



COVALENT
METROLOGY

Welcome

LASER ABLATION INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY: NOT JUST ROCKS

SPEAKER:

Lucas Smith

Director of Business Development
for the Americas,
Teledyne CETAC

February 10, 2022 | 11am PT



COVALENT ACADEMY

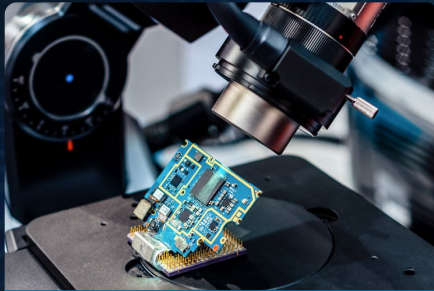
Advancements in
Instrumentation Series

Episode 30



COVALENT METROLOGY

Silicon Valley-based analytical labs and platform delivering quality data and expert analysis for advanced materials and device innovation



Comprehensive Solutions Stack

*50+ cutting-edge
instruments, offering
100+ Techniques*

Analytical Services

Advanced Modeling

Method Development

Temp. Staffing Solutions



Affordable and Fast

*Fast Turnaround Times,
No Expedite Fees*

Volume Savings

*Instant Access to Data
and Reports in Secure
Portal*



Flexible Business Model

*Custom Consulting
Solutions and Certified
Onsite Support*

*Training and Certification
on Instrumentation*

*Co-op and Tool-Share
Opportunities*

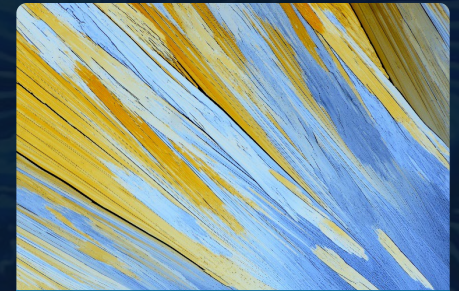
Laboratory Audits



Rich Network of Partnerships

*Partner to World's
Leading Instrument
Manufacturers and Labs*

*Expanding
Instrumentation, Lab
Connections and
Learning*



Who We Are, Who We Serve

*500+ Clients,
40-60 Added / Quarter*

50+ People, 14 PhDs

*Cutting-edge Analytical
Capabilities*

*Lab Location:
Sunnyvale, CA*

Covalent Technical Groups and Organization

4

PCBA, Semiconductor, and Electronic Device Metrology & Failure Analysis

- DPA / Mechanical Cross-section
- Dye & Pry Test
- EBIC / OBIC failure analysis
- Hot Spot Detection
- IR Imaging / Emission Microscopy
- NIR Imaging
- Root-Cause Failure Analysis

Electron Microscopy and Scanning Probe Microscopy

- AFM & Advanced AFM Modes (EFM, KPFM, MFM, PFM)
- Scanning Acoustic Microscopy (SAM)
- SEM (+ EDS)
- FIB-SEM (+ EDS)
- S/TEM (+ EDS / + EELS)
- Nano-indent / Nano-scratch

Optical Microscopy & Spectroscopy

- Chromatic Aberration
- Digital Optical Microscopy
- FTIR and ATR-FTIR
- Laser Scanning Confocal Microscopy
- Spectral Ellipsometry
- UV-Vis-NIR Spectroscopy
- White Light Interferometry

X-Ray Characterization

- X-Ray Diffraction (XRD)
- X-Ray Reflectometry (XRR)
- Micron-spot ED-XRF
- WDXRF
- Micro-computed X-ray Tomography (Micro-CT)
- 2D X-ray Inspection & X-ray Radiography

Elemental / Chemical Composition Analysis

- EPMA
- GD-OES
- GC-MS
- ICP-MS and LA-ICP-MS
- Raman Microscopy & Spectroscopy
- NMR (1D or 2D; solid / liquid)

Particle Analysis

- Dynamic Light Scattering (DLS)
- Laser Diffraction Particle Size Analysis (PSA)
- Particle Zeta Potential

Material Property Characterization

- DSC
- DMA & TMA
- Rheometry
- TGA
- Surface Zeta Potential
- Porometry / Porosity
- Gas Adsorption
- Gas Pycnometry
- Foam Density
- Tap Density

Surface Spectroscopy Analysis

- Dynamic-SIMS
- ToF-SIMS (Static-SIMS)
- Ion Scattering Spectroscopy (ISS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- X-ray Photoelectron Spectroscopy (XPS)



- Teledyne CETAC is a global leader in the development of advanced sample-introduction and sample-handling equipment for elemental analysis
- Partnership with Covalent announced in Fall, 2021 to:
 - Jointly investigate and develop new applications for Teledyne's element analysis tools
 - Bring cutting-edge analytical chemistry solutions to Covalent clients
- First Teledyne instrument installed at Covalent is the *Iridia Laser Ablation System*

Other Covalent Partners



Lucas Smith

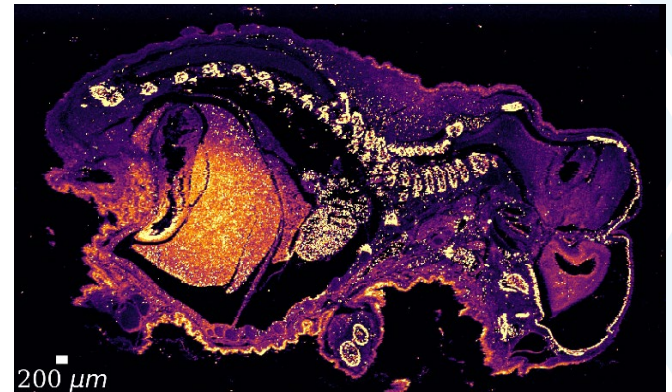
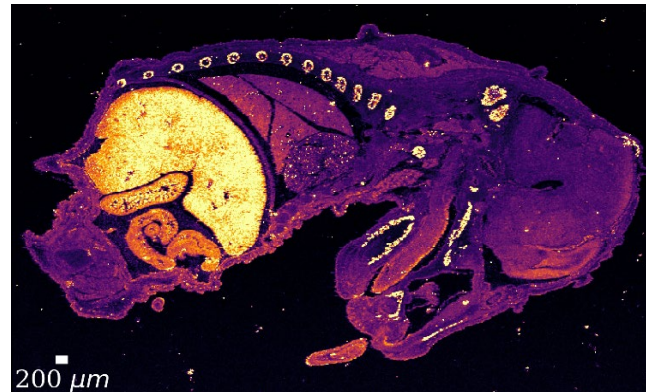
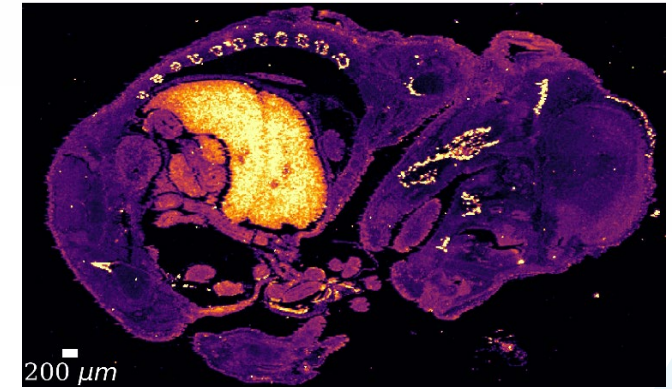
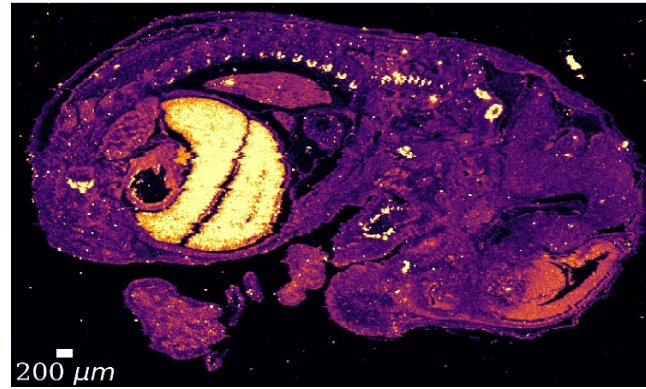
Director for Business Development for the Americas,
Teledyne CETAC

- Degrees in Microbiology and Biochemistry from The University of Michigan (Go Blue!)
- Manages laser opportunities and customer relationships throughout the Western Hemisphere
- Member of the Teledyne team for just over 7 years
- Prior to joining Teledyne Lucas held a variety of roles at
 - Pfizer Pharmaceuticals
 - LECO Corporation
- Performed mobile analytics for the US Navy & Airforce



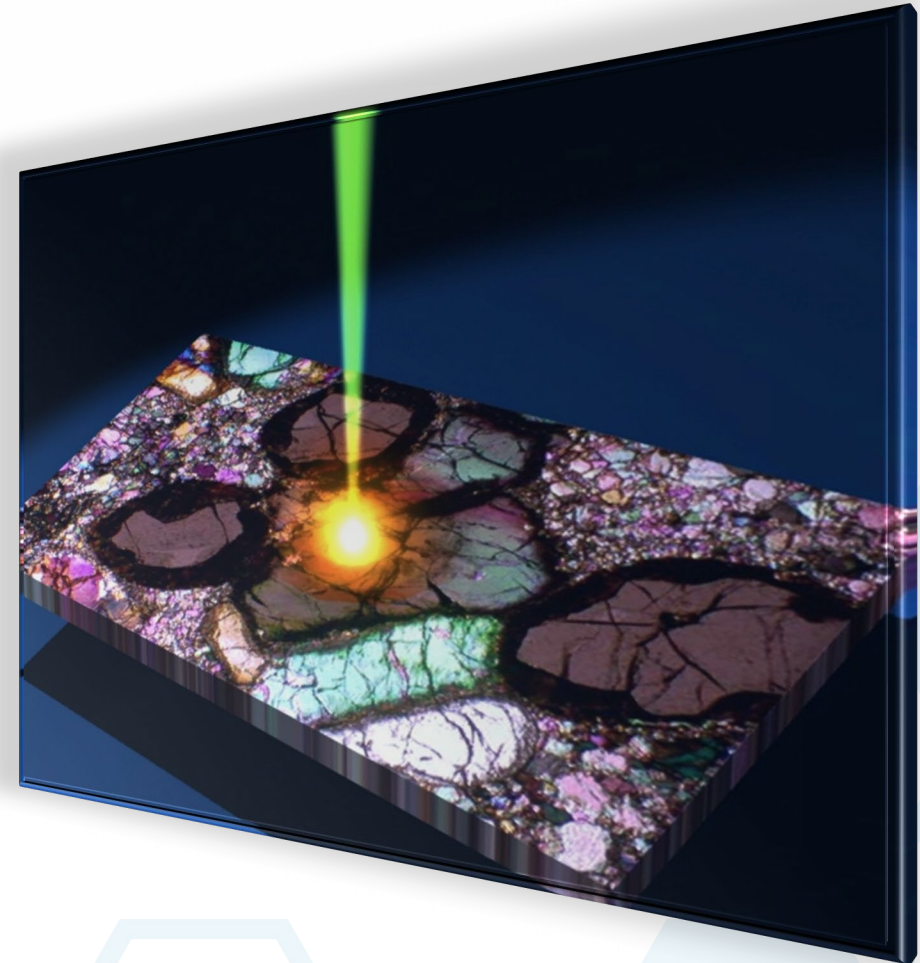
Content

- What is laser ablation ICP-MS?
- Why laser ablation (advantages vs disadvantages)
- Laser ablation vs other analytical techniques
- Spatial Analysis
- Volumetric Ablation
- Application examples
- Q&A



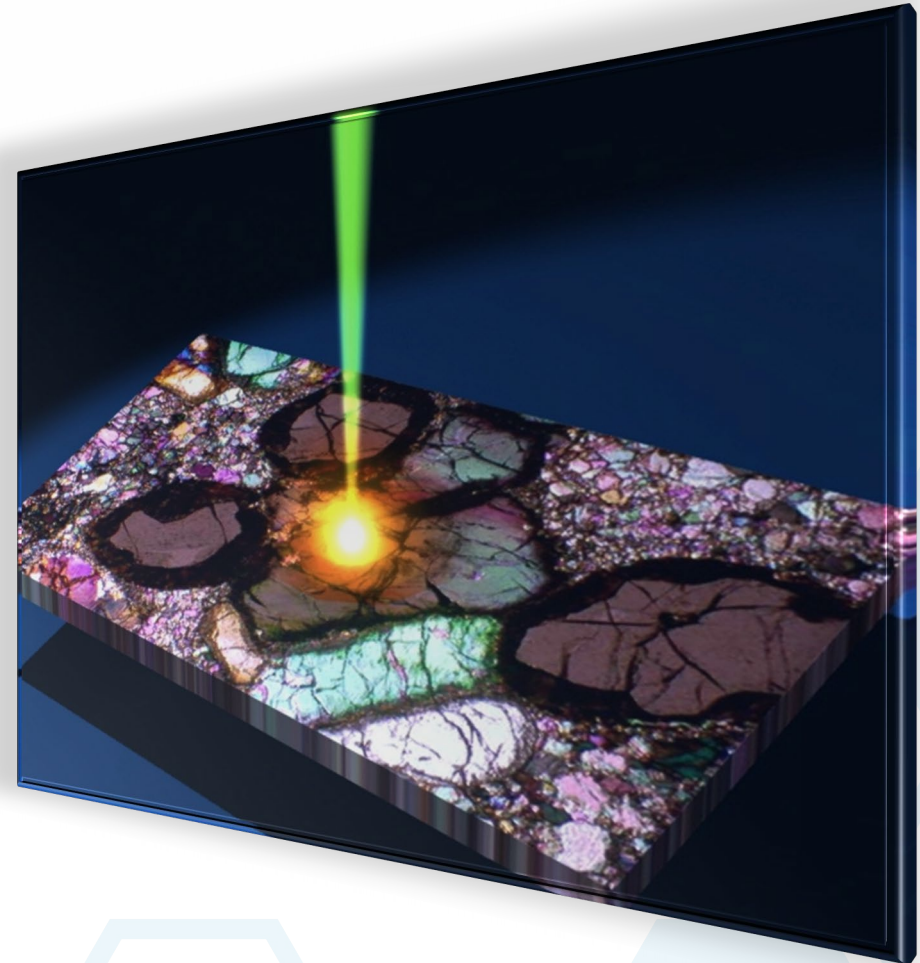
What is Laser Ablation?

