

# MERC Isotope Geochemistry Laboratory:

*Analytical Capabilities and  
Recent Developments*

Jeff Marsh

MERC-Laurentian University

SEM HV: 10.0 kV	WD: 17.20 mm	VEGA3 TESCAN
View field: 361 µm	Det: CL	100 µm
SEM MAG: 576 x	Date(m/d/y): 02/22/19	MERC-IGL Laurentian Univ.



A new Canadian research initiative funded  
by Canada First Research Excellence Fund.

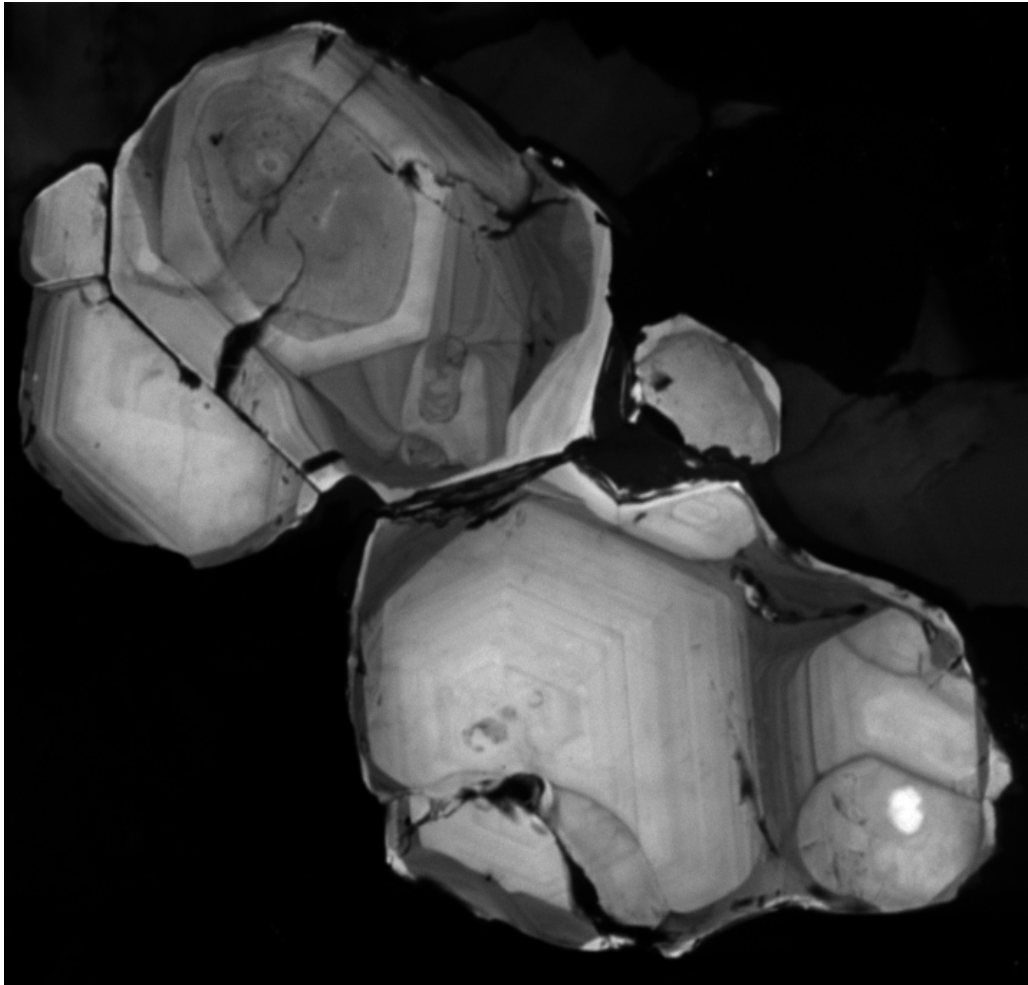


Canada



# MERC - Isotope Geochemistry Lab

## Talk Overview



- New SEM installation
  - BSE & CL imaging capabilities
  - EDS capabilities (after next week)
- LA-ICPMS analytical performance
  - U-Pb data: quadrupole vs. multicollector
  - U-Pb data: precision & reproducibility
  - Hf data: precision & reproducibility
  - LASS: comparisons with single stream
- Maniitsoq zircon: a new Archean RM?
  - U-Pb systematics: TIMS vs. LA-ICPMS
- Hf isotope analysis of detrital zircon

# LA-ICPMS Instrumentation

## \* Photon Machines Analyte G2 ArF excimer laser

- 193 nm wavelength, <5 ns pulse duration
- Large format, two-volume Helex II cell (~1 sec. washout)
  - \* Holds nine 1" rounds or four thin sections + three 1" rounds

## \* Two plasma source mass spectrometers (ICP-MS)

- Thermo iCap-TQ: triple quadrupole ICP-MS
- Thermo Neptune: multicollector sector-field ICP-MS



*Neptune*



*Analyte G2*



*iCap-TQ*

# LA-ICPMS Instrumentation + SEM

## \* Photon Machines Analyte G2 ArF excimer laser

- 193 nm wavelength, <5 ns pulse duration
- Large format, two-volume Helex II cell (~1 sec. washout)
  - \* Holds nine 1" rounds or four thin sections + three 1" rounds

## \* Two plasma source mass spectrometers (ICP-MS)

- Thermo iCap-TQ: triple quadrupole ICP-MS
- Thermo Neptune: multicollector sector-field ICP-MS

## \* Tescan Vega 3 SEM (w/BSE, CL, EDS)

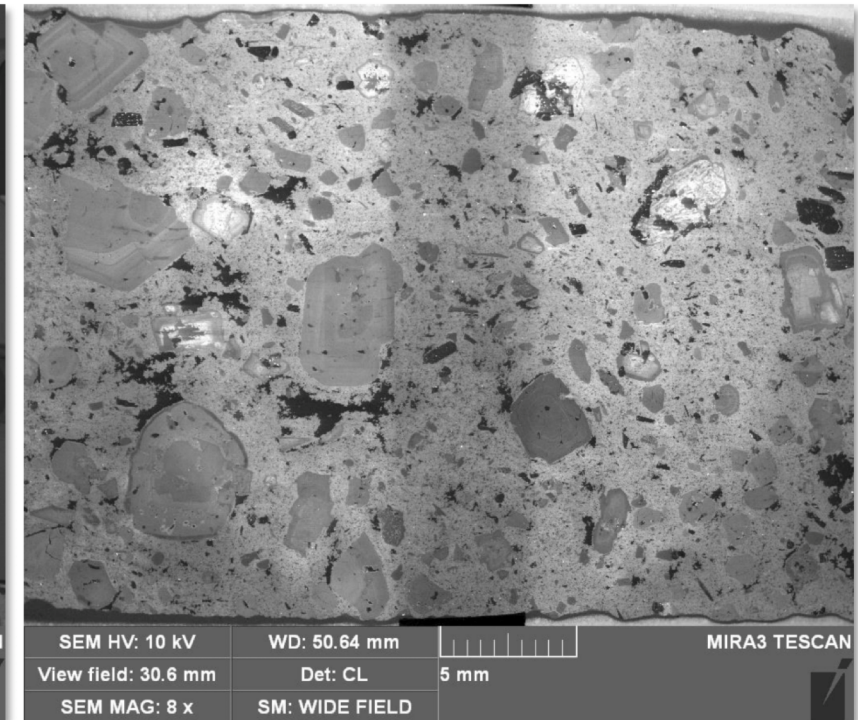
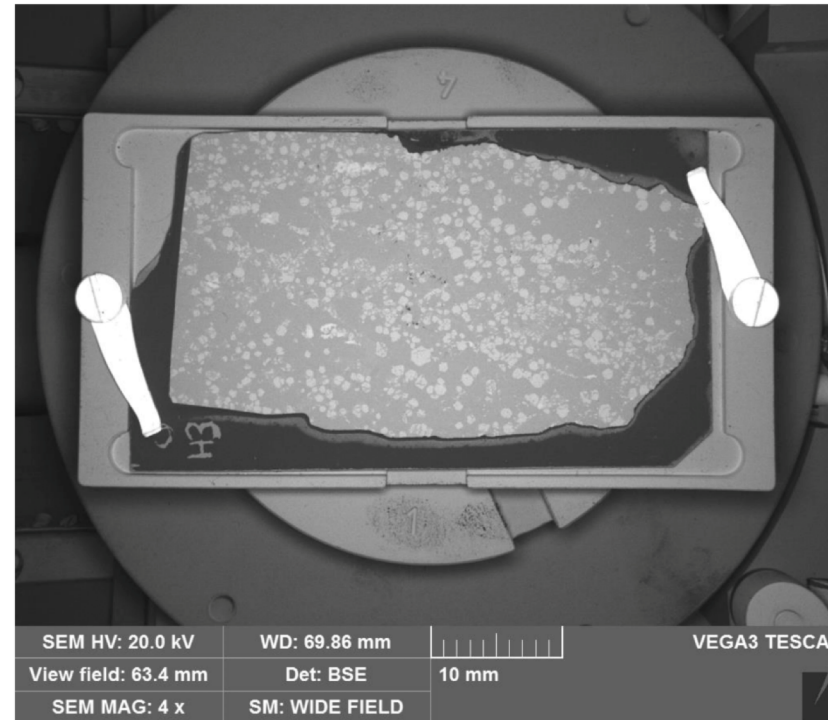
- Tescan monochromatic CL detector (w/simultaneous BSE/SE/EDS)
- Bruker EDS system, 60 mm<sup>2</sup> chip
- Motorized stage automation
- Widefield imaging
- 4 thin section/6 puck capacity



# SEM-EDS system

Tescan Vega 3 - LMH

- Motorized stage automation
- Widefield imaging
- 4 thin section/6 puck capacity

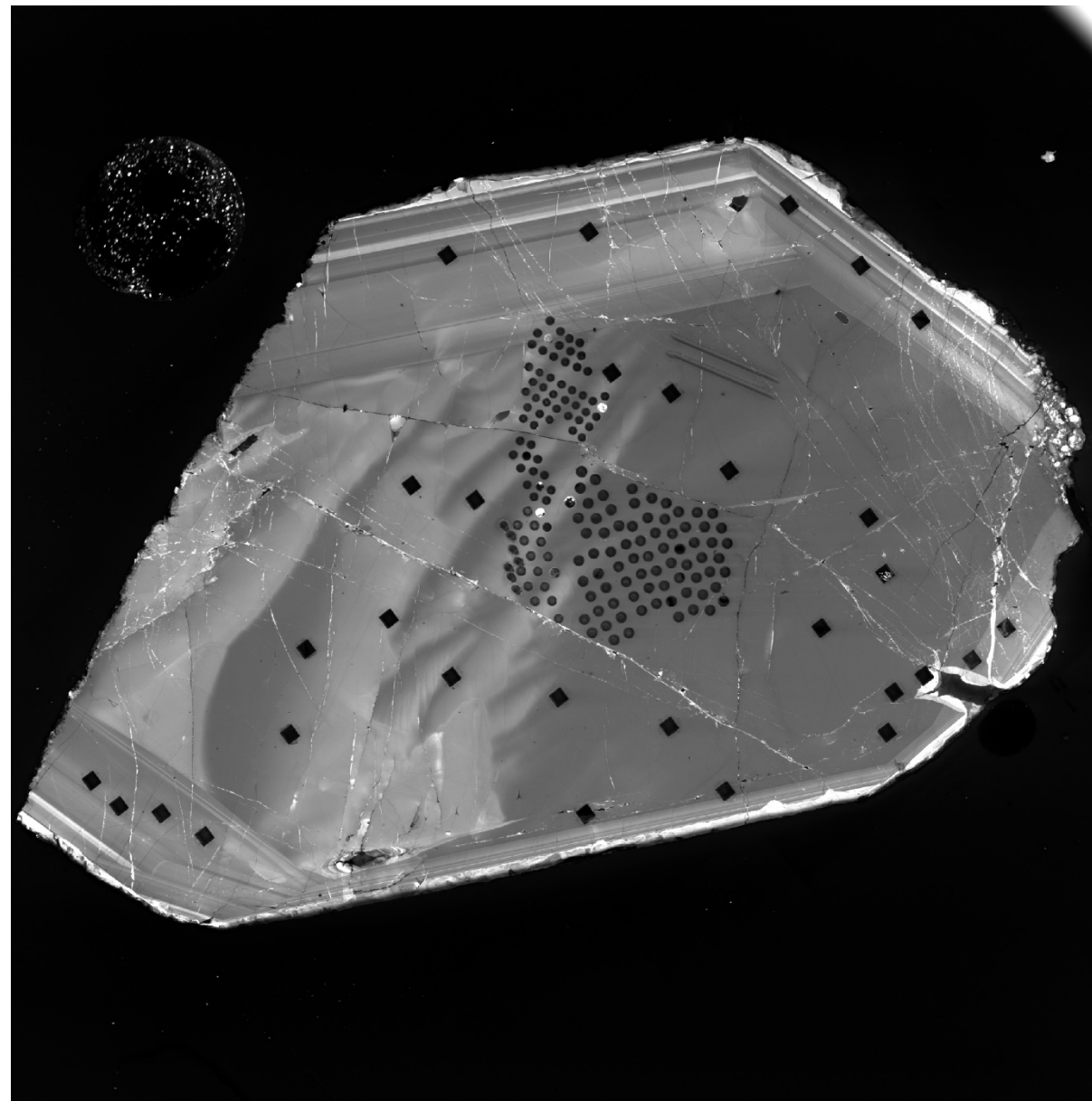


Widefield SE/BSE/CL imaging and Positioner tool enables rapid (~1 minute) imaging of entire specimen and single click stage movement to feature of interest.

# SEM-EDS system

Tescan Vega 3 - LMH

- Monochromatic CL Imaging
- High resolution imaging of large areas (up to 25 mm<sup>2</sup>) in ~15 minutes
- High resolution imaging of small areas (~1 mm<sup>2</sup>) in ~1 minute



Monochromatic  
CL image of  
Maniitsoq  
zircon

2560<sup>2</sup> pixel  
resolution over  
~3mm<sup>2</sup> area

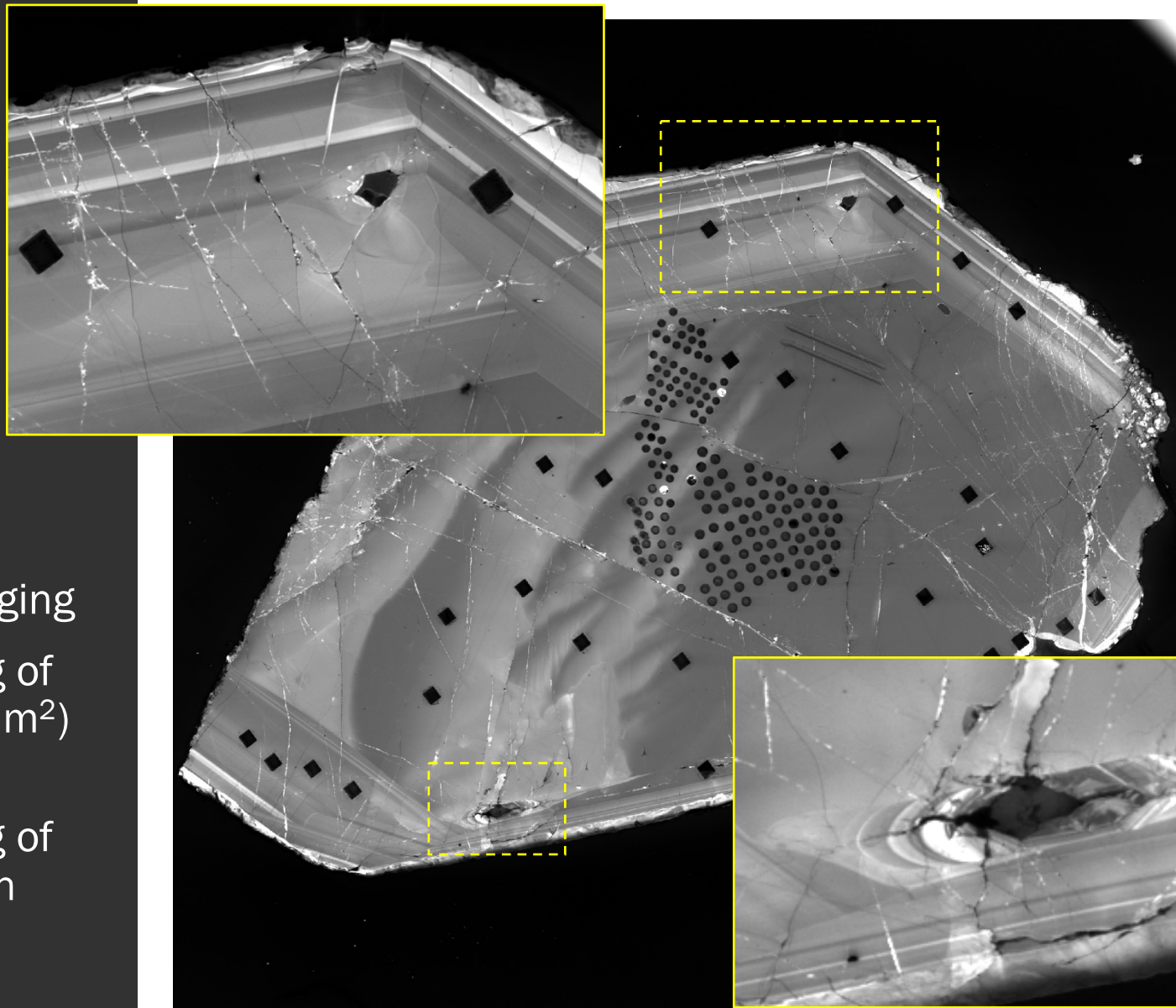
11 minute long  
scan

SEM HV: 10.0 kV	WD: 15.18 mm	VEGA3 TESCAN
View field: 2.89 mm	Det: CL	500 μm
SEM MAG: 239 x	Date(m/d/y): 02/07/19	Laurentian University

