

A comparison of different calibration strategies for trace and ultra-trace analysis by inductively coupled plasma mass spectrometry (ICP-MS)

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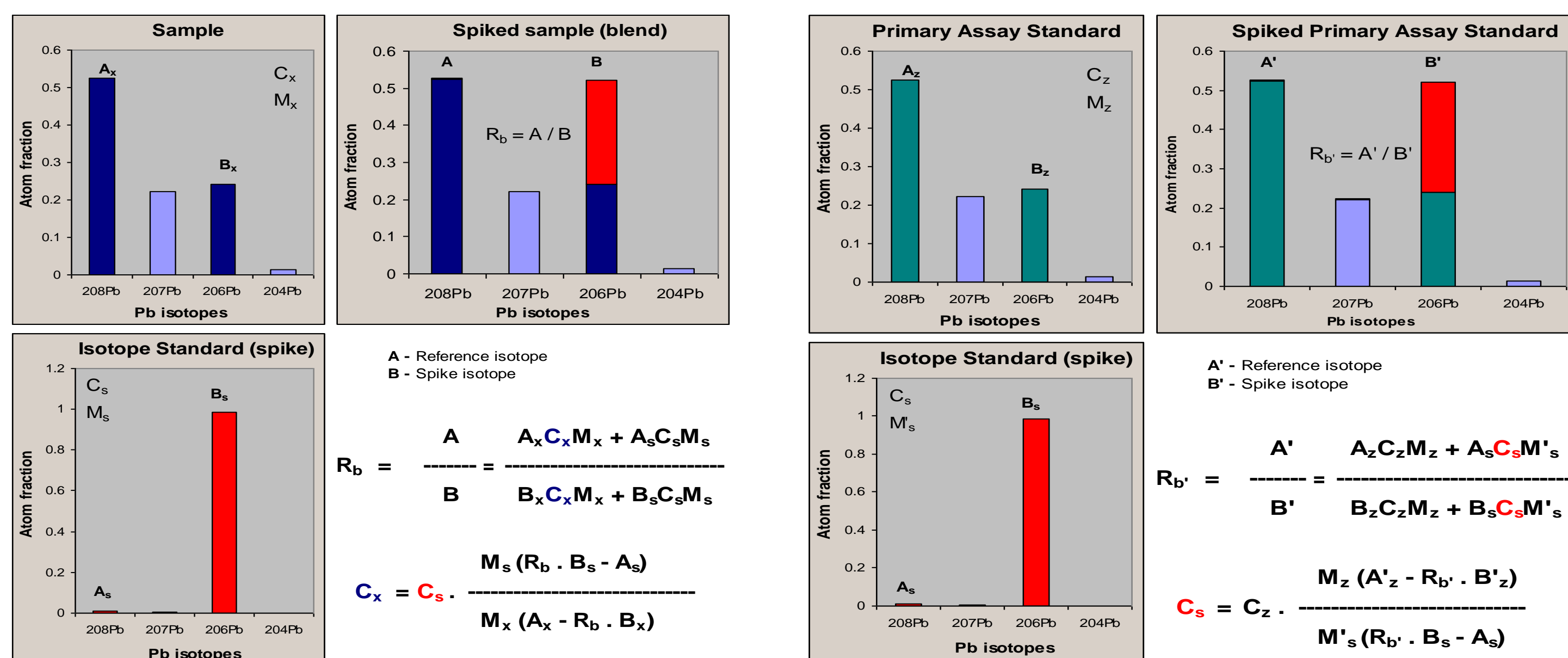
Introduction

The National Metrology Institute of South Africa (NMISA) is expanding its capabilities in the field of the analysis of trace and ultra-trace levels of toxic and minor elements in environmental samples, with a special focus on soils to support University of Pretoria (UP) projects in the field of veterinary geology for the One Health Platform.