- A laser ablates a solid sample and delivers material directly to the ICPMS for elemental analysis
- Eliminates dissolution step and most sample prep
- Maintain spatial resolution



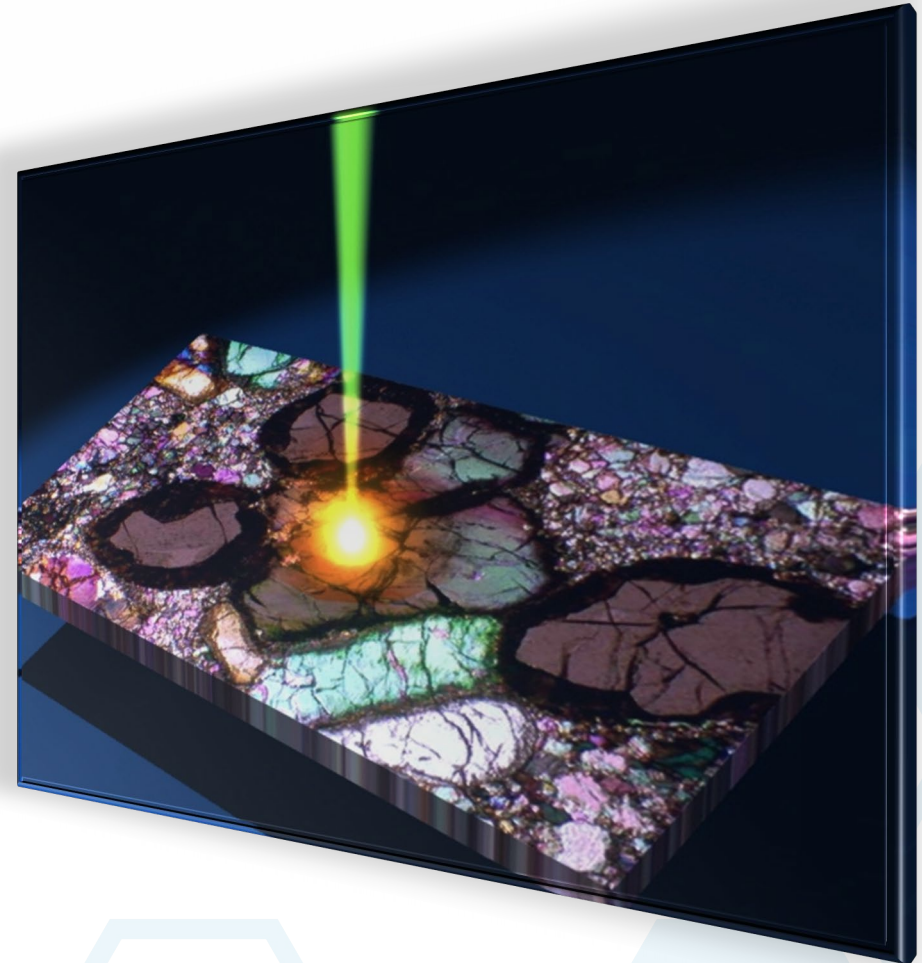
Why Laser Ablation?

- Rapid, sensitive multi-element detection at sub-ppm levels.
- Eliminates (most) labor-intensive sample preparation
- Can sample virtually any material (including liquids)
- Flexible method development
- High spatial resolution of microfeatures
- Sample mapping and depth profiling capabilities
- No contamination of reagents from digestion/dilutions steps

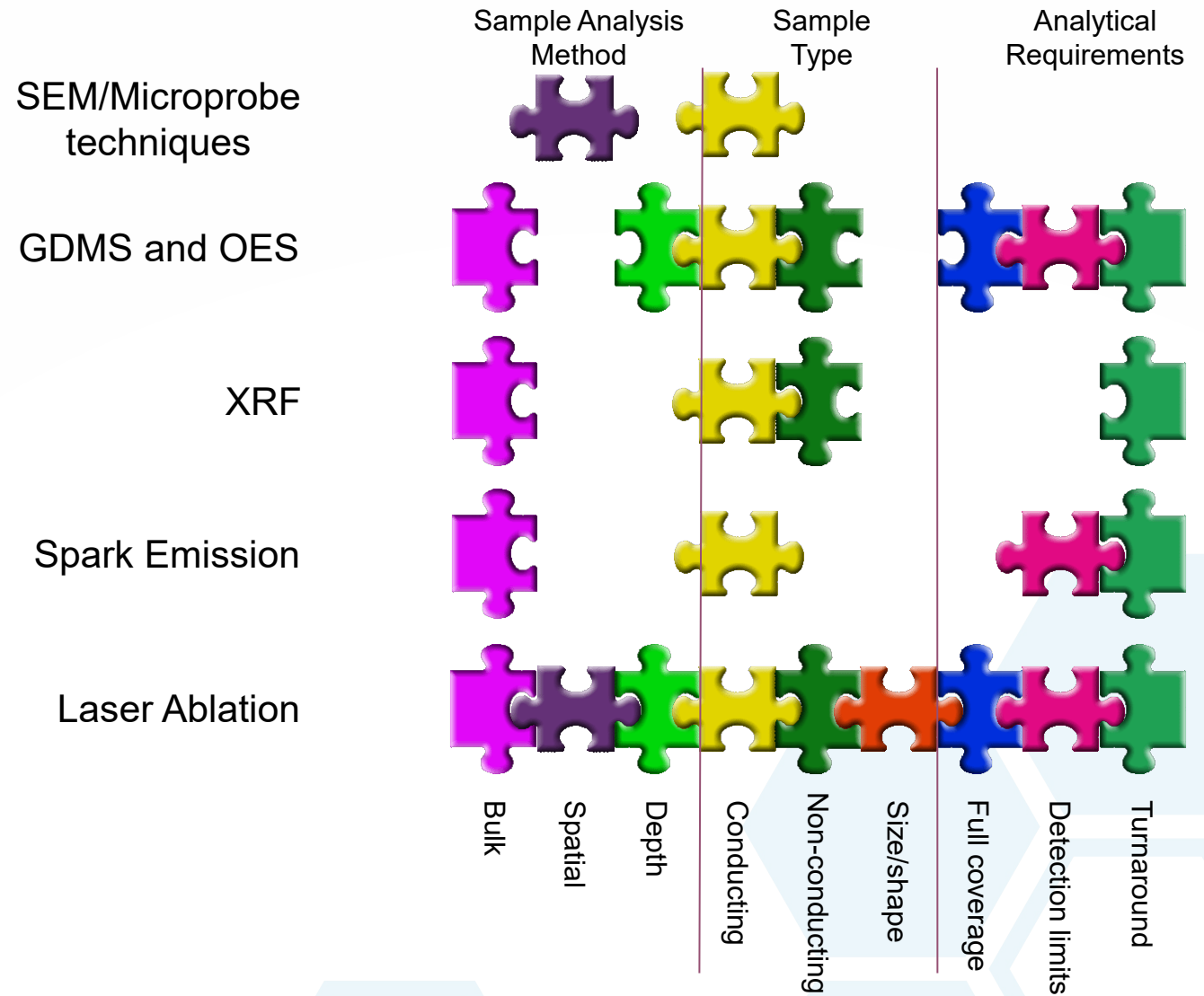


Challenges of the Technique

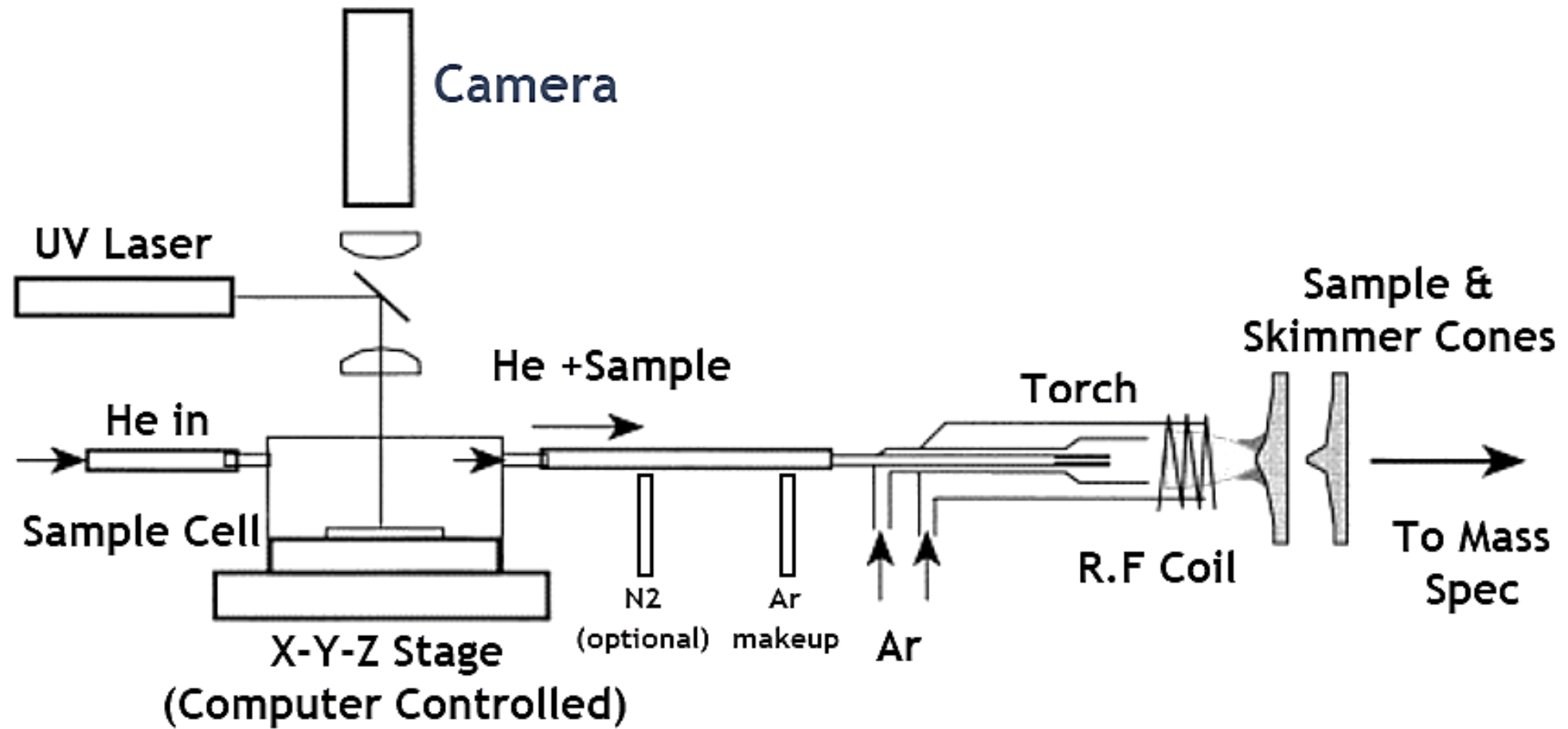
- Historically difficult acquiring standards
- Upper limit in sensitivity due to detector saturation
- (micro)Destructive technique
- Initial cost



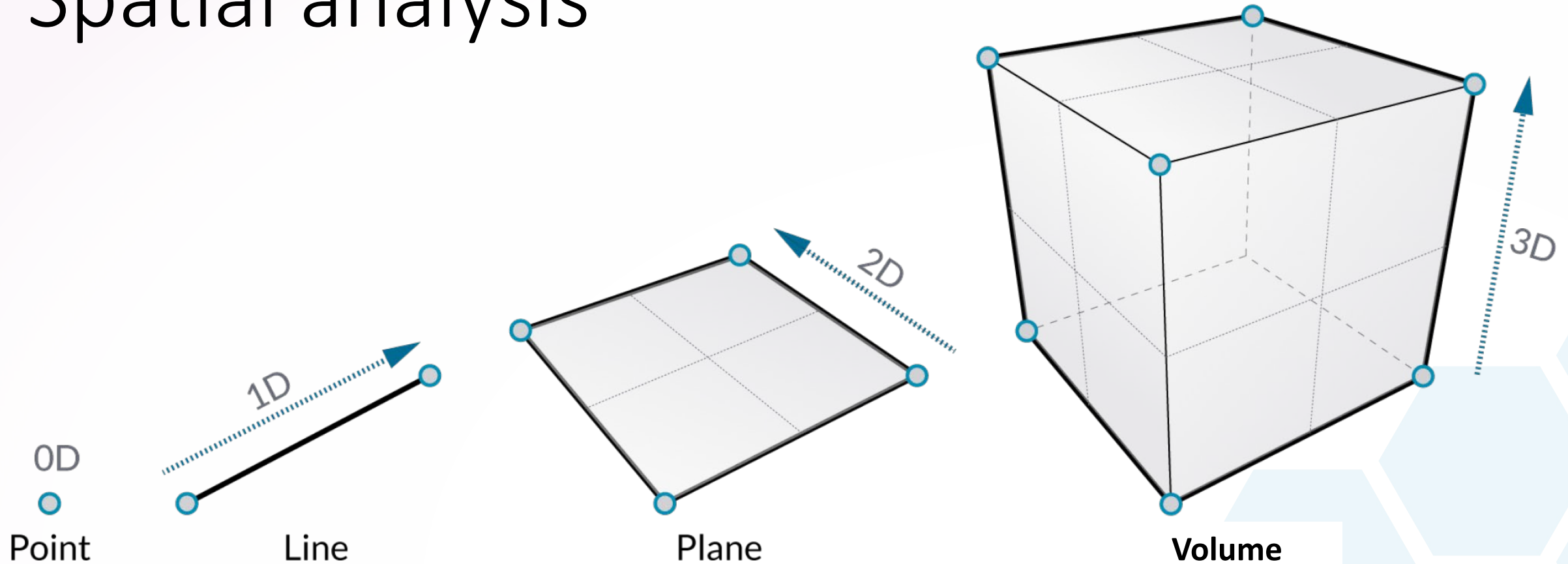
Laser Ablation VS Other Techniques



What is Laser Ablation ICP-MS?

