The Maia detector at PETRA III P06
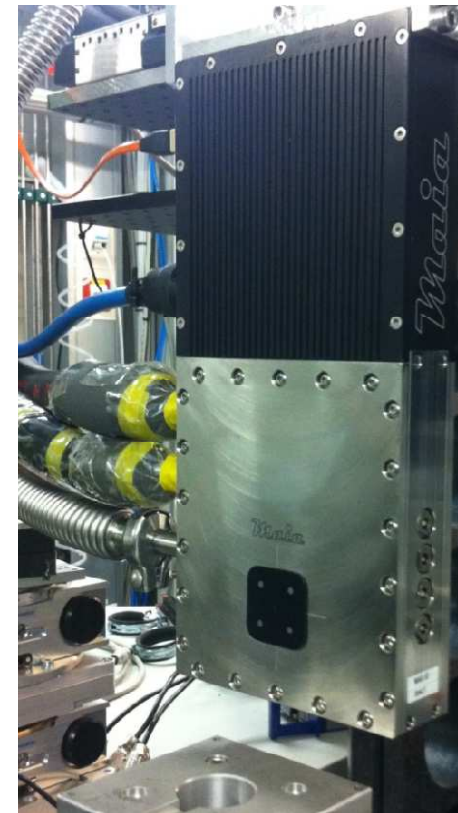
Gerald Falkenberg
Deutsches Elektronen-Synchrotron DESY,
Hamburg, Germany

HELMHOLTZ | ASSOCIATION

DESY

Outline

> The Hard X-ray Micro/Nano-Probe beamline P06 and Maia

> Brief characterization of the Maia detector system

> Application examples: large 2D, cryo-XRF Tomo, Xanes-imaging

> Conclusions

Acknowledgements
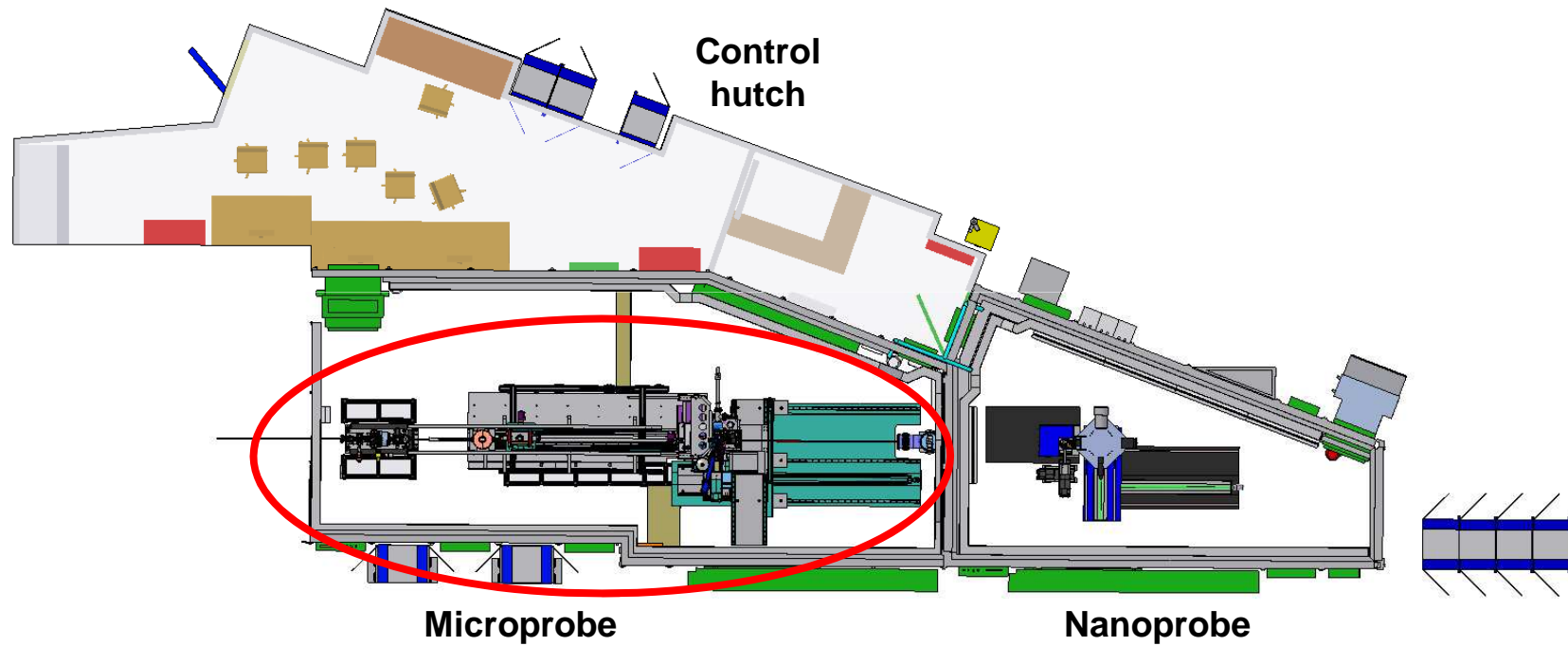
Beamline P06

Experimental hutches of the Hard X-ray Micro/Nano-Probe beamline P06

Beamline P06: Microprobe Experiment

Detectors:

Si Drift (SII Vortex 1+4 element)
Si(Li), HPGe
High-res. CCD (PCO4000)
MAR 165
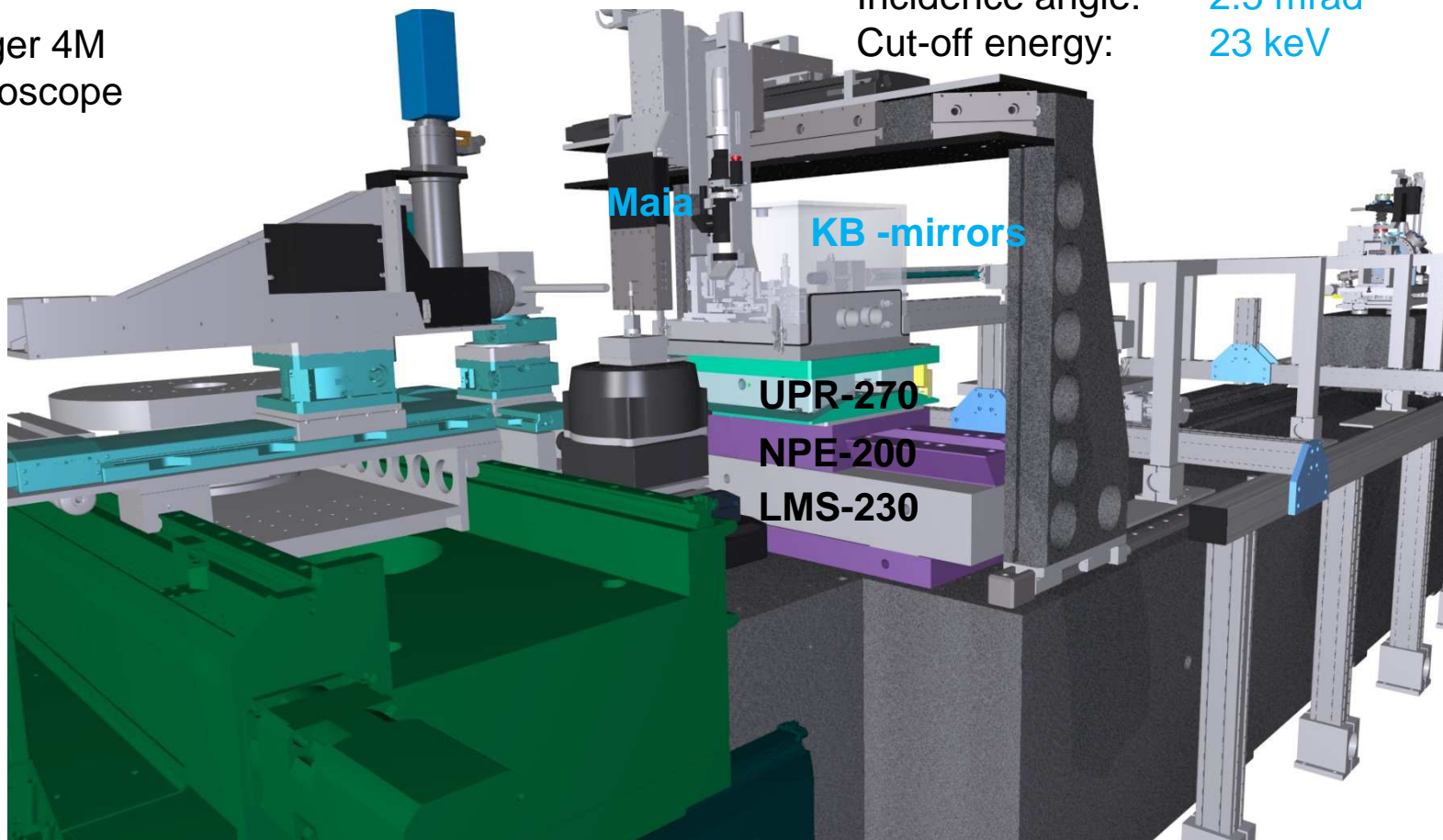Pilatus, Eiger 4M
Video-microscope
Maia

