Microspectroscopy Beamline

Accelerator Science School and Workshop
25 March 2008

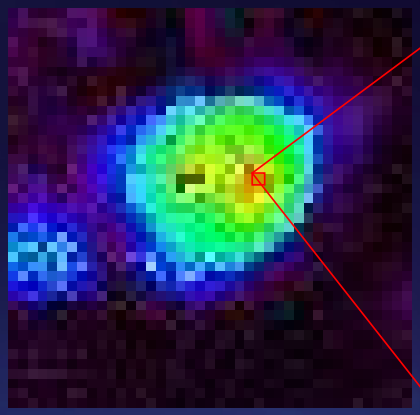
David Paterson

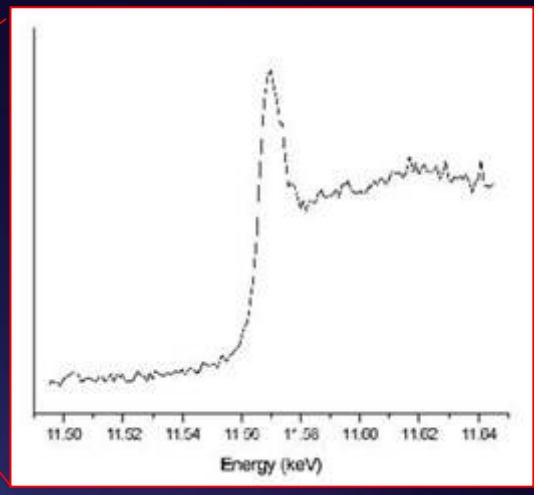
The Synchrotron World Map

– as seen from Australia

Operating at full speed = 200 mA @ 3 GeV

Microspectroscopy beamline – vital statistics

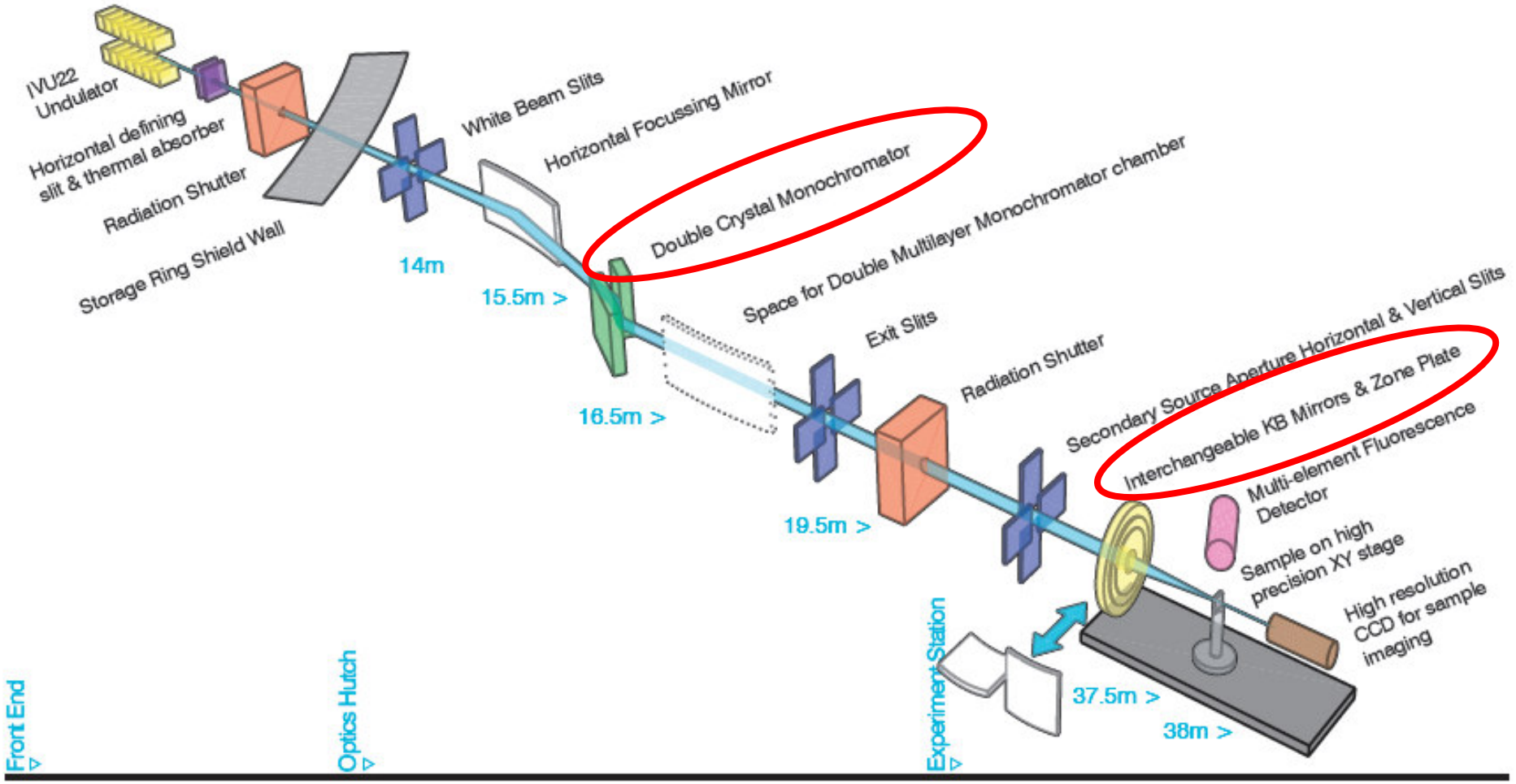




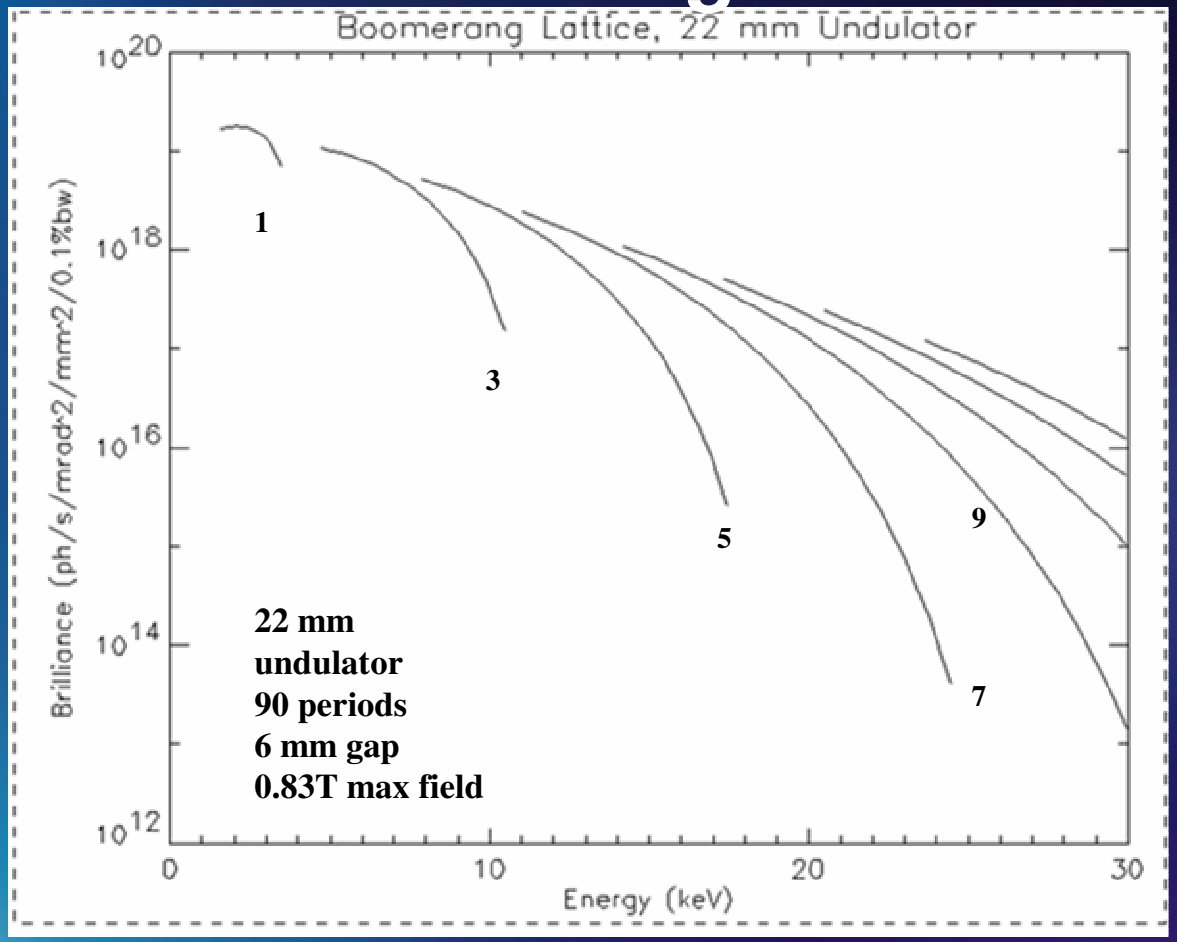
Pt spectrum located in a tumour cell
Hambley et al, U Sydney

- **Energy range**
  4 keV to 25 keV
- **Nanoprobe**
  100 nm spatial resolution
- **Elements accessible**
  atomic number ≥ 11, sodium & heavier by XRF
  atomic number ≥ 22, titanium & heavier by XAS
- **Measurements**
  X-ray fluorescence (XRF), X-ray absorption spectra (XAS),
  μXANES & μEXAFS – spatially resolved
- **Information**
  Elemental mapping, **chemical state** mapping, **ppm sensitivity**, long & short range **structure**
- **Synchrotron benefits compared to lab source**
  Spot size (0.1-10 μm), energy scanning, intensity/sensitivity

Conceptual layout



Undulator tuning curves



Tuning Curves for in vacuum 22mm, 90 period, 6 mm minimum gap undulator with 0.83 T max field. Harmonics to 15 are shown. (achieved 0.97 T!)

Brightness

5 keV on 3rd harmonic

8.7×10^{18} ph/s/0.1%BW/mrad^2/mm^2

25 keV on 9th harmonic

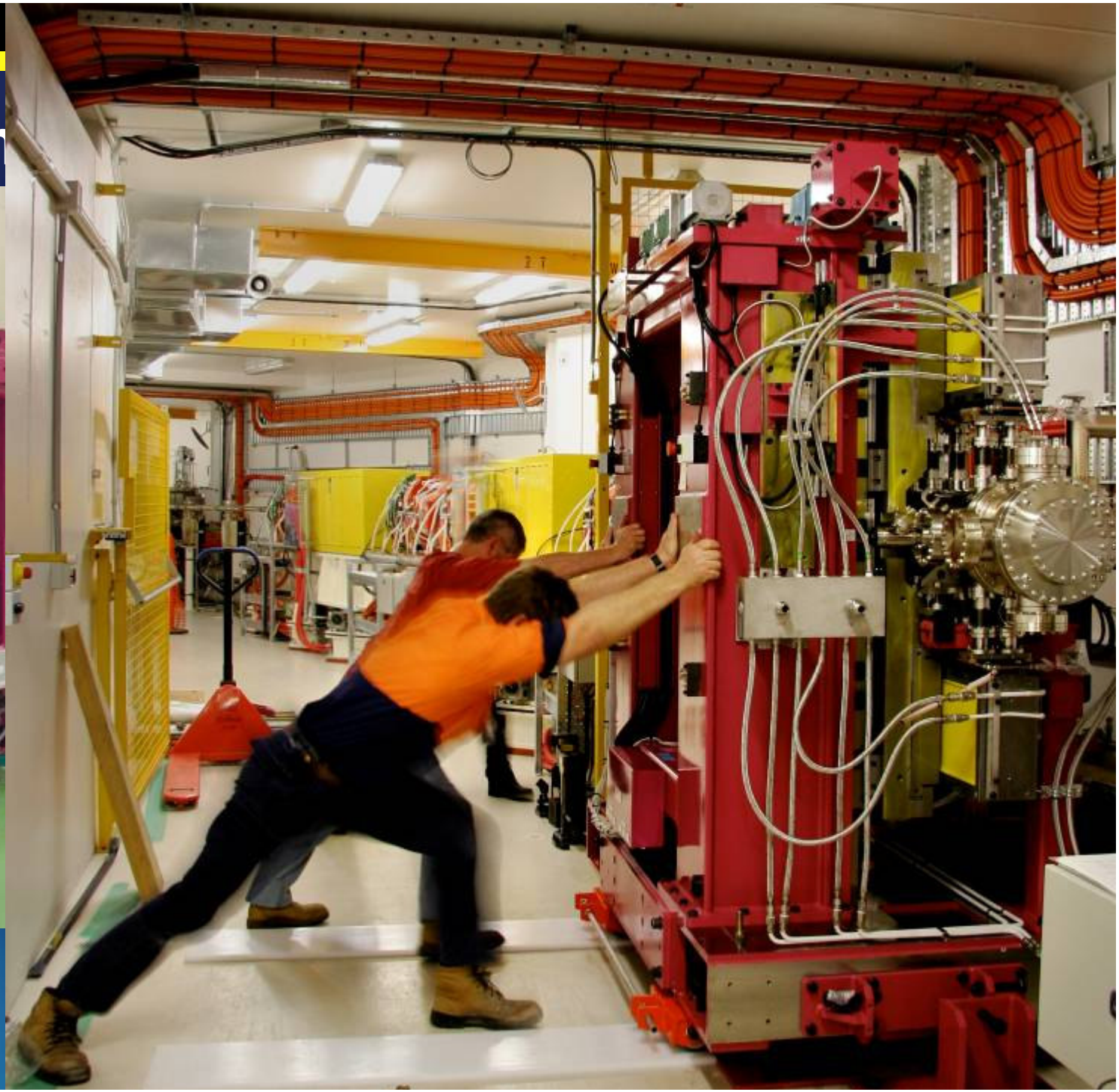
4.6×10^{15} ph/s/0.1%BW/mrad^2/mm^2.

Specified > 90% of theoretical flux at peak 7th harmonic, > 85% of theoretical flux in the peak at the 9th harmonic.

Curves assume zero phase errors but include allowance of 0.1% for energy spread

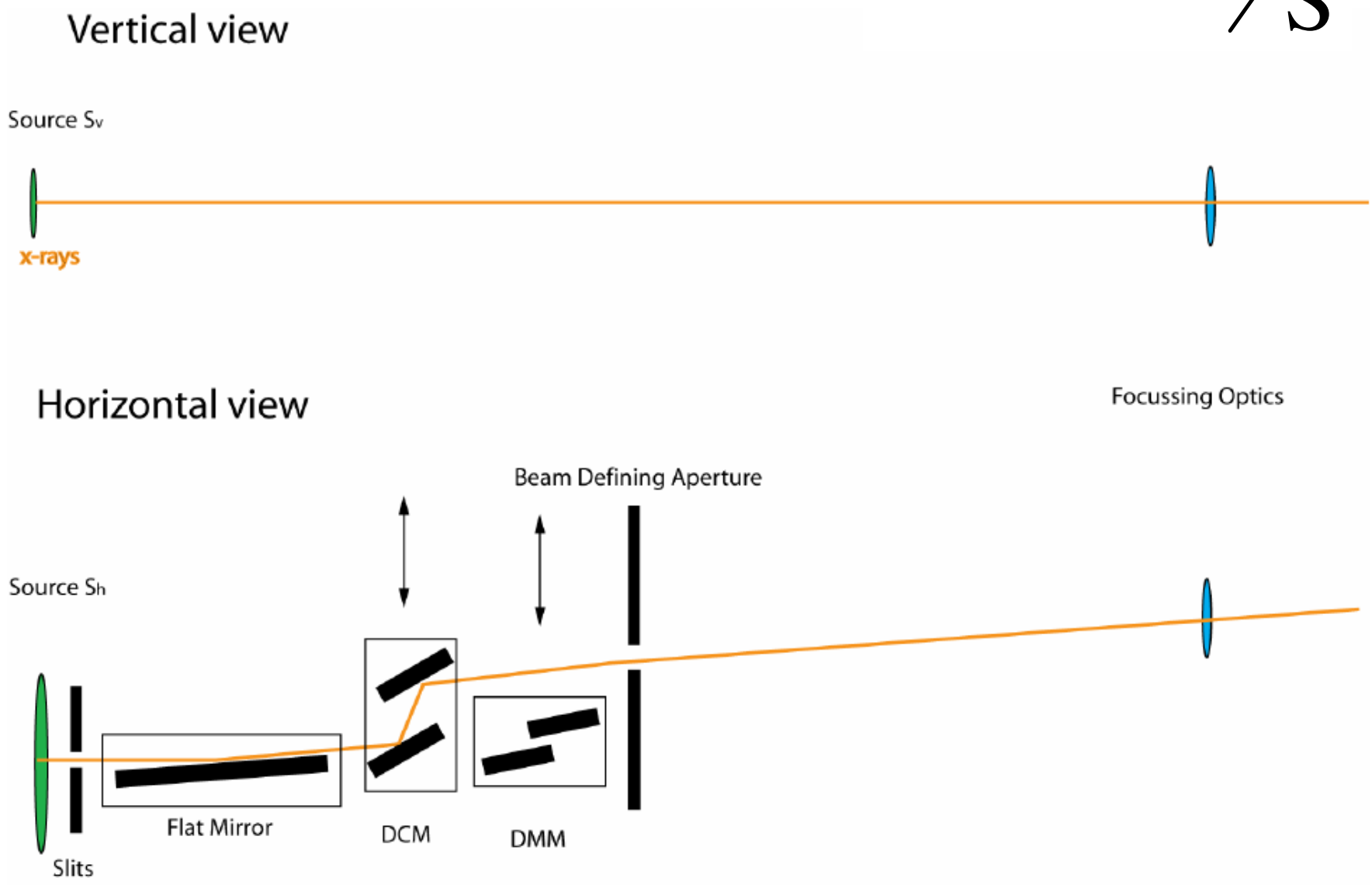
Phase errors on undulator specified at <2.5 degrees