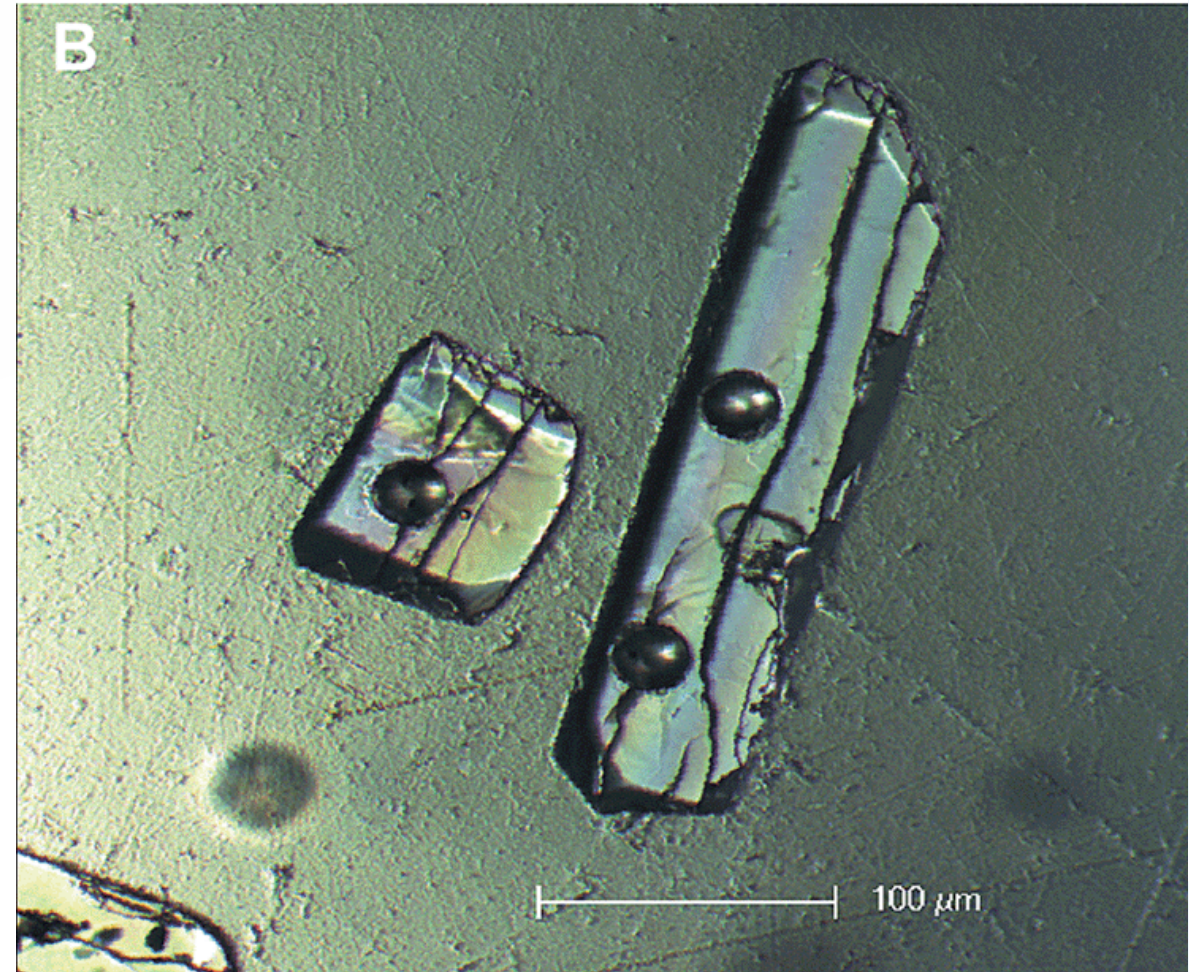
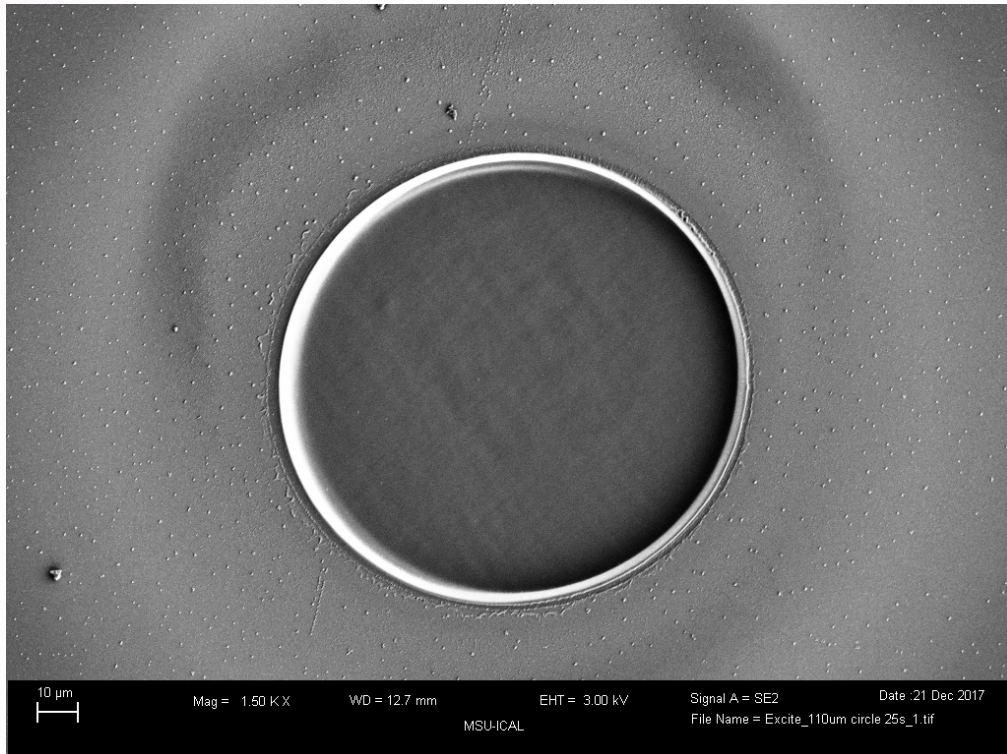


Spatial analysis



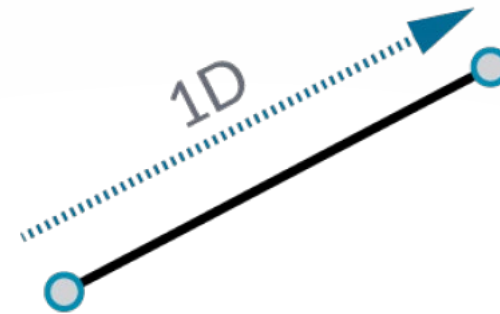
Spatial analysis

- OD – Single Spot - this is (still..!) the typical LA-ICP-MS analysis
- Suitable for bulk analysis, depth profiling, isotope ratio, etc.



Courtesy of Dr. Maurizio Petrelli, University of Perugia

Spatial analysis



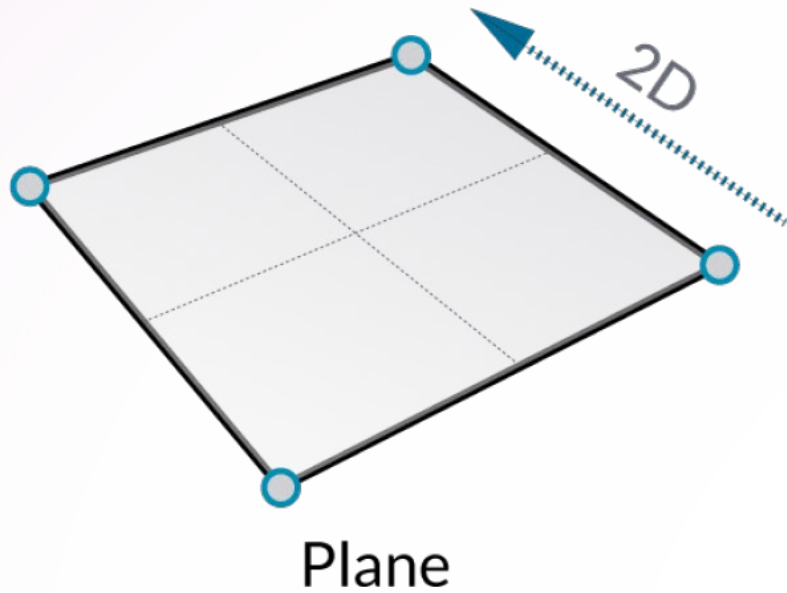
Line

- 1D – Transect
- Useful for the study of diffusion profiles, bulk analysis of homogeneous samples (e.g., Particles trapped on filters, etc.)

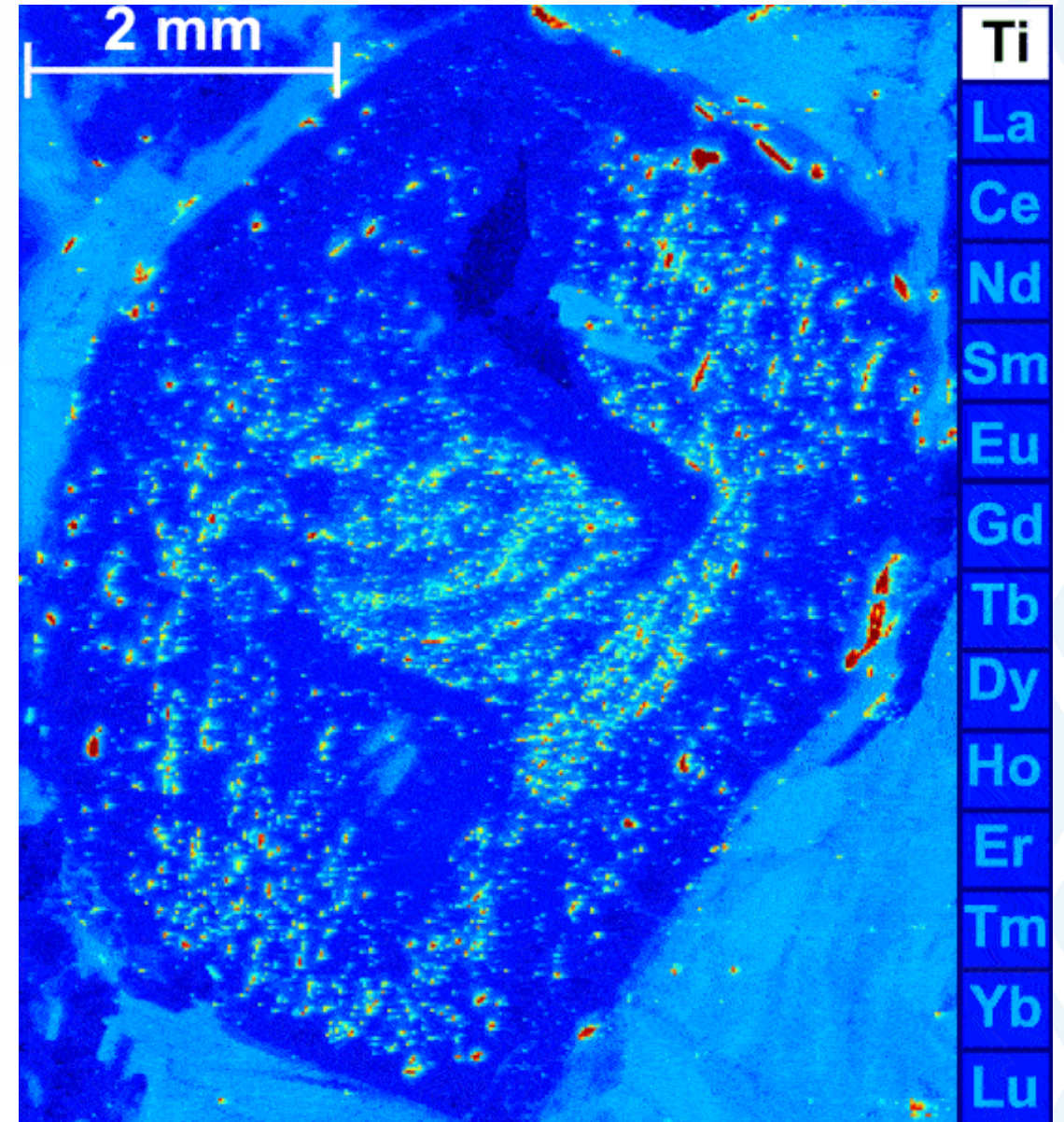
Ogrizek, M., Jaćimović, R., Šala, M., & Kroflič, A. (2021). No more waste at the elemental analysis of airborne particulate matter on quartz fibre filters. *Talanta*, 226.



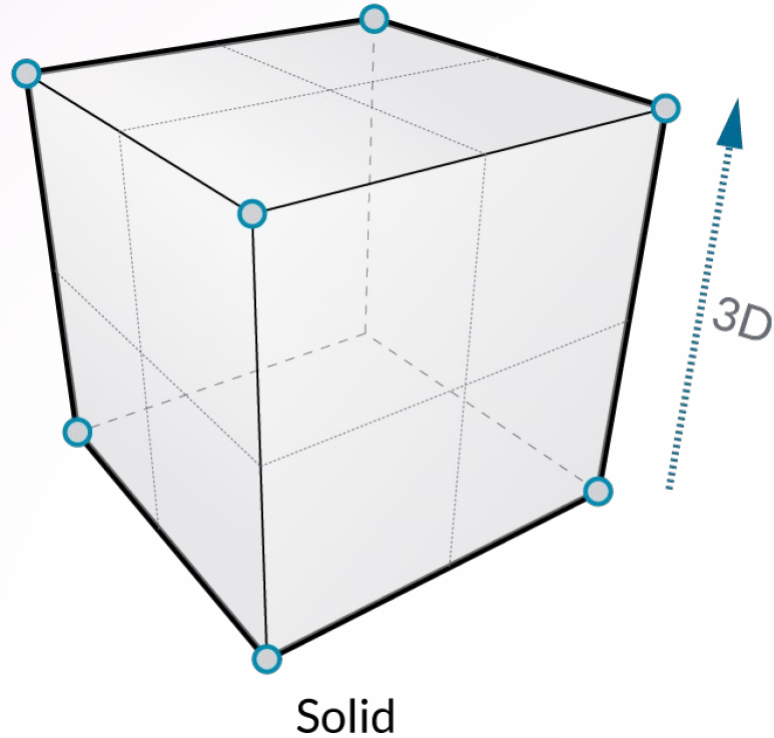
Spatial analysis



- 2D – the spatial distribution of elements (isotopes) in samples



Spatial analysis



- 3D – the spatial distribution of elements (isotopes) in 3D



Zircon from the Ditrau alkaline massif (Eastern Carpathians, Romania).
Courtesy of Dr. Gavril Sabau, Geological Institute of Romania