KB-mirrors

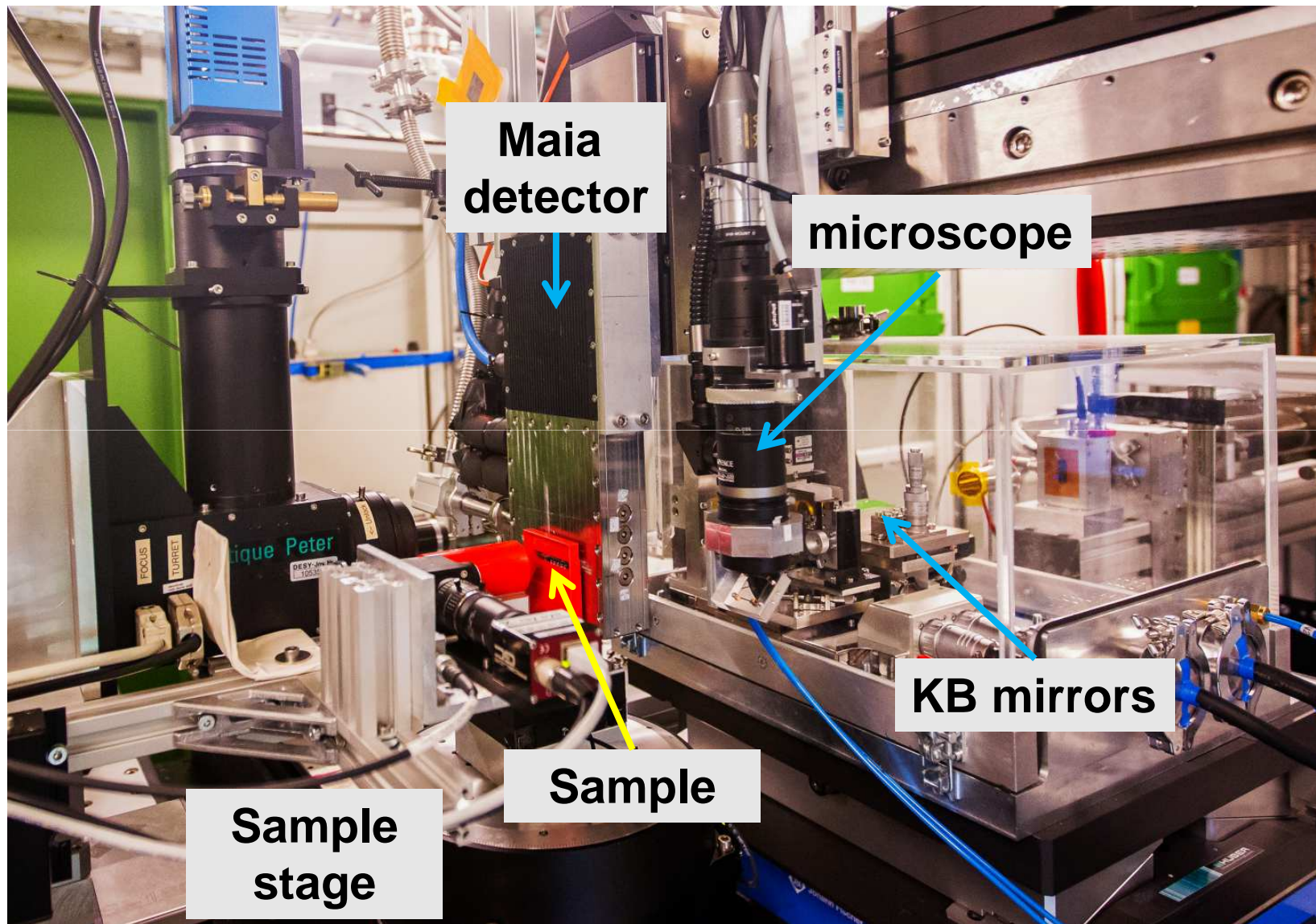Fixed elliptical shape (JTEC)
Shape error: < 2nm (peak-valley
Working distance: 200 mm
Incidence angle: 2.5 mrad
Cut-off energy: 23 keV

Maia

KB -mirrors

UPR-270

NPE-200

LMS-230

Beamline P06: Microprobe Experiment

Maia detector

microscope

Sample

Sample stage

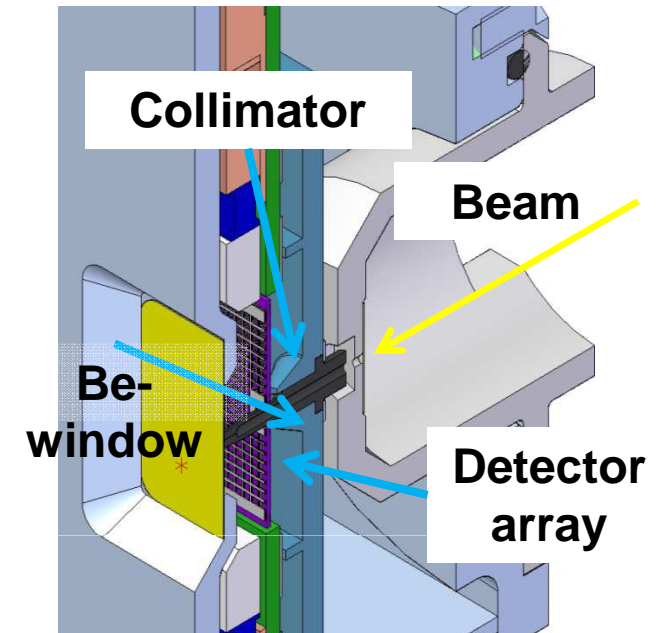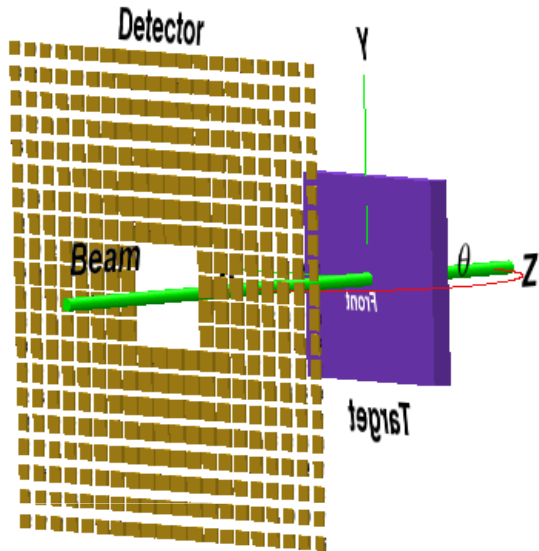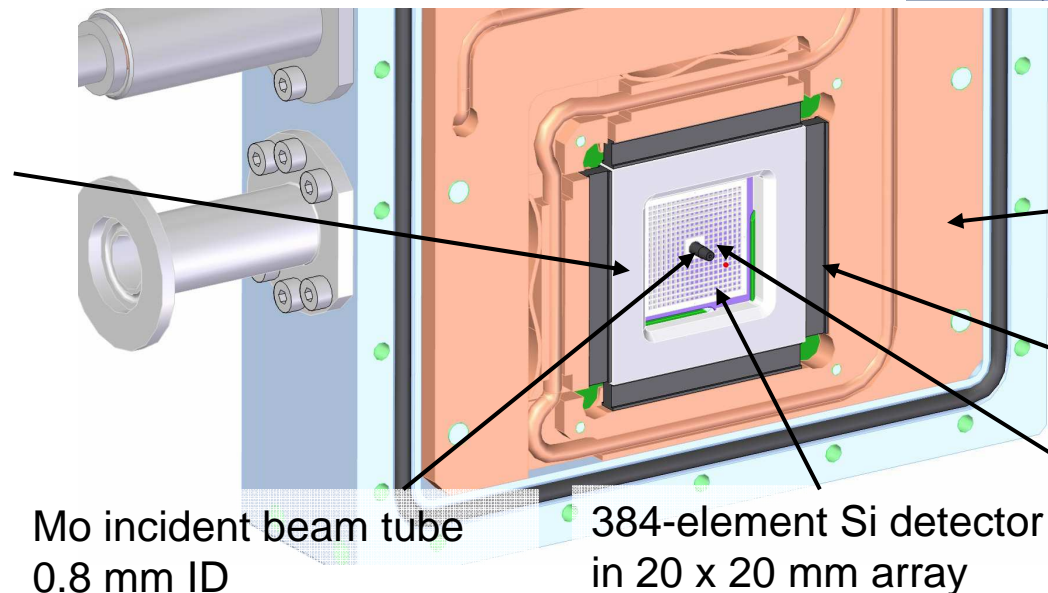KB mirrors