In-vacuun



Optics only in horizontal

$$D = 0.5L\,\lambda/S$$



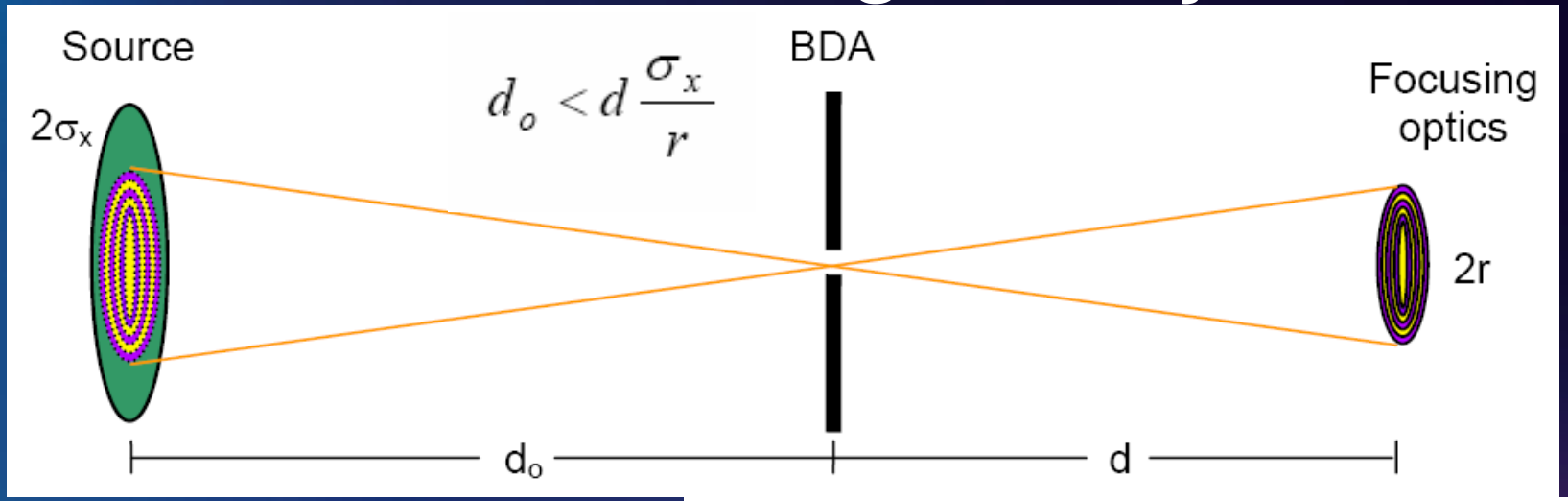
Coherent illumination - van Cittert-Zernike theorem

the spatial coherence over a space illuminated by an incoherent extended source is described by the Fourier transform of the intensity distribution over the source

$$D = 0.5L\,\lambda/S$$

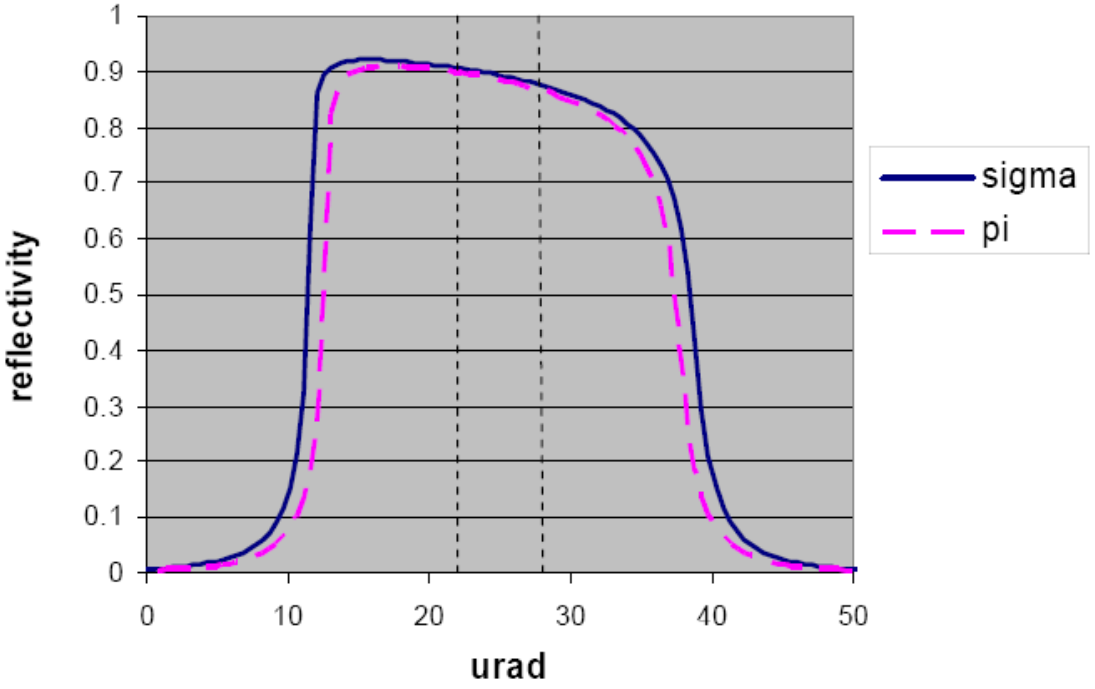
	Source Size 1σ (μm)	Source Size FWHM (μm)	Energy (keV)	λ (A)	L (m)	Coh illum Diam (μm)	Note
Horiz	320	753.536	5	2.48	37	14	Horizontal 320 μm
Vert	16	37.6768	5	2.48	37	287	
BDA	8	18.8384	5	2.48	20	310	BDA @ 17 m
Horiz	320	753.536	10	1.24	37	7	
Vert	16	37.6768	10	1.24	37	143	Vertical source size
BDA	10	23.548	10	1.24	20	124	16 μm
Horiz	320	753.536	20	0.62	37	4	
Vert	16	37.6768	20	0.62	37	72	
BDA	8	18.8384	20	0.62	20	77	

Horizontal diffraction geometry

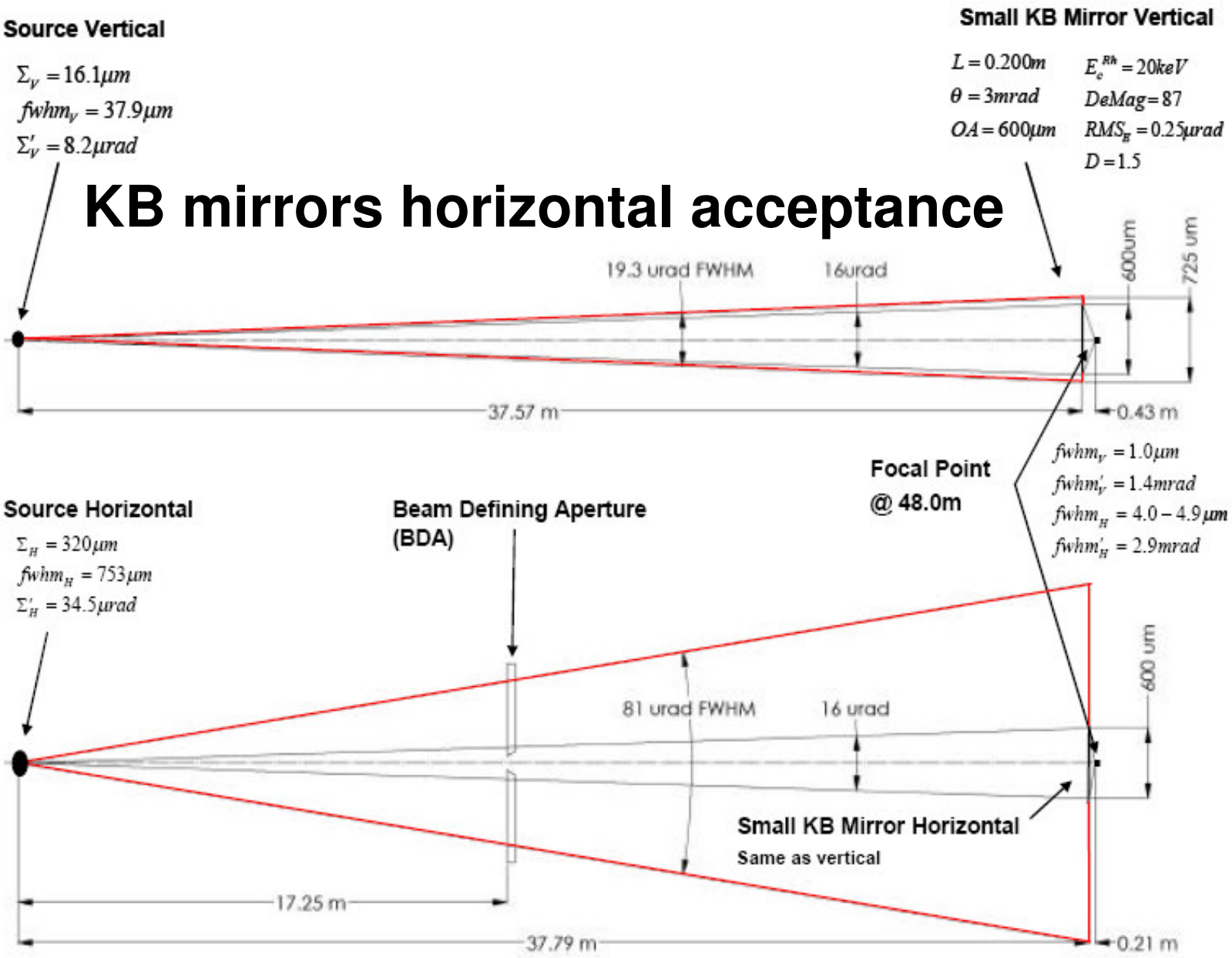


Polarization losses?
Pi polarization

Acceptance of nanoprobe optics = 6.7 µrad
Throughput becomes
5.0 keV 50% -> **80**%
10 keV 91% -> **99**%

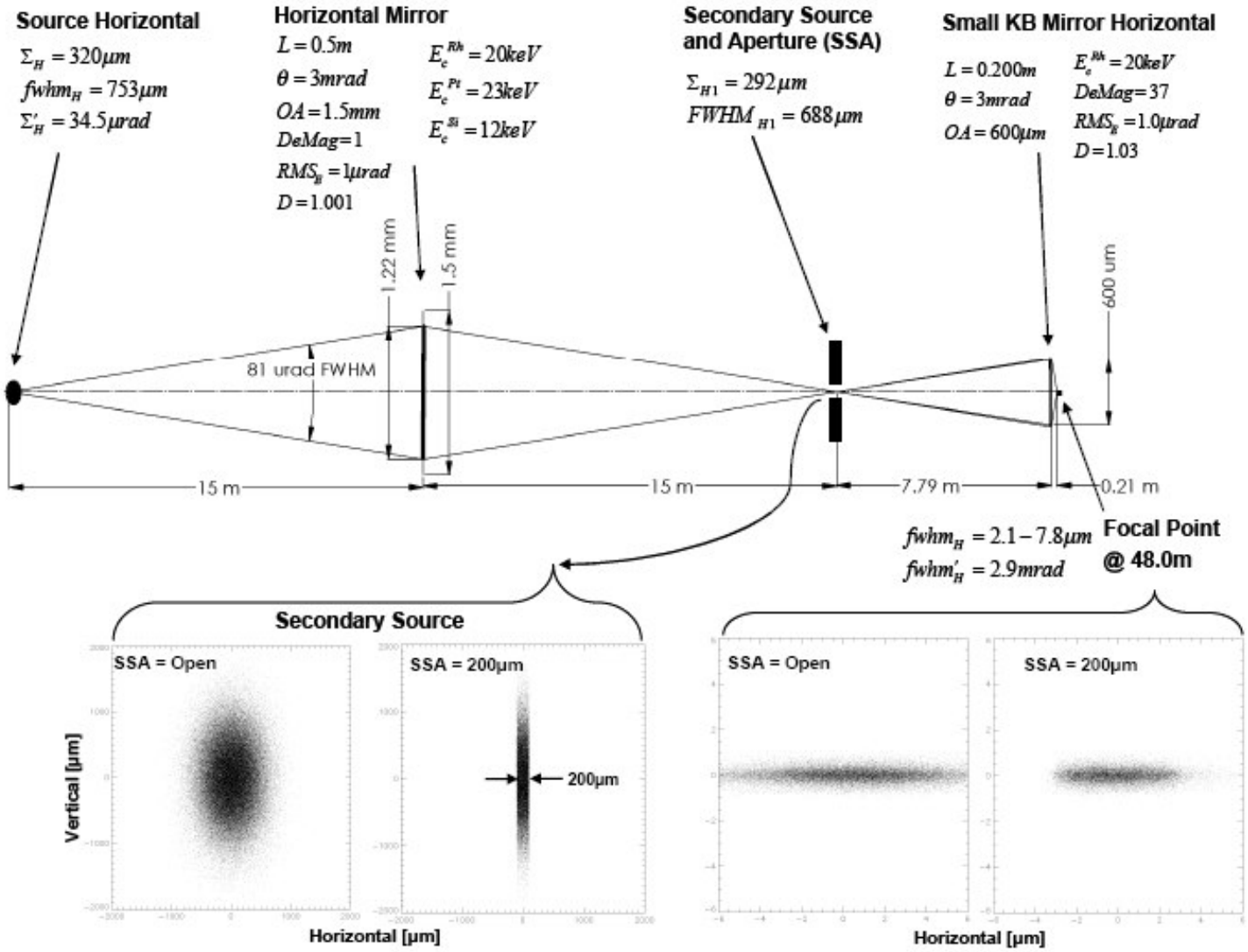


KB mirrors horizontal acceptance



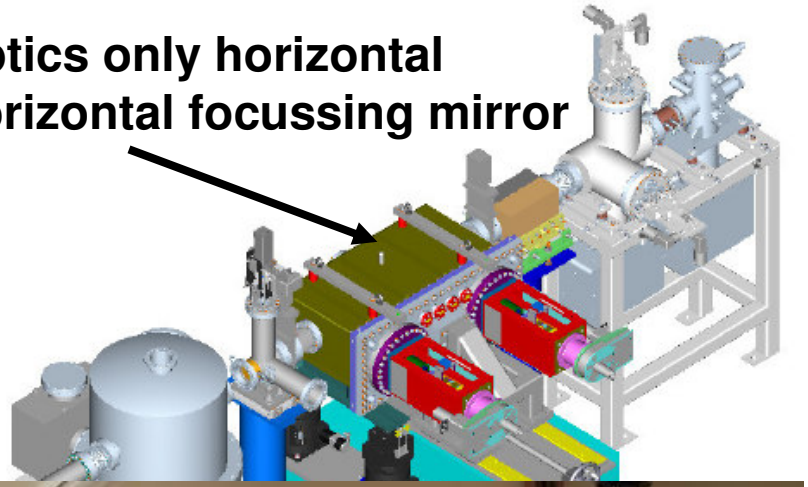
1-1. Optics layout for the small kb only focusing optics mode. Top vertical focusing, bottom horizontal.

Horizontal compound focusing



Optics layout horizontal compound focusing (HCF) mode optics mode. Vertical same as small KB only mod

Detailed final design



Double multilayer monochromator

Integrated design:

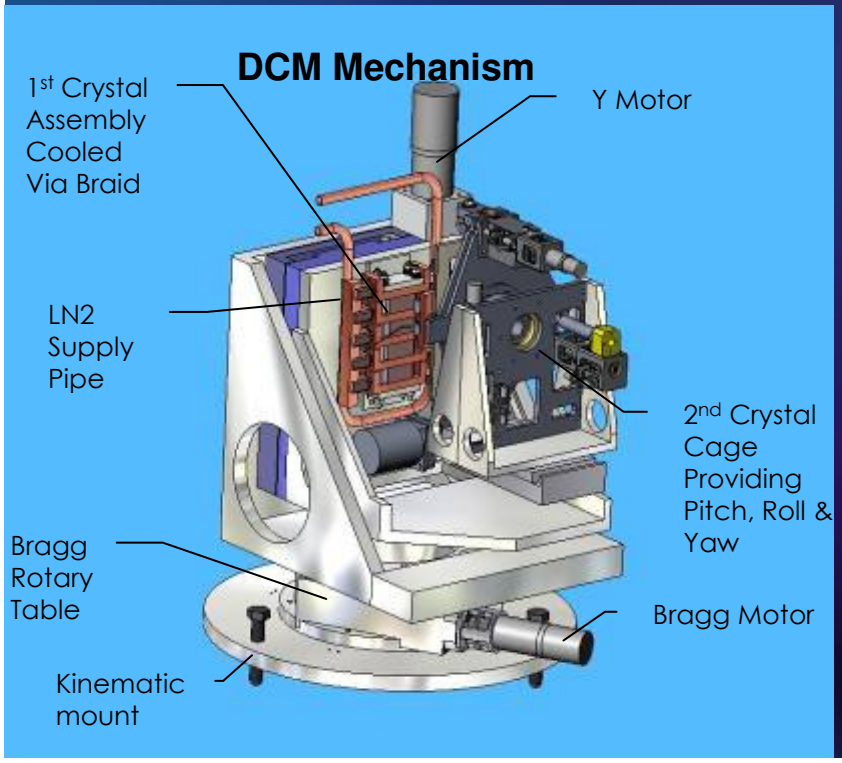
Double Crystal Monochromator &
Double Multilayer Monochromator