# SEM-EDS system

Tescan Vega 3 - LMH

- Monochromatic CL Imaging
- High resolution imaging of large areas (up to 25 mm<sup>2</sup>) in ~15 minutes
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View field: 2.89 mm	Det: CL	500 μm
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# SEM-EDS system

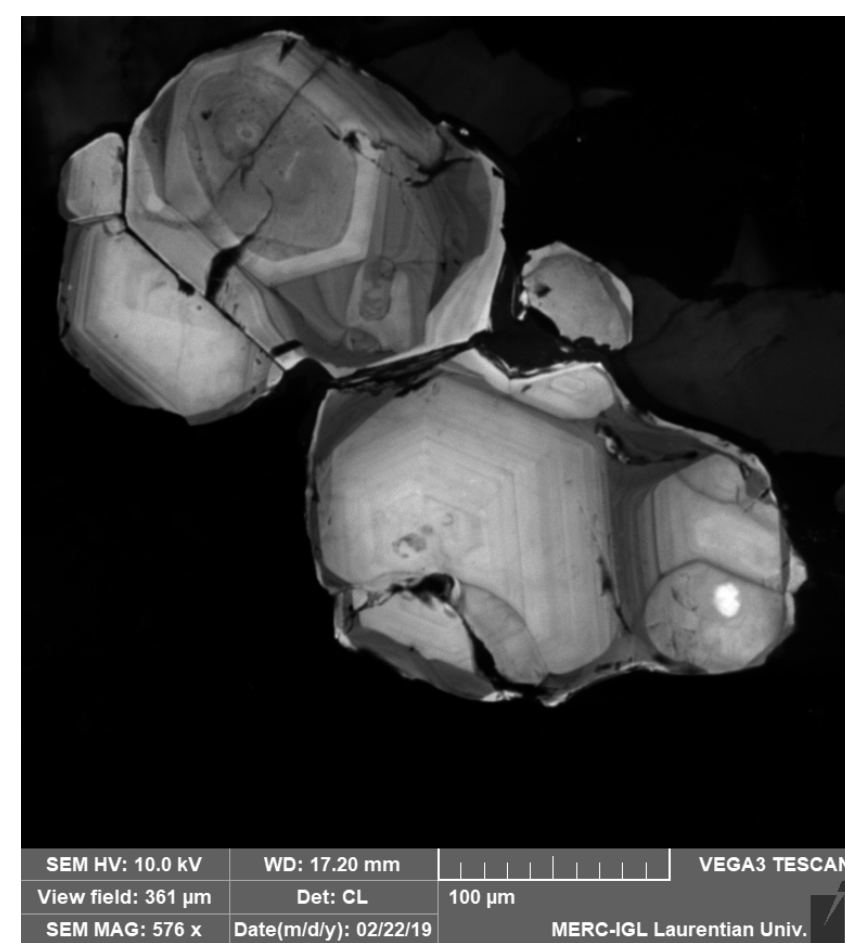
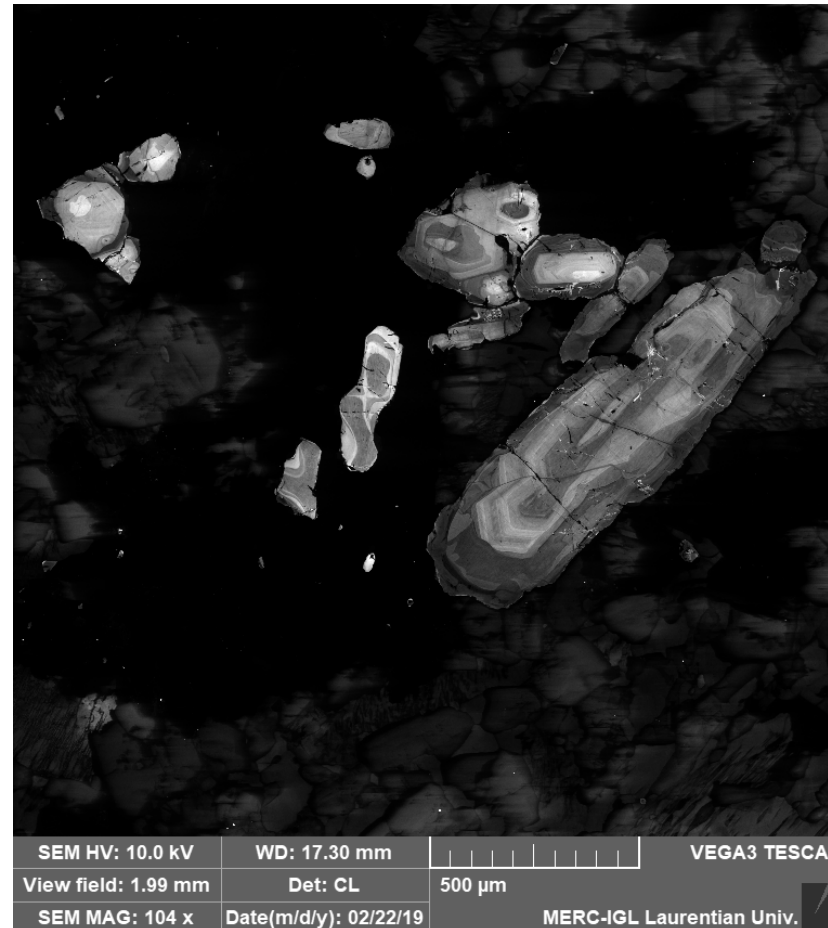
Tescan Vega 3 - LMH

- Monochromatic CL Imaging
- High resolution imaging of large areas (up to 25 mm<sup>2</sup>) in ~15 minutes
- High resolution imaging of small areas (~1 mm<sup>2</sup>) in ~1 minute

Monochromatic CL image of  
Chibougamau apatite in thin section

768<sup>2</sup> pixel resolution

1 minute long scan

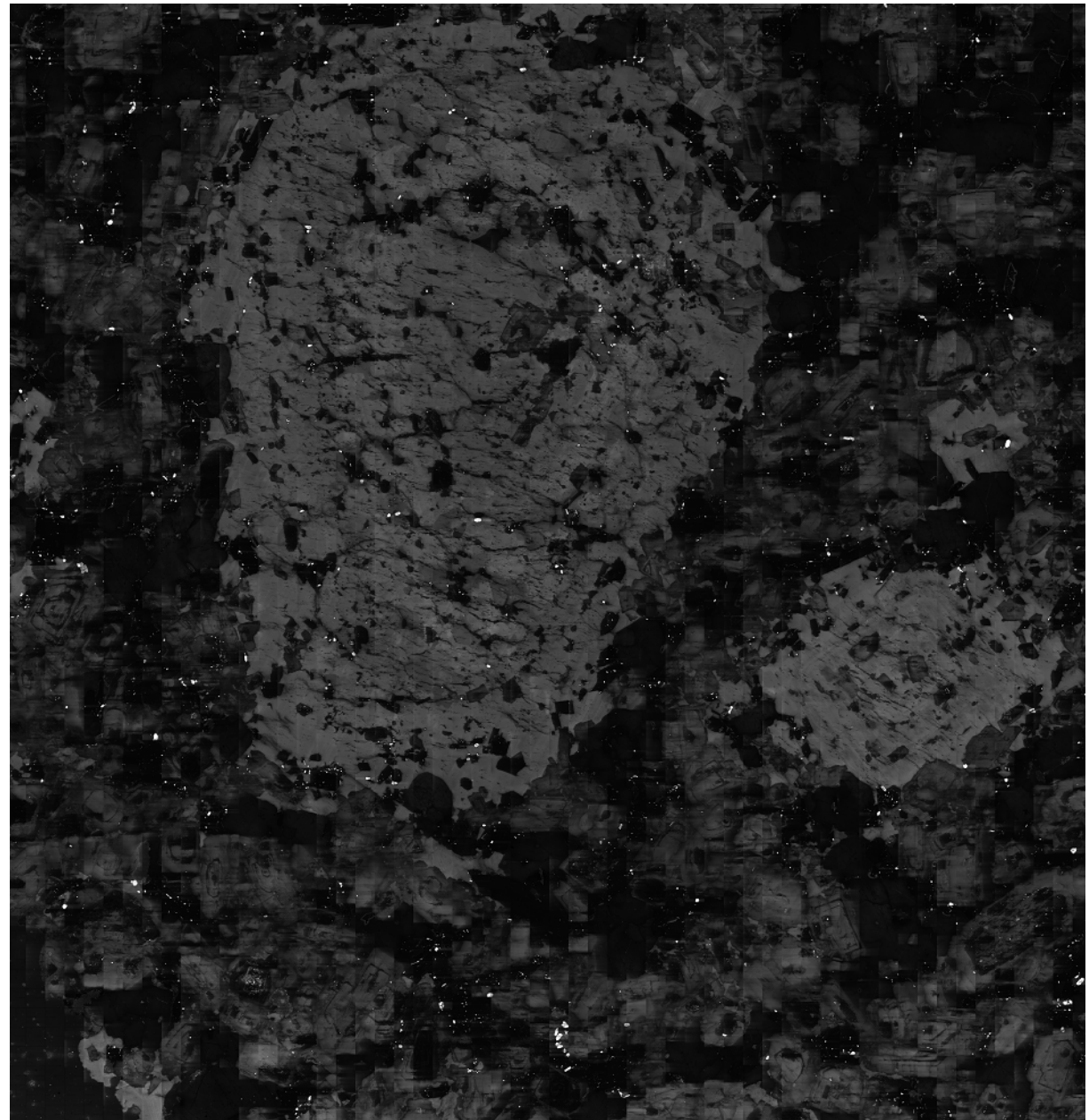




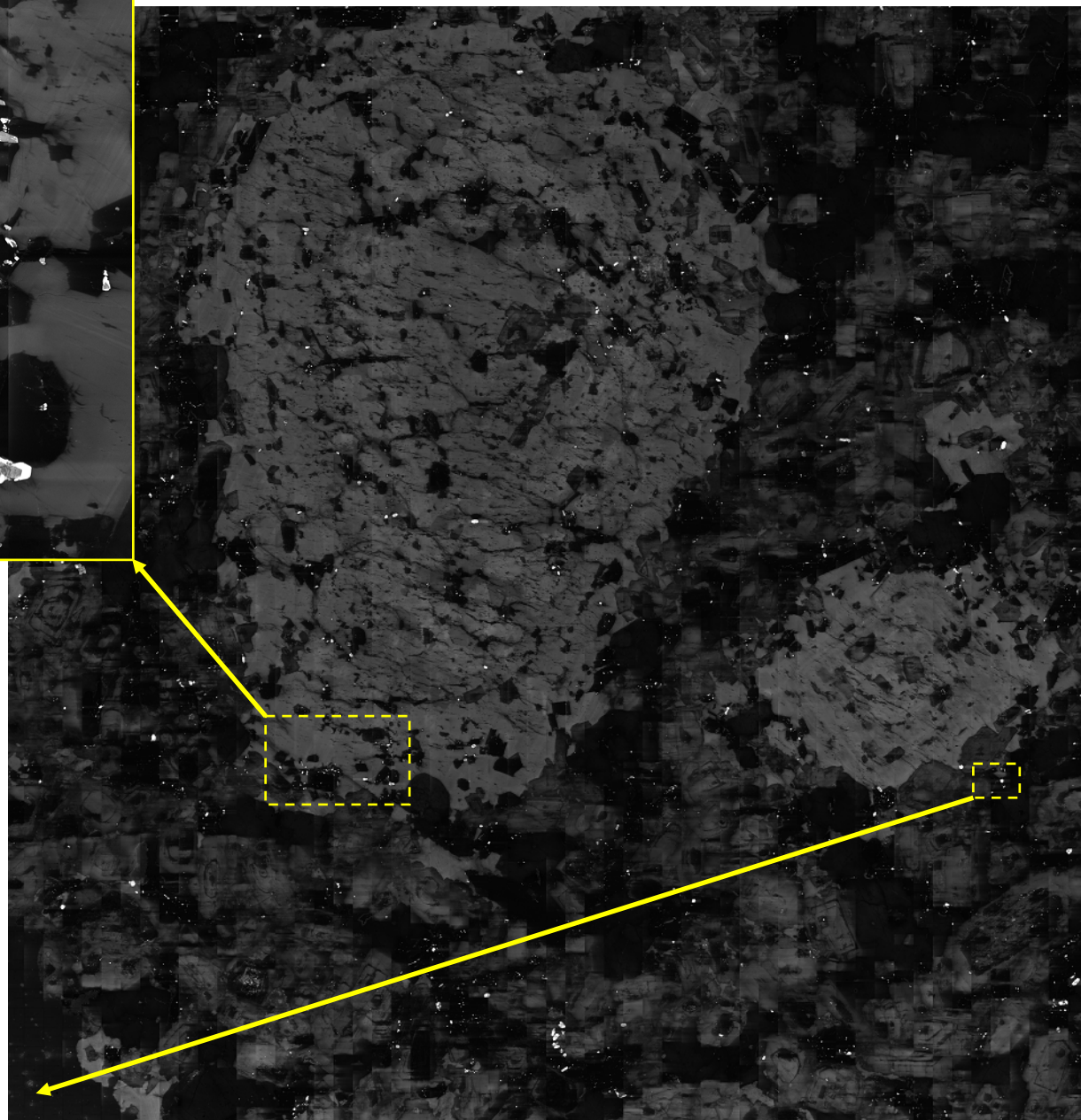
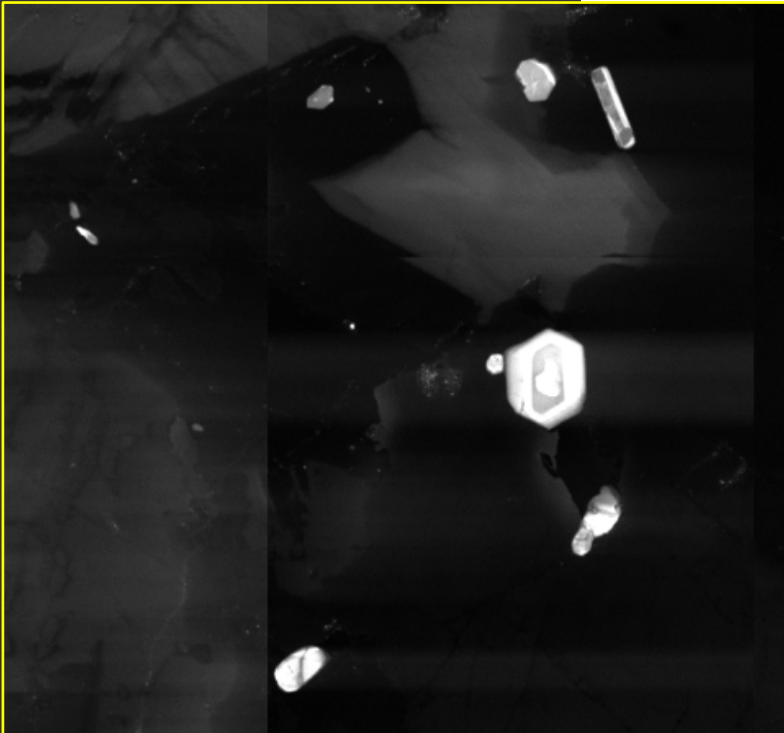
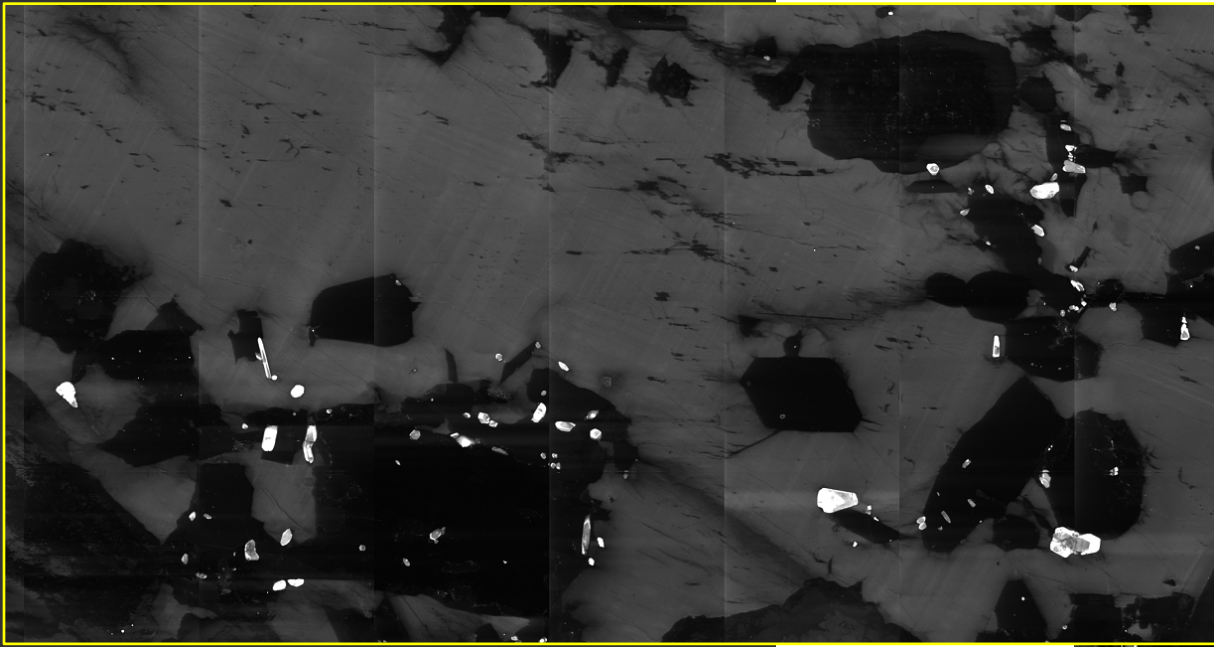
# SEM-EDS system

Tescan Vega 3 - LMH

- Large Area CL Imaging with Image Snapper
- Stitching together of hundreds/thousands of high-resolution images (covering full thin section) in ~1.5 hours



Monochromatic CL image of Chibougamau syenite in thin section (~15 \* 20 mm)