The Maia Detector

Maia

386 detector elements
1 mm^2 active area/element
Solid angle ~1.3 sr
Count rate: 50 Mio photons/s
Typical dwell time: 1ms/pixel
Energy resolution: 260eV

Detector
Beam
Front
Target

Collimator
Beam
Be-window
Detector array

AlN detector support frame

Cu cooling/support block

Peltier coolers

Mo charge-sharing suppression mask

Mo incident beam tube
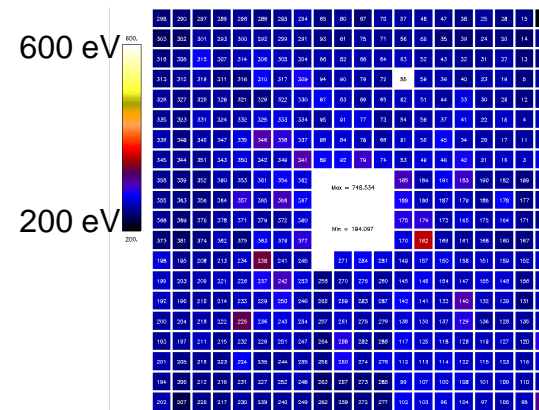0.8 mm ID

384-element Si detector
in 20 x 20 mm array
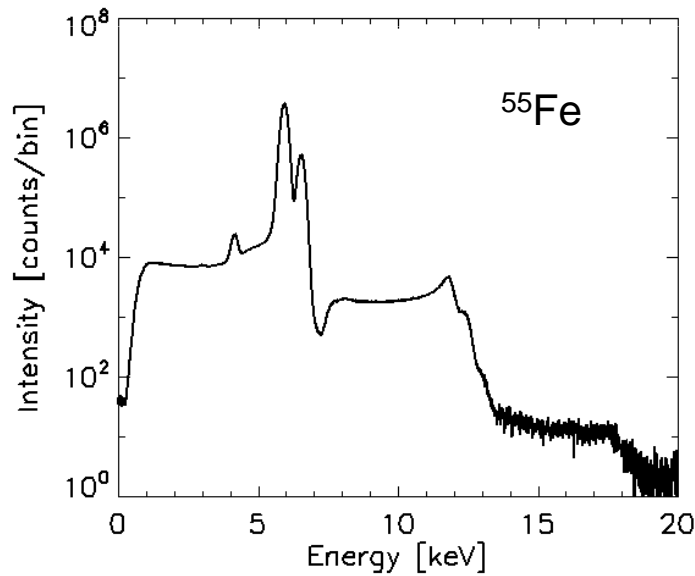
Energy resolution (Maia C type)

Tested with a ^{55}Fe source (PETRA III SHUTDOWN)

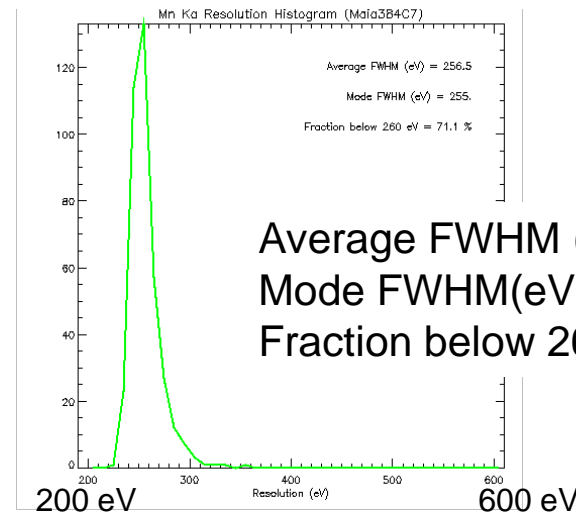Each detector pad is individually trimmed, linearized, calibrated…

Energy resolution of the individual detector elements

600 eV

200 eV

Sum spectrum over all detector elements

^{55}Fe

Mn Kα resolution Histogram

200 eV   600 eV

Average FWHM (eV) =256.5
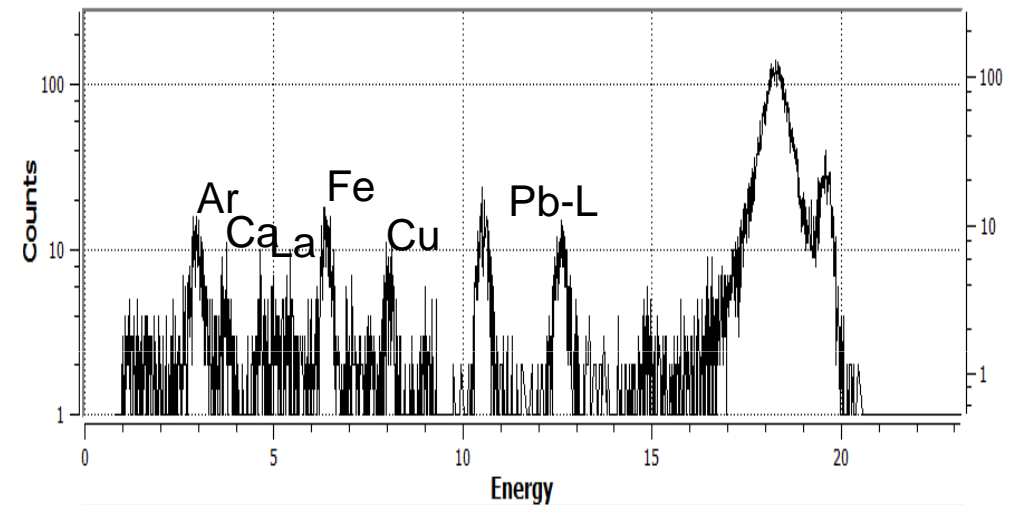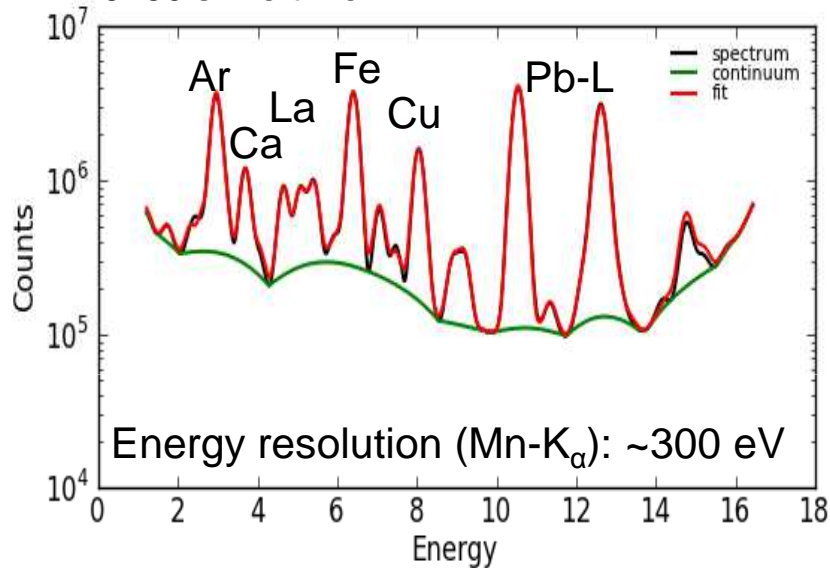Mode FWHM(eV) =255
Fraction below 260eV =71.1%

Limits of detection (Maia B type)

