Nu Plasma II - Collector Configuration
U-Th-Pb mass array

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<th>Analysis Mass table - C:\Nu Plasma II\Analyses\In_situ_U_Pb_v1.nrf</th>
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### Mass Separation: 1

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<td>Magnet delay time [s]: 1</td>
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- **238U**
- **232Th**
- **235U**
- **208Pb**
- **207Pb**
- **206Pb**
- **204Pb**
- **204Hg**
- **202Hg**
Peak Alignment/Coincidence

- $^{238}\text{U}$
- $^{232}\text{Th}$
- $^{208}\text{Pb}$
- $^{207}\text{Pb}$
- $^{206}\text{Pb}$
- $^{204}\text{Pb}$
- $^{204}\text{Hg}$
- $^{202}\text{Hg}$
Detectors

COLLECTOR HOUSING (Top plate off)

Ion counter entry slits  Faraday collectors

Faraday collector
Ion counters (5 of them on NuPlasmaII)—discrete dynode electron multipliers
Simonetti et al. (2008)
Faraday – ion counter calibration

Simonetti et al. (2008)
• Use Certified Reference Material (CRM-112a) uranium (natural) isotope standard or any other appropriate standard material

• Measure ratios, e.g., $^{238}\text{U}/^{234}\text{U}$ or $^{238}\text{U}/^{235}\text{U}$ at first involving mixed Faraday-ion counter combinations, and then ion counter-ion counter combinations
### Analysis Mass Table - C: \ Nu Plasma II \ Analyses \ U_Gain.nrf

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### Mass Separation: 1.

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- Measurements per block: 10
- Number of blocks: 1
- Magnet delay time (s): 2

**Options:**
- Centre each Block
- Zero each cycle
- Sit on set (Delta M)
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**Mass Separation:** 1.

**Settings:**
- Measurements per block: 20
- Number of blocks: 2
- Magnet delay time [s]: 2

**Options:**
- Centre each Block
- Zero each cycle
- Sit on set (Delta M)
### Mass Separation: 1.

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### Options:

- Centre each Block
- Zero each cycle
- Sit on set (Delta M)

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- IC2 232.50
- Integ Time

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- L6
- IC0
- IC1
- L2
- L3
- A
- L1
- H1
- H3
- H4
- H5
- H6
- H7
- H8
- H9

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Ion counter calibration exercise

- You are assigned the responsibility to calibrate the ion counter efficiencies on your MC-ICP-MS instrument.
- You are given *three possible isotopic standard solutions* that could be used to conduct the ion counter efficiency calculations.
- Decide on which element you would use for the calibration and explain your choice, and then demonstrate/outline the measurement sequence.
- The possible three isotope standard solutions are:

Lu: $^{176}\text{Lu}/^{175}\text{Lu} = 0.02656$

Tl: $^{205}\text{Tl}/^{203}\text{Tl} = 2.3781$

In: $^{115}\text{In}/^{113}\text{In} = 22.31$
Data Reduction Protocols–Isotope Ratio Measurements

Antonio Simonetti
University of Notre Dame
Possible Issues with (LA)-MC-ICP-MS Analyses?

- Precision and accuracy on individual isotopic measurements suffer due to matrix effects and isobaric interferences?
- Availability of suitable reference materials, in relation to matrix-matching in studies of mass-dependent isotopic fractionation, and laser-induced isotopic fractionation
- Factors that contribute to the accuracy and precision of in-situ measurements-
  - Interplay related to **sample, laser operating conditions** and **processes in the mass spectrometer**
Not accurate
Not precise

Not accurate
Precise

Accurate
Not precise

Accurate
Precise
Parameters affecting accuracy and precision

Sample
- grain size
- concentration of element/isotopes of interest (major, minor, trace)
- parent-daughter element ratio

Laser operating conditions
- laser wavelength
- frequency
- energy
- spot vs raster

Accuracy
- spot size
- overlap corrections
- matrix effects

Precision
- volume of sample ablated
- laser induced fractionation
- matrix effects
- ablation rate

Mass spectrometry
- sensitivity
- mass bias corrections
- overlap corrections
- background and/or blank
- counting time
- reference materials
- standard/sample bracketing

Pearson et al. (2008)
In-situ Isotope Studies

- Fall into 2 groups:
  - Measurement of radiogenic isotopes for trace elements in common rock-forming minerals; e.g., Sr in clinopyroxene, carbonate, feldspar; Hf in zircon; Pb in feldspar, clinopyroxene
  - Study of mass-dependent isotopic fractionation of elements that are major constituents in minerals of interest; e.g., Cu in chalcopyrite, Fe in pyrite
Size of mineral grain and sensitivity of mass spectrometer will dictate the **sampling strategy**.

- **rastering** so as to maximize volume of sample ablated and therefore improve precision (i.e. higher ion signal) and 'homogenize' the sample.

- **stationary** analysis, mimic a microprobe and hence attempt to decipher internal variations combined with trace element data, images, etc.
Instrumental Mass Bias

- Probably is the most important factor affecting the accuracy and external precision.

- Mass bias is isotopic fractionation (i.e. artificial change in isotope ratios) produced by variable transmission of the ion beam in mass spectrometer.