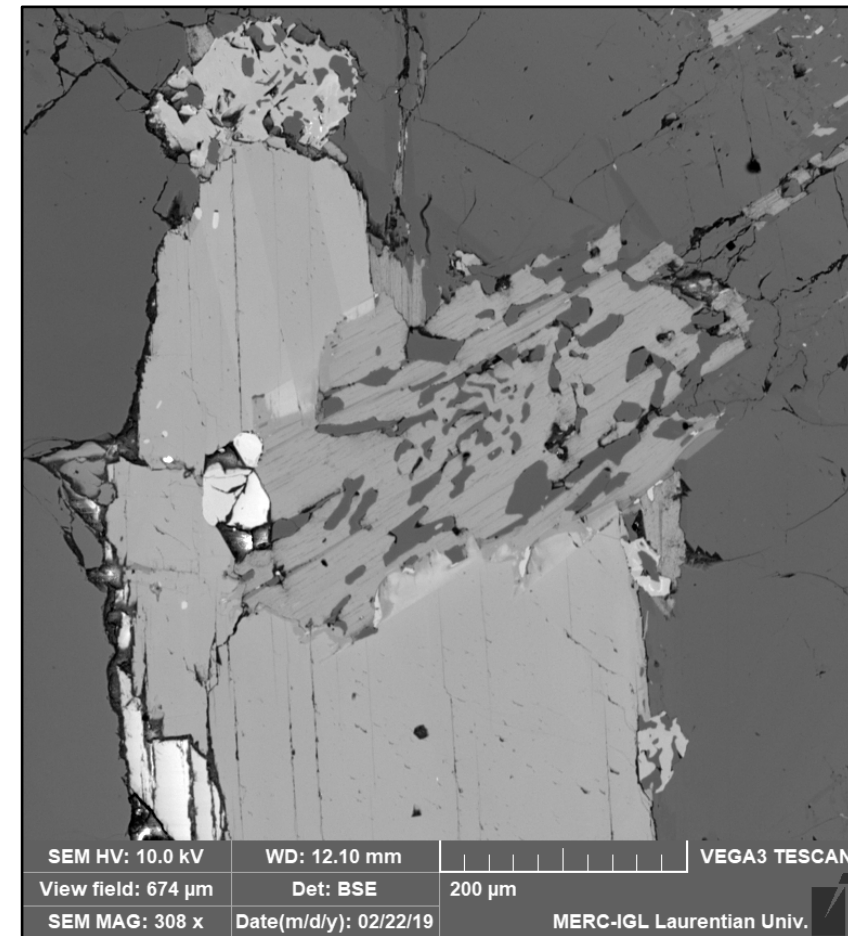
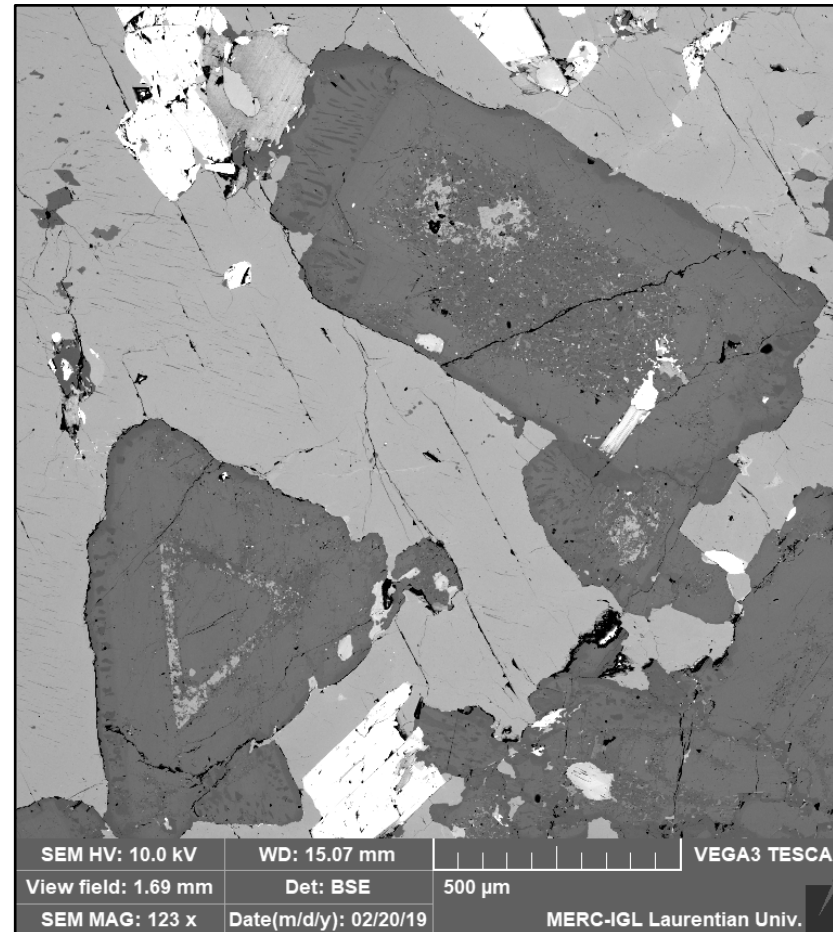
Monochromatic CL image of Chibougamau syenite in thin section (~15 \* 20 mm)

# SEM-EDS system

Tescan Vega 3 - LMH

- High resolution and large area BSE Imaging with Image Snapper

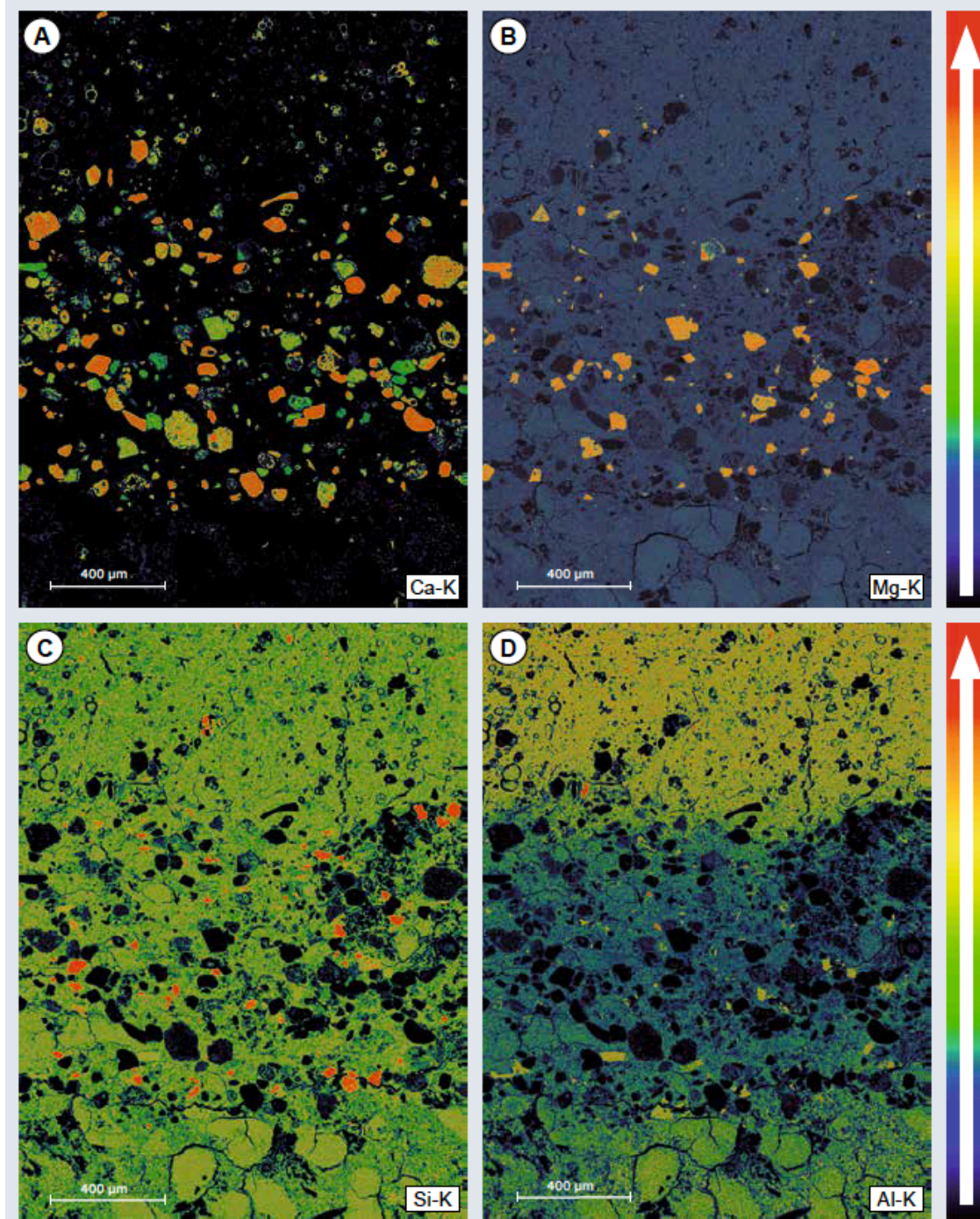
BSE image of reaction textures from Chibougamau  
granodiorite in thin section



# SEM-EDS system

Tescan Vega 3 - LMH

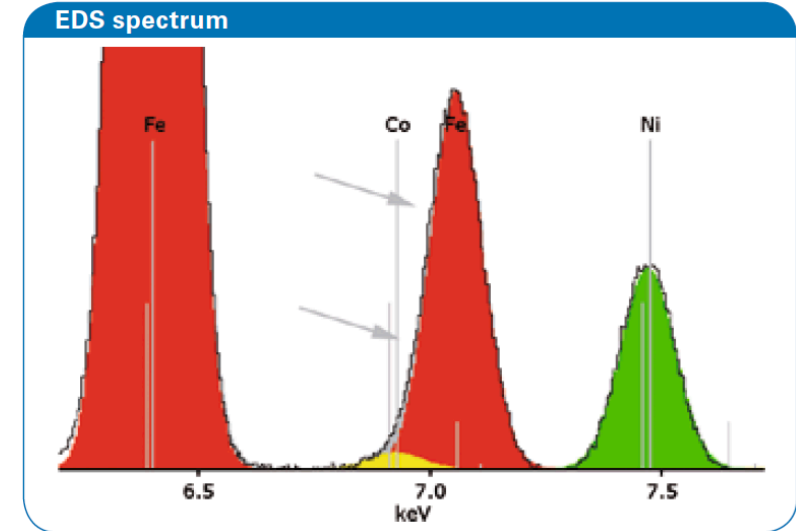
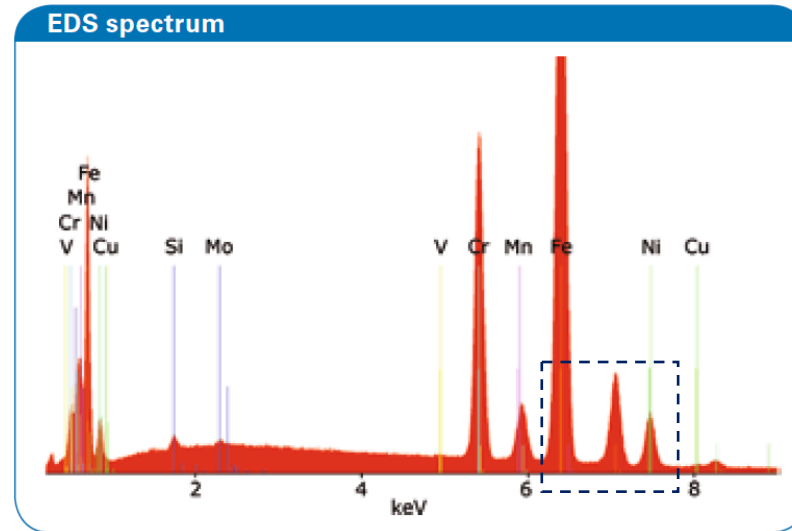
- Bruker Quantax – with X-Flash 60 mm<sup>2</sup> detector
- Large area mapping: elemental concentration maps for entire periodic table (nearly), with peak deconvolution capabilities for overlapping X-ray energies



# SEM-EDS system

Tescan Vega 3 - LMH

- Bruker Quantax – with X-Flash 60 mm<sup>2</sup> detector
- Standard-based quantification: accurate elemental concentration determination, down to <<wt.% (peak deconvolution)

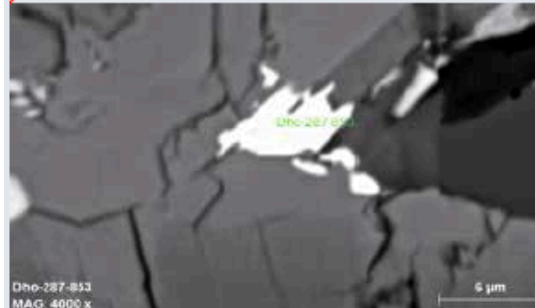
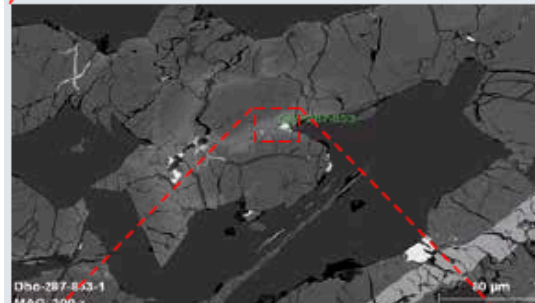
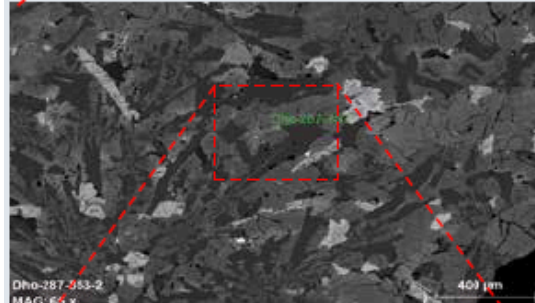
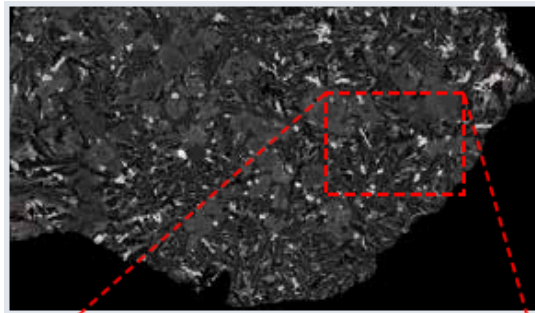


Method	Si	V	Cr	Mn	Fe	Co	Ni	Cu	Mo
P/B-ZAF, reference for cobalt	0.27	0.13	17.83	1.55	71.64	0.12	7.74	0.43	0.29
$\Phi(\rho, z)$ standard-based	0.33	0.13	18.27	1.51	70.90	0.12	8.07	0.37	0.31
Comparison S-OES, XRF	0.33	0.09	18.30	1.61	70.56	0.14	8.13	0.37	0.29

# SEM-EDS system

Tescan Vega 3 - LMH

- Bruker Quantax – with X-Flash 60 mm<sup>2</sup> detector
- Particle analysis: rapid search of entire specimen for particles with specified BSE intensity, followed by analysis, chemical/physical characterization, and automated revisitation



ID	Area /μm <sup>2</sup>	Max. length /μm	Width /μm	Perimet. /μm	Zr Wt.%
01-00073	8.1	4.9	4.0	16.5	63.2
01-00143	5.3	3.4	2.6	9.5	60.3
01-00365	12.2	6.3	3.8	16.4	62.0
01-00381	13.4	6.7	3.2	16.0	62.7
01-00399	14.2	9.4	3.9	28.0	59.5
01-00571	6.1	4.3	2.6	10.7	67.9
01-00620	5.7	4.3	2.3	10.3	63.5
01-00815	13.0	6.4	4.3	17.5	66.2
01-00853	19.1	8.1	4.5	19.9	66.5
01-00870	9.8	4.9	3.2	12.2	66.9
01-00951	11.4	5.1	4.4	17.0	60.6

# LA-ICPMS analytical development

## U-Pb geochronology

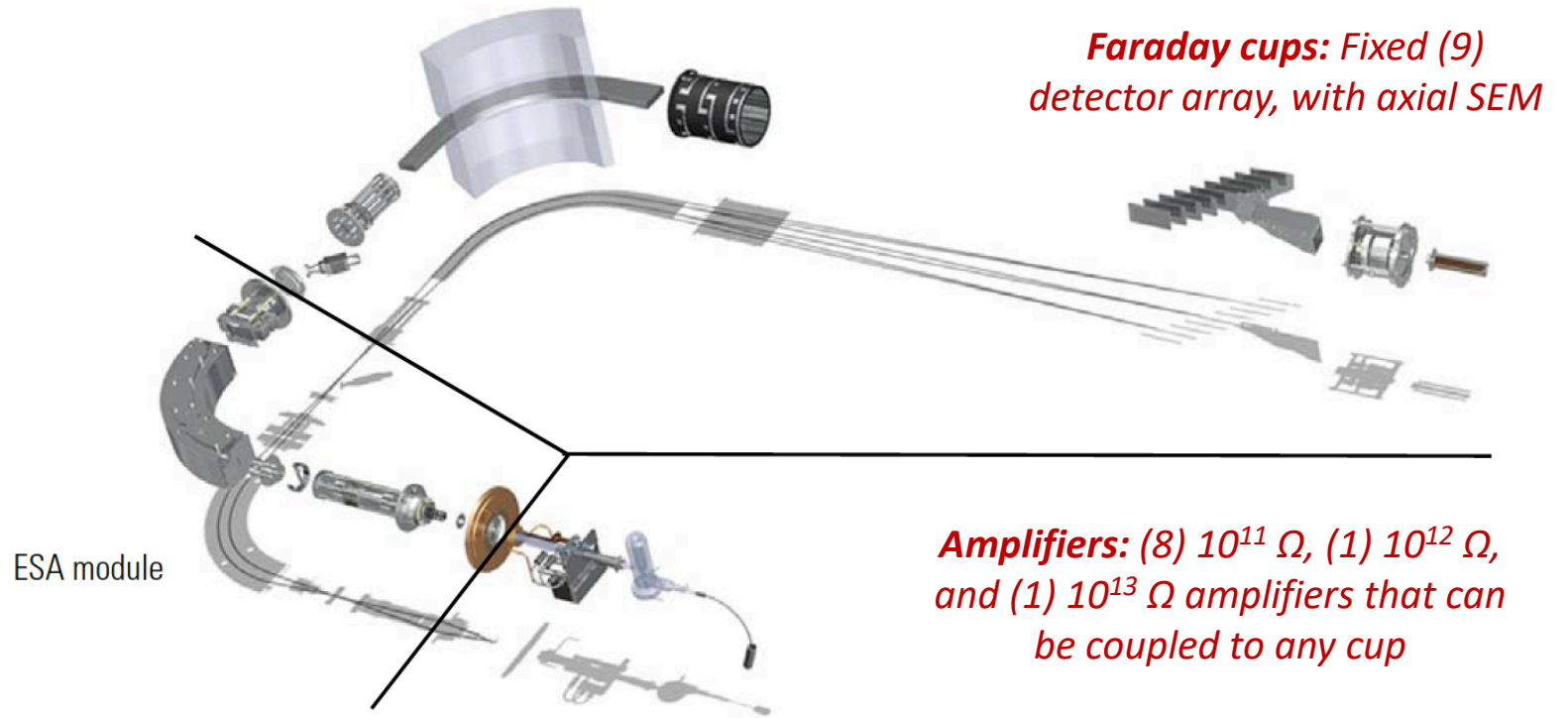
- U-Pb on Neptune developed in July 2018
- No ion counters for Pb
- 238-204 is maximum mass range (can't measure  $^{202}\text{Pb}/\text{Hg}$ )
- Not good for low Pb\* (very young/low U) samples