AXO thin film standard @ 19.5 keV

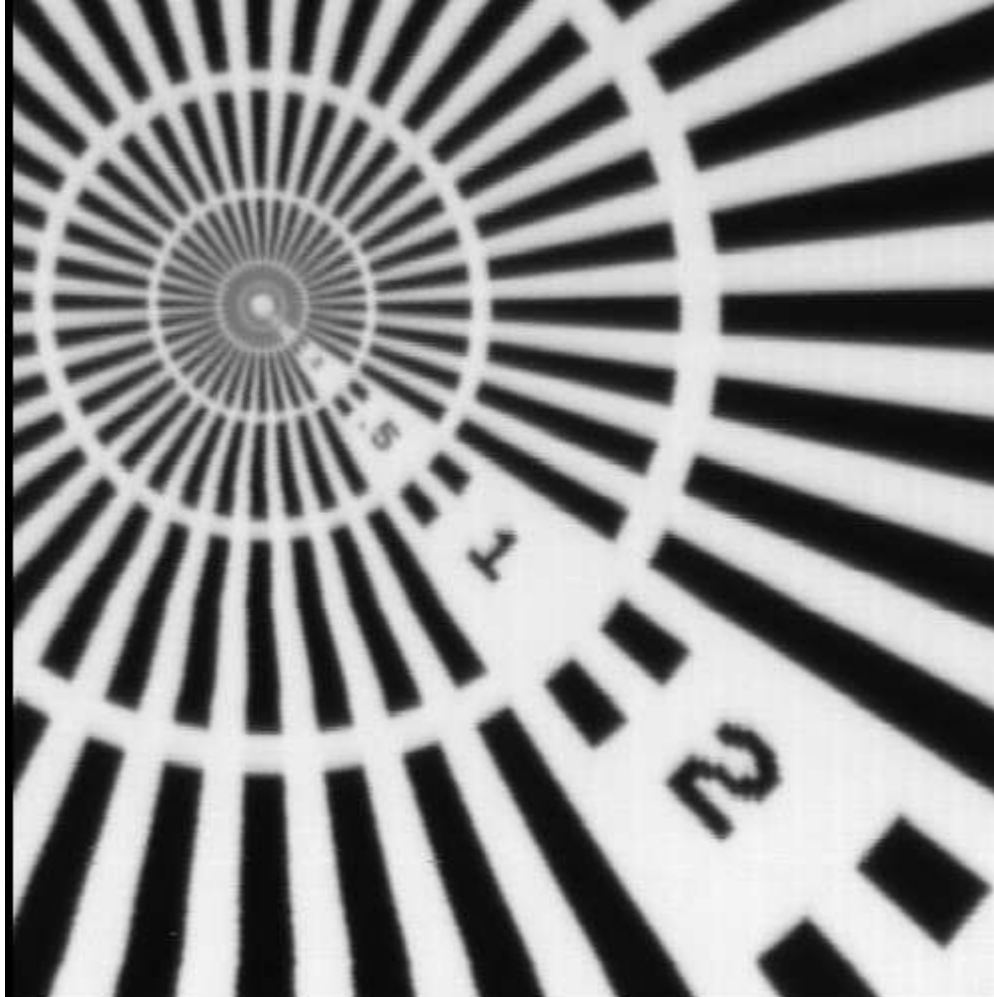**Sum spectrum** of 1904 x 1370 scanned pixels, 9130 s live time

Random **single pixel spectrum**, 3.5 ms dwell time

Energy resolution (Mn-K_α): ~300 eV

Element	Concentration [ng/mm^2]	LOD [ng/mm^2] for 1 s	LOD [ng/mm^2] for 1 ms
Pb-L$_\alpha$ (10.6 keV)	7.7±1.3	0.045	1.4
La-L$_a$ (4.6 keV)	9.0±1.9	0.880	28
Cu (8.0 keV)	2.4±0.5	0.047	1.6
Fe (6.4 keV)	4.0±0.4	0.110	3.4
Ca (3.7 keV)	11.4±5.5	0.760	24

The Maia detector system: fast scanning performance

100nm pitch 100ms dwell time

MAIA controller combines
- XRF data
- encoder position
- flux monitor

Limited Performance at dwell times smaller 1 ms propably caused by limited stiffness of sample (-stage) mount
-> use interferometer on sample position

GeoPIXE

GeoPIXE - Quantitative PIXE/SXRF Imaging and Analysis

GeoPIXE

The control hard- and software of the Maia allows for the fast acquisition of elemental distribution images (less than 1 ms dwell time).

It includes the GeoPIXE software package. It is an integral part of the Maia system and allows for:

- Real time processing of the XRF data acquired by 384 detector elements via dynamic analysis
- Correction for variation of dwell time, primary flux, dead-time and pileup
- Export of high quality images
- Quantification of XRF data

C.G. Ryan, M. Jensen, B.E. Etschmann, D.R. Cousens
CSIRO Earth Science and Resource Engineering
CSIRO (c) 1987-2011

Continue

C.G. Ryan, D.R. Cousens, S.H. Sie, W.L. Griffin, G.F. Suter, E. Clayton, Nucl. Instrum. Methods Phys. Res., Sect. B 47 (1990) 55-71.

Application Examples

> Fast 2D XRF imaging: U in freeze dried fish gill

> Cryo-XRF tomography of shock-frozen *Ceratophyllum demersum*

> *XANES imaging: degradation of Lead chromate yellow*

Fast 2D XRF imaging: U uptake in Salmon gills

Backscatter

2230 x 0.5 µm

8000 x 0.5 µm

Question: Is U actively taken up in the gills of Atlantic Salmon (*Salmo Salar*)?

- Atlantic Salmon was exposed for 96 hours to U (6 mg/L)

- Gills were removed from the fish and freeze dried

- Scanned with Maia at P06:

    - 0.5x0.5 µm step size

    - 4x1.165 mm^2 Area

    - 1.2 ms dwell time

    - 18 keV
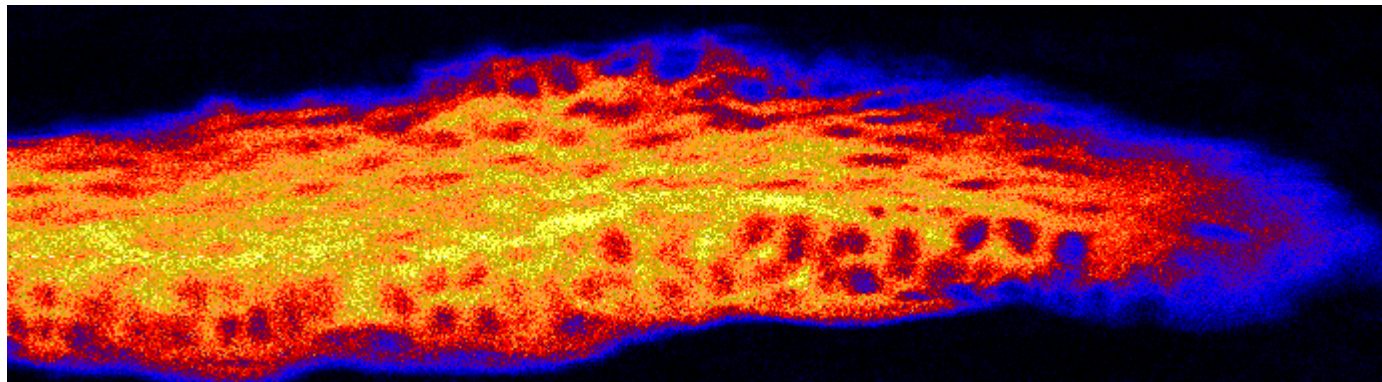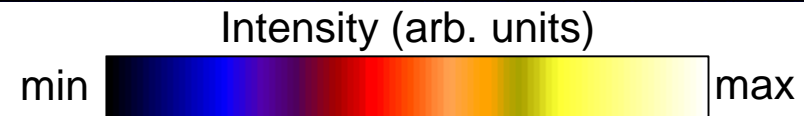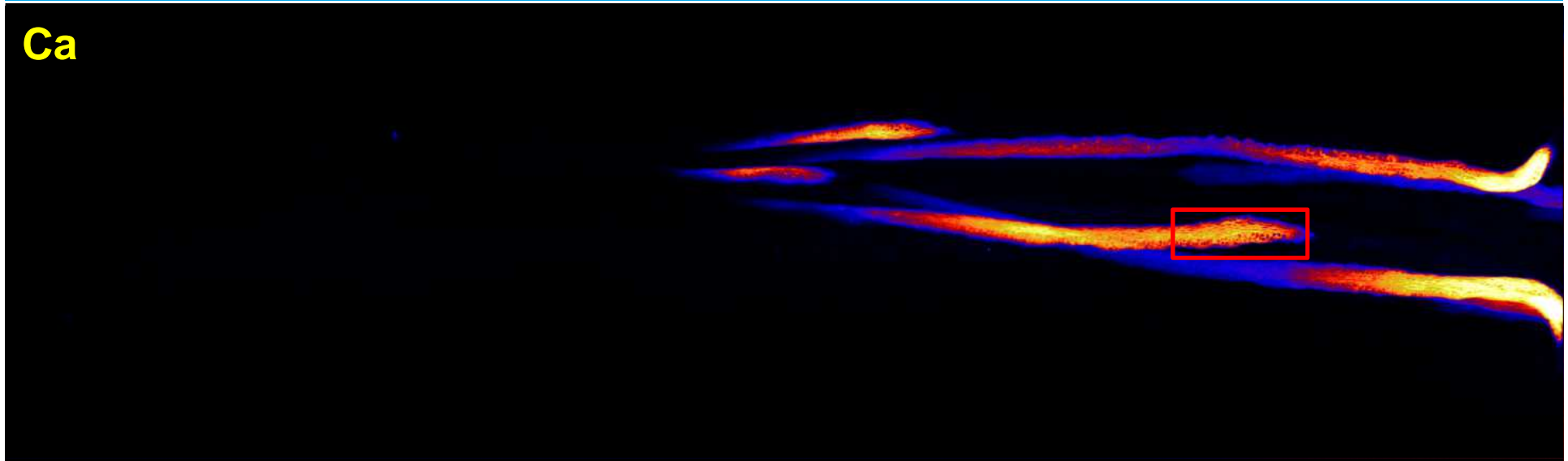
    - ~8 hours measurement time