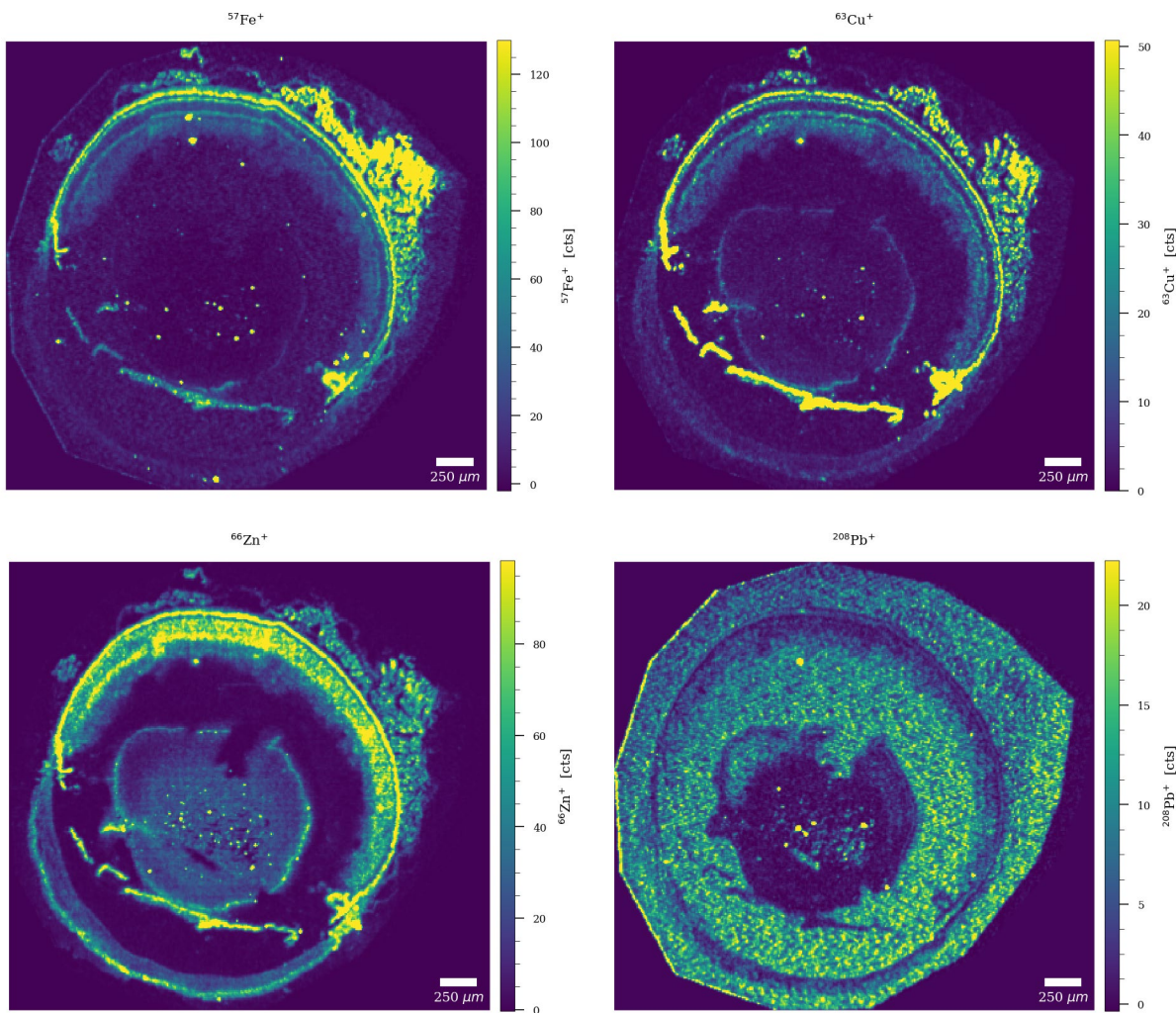
Data Collected at 375Hz

Mouse eye

- 3.0mm diameter
- Unpublished data

**10 μm resolution
375 Hz**

- Agilent 8900 ICP-MS
- Cobalt *Long Pulse* cell
- Parameters optimized and images generated with Teledyne HDIP software
- **5 elements measured**
 - C^{13} , Fe^{57} , Cu^{63} , Zn^{66} , Pb^{208}

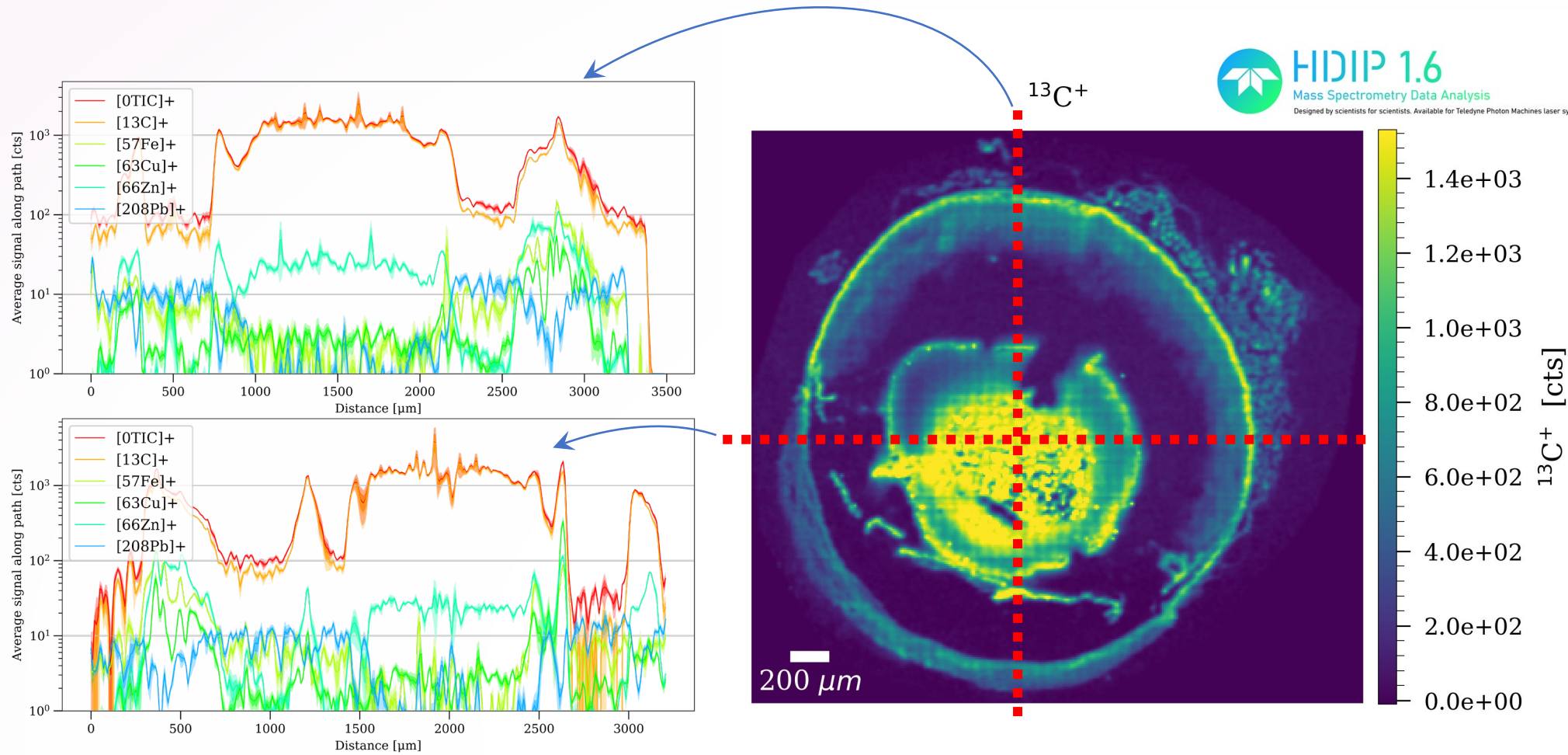


50 minutes

N NATURAL
HISTORY
MUSEUM

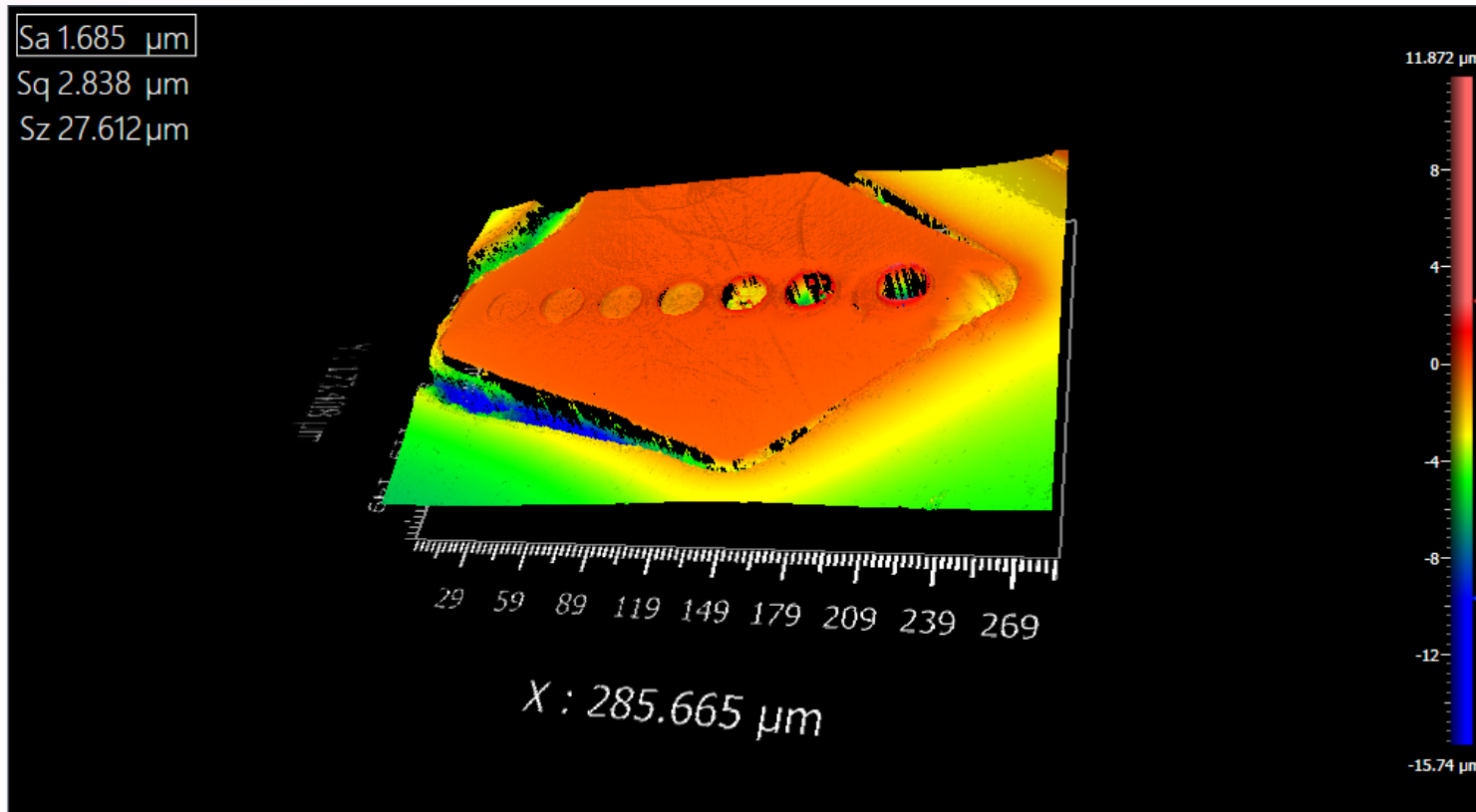
**Sheffield
Hallam
University**

Horizontal & Vertical Data Transects



Transects extracted with Teledyne HDIP software

Volumetric Ablation – for Depth Profiling and Thin Film Analysis



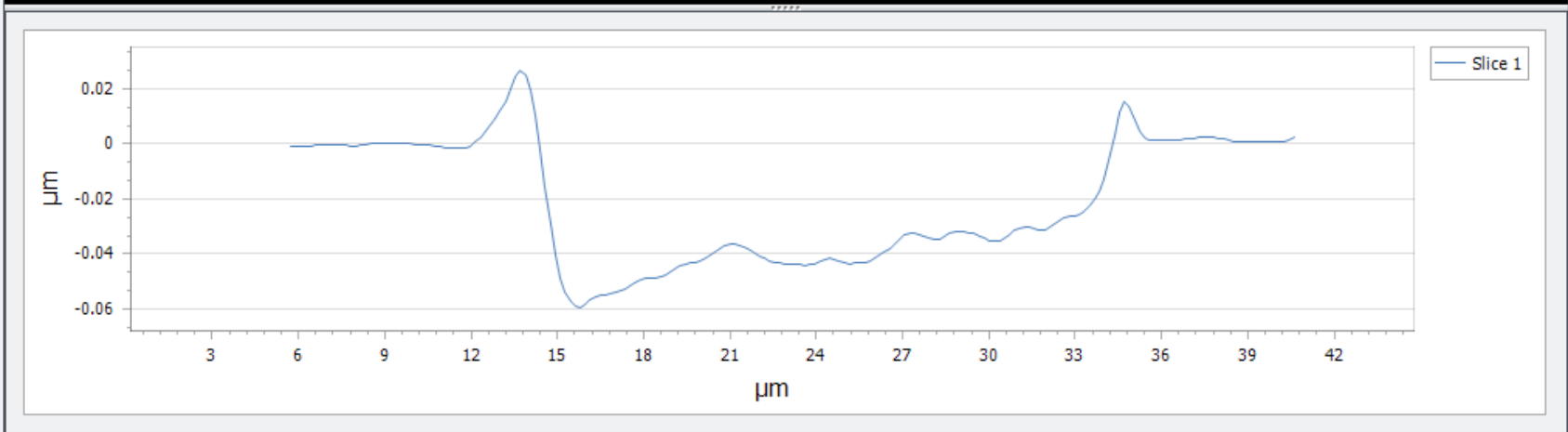
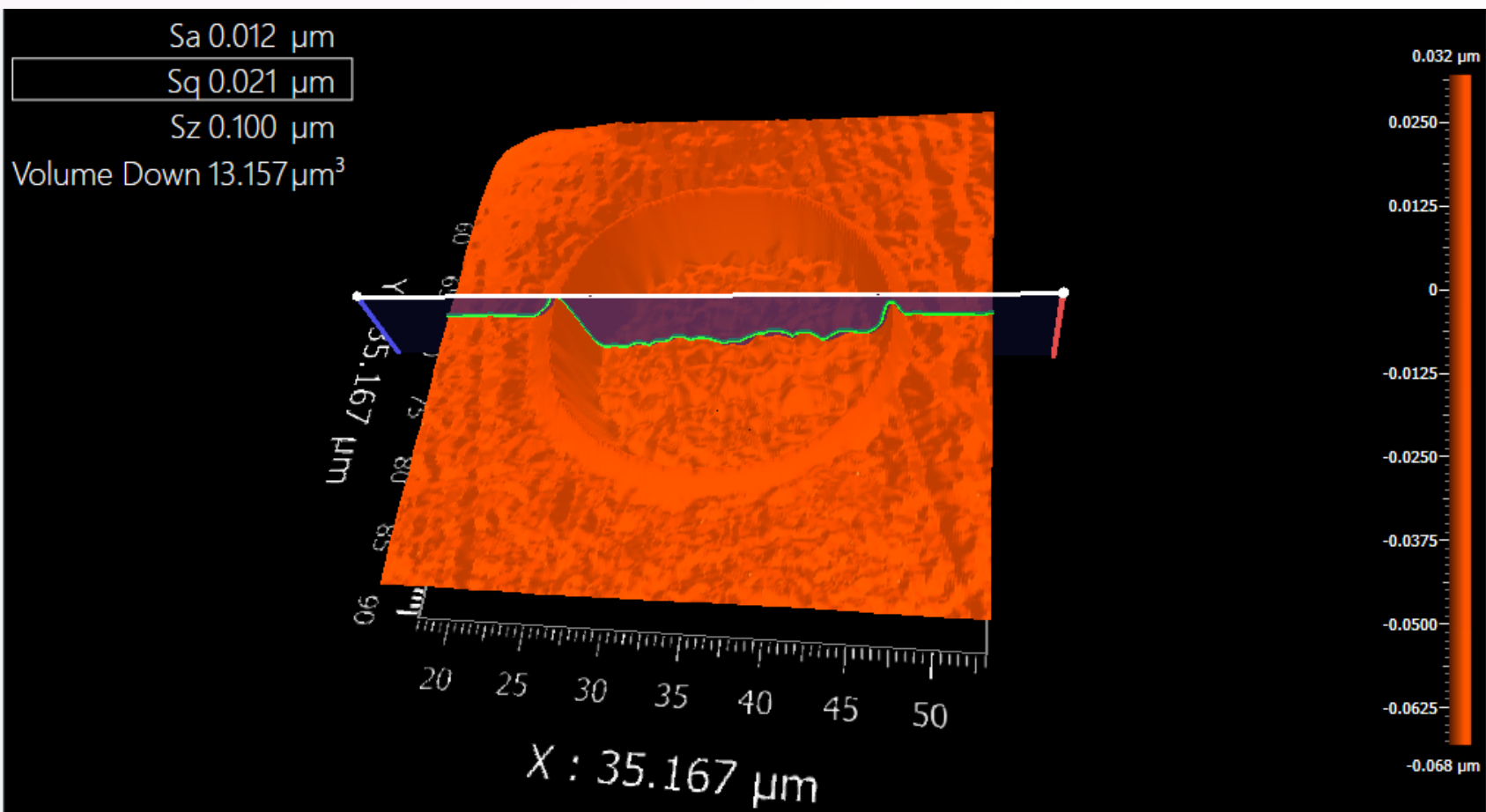
FC-1 zircon

