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Pb Isotope Analysis of K Feldspars – A Challenge for LA-ICP-MS

Introduction

Feldspars are a useful provenance indicator but are easily weathered into small grains and are poor in uranium, making isotope ratio work difficult. Analyses are usually conducted by LA-ICP-MC-MS to improve precision and deal with the low sensitivity. It would be preferable to perform this analysis via LA-ICP-QMS, however the detection limits and precision are challenging and LA-ICP-MC-MS is typically used.

Here we explore the use of cutting edge LA-ICP-QMS techniques for provenancing of K feldspars. An iCAP Q (Thermo Fisher Scientific) was configured in high-sensitivity mode and running a He CCT method to maximize the high mass signal. A NWR213 (Elemental Scientific) with TwoVol2 ablation cell was used to ablate mounted and polished grains of K Feldspar (identified by SEM). The resulting data concurred with previously published studies using LA-ICP-MC-MS. Although precision from the LA-ICP-QMS method was slightly poorer it was within a useful range for the application.



Experimental

Grains were set in a 1" resin mount and analyzed by SEM prior to analysis to identify K feldspar grains (Figure 1). The SEM images were imported into the ActiveView software to omit any human translation error. Each grain was analyzed by spot ablation for 60 seconds (Figure 2. Experimental conditions in Table 1). NIST612 was used as a standard and a sample of Shap granite was used as a "known" sample. Data was reduced using lolite.

Table 1. Data was reduced using lolite.

Parameters Employed	
Laser Ablation	NWR213
Spot Size	60 μm
Repetition Rate	10 Hz
Fluence	6 J/cm ²
Ablation Cell	TwoVol2
He Flow Rate	0.70 mL/min
ICP-MS	Thermo iCAP Q
RF Power	1300 W
Neb Ar Flow Rate	0.69 mL/min
Collision Cell Mode	CCT
Isotopes Monitored	202, 204, 206, 207, 208
Interface	High Sensitivity Insert

