INNOVATIVE SURFACE ANALYSIS

Latest Developments in 2D and 3D TOF-SIMS Analysis

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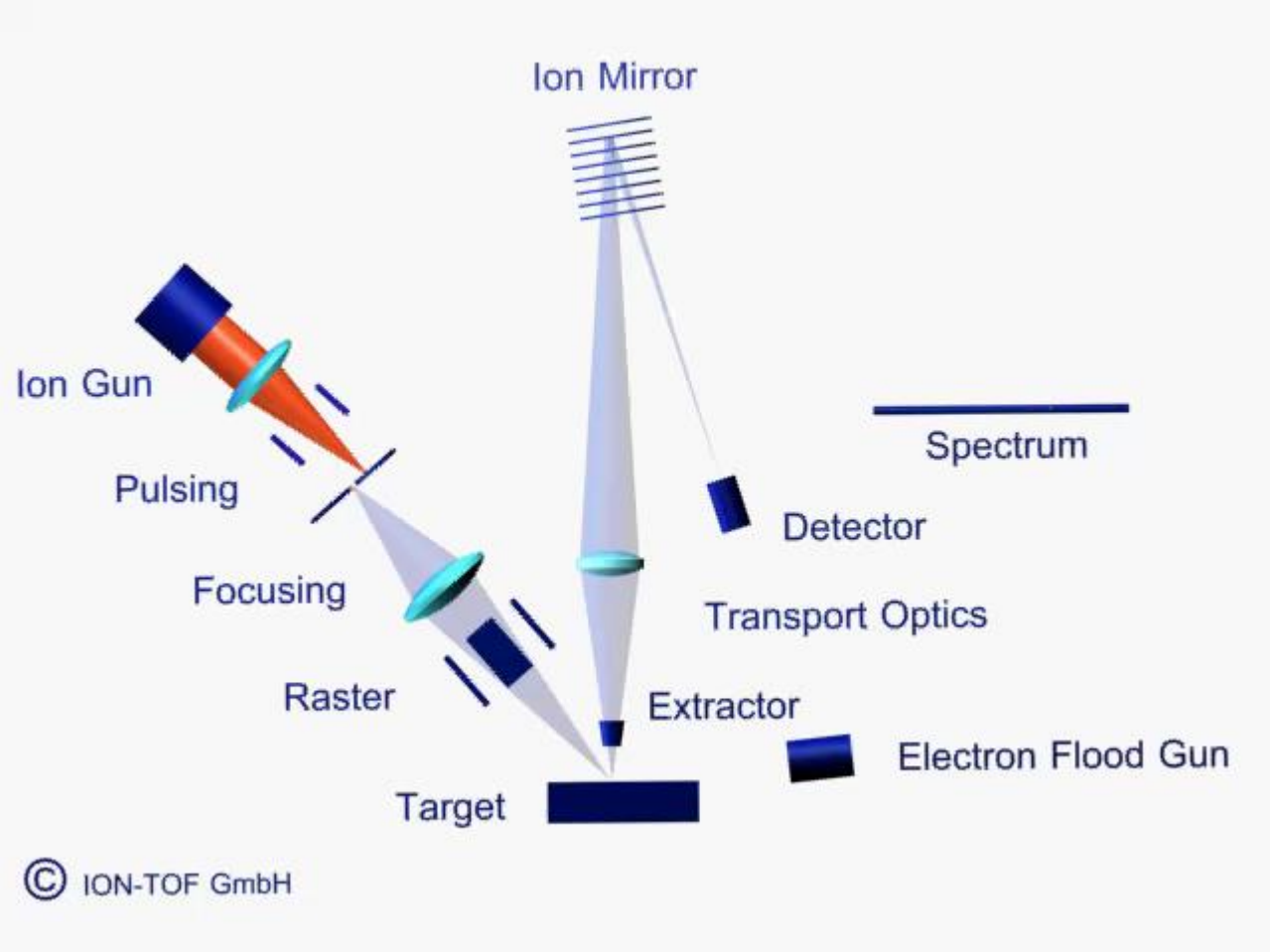
IONTOF

Superior Performance for all SIMS Applications

INNOVATIVE SURFACE ANALYSIS

Secondary Ion Mass Spectrometry



Modes of Operation

INNOVATIVE SURFACE ANALYSIS

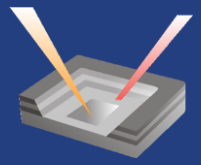
Surface Spectrometry

quasi non-destructive surface analysis of the outer monolayers
elemental and molecular information
ppm/ppb sensitivity

Surface Imaging

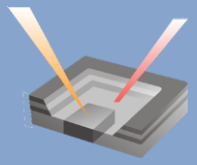
chemical mapping of the surface
lateral distribution of elements and molecules
lateral resolution down to 50 nm
parallel acquisition of all images

Depth Profiling



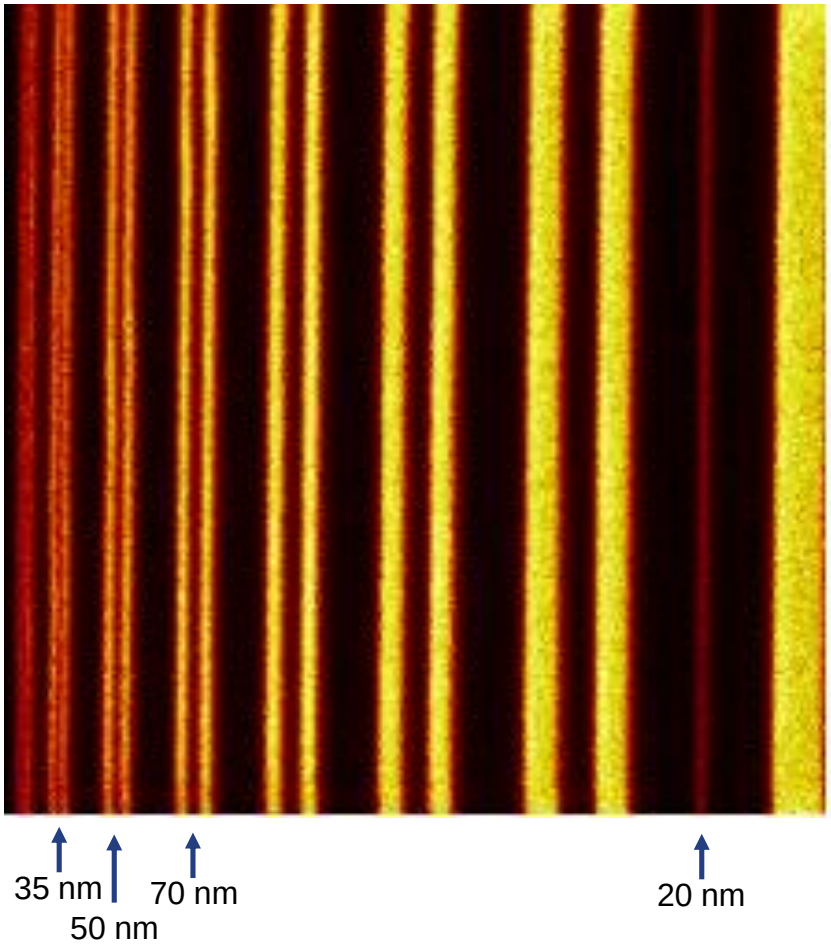
analysis of the in-depth distribution
elemental and molecular information
depth resolution < 1 nm
from a few nm to several µm

3D Analysis

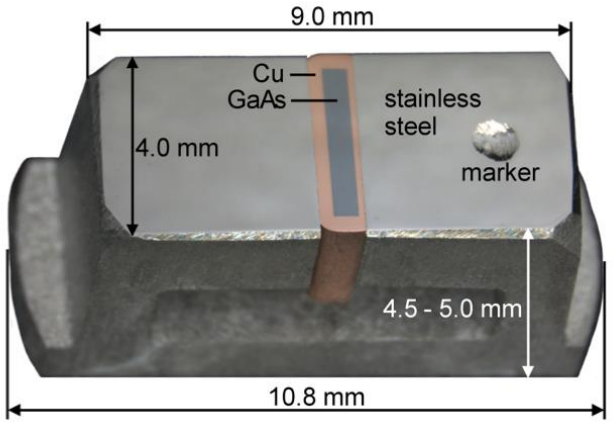


combination of imaging and in-depth information
elemental and molecular information

Ultimate 2D Imaging Resolution

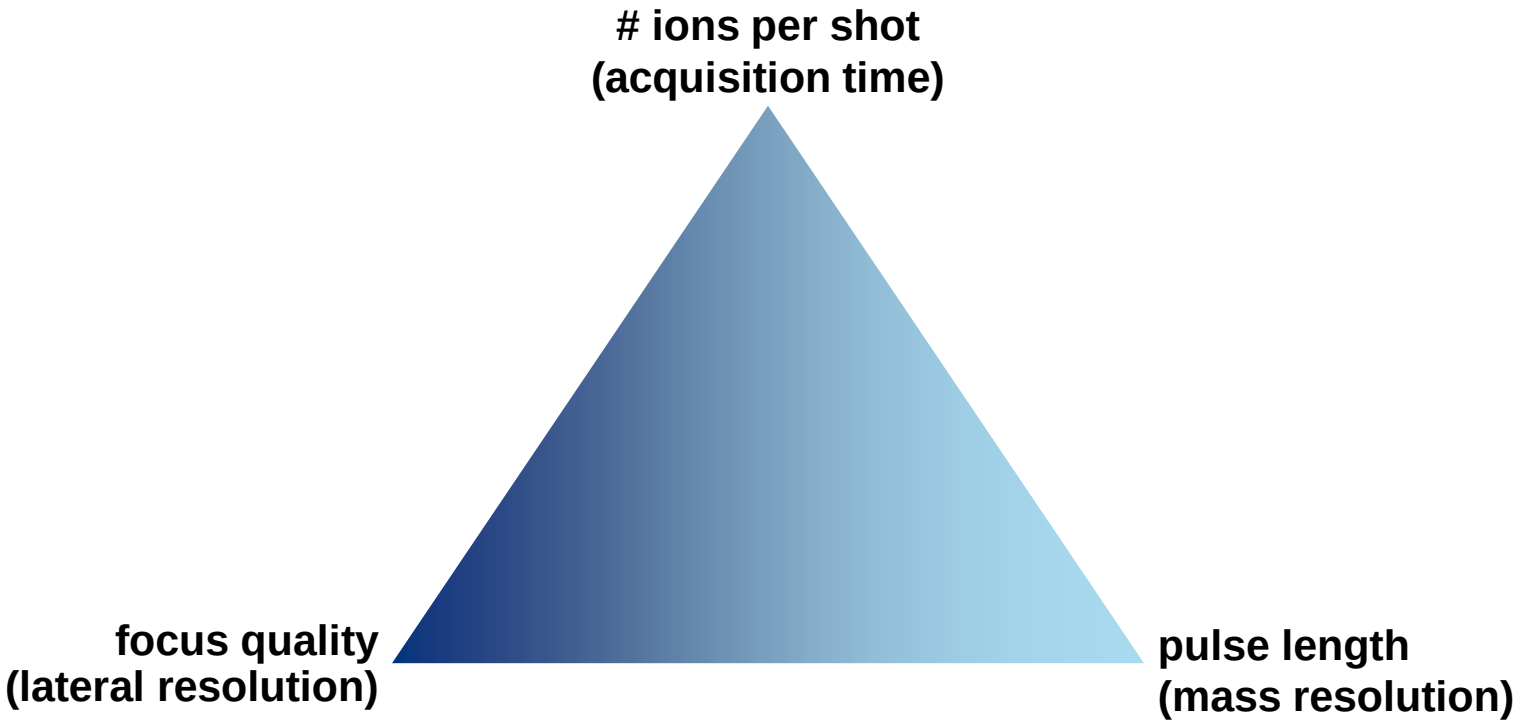


Primary ion:	Bi_1^+
Field of view:	5 x 5 µm²
Pixel resolution:	512 x 512 pixel
Resolution:	**< 50 nm**

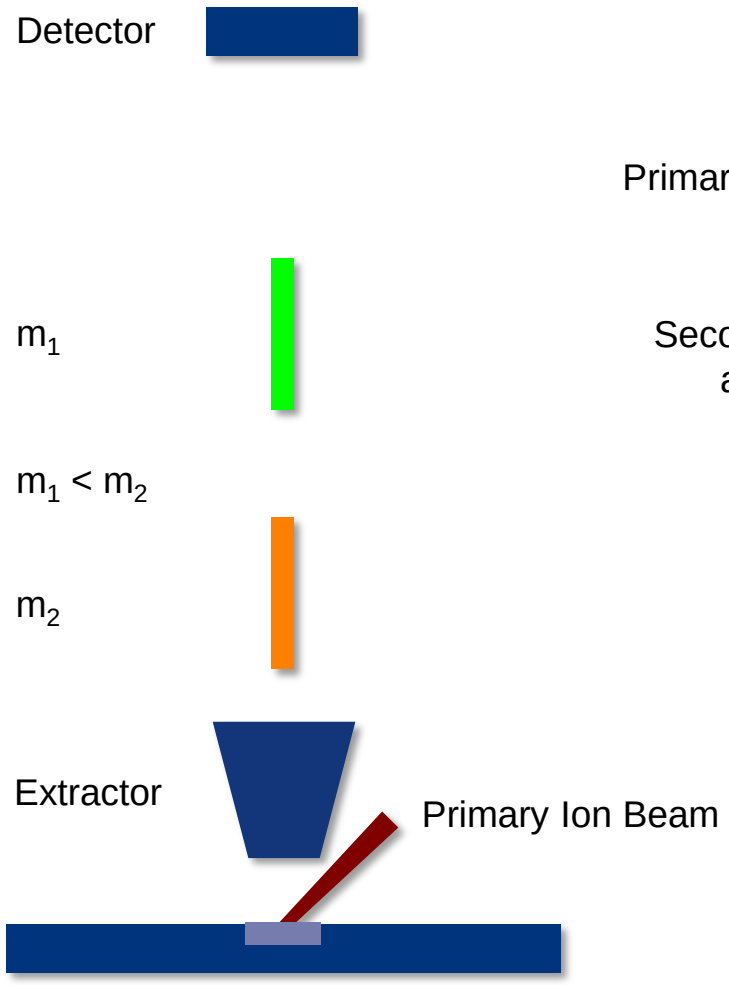


BAM test sample L-200

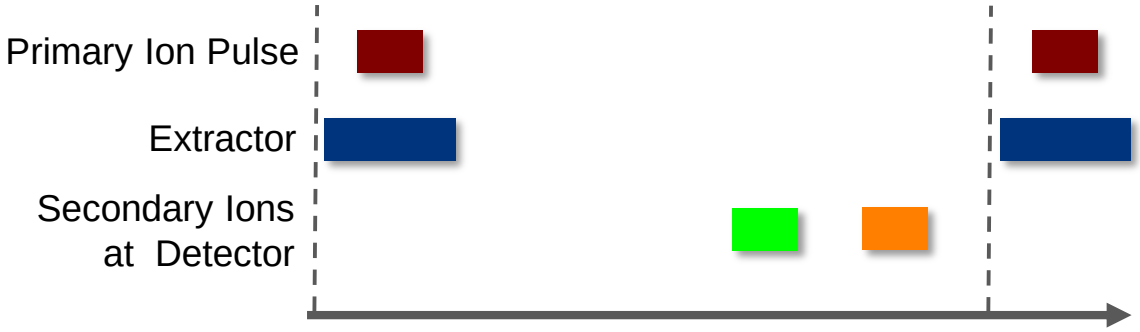
Mass Resolution vs. Lateral Resolution



TOF Principle

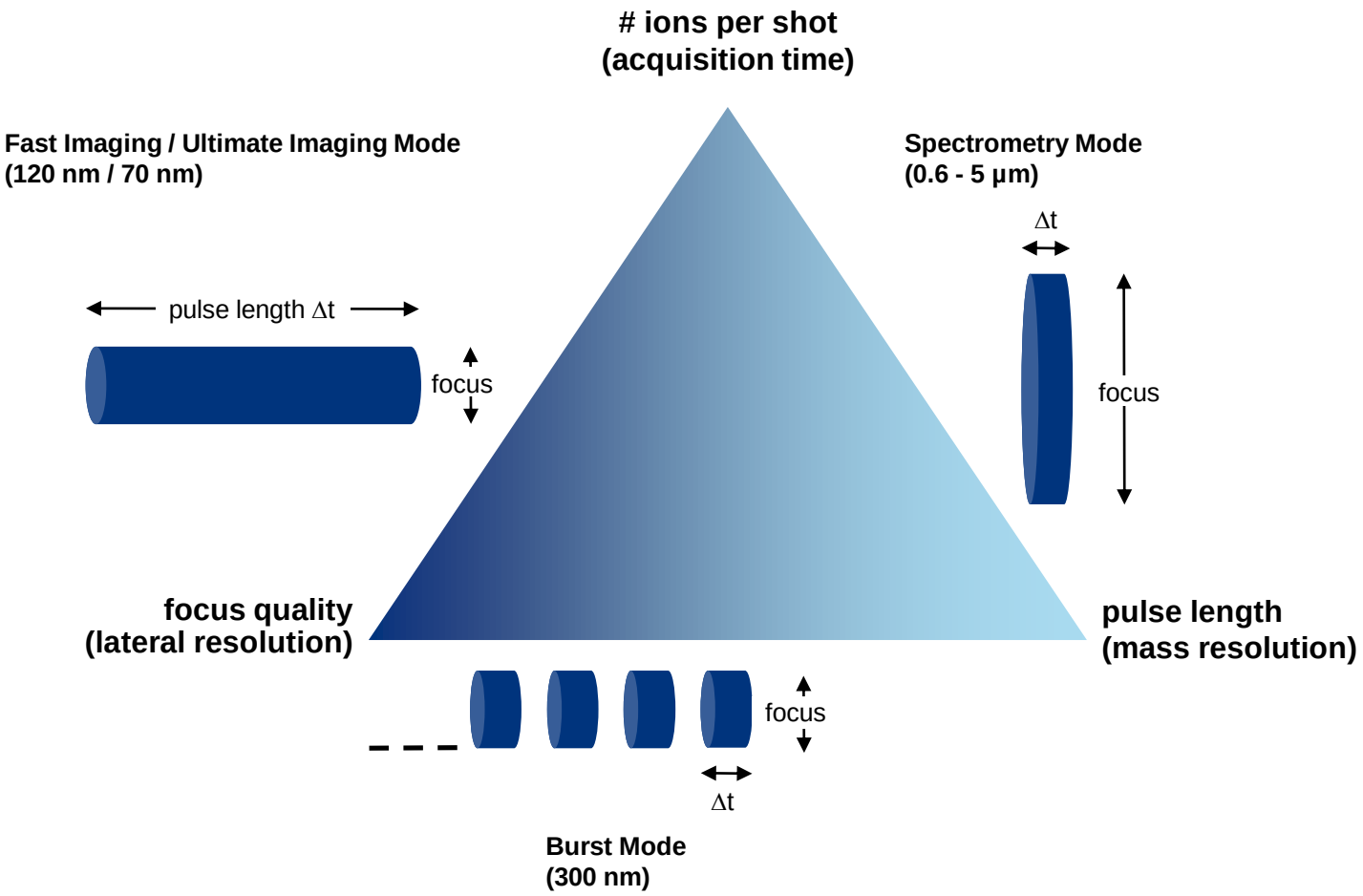


Static Extraction

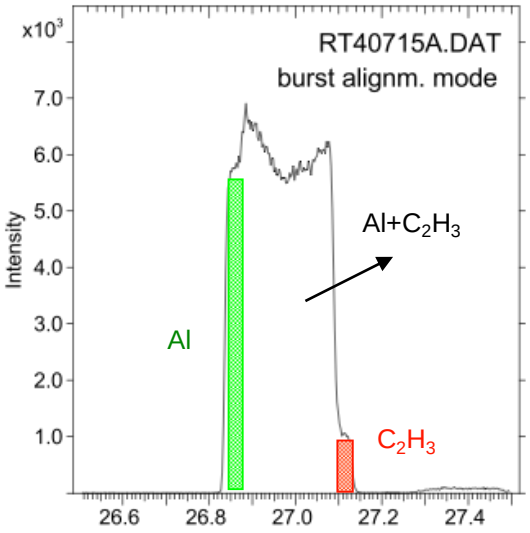


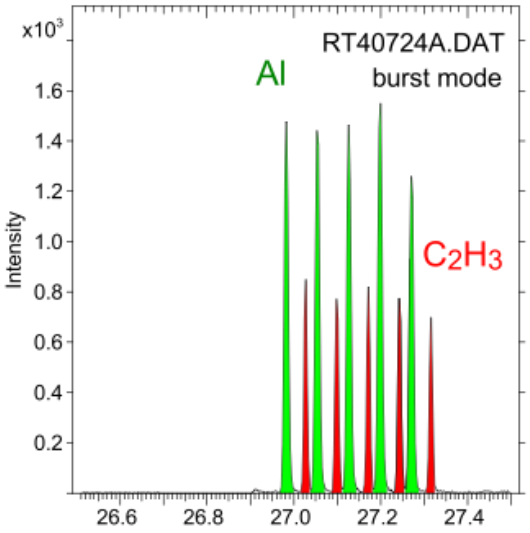
- High mass resolution requires short primary ion pulses