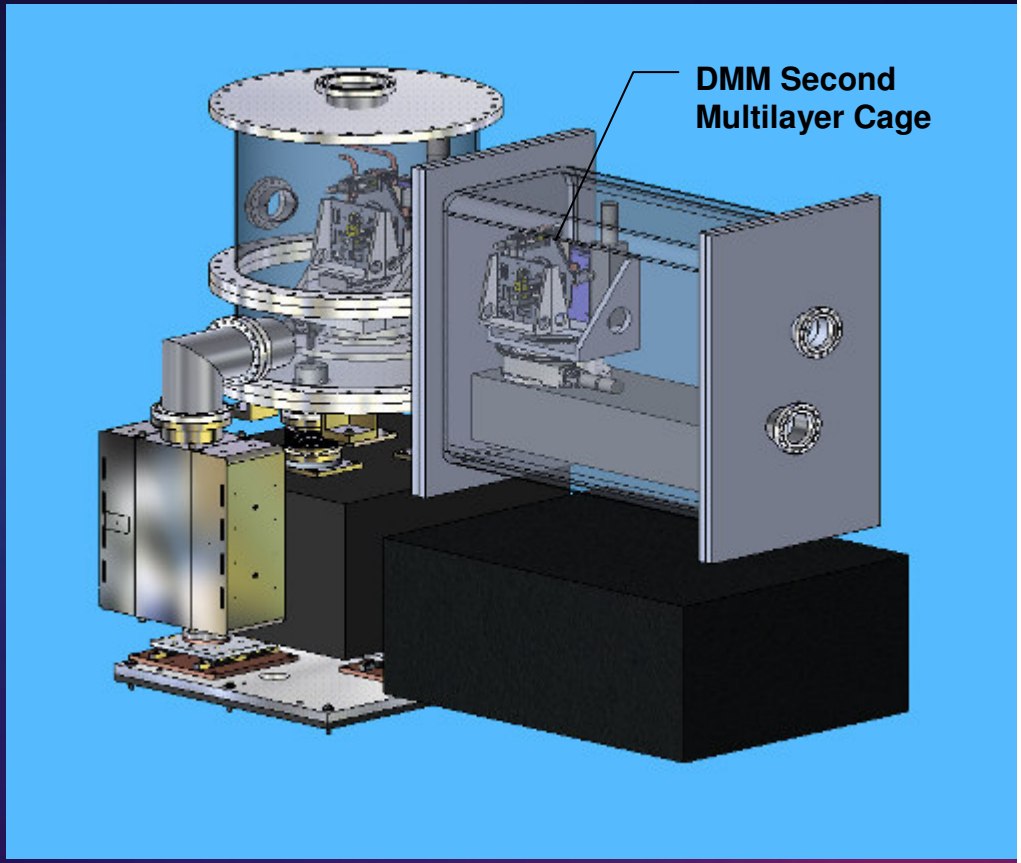
DCM-Si(111): 0.02% ΔE/E

1×10^{12} ph/s @ 12 keV in 10 μm

4×10^{10} ph/s @ 12 keV in 1 μm

DMM: 2% ΔE/E, 1×10^{13} ph/s in 10 μm





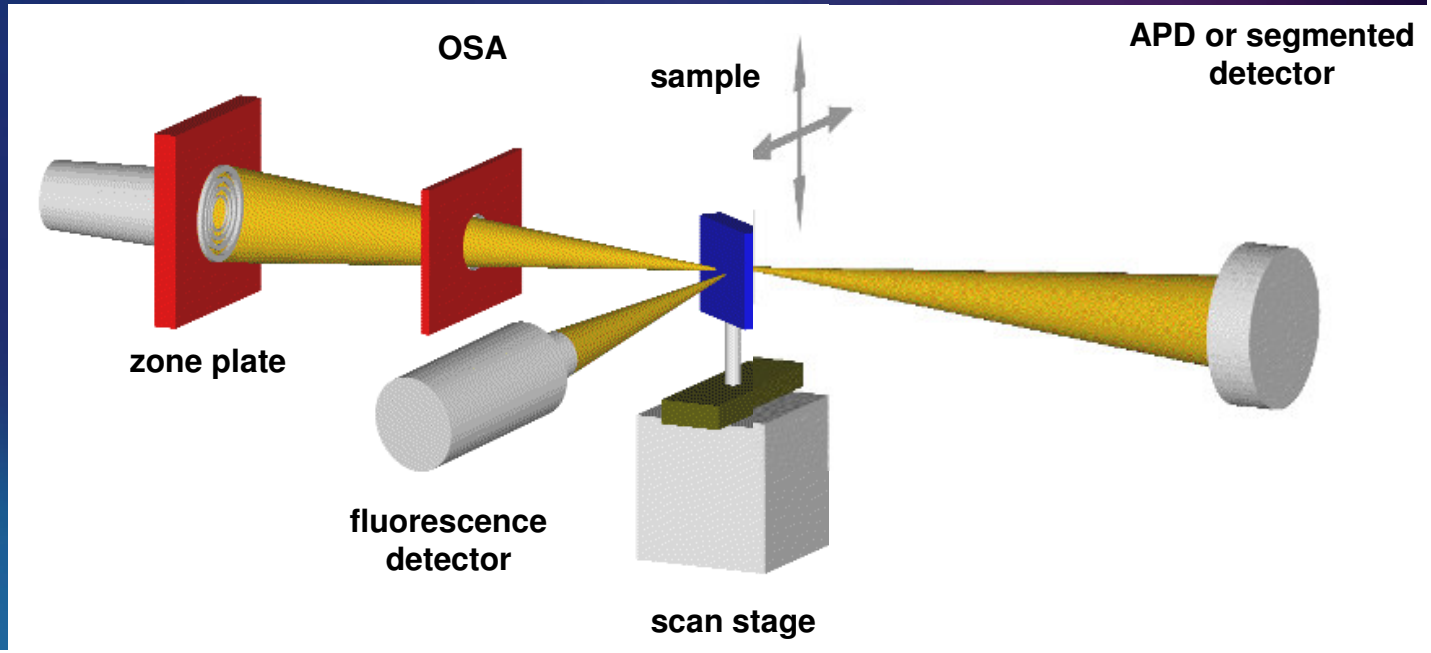
Microprobe focusing optics

KB mirrors

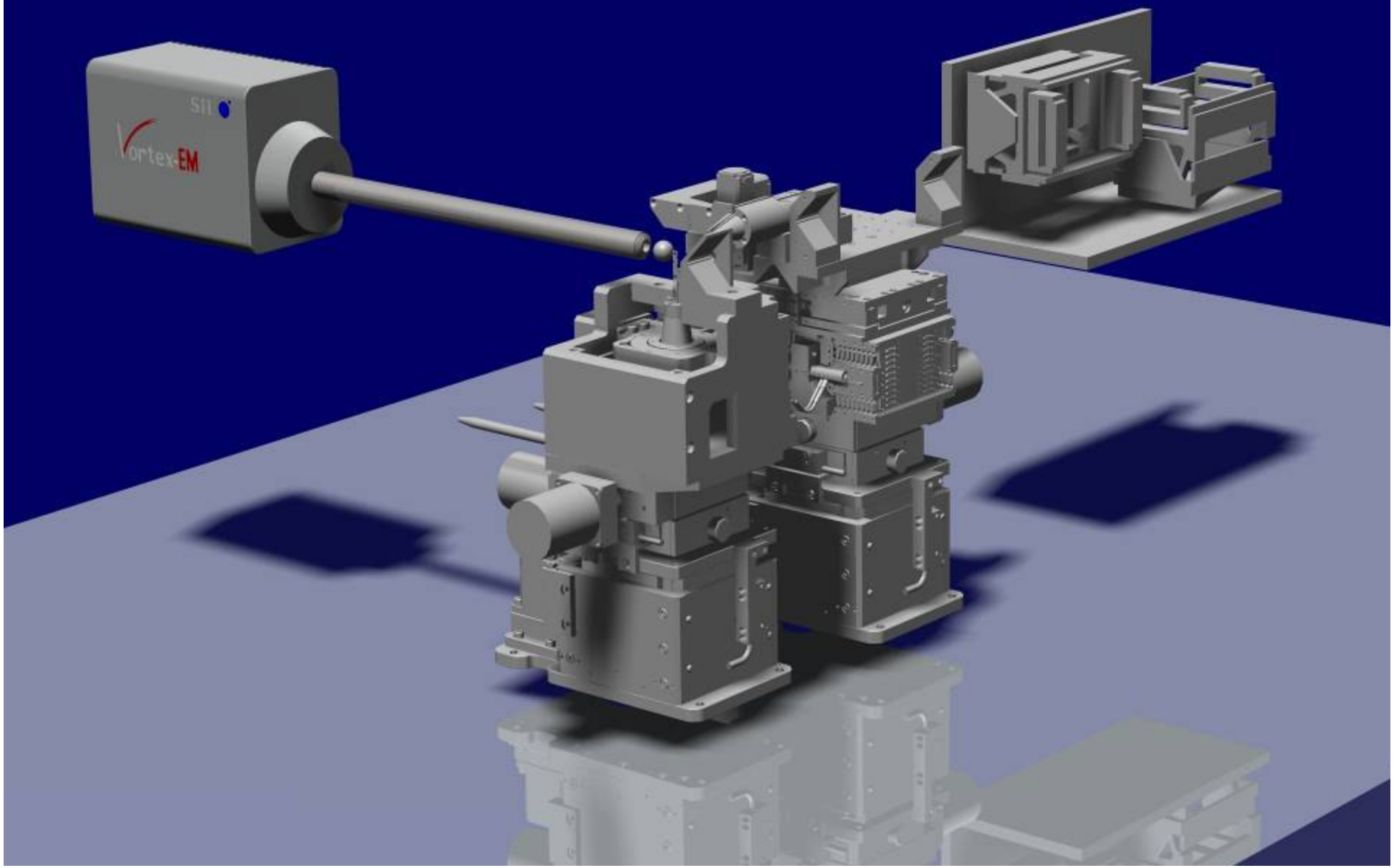
- A mirror pair of the Kirkpatrick-Baez geometry will be used for high throughput achromatic studies. Capable of focusing to 1 μm maintaining maximum acceptance.

Fresnel zone plates

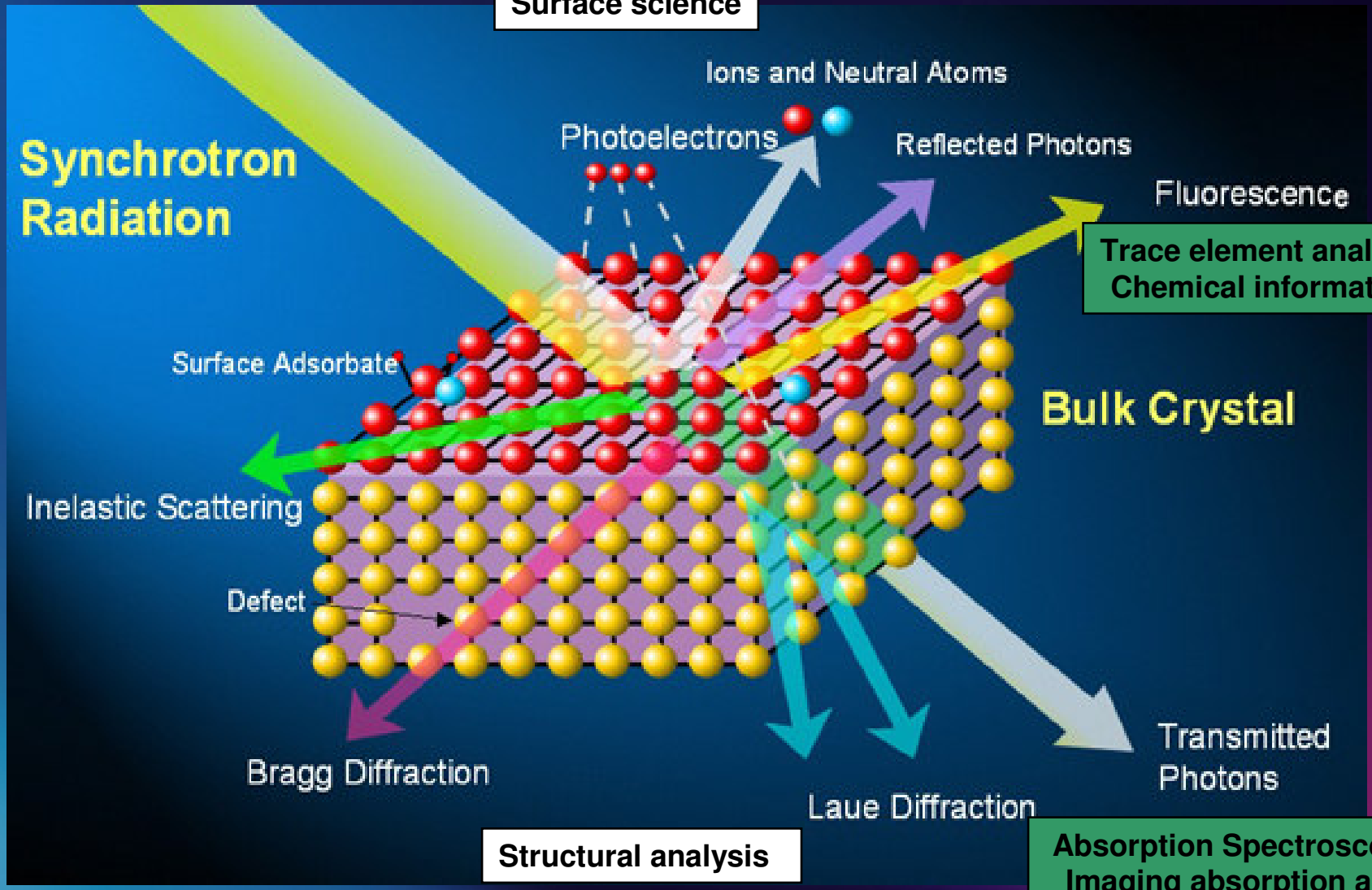
- The beamline will also accommodate and provide the correct coherent illumination to Fresnel zone plates. Capable of focusing to 60 nm for studies requiring sub-micron resolution.



Integrated KB mirrors and zone plate optics

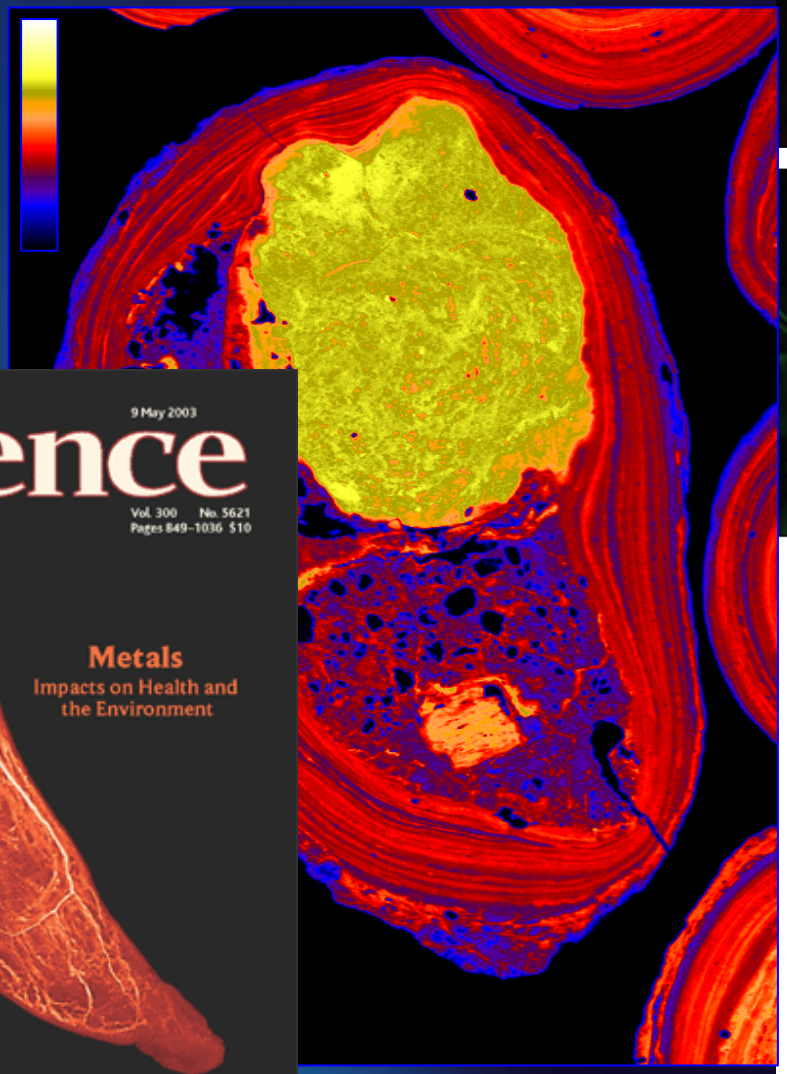


Interaction of X-rays with matter

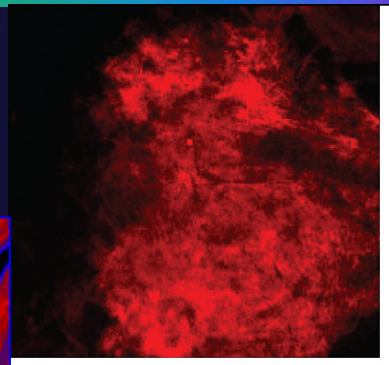


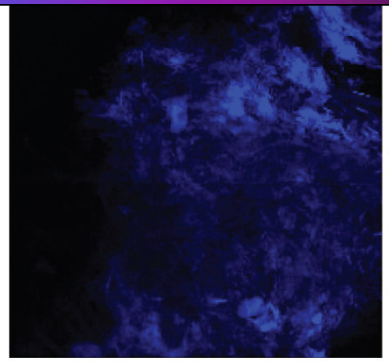
Courtesy SRRC, Taiwan

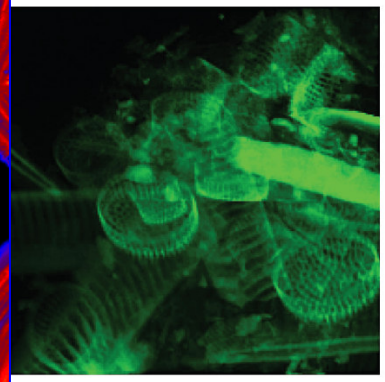
Applications: biological & environmental sciences



Trace metal mapping and speciation







SPECTROMICROSCOPY— A Tool for Environmental Sciences

High spectral and spatial resolution makes synchrotron-based spectromicroscopy well suited for environmental applications.

JÜRGEN THIEME
UNIVERSITY OF GÖTTINGEN (GERMANY)
IAN McNULTY
STEFAN VOGT
ARGONNE NATIONAL LABORATORY
DAVID PATERSON
AUSTRALIAN SYNCHROTRON

Environmental science is an extremely diverse field that impacts many facets of science, industry, and everyday life. Because of the complexity of environmental processes and the many mutual interactions involved, competence is required in more than one area of expertise, and a multidisciplinary approach is often necessary. The experts in geochemistry, hydrology, microbiology, and atmospheric and soil sciences, to name a few, are essential in this regard. For example, the complex roles of carbon, nitrogen, phosphorus, and sulfur cycles in the environment are incompletely understood, as are their mutual interactions. Similarly, our knowledge of uptake and metabolism of environmental contaminants by biological organisms is far from complete. Elemental transport, redox reactions, microbial activity, and anthropogenic influences all affect these processes. Knowledge of each individual piece of the puzzle contributes to a better understanding of the overall picture.

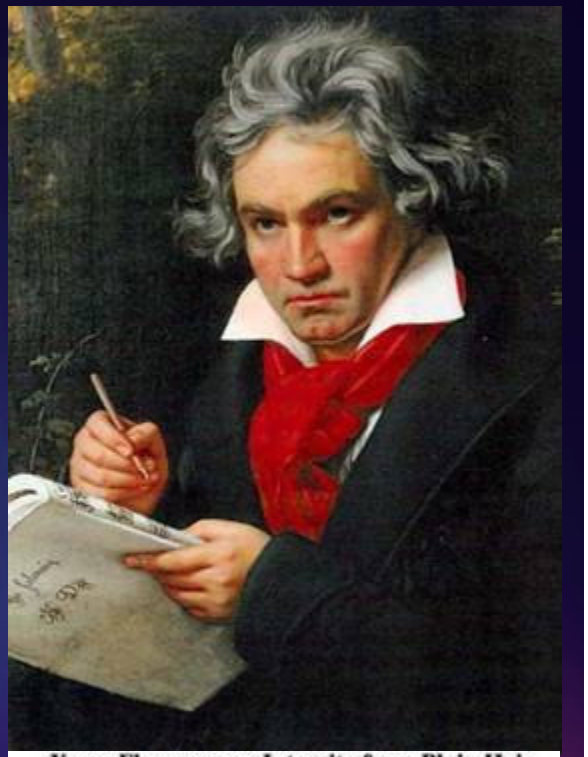
Uses and applications

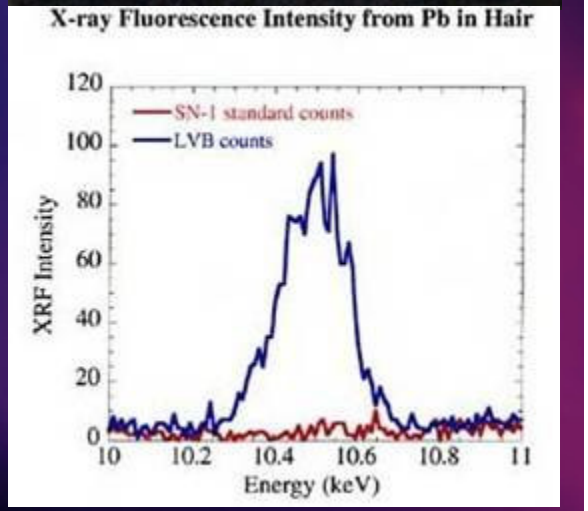
Microspectroscopy

What killed Beethoven?

Using synchrotron light to analyse six strands of Beethoven's hair, US scientists discovered 100 times the safe level of lead.

The result showed his deafness, other illnesses and death were likely to have been caused by lead poisoning.

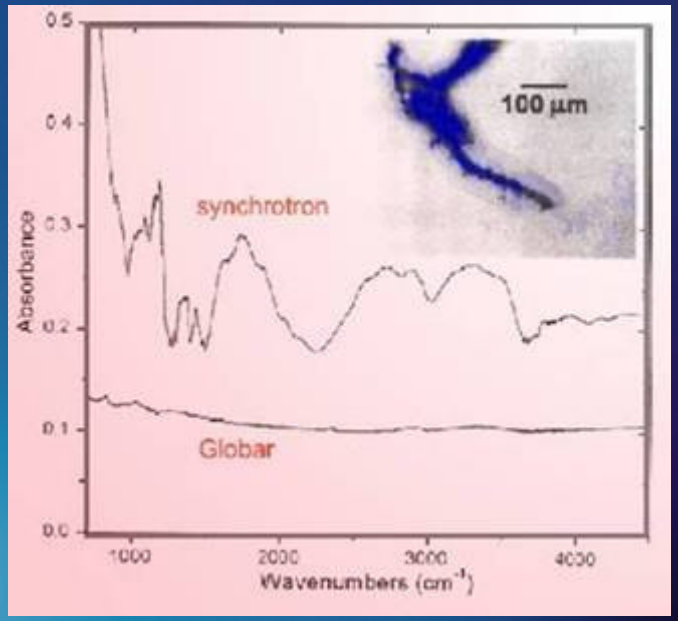




Characterise at the crime scene - safeguarding Australia

Forensics

Extremely small samples from crime scenes can be analysed using synchrotron technology.



Forged documents and counterfeit money can be detected using synchrotron techniques.