Cup  
Mass  
Amplifier

### U-Pb cup configuration (Faraday's only)

Cup	L4	L3	L2	L1	Ax	H1	H2	H3	H4
Mass	$^{204}\text{Pb}$	$^{206}\text{Pb}$	$^{207}\text{Pb}$	$^{208}\text{Pb}$	~220.65		$^{232}\text{Th}$		$^{238}\text{U}$
Amplifier	$10^{11}$	$10^{11}$	$10^{12}$	$10^{11}$	$10^{11}$		$10^{11}$		$10^{11}$

ESA module



**Faraday cups: Fixed (9)  
detector array, with axial SEM**

**Amplifiers: (8)  $10^{11} \Omega$ , (1)  $10^{12} \Omega$ ,  
and (1)  $10^{13} \Omega$  amplifiers that can  
be coupled to any cup**

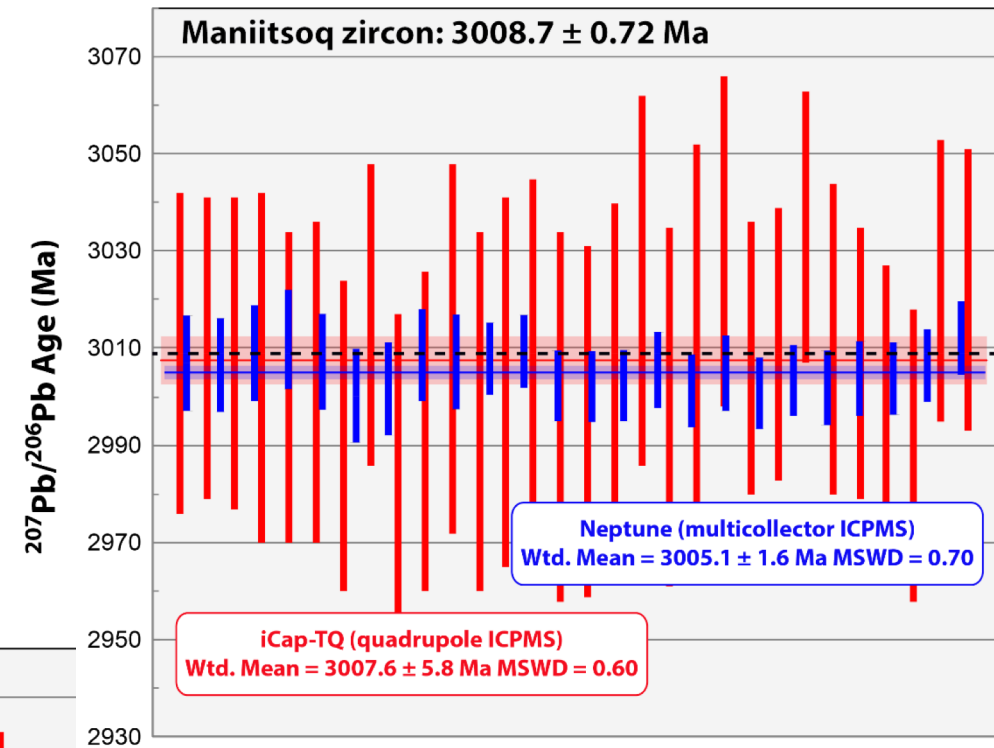
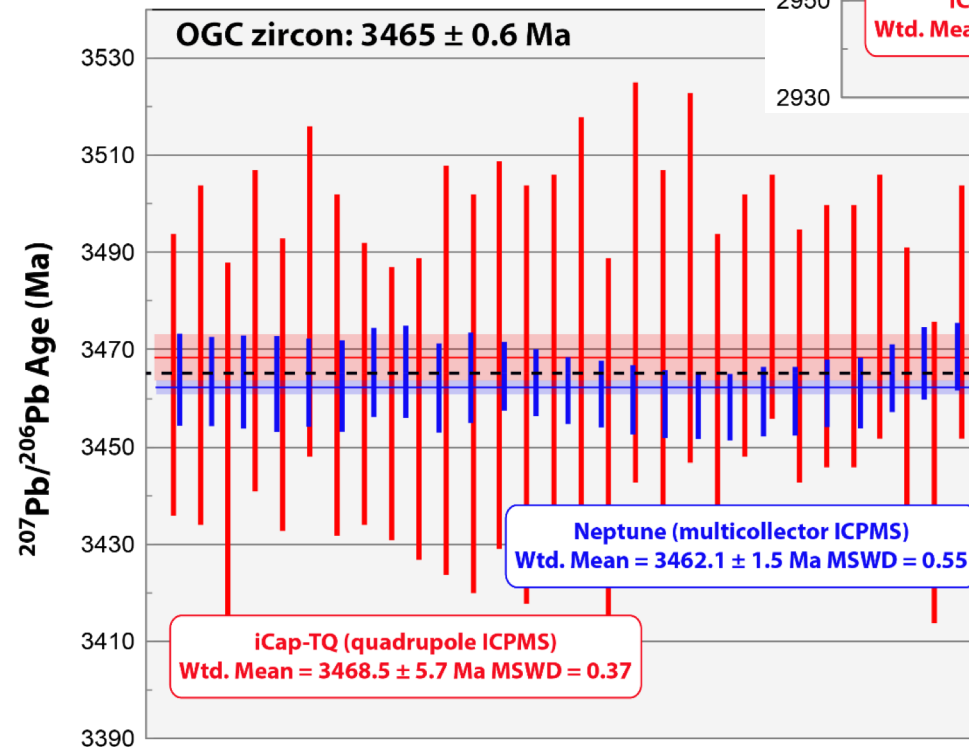
# LA-ICPMS analytical performance

U-Pb geochronology

- iCap-TQ vs. Neptune

MC-ICP provides ~5x better internal precision on U/Pb and Pb/Pb ratios than Q-ICP.

Single spot date uncertainty ~0.2-0.3% (i.e. 3000 ± 6-10 Ma)



High instrumental precision exposes other contributions to variability inherent to LA-ICPMS

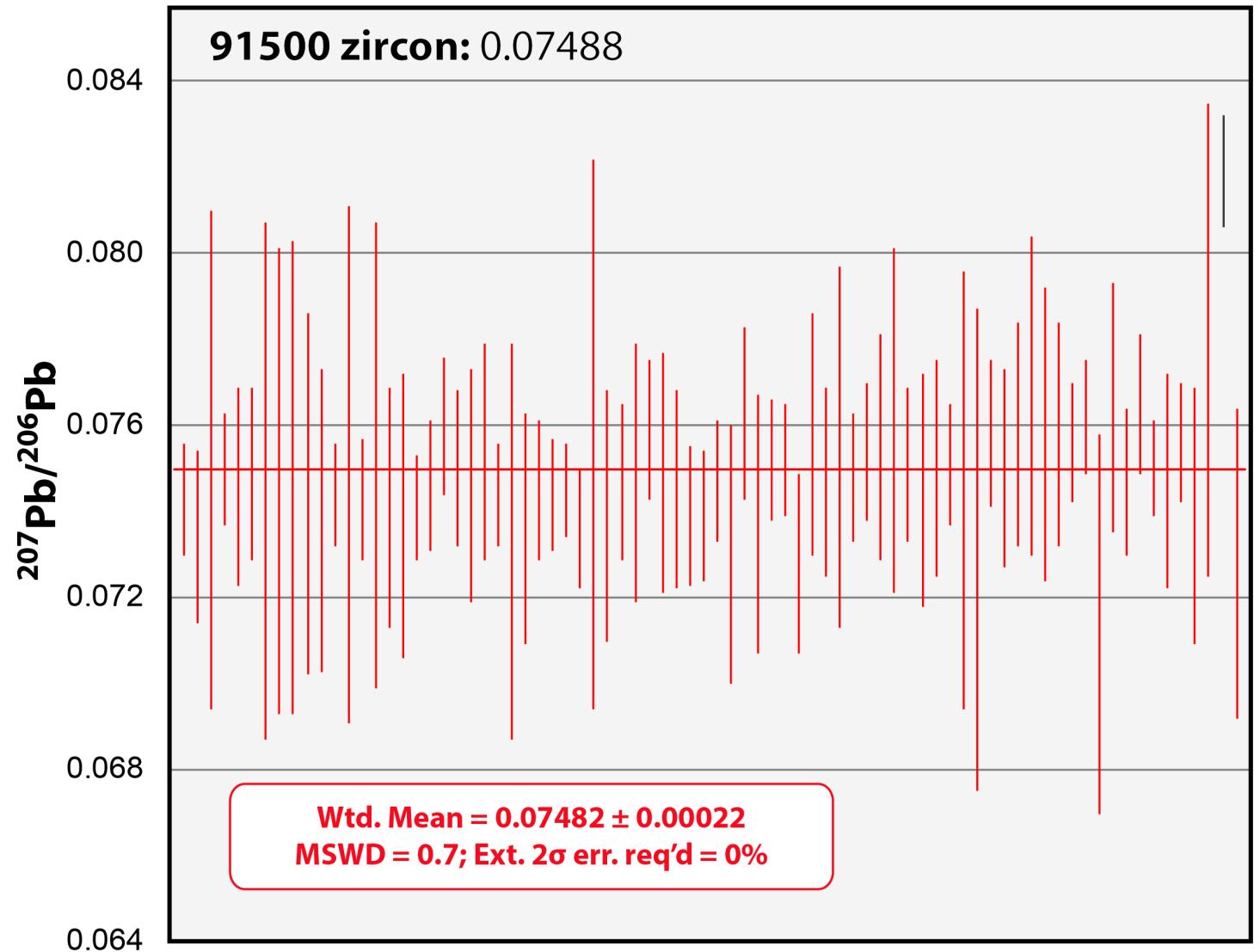
Demonstrates need for additional uncertainty propagation



# LA-ICPMS analytical performance

## U-Pb geochronology

- Precision & reproducibility
- $2\sigma$  RSD  $^{206}\text{Pb}/^{238}\text{U} = 6.31\%$ ;  
 $^{207}\text{Pb}/^{206}\text{Pb} = 2.74\%$

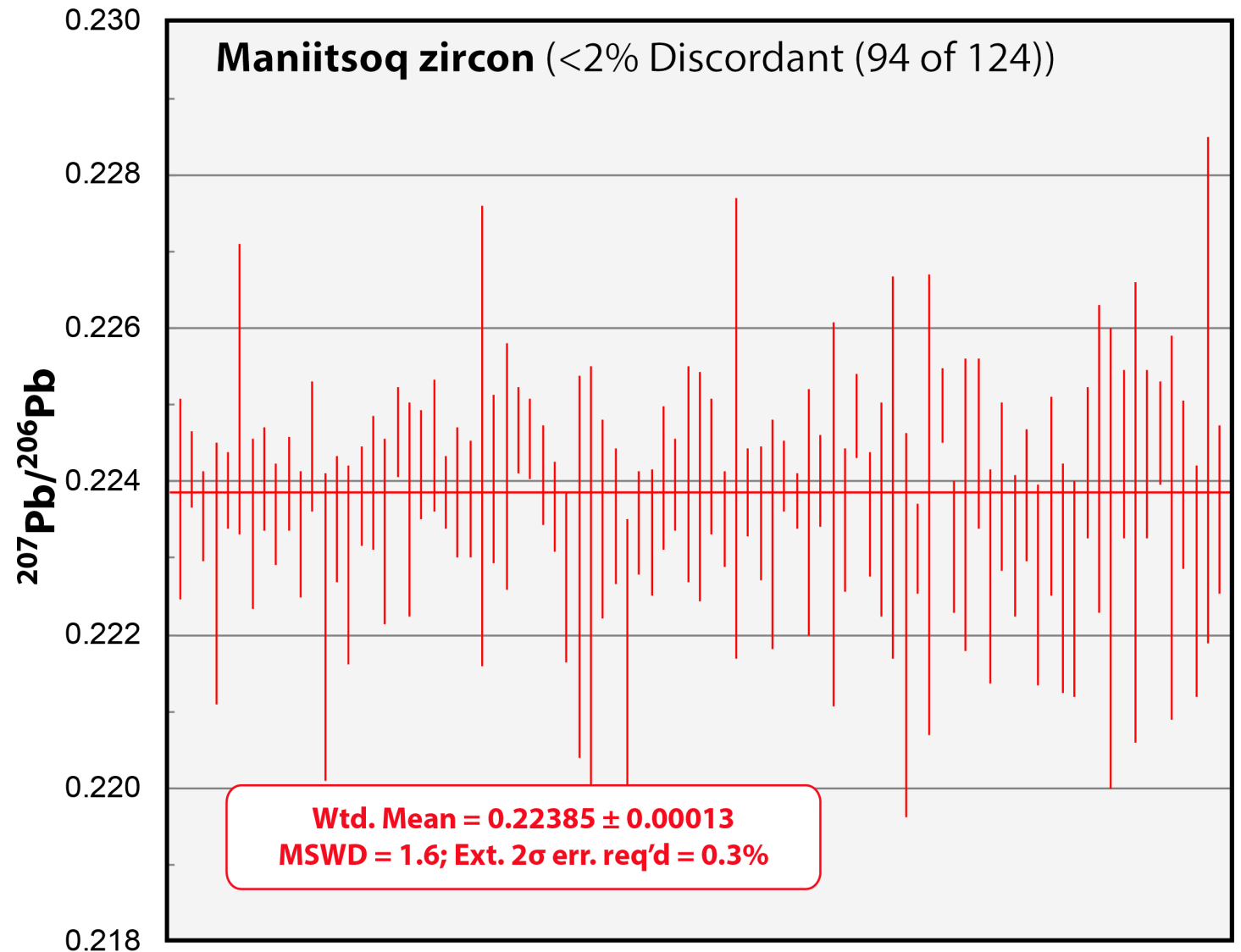


$^{207}\text{Pb}/^{206}\text{Pb}$  ratios for 91500 zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM)

# LA-ICPMS analytical performance

## U-Pb geochronology

- Precision & reproducibility
- $2\sigma$  RSD  $^{206}\text{Pb}/^{238}\text{U} = 2.15\%$ ;  
 $^{207}\text{Pb}/^{206}\text{Pb} = 0.62\%$

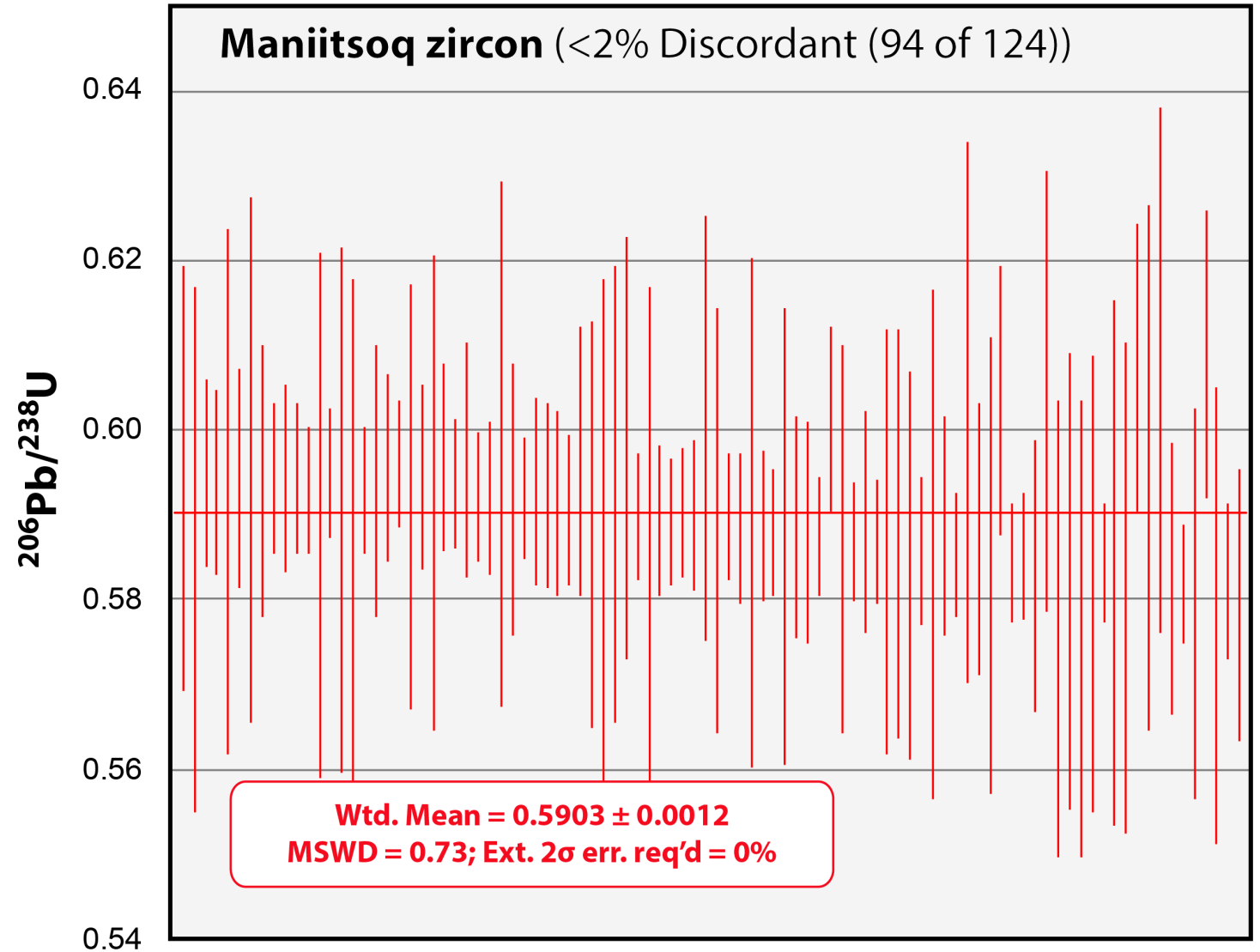


$^{207}\text{Pb}/^{206}\text{Pb}$  ratios for Maniitsoq zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM)