3 Jcm^{-2} (measured at the sample surface)

10 Hz

20 μm spot

1, 5, 10, 20, 50, 100 and 157 pulses



FC-1 zircon

3 Jcm⁻²

10 Hz

20 μm spot

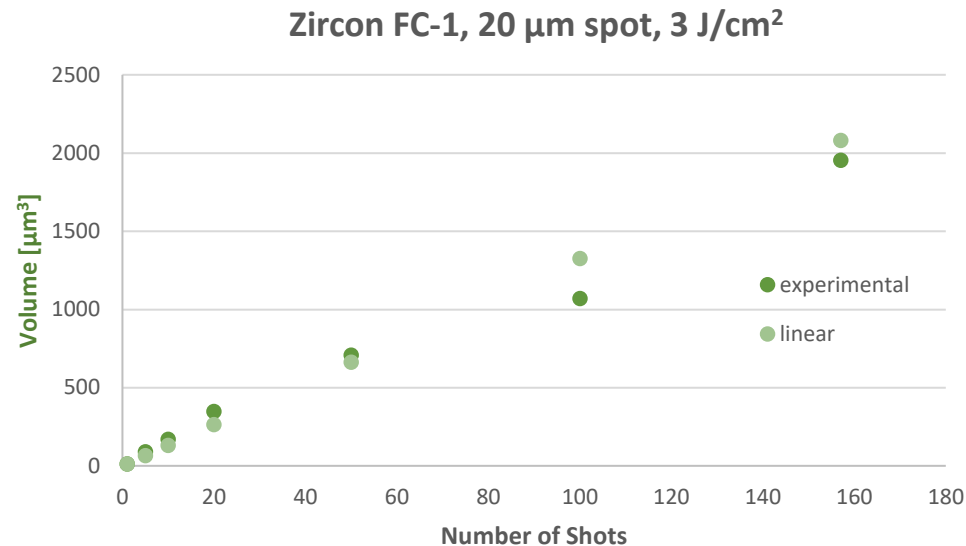
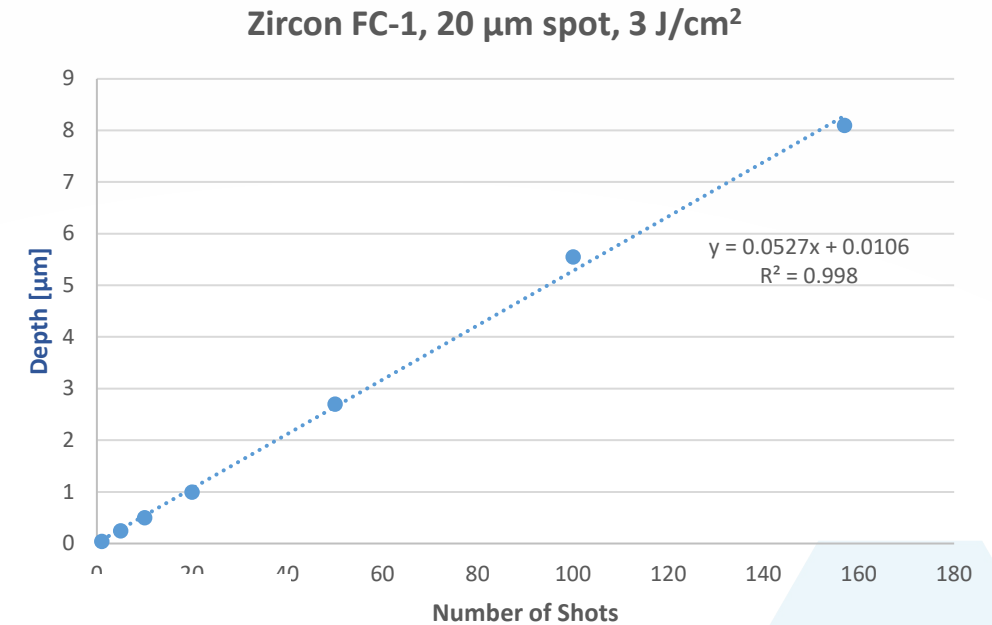
1 pulse



NATIONAL INSTITUTE OF CHEMISTRY

Volumetric Ablation – for Depth Profiling and Thin Film Analysis

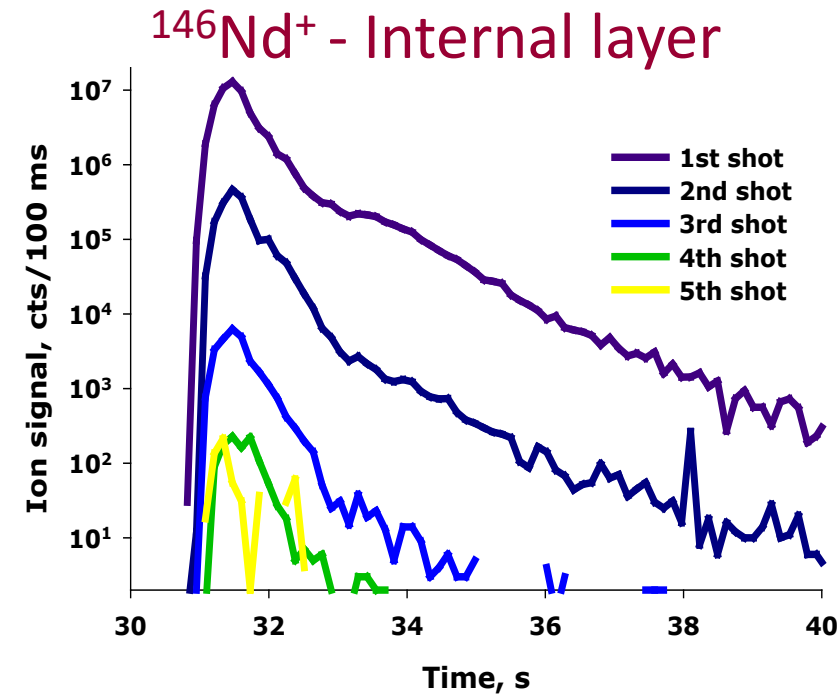
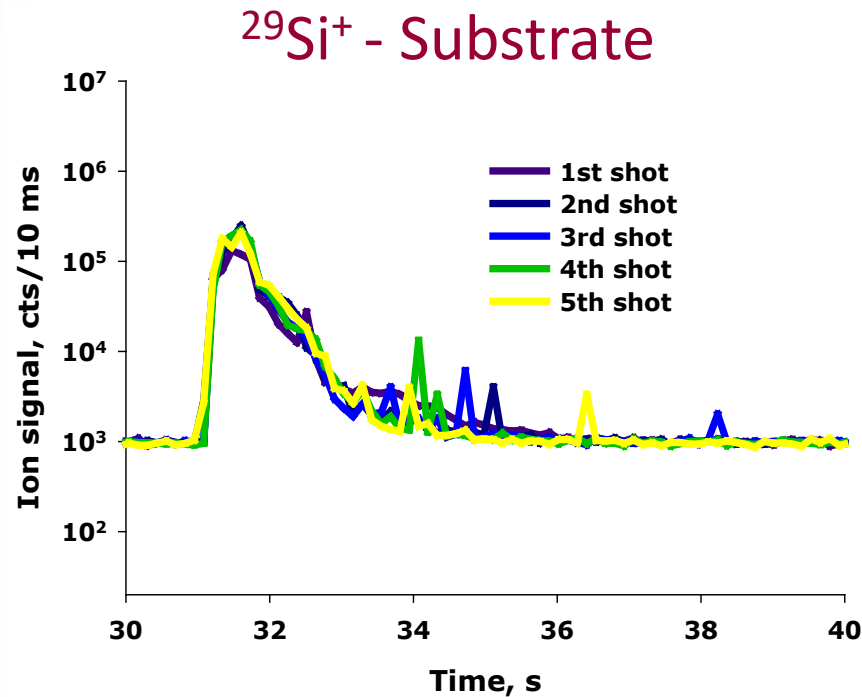
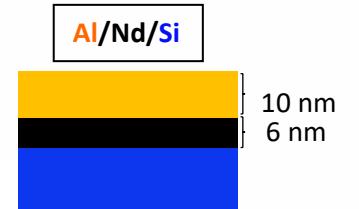
Shots	Depth	Volume	Expected
1	0.04	13.26	13.26
5	0.25	91.56	66.28
10	0.5	171.0	132.6
20	1	349.0	265.1
50	2.7	708.5	662.8
100	5.55	1070	1326
157	8.1	1954	2081



Thin Film Analysis

Ion signal evolution for 5 individual laser pulses

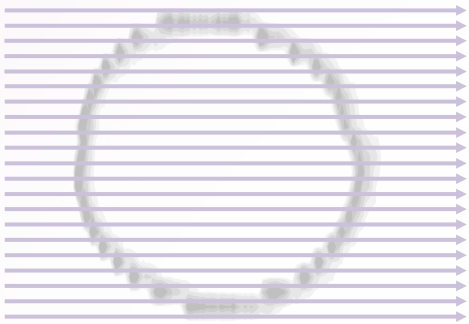
Sample: 6 nm Nd internal layer



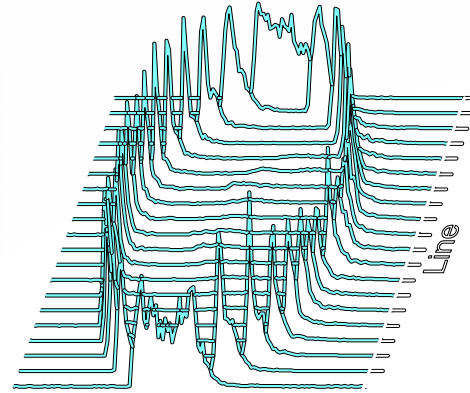
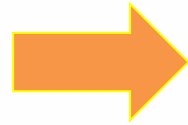
High energy laser pulses can remove thin layers within a few laser pulses



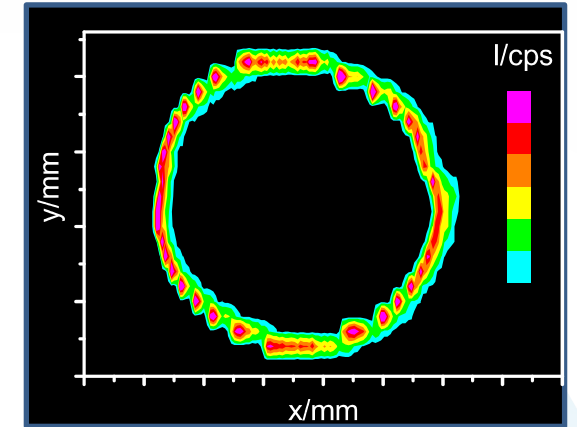
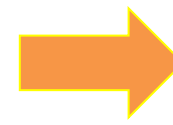
Elemental Imaging – Basics of Map/Image Generation