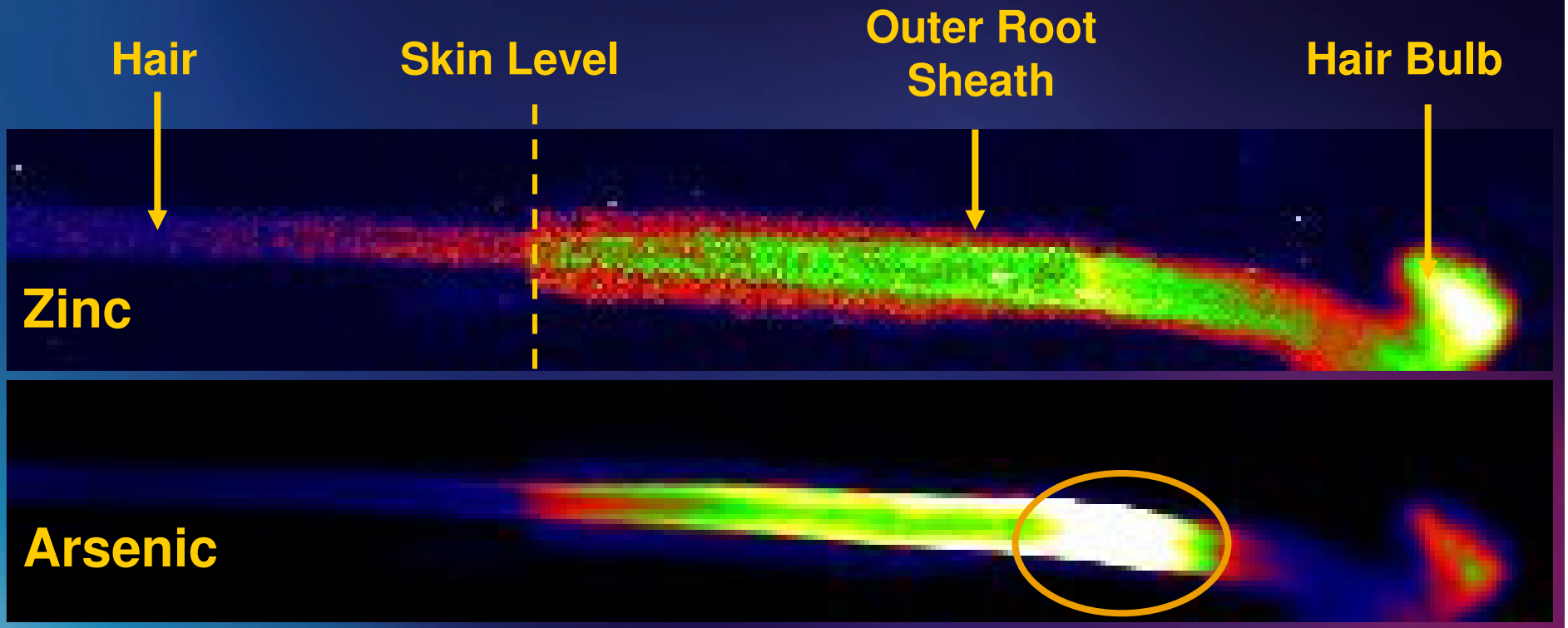
Pollution Control

Air pollution control strategies are very expensive so it's important to target the right sources.

Airborne particles can be "fingerprinted" to identify their source - and dealt with !

A Legendary Cold Case

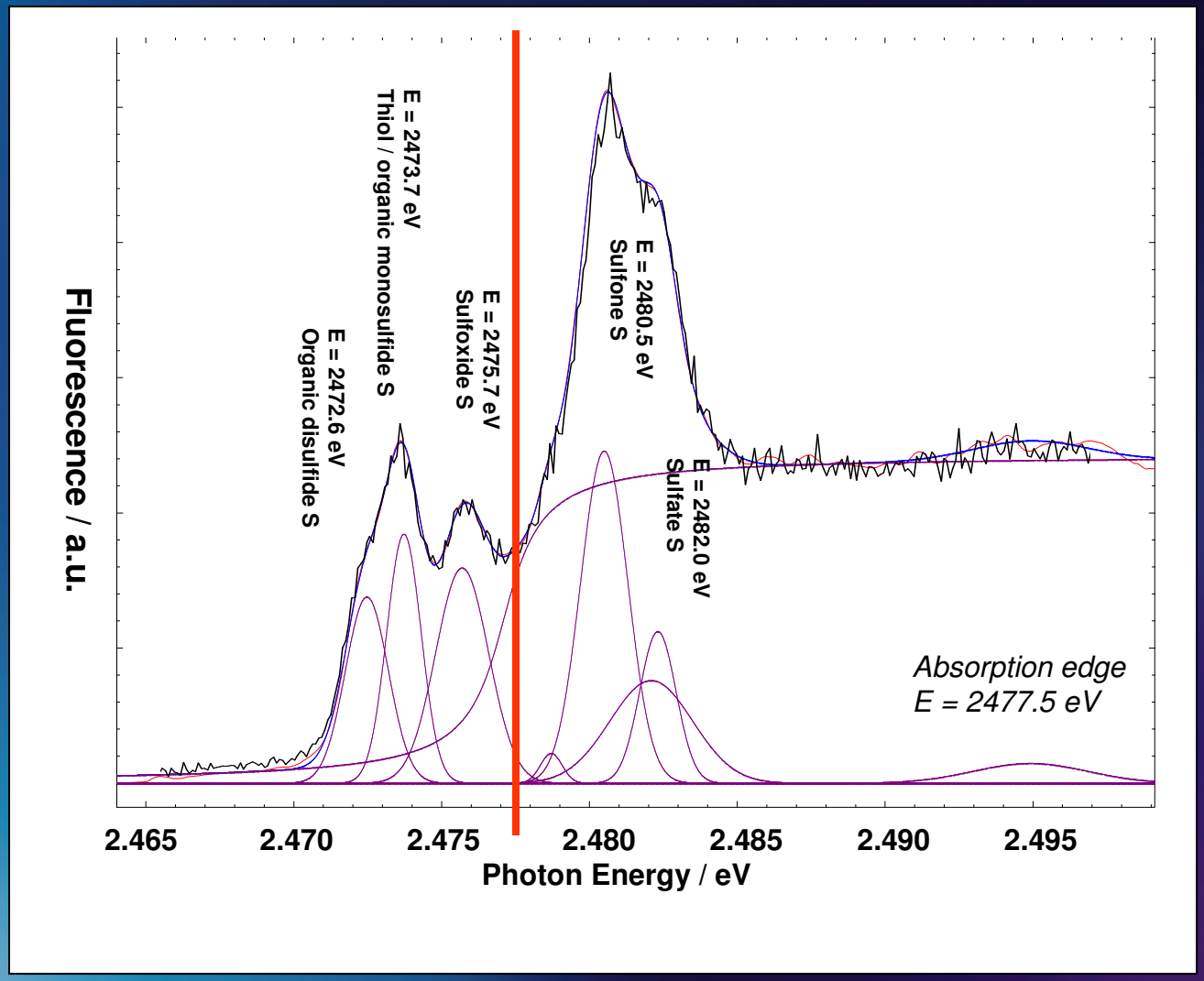
Phar Lap's hair was analysed for heavy metals



High arsenic consistent with a large amount of arsenic ingested in the champion's last 30 hours of life

Australian Research

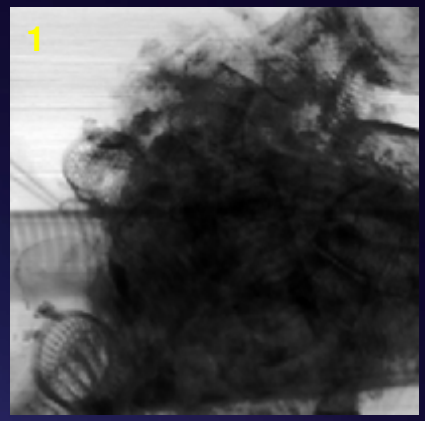
Spectromicroscopy examples



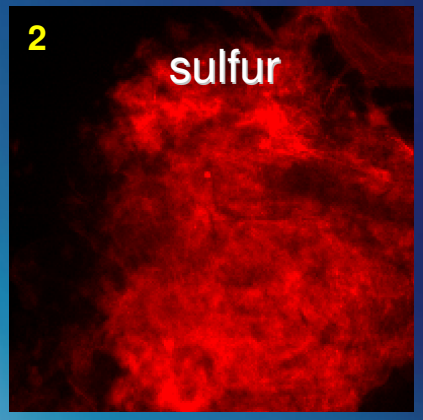
Chemical analysis:
Integrated
Reduced ⇔ Oxidized

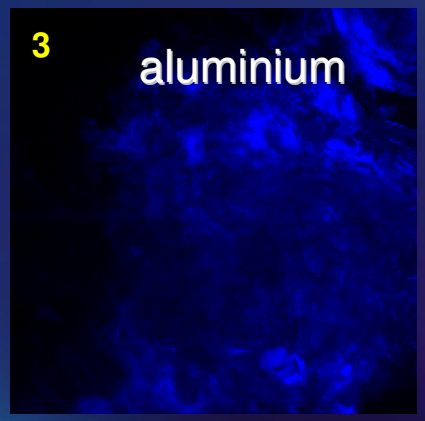
J. Prietzel, J. Thieme, U. Neuhäusler, J. Susini, I. Kögel-Knabner, EJSS 54(2003) 1-11

Spectromicroscopy examples



50×50 μm², 400×400 pixels, 125 nm, 0.5 sec dwell/pixel, full spectrum @ a pixel





Australian research example: Speciation and distribution of phosphorus in fertilized soil

In calcareous soils of southern Australia the agronomic efficiency of P fertilizers is strongly dependent on the form of fertilizer

Liquid technical grade monoammonium phosphate (TG-MAP) was 4 to 15 times more effective in increasing the grain yield of wheat than granular MAP

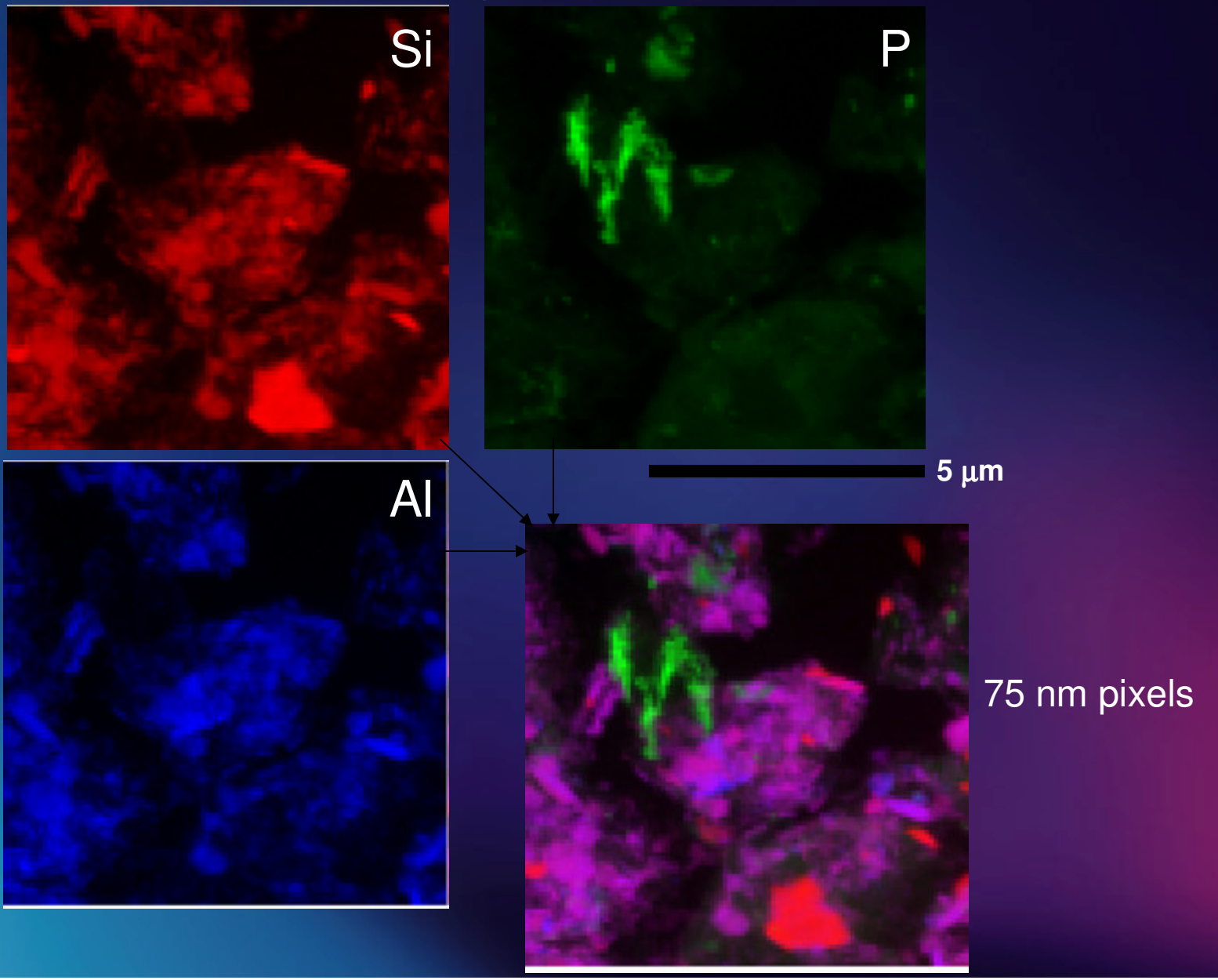
Similar results were obtained when a number of fluid and granular products were compared in both greenhouse and field studies

Chemical and physical factors in combination cause fluid fertilizers to be more efficient than similar granular products

- Soil diffusion of P from granular products is more limited than when P is supplied with fluid fertilizers
- may be the result of an increase precipitation in or around P fertilizer granules,

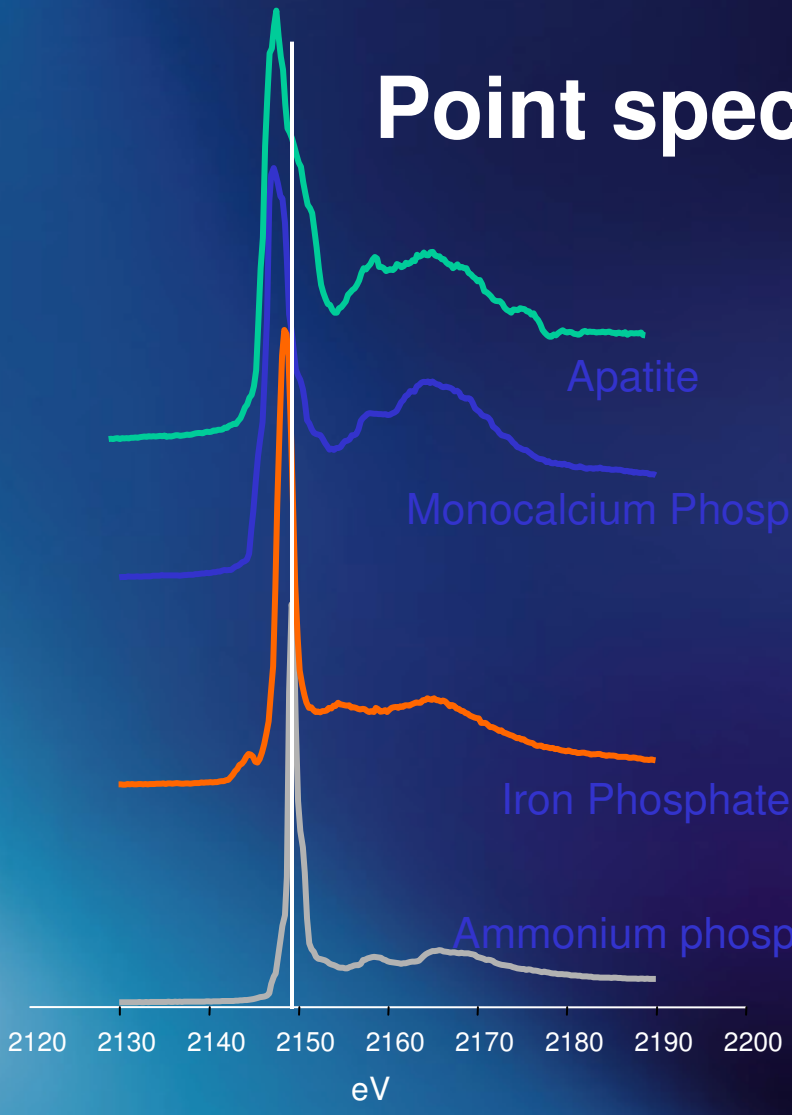
Specific mechanisms responsible for the differential response are still poorly understood.

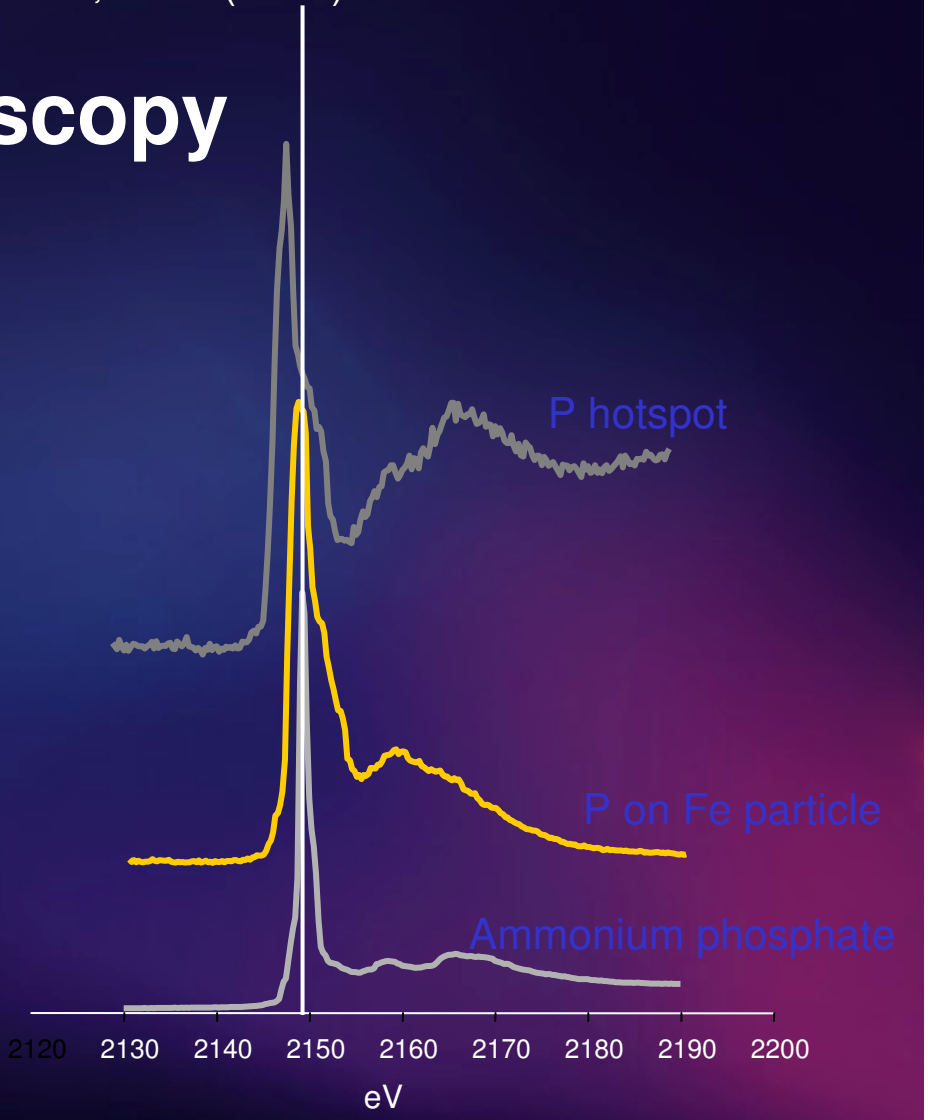
P μXANES: identifying the forms of P in soil



E. Lombi, K. G. Scheckel, R.D. Armstrong, J.N. Cutler and D. Paterson
American Soil Science Society Journal **70**, 2038 (2006).