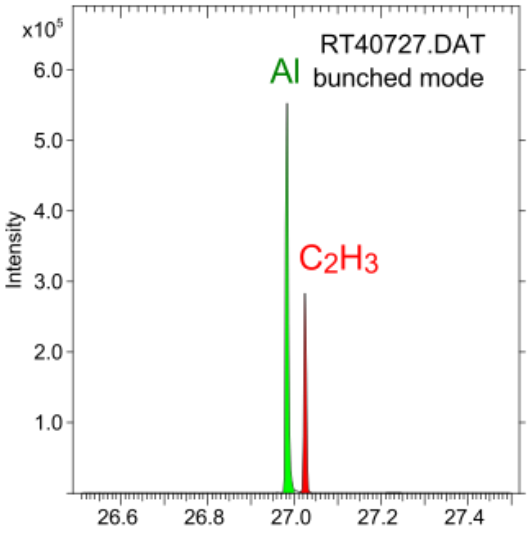
Modes of Operation

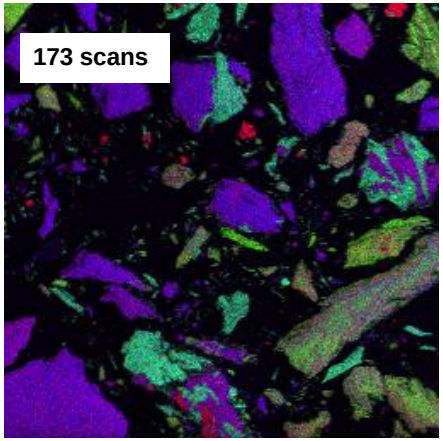


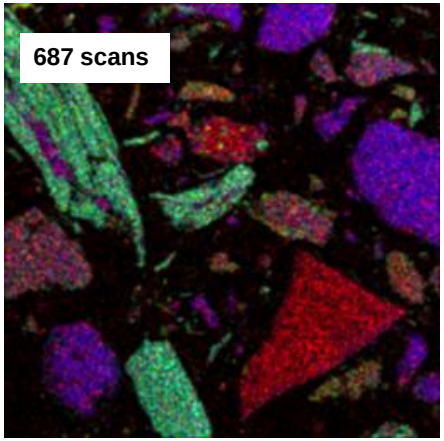
Mass Resolution vs. Lateral Resolution

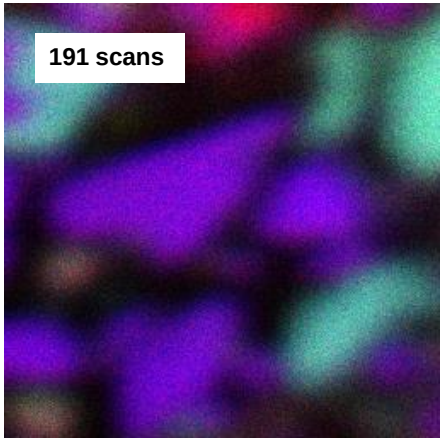






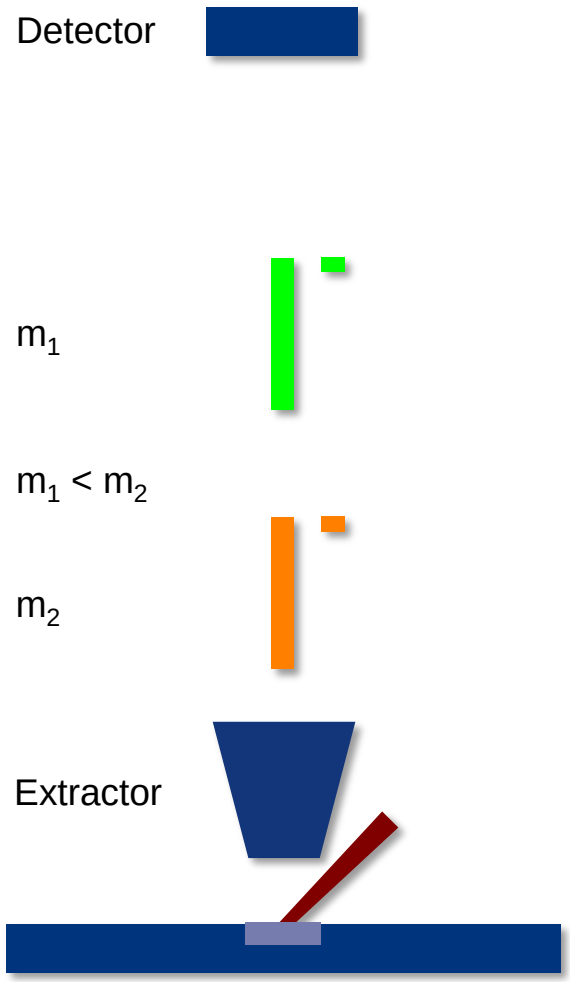




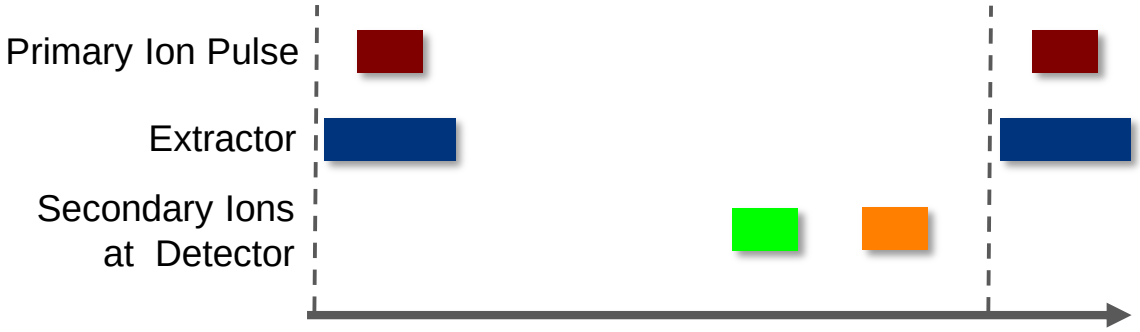


samples provided by Dr. van Leerdam, Akzo Nobel Chemicals bv, The Netherlands

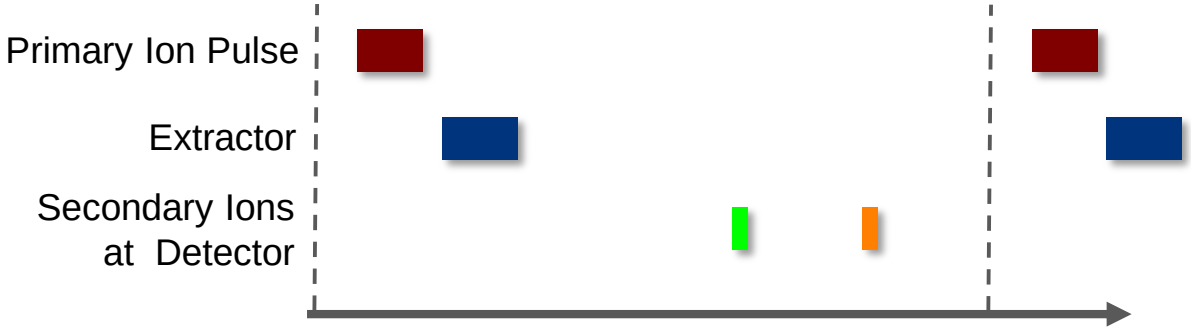
Separating Mass and Lateral Resolution



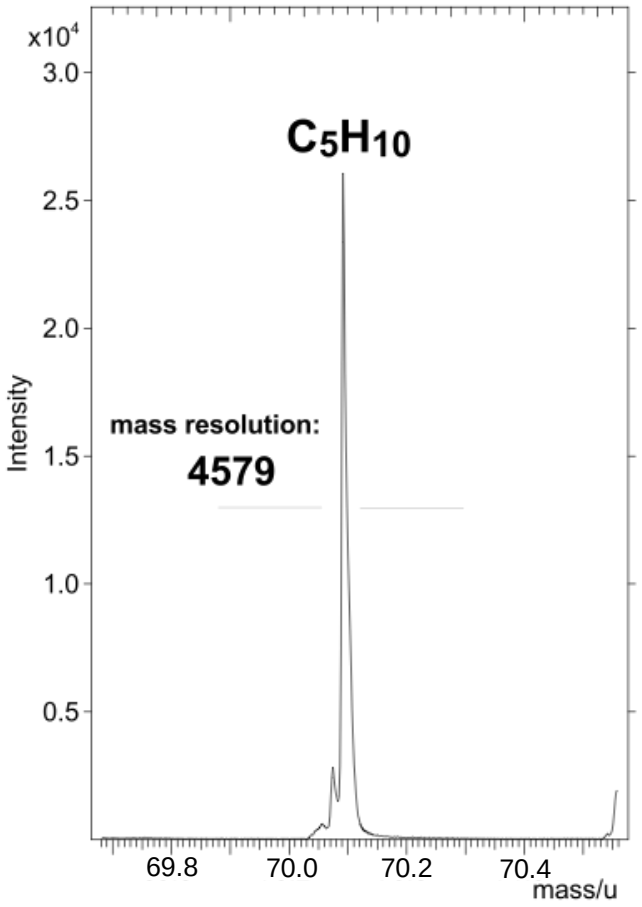
Static Extraction

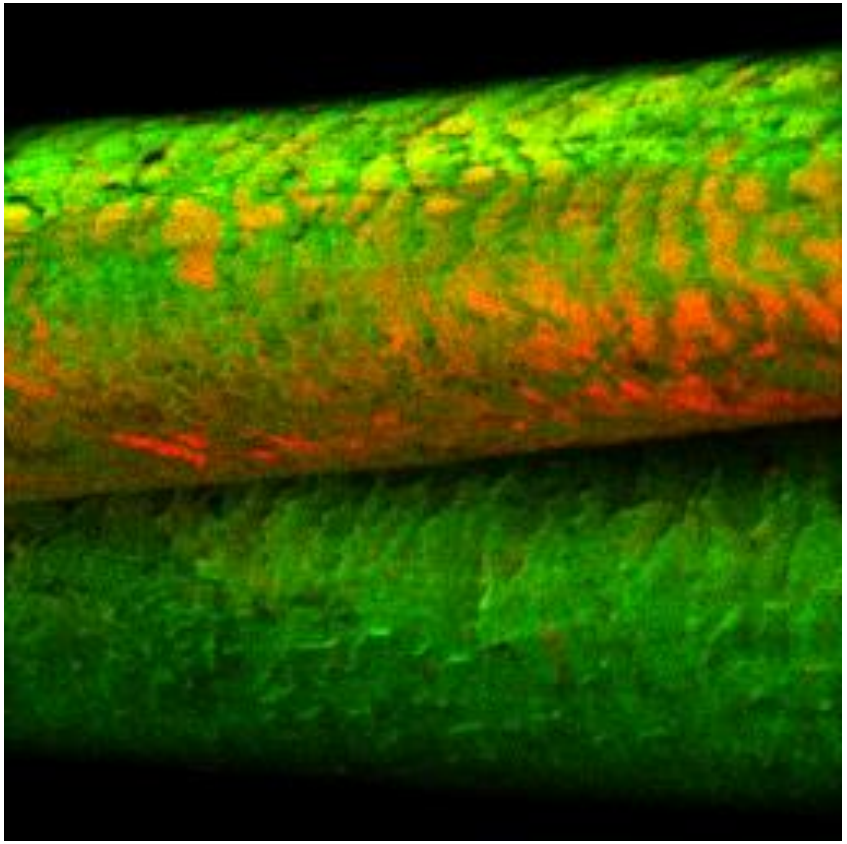


Delayed Extraction



Hair Sample


x10⁴
3.0
2.5
2.0
1.5
1.0
0.5
Intensity
C5H10
mass resolution:
4579
69.8
70.0
70.2
70.4
mass/u