# LA-ICPMS analytical performance

## U-Pb geochronology

- Precision & reproducibility
- $2\sigma$  RSD  $^{206}\text{Pb}/^{238}\text{U} = 2.15\%$ ;  
 $^{207}\text{Pb}/^{206}\text{Pb} = 0.62\%$

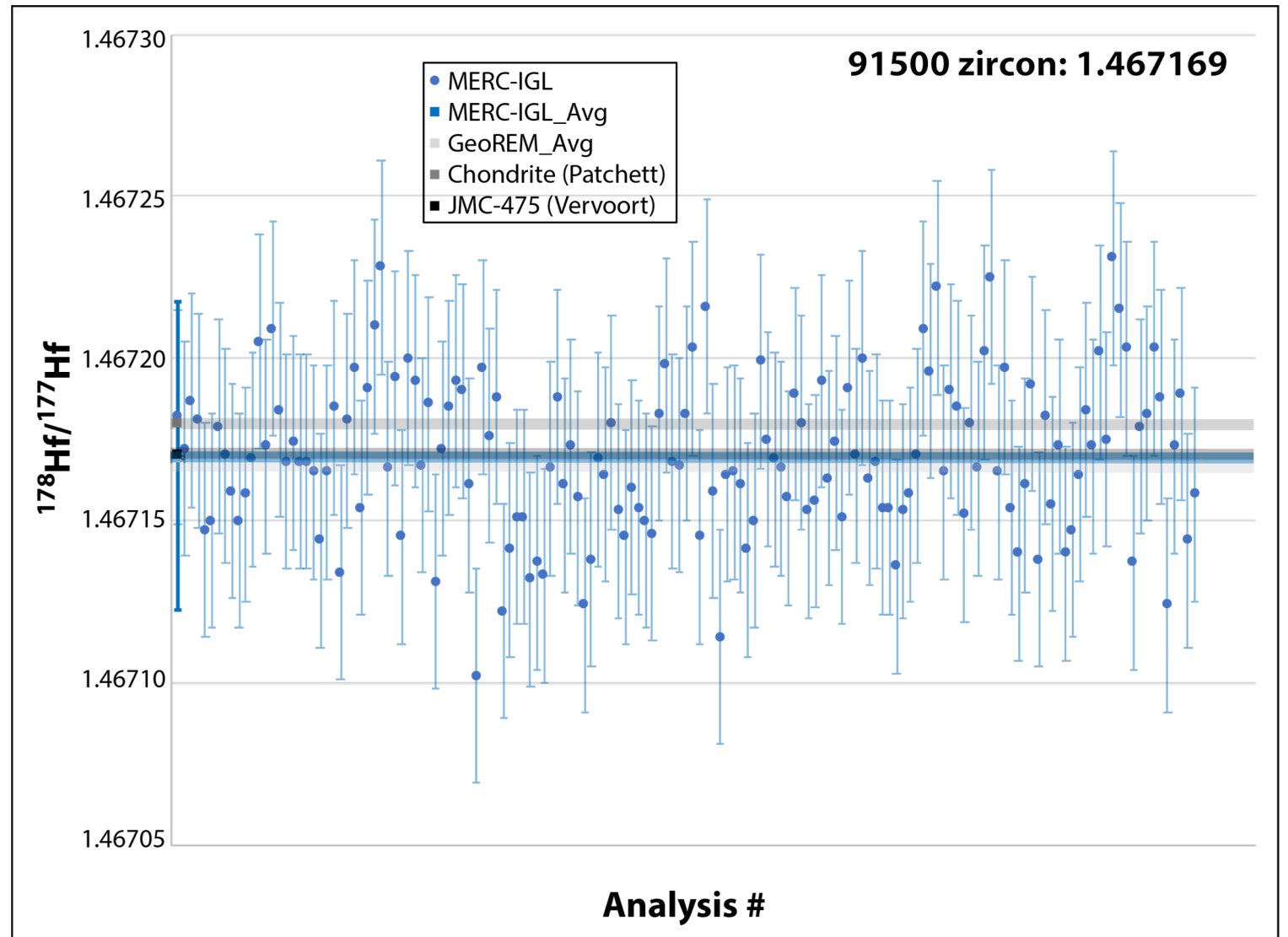


$^{206}\text{Pb}/^{238}\text{U}$  ratios for Maniitsoq zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM)

# LA-ICPMS analytical performance

Hf isotope analysis

- Precision & reproducibility

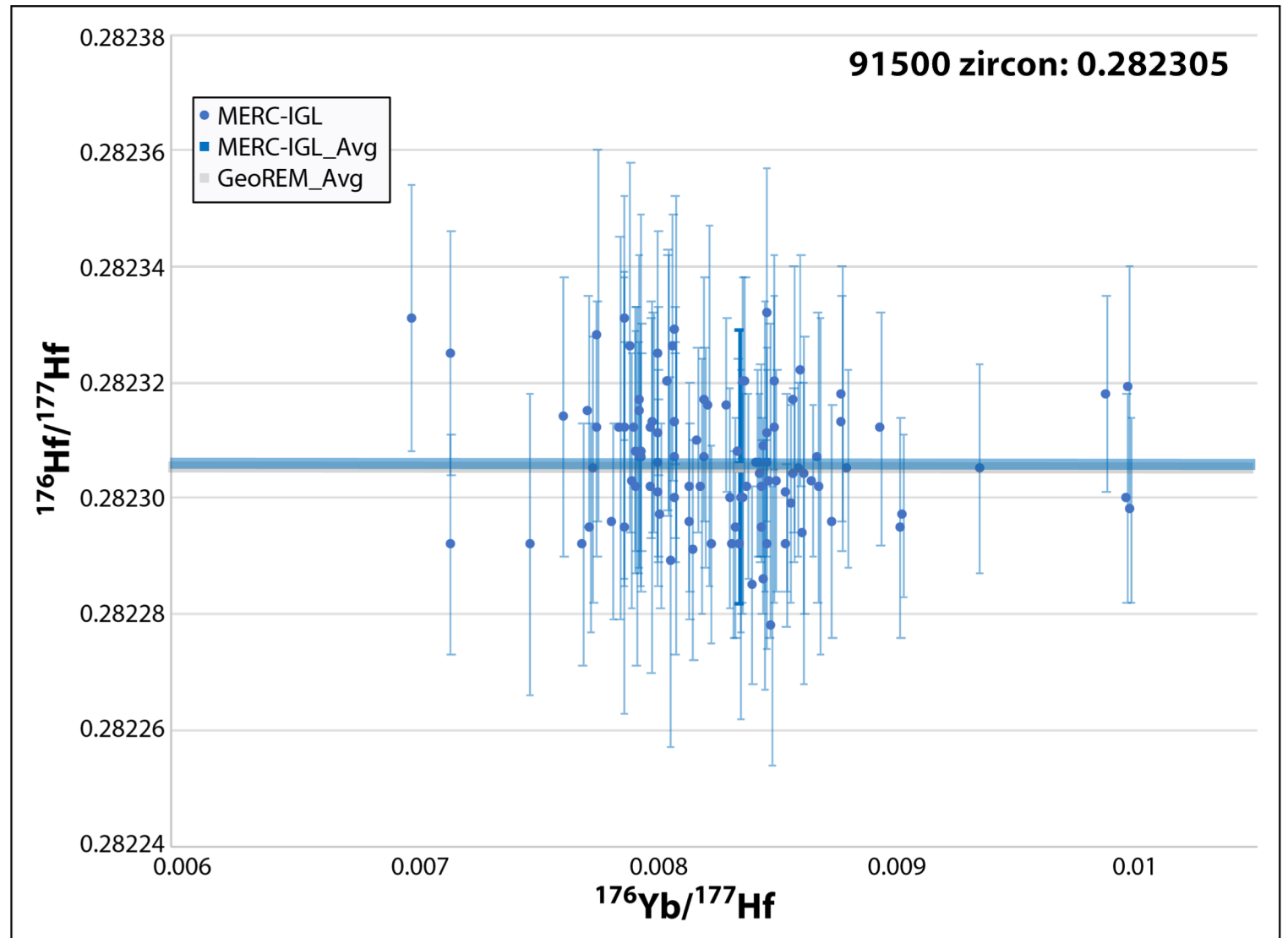


$^{178}\text{Hf}/^{177}\text{Hf}$  ratios for 91500 zircon analyzed by single stream (Hf isotopes only) between Aug 2018 present (normalized to Plesovice zircon PRM)

# LA-ICPMS analytical performance

Hf isotope analysis

- Precision & reproducibility

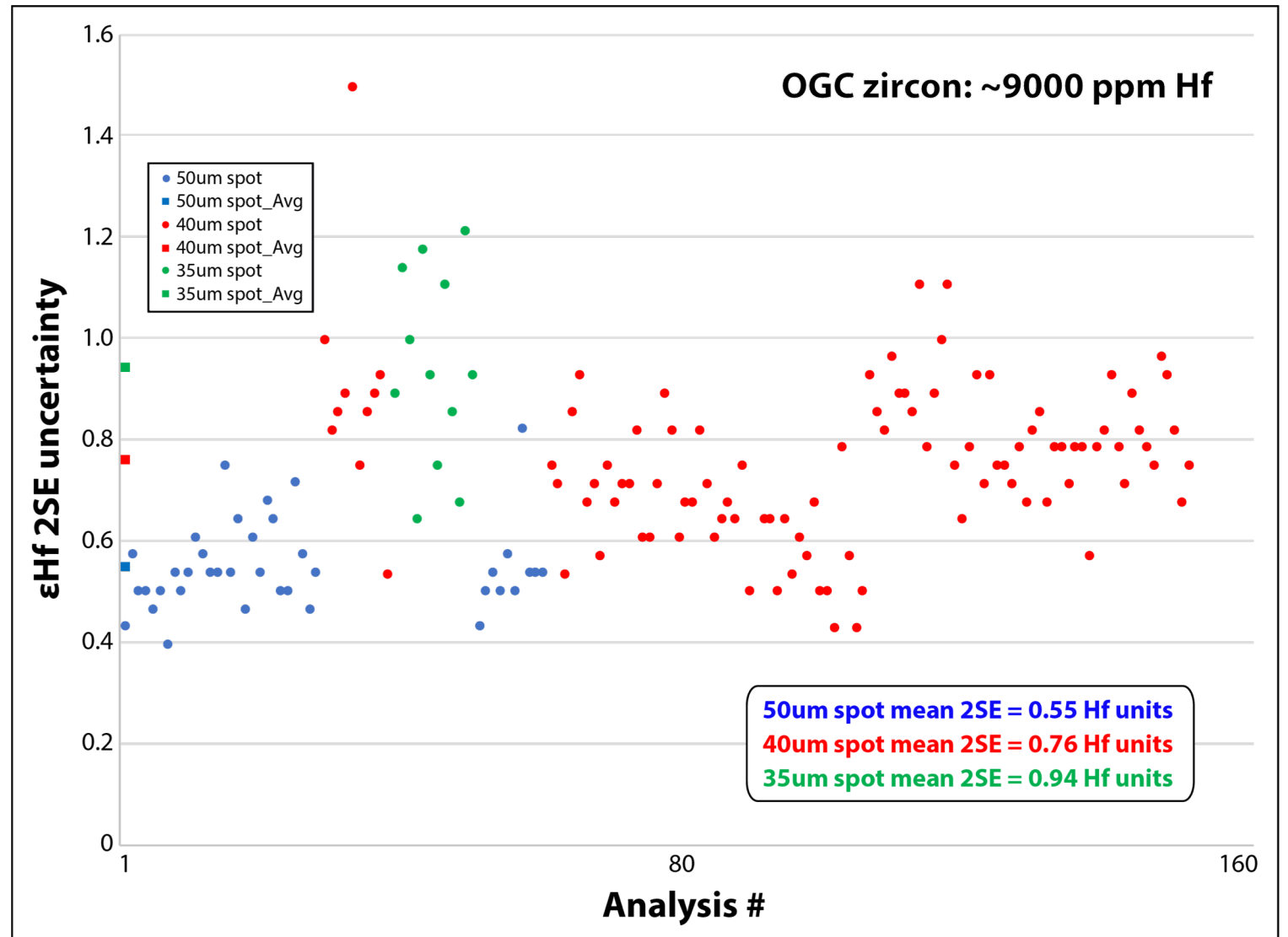


$^{178}\text{Hf}/^{177}\text{Hf}$  ratios for 91500 zircon analyzed by single stream (Hf isotopes only) between Aug 2018 present (normalized to Plesovice zircon PRM)

# LA-ICPMS analytical performance

Hf isotope analysis

- Precision & reproducibility



$\epsilon$ Hf uncertainty values for OGC zircon (3465 Ma) analyzed by single stream with a range of spot sizes between Aug 2018 present (normalized to Plesovice zircon PRM)

# LA-ICPMS analytical performance

Laser Ablation Split Stream  
capabilities

- Analyzing different isotopic systems or elemental groups simultaneously on different ICPMS
- U-Pb + TE
- Hf + U-Pb
- Hf + U-Pb + TE
- Sm-Nd, Rb-Sr, metal isotopes



*Solid rock/mineral sample*



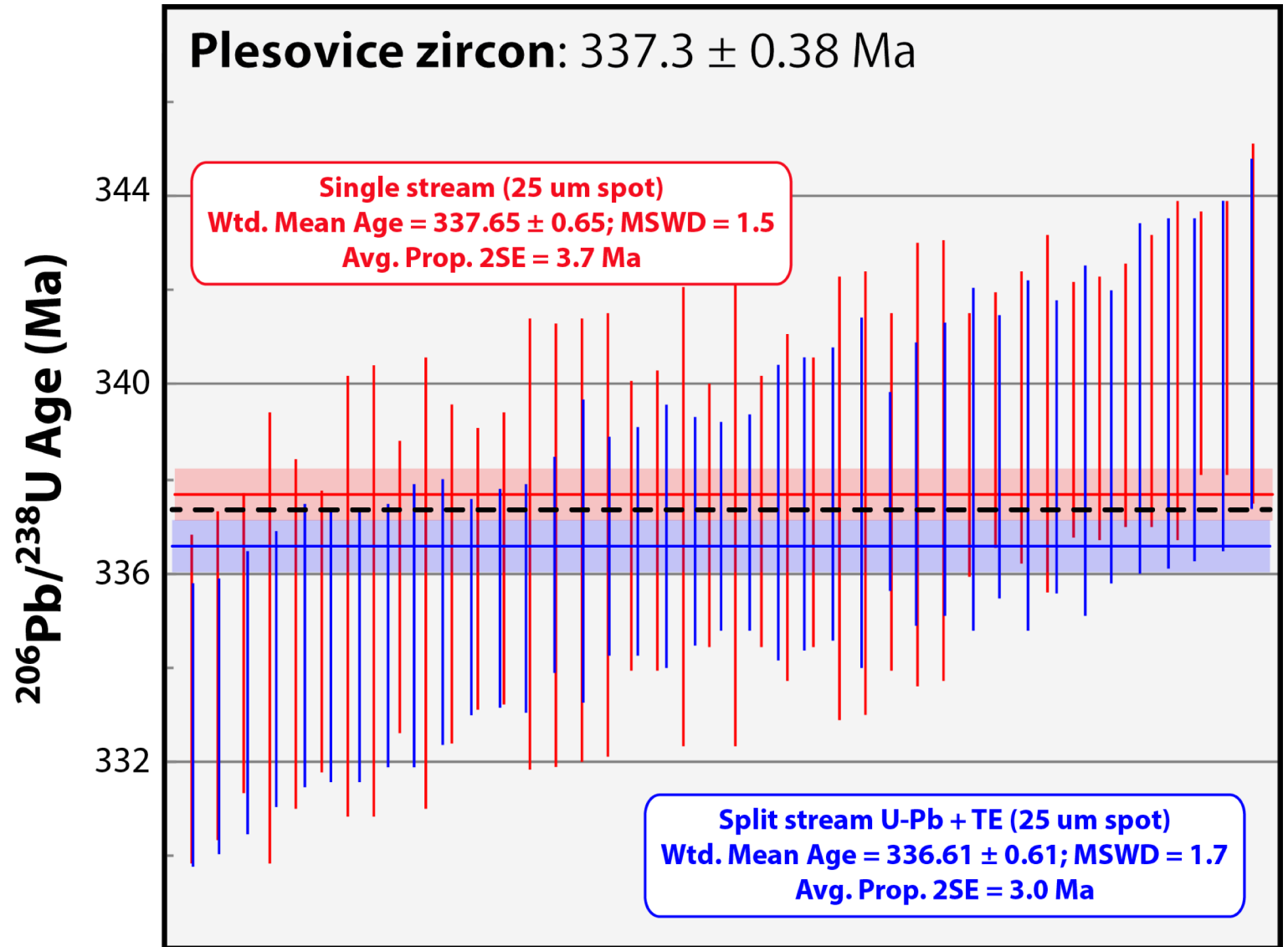
*Ablated aerosol  
transported in  
nylon tubing*