In collaboration with:
S. Cagno, O. Lind, B. Salbu: Norwegian University of Life Sciences (NO)
G. Nuyts, F. Vanmeert, K. Janssens: University of Antwerp (BE)

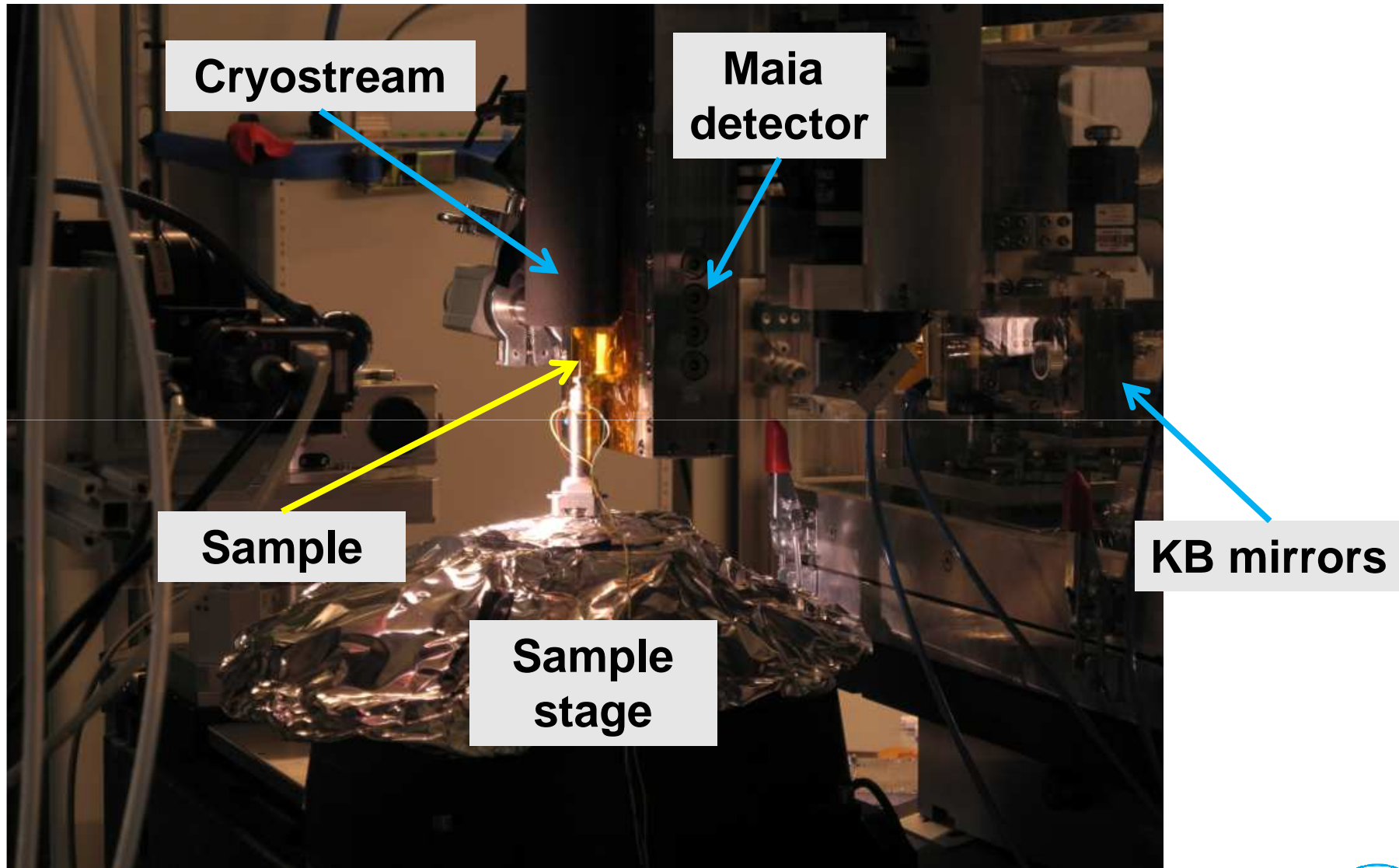# Fast 2D XRF imaging: U uptake in Salmon gills

Ca

Intensity (arb. units)

min ——— max

700x190 µm

The Maia system at P06 allows to acquire high resolution elemental distribution images of large areas.

Cryo-XRF tomography
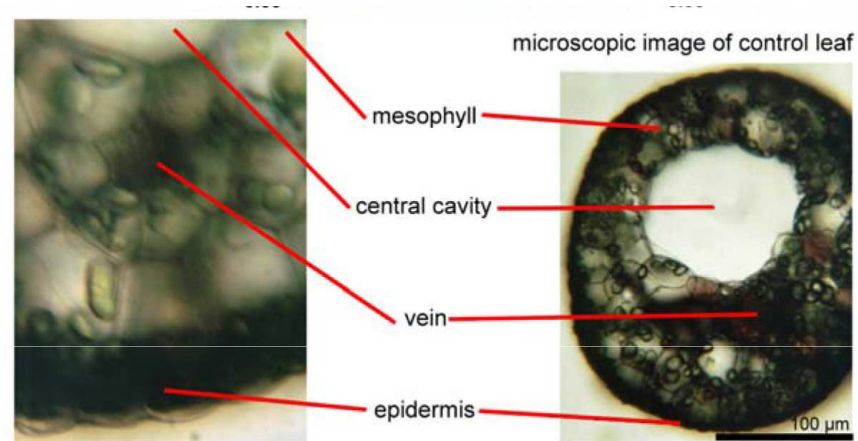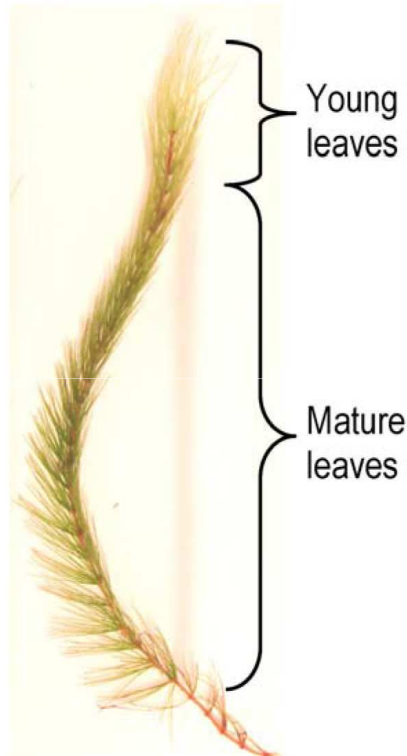

Cryostream

Maia detector

Sample

Sample stage

KB mirrors

Cryo-XRF tomography

Distribution of As in *Ceratophyllum demersum*

Young leaves

Mature leaves

microscopic image of control leaf

mesophyll

central cavity

vein

epidermis

100 µm

- *Ceratophyllum demersum* was grown in 0 to 5 µM As solution.
- After harvesting leafs and cleaning: Transfer to glass capillaries
- Shock-freezing in supercooled isopentane
- First XRF tomography experiment with Maia at P06

Data acquired in collaboration with:
Seema Mishra (University of Konstanz, DE)
Hendrik Kuepper (University of Konstanz, DE)