Line-by-line ablation



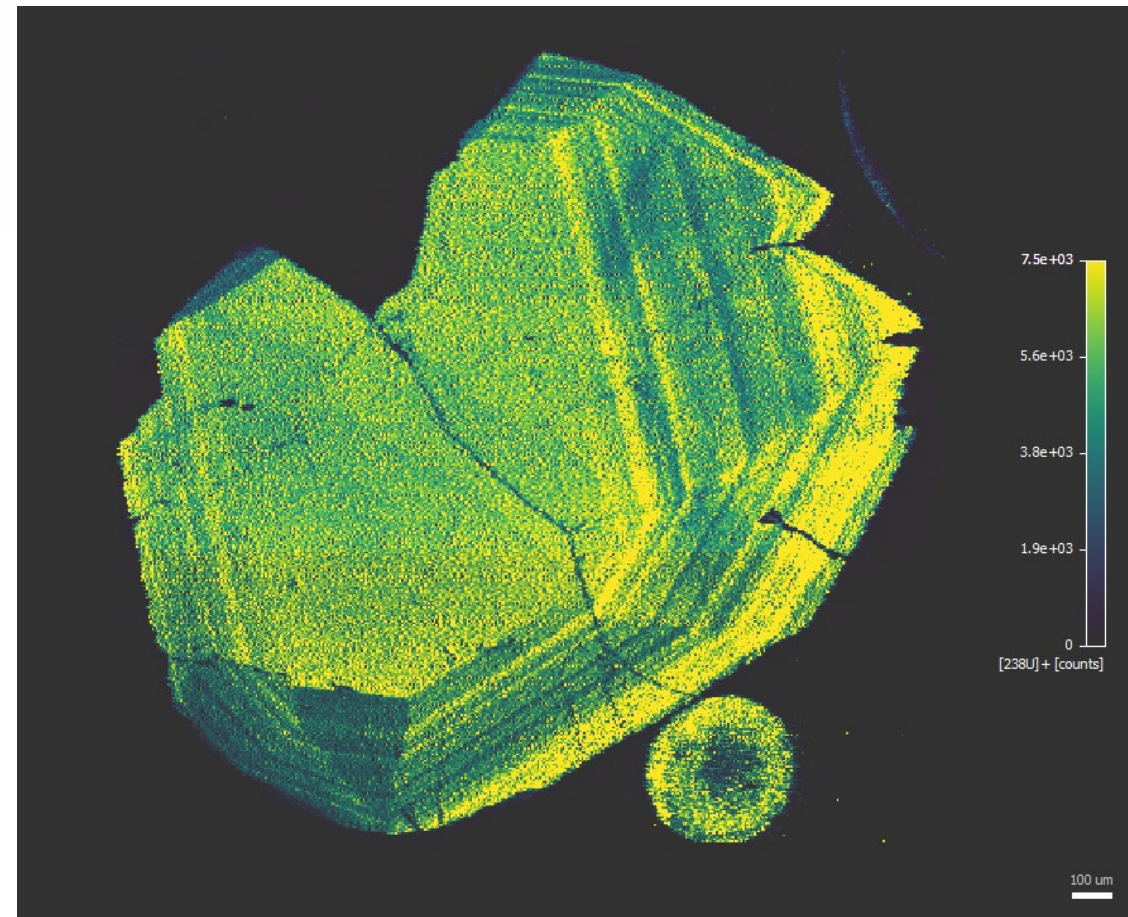
Transient ion traces



Ion intensity image

Elemental Imaging

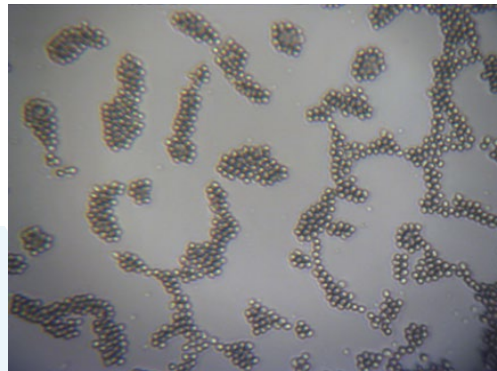
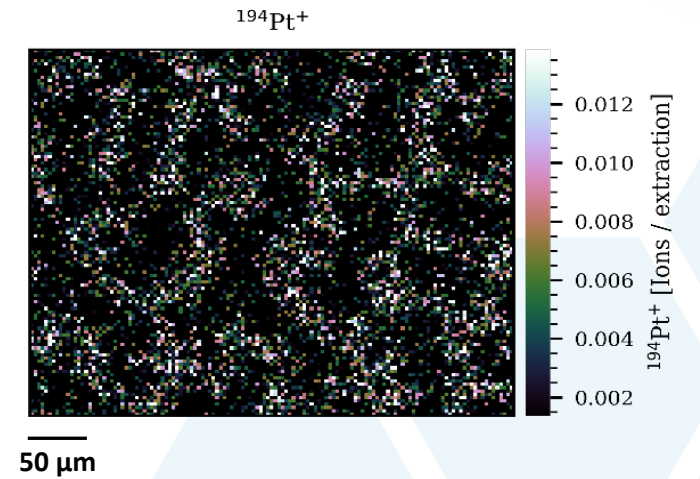
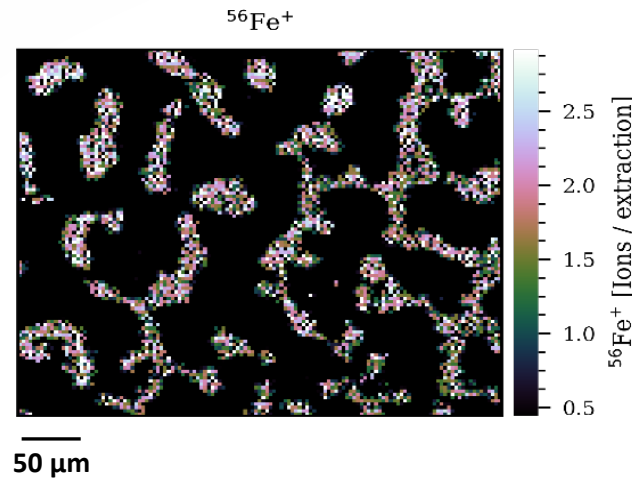
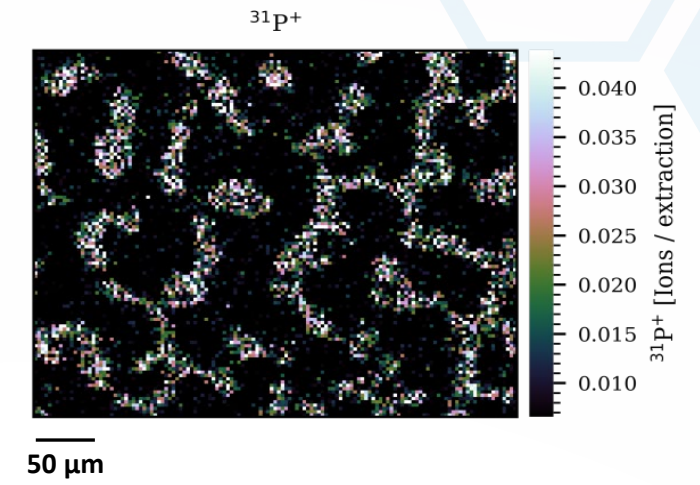
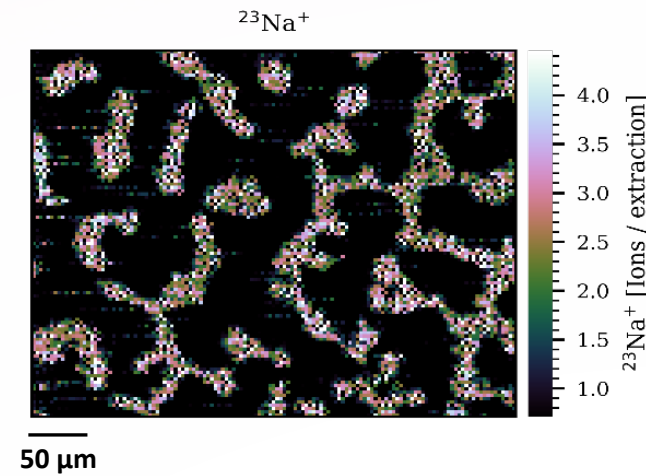
	A	B	C	D	E	F	G	H	I	J	K	L	M
8	0.096912	0.114887	0.583607	0.660054	0.852923	0.218161	0.397852	0.78179	0.633538	0.383446	0.096522	0.502717	1.18885
9	0.146324	0.161749	0.464136	0.69083	0.623064	0.204618	0.404379	0.787971	0.633808	0.384757	0.198939	0.33847	1.19359
10	0.16358	0.155945	0.601307	0.395216	0.692919	0.485034	0.198672	0.556445	0.57554	0.364166	0.13546	0.333279	1.07667
11	0.235245	0.048706	0.774869	0.412034	1.03772	0.49413	0.396634	0.292094	0.385738	0.438952	0.193673	0.304141	1.02072
12	0.39712	0.14578	0.842969	0.40835	0.946479	0.643883	0.193839	0.556434	0.453219	0.189042	0.234477	0.263653	0.662822
13	0.228604	0.079104	1.19217	0.830759	1.16044	0.536759	0.131089	0.130565	0.244766	0.250621	0.202918	0.497544	0.532865
14	0.259311	0.237749	0.864093	0.823291	1.2092	0.696092	0.162978	0.415797	0.220872	0.089312	0.210114	0.775358	0.56165
15	0.390668	0.190997	1.06221	0.999354	1.30664	0.866227	0.227986	0.176978	0.086577	0.13053	0.248586	0.472006	0.35736
16	0.104634	0.409608	1.61818	1.43177	1.31183	0.545197	0.293767	0.264986	0.16443	0.072054	0.091039	0.754845	0.253603
17	1.16384	0.385957	1.65124	0.798049	1.56494	0.66774	0.415711	0.573101	0.046124	0.074338	0.611663	0.610238	0.226235
18	0.369656	0.715347	1.53221	6.00476	1.38987	1.05974	0.666762	1.13793	0.026259	0.211942	0.457764	0.350029	0.116701
19	0.352381	1.07845	4.65948	2.63666	1.17151	1.08914	0.720548	1.27075	0.068776	0.145108	0.386675	0.258985	0.04538
20	0.505702	1.10533	1.41073	1.47368	1.21054	0.930509	0.639065	0.895207	0.026999	0.050367	0.28623	0.430147	0.12626
21	0.599119	0.896266	0.525086	1.20172	1.30803	0.803165	0.563338	0.772326	0.156519	0.096277	0.404549	0.324726	0.103211
22	0.414209	0.801911	0.843391	1.09828	1.25331	0.985016	0.449396	0.53338	0.099202	0.072237	0.305811	0.368388	0.133242
23	0.366787	0.768113	0.531492	1.07642	1.11176	0.987023	0.758482	0.611338	0.056195	0.045279	0.151004	0.23329	0.064937
24	0.458827	0.845649	0.824528	1.03083	1.07516	1.18947	0.514221	0.293982	0.159779	0.113608	0.22589	0.171253	0.041933
25	0.716285	0.663331	0.689056	0.820868	1.02739	1.16858	0.718875	0.474105	0.100959	0.083564	0.164511	0.105662	0.083173
26	0.819503	1.24763	1.42895	0.707344	1.52214	1.09088	0.763535	1.13511	0.033873	0.142139	0.167168	0.099386	0.043056
27	0.912334	2.01484	1.20841	0.724997	1.44369	1.09532	0.917739	1.39346	0.048793	0.14082	0.097433	0.074208	0.03787
28	1.15607	3.04507	1.26789	0.62716	1.25552	0.793643	1.00461	0.989637	0.03223	0.085011	0.113863	0.018183	0.098037
29	1.15575	2.83549	1.06188	0.610768	1.54787	0.909811	0.55543	1.672	0.064739	0.10096	0.171191	0.05557	0.056548
30	1.12294	1.72703	0.717315	0.934489	1.30029	1.02686	0.785505	1.44898	0.024459	0.053282	0.110676	0.115487	0.096095
31	1.67334	1.15917	0.571676	0.631869	1.5819	0.646488	0.656882	1.15551	0.027585	0.079305	0.118181	0.086831	0.154575
32	1.6434	0.868305	0.285532	0.760374	1.91634	0.746576	0.729745	1.14162	0.033734	0.076443	0.050522	0.097287	0.204627
33	1.65086	0.671309	0.166098	0.58059	1.78824	0.983115	0.774283	1.11937	0.034717	0.162403	0.054783	0.087952	0.131606
34	1.30019	0.857228	0.160557	0.581421	1.67726	0.202466	1.25894	0.751958	0.016627	0.103778	0.056284	0.066515	0.162199
35	1.00326	0.564484	0.228819	0.760232	1.27207	1.84559	1.43669	0.496919	0.041127	0.173992	0.057838	0.080648	0.181131
36	0.775591	0.41202	0.417392	0.642002	0.946925	1.4467	1.69969	0.354116	0.054937	0.128461	0.08167	0.083234	0.138208
37	0.662539	0.370612	0.532953	0.636069	0.983973	1.55404	1.32917	0.202616	0.021751	0.119431	0.072847	0.085618	0.138608
38	0.463375	0.31527	0.358647	0.586981	1.13203	1.17624	1.12798	0.188072	0.03207	0.065468	0.060936	0.055543	0.088213
39	0.540517	0.30932	0.42259	0.42403	1.23246	0.904797	0.651712	0.114582	0.110343	0.144098	0.092224	0.05494	0.060053
40	0.38211	0.329588	0.45556	0.863497	0.902195	0.664446	0.376653	0.074541	0.080114	0.064662	0.094256	0.129623	0.165235
41	0.421603	0.392585	0.279268	0.932795	0.900197	0.355988	0.44882	0.091691	0.170302	0.099841	0.138096	0.078876	0.125028
42	0.390633	0.381289	0.360255	0.811816	0.628627	0.361066	0.198197	0.126083	0.085758	0.11067	0.039471	0.077577	0.05944
43	0.347463	0.272831	0.298817	0.742719	0.252145	0.301427	0.175459	0.117354	0.067837	0.076817	0.032003	0.072841	0.083575



Courtesy of Dr. Gavril Sabau, Geological Institute of Romania

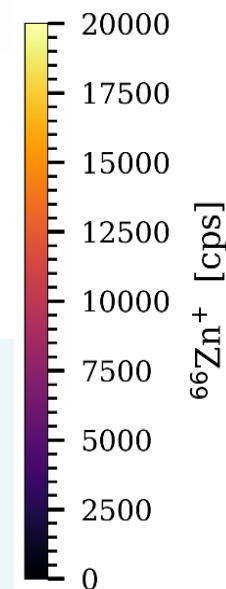
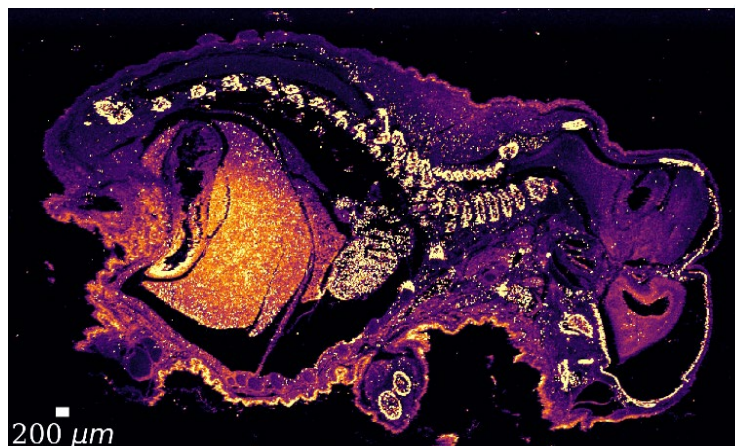
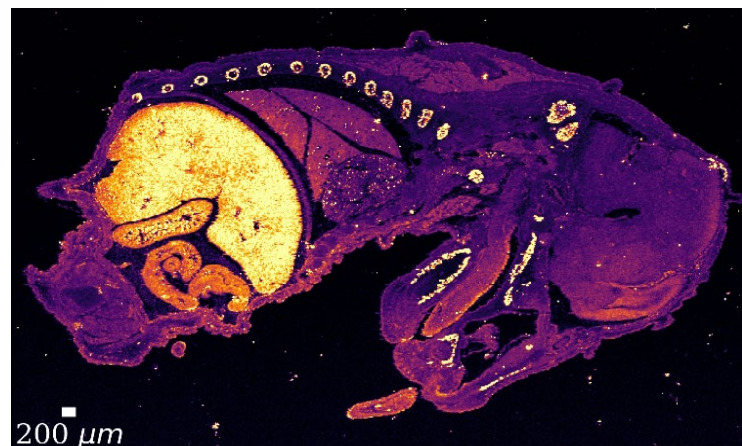
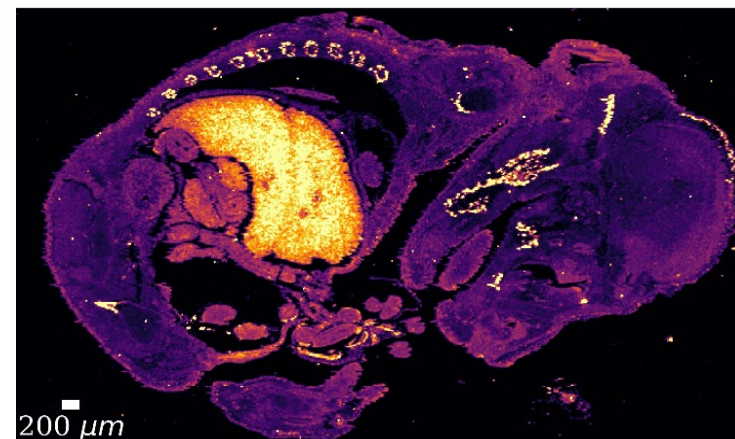
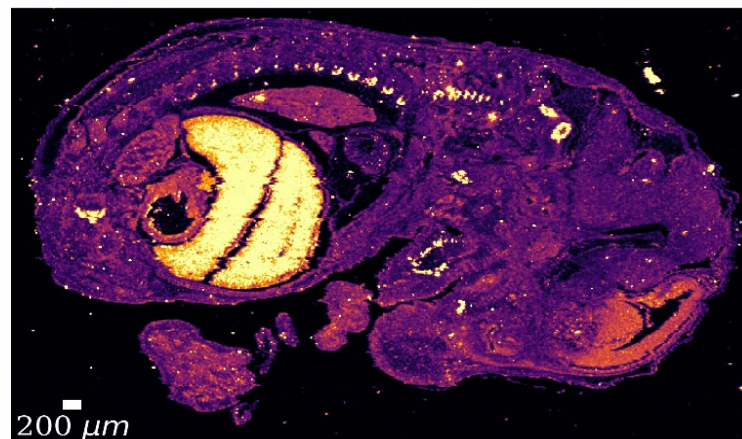
Elemental Imaging