Human Hair Sample

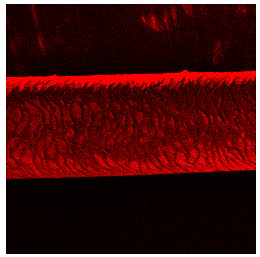
INNOVATIVE SURFACE ANALYSIS

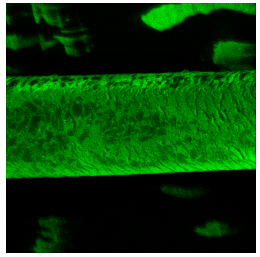
238 x 238 µm²

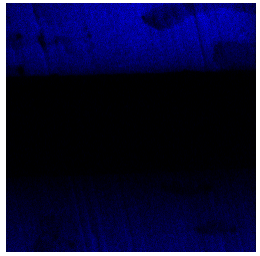
polysiloxans

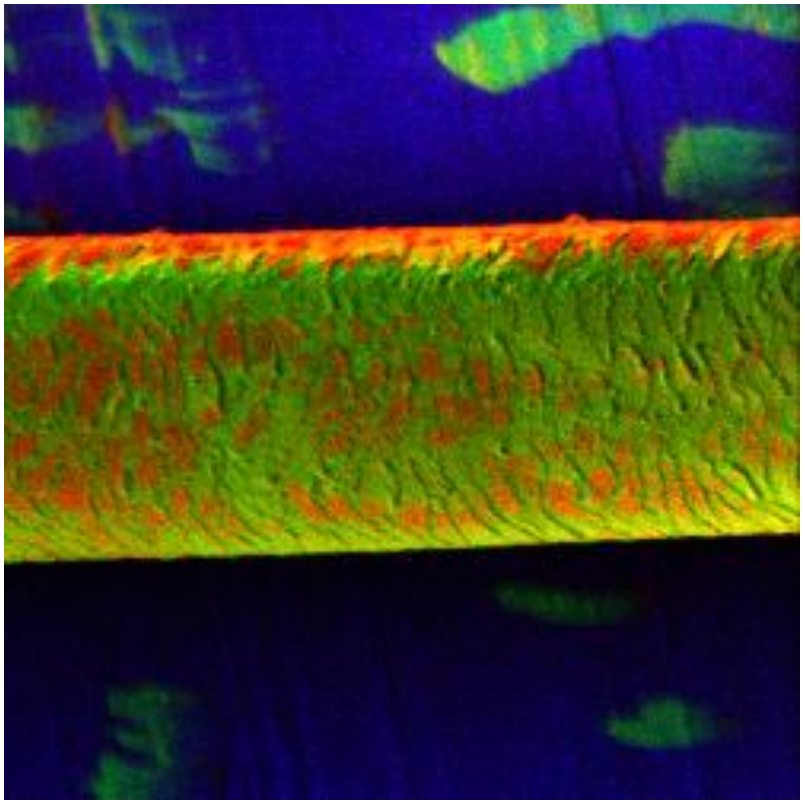
K+

$Al_xO_yH_z^+$

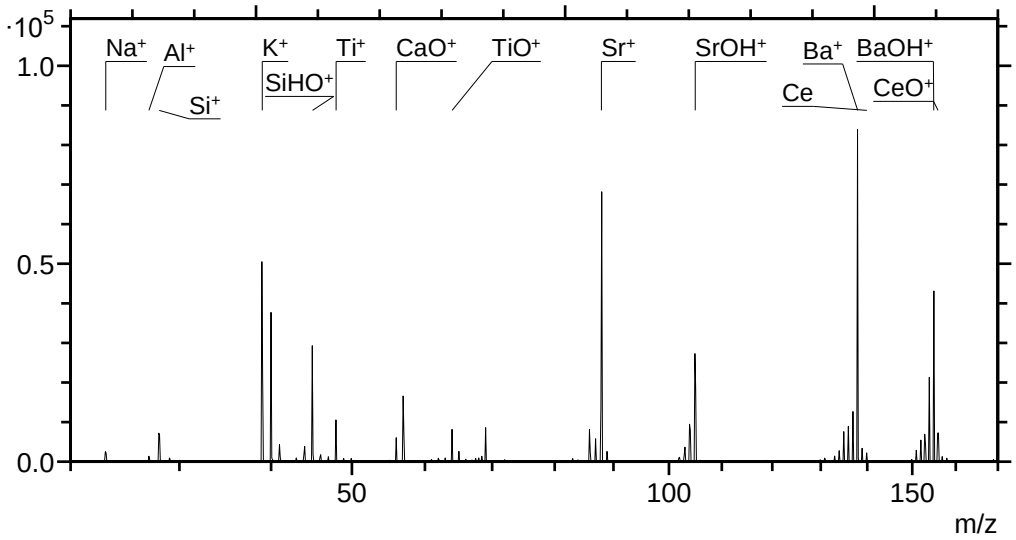


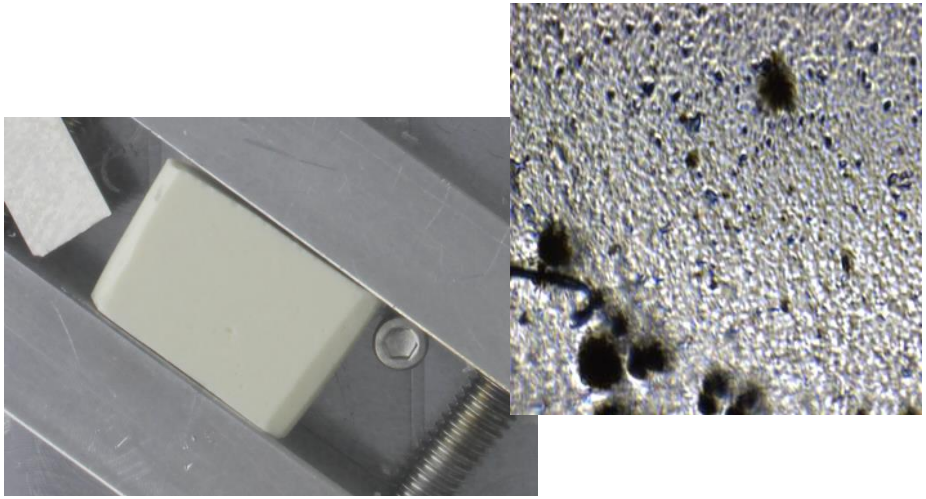


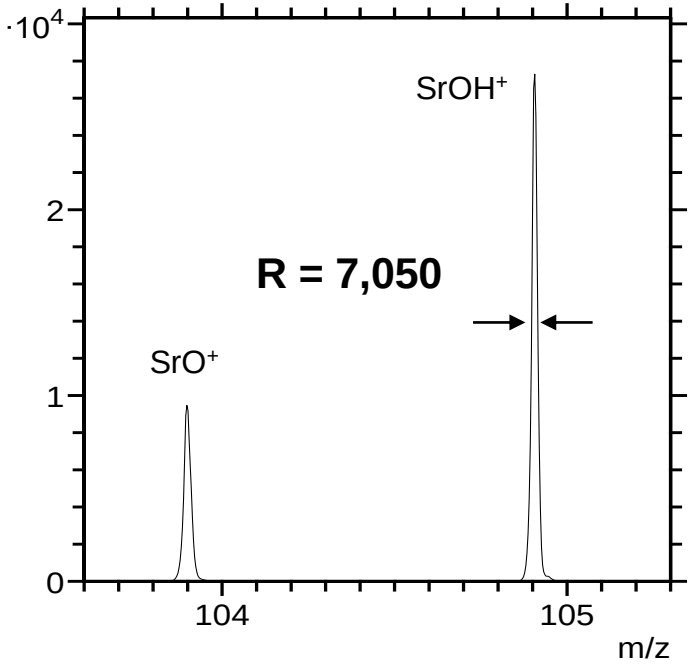




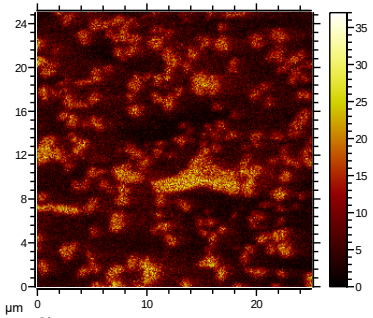
Glass Ceramic, FoV 25 x 25 µm²



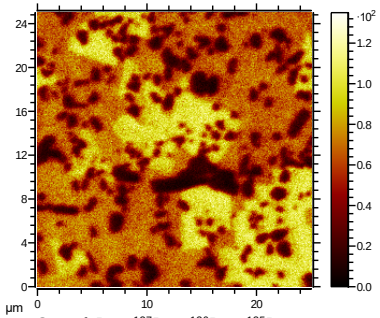




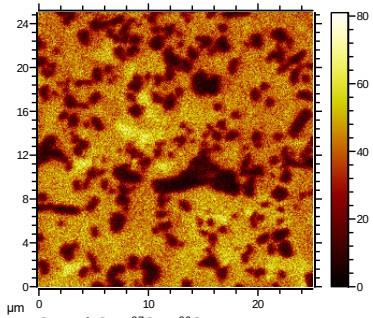
Glass Ceramic, FoV 25 x 25 µm²



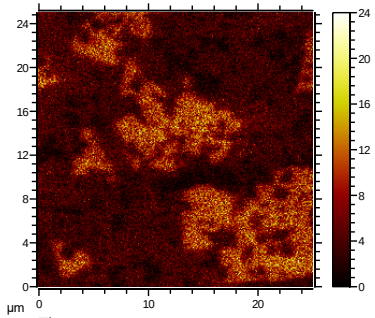
Si+
MC: 37; TC: 4.859e+005



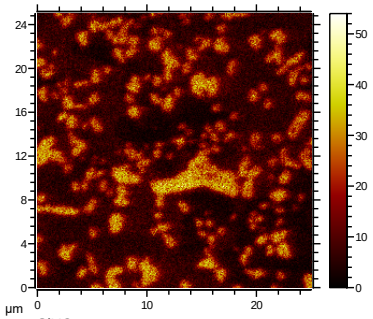
Sum of: Ba+, ^{137}Ba+, ^{136}Ba+, ^{135}Ba+
MC: 135; TC: 4.155e+006



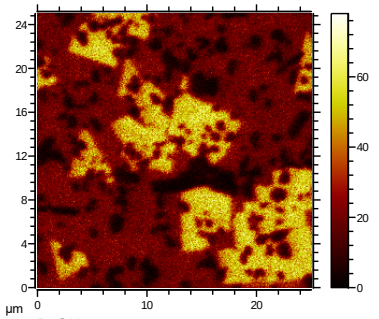
Sum of: Sr+, ^{87}Sr+, ^{86}Sr+
MC: 81; TC: 2.380e+006



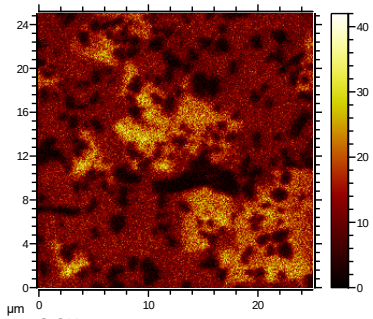
Ti+
MC: 24; TC: 3.201e+005



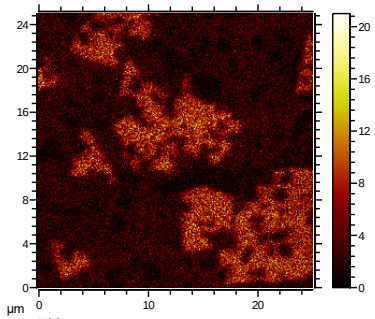
SiHO+
MC: 54; TC: 7.446e+005



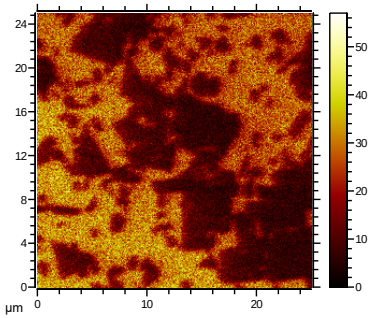
BaOH+
MC: 78; TC: 1.569e+006



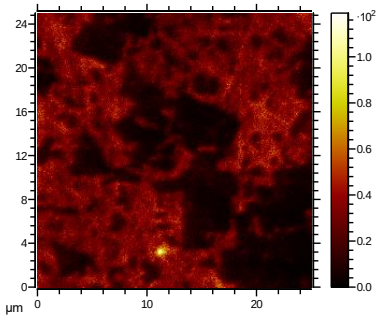
SrOH+
MC: 42; TC: 7.422e+005



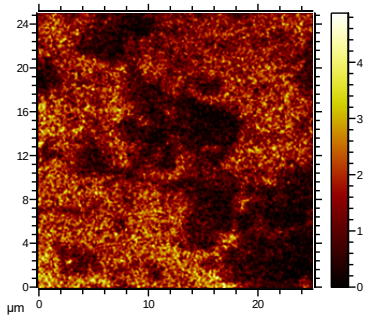
TiO+
MC: 21; TC: 2.266e+005



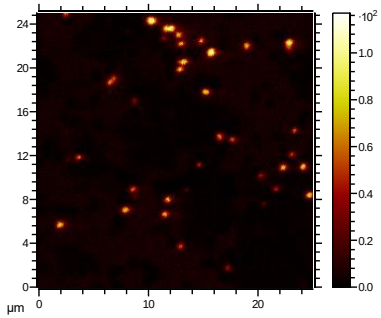
Ca+
MC: 57; TC: 1.293e+006



K+
MC: 119; TC: 1.399e+006



Al+ (filtered)
MC: 5; TC: 8.749e+004



CeO+
MC: 117; TC: 3.284e+005

Glass Ceramic, FoV 25 x 25 µm²

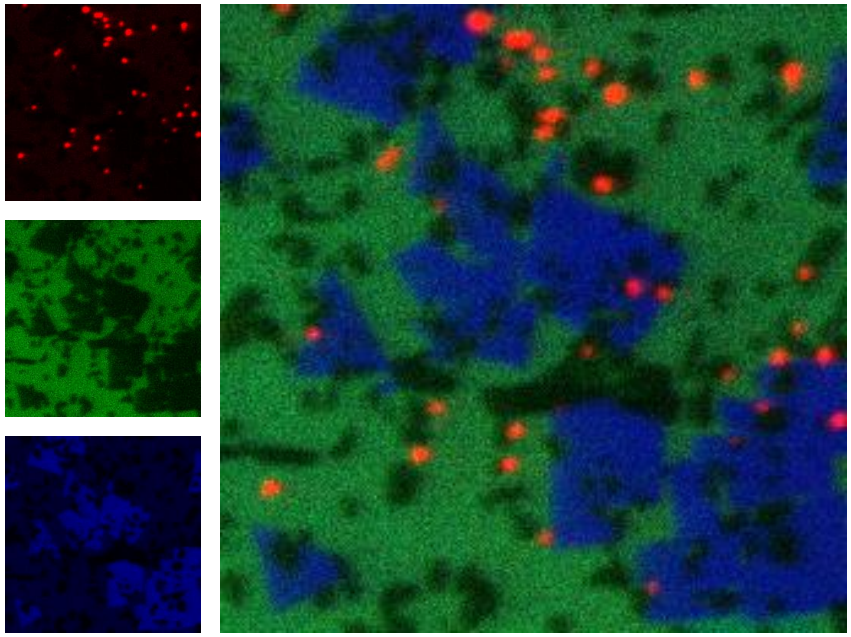
INNOVATIVE SURFACE ANALYSIS

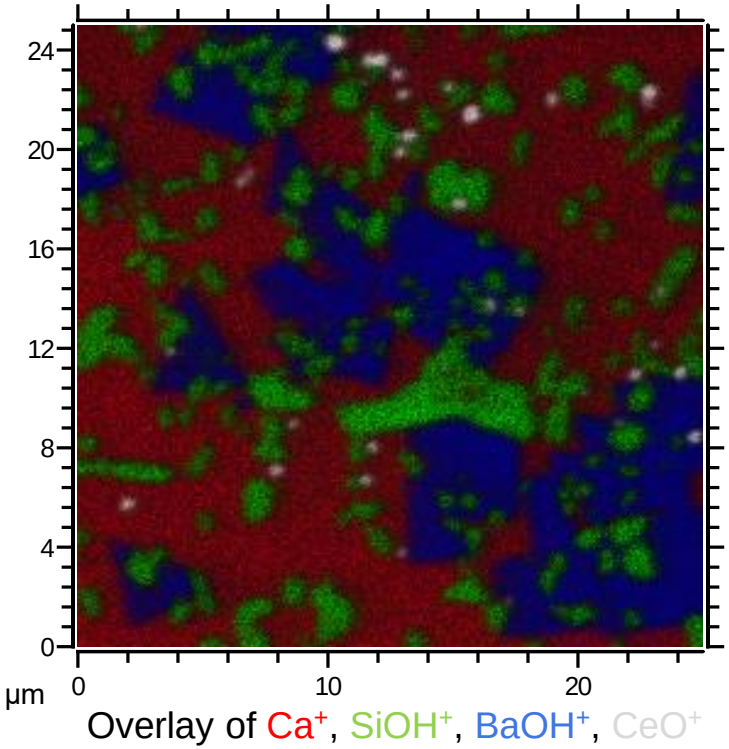
25 x 25 µm²

CeO+

Ca+

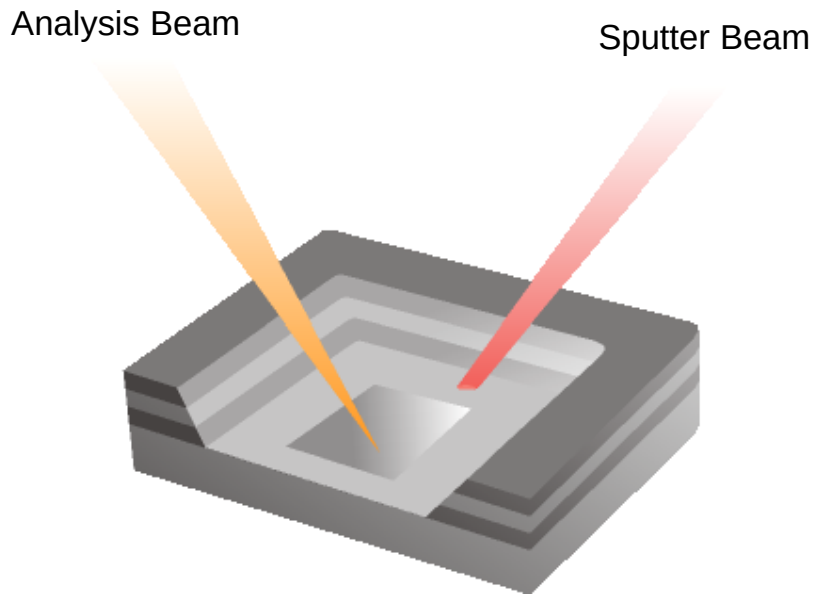
BaOH+





Dual Beam Depth Profiling

- Sputter beam and analysis beam conditions are optimised independently
- Analysis is performed by a short pulse length and small spot size ion beam for high mass and lateral resolution
- Sputtering is achieved by a beam of reactive species(O_2 or Cs) or clusters (Ar_{1500}) at low energy for increased sensitivity, high depth resolution, and short transients

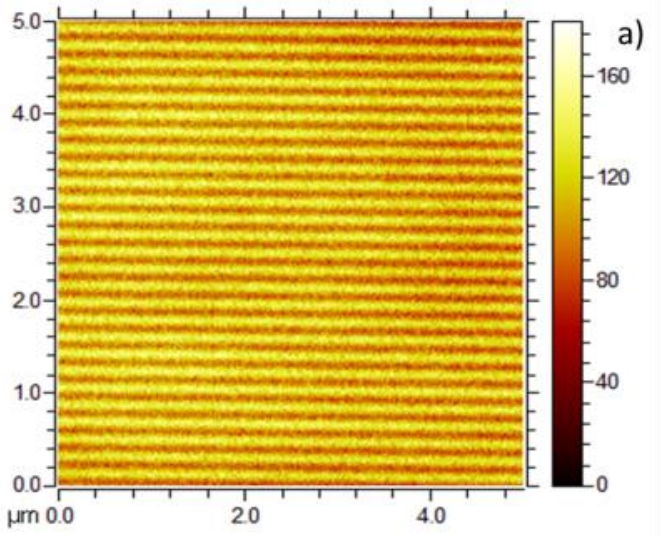


Micro Area Profiling of III-V Trenches

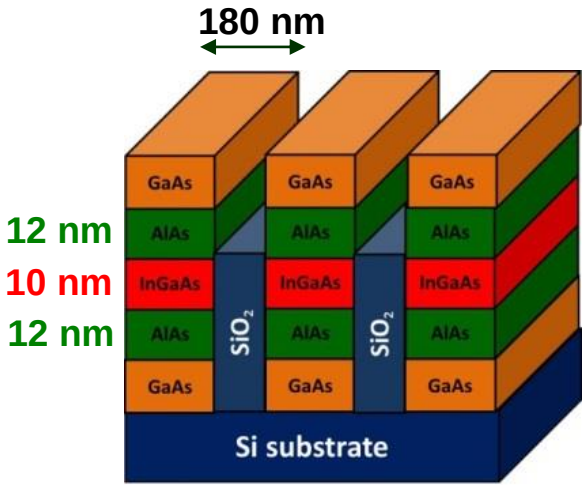
3D analysis of patterned InGaAs QWs

Example provided by CEA LETI: V. Gorbenko et al., presented at 9th SIMS Europe 2014

Analysis: Bi_3^{++} 60 keV energy, 5 x 5 µm²
Sputtering: O_2^+ at 500eV, 200 x 200 µm².