Images reproduced from:
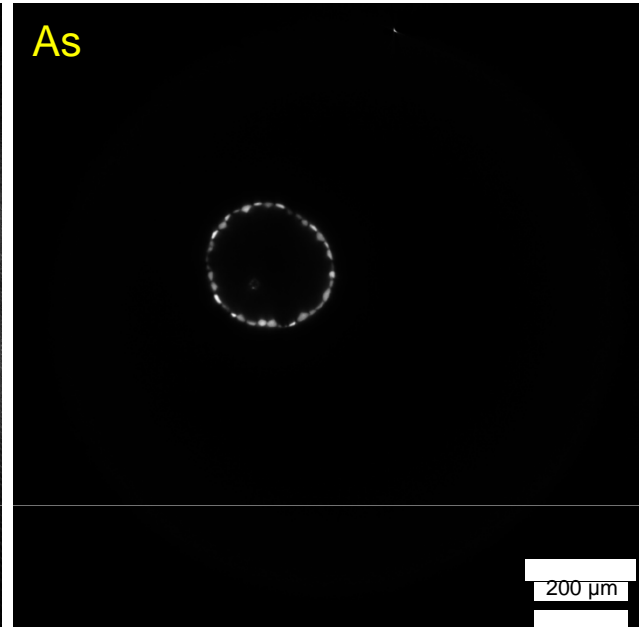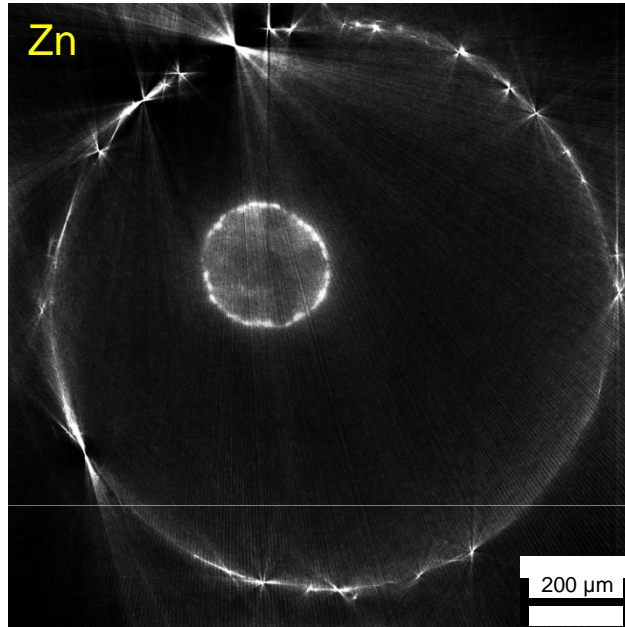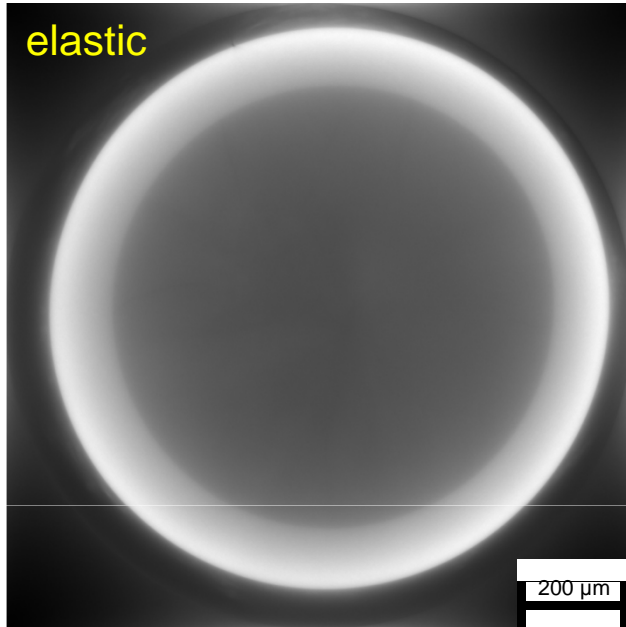**Mishra S et al. Plant Physiol. 2013;163:1396-1408**

As
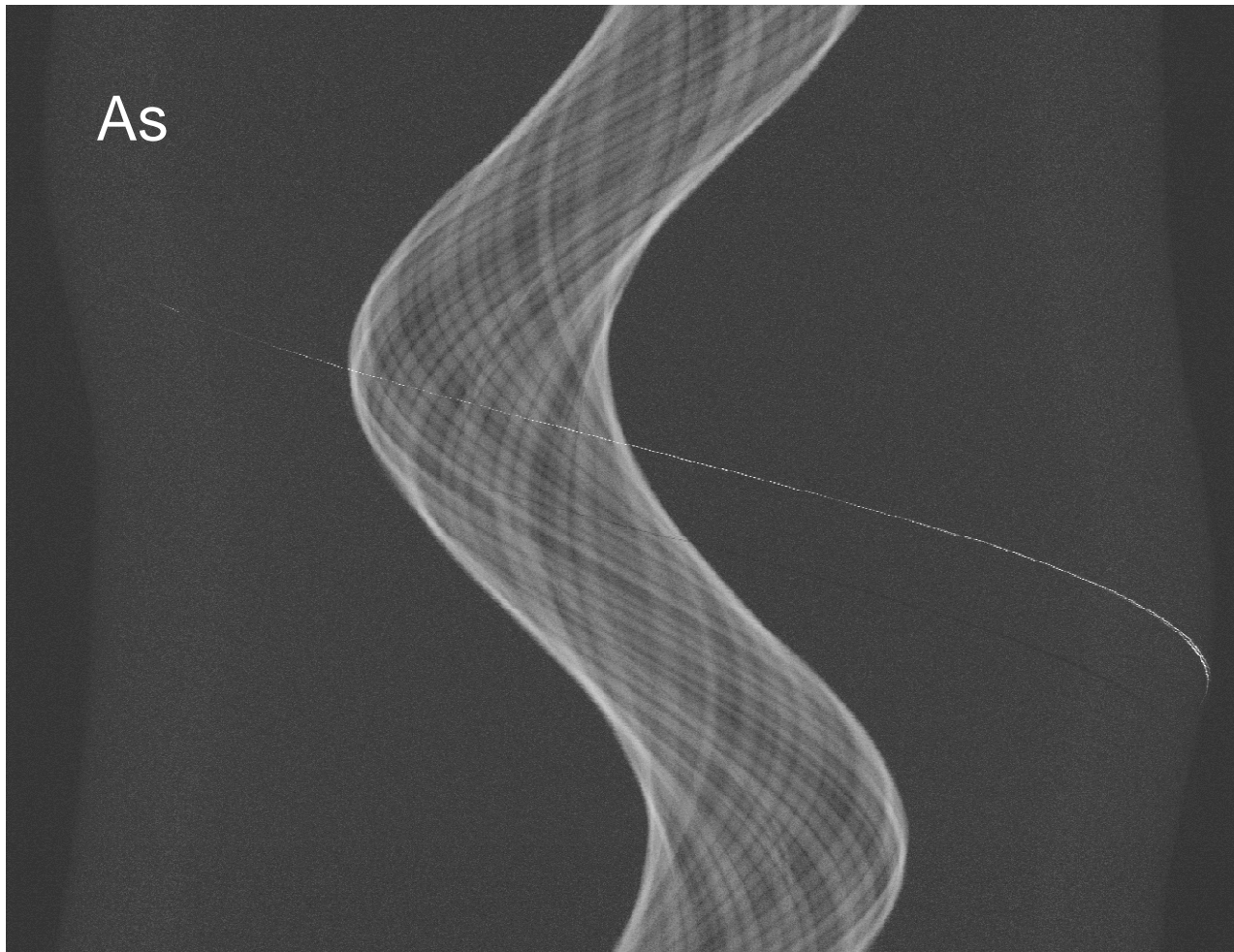
- 4709 x 240 nm steps
- 3600 x 0.1 degree steps
- 1.2 ms dwell time
- 8 hours aquisition
- The sinograms were drift corrected