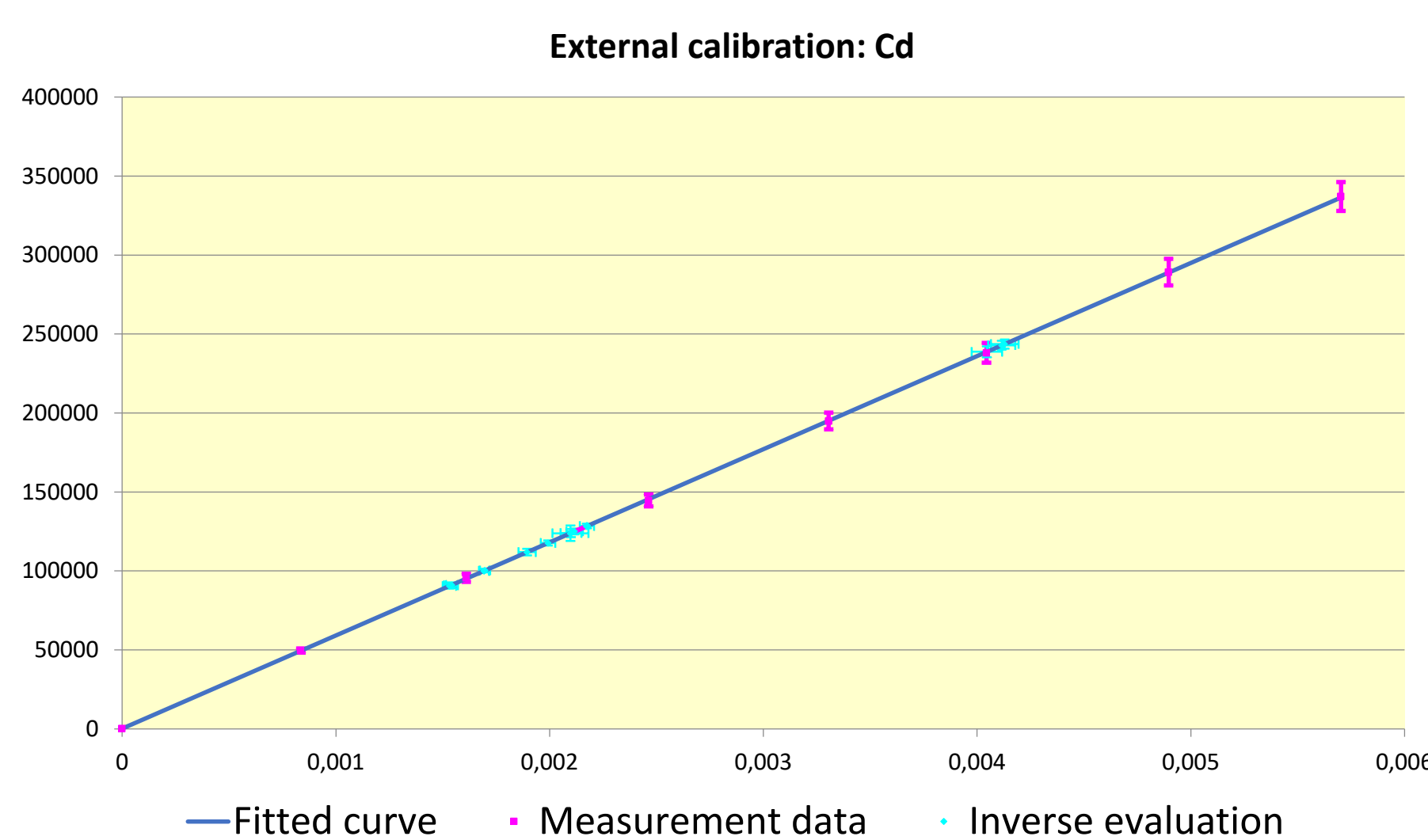
The purpose of this study was to compare the accuracy of three different calibration strategies for the analysis of trace elements in soil using inductively coupled plasma sectorfield-mass spectrometry (ICP-SFMS). The most accurate strategy employed in the laboratory is isotope dilution analysis and the other two strategies used are gravimetric external calibration with gravimetric internal standardisation and gravimetric standard addition with gravimetric internal standardisation.