$^{28}Si^+$ ion image (top view)

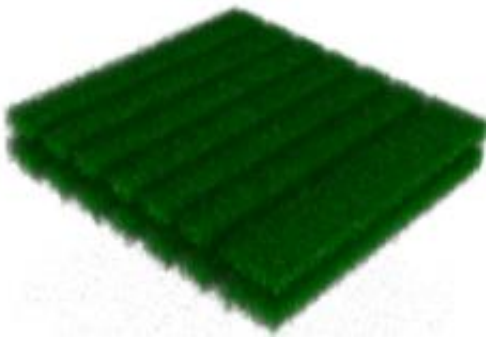


Micro Area Profiling of III-V Trenches

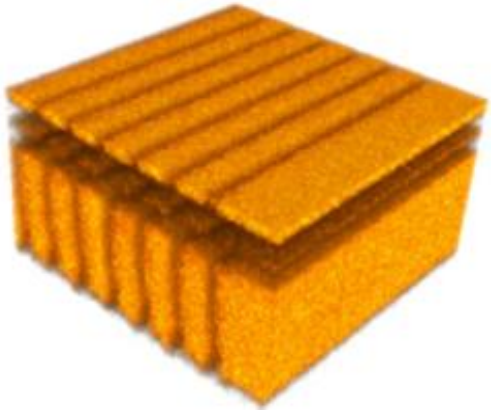
INNOVATIVE SURFACE ANALYSIS

10 µm x10 µm x 200 nm

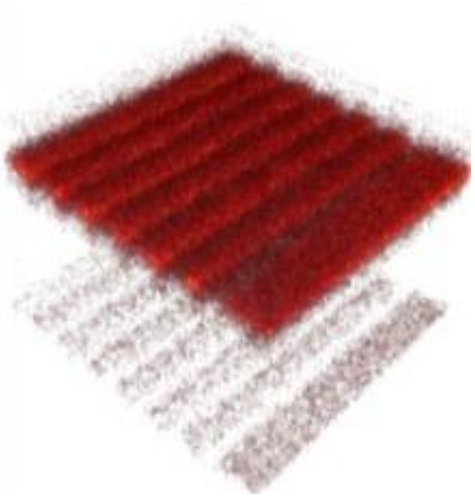
Al^+



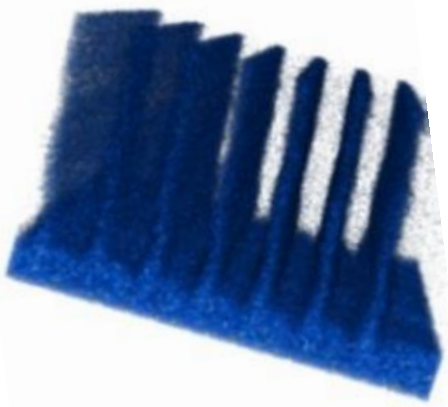
Ga^+



In^+

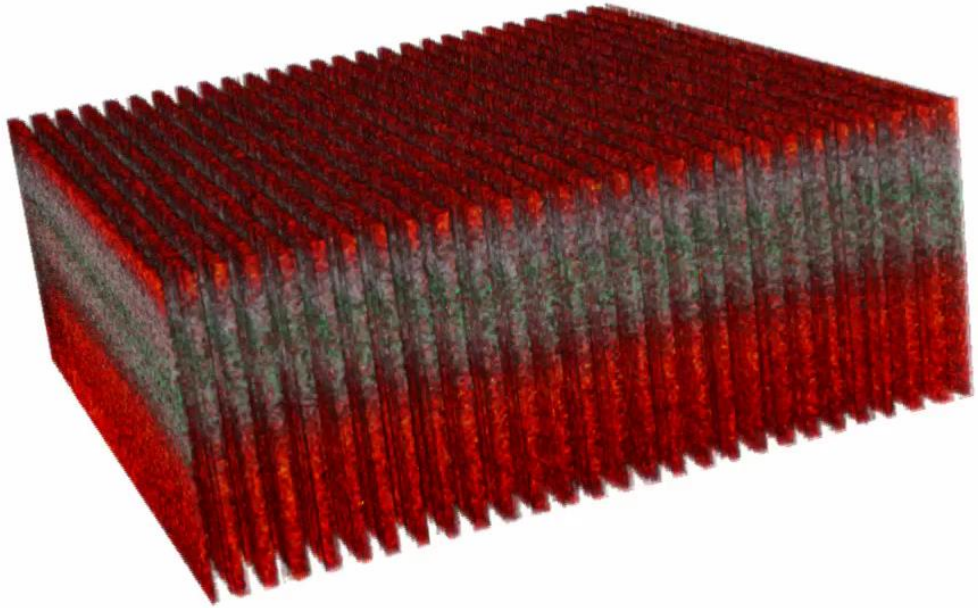


Si^+



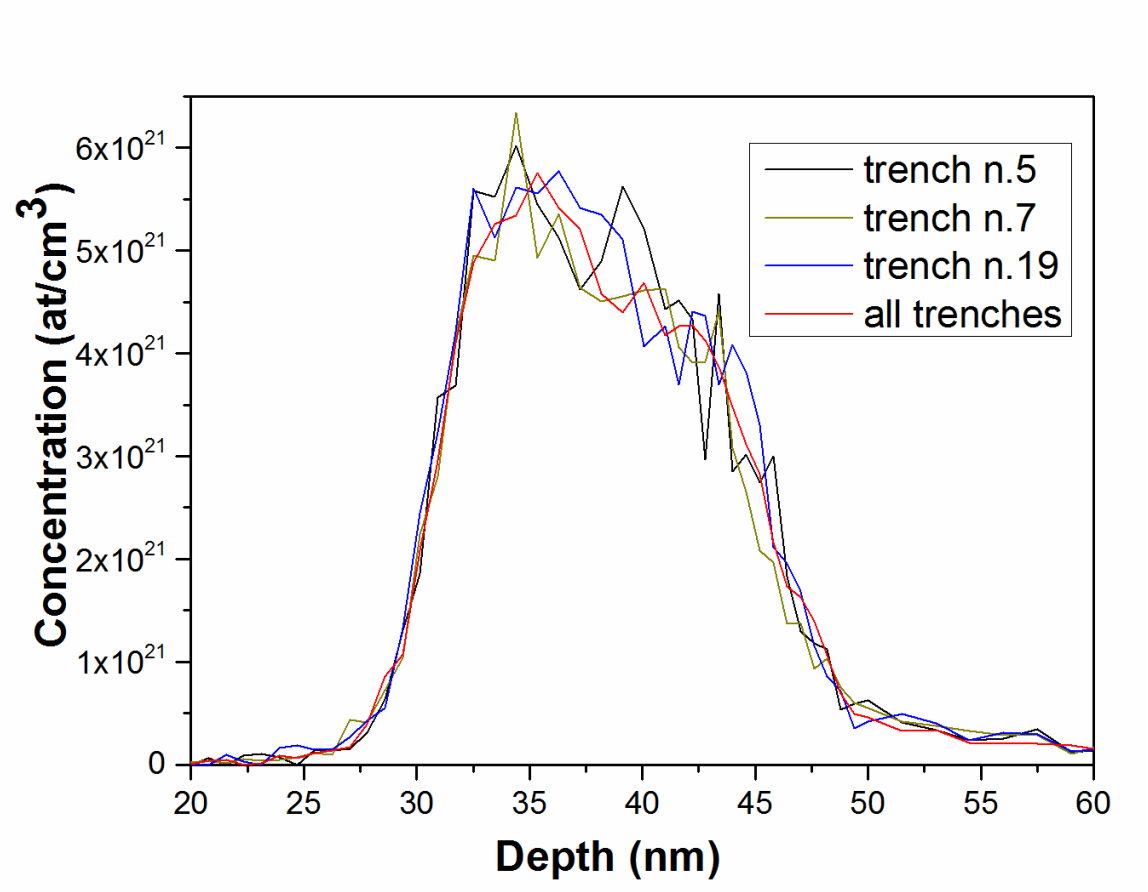
128 x 128 pixels (78,1 nm/pixel)

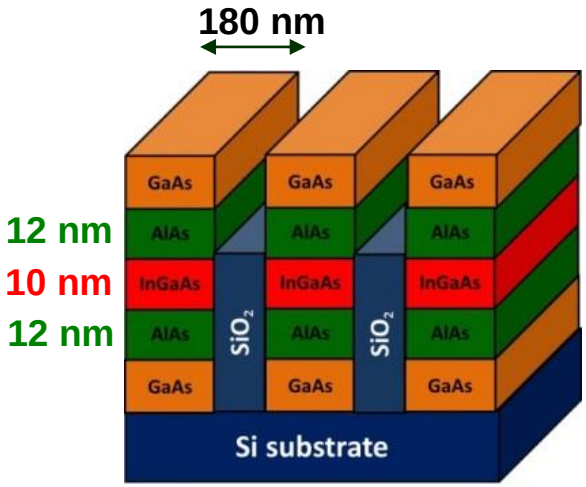
Micro Area Profiling of III-V Trenches



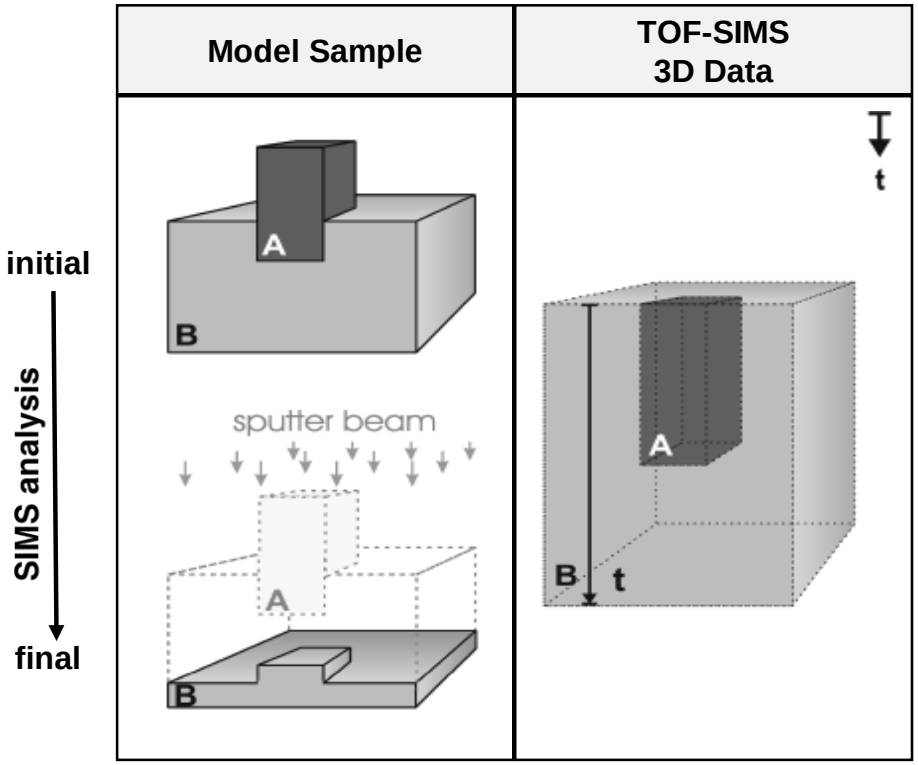
Micro Area Profiling of III-V Trenches

Quantification of individual QWs without contribution from SiO_2





Influence of Topography and Sputter Rates

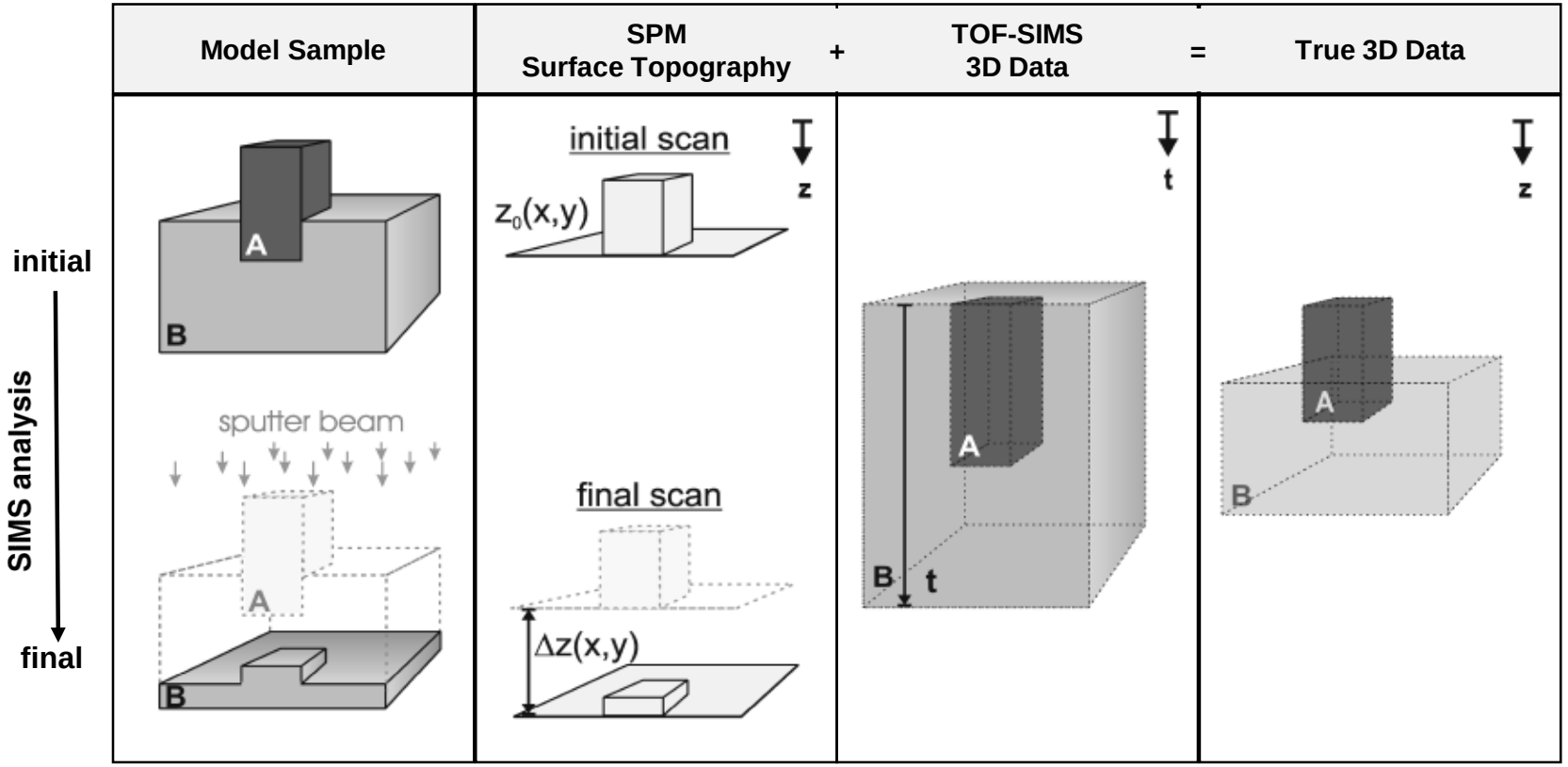


SIMS does not provide any information about ...

...the topography or...

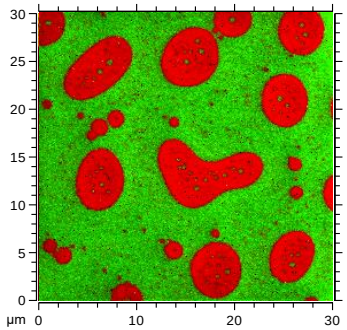
...the changes of the topography due to different sputter rates

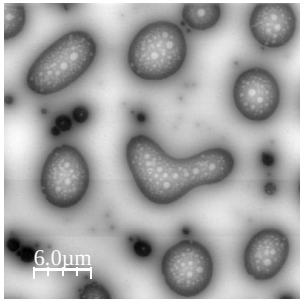
Concept of the Combined Instrument




Space for SPM..?

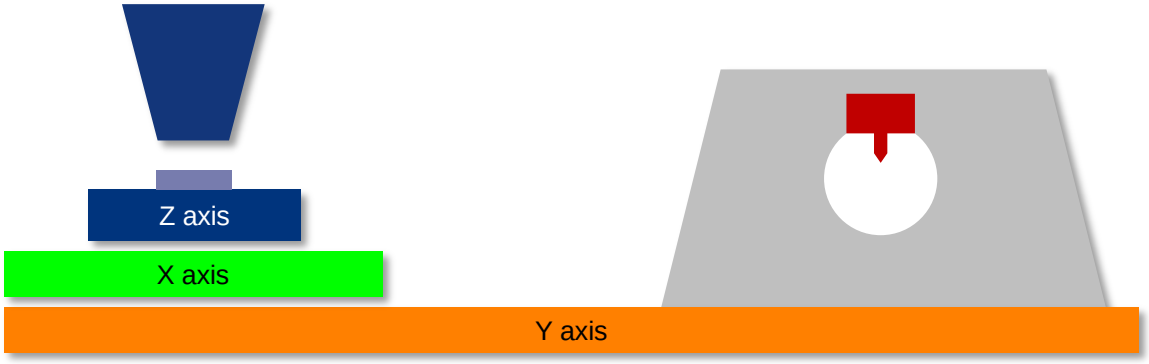
TOF-SIMS / SPM Setup





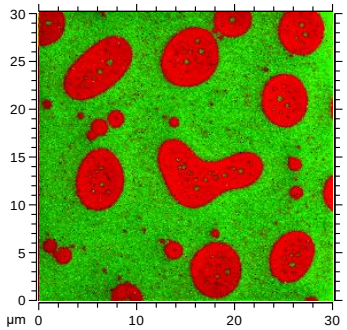
TOF-SIMS Module

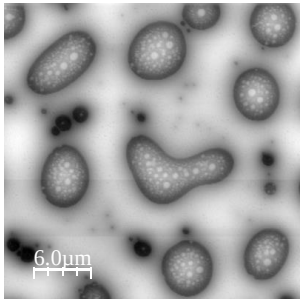
SPM Module



high precision sample transfer (accuracy < 1 µm)