# LA-ICPMS analytical performance

U-Pb geochronology

- Precision & reproducibility
- Single stream vs. LASS



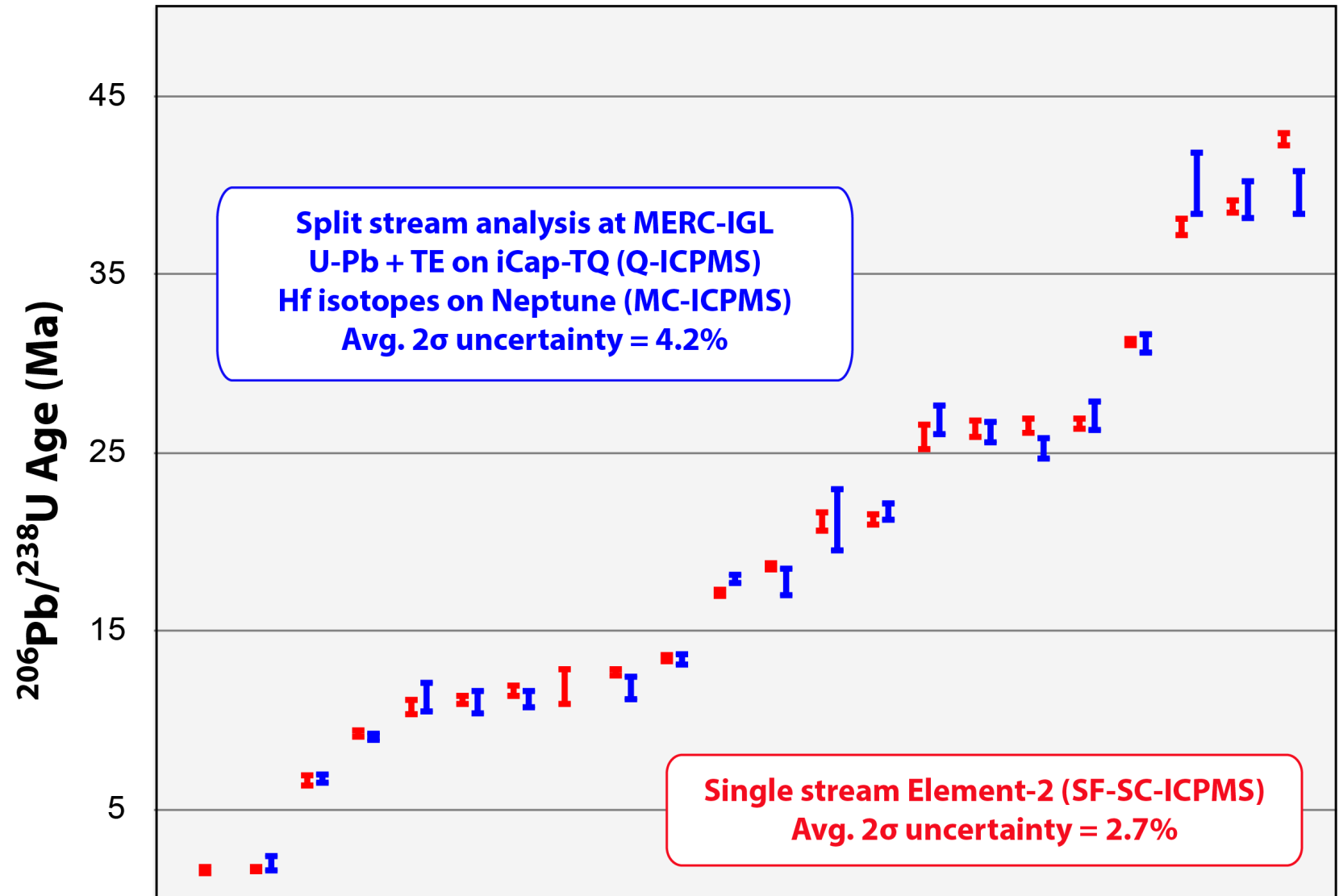
$^{206}\text{Pb}/^{238}\text{U}$  dates for Plesovice zircon analyzed by single stream (U-Pb only) and split stream (U-Pb+TE; LASS) between Aug-Oct 2018 (normalized to 91500 zircon PRM)



# LA-ICPMS analytical performance

## U-Pb+TE+Hf isotope analysis

- Precision & reproducibility
- Single stream vs. LASS
- Hf (Neptune) + U-Pb & TE (iCap)

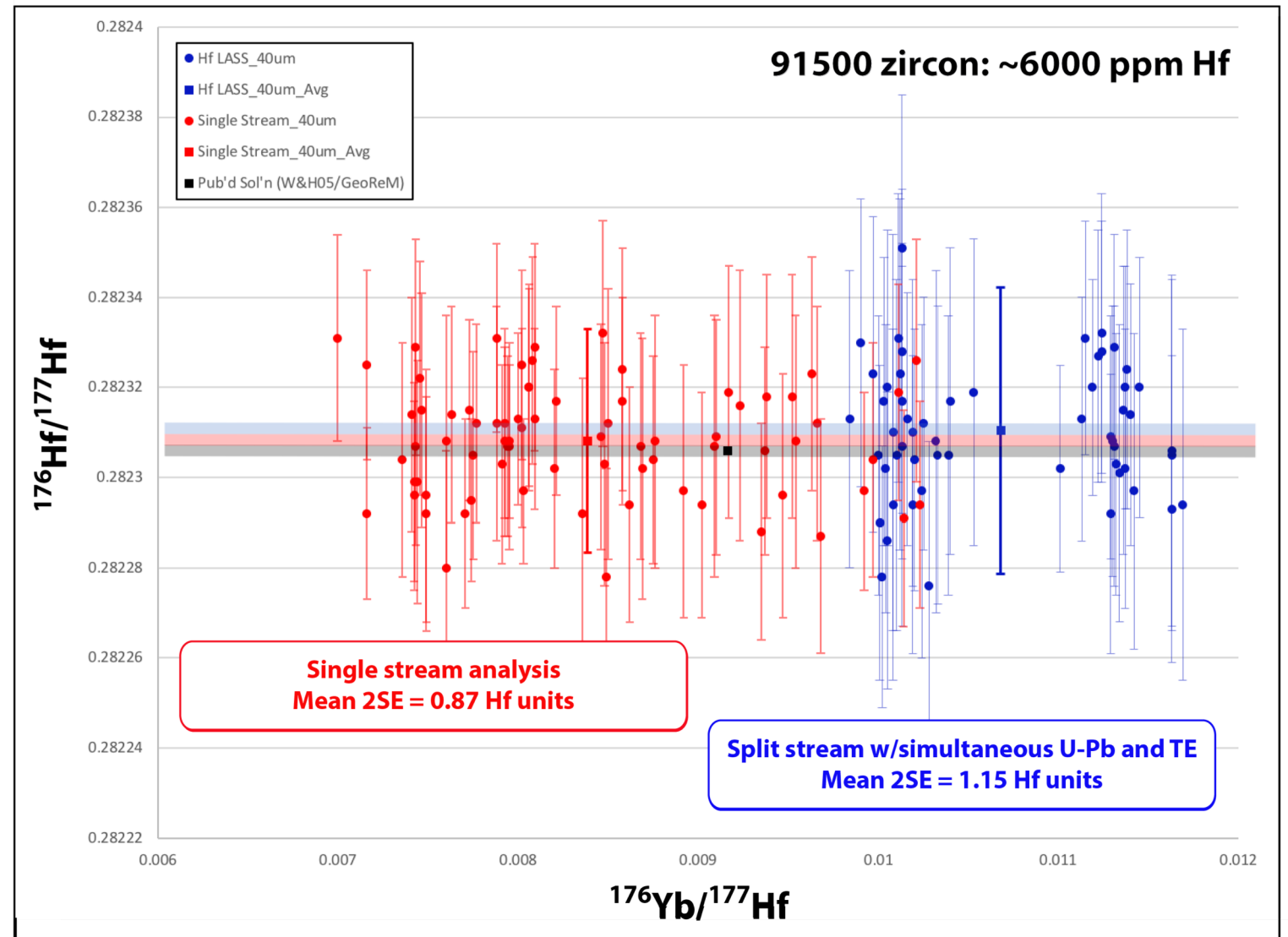


$^{206}\text{Pb}/^{238}\text{U}$  dates for zircon previously dated by single stream analysis on an Element-2 SF-SC-ICP (in red) and split stream analysis with simultaneous TE+Hf (U-Pb isotopes with TE on iCap quadrupole ICPMS)

# LA-ICPMS analytical performance

U-Pb+TE+Hf isotope analysis

- Precision & reproducibility
- Single stream vs. LASS
- Hf (Neptune) + U-Pb & TE (iCap)

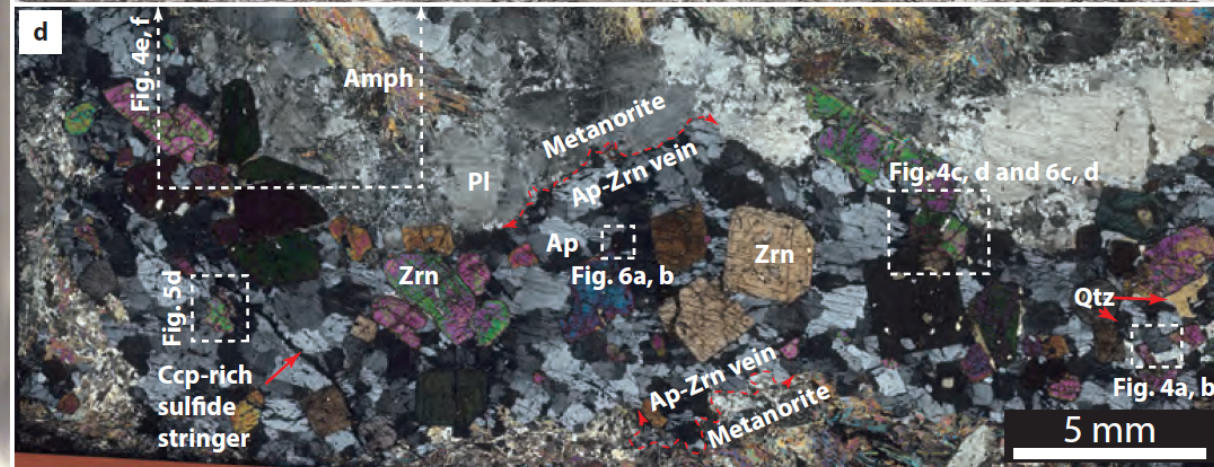
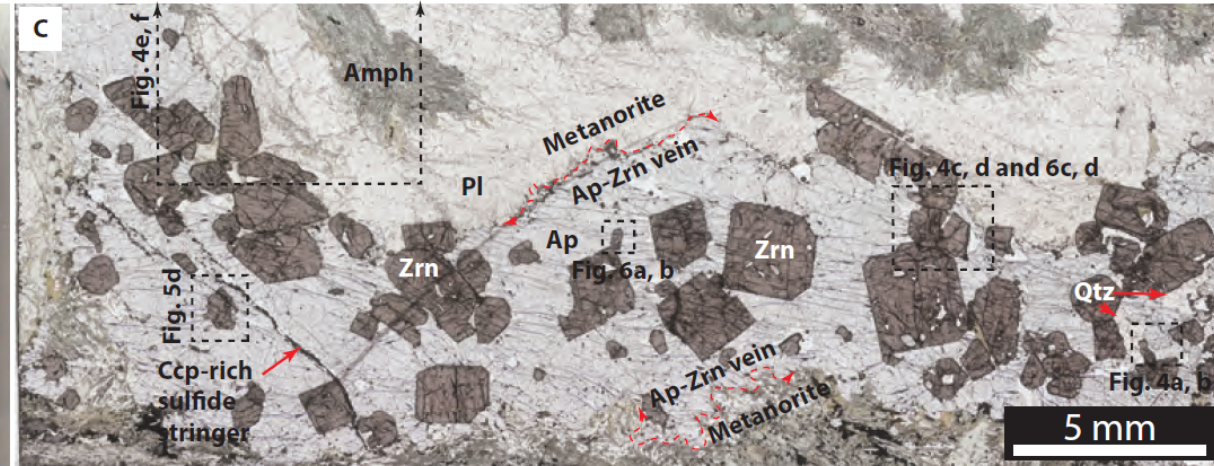
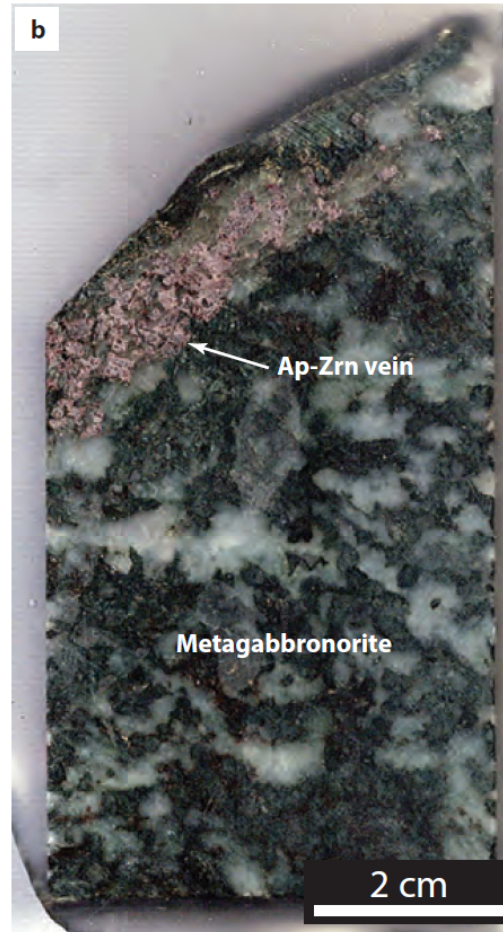


$^{176}\text{Hf}/^{177}\text{Hf}$  ratios for 91500 zircon analyzed by single stream (Hf isotopes only) and split stream (Hf+U-Pb+TE; LASS) between Aug 2018 present (normalized to Plesovice zircon PRM)

# Manitsoq zircon

A potential Archean zircon U-Pb and Hf isotope reference material developed in the MERC-IGL and JSL

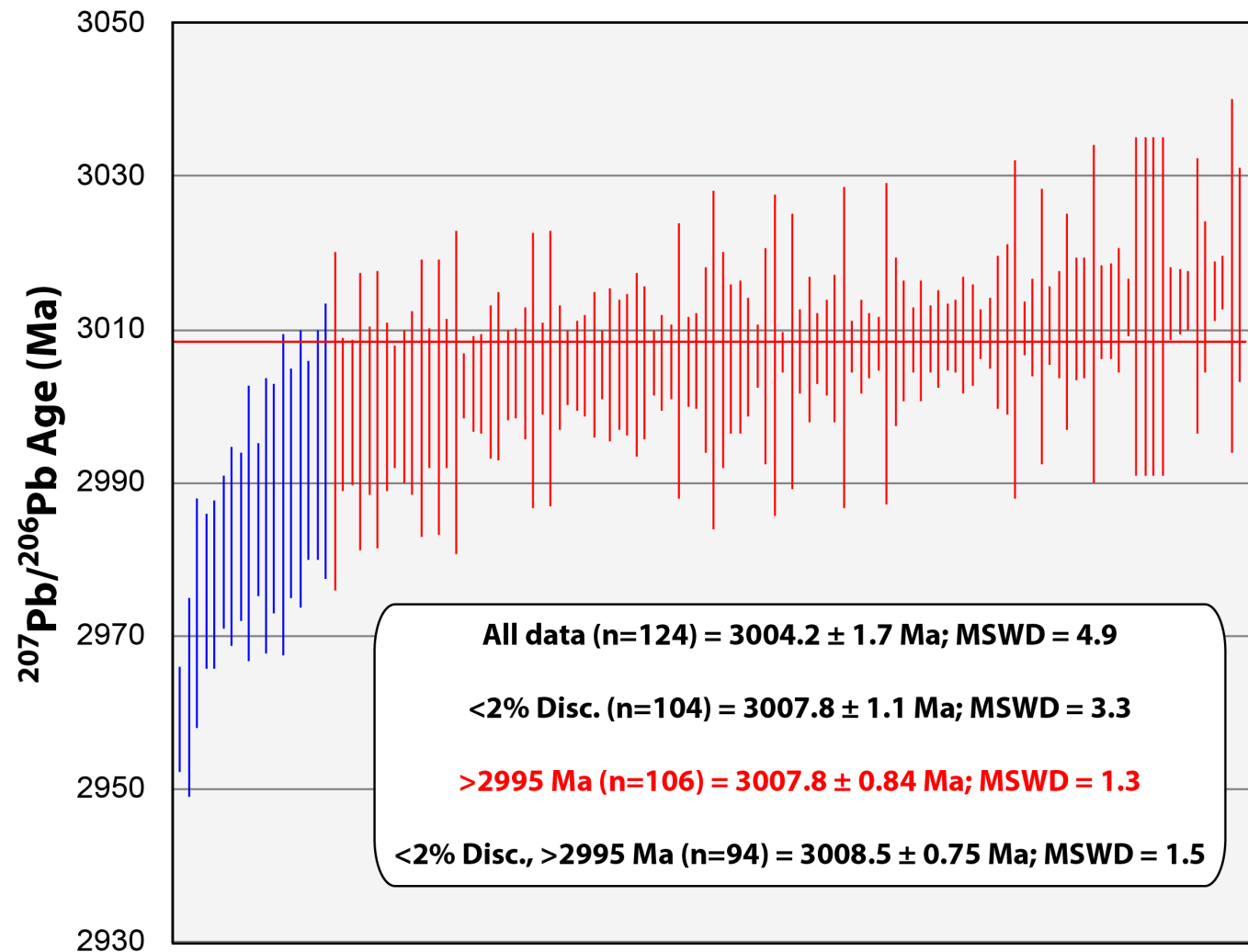
- Large, euhedral crystals in Plag+Qtz+Ap vein hosted by gabbro norite gneiss
- Norites post-kinematic, have yielded zircon U-Pb dates of  $3016 \pm 23$ ,  $3006 \pm 13$ , and  $2976 \pm 13$  (SHRIMP, Garde et al., 2000).