- Red blood cells of a patient after cisplatin chemotherapy (Courtesy of Dr. Sarah Theiner, University of Vienna)
- 2 μm resolution
- 100 Hz
- Dosage: 4
- Imaging Time: 20 min



Elemental Imaging

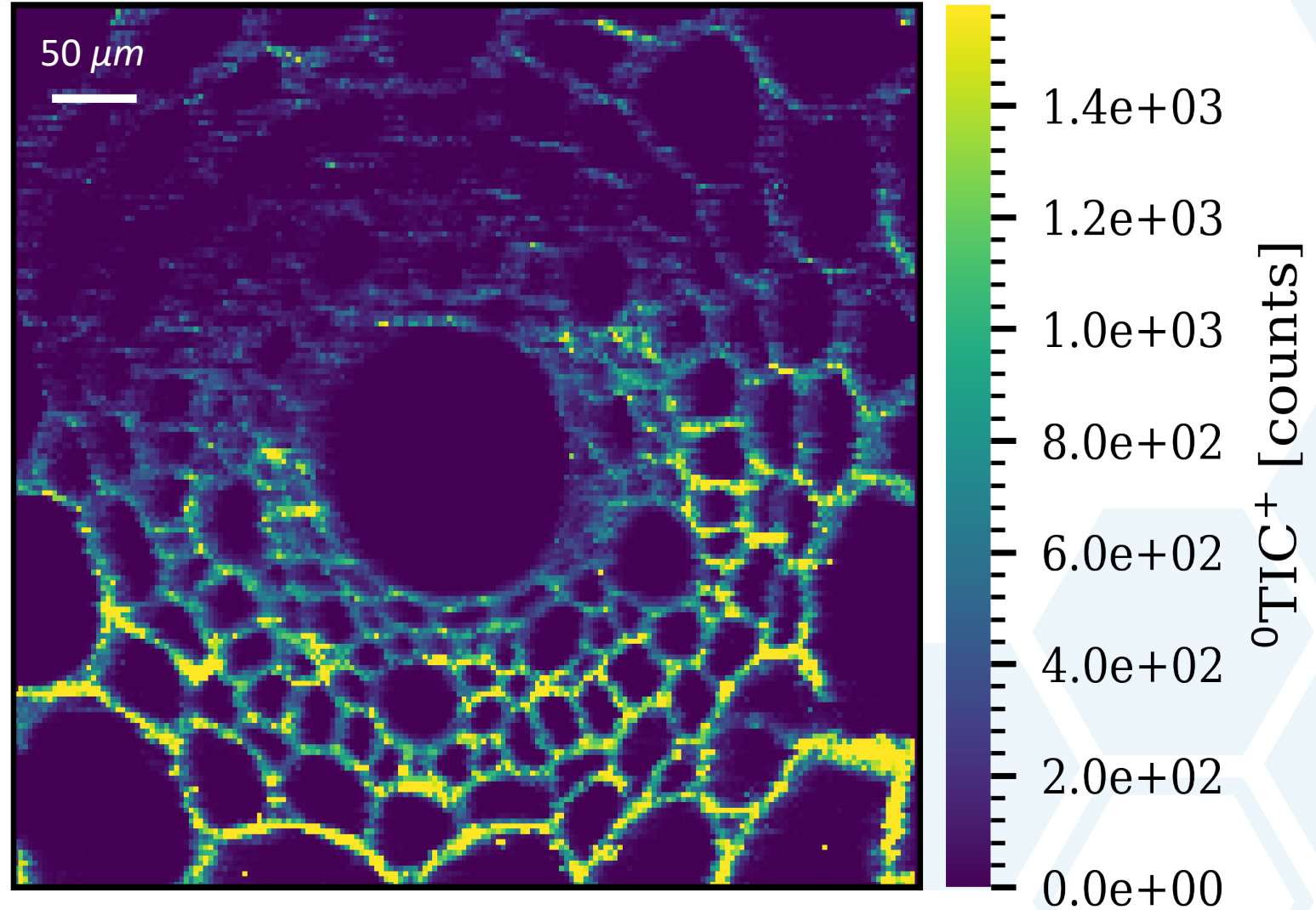
- Mouse Embryo (unpublished data)
- 5 μm thickness, deparaffinized prior to analysis
- 8 hours analysis time for 18 samples
- 19 μm Spot
- Dosage: 1
- Energy Density: 0.93 J cm^2
- Scan Speed: 2 mm/s
- Rep Rate: 100Hz



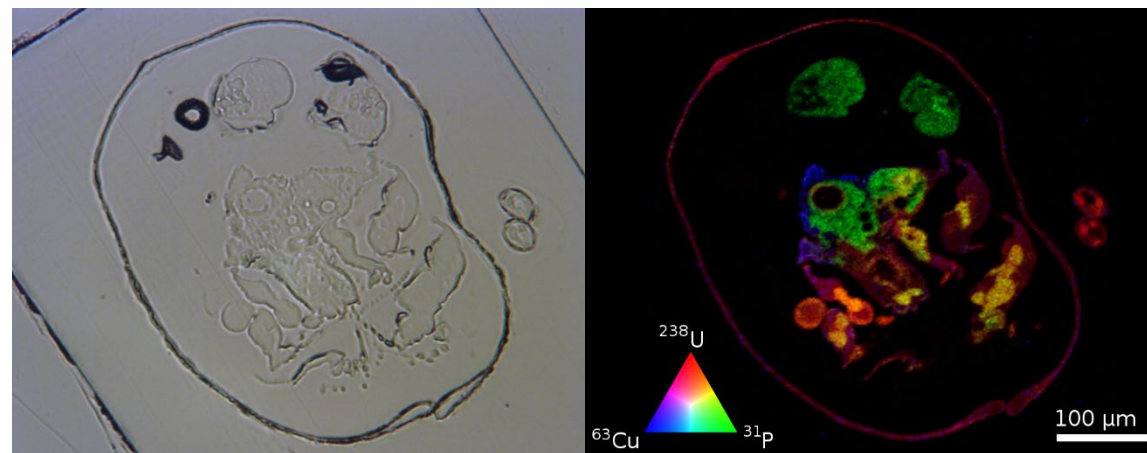
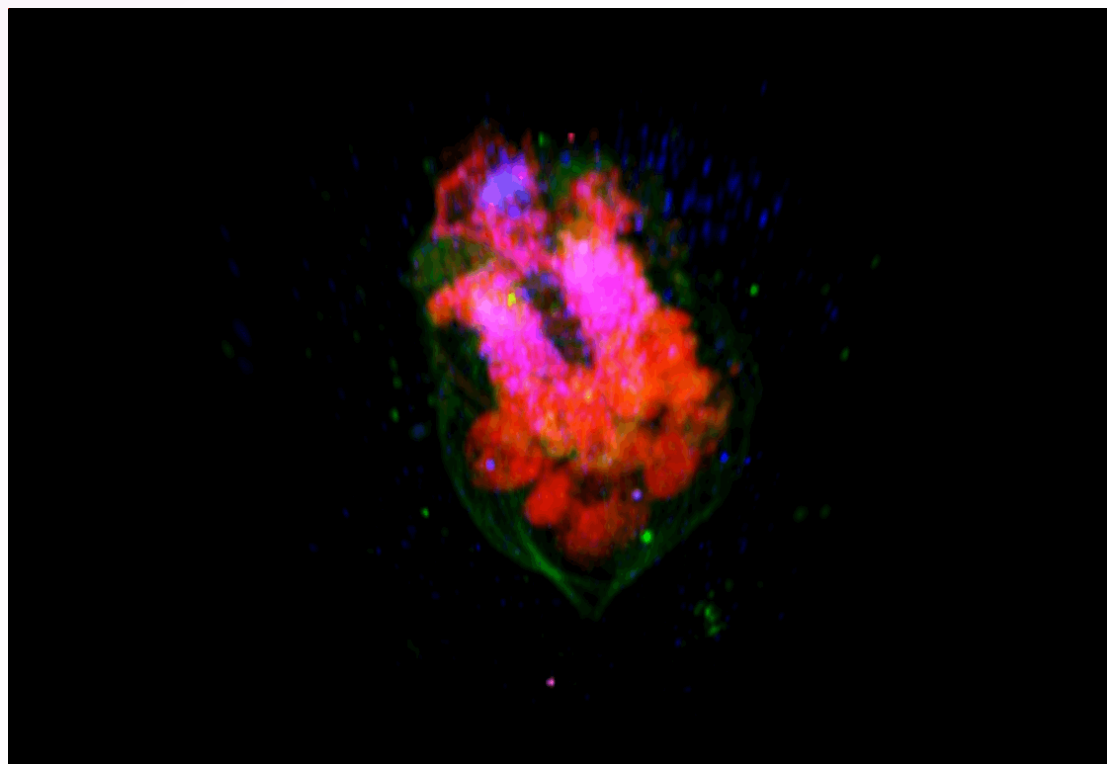
Elemental Imaging

- Barley root section
- 1 μm resolution,
- 300 Hz
- 1 pulse/pixel
- Total Imaging Time: 3.2 min

Root4_Mg_1 μm



HDIP Software – Powerful Simplicity (3D Imaging)



Three-Dimensional Reconstruction of the Tissue-Specific Multi-elemental Distribution within *Ceriodaphnia dubia* via Multimodal Registration Using Laser Ablation ICP-Mass Spectrometry and X-ray Spectroscopic Techniques. Stijn J. M. Van Malderen et al, DOI: [10.1021/acs.analchem.7b00111](https://doi.org/10.1021/acs.analchem.7b00111)

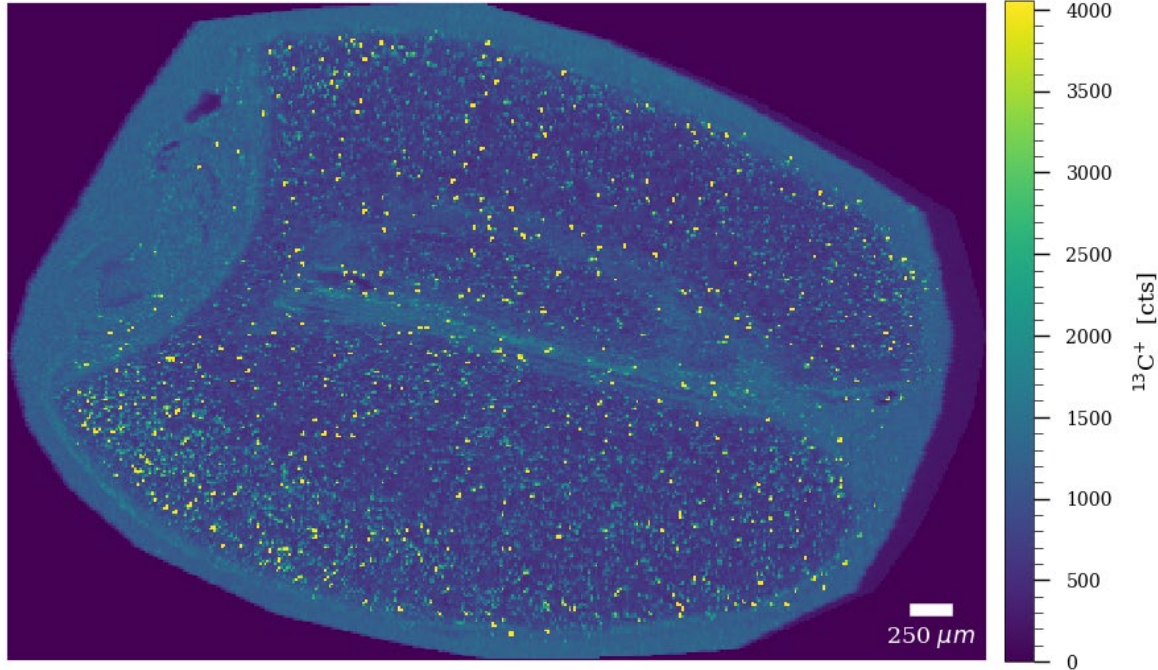
Data Collected at 400Hz



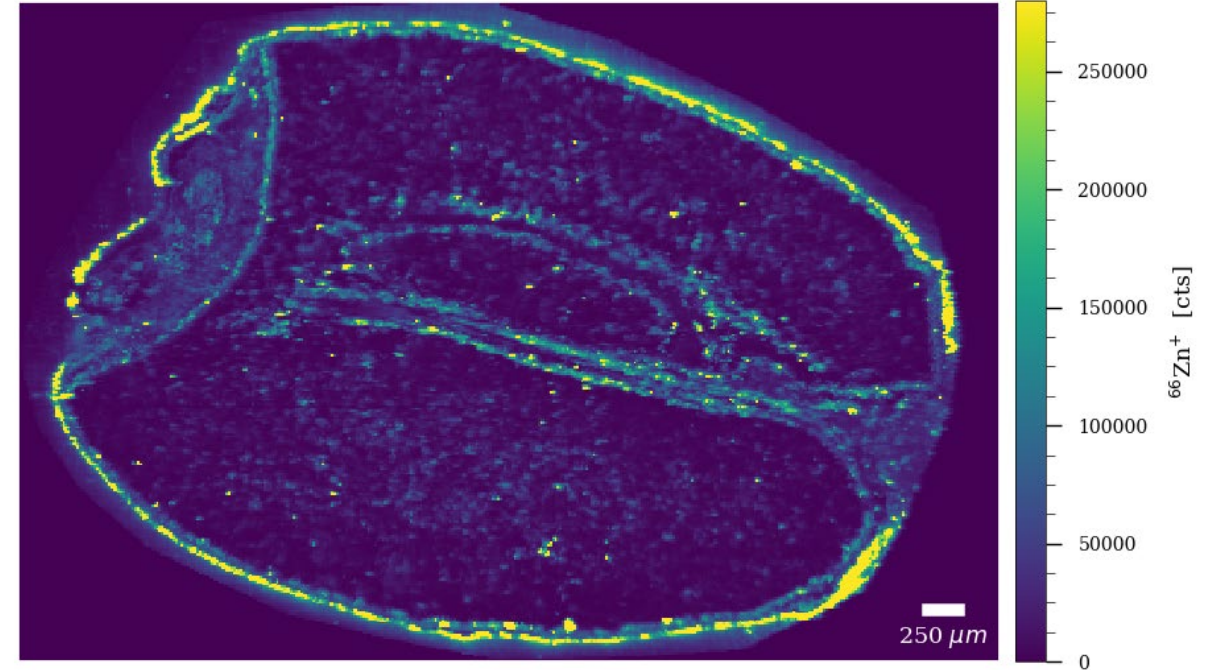
- Wheat grain (unpublished data)
- Mounted in epoxy resin and polished to give a flat surface
- Interested in zinc uptake
 - Concentration & location