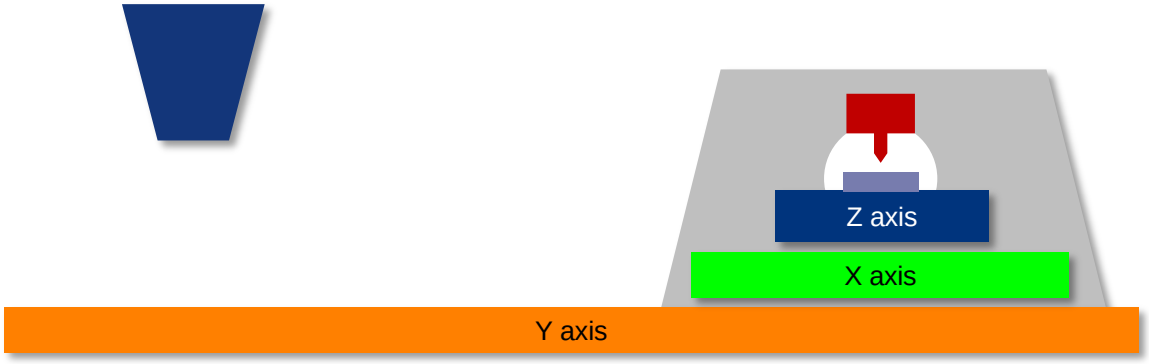
TOF-SIMS / SPM Setup





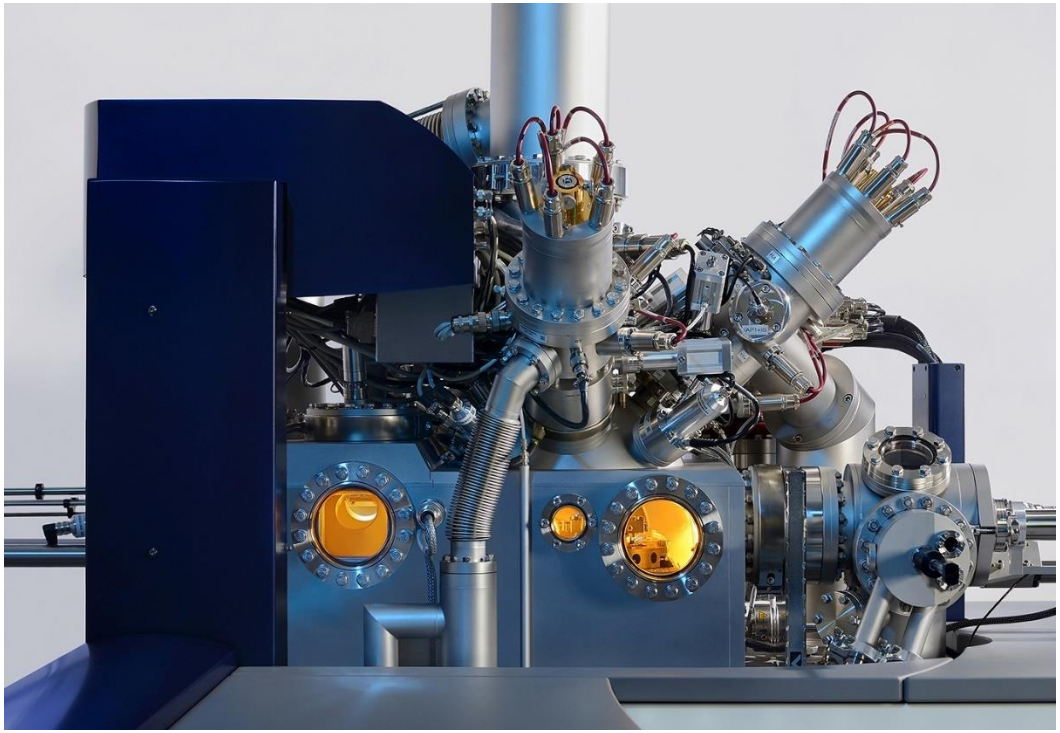
TOF-SIMS Module

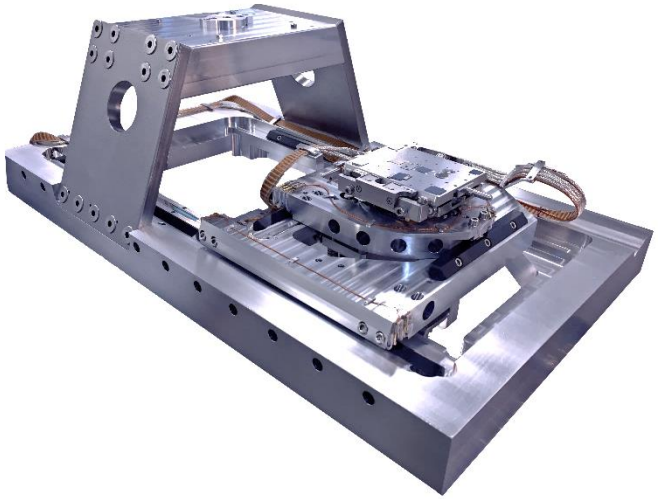
SPM Module



high precision sample transfer (accuracy < 1 µm)

Combined TOF.SIMS – SPM Instrument





NanoScan UHV SPM module

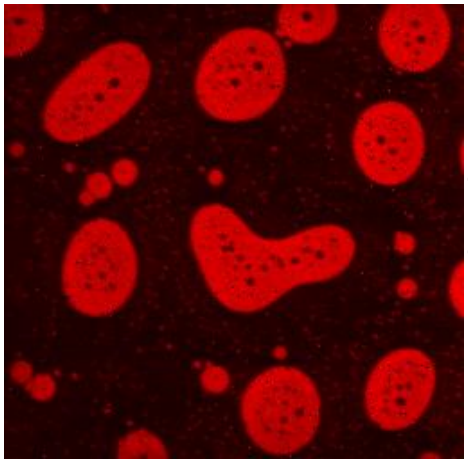
- Flexure stage scanner with 80 x 80 x 10 µm³
- 4-axes high precision piezo stage (XYZR)
- Various static and dynamic SPM modes
- Fast cantilever exchange (storage of 4)

High precision piezo stage (XYZR)

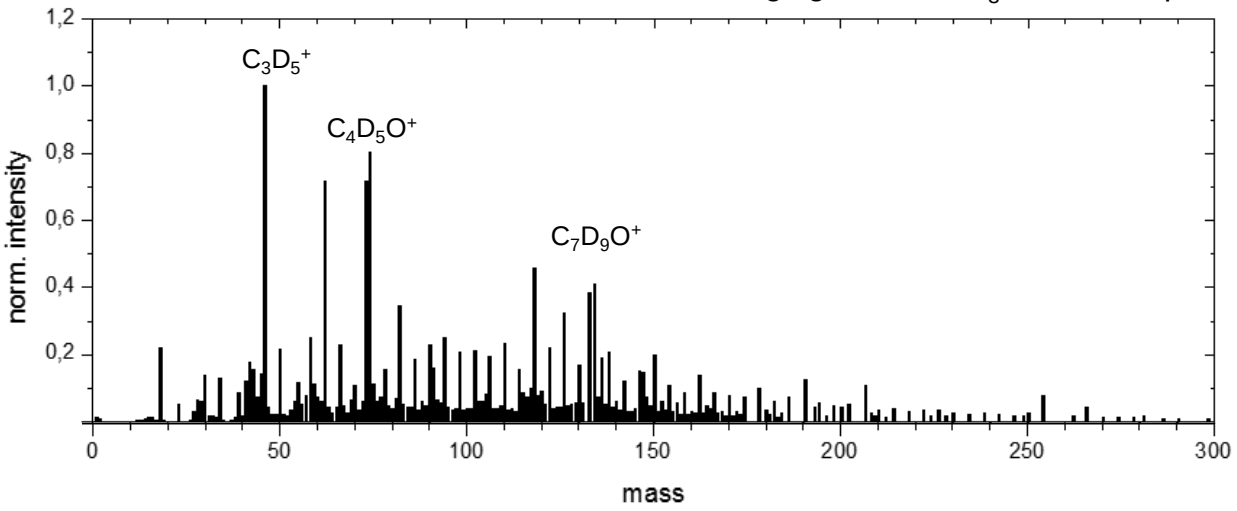
- speed: 10 mm/s
- encoder resolution: 10 nm
- positioning accuracy: < 1 µm