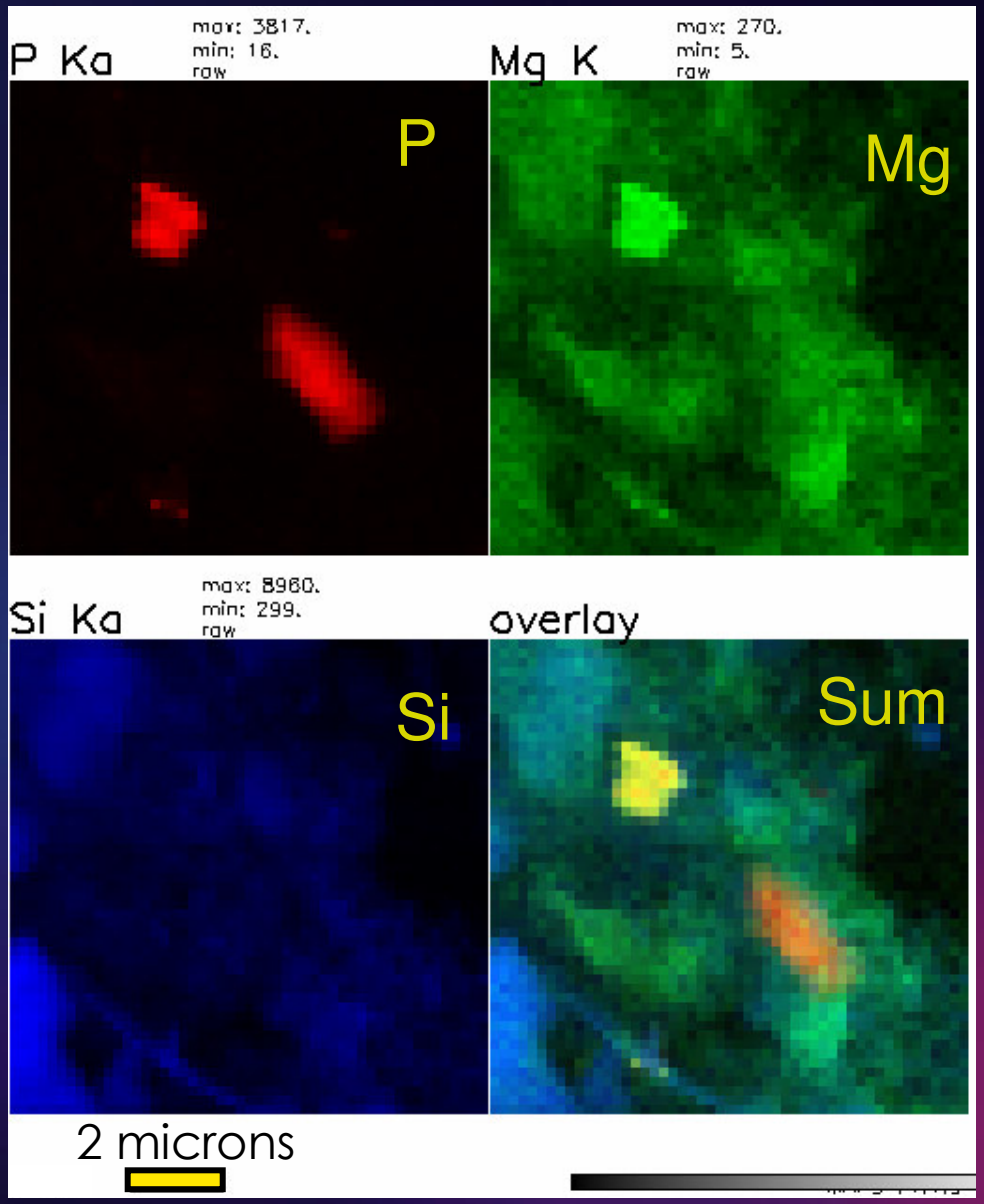
Point spectroscopy



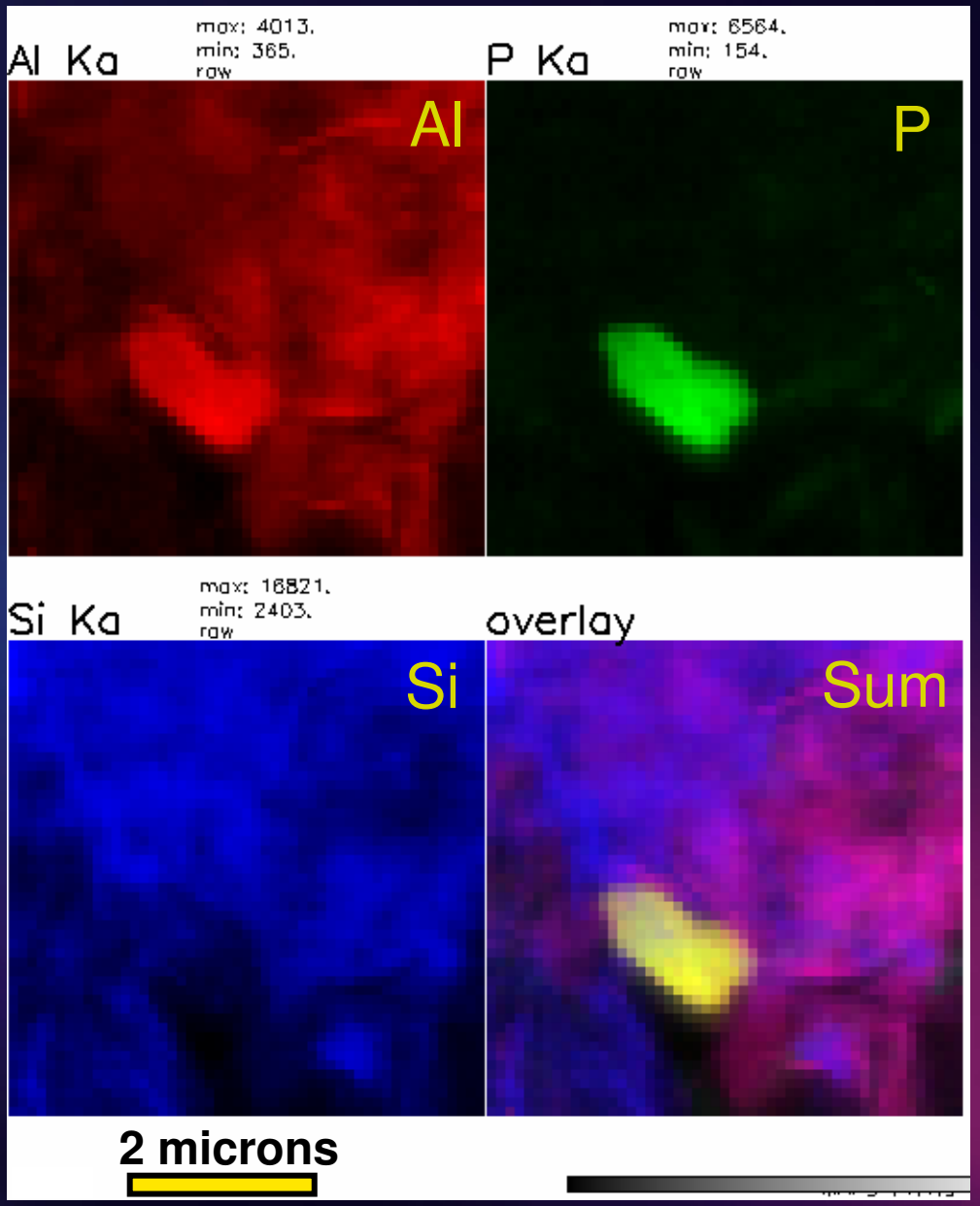


Fine scale fluorescence maps show elemental associations with P

Mg Phosphates?

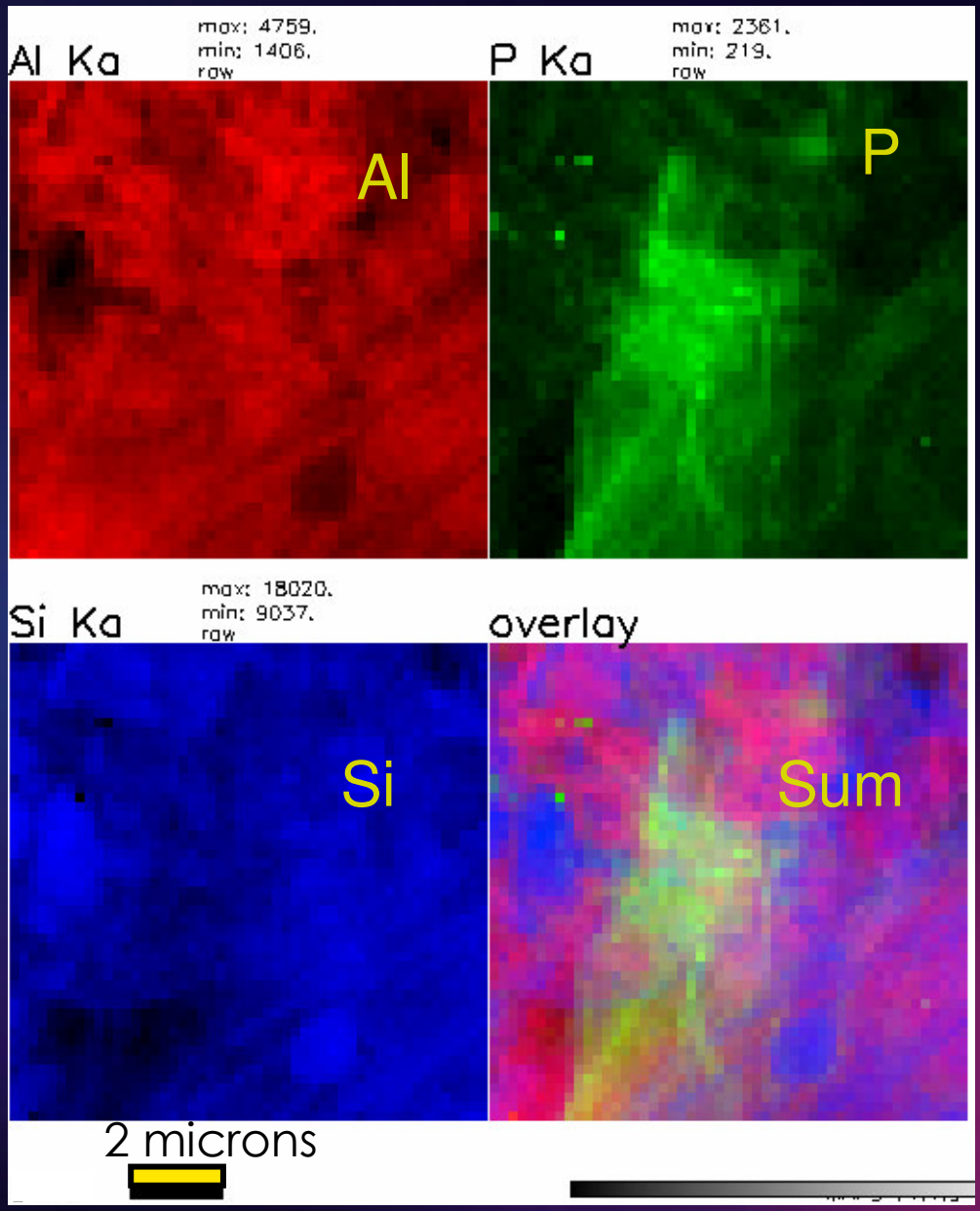


Al Phosphates?



Organic phosphates?

Diffuse phosphorus not associated with Mg, Na, Al or Si may be organic materials like cell walls

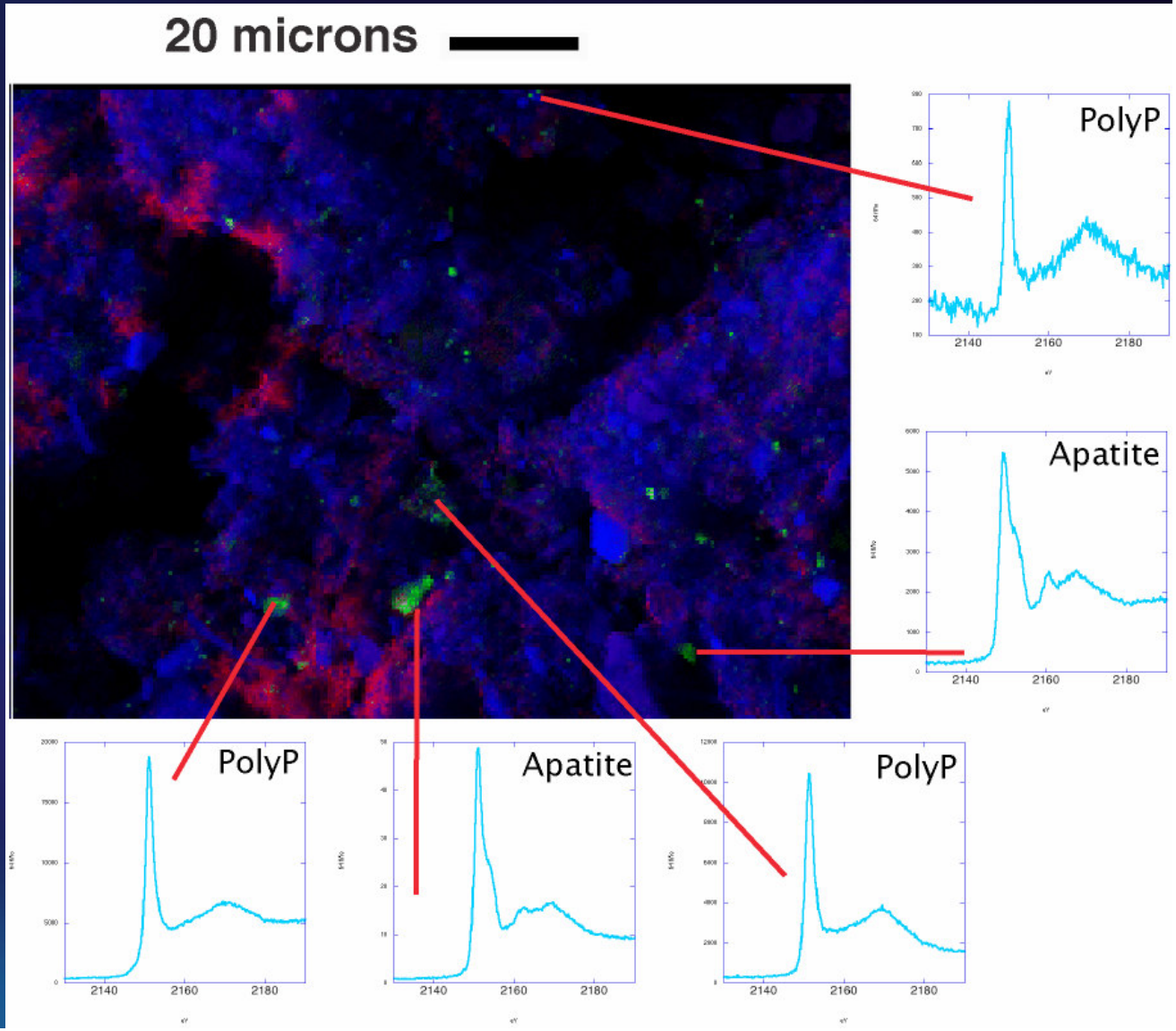


Elemental mapping and spectroscopy

Effingham Inlet Oxic Site Surface Sediment

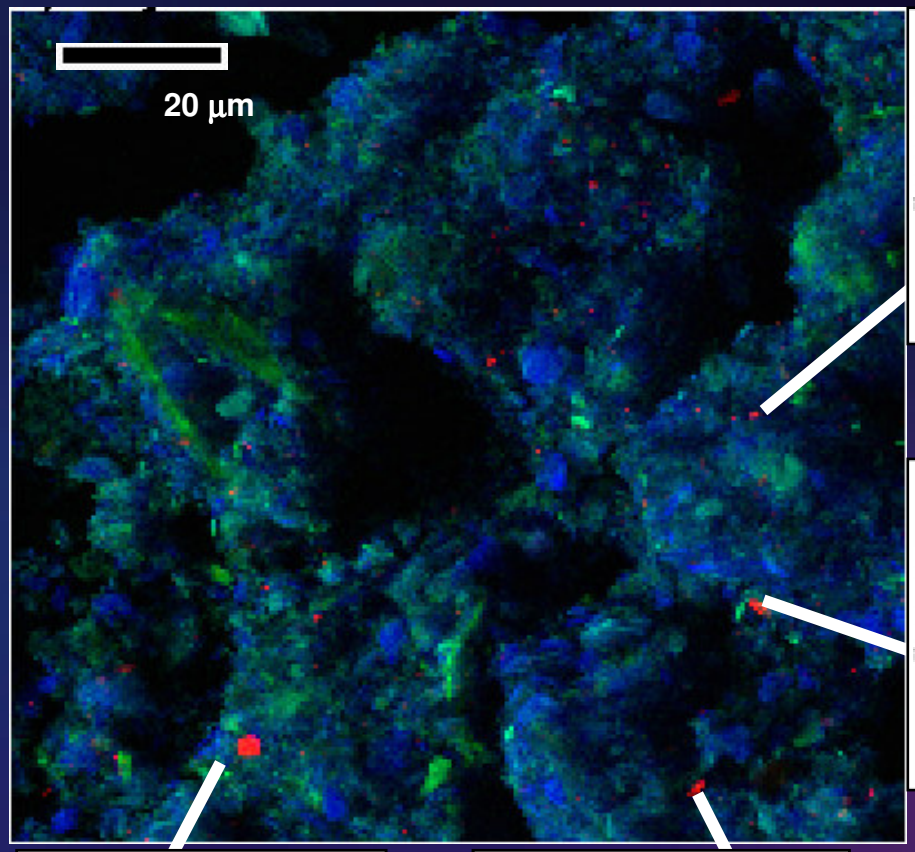
Polyphosphates occur in submicron scale blobs - similar to those found in bacterial cells

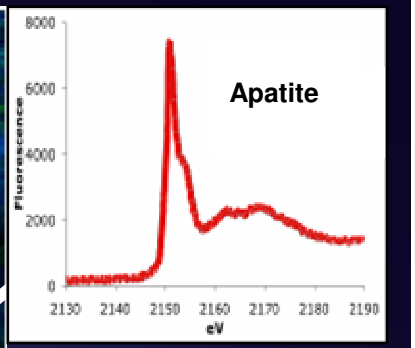
Polyphosphates seen primarily in Effingham oxic surface sediments and rarely in anoxic sediments

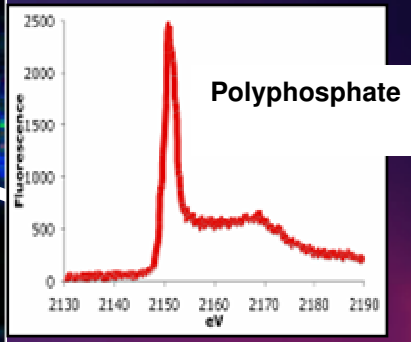


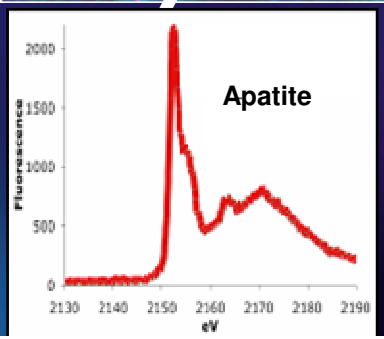
X-ray fluorescence map and spectra of marine sediment

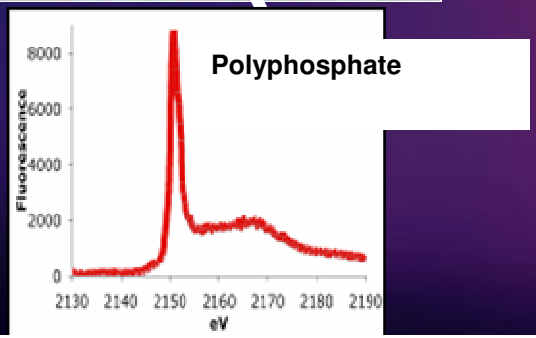
Red = phosphorus
Blue = aluminum
Green = magnesium