# Maniitsoq zircon

A potential Archean zircon  
U-Pb and Hf isotope  
reference material developed  
in the MERC-IGL and JSL

- Recently obtained TIMS U-Pb ratios from JSL
- Slight normal discordance, reflecting minor Pb-loss

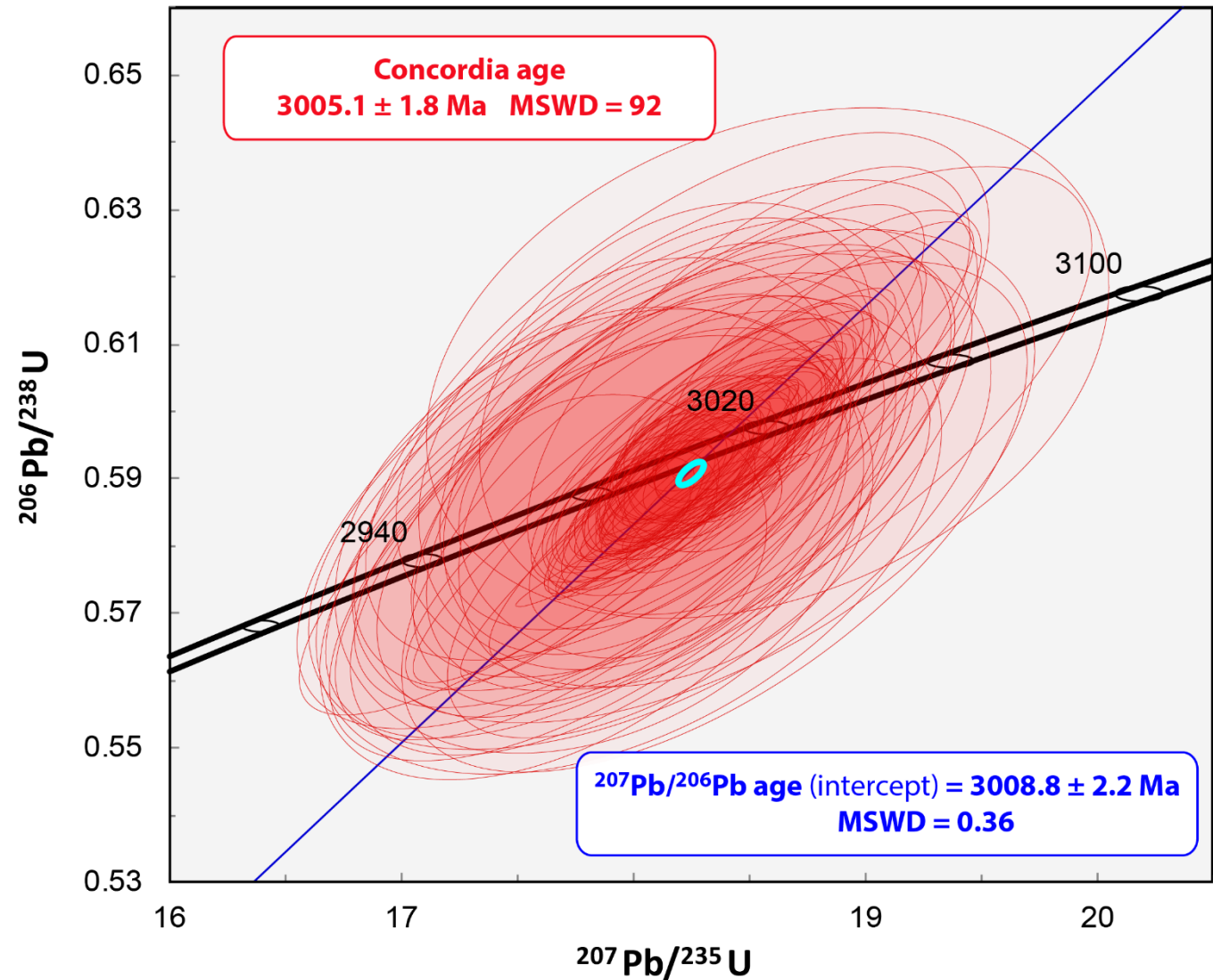


$^{207}\text{Pb}/^{206}\text{Pb}$  weighted mean age plot for Maniitsoq zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM).  $^{207}\text{Pb}/^{206}\text{Pb}$  weighted mean age for 106 of 124 analyses with age >2995 shown in red

# Maniitsoq zircon

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U-Pb and Hf isotope  
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- Recently obtained TIMS U-Pb ratios from JSL
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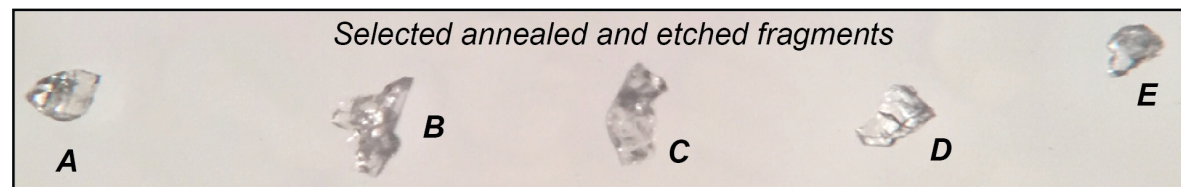
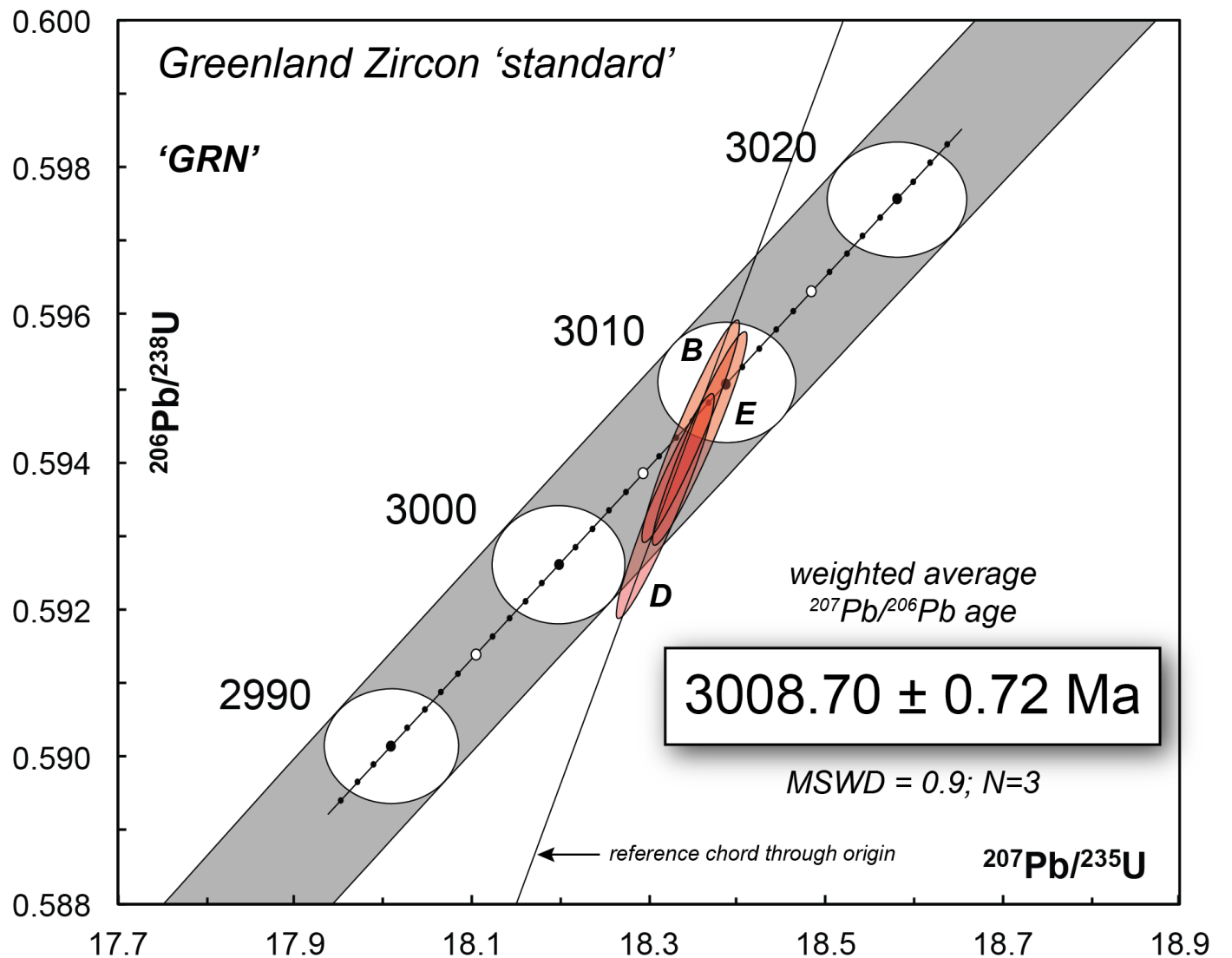


Concordia plot for Maniitsoq zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM). 94 of 124 analyses plotted, with <2% discordance and  $^{207}\text{Pb}/^{206}\text{Pb}$  age >2995

# Manitsoq zircon

A potential Archean zircon U-Pb and Hf isotope reference material developed in the MERC-IGL and JSL

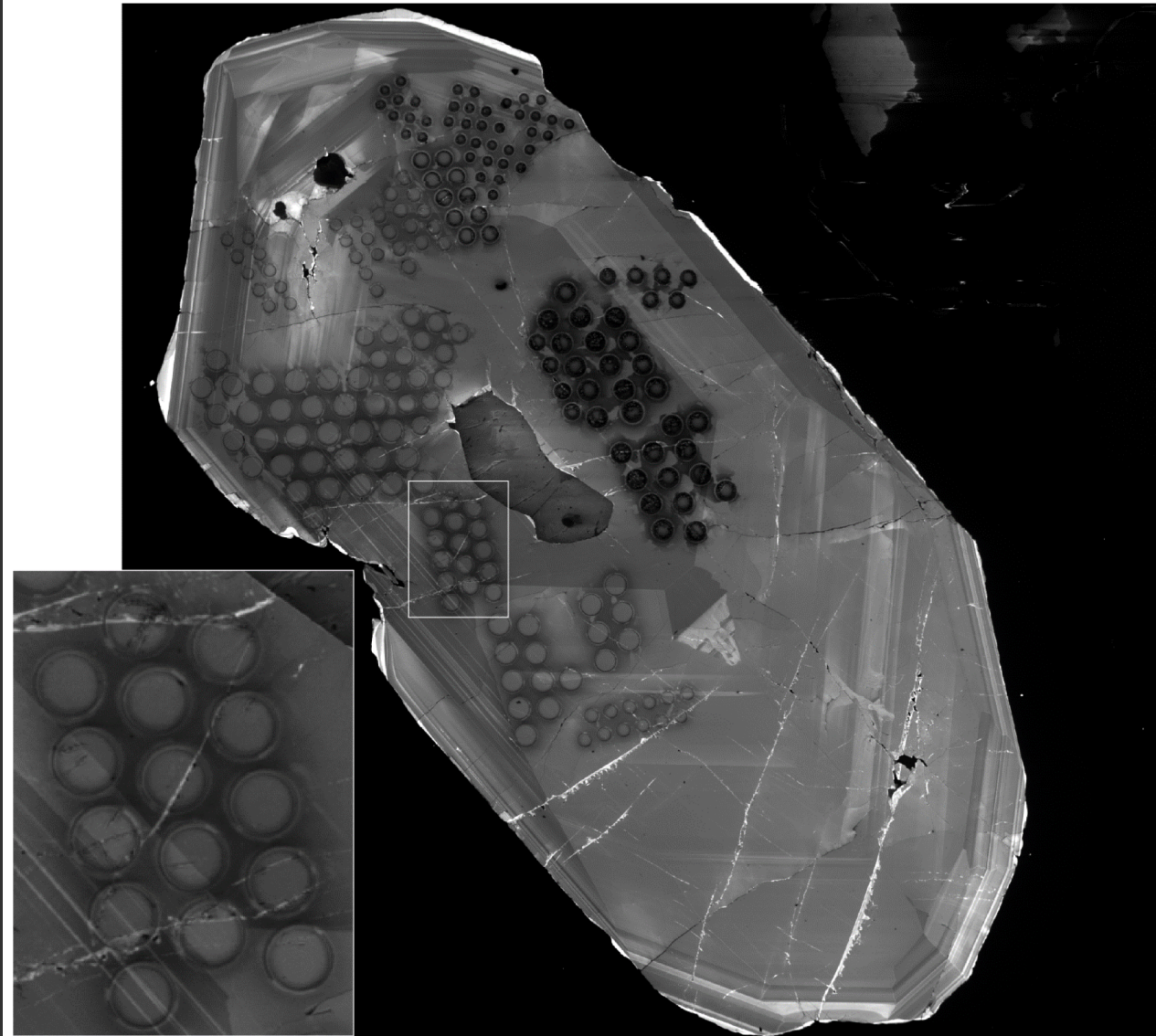
- Recently obtained TIMS U-Pb ratios from JSL
- Slight normal discordance, reflecting minor Pb-loss



# Manitsoq zircon

A potential Archean zircon  
U-Pb and Hf isotope  
reference material developed  
in the MERC-IGL and JSL

- Recently obtained TIMS U-Pb ratios from JSL
- Slight normal discordance, reflecting minor Pb-loss



SEM HV: 10.0 kV

View field: 2.34 mm

SEM MAG: 296 x

WD: 15.05 mm

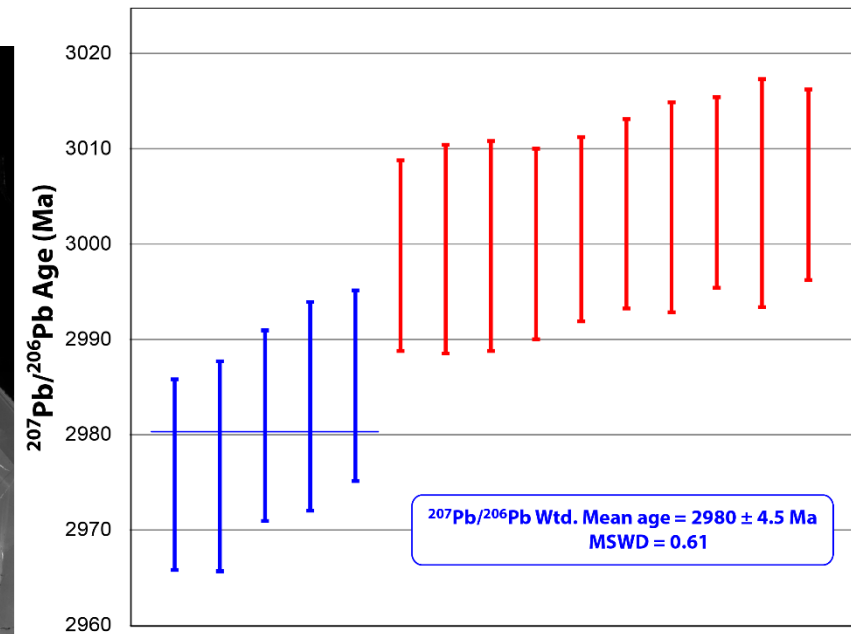
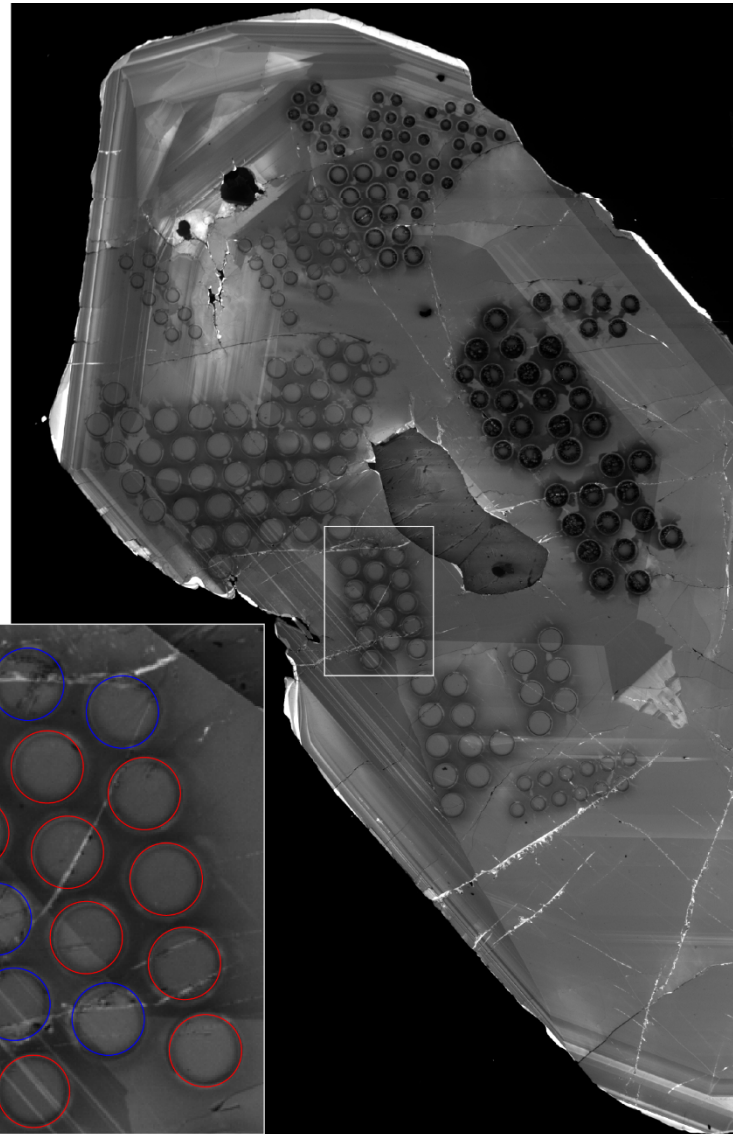
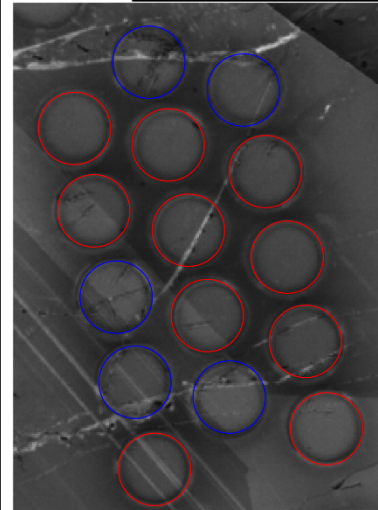
Det: CL, SE