Calibration strategies

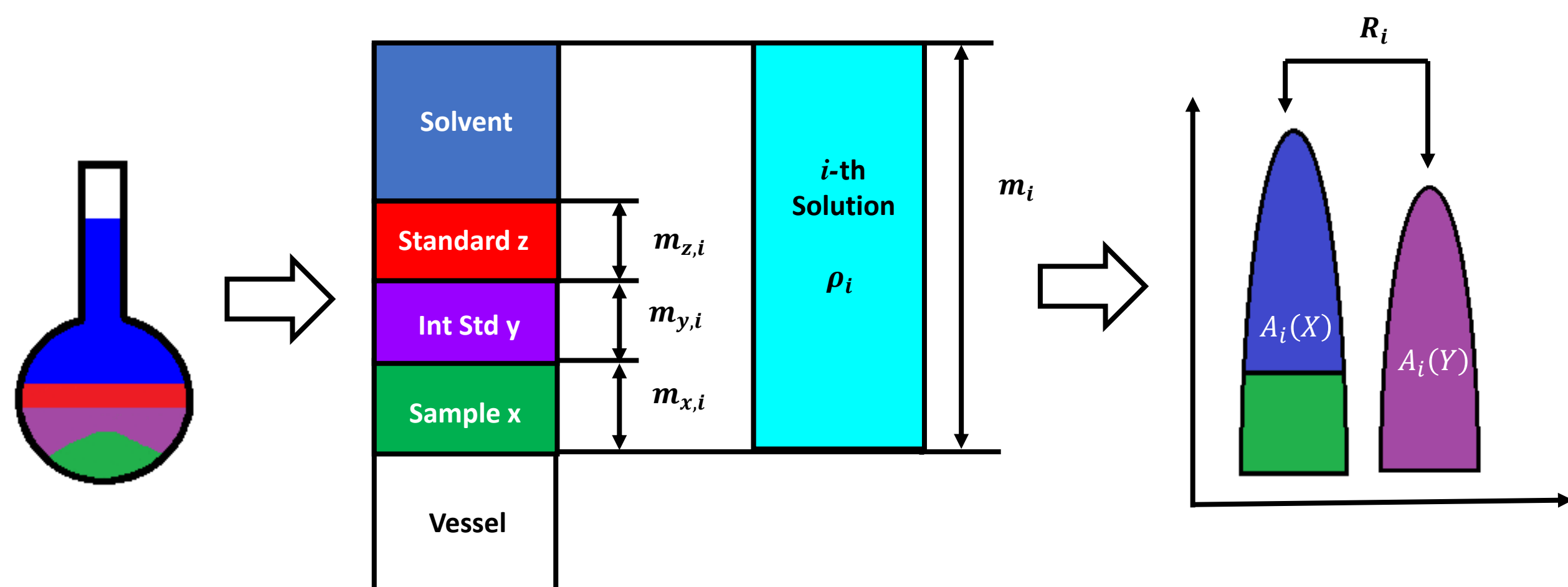
Double isotope dilution mass spectrometry