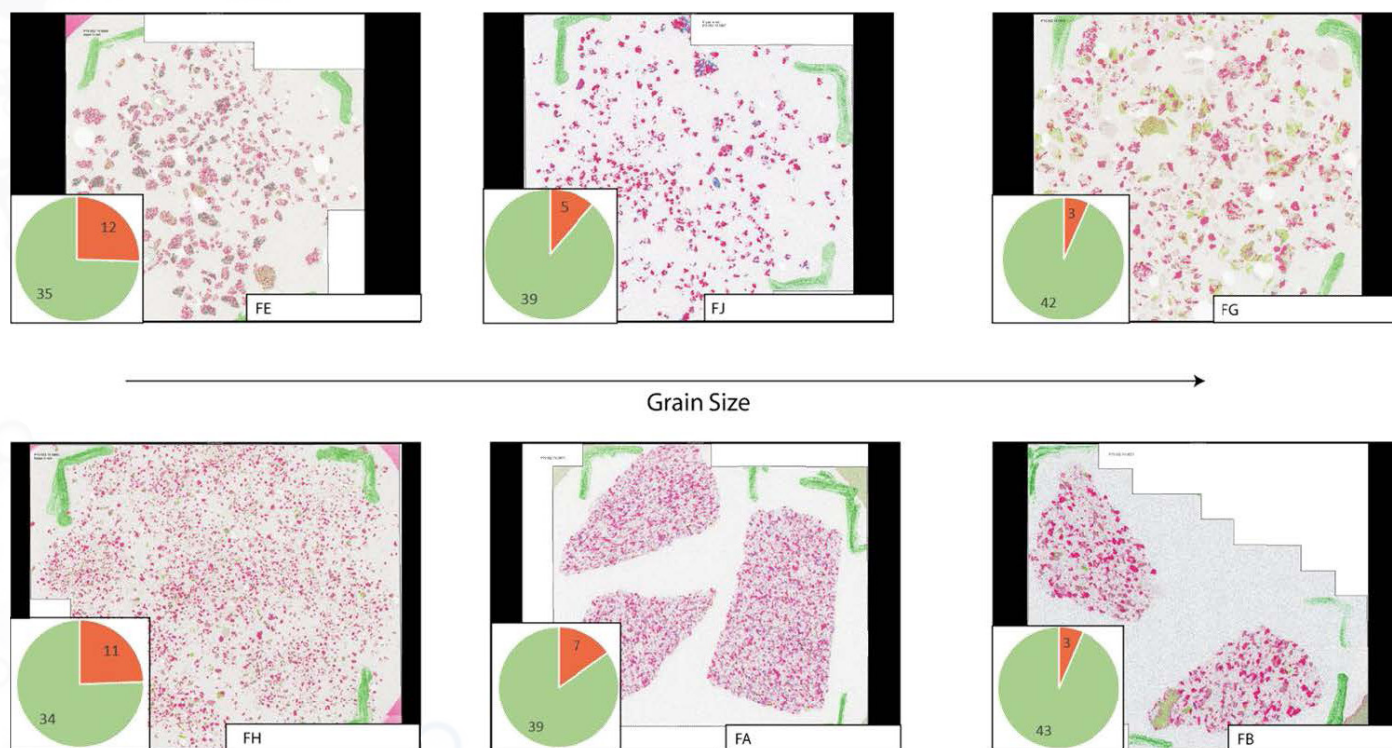


Figure 1. Color-phase SEM images of K feldspar grains (pink). Grain sizes increase left to right, and pie charts show number of grains where Pb isotope ratios are >0.5% (green) or <0.5% (red) based on lolite data reduction post-laser ablation.

Pb feldspar data	SEM image
■ Pb data <0.5 uncertainty	■ K-feldspar
■ Pb data >0.5 uncertainty	

Results

Data for time-resolved scans is shown in Figure 2, showing good sensitivity for all Pb isotopes. The data for Shap granite was compared to published results from Tyrrell et al.† (Figures 3 and 4) and shows that the LA-ICP-QMS method has good agreement with equivalent LA-ICP-MCMS methodology. (NB: Tyrrell et al. does not provide error data)

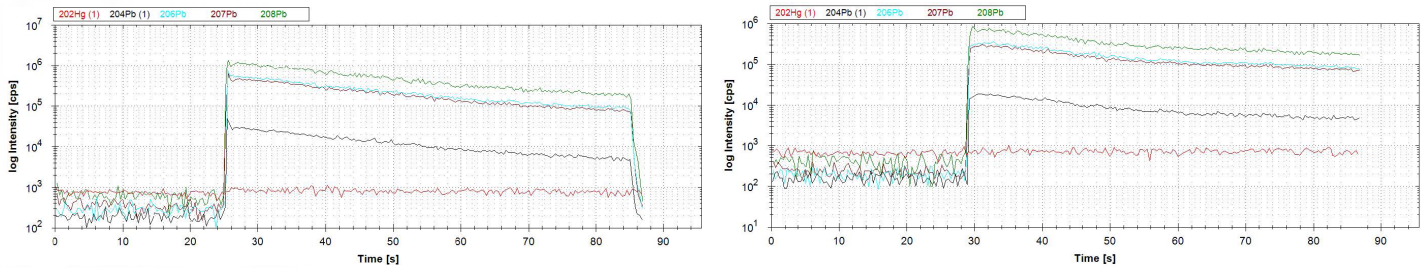


Figure 2. Time-resolved plot of Pb and Hg isotopes from laser ablation of Shap granite and a K feldspar grain.

