

# Analysis of Pb/U Ratios in Zircons Using Laser Ablation Coupled with XSeries<sup>II</sup> ICP-MS

## Key Words

- ICP-MS
- Laser Ablation
- Pb/U ratios
- 193nm Solid State Laser
- Zircons

## Introduction:

For U-Pb dating of Zircons  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{207}\text{Pb}/^{235}\text{U}$  isotope ratios are typically measured on multiple grain aliquots or single crystals to assess concordance and to study the evolution of igneous, metamorphic and sedimentary rocks. The high accuracy with which U decay constants are known today has established this technique as the benchmark for provision of absolute age control and geochronology of crustal rocks on the geological time scale.

Thermal Ionisation Mass Spectrometry (TIMS) instrumentation has traditionally been used for high precision measurements of Pb/U isotope ratios in zircon populations. However, the major drawback with this technique is the length of time required for a single sample analysis, which usually extends to a period of several weeks. LA-ICP-MS instrumentation does not compete with TIMS in terms of analytical precision. However, the popularity of this technique is growing considerably as a result of its high flexibility for in-situ analysis, characteristic speed of analysis (i.e. fully automated data acquisition taking only a few minutes for a single zircon), low operating costs and wide availability of instrumentation.

This application note describes coupling of the New Wave Research UP193 Solid State laser ablation system with the XSeries<sup>II</sup> ICP-MS from Thermo Electron Corporation for analysis of two well characterised zircon standards. Standards analyzed include the well characterised 91500 zircon standard (1065Ma), as derived from a single large crystal in syenite collected from Renfrew country, Ontario, and a young Fish Canyon Tuff zircon (28.4Ma), collected from an ash flow deposit in the San Juan Mountains, Colorado.



## Sample Analysis:

Zircons were mounted in a single polished epoxy resin block and were ablated in a He gas flow of  $0.6 \text{ L min}^{-1}$  using the laser ablation parameters shown in Table 1. Resultant sample ablation characteristics for the Fish Canyon Tuff zircon are shown for reference in Figure 1.

Laser Warm-up	60 secs (laser firing with shutter closed)
Spot Size	35 $\mu\text{m}$ (Aperture Imaged)
Frequency	10 Hz
Laser Output	48 %
Laser Pattern	100 $\mu\text{m}$ Line Raster (9 passes at 10 $\mu\text{m}/\text{sec}$ scan)
Irradiance	1.27 $\text{GW}/\text{cm}^2$
Fluence	3.52 $\text{J}/\text{cm}^2$

Table 1: Laser Ablation Parameters

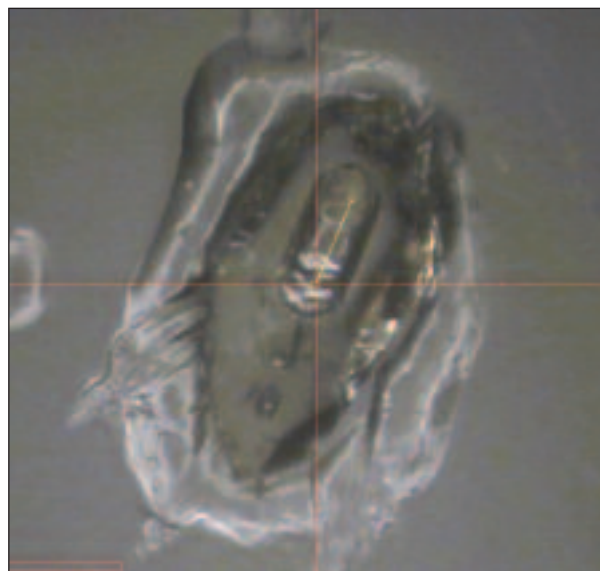


Figure 1: Fish Canyon Tuff zircon following ablation using the 100  $\mu\text{m}$  line raster.

The XSeries<sup>II</sup> ICP-MS was configured with the Xs interface cones and operated under standard hot plasma conditions in the Xs<sup>+</sup> mode. PlasmaLab ICP-MS data acquisition parameters are shown below in Table 2 and analytical signals derived from ablation of the 91500 zircon standard are shown for reference in Figure 2.

Data Acquisition Mode	Profile TRA
Analyte dwell times	10 ms $^{206}\text{Pb}$ , $^{238}\text{U}$
	30 ms $^{207}\text{Pb}$ , $^{235}\text{U}$
Acquisition Time	140 s (including 60 s gas blank)

Table 2: ICP-MS Data Acquisition Parameters

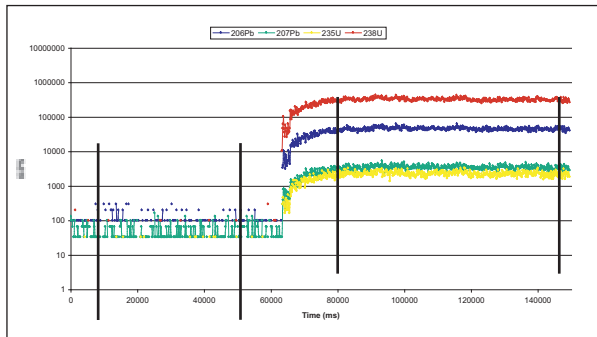


Figure 2: Ablation signals for 91500 zircon standard

The PlasmaLab ICP-MS software triggers and controls the UP193nm laser automatically through completely integral/embedded software functionality. Signals for  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{235}\text{U}$  and  $^{238}\text{U}$  rise sharply at around 65 seconds when the shutter of the laser ablation system opens and initial sample ablation occurs. Flat, stable analyte signals are observed for a period of over 60 seconds during the zircon ablation enabling an optimum time period for measurement of the required analyte signals.