Gravimetric external calibration with gravimetric internal standardisation



Gravimetric standard addition with gravimetric internal standardisation



Experimental set up

Microwave digestion

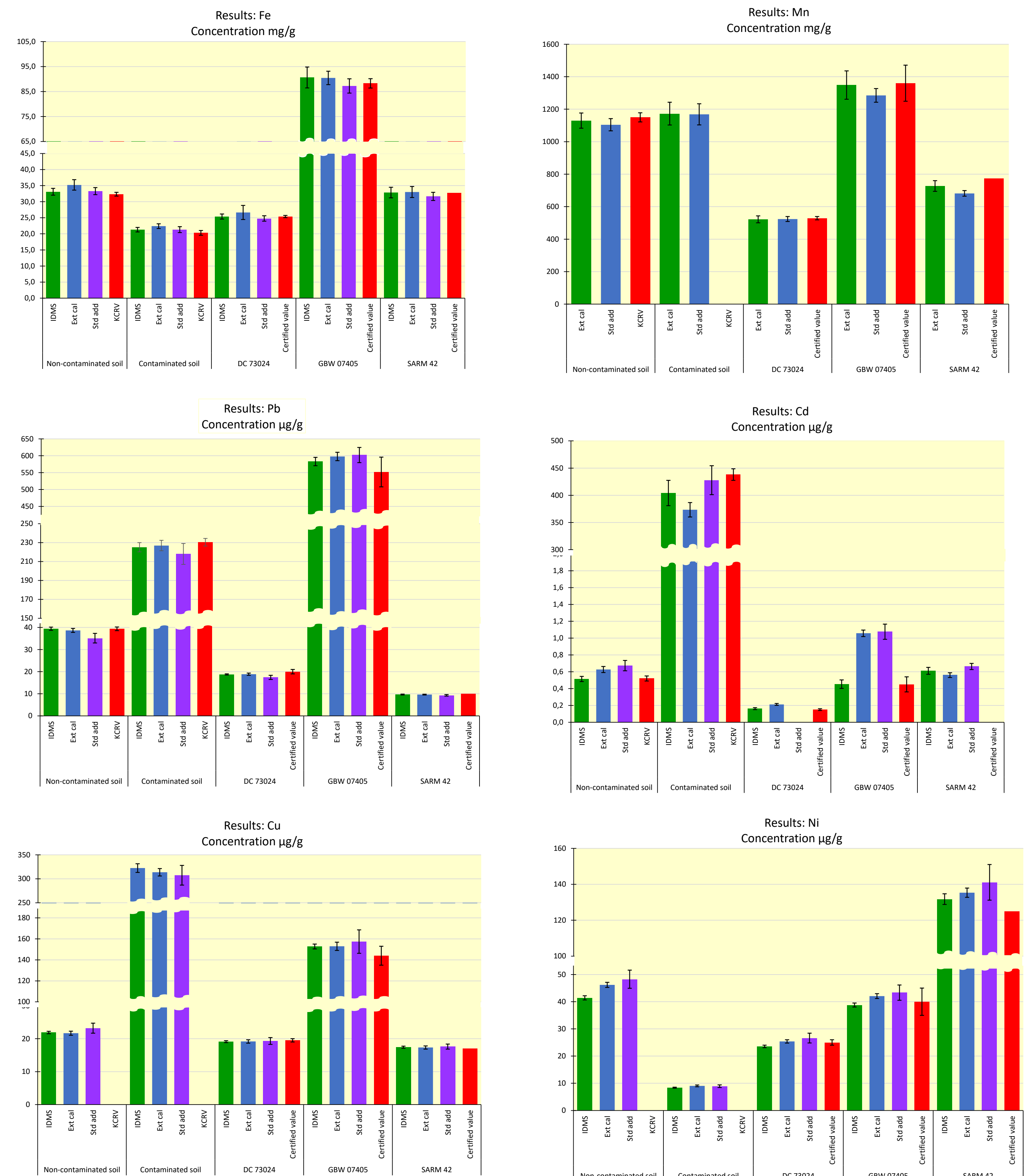
Reagents:

Step 1: 8 mL HNO₃, 5 mL HCl, 3 mL HF



Step 2: 1 mL HF, 15 mL 5% H₃BO₃

Results



Conclusions and Recommendations

The results and uncertainties for all the elements across all three calibration strategies compare well with the certified values or the comparison reference values of the soil samples. The estimated uncertainties of the results for the external calibration and standard addition methods can be improved by increasing the number of replicates.

This study shows that external calibration and standard addition methods can be applied with comparable precision to isotope dilution analysis. It opens doors to routine analytical laboratories that require higher precision and accuracy for reporting results, but do not have access to the expensive instrumentation required for isotope dilution analysis.

These approaches will be best suited to homogeneity and stability studies where many of the same samples are analysed. Due to the fact that many replicates of essentially the same samples are analysed, measurement precision comparable to the IDMS method that will typically be used for the characterisation study can be obtained. In the case of these studies the experimental design will be simplified compared to the study presented here where samples were analysed over an extended range of concentrations for the different elements.

Future work is planned to simplify the experimental design of the standard addition method by spiking the samples before digestion. This approach will reduce the amount of gravimetric sample preparation required.

References

Torres, M. Rocio Arvizu, et al, "Contaminant and other elements in soil (CCQM-K127)", *Metrologia* 54.1A: 08010.

Davis, Anthony J., and Jan G. Myburgh, "Investigation of stillbirths, perinatal mortality and weakness in beef calves with low-selenium whole blood concentrations", *Journal of the South African Veterinary Association* 87.1 (2016): 1-6.