Image reconstruction: MLEM routine from XRDUA (W. De Nolf, F. Vanmeert and K. Janssens, J. Appl. Crystallogr. 2014, 47, 1107-1117)
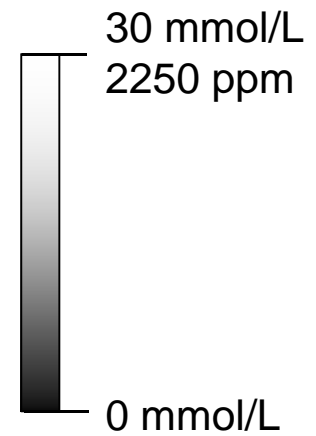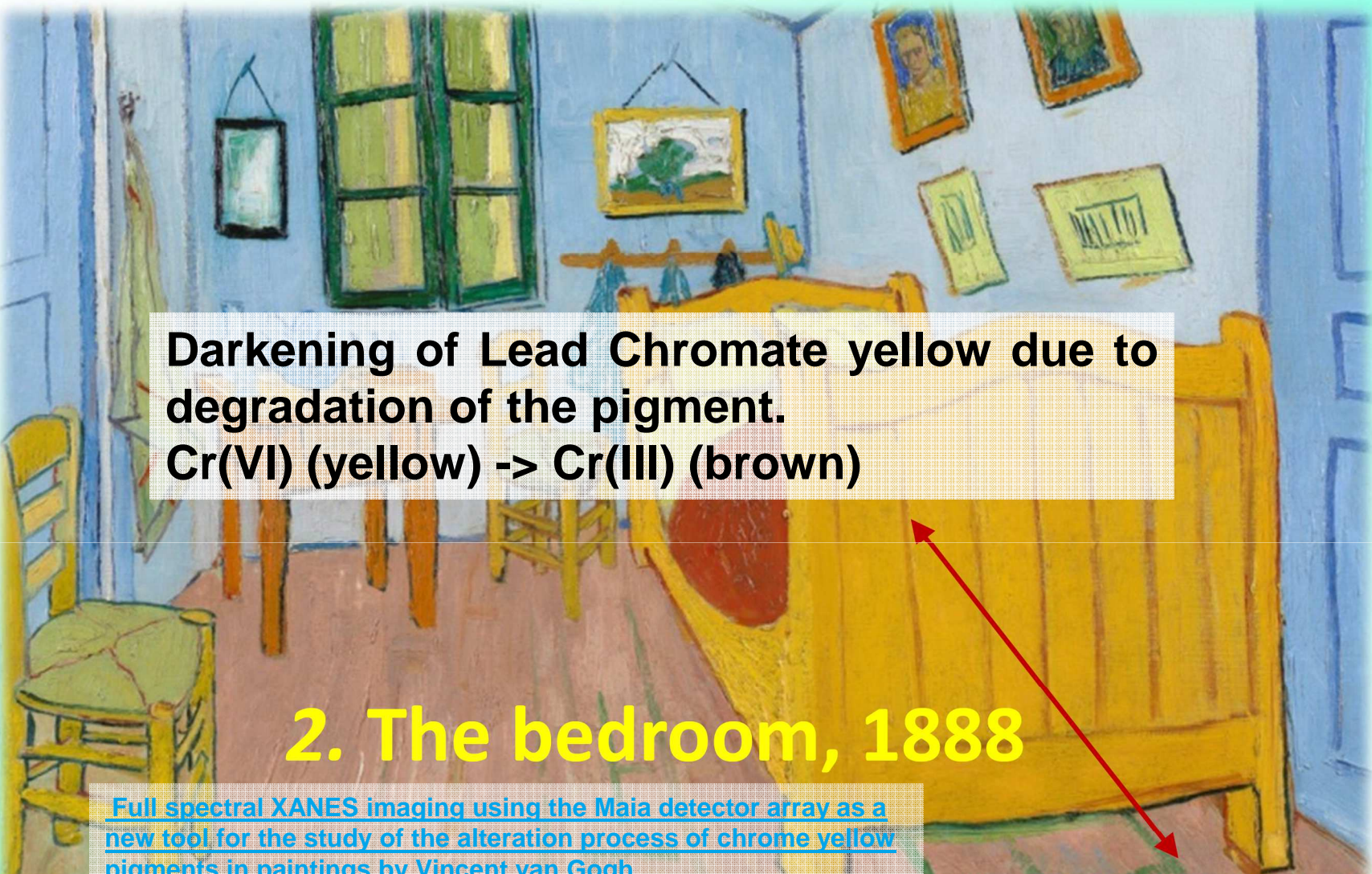
Cryo-XRF tomography

elastic — 200 µm

Zn — 200 µm

As — 200 µm

Cryo-XRF tomography

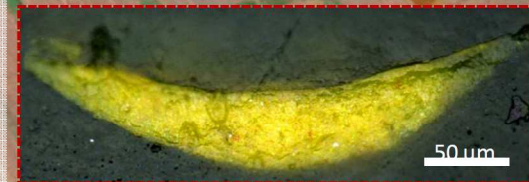- The Maia allows for the acquisition of quantitative XRF tomograms with sub-cellular resolution.

Darkening of Lead Chromate yellow due to degradation of the pigment.
Cr(VI) (yellow) -> Cr(III) (brown)

2. The bedroom, 1888

50 µm

Van Gogh Museum
Amsterdam
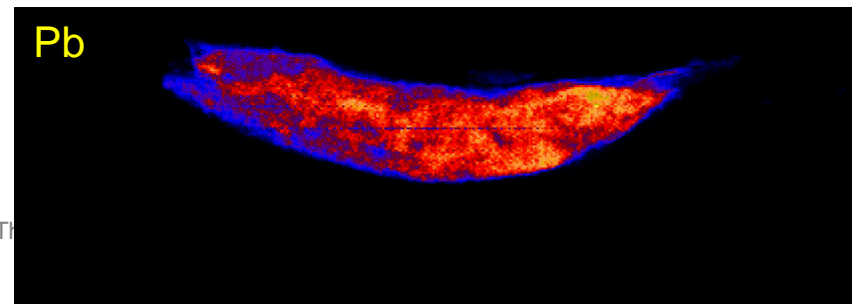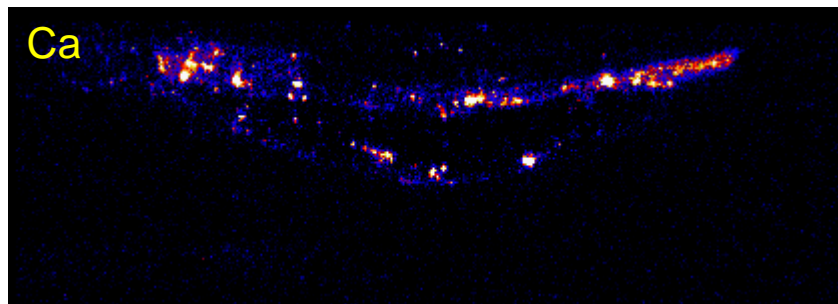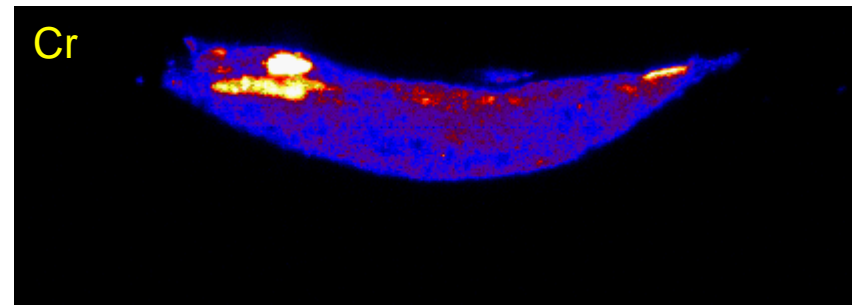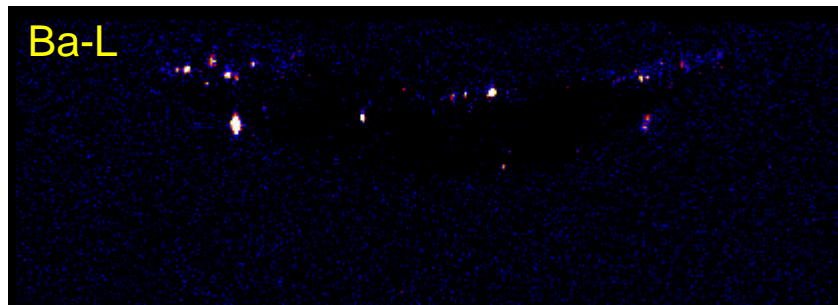
Scan area: 420x150 µm
Step size: 1 µm
Dwell time: 3 ms/pixel
Energy range: 5.96 to 6.088 keV in **125 scans**
Measurement time: 3.5 minutes/slice; 8 hours/XANES stack

XANES: X-ray Absorption Near Edge Structure

XANES: X-ray Absorption Near Edge Structure

Area #0 (Cr III)

Area #2 (Cr VI)