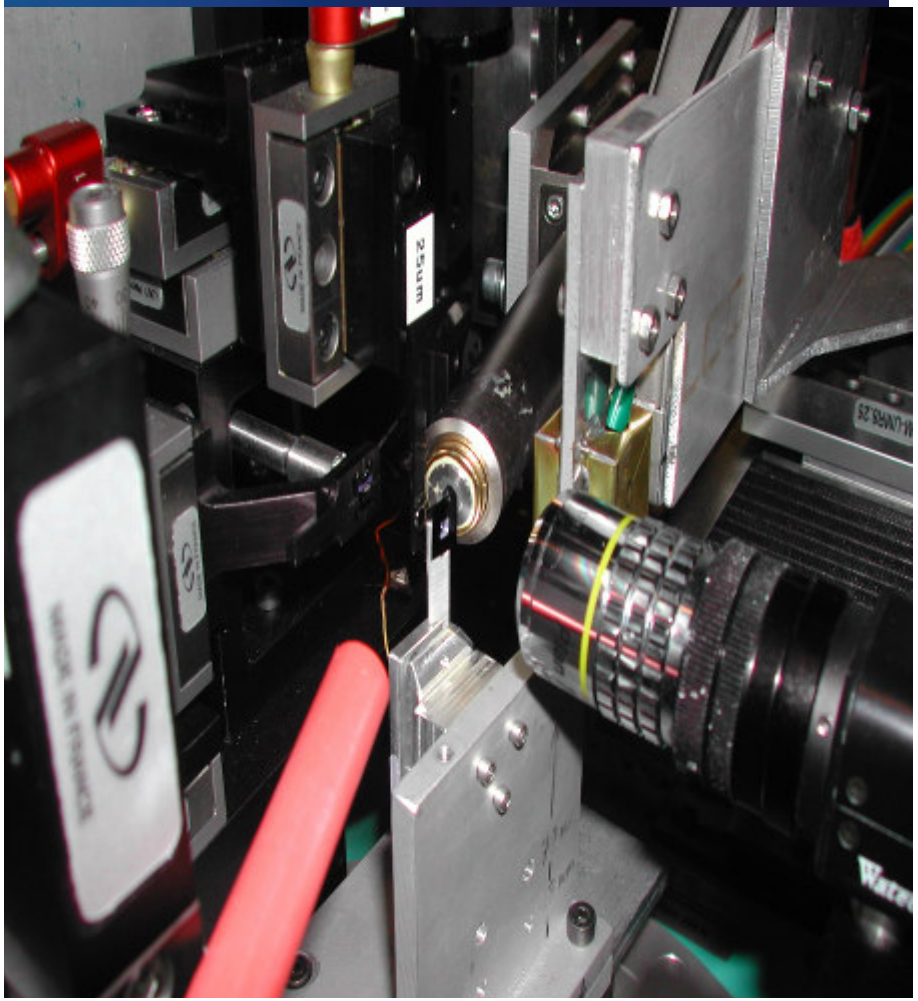


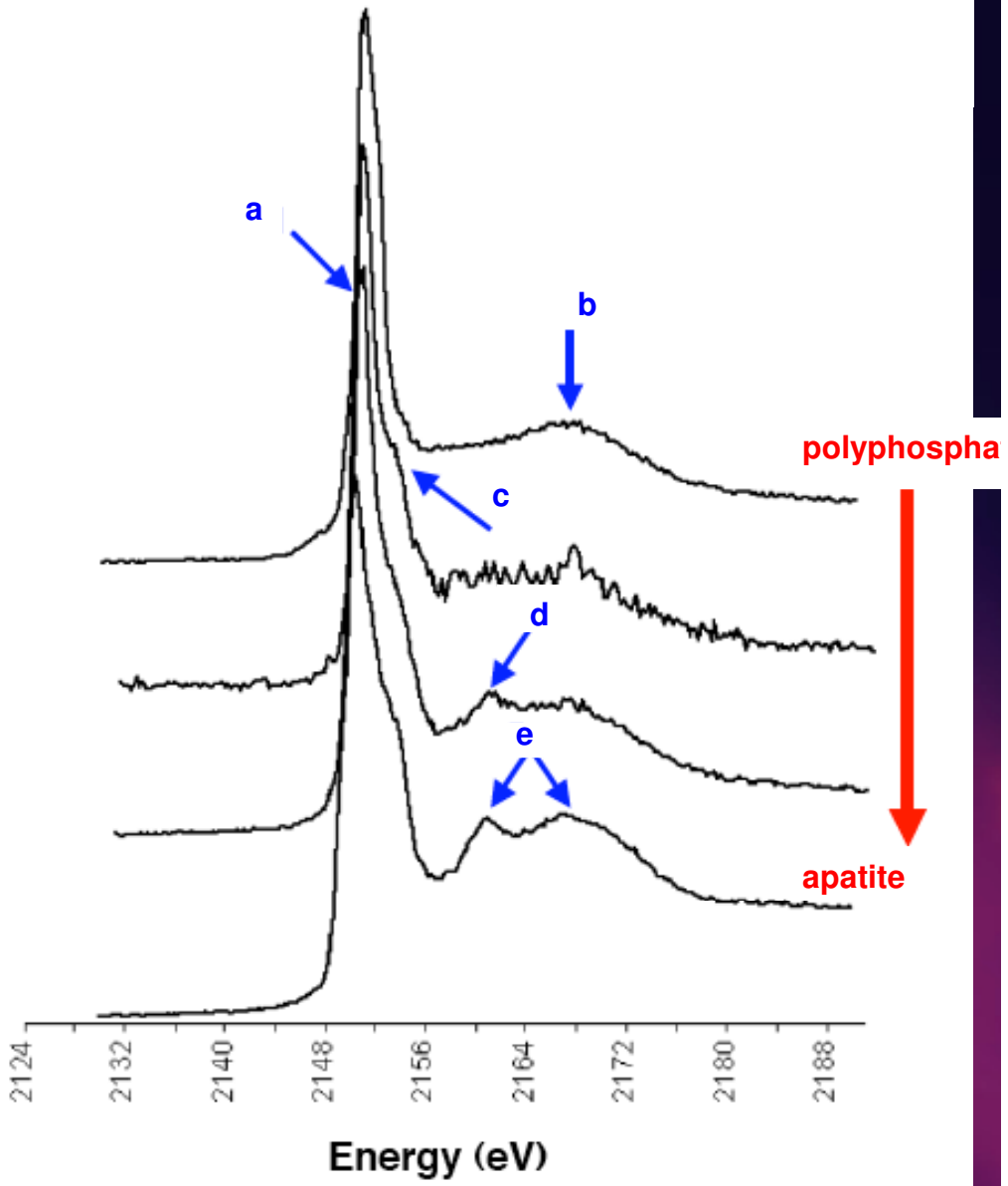




Diagenetic transformation of polyphosphate to apatite

Diaz et al Science , in press





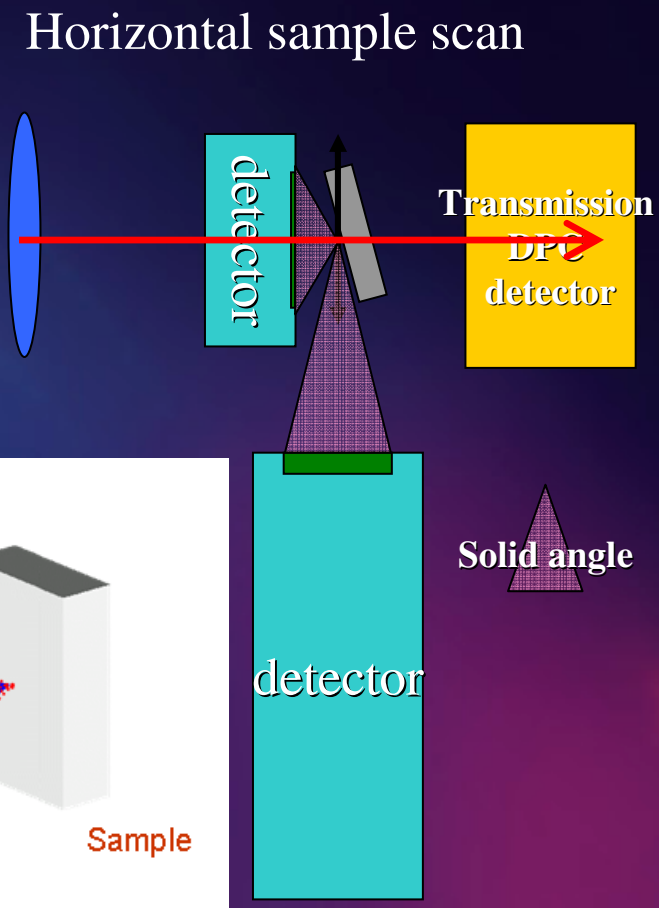
Detectors: optimum geometry for fluorescence detection

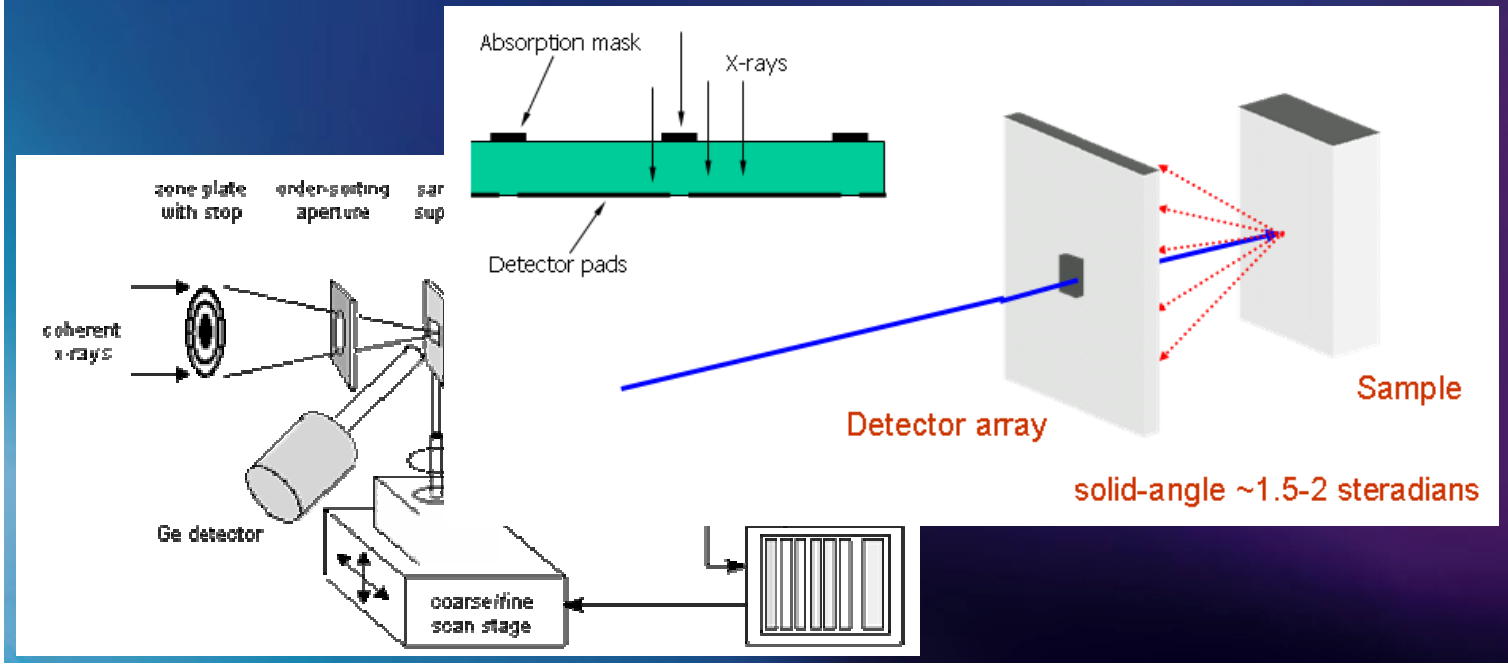
Traditional geometry

- Detector perpendicular to incident beam
- sample @ 75-45°
- Minimises elastic scatter detection
- Limits solid angle, lateral sample size and scan range

Annular geometry

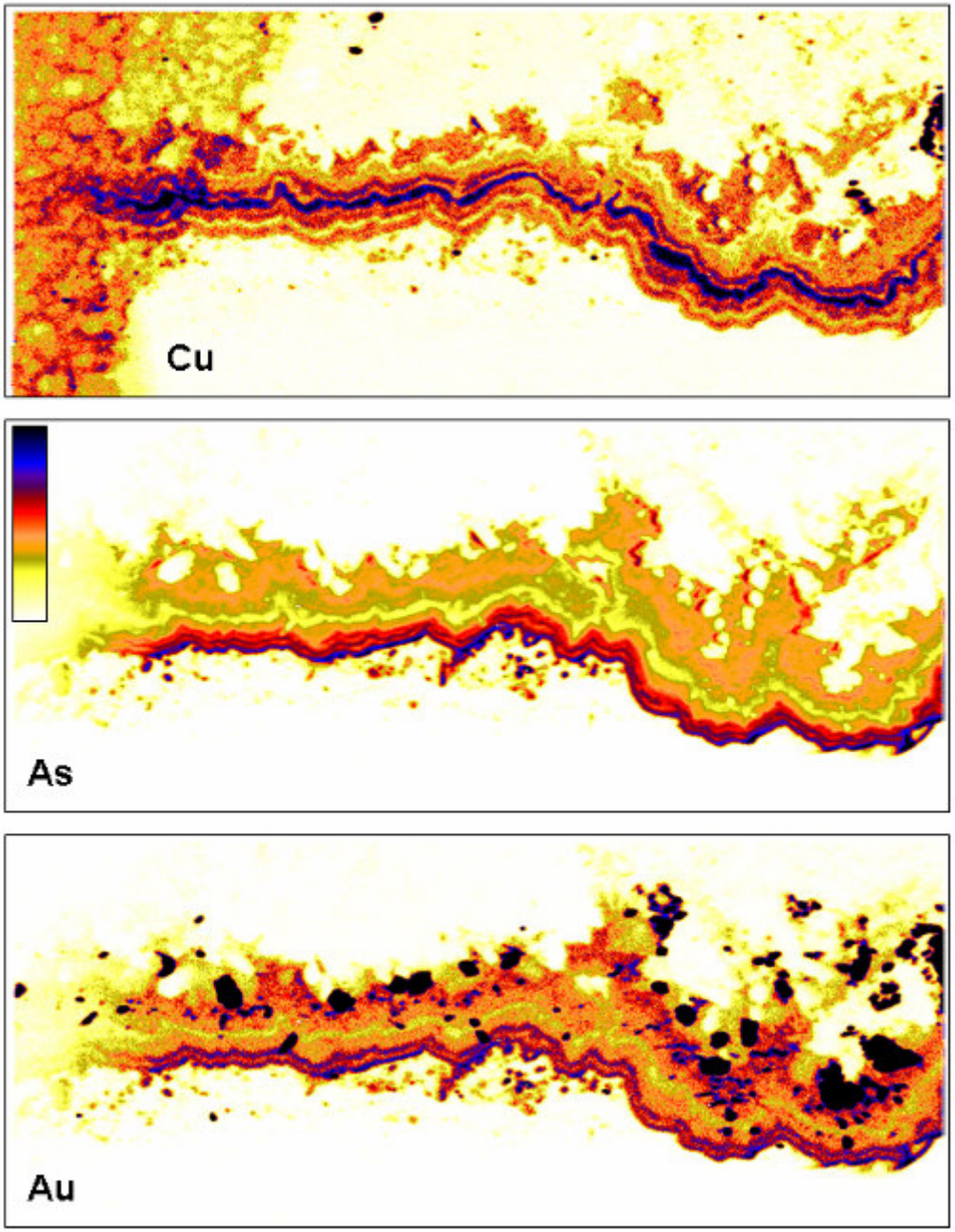
- Maximises solid angle, sample @ 90°
- No constraint on lateral sample size and scan range



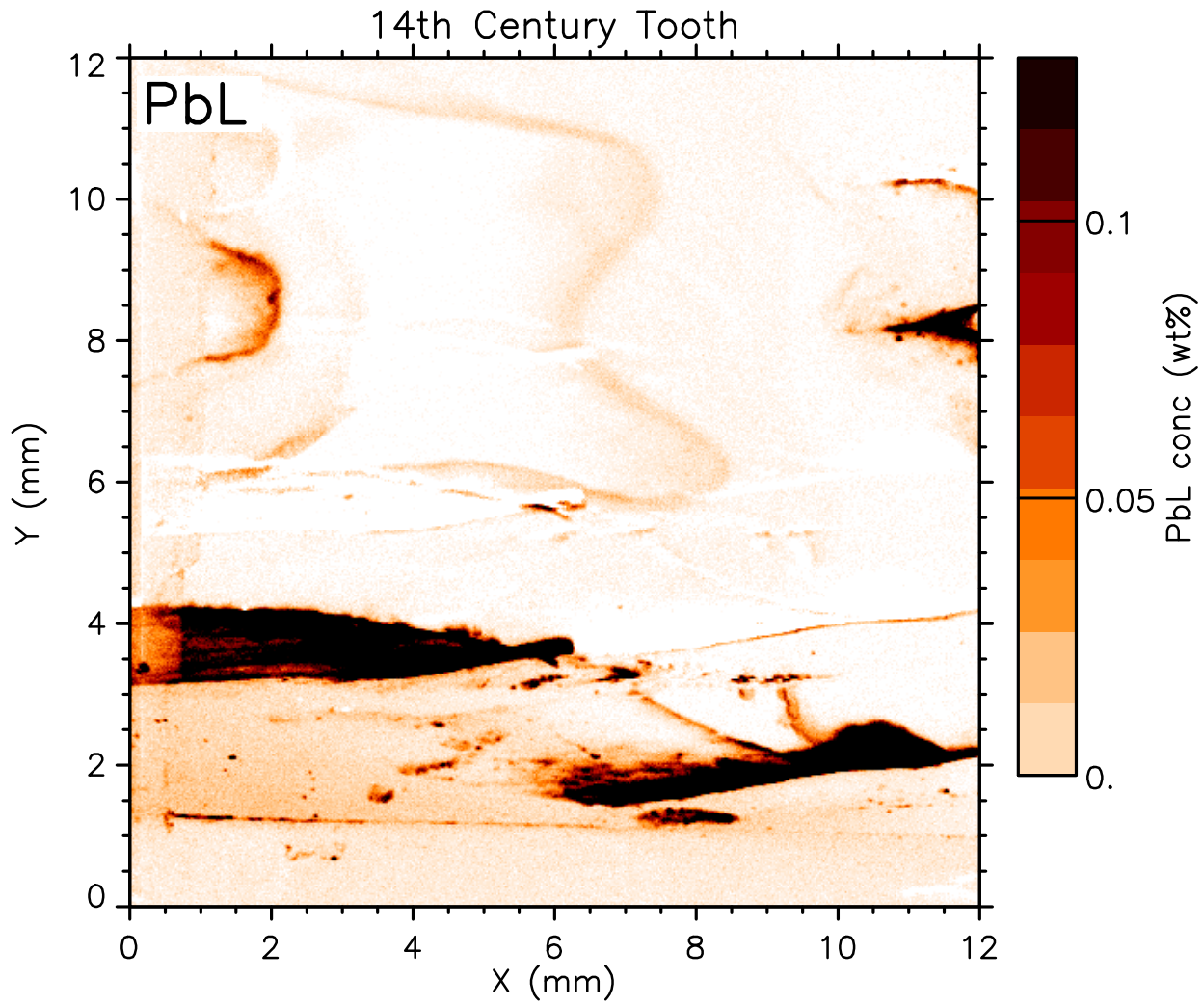


50 msec dwell Emperor pyrite 800 x 500 10 µm pixel acquisition time in 5.5 hours

Next generation x-ray microspectroscopy: Fluorescence tomography and



6 ms/pixel = 2000 X 2000 pixels in 6.7 hours



XANES image stack collected at 20 photon energies, each with 300 x 300 pixels at a dwell time of 10 ms could be achieved in 5 hours

Shades of things to come ...

