

OPTIMIZATION OF LEAD (Pb) ISOTOPES MEASUREMENT USING QUADRUPOLE-INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER (Q-ICP-MS).

Abida Usman^a, S. D. Young^a, E. H. Bailey^a, E. L. Ander^b, S. R. Chenery^b

^a*Division of Agricultural and Environmental Sciences, School of Biosciences, University of Nottingham, Sutton Bonington Campus, Loughborough, Leicestershire LE12 5RD, United Kingdom*

^b*British Geological Survey, Nicker Hill, Keyworth, Nottingham NG12 5GG, United Kingdom*

mbxau@nottingham.ac.uk

Keywords: Q-ICP-MS; Pb isotopes; Peak compression; Mass bias correction

Introduction

In isotope studies, multi-collector inductively coupled plasma mass spectrometers (MC-ICP-MS) and thermal ionization mass spectrometers (TIMS) are generally preferred to quadrupole ICP-MS because of its inability to measure all relevant isotopes simultaneously. Measured isotope intensities can be affected by variable interferences such as plasma flicker; this is especially important to the measurement of less abundant isotopes (Vanhaecke et al., 1997; Komárek et al., 2008). The other major drawback of Q-ICP-MS relates to differences in the efficiency of ion transfer through the instrument which causes mass discrimination effects; heavier isotopes are more efficiently transferred mainly due to their greater kinetic energy (Heumann et al., 1998). The objective of this study was to test approaches for improving Pb isotope measurement by Q-ICP-MS.

Methods

An optimized methodology for Pb isotope (²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb) determination was developed using a Q-ICP-MS system (Model iCAPQ; Thermo Scientific, Bremen, Germany). Reference materials e.g. NIST-981 were used to optimize the detector dead time correction factor (Df) and the dwell time. Internal (thallium) and external (NIST-981) standards were used to determine a mass discrimination correction factor (Kf). An attempt was made to reduce the problem of counting statistics due to the low isotopic abundance of ²⁰⁴Pb using 'peak compression' for the more abundant isotopes. Compression factors (Kc) were calculated individually for ²⁰⁵Tl and with NIST-981 (for ²⁰⁸Pb, ²⁰⁷Pb and ²⁰⁶Pb). Data derived in pulse counting mode were back-converted to an equivalent uncompressed intensity by correction with calculated Kc values determined with Tl (internal conversion) and NIST-981 (interpolated external conversion). Blank corrections were applied to the converted signals for Pb and Tl isotopes using calculated compression factors. The resulting data was then further processed for mass bias correction with both Tl (internal standard) and NIST-981 (external standard). Both mass bias corrections were applied sequentially so that Tl reduced noise caused by variation in sample matrix whereas NIST-981 allowed for long term instrumental drift during the experiment. A comparison was made between data collected using Q-ICP-MS and pre-existing multi-collector (MC-ICP-MS) for the same samples. The purpose of this comparison was to select the best possible Q-ICP-MS option assuming that MC-ICP-MS provides the most accurate and precise data.

The developed methodology was applied to a range of samples including urban soil samples Nottingham and Wolverhampton), urban allotment soils (Nottingham) and sewage amended soils (Pakistan).

Results

The isotopic ratios with ^{204}Pb as the denominator ($^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$) in uncompressed mode provided the best fit overall in terms of lower RSD values and slope values which were closest to 1.0.

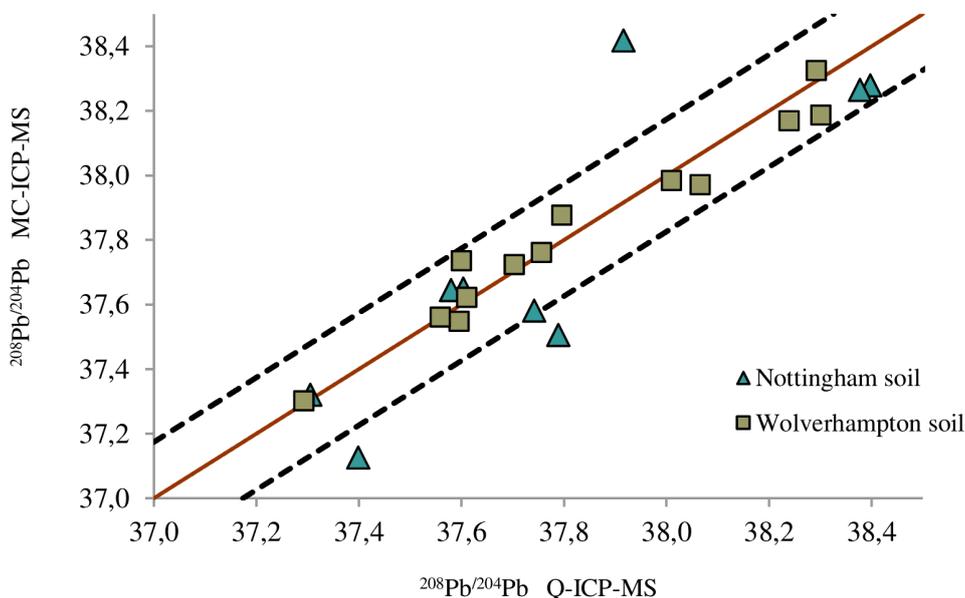


Figure 1: Comparison of $^{208}\text{Pb}/^{204}\text{Pb}$ isotopic ratios (uncompressed data) in urban soils measured by MC-ICP-MS with those measured using an optimised Q-ICP-MS approach. Solid line gives the 1:1 relationship plus or minus one standard deviation (dashed lines).

Conclusion

Optimization of analytical procedure can improve Pb isotopes measurement with Q-ICP-MS that can therefore be used as an alternative for MC-ICP-MS in terms of source apportionment of Pb sources and their pathways in soil.

References

- G. Heumann, K., M. Gallus, S., Rädlinger, G., & Vogl, J. (1998). Precision and accuracy in isotope ratio measurements by plasma source mass spectrometry. *Journal of Analytical Atomic Spectrometry*, 13(9), 1001-1008
- Komárek, M., Ettler, V., Chrástný, V., & Mihaljevič, M. (2008). Lead isotopes in environmental sciences: A review. *Environment International*, 34(4), 562-577.
- Vanhaecke, F., Moens, L., Dams, R., Papadakis, I., & Taylor, P. (1997). Applicability of High-Resolution ICP-Mass Spectrometry for Isotope Ratio Measurements. *Analytical Chemistry*, 69(2), 268-273.