#### Data Interpretation:

Raw time resolved data (including both gas blank and ablation signal) was exported from the PlasmaLab Profile TRA experiment in .csv file format using the integral software tools. Preliminary selection of background and ablation signal intensities was then performed using 'LAMTRACE', a Lotus 123 macro based spreadsheet package written by Simon Jackson of Macquarie University, Australia. Mass bias and background corrected signal intensities were calculated for each isotope using this software package and Concordia age calculations and diagrams were then established for each zircon based upon the correlation of the measured  $^{207}\text{Pb}/^{235}\text{U}$  and  $^{206}\text{Pb}/^{238}\text{U}$  ratios. LAMTRACE time-resolved isotope ratio data derived from the 91500 zircon sample is shown below in Figure 3 for reference and Concordia plots for both the 91500 standard and the Fish Canyon Tuff zircons are shown in Figures 4 and 5 respectively.

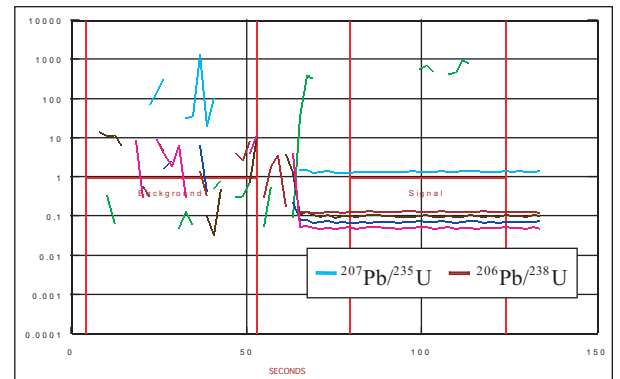


Figure 3: Signal ratios for 91500 zircon using 'LAMTRACE'

Flat, stable, signals are observed for  $^{207}\text{Pb}/^{235}\text{U}$  and  $^{206}\text{Pb}/^{238}\text{U}$  ratios in the above plot and there is little evidence of temperature or particle induced fractionation effects when using the LA-ICP-MS parameters described above.

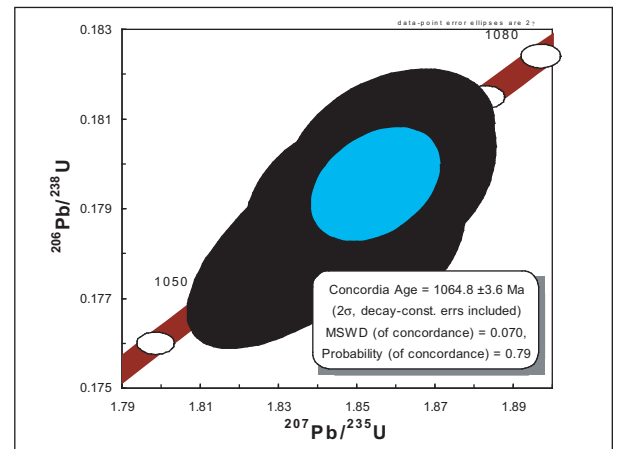


Figure 4: Concordia plot for 91500 zircon (1065Ma)

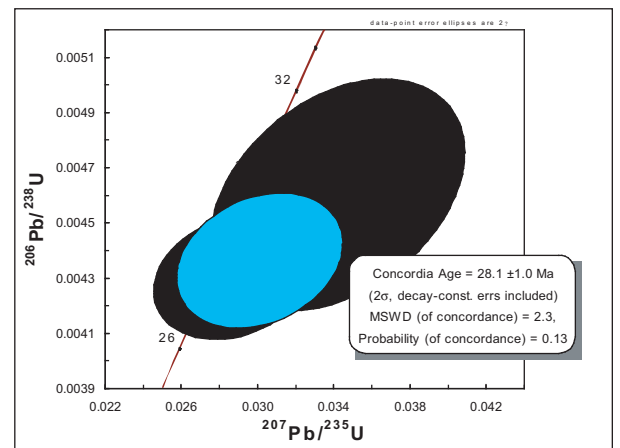


Figure 5: Concordia plot for Fish Canyon Tuff zircon (28.4Ma)

Pb/U isotope ratios correlate well for the 91500 and Fish Canyon Tuff zircons and the Concordia ages derived from the above plots match closely with the expected ages of the samples. The Concordia ages of the 91500 standard and Fish Canyon Tuff zircons are determined as  $1064.8 \pm 3.6$  Ma and  $28.1 \pm 1.0$  Ma respectively.

## Summary:

The New Wave Research 193nm Solid State Laser Ablation system is coupled seamlessly to the XSeries<sup>™</sup> ICP-MS to facilitate accurate assessments of U-Pb age and concordance in zircon populations with highly variable age characteristics. The unparalleled integration of the laser ablation and ICP-MS instrumentation provides a rapid, flexible and cost effective analytical solution for zircon analyses in the field of geochemical research.

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