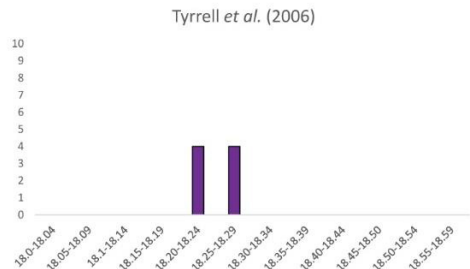
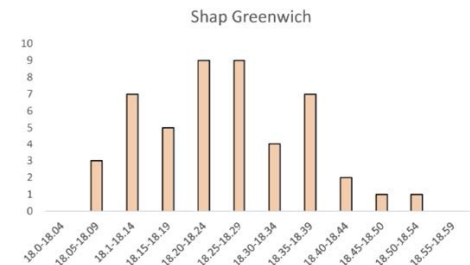
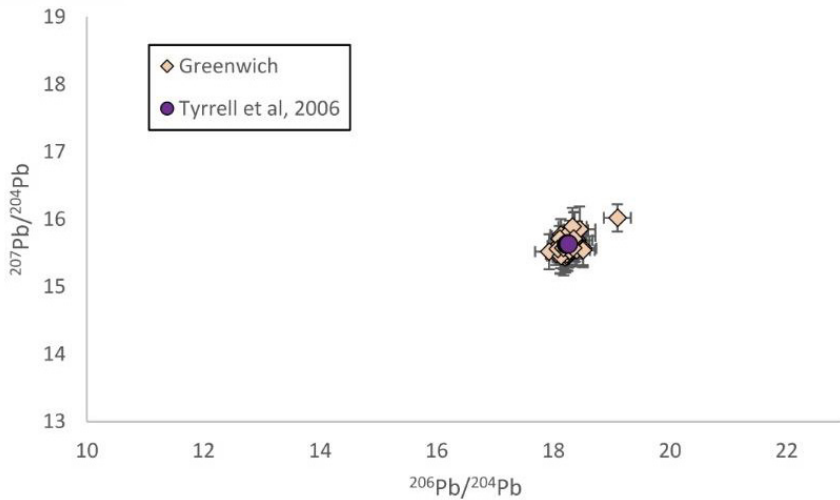


Figure 3. Pb isotope ratio plots of ablations of Shap granite using an NWR213 from Elemental Scientific laser ablation system and Thermo Scientific iCAP Q showing individual data points (n=50). No precision data is available for Tyrrell et al.

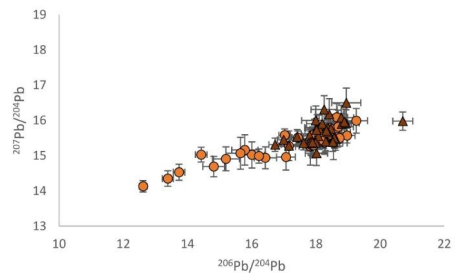
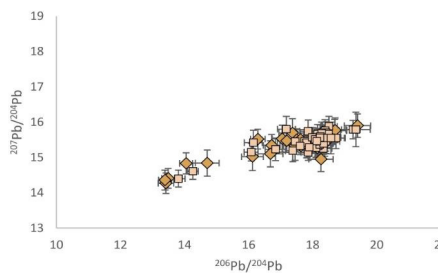
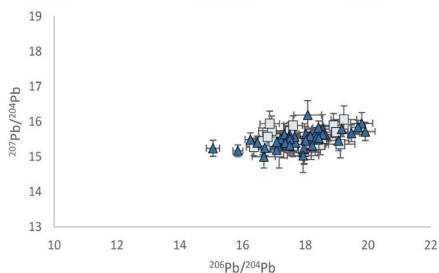


Figure 4. Pb isotope ratio plots of ablations of K feldspars using an NWR213 from Elemental Scientific laser ablation system and Thermo Scientific iCAP Q. Each plot shows a pair of K feldspar populations that were expected to match, which has been shown. Some contribution from common lead at m/z 204 is observed for smaller, more weathered grains.

Conclusions

Pb isotope ratio analysis of K feldspars has been achieved by LA-ICP-QMS with precision approaching that of LA-ICP-MC-MS.

Improvements in sample transport efficiency and reproducibility from the TwoVol2 ablation chamber on the NWR213, and sensitivity, stability and negligible background on the iCAP Q have lead to significant improvements in Pb & U isotopic measurements.

Advances in laser instrument design coupled to software advances such as ImageImport, ImageLock and a software bridge between the NWR and iCAP Q facilitate long, unattended instrument runs and compliment MS developments.

References

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