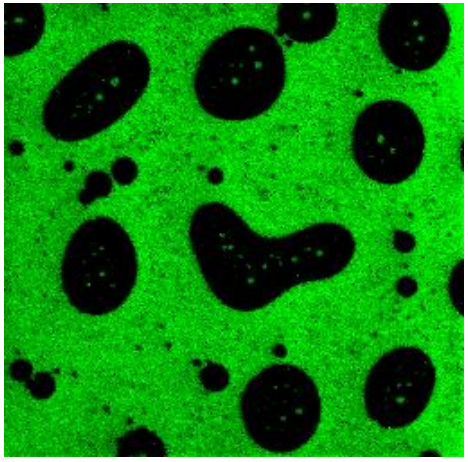
PMMA / PS Polymer Blend

Surface Imaging: 60 keV, Bi_3^{++}, FoV: 30 µm²

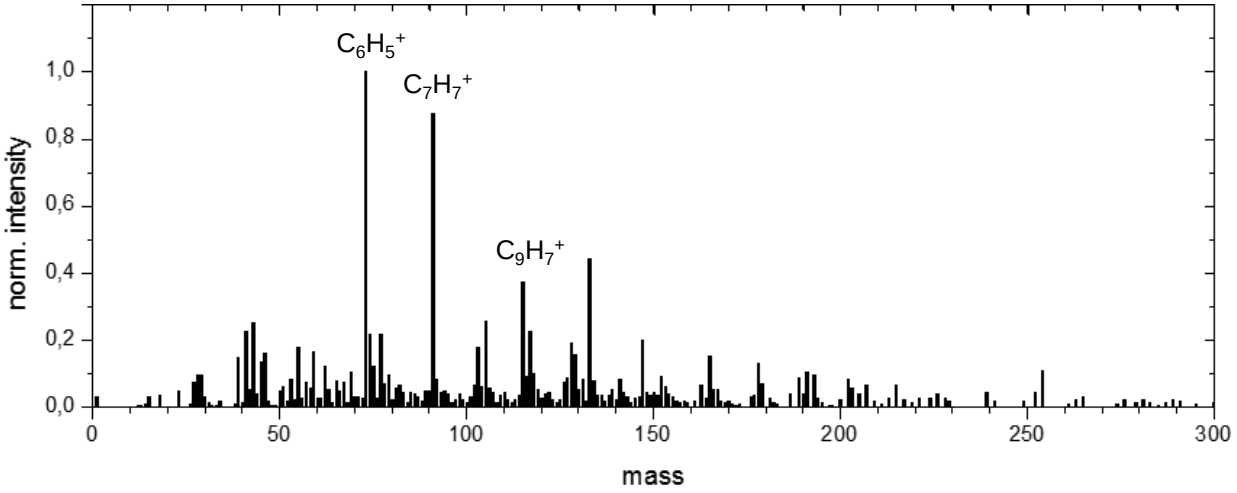


PMMA

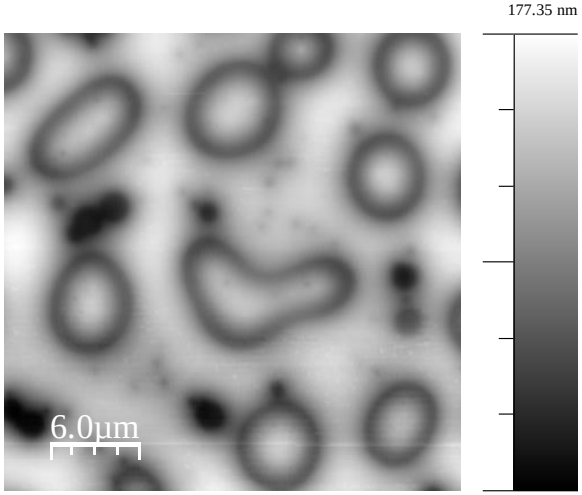




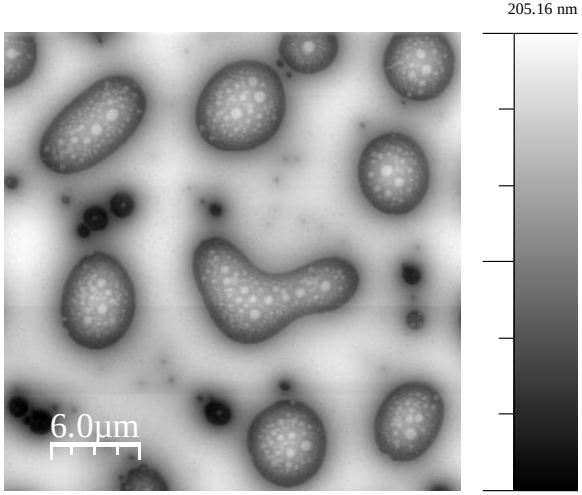
PS



PMMA / PS Polymer Blend

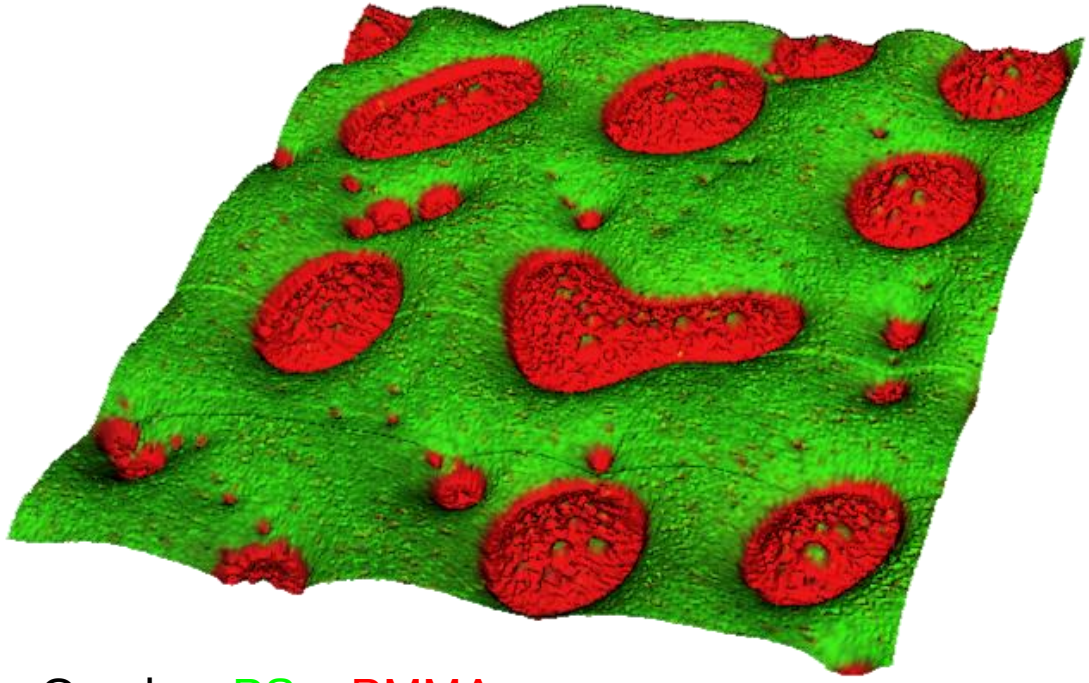


SPM: before TOF analysis



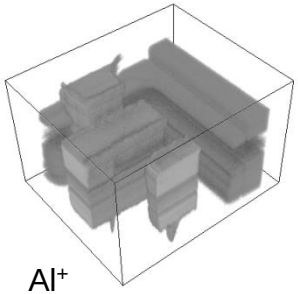
SPM: after TOF analysis

Combined TOF-SIMS / SFM 2D analysis: Topography and chemical information

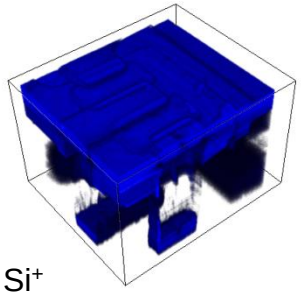


Overlay: PS + PMMA

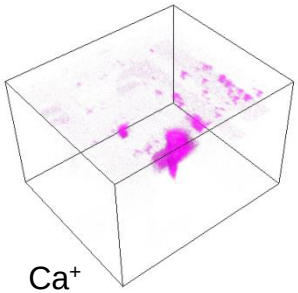
3D Overlay - Volume Plot



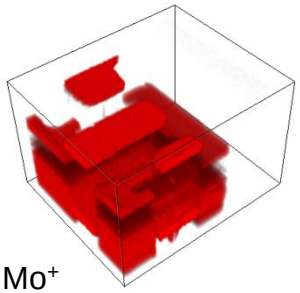
Al^+



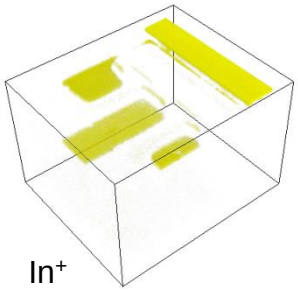
Si^+



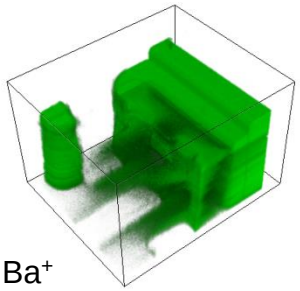
Ca^+



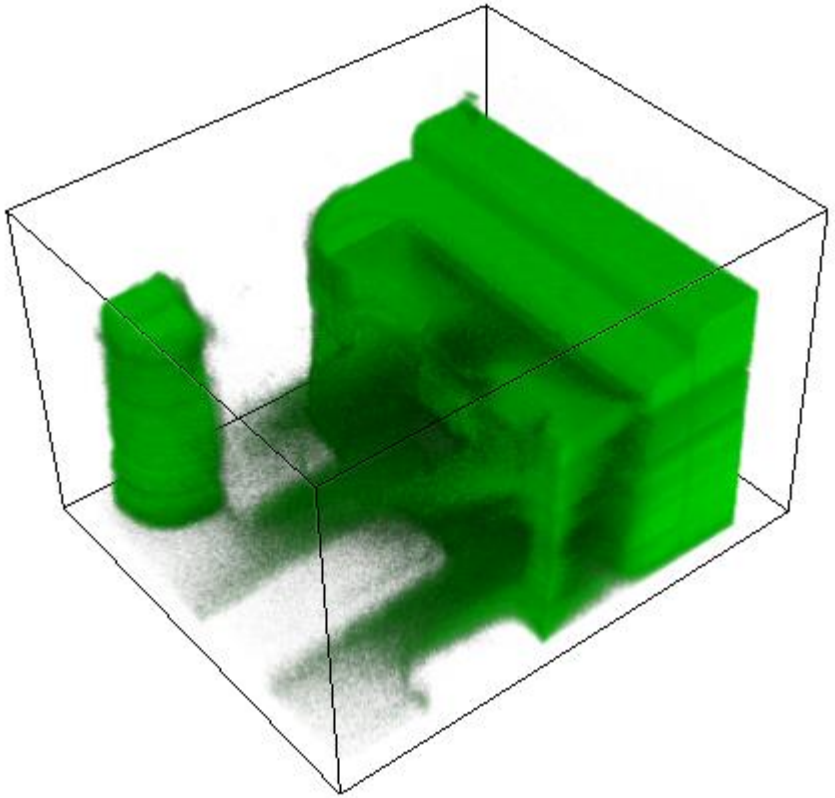
Mo^+



In^+

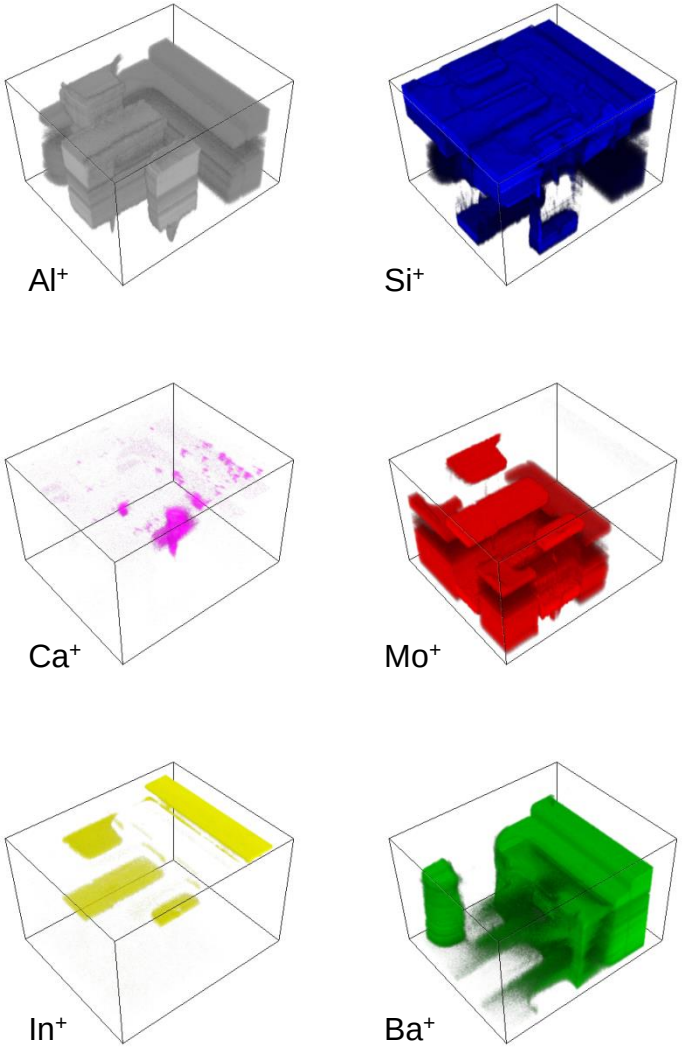


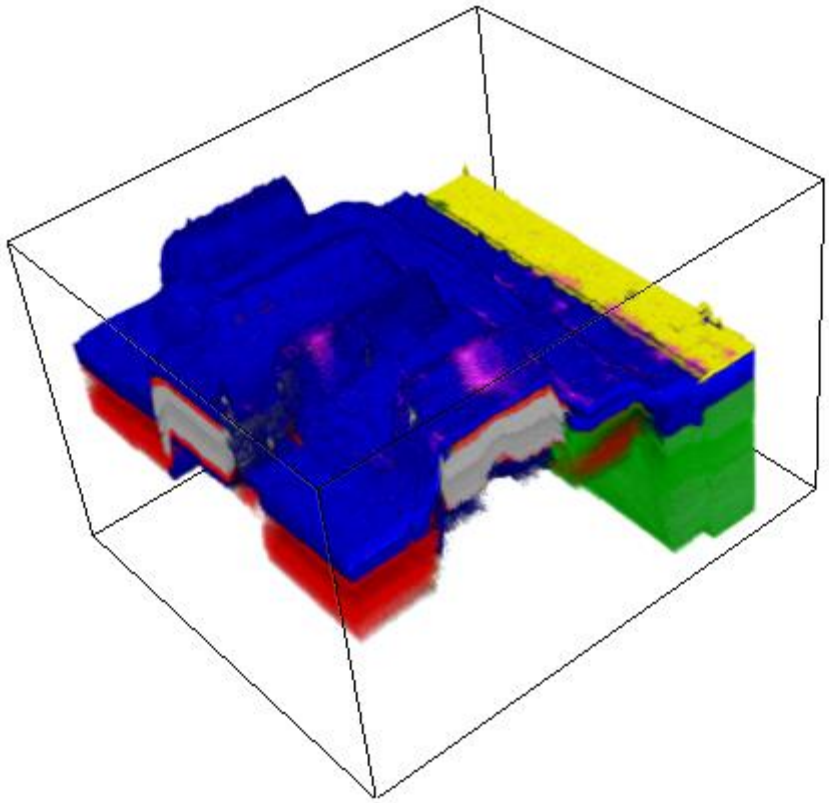
Ba^+



TOF-SIMS data only

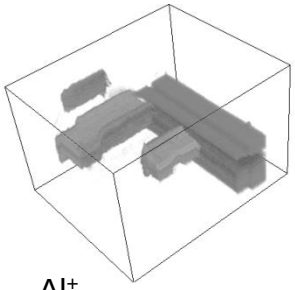
3D Volume Plot vs. 3D Image



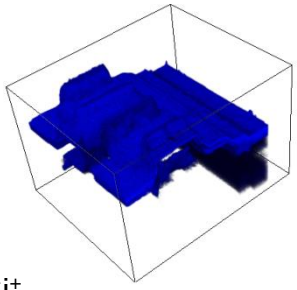


Combination of TOF-SIMS and SPM data

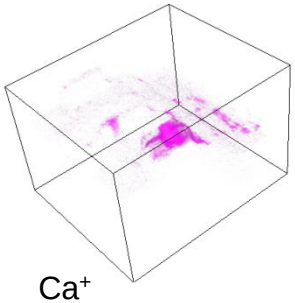
Cross Section through 3D Image



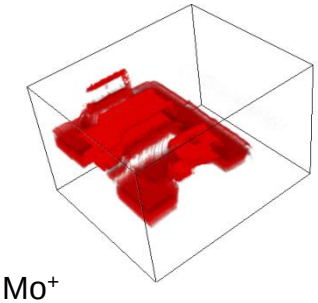
Al^+



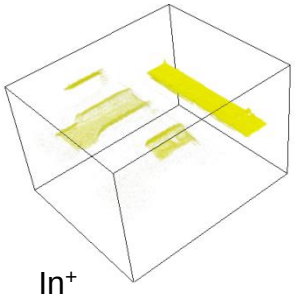
Si^+



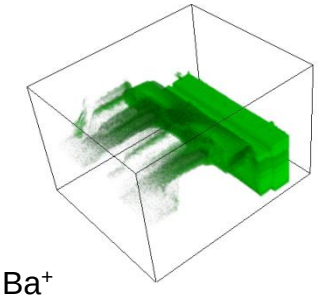
Ca^+



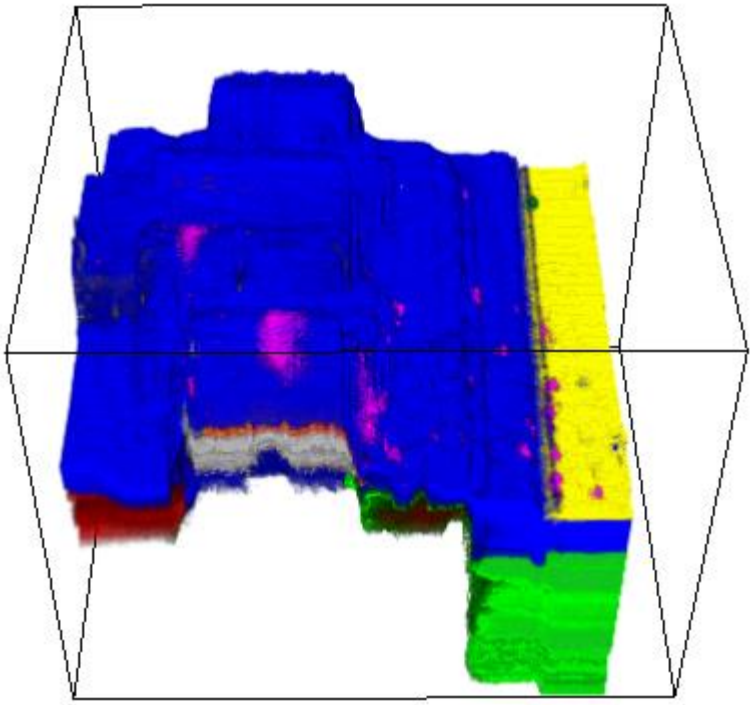
Mo^+



In^+



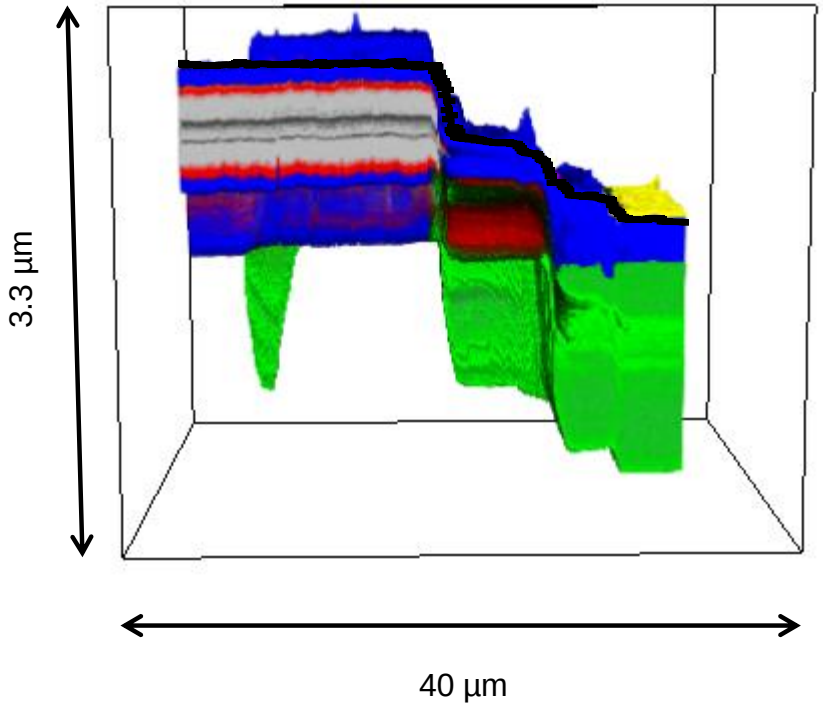
Ba^+



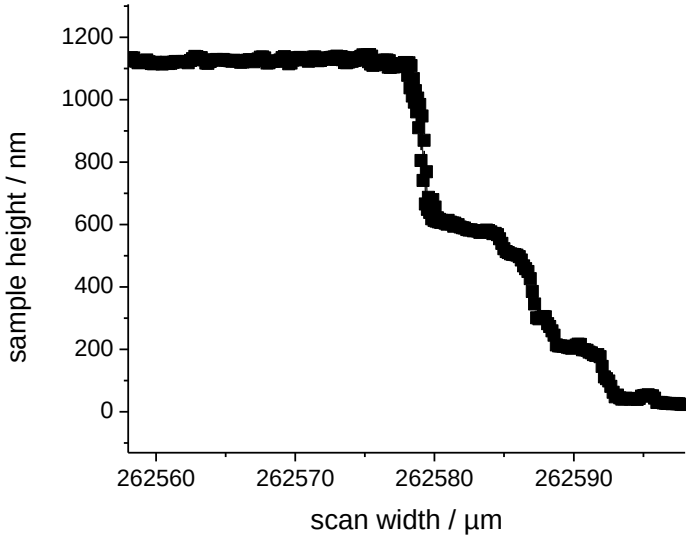
Combination of TOF-SIMS + SPM data

Topography: Data Evaluation

Cross section:

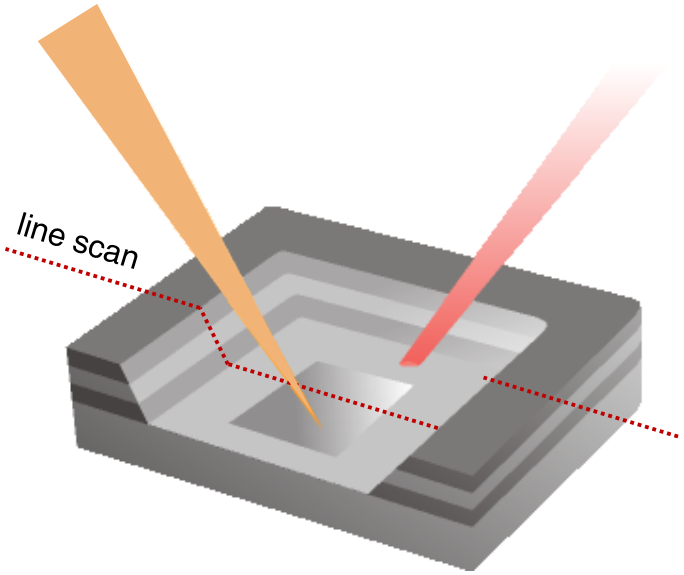


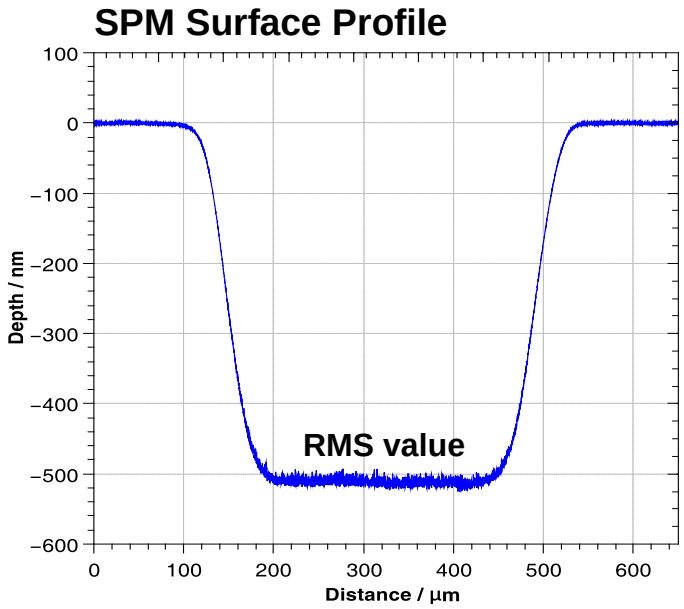
Height profile:



Combination of TOF-SIMS + SPM data

Depth Scale Calibration in SIMS

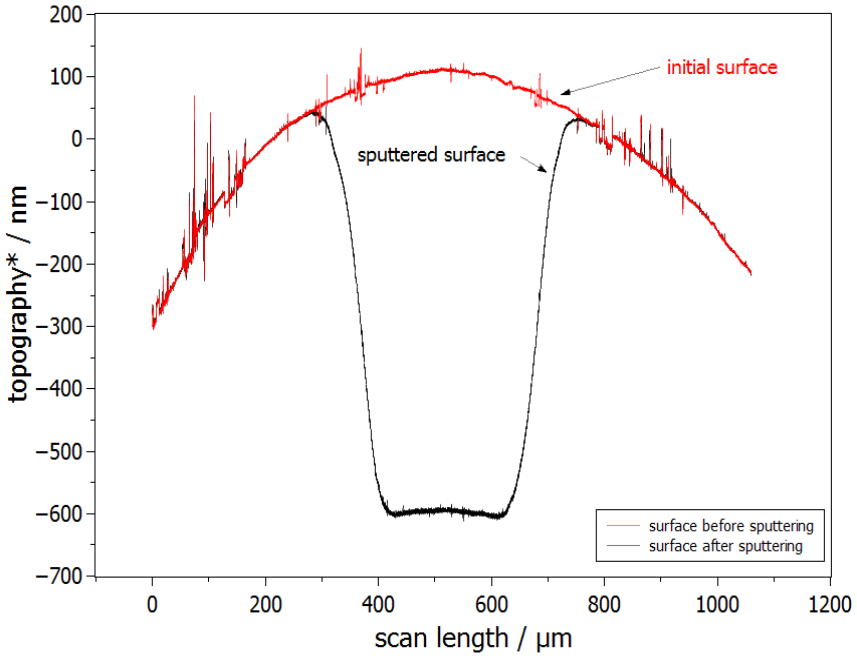




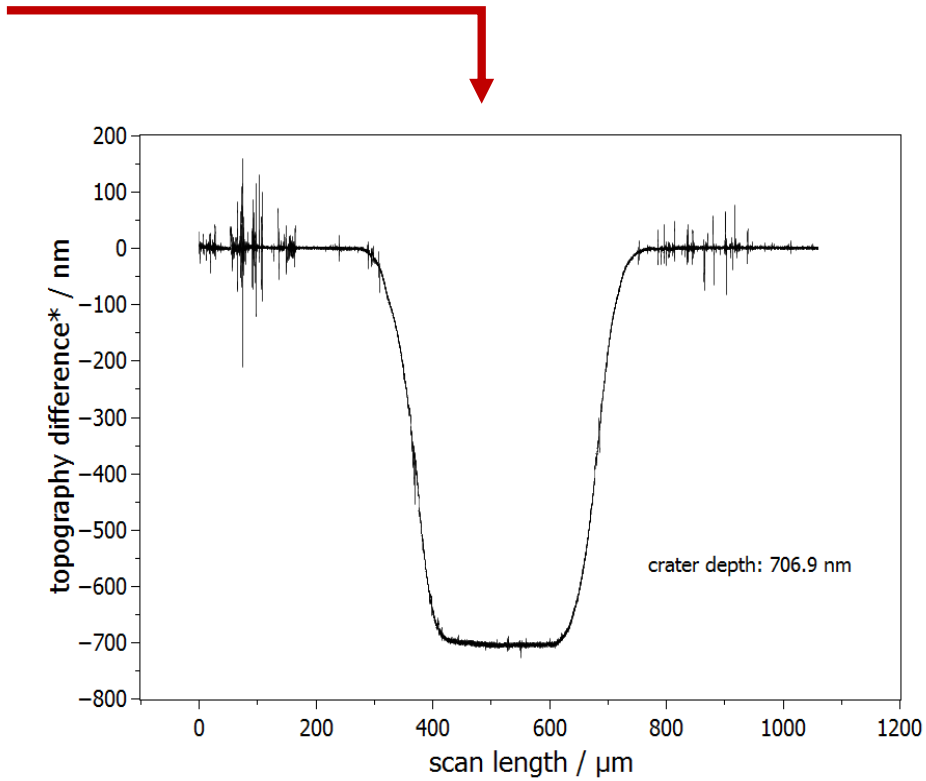
SPM Profiler Mode

- Depth profiles and 3D data sets need depth calibration (t → z)
- Crater depth needs to be measured relative to the initial surface
- Typical SIMS crater dimensions: 200 - 500 µm
- Limited SPM scan range: 80 µm

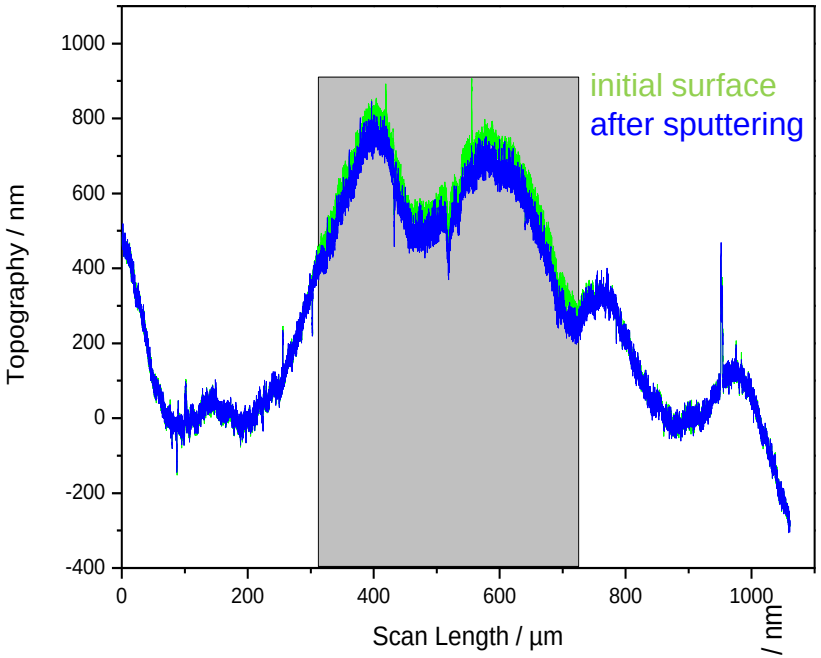
Curved Glass Surface with Polymer Coating