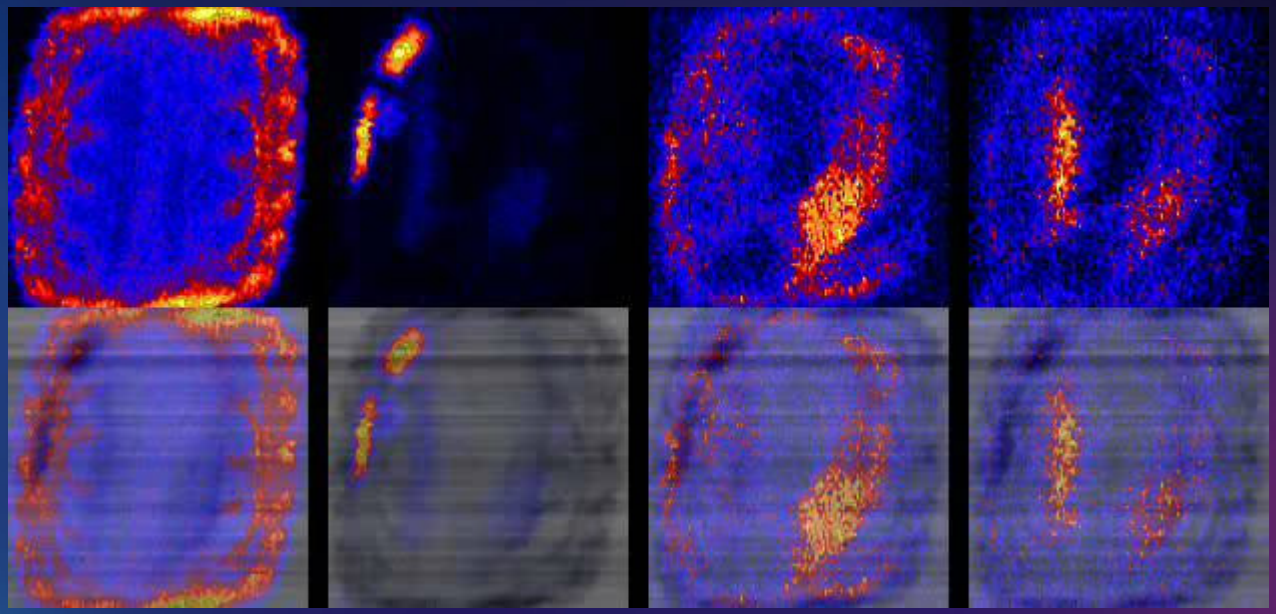
Iron oxide nodules, Rose Dam WA

5 ms dwell, 6 hours total scan time!

Fe-Y-Cu RGB composite (1500 x 2624 pixels, 13 x 21 mm^2)

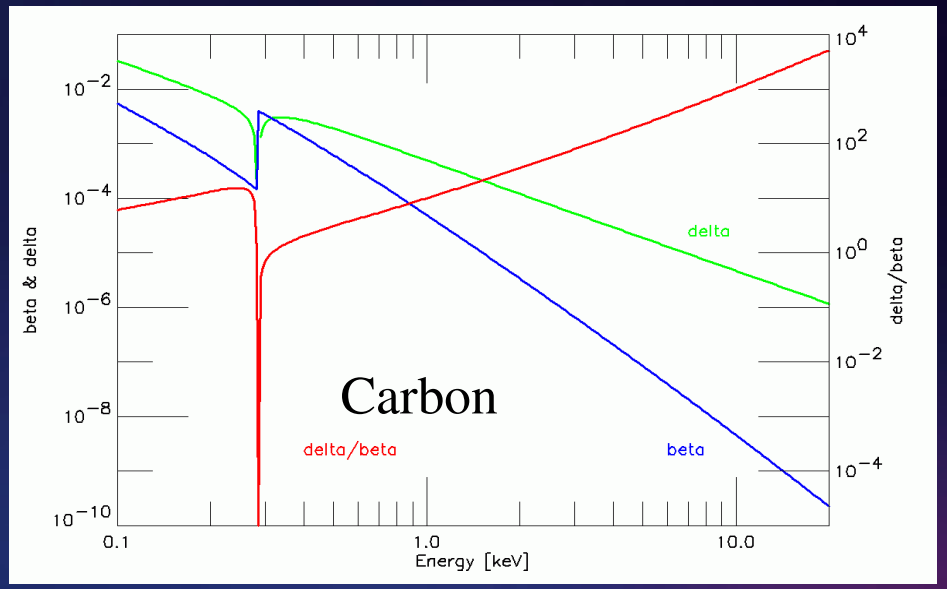
Australian Synchrotron

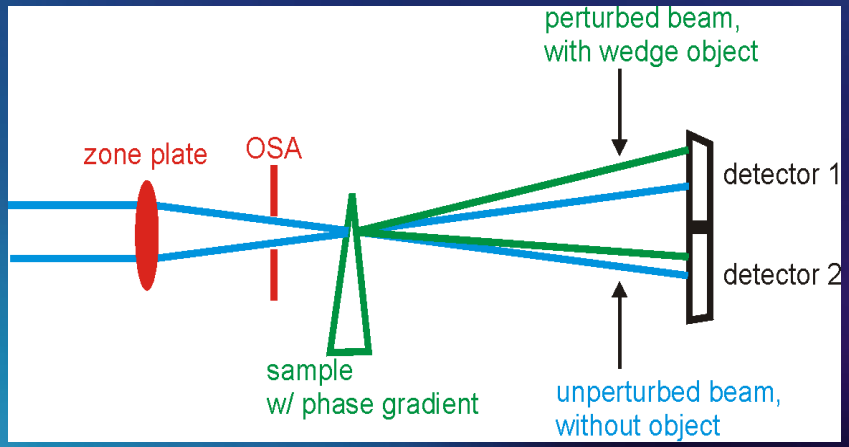
Fluorescence tomography

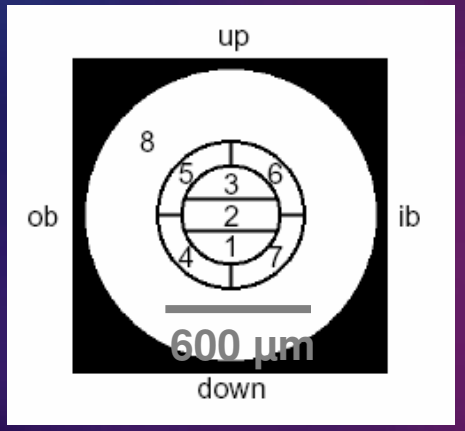


Detectors: Differential Phase Contrast

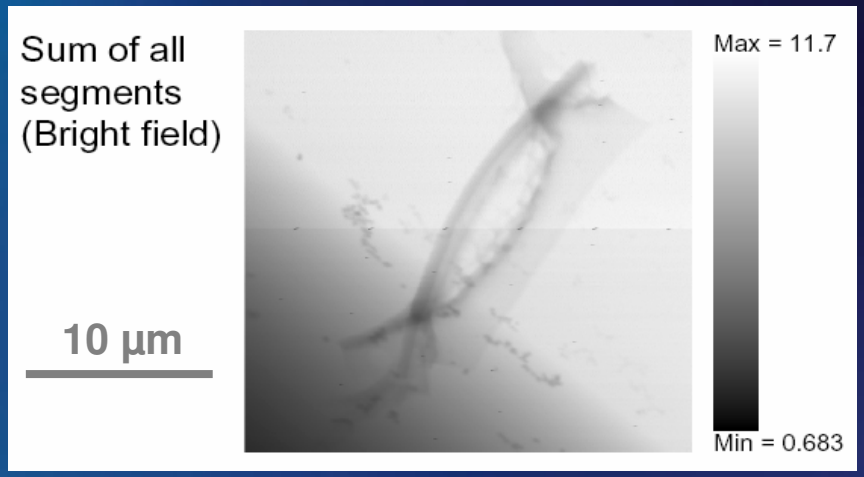
Why phase contrast?

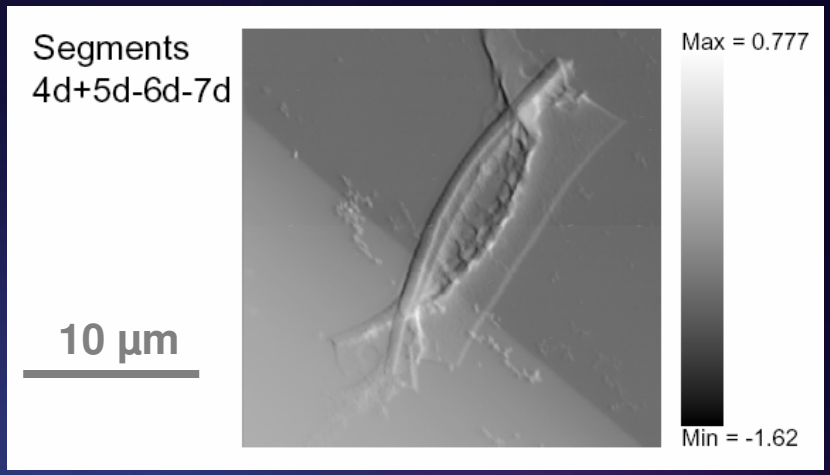






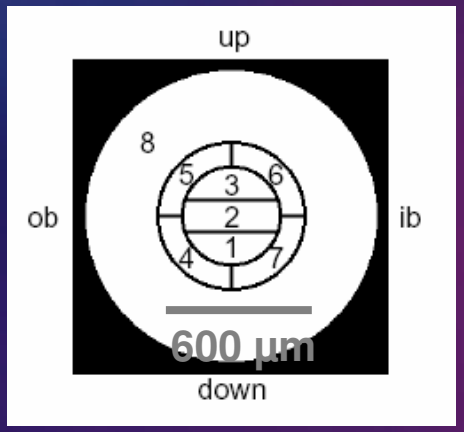
Phase contrast imaging





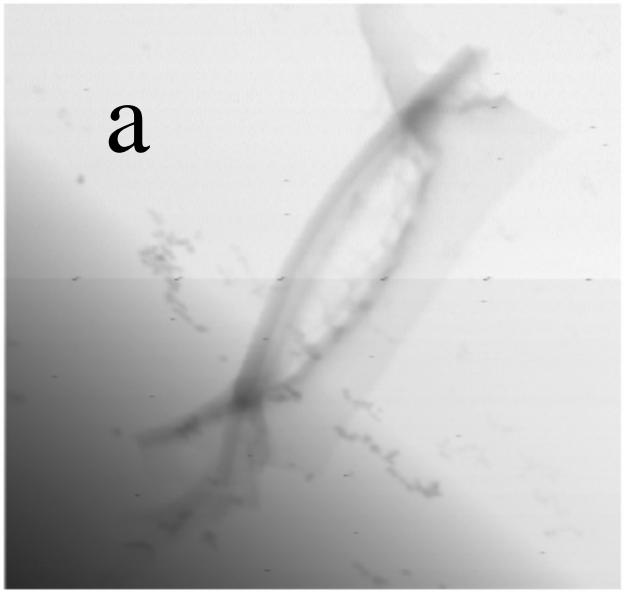
Trials @ APS 2-ID-B

- 2 msec dwell, sub-msec achievable
- Diatom @ 1790 eV fly scan
- 25 nm pixel, 1000 X 1000 pixels, 40 min
- Xradia 50 nm zone plate



Phase contrast imaging - intermediate energy

Sum of all segments (Bright field)

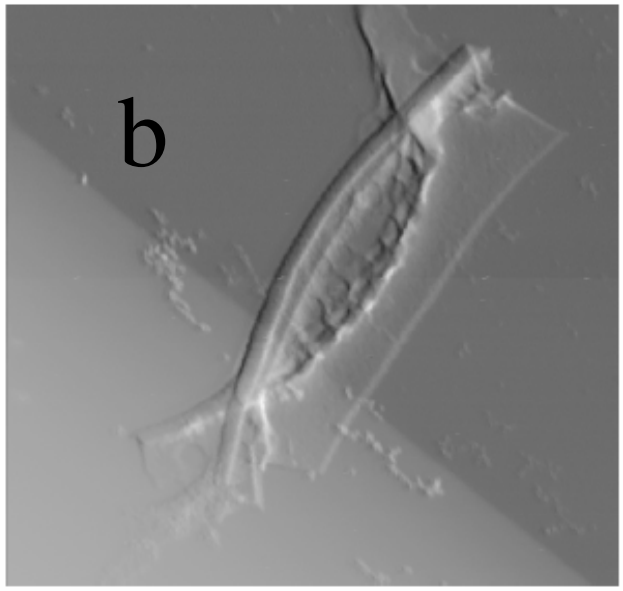


Max = 11.7

Min = 0.683

10 µm

Segments 4d+5d-6d-7d

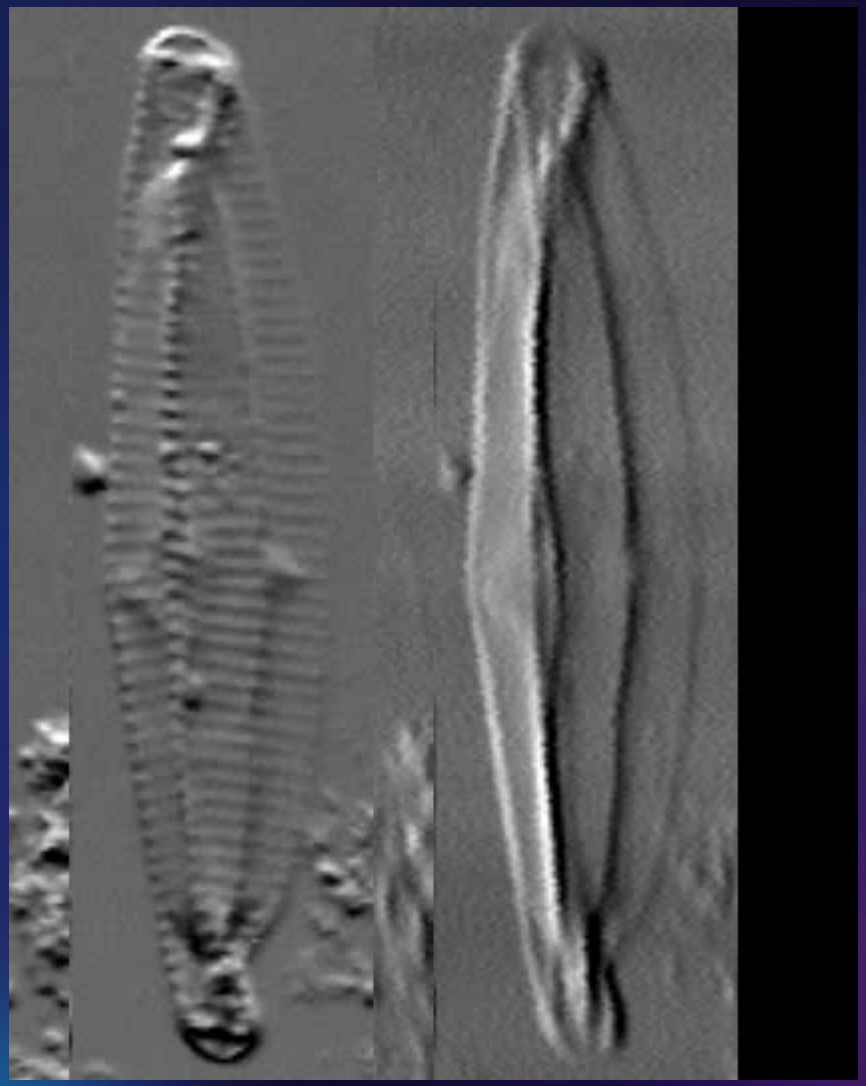


Max = 0.777

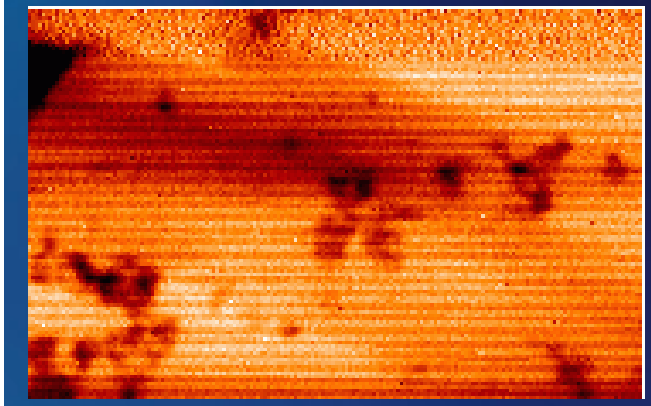
Min = -1.62

10 µm

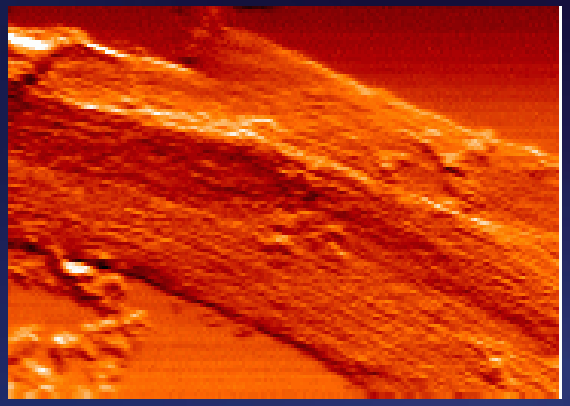
DPC tomography



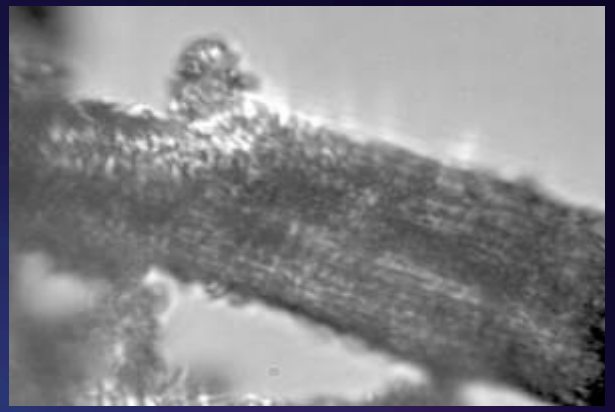
Hard x-ray example - differential phase contrast



ABS



DPC



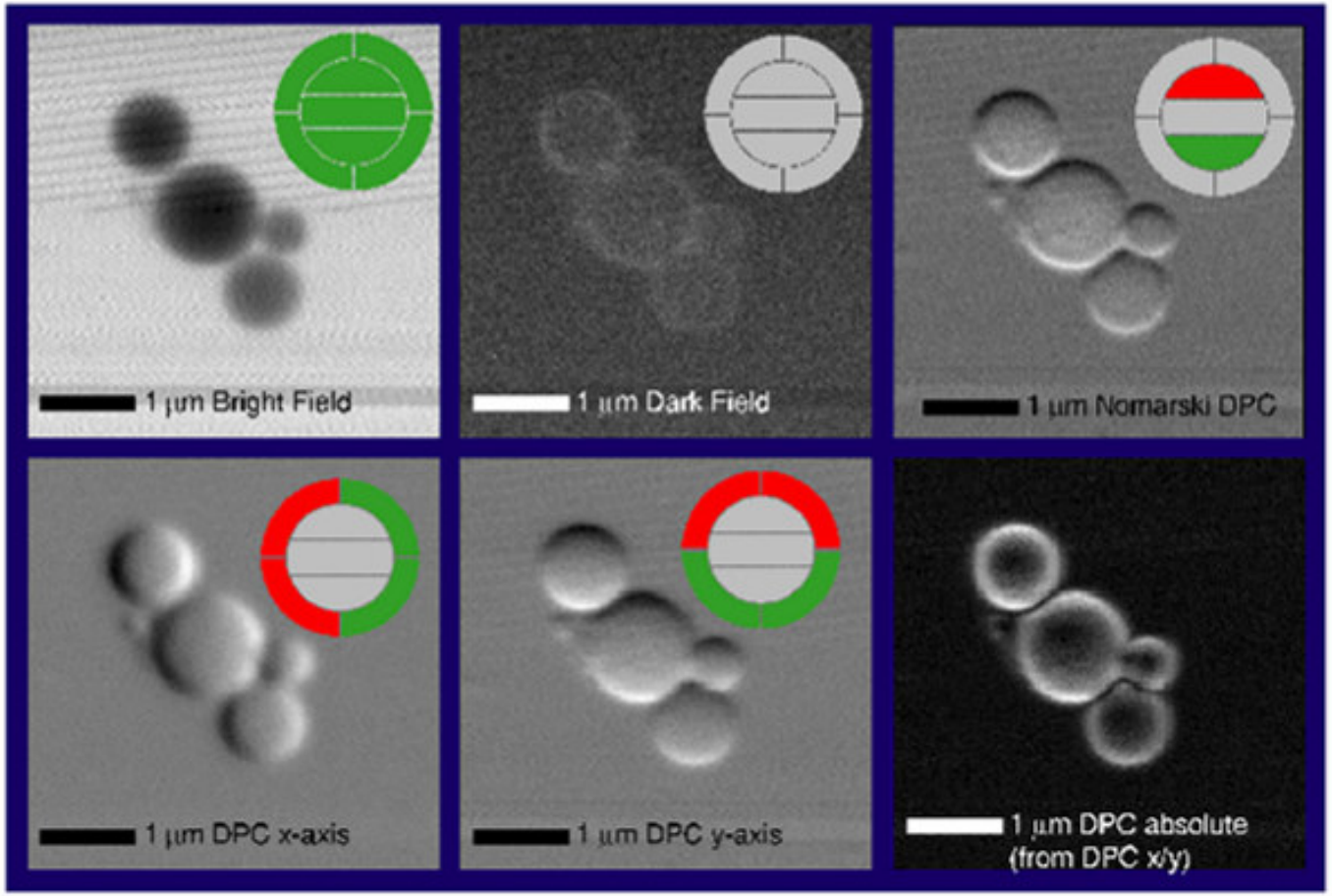
VLM

Scans of a cardiac myocyte, taken at 10 keV incident X-ray energy at 2-ID-E:
ABS absorption contrast image showing mostly salt deposits that remained from sample preparation, note beam noise and intensity fluctuations
DPC differential phase contrast - visualizes the biological structure of the cell
VLM visible light micrograph of the same cell
DPC image gives significantly better representation of the biological mass of the specimen than the absorption contrast, and self normalizes against intensity fluctuations, ideal for fast fly scans

Advantages

Simultaneous Availability Of Contrast Modes

- Silica spheres 1 μm diameter or less
- Differential phase contrast filters out intensity fluctuations of the source!