Date(m/d/y): 02/07/19

# Manitsoq zircon

A potential Archean zircon  
U-Pb and Hf isotope  
reference material developed  
in the MERC-IGL and JSL

- Recently obtained TIMS U-Pb ratios from JSL
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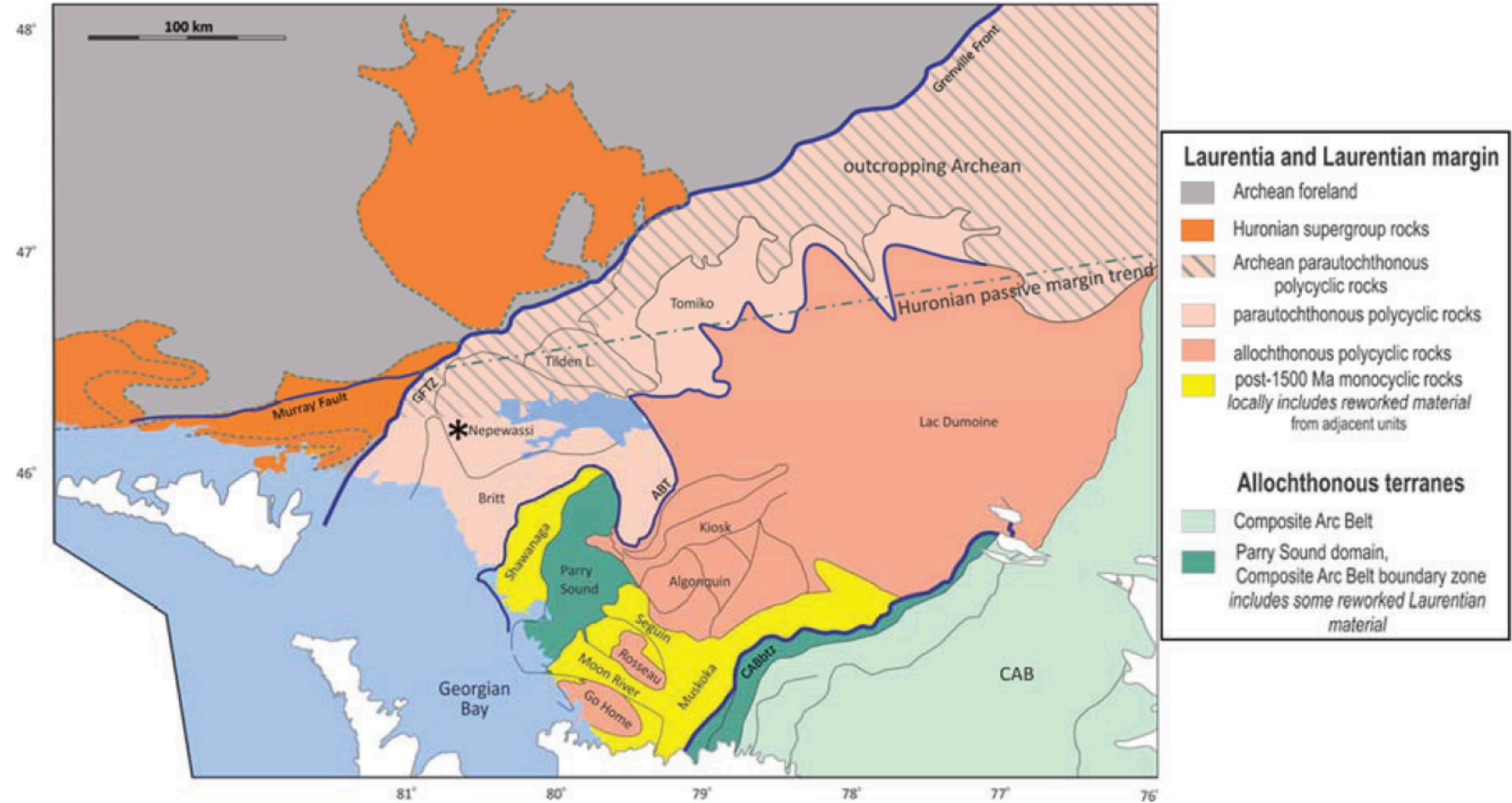


SEM HV: 10.0 kV	WD: 15.05 mm
View field: 2.34 mm	Det: CL, SE
SEM MAG: 296 x	Date(m/d/y): 02/07/19



# Detrital zircon Hf isotope analysis

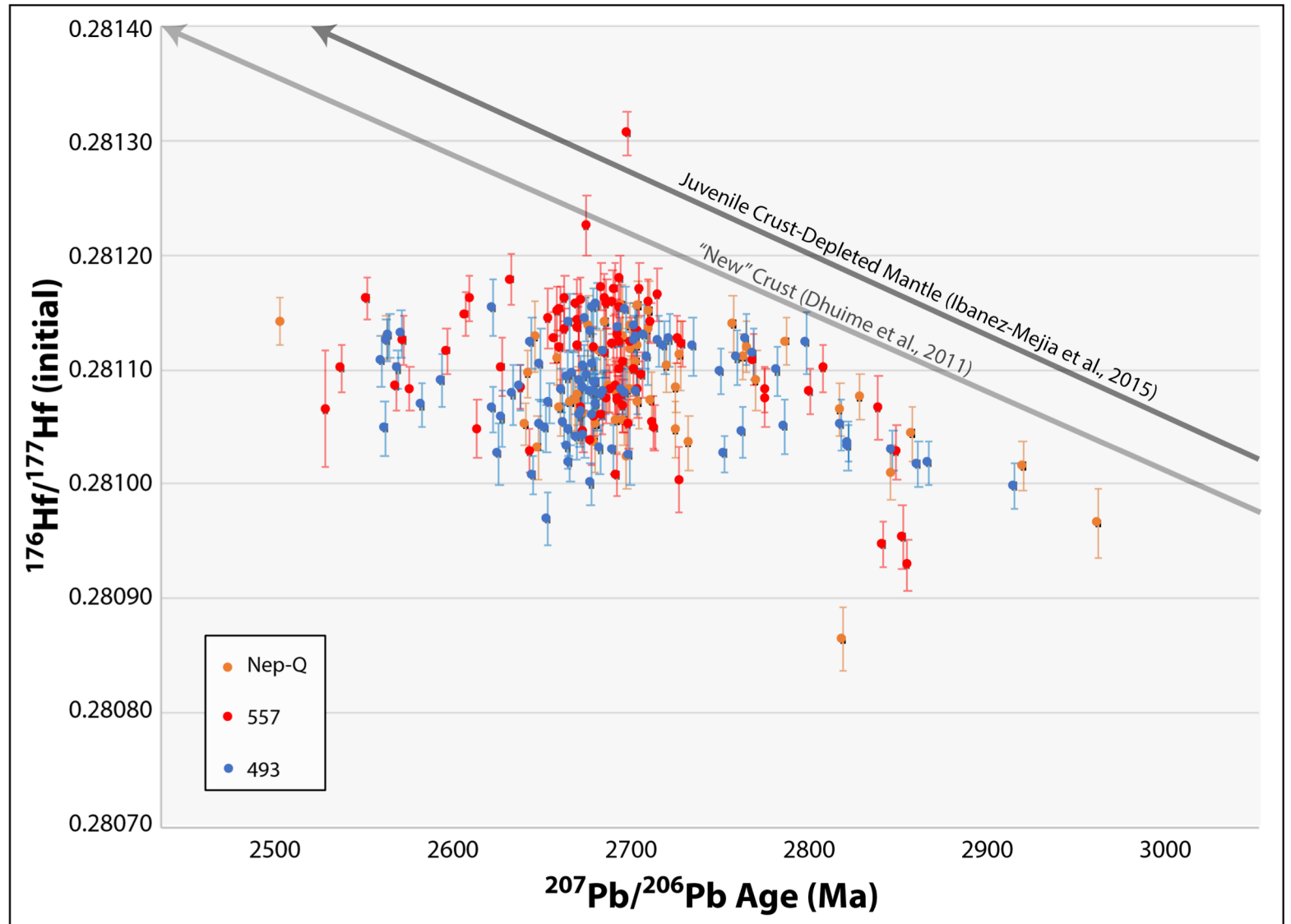
- A potential tool for recognizing tectonic/magmatic styles and isotopic fingerprints
- Case study from the Grenville Province



Geological map of the Grenville Province, southern Ontario. DZ samples come from Nepewassi Domain, in the Parautochthonous belt south of GFTZ

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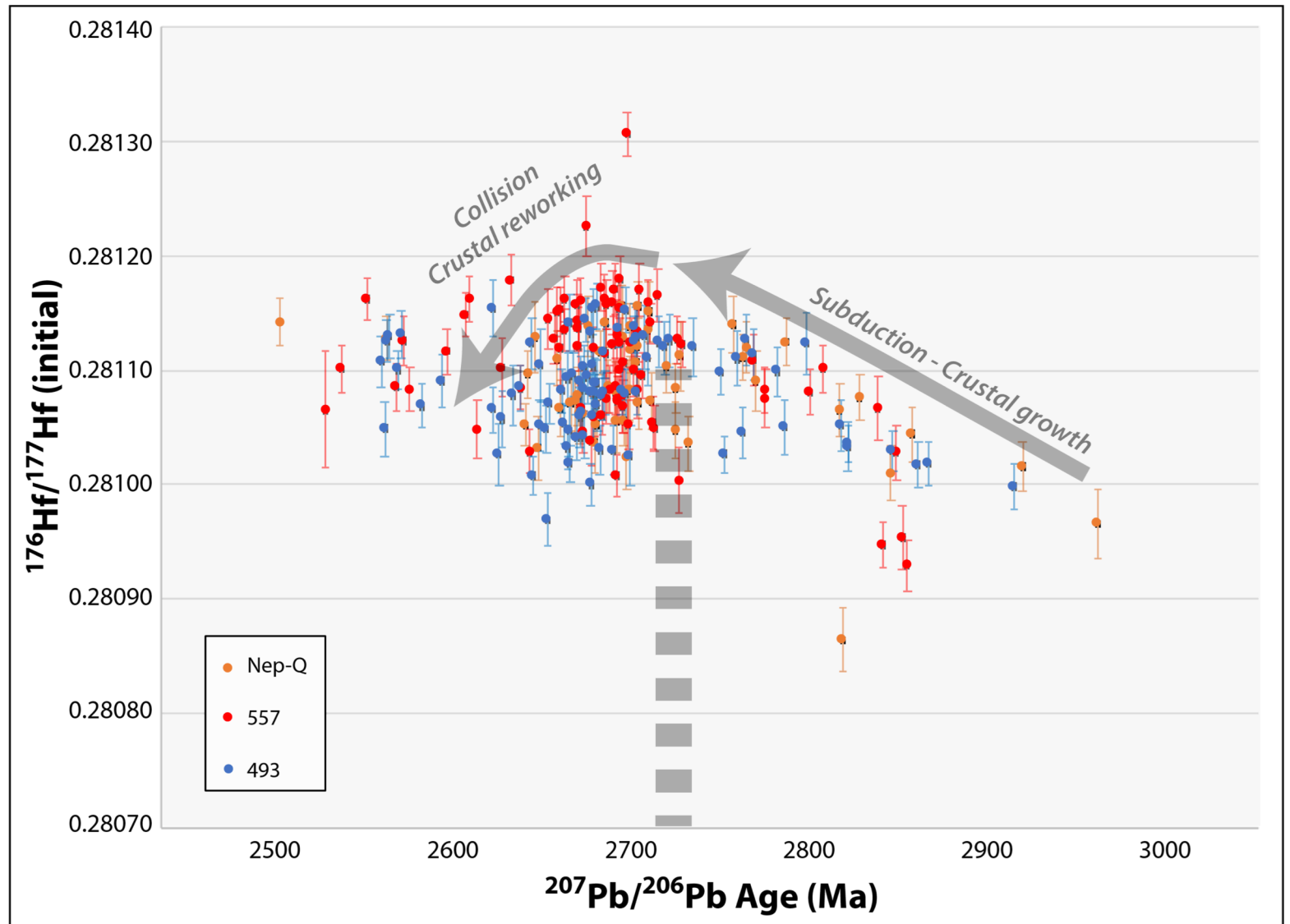
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# MERC-IGL: Student activity & Method development

- LU PhD student assistants working on analytical method development associated

**Xuyang Meng:** Monazite, apatite, and allanite U-Pb geochronology and Nd isotope analysis

**Nicolas Estrada:** Titanite U-Pb geochronology and Nd isotope analysis and SEM-EDS applications

**Ijaz Ahmad:** PGE analysis of sulfide minerals and signal deconvolution for quantitative analysis of small inclusions

*Please contact me to discuss your analytical needs for the Metal Earth project*

*jmarsh@laurentian.ca*

*Thank you.*

SEM HV: 10.0 kV	WD: 17.20 mm	VEGA3 TESCAN
View field: 361 µm	Det: CL	100 µm
SEM MAG: 576 x	Date(m/d/y): 02/22/19	MERC-IGL Laurentian Univ.



A new Canadian research initiative funded by Canada First Research Excellence Fund.



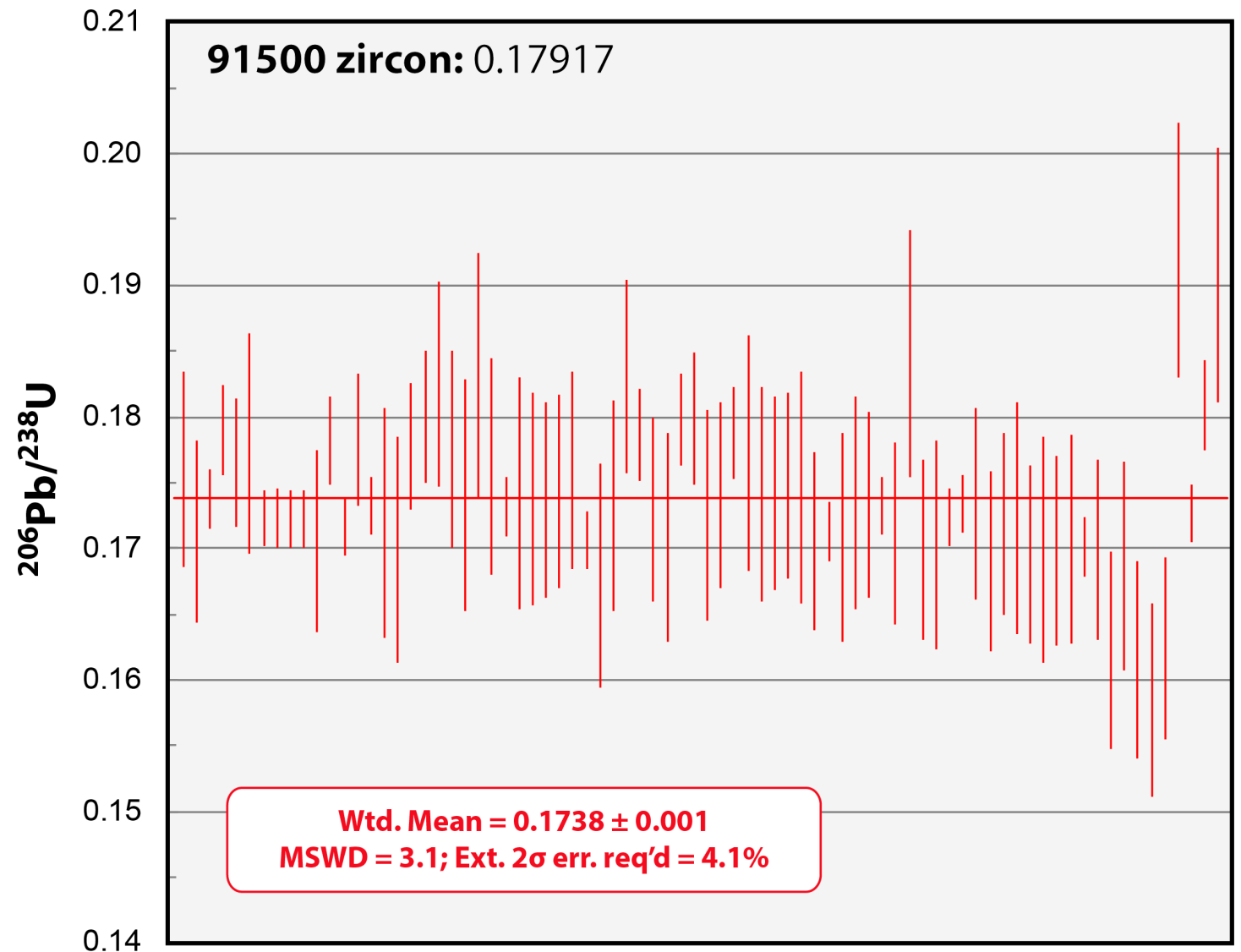
Canada



# LA-ICPMS analytical performance

## U-Pb geochronology

- Precision & reproducibility
- $2\sigma$  RSD  $^{206}\text{Pb}/^{238}\text{U} = 6.31\%$ ;  
 $^{207}\text{Pb}/^{206}\text{Pb} = 2.74\%$



$^{206}\text{Pb}/^{238}\text{U}$  ratios for 91500 zircon analyzed as a verification RM during U-Pb/TE LASS runs from Aug 2018 to present (normalized to OGC zircon PRM)