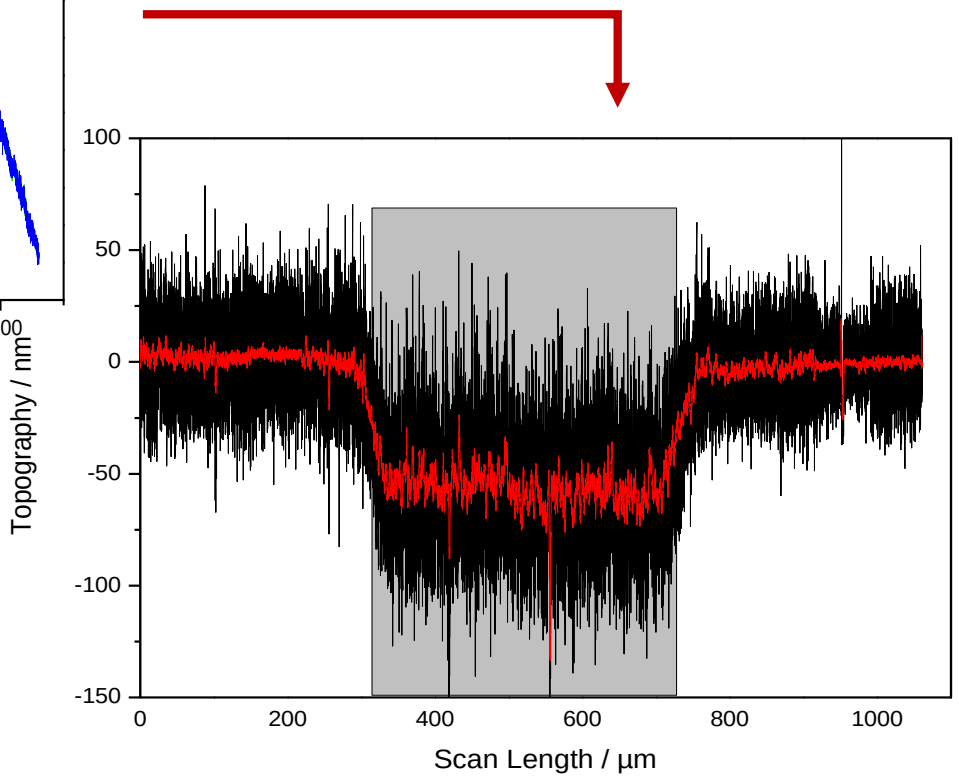


* arbitrary levelling



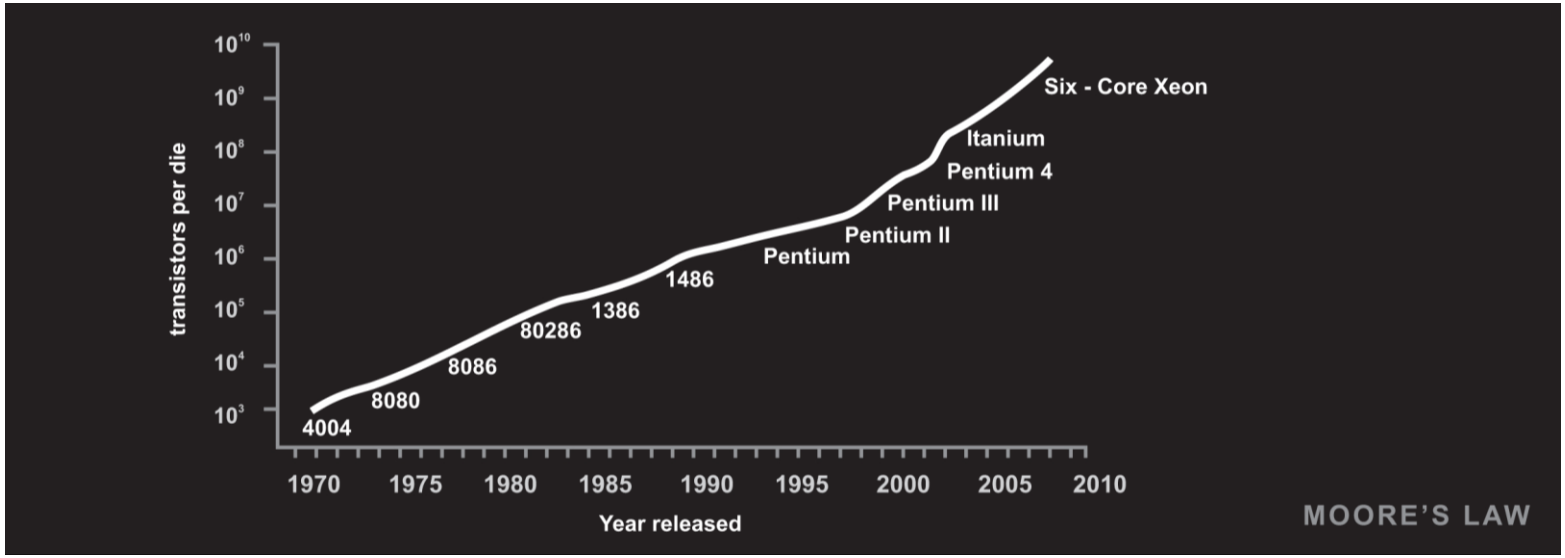
Crater on Glossy Photopaper



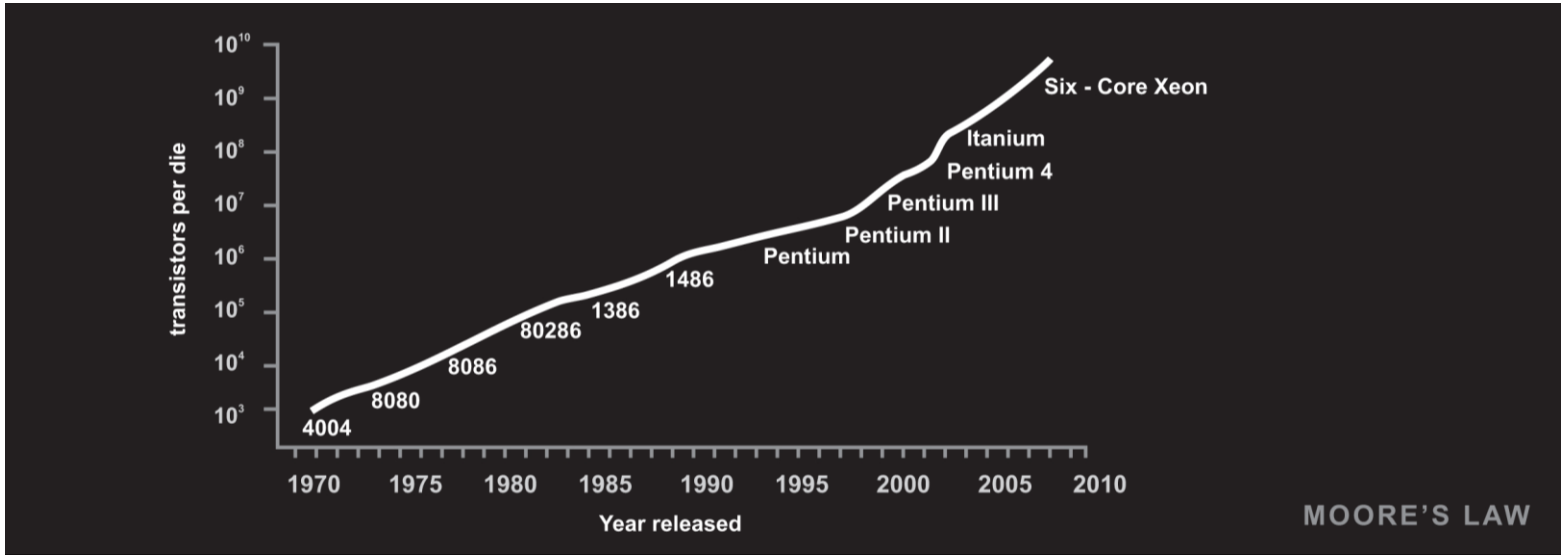


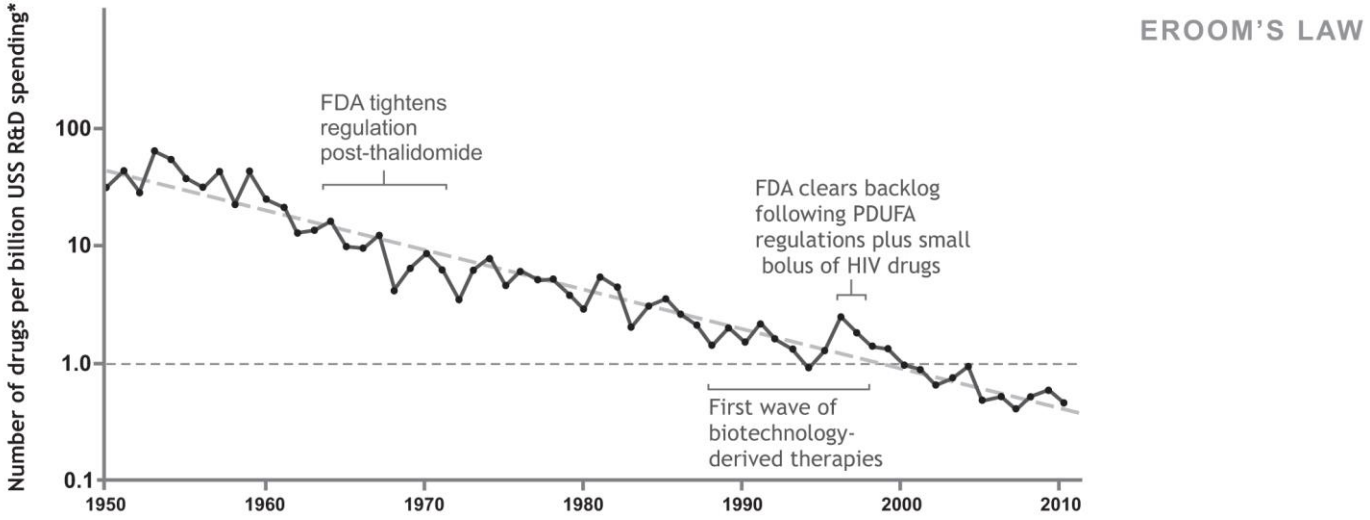
MOORE'S and EROOM'S LAW

INNOVATIVE SURFACE ANALYSIS

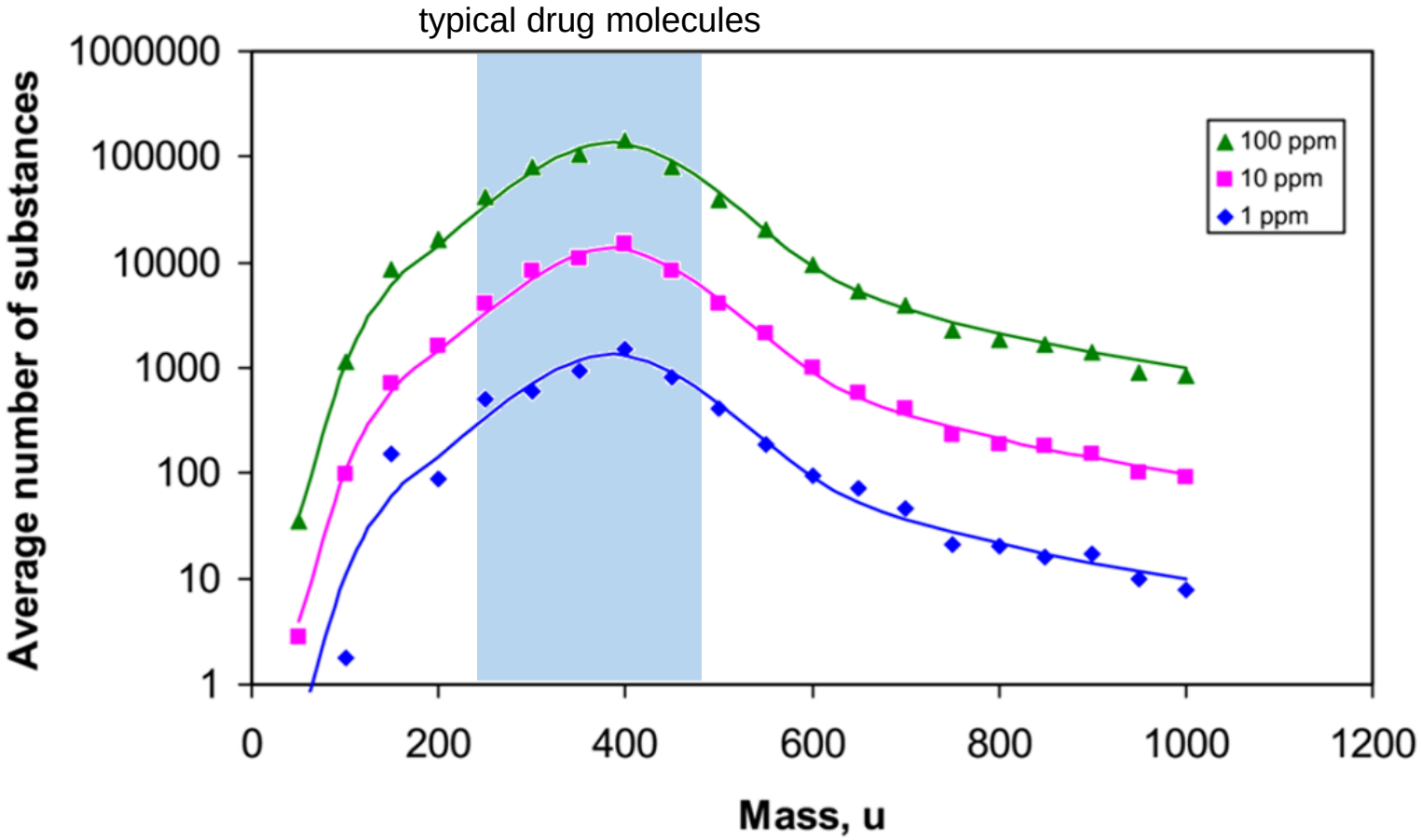


MOORE'S LAW and EROOM'S LAW





Average Number of Molecules in PubChem DB



Ian S. Gilmore: “SIMS of organics—Advances in 2D and 3D imaging and future outlook”, Journal of Vacuum Science & Technology A **31**, 050819 (2013);

Average Number of Molecules in PubChem DB

Requirement for...

high mass accuracy
high mass resolution
isotopic pattern matching

Ian S. Gilmore: “SIMS of organics—Advances in 2D and 3D imaging and future outlook”, Journal of Vacuum Science & Technology A **31**, 050819 (2013);

Combined TOF.SIMS 5 / Q Exactive ™ Instrument

INNOVATIVE SURFACE ANALYSIS

Hybrid SIMS Instrument

- Dual analyser configuration with ToF and Orbitrap ™
- Pulsed and DC mode operation
- Single and dual beam analysis modes

Thermo Scientific ™ Q Exactive ™ HF

- Mass resolution 240,000 @ m/z 200
- Scan rate up to 18 Hz
- Mass accuracy < 1 ppm
- MS/MS with precise precursor selection, full mass resolution and mass accuracy

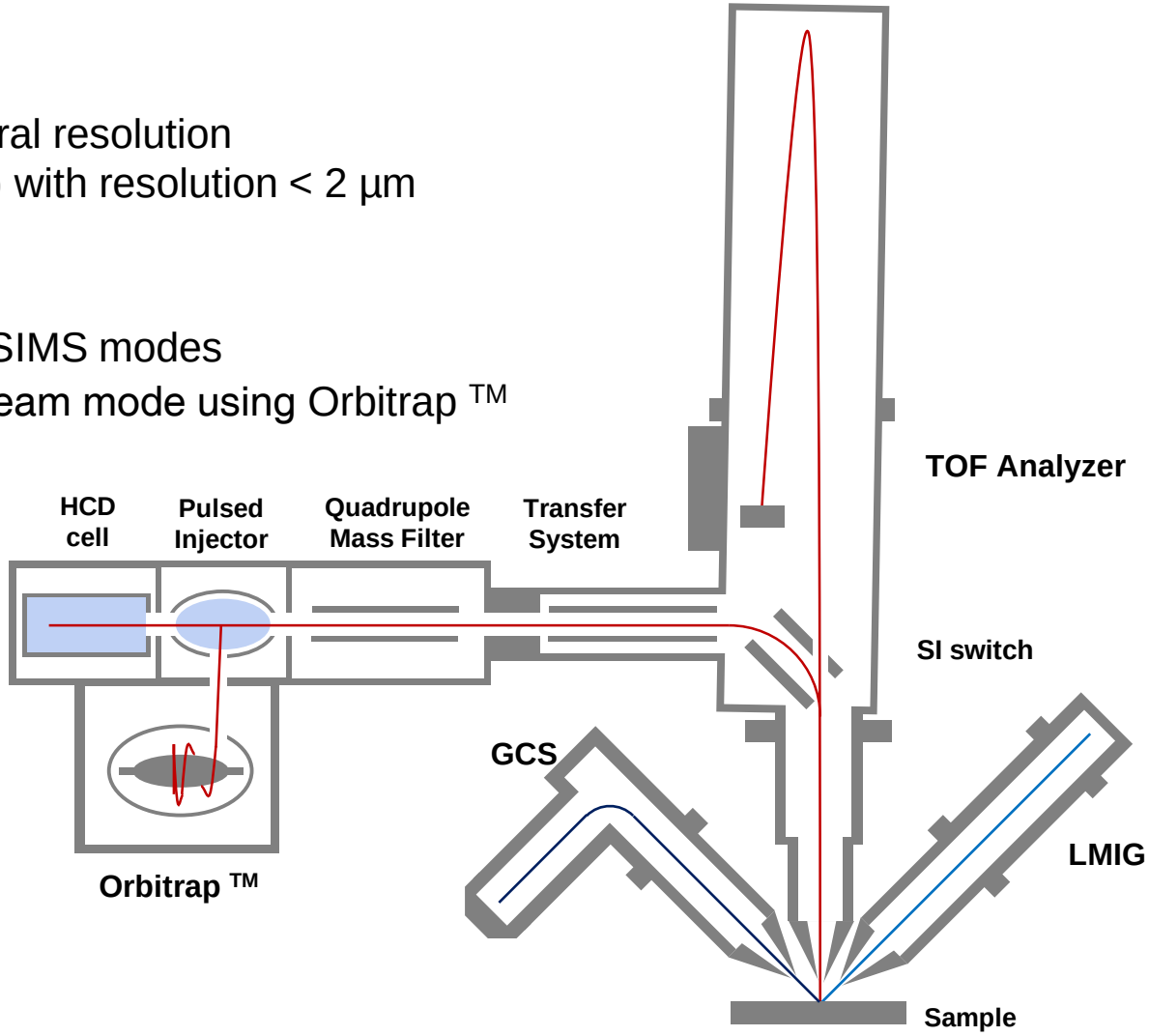
Combined TOF.SIMS 5 / Q Exactive ™ Instrument