How to correlate element distribution with biological structure ?

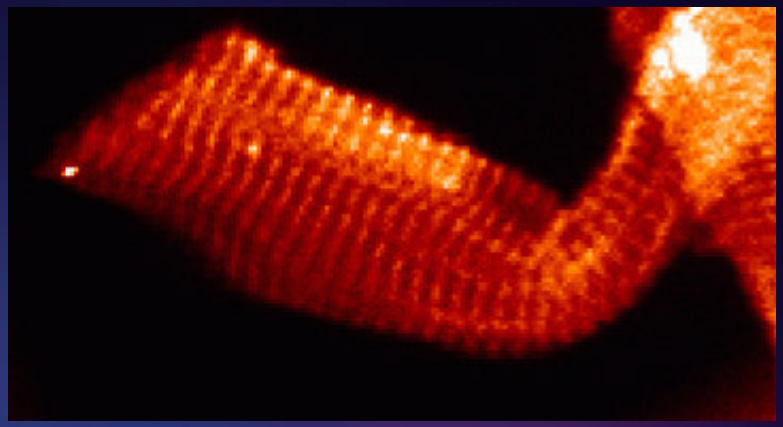
Hard X-ray microscopy: great sensitivity for medium/high Z elements,

but mapping of biological mass and structure (mostly C,N,H) difficult:

very low photoelectric absorption (hard x-rays !)

very low fluorescence yield

Zn fluorescence

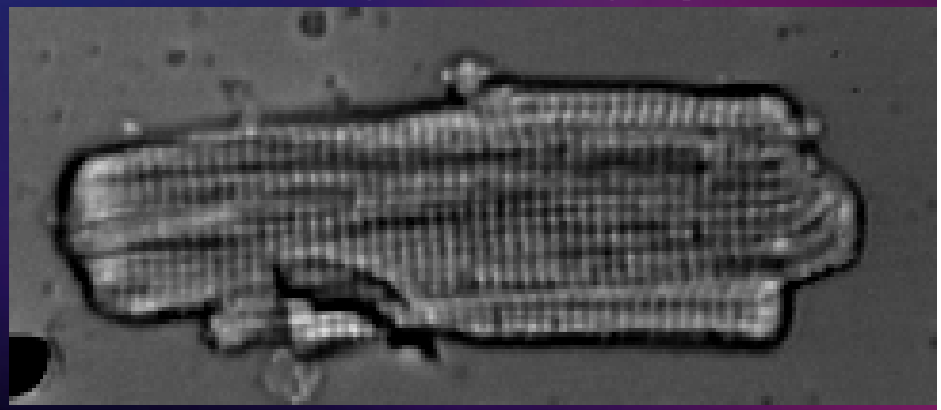


Are these the same striations ???

at the same time:

• exact correlation of elemental maps with biological structure critical !!

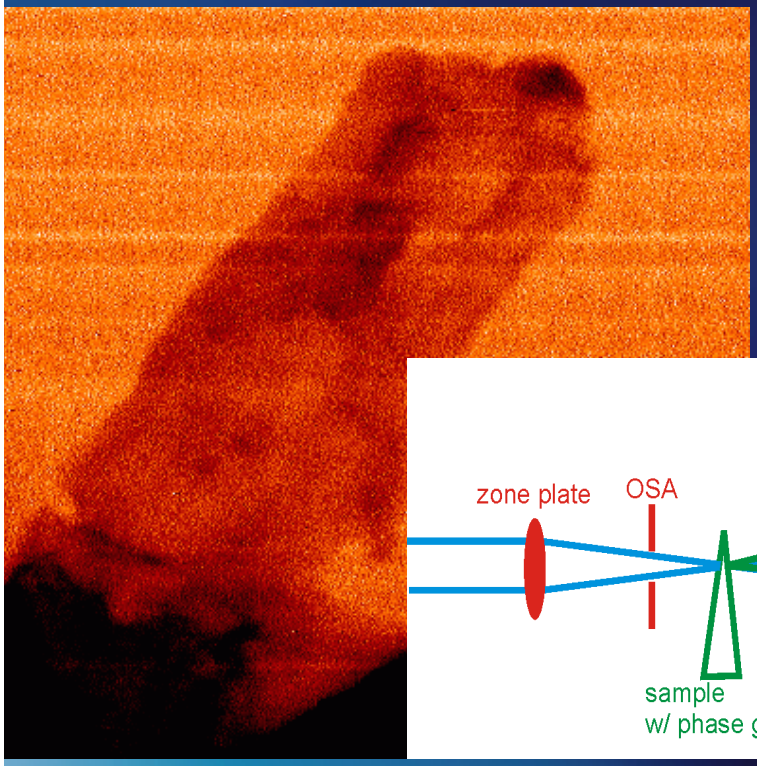
Visible light micrograph

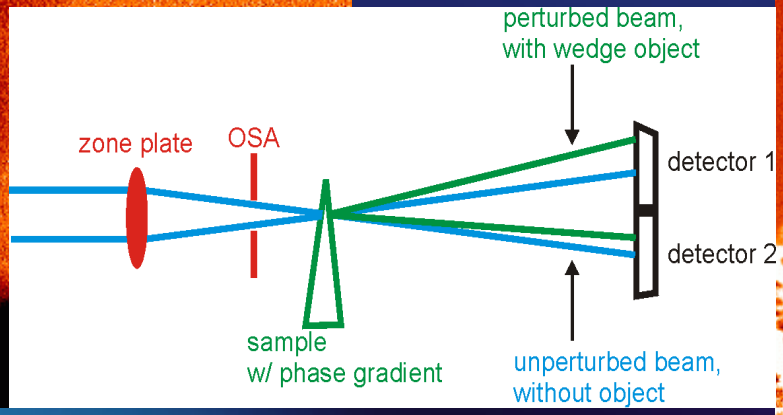


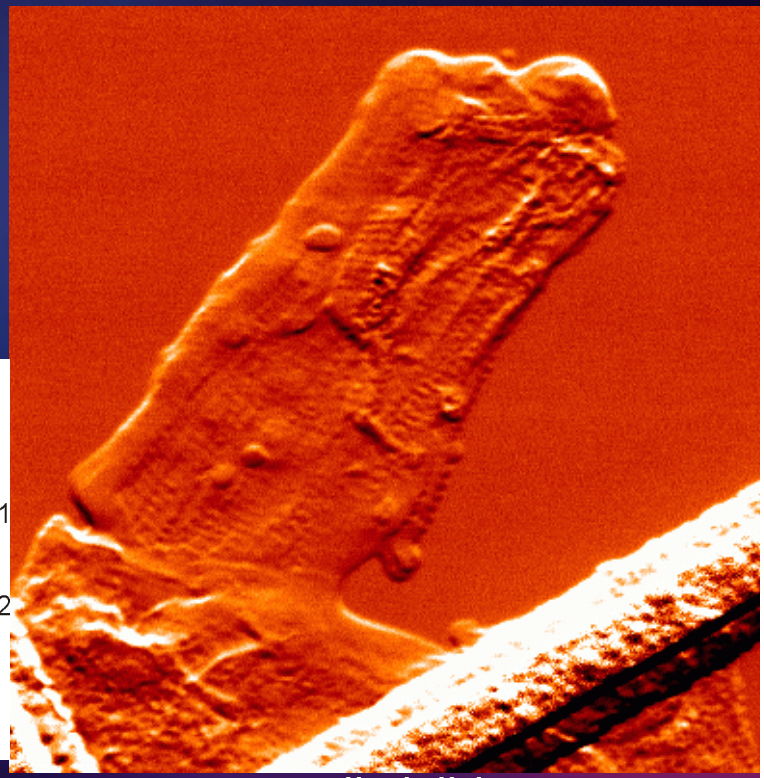
Differential Phase Contrast (DPC) for hard X-ray microscopy

detect phase contrast in addition to absorption contrast

- for fast scans for targeting
- to acquire structural, C-based information from the specimen







Here, striations in heart muscle cells due to sarcomere pattern are well visible
We will use DPC routinely to locate cells and low contrast samples

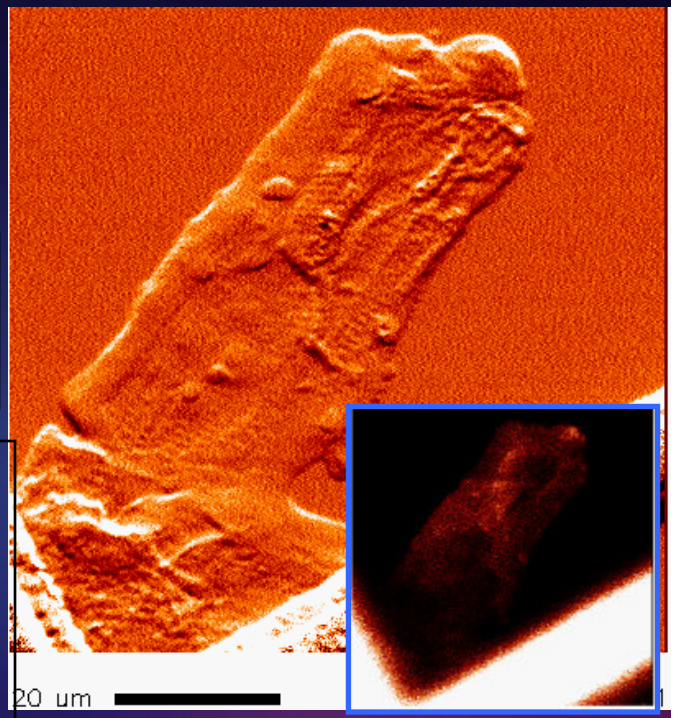
Australian Synchrotron

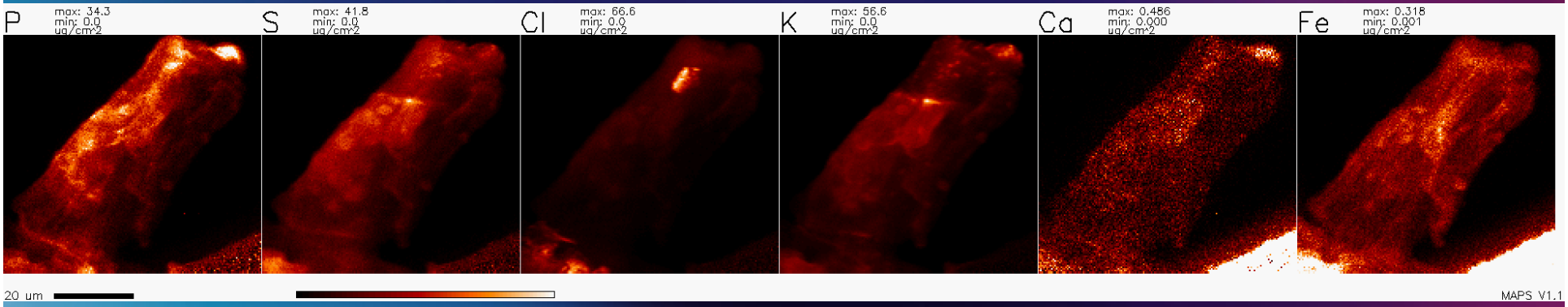
Differential Phase Contrast to co-localize structure with elemental content & acquire fast overview scans in scanning microprobes

to visualize cell structure in hard X-ray microscopy, use phase contrast instead of absorption, e.g., for scanning probe: differential phase contrast

bottom: elemental maps of myocyte acquired using the exact coordinates from the preview scan. Scan time: 10h

right: Targeting scan of a cardiac myocyte, acquired in 15 min. The DPC image shows striations caused by the regular arrangement of myofilaments. Inset: total fluorescence yield gives a preview of elemental content





S. Vogt, B. Hornberger, M. Feser, B. Palmer, D. Legnini, C. Jacobsen, J. Kirz, J. Maser

Acknowledgements

Chris Ryan CSIRO
David Cohen ANSTO
Martin de Jonge, Jonathan McKinlay Australian Synchrotron
Jörg Maser, Stefan Vogt, Ian McNulty, Barry Lai APS

- Invitation to postdocs and students interested in studying at the Australia Synchrotron

- Contact the relevant beamline scientist
  david.paterson@synchrotron.org.au

Australian Synchrotron

www.synchrotron.org.au

Initial Beamlines

1.	High-throughput protein crystallography	3-BM1	5–23 keV	
2.	Protein microcrystal & small molecule	3-ID	5.5–20 keV	
3.	Powder X-ray diffraction	10-BM1	4–37 keV	(0.41-3.1 Å)
4.	Small and wide angle X-ray scattering	13-ID	5.5–20 keV	
5.	X-ray absorption spectroscopy	12-ID	4–65 keV	
6.	Soft X-ray spectroscopy	14-ID	0.1–2.5 keV	
7.	Vacuum ultraviolet (VUV)	ID	10–350 eV	
8.	Infrared spectroscopy	1-BM	0.001–1 eV	(2–10,000 cm^{-1})
9.	**Microspectroscopy**	**5-ID**	**4.5–20 keV**	
10.	Imaging & medical therapy	8-ID	10–65 keV	
11.	Microdiffraction and fluorescence probe	BM	4–37 keV	
12.	Circular dichroism	BM	2–10 eV	
13.	Lithography	BM	2–25 keV	

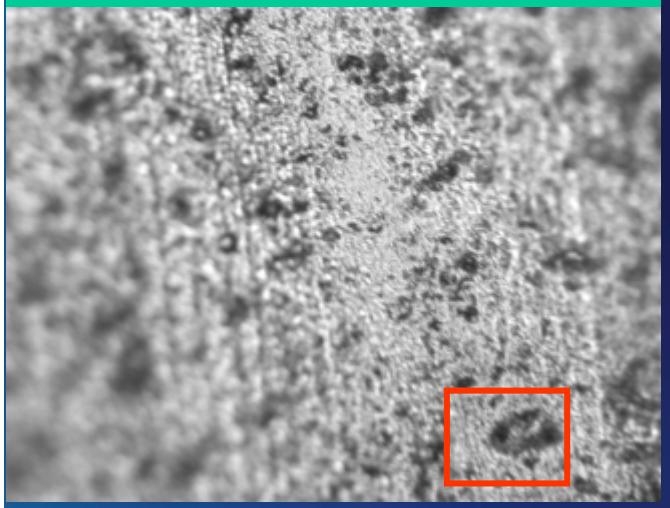
ID = Insertion Device

BM = Bending Magnet

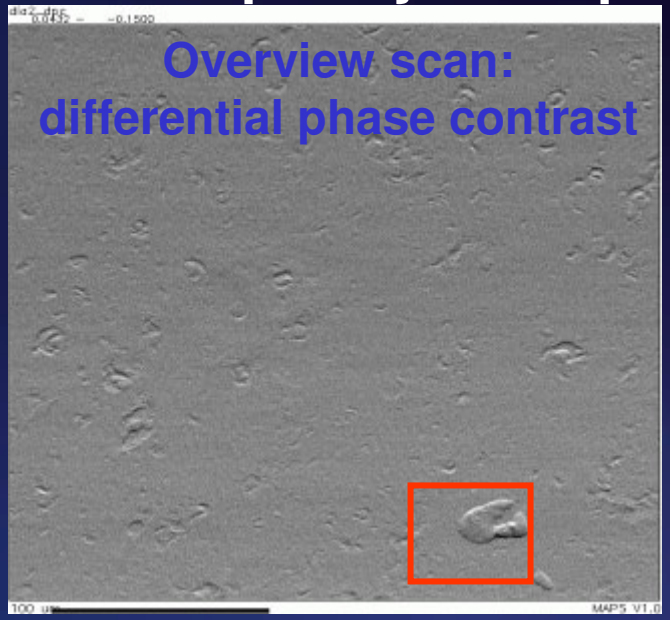
Australian Synchrotron

DPC and total fluorescence to quickly find specific sample areas

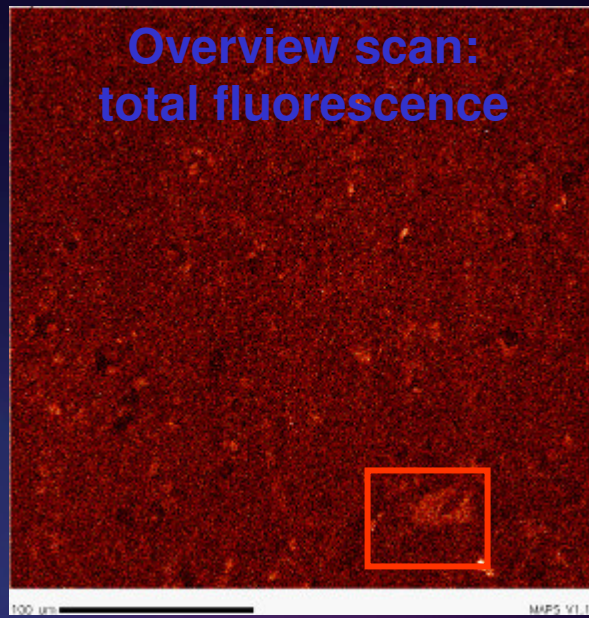
visible light microscope



Overview scan: differential phase contrast

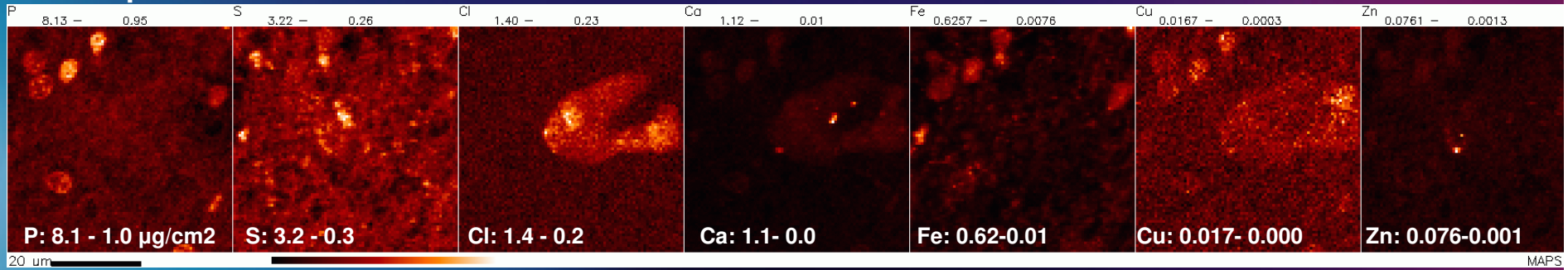


Overview scan: total fluorescence



TOP: Samples are previewed in the light microscope. Potentially interesting areas are then fast-scanned in the microprobe (e.g. 300x300 micron area scan of a human brain tissue section, acquired in 45 min), DPC shows specimen structure, and TFY gives elemental content preview. Based on this, a target area for slow steps scans is chosen.

BOTTOM: elemental maps of a selected 50x50 micron area, acquired in 3h using slow step scan modes. The P maps shows several cell nuclei.



S. Vogt, B. Hornberger, M. Feser, E. Bigio, D. Legnini, T. Paunesku, C. Jacobsen, J. Kirz, J. Maser