Data Collected at 400Hz

Carbon¹³

¹³C⁺

Zinc⁶⁶

⁶⁶Zn⁺

50 minutes

- **2 elements measured:** Carbon and Zinc
- **15 μm** circular spot; **400 Hz**
- Cobalt *Long Pulse* cell
- Agilent 8900 ICP-MS
- Parameters optimized and images generated with Teledyne HDIP software



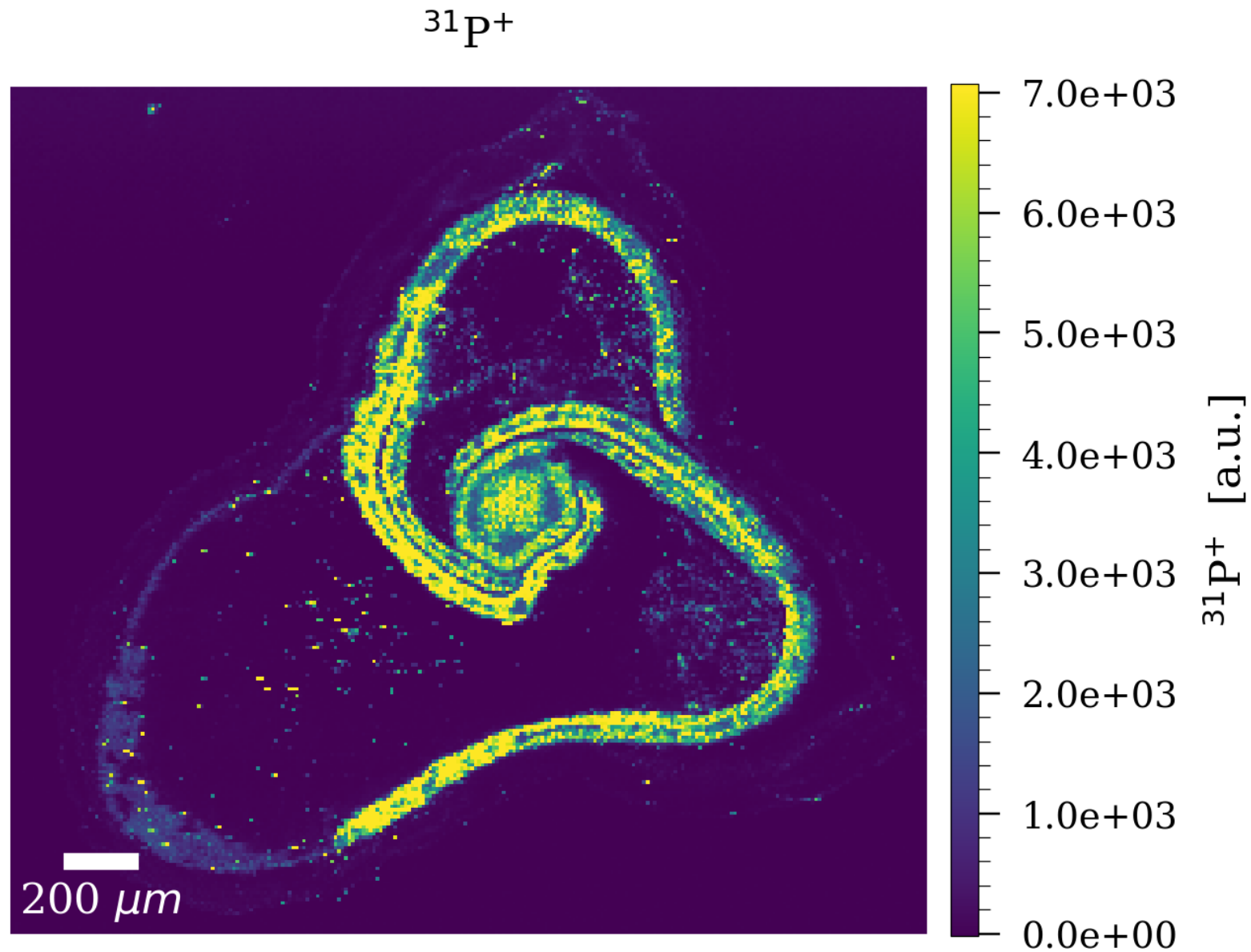
HDIP 1.6

Mass Spectrometry Data Analysis

Designed by scientists for scientists. Available for Teledyne Photon Machines laser systems.

Elemental Imaging

- Buckwheat section (unpublished data).
Courtesy of Dr. Martin Sala, National
Institute of Chemistry Slovenia
- 10 μm resolution
- 300 Hz
- 4 pulse/pixel



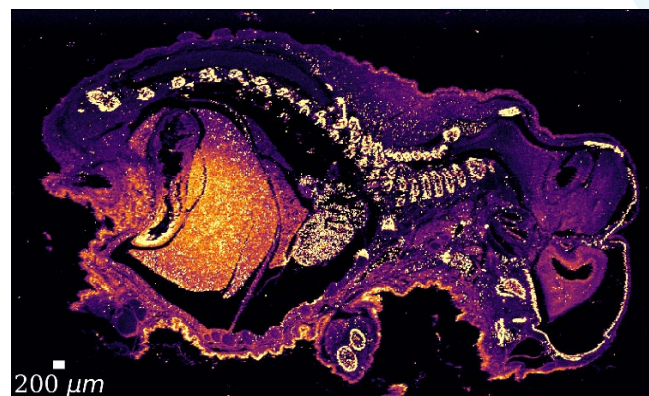
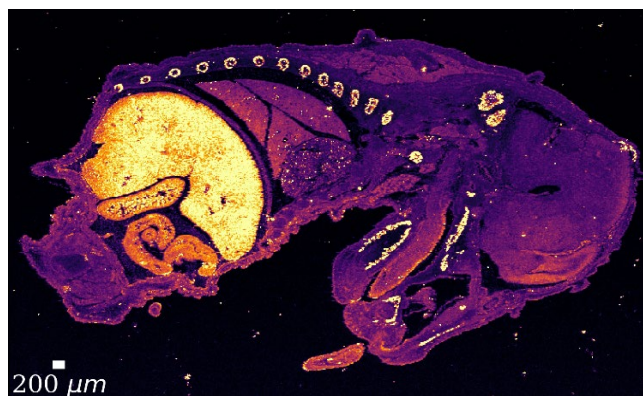
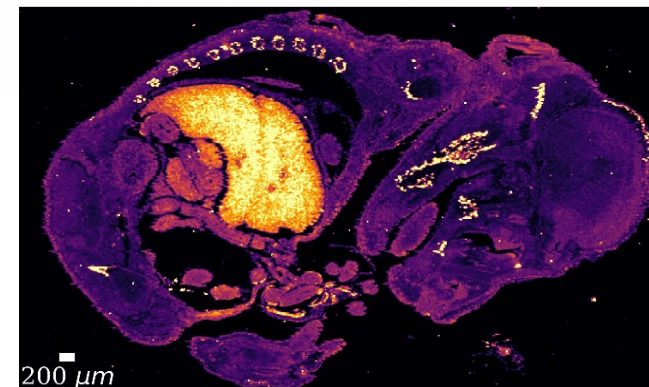
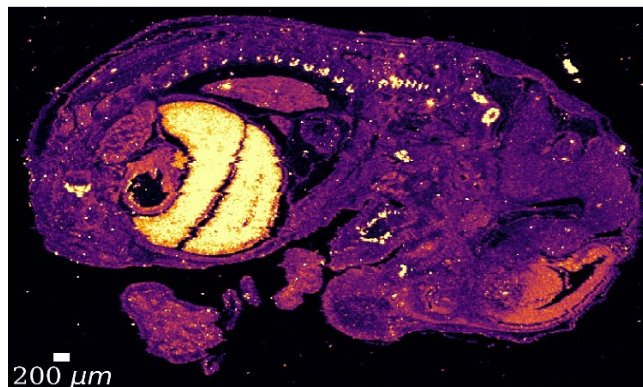
Bulk Analysis

	Test 1 (10 Hz; Total Flow: 0.65 LPM)				Test 2 (50 Hz; Total Flow: 0.75 LPM)				Test 3 (50 Hz; Total Flow: 1.05 LPM)			
	[7Li]+	[89Y]+	[139La]+	[238U]+	[7Li]+	[89Y]+	[139La]+	[238U]+	[7Li]+	[89Y]+	[139La]+	[238U]+
Replicate 1 mean [cts]	2.62E+03	7.03E+03	8.89E+03	8.66E+03	8.81E+03	2.65E+04	3.20E+04	2.75E+04	8.81E+03	2.71E+04	3.25E+04	2.90E+04
Replicate 2 mean [cts]	2.66E+03	7.00E+03	8.74E+03	8.43E+03	8.97E+03	2.65E+04	3.22E+04	2.88E+04	8.84E+03	2.68E+04	3.22E+04	2.88E+04
Replicate 3 mean [cts]	2.71E+03	6.95E+03	8.68E+03	8.70E+03	8.98E+03	2.65E+04	3.24E+04	2.85E+04	9.09E+03	2.67E+04	3.24E+04	2.86E+04
Replicate 4 mean [cts]	2.74E+03	7.02E+03	8.69E+03	8.53E+03	9.13E+03	2.67E+04	3.22E+04	2.92E+04	9.03E+03	2.69E+04	3.25E+04	2.88E+04
Replicate 5 mean [cts]	2.73E+03	6.95E+03	8.85E+03	8.53E+03	9.16E+03	2.67E+04	3.26E+04	2.88E+04	9.06E+03	2.66E+04	3.22E+04	2.87E+04
Replicate 6 mean [cts]	2.81E+03	6.93E+03	8.62E+03	8.56E+03	9.09E+03	2.65E+04	3.24E+04	2.91E+04	9.06E+03	2.68E+04	3.24E+04	2.85E+04
Replicate 7 mean [cts]	2.72E+03	7.00E+03	8.81E+03	8.54E+03	9.15E+03	2.67E+04	3.25E+04	2.85E+04	8.90E+03	2.68E+04	3.25E+04	2.87E+04
Replicate 8 mean [cts]	2.68E+03	6.99E+03	8.78E+03	8.56E+03	8.96E+03	2.66E+04	3.22E+04	2.81E+04	8.95E+03	2.68E+04	3.23E+04	2.87E+04
Replicate 9 mean [cts]	2.65E+03	6.94E+03	8.75E+03	8.48E+03	8.98E+03	2.65E+04	3.22E+04	2.86E+04	8.92E+03	2.68E+04	3.25E+04	2.89E+04
Replicate 10 mean [cts]	2.65E+03	7.10E+03	8.77E+03	8.65E+03	8.92E+03	2.66E+04	3.22E+04	2.83E+04	8.95E+03	2.71E+04	3.26E+04	2.90E+04
Mean signal [cts]	2.70E+03	6.99E+03	8.76E+03	8.56E+03	9.02E+03	2.66E+04	3.23E+04	2.85E+04	8.96E+03	2.68E+04	3.24E+04	2.88E+04
Median signal [cts]	2.58E+03	6.83E+03	8.49E+03	8.21E+03	8.98E+03	2.65E+04	3.22E+04	2.85E+04	8.93E+03	2.68E+04	3.23E+04	2.86E+04
Measurement uncertainty mean (1SD) [cts]	1.65E+01	1.59E+01	2.42E+01	2.54E+01	3.41E+01	2.61E+01	5.31E+01	1.46E+02	2.89E+01	5.24E+01	3.85E+01	5.18E+01
Measurement uncertainty replicates (1SD) [cts]	5.22E+01	5.01E+01	7.64E+01	8.02E+01	1.08E+02	8.26E+01	1.68E+02	4.62E+02	9.12E+01	1.66E+02	1.22E+02	1.64E+02
Absolute total combined uncertainty on the mean (1SD) [cts]	1.66E+01	1.59E+01	2.42E+01	2.54E+01	3.42E+01	2.61E+01	5.31E+01	1.46E+02	2.89E+01	5.24E+01	3.85E+01	5.18E+01
Relative total combined uncertainty on the mean (1SD) [%]	0.62%	0.23%	0.28%	0.30%	0.38%	0.10%	0.16%	0.51%	0.32%	0.20%	0.12%	0.18%

- 0.01 s dwell time; 15 μm spot size; 3.5 Jcm^{-2}
- 10 lines of 30s each @ different lasing parameters:
 - Test 1(light green) – 10 Hz, total gas flow of 0.65 LPM (0.35 LPM in MFC1 and 0.3 LPM in MFC2)
 - Test 2 (light blue) – 50 Hz, total flow of 0.75 LPM (0.45 LPM in MFC1 and 0.3 LPM in MFC2)
 - Test 3 (light orange) – 50 Hz, total flow of 1.05 LPM (0.525 LPM in MFC1 and 0.525 LPM in MFC2)

Summary

- Laser ablation provides outstanding sample flexibility
- Capable of single spot or bulk analysis
- Capable of generating sample maps in either 2D or 3D
- Technique developed need for automated data interrogation software
- An excellent addition to your “analytical toolbelt”



ADVANCED ANALYTICAL SCANNING TRANSMISSION ELECTRON MICROSCOPY (STEM), AND FUTURE DIRECTIONS

SPEAKER:

Patrick Phillips, PhD

Asst. TEM Product Manager,
JEOL USA

March 24, 2022 | 11am PT



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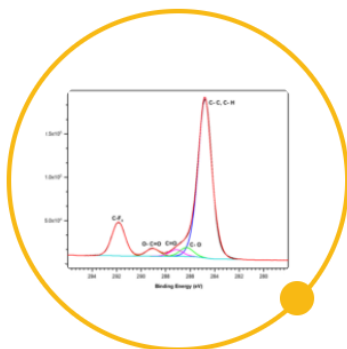
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Q & A Session



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Thank you