Imaging XANES with the Maia detector system allows to acquire chemical information of large areas
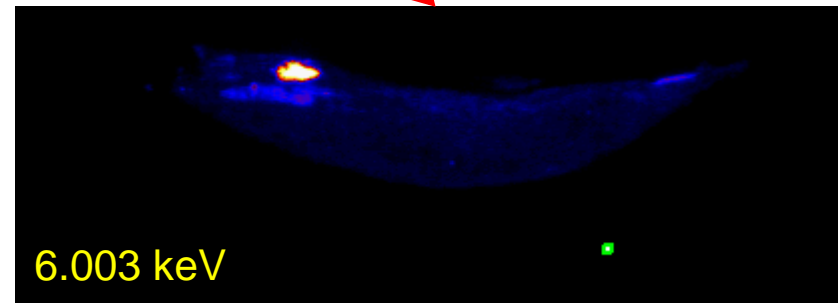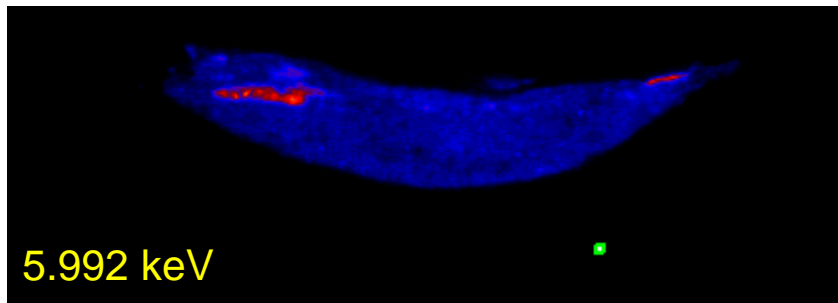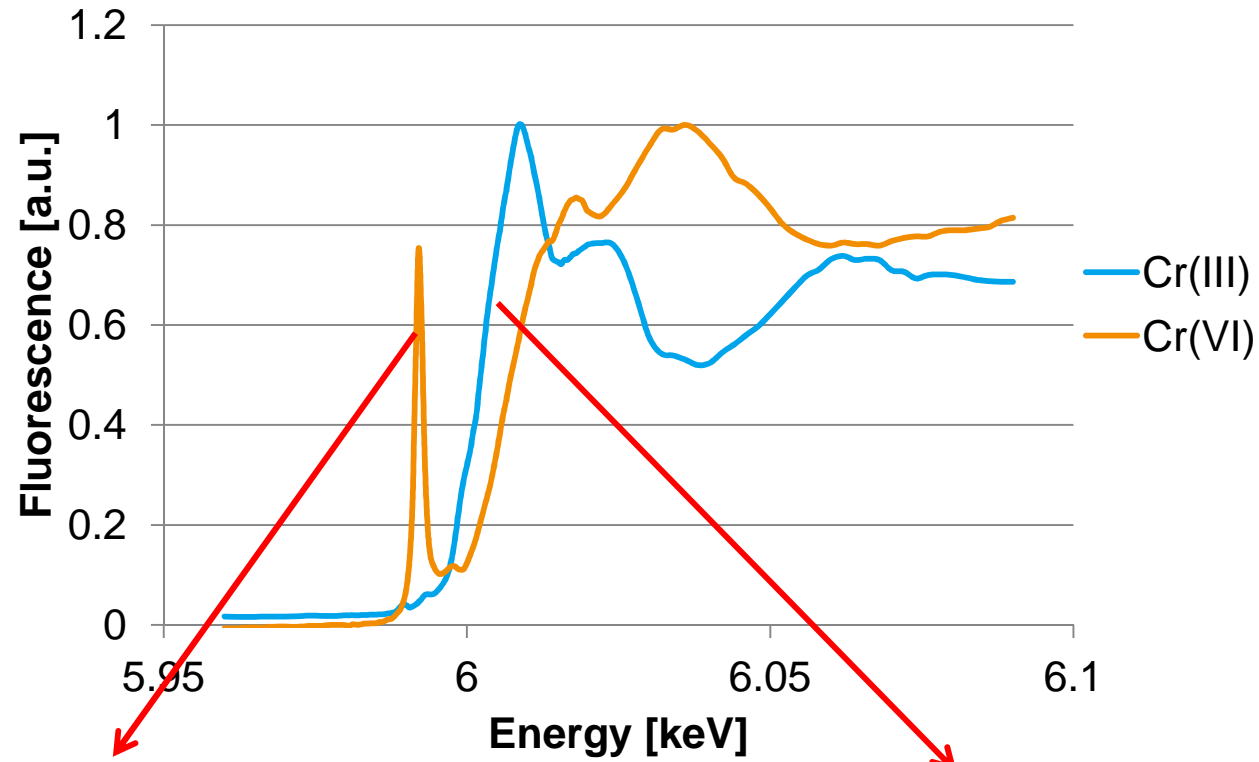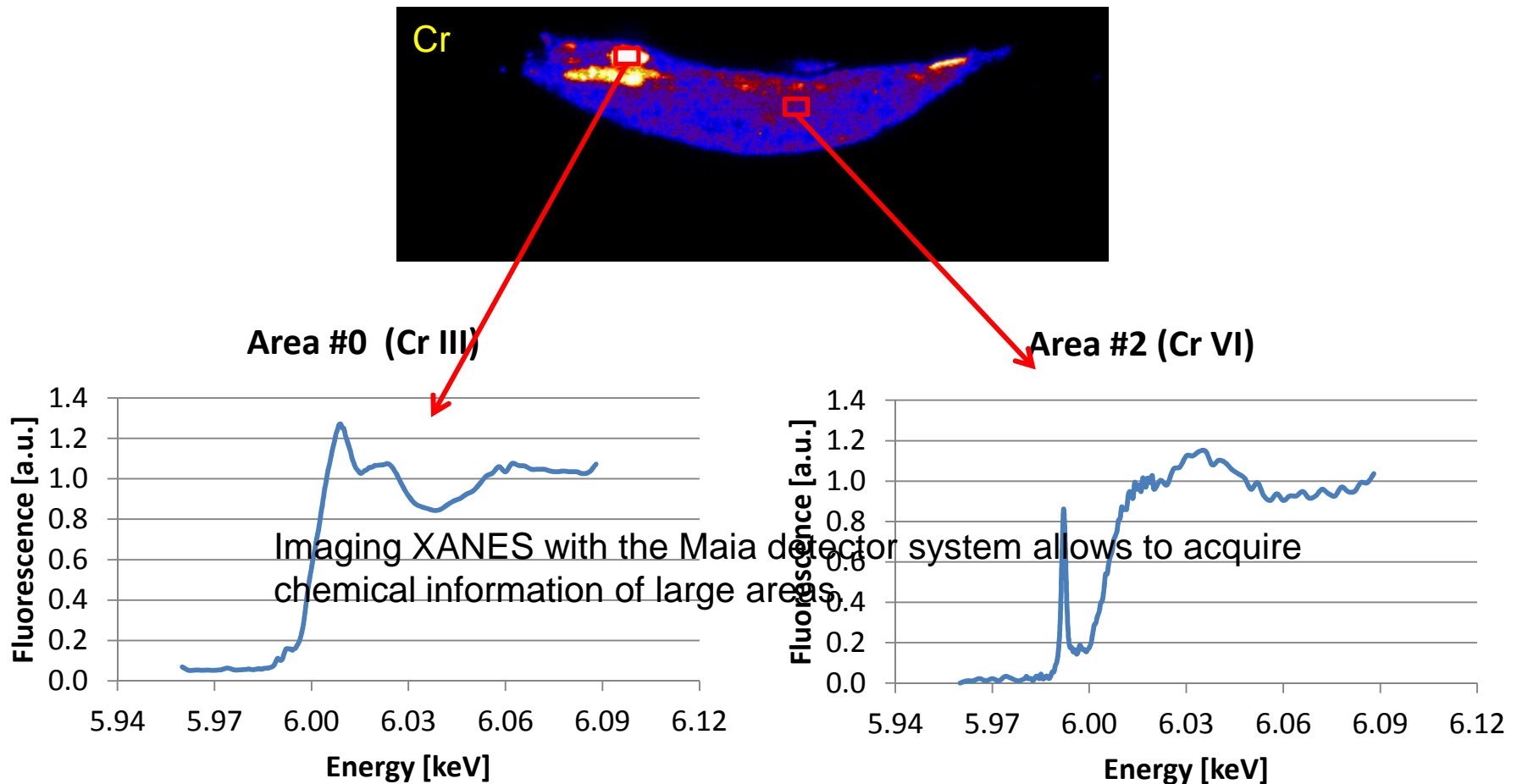
Conclusions

> The Maia system at P06 allows fast XRF measurements

- ms dwell time

- Sub-micrometer spatial resolution

- Multi-dimensional

> Can be coupled with sample environments

> Maia system including GeoPixe software cares for complete signal workflow from signal detection, data acquisition, data processing to quantitative data analysis and export of high quality images

> Maia is not good for everything (geometry, resolution, size,…)

maia scan	step width /nm	dwell/ms	accl. Hydra /mms^{-2}	scan time /min	spatial resolution /nm
January 2014 E=11.5 keV, ~500x500nm beamsize					

maia scan	step width /nm	dwell/ms	accl. Hydra /mms^{-2}	scan time /min	spatial resolution /nm
5970	200	0,1	20	3	452,08
5971	200	0,5	20	3	414,94
5972	200	1	20	3	352,61
5973	200	10	20	12	313,28
5974	200	100	20	105	262,74
5975	100	0,1	20	5	353,36
5976	100	0,5	20	6	265,11
5977	100	1	20	7	263,30
5978	100	10	20	44	245,70
5979	100	100	20	247-aborted	
Y stage mounted at the top					
5987	100	0,1	10	4	275,48
5988	100	0,3	10	4	263,02
5989	100	1	10	8	212,59
5990	100	0,1	20	4	269,98
5991	100	0,3	20	6	235,96
5992	100	1	20	8	230,95
5993	100	10	20	44	225,02
5994	100	100	20	417	**201,21**
5995	200	0,1	20	3	382,26
5996	200	0,3	20	3	347,22
5997	200	1	20	4	335,80
5998	200	10	20	12	313,09
5999	200	100	20	7- aborted	

January 2014 E=11.5 keV, ~500x500nm beamsize
Y stage mounted at the bottom