Hybrid SIMS Instrument

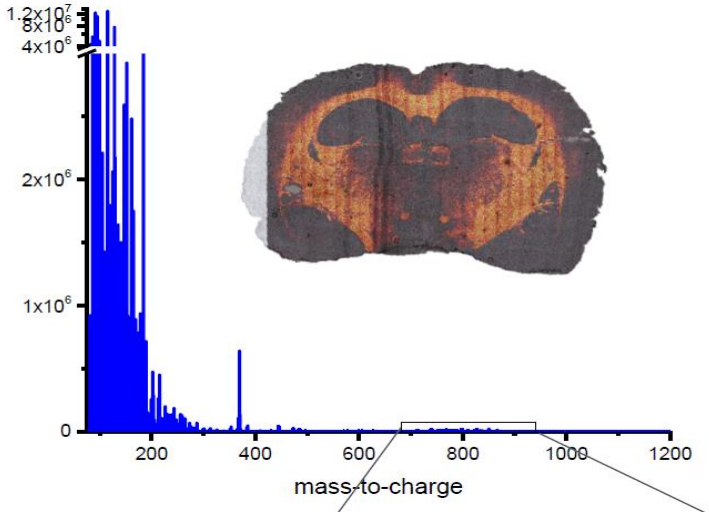
- TOF.SIMS 5 platform
- Bi Nanoprobe for highest lateral resolution
- Ar Gas Cluster Source (GCS) with resolution < 2 µm

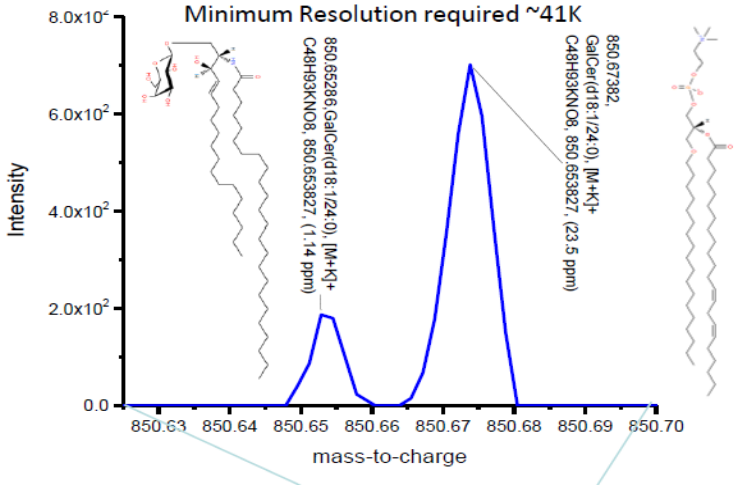
Operational modes

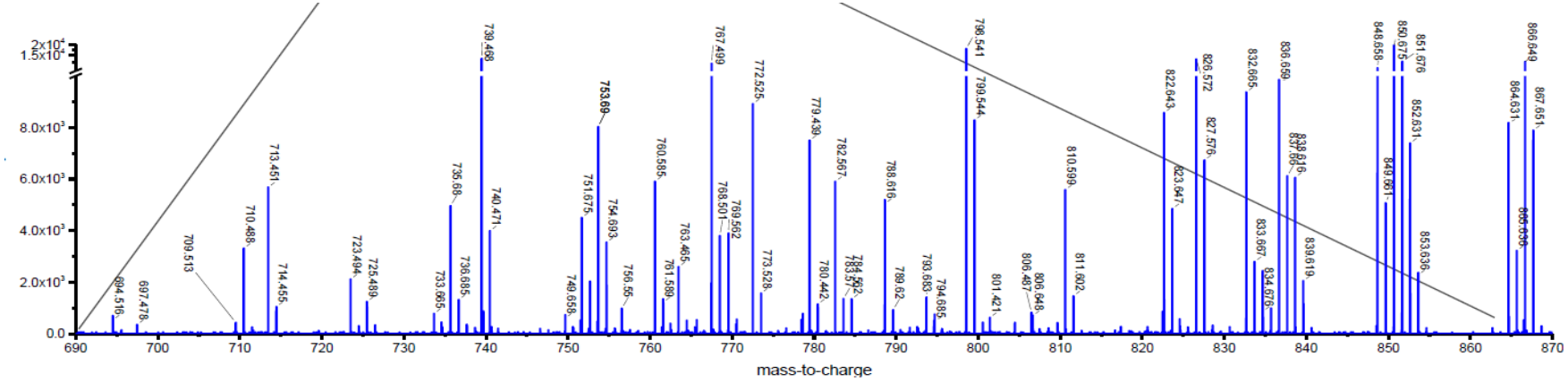
- all conventional pulsed ToF-SIMS modes
- “DC” operation with single beam mode using Orbitrap ™



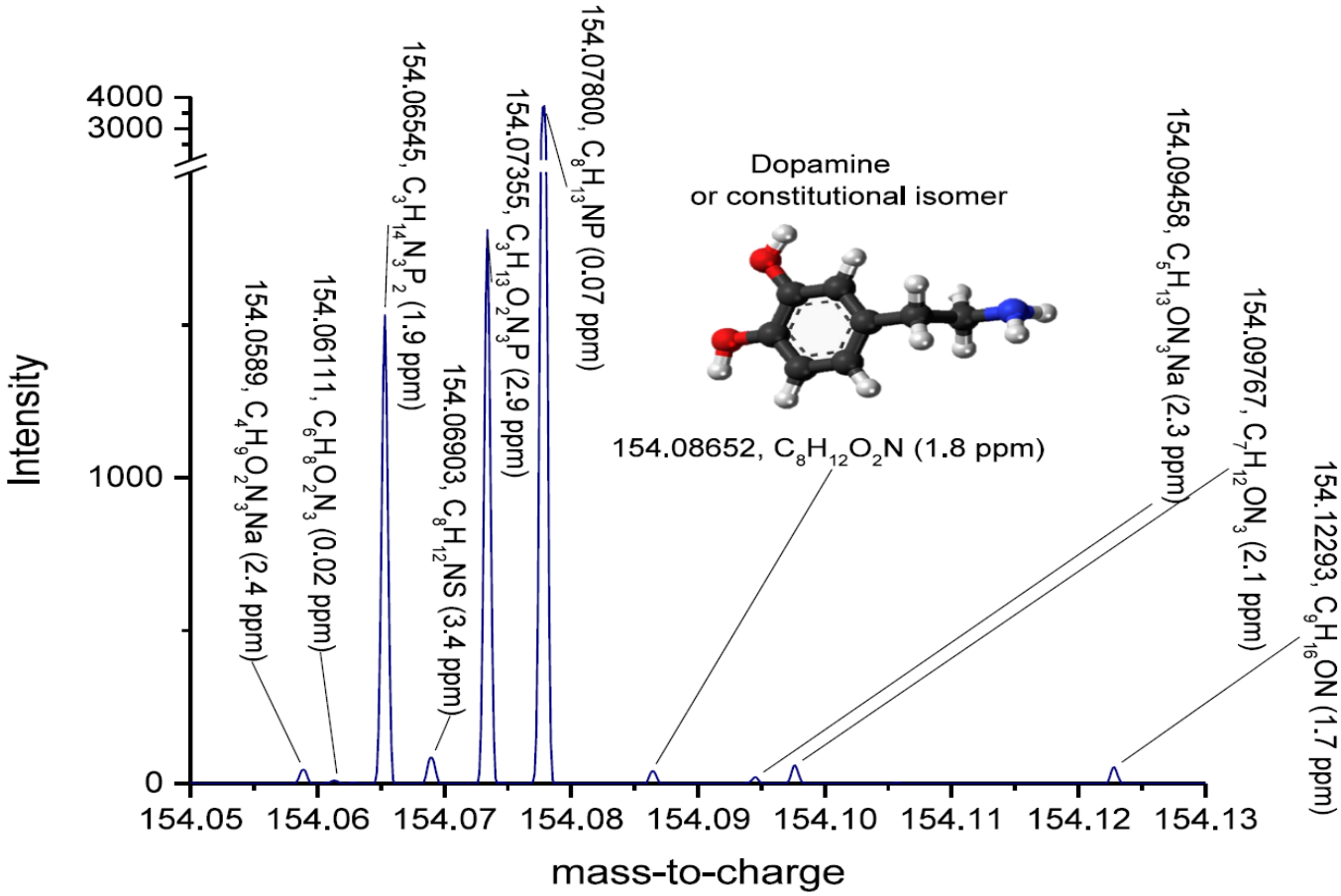
Ultra High Mass Resolution Lipidomics





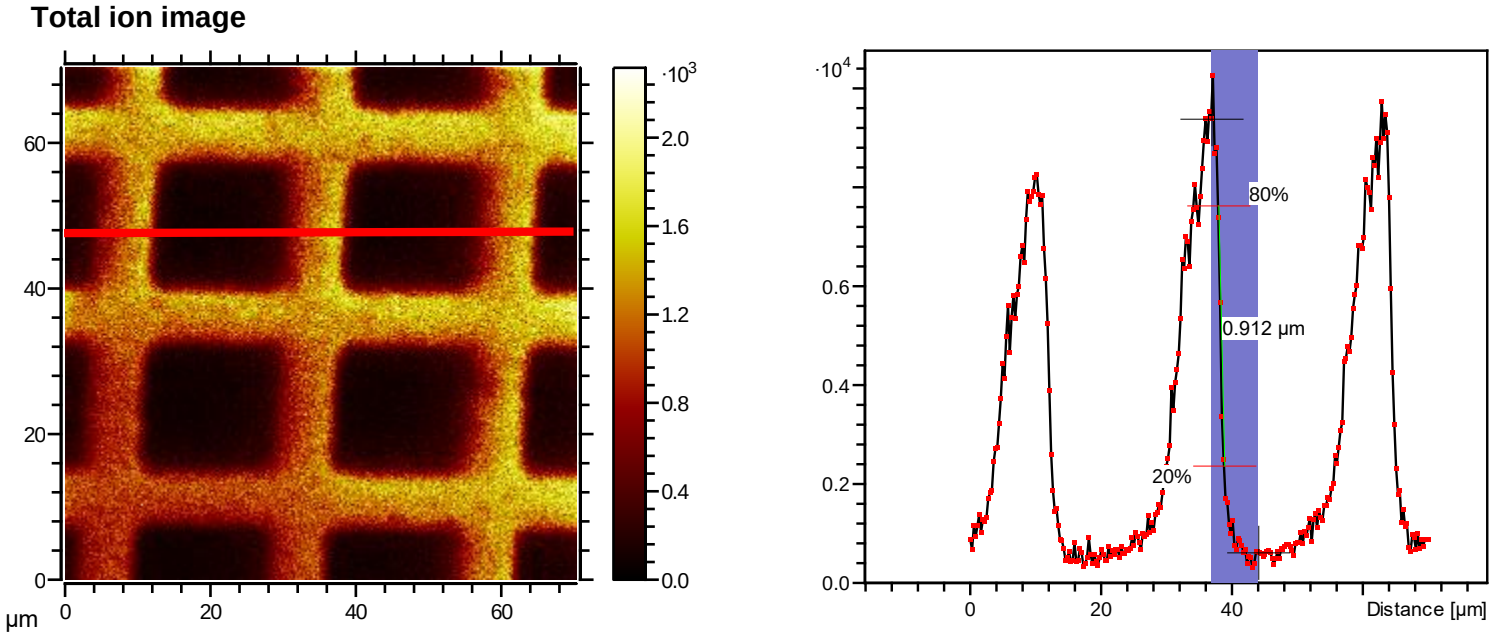


Detection of Neurotransmitter Dopamine



High Resolution Gas Cluster Source

INNOVATIVE SURFACE ANALYSIS



- Ion source: Ar Gas cluster ion source
- Beam energy: 20 keV
- Pulsed target current: 6 pA

- Lateral resolution: **< 1 µm**

Submicron Argon Gas Cluster SIMS Imaging

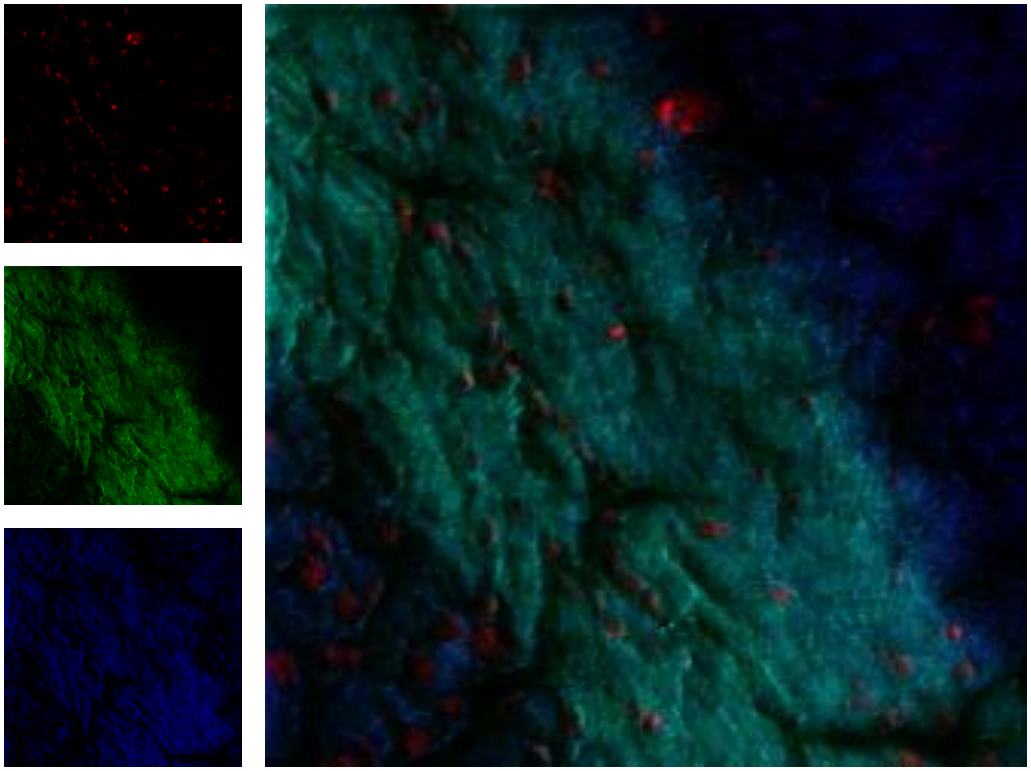
INNOVATIVE SURFACE ANALYSIS

250 x 250 µm²

$C_5H_4N_5^-$
Adenine

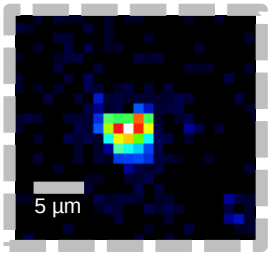
$C_{48}H_{90}NSO_{11}^-$
C24:1 Sulfatide

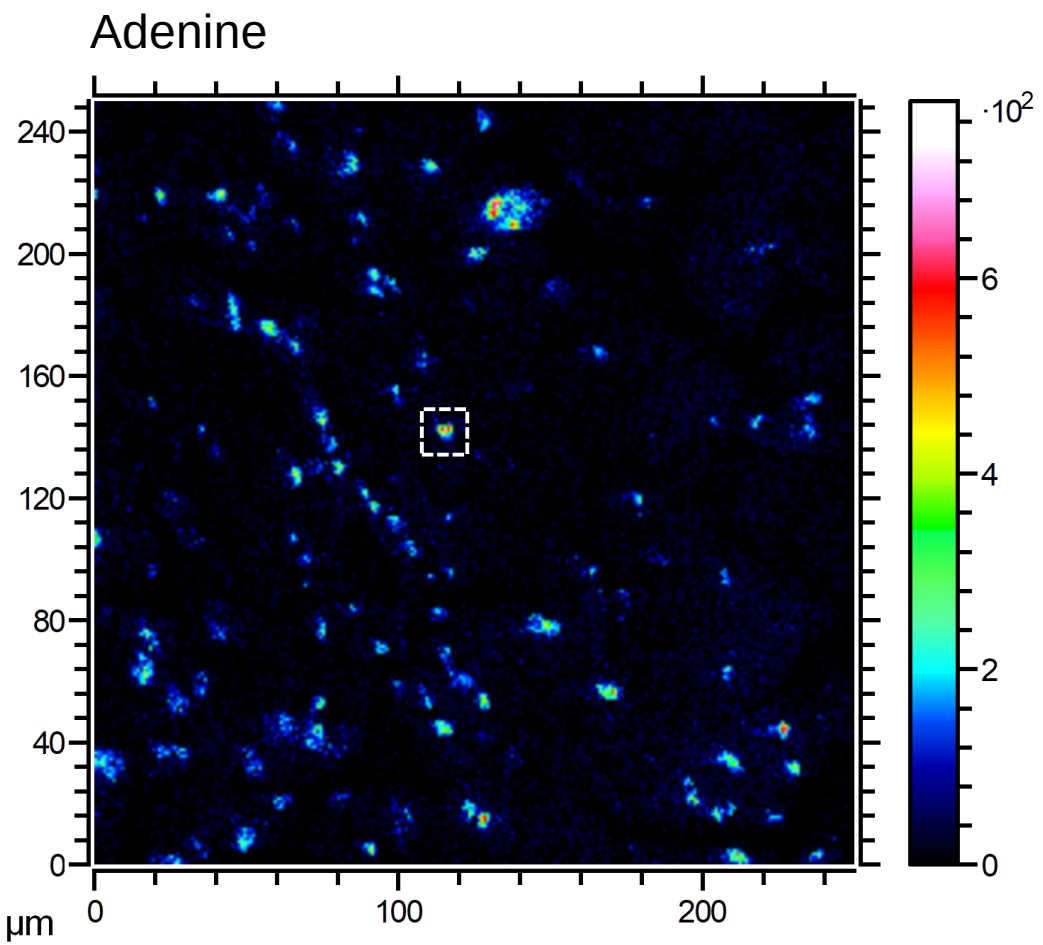
$C_6H_{10}PO_8^-$
PI headgroup fragment



Submicron Ar Cluster SIMS Imaging

FOV: 250 µm
Raster size: 250x250
PI current: 3.67 pA
Dose density: 1.94E15 PI/cm²





Thank you for your attention!