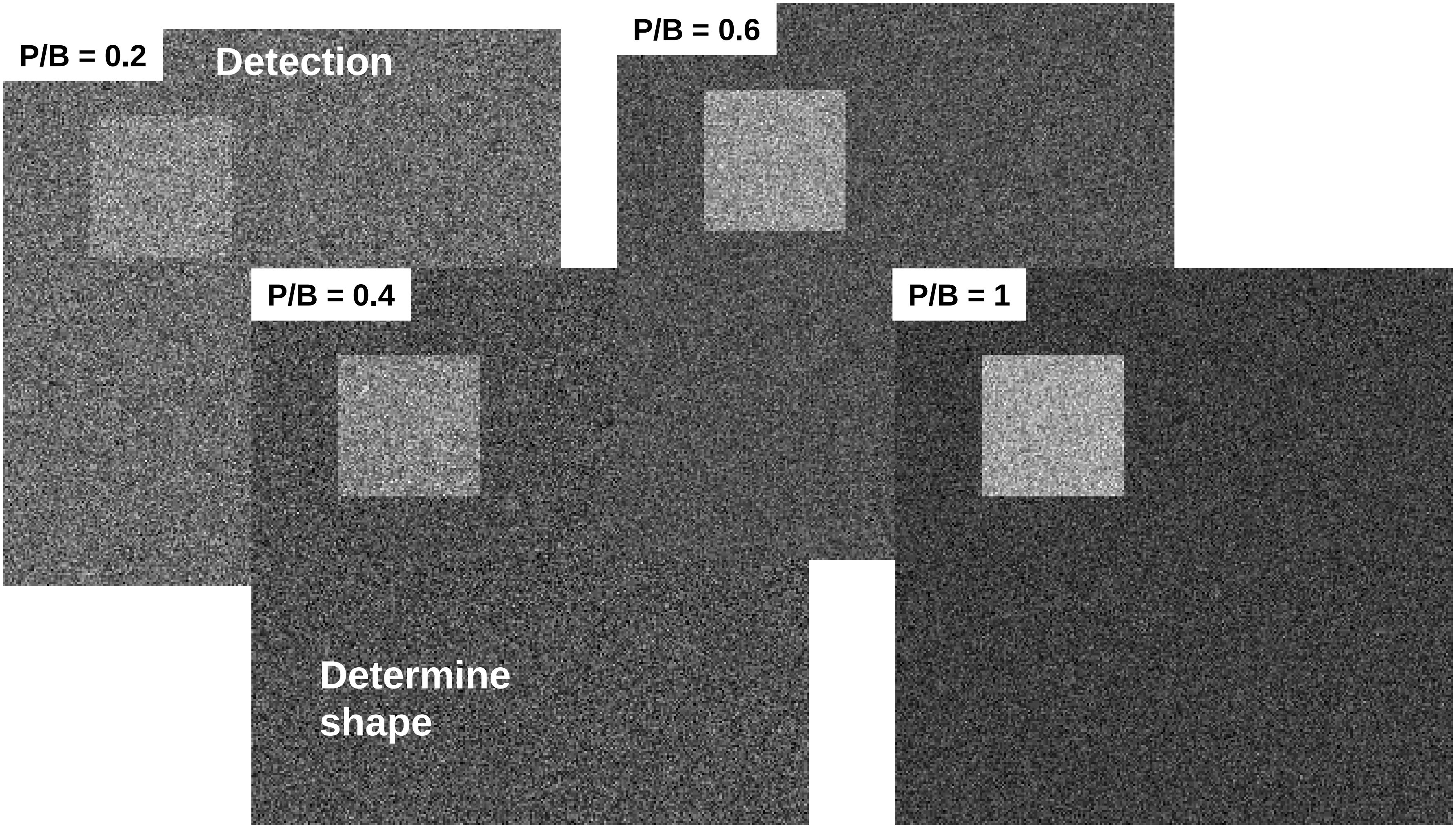
Feature: 5 pixels square = 2% of image width (256 pixels)



B = 25 counts

Size Matters! Effect of Feature Size on Visibility

Feature: 64 pixels square = 25% of image width (256 pixels)



B = 25 counts

See: Bright, D. S., Newbury, D. E., and Steel, E. B., “Visibility of objects in computer simulations of noisy micrographs”, J. Micros., 189 (1998) 25-42.

Conclusions

- SDD performance enables XSI mapping in 10 to 200 seconds (512x394_1ms) if the specimen can withstand the high beam current necessary to generate high x-ray flux.
- NIST LISPIX interactive tools enable detection and recovery of compositional features, including those that are rare and unanticipated.
- Strategy for mapping can be developed with the spectral simulation tool in NIST DTSA and the new image simulation tool embedded in NIST LISPIX.

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