- In the MC-ICP-MS instrument, this occurs primarily in the plasma and interface regions.
For MC-ICP-MS instruments,

- The ‘measured’ isotope ratio > the ‘true’ ratio
- E.g., measured $^{87}\text{Sr}/^{86}\text{Sr}_{\text{sample}} > \text{‘true’} ^{87}\text{Sr}/^{86}\text{Sr}_{\text{sample}}$

For TIMS (thermal ionization mass spectrometers) instruments,

- The ‘measured’ isotope ratio < the ‘true’ ratio
- E.g., measured $^{87}\text{Sr}/^{86}\text{Sr}_{\text{sample}} < \text{true} ^{87}\text{Sr}/^{86}\text{Sr}_{\text{sample}}$
In-situ analyses are also affected by the “matrix” of the samples, which may produce isotopic interferences (isobaric and molecular overlaps) – these also need to be corrected (if possible)!

Isobaric (equivalent atomic mass) interferences in radiogenic isotope systems include:

- $^{87}\text{Rb} \rightarrow ^{87}\text{Sr}$
- $^{144}\text{Sm} \rightarrow ^{144}\text{Nd}$
- $^{176}\text{Lu} \rightarrow ^{176}\text{Hf}$
For example, matrix-based molecular interferences include:

- \( ^{40}\text{Ca} + ^{31}\text{P} + ^{16}\text{O} = \text{mass 87} = ^{87}\text{Sr} \)

The principal inorganic component of enamel is Hydroxyapatite - \( 3\text{Ca}_3(\text{PO}_4)_2.\text{CaX} \) [where X can represent a mixture of F, Cl, CO\(_2\), OH]

In-situ LA-MC-ICP-MS analyses of fossilized teeth from human remains for their Sr isotope composition (Simonetti et al., 2008)

Tracing migration patterns for ancient civilizations
Simonetti et al. (2008, Archaeometry)
Simonetti et al. (2008, Archaeometry)
Magnitude (i.e., deviation of measured ratio compared to the ‘true’ value) of mass bias for MC-ICP-MS instrument is larger than that associated with TIMS (thermal ionization mass spectrometry).

However, the same fractionation laws are applied to correct for instrumental mass bias.
Previous studies have shown that instrumental mass bias can be corrected using a "generalized power law"

\[ R_{true} = R_{meas} \cdot f^{(M_2 - M_1)} \]

- \( R_{true} \) = true isotope ratio of the two isotopes of mass \( M_1 \) and \( M_2 \) (\( M_2/M_1 \))
- \( R_{meas} \) = is the isotope ratio (\( M_2/M_1 \)) measured by the mass spectrometer
- \( f \) = mass fractionation coefficient
Equation can be rewritten into “Exponential Law” form:

\[ R_{true} = R_{meas} \cdot \left( \frac{M_2}{M_1} \right)^f \]
Correction of instrumental mass bias is best achieved by “internal normalization”

Mass fractionation coefficient ($f$) can be determined using a pair of stable isotopes with a known or “true” isotopic ratio

E.g.,
- $^{86}\text{Sr}/^{88}\text{Sr} = 0.1194$
- $^{146}\text{Nd}/^{144}\text{Nd} = 0.7219$
- $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$
- $^{205}\text{Tl}/^{203}\text{Tl} = 2.3871$
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*Note: The above table represents a collector configuration for Nu Plasma II.*
$R_{true} = R_{meas} \cdot (M_2 / M_1)^f$

Pearson et al. (2008)
**Fig. 7-3.** Plot of $\ln(^{178}\text{Hf}/^{177}\text{Hf})$ vs. $\ln(^{179}\text{Hf}/^{177}\text{Hf})$ for the JMC475 Hf solution. The data points are from 132 analyses over a period of 5 years and show the long-term robustness of the instrumental mass fractionation on the Nu Plasma.
Linear relationship on log-log plots also holds for when isotope ratios of different elements are plotted, which formed the basis for the ‘external’ or ‘doped’ correction procedure.

This technique has been successfully applied to a number of applications as shown by previous investigations:

- Cu-Zn (e.g., Maréchal et al., 1999)
- Pb-Tl (e.g., Belshaw et al., 1998; Woodhead, 2002)
Fig. 7-4. Plot of \( \ln(\frac{^{208}\text{Pb}}{^{206}\text{Pb}}) \) vs. \( \ln(\frac{^{205}\text{Tl}}{^{203}\text{Tl}}) \) of a mixed Pb (SRM981) and Tl (SRM997) solution. The data points are from 55 analyses over a 3-year period. The slope of the line (1.001±0.018) is slightly higher than the theoretical value derived from the mass relationship of the exponential (0.9855).
Fig. 7-5. Plot of $\ln(\frac{^{173}Yb}{^{172}Yb})$ vs. $\ln(\frac{^{179}Hf}{^{177}Hf})$ of a mixed Hf (JMC475) and Yb solution showing the similarity of mass bias behavior between three Nu Plasma MC–ICP–MS (Nu005 with and without the Big80 pump, Nu034 and Nu factory. 

Pearson et al. (2008)
Fig. 7-6. Plot of exponential mass bias coefficient $f$ for Yb ($^{173}\text{Yb}/^{172}\text{Yb}$) vs Hf ($^{179}\text{Hf}/^{177}\text{Hf}$). Data as in Fig 7-5. The linear array gives a constant relationship between $f_{\text{Hf}}$ and $f_{\text{Yb}}$, but because the slope is not equal to one it indicates that the mass fractionations of Yb and Hf are not the same.