Evaluation of major to ultra trace element bulk rock chemical analysis of nanoparticulate pressed powder pellets by LA-ICP-MS

D. PETERS AND T. PETTKE

Institute of Geological Sciences, University of Bern, Baltzerstrasse 1+3, 3012 Bern, Switzerland

(correspondence: daniel.peters@geo.unibe.ch, pettke@geo.unibe.ch)

Introduction

We present [1] a method combining the advantages of nanoparticulate pressed powder pellets (PPP) [2] and the addition of a mechanical and laser light absorbing binder [3] to achieve precise and accurate major to trace element analysis of bulk rock samples via 193 nm LA-ICP-MS. Focus is put on major elements; unconventional fluid-tracers, e.g., B, As and Sb, and on trace elements not commonly measured. Data are calculated with GSD-1G as external standard (if not indicated otherwise) and internally standardised to the sum of major element oxides.

Materials

The LA-ICP-MS PPP analytical procedure was optimised and evaluated using six different geological reference material (GRM) powders (JP-1, UB-N, BCR-2, GSP-2, OKUM, and MUH-1). Calibration based on external standardisation using NIST SRM 610, SRM 612, BCR-2G, and GSD-1G glasses allows for evaluation of possible matrix effects during LA-ICP-MS analysis.

PPP analytical procedure

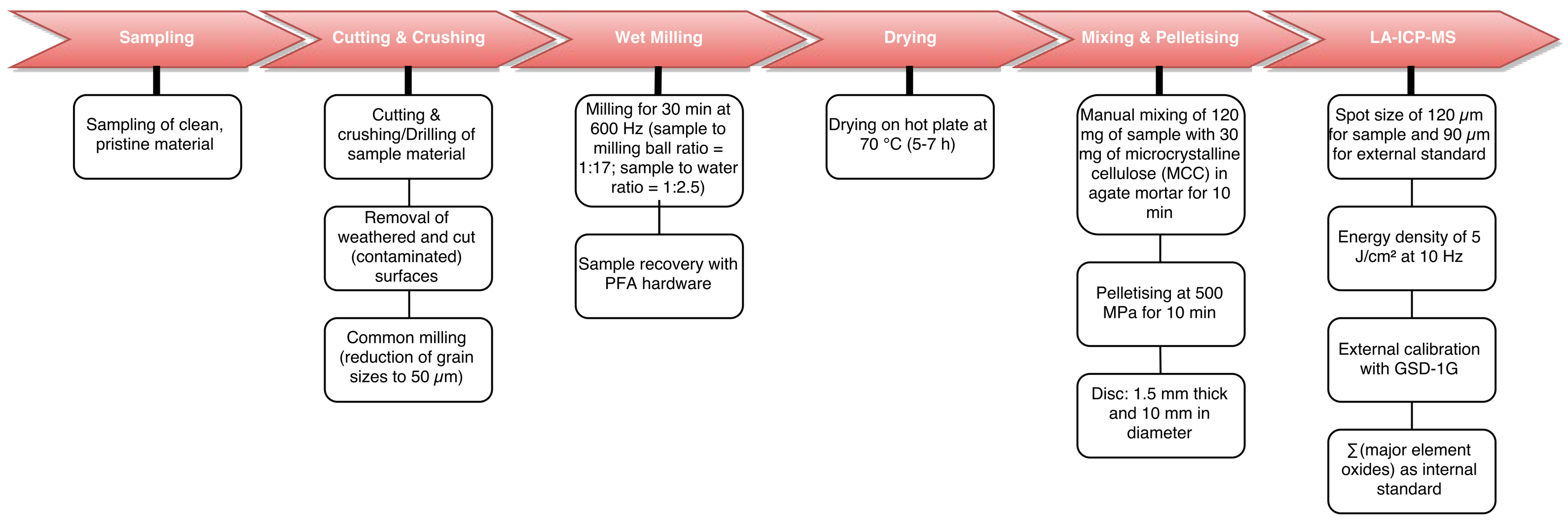

Fig. 1 Scheme of PPP analytical procedure. A planetary ball mill (Retsch PM 100) and agate milling equipment are used. LA-ICP-MS measurements are conducted with a GeoLas-Pro 193 nm ArF Excimer laser system (Lambda Physik, Germany) in combination with an ELAN DRC-e quadrupole mass spectrometer (Perkin Elmer, USA) at the University of Bern, Switzerland. Robust plasma conditions (S(U) = S(Th)) and ThO production rate < 0.5 % are used, and drift is linearly corrected for by bracketing external standardisation.

Evaluation of different matrices with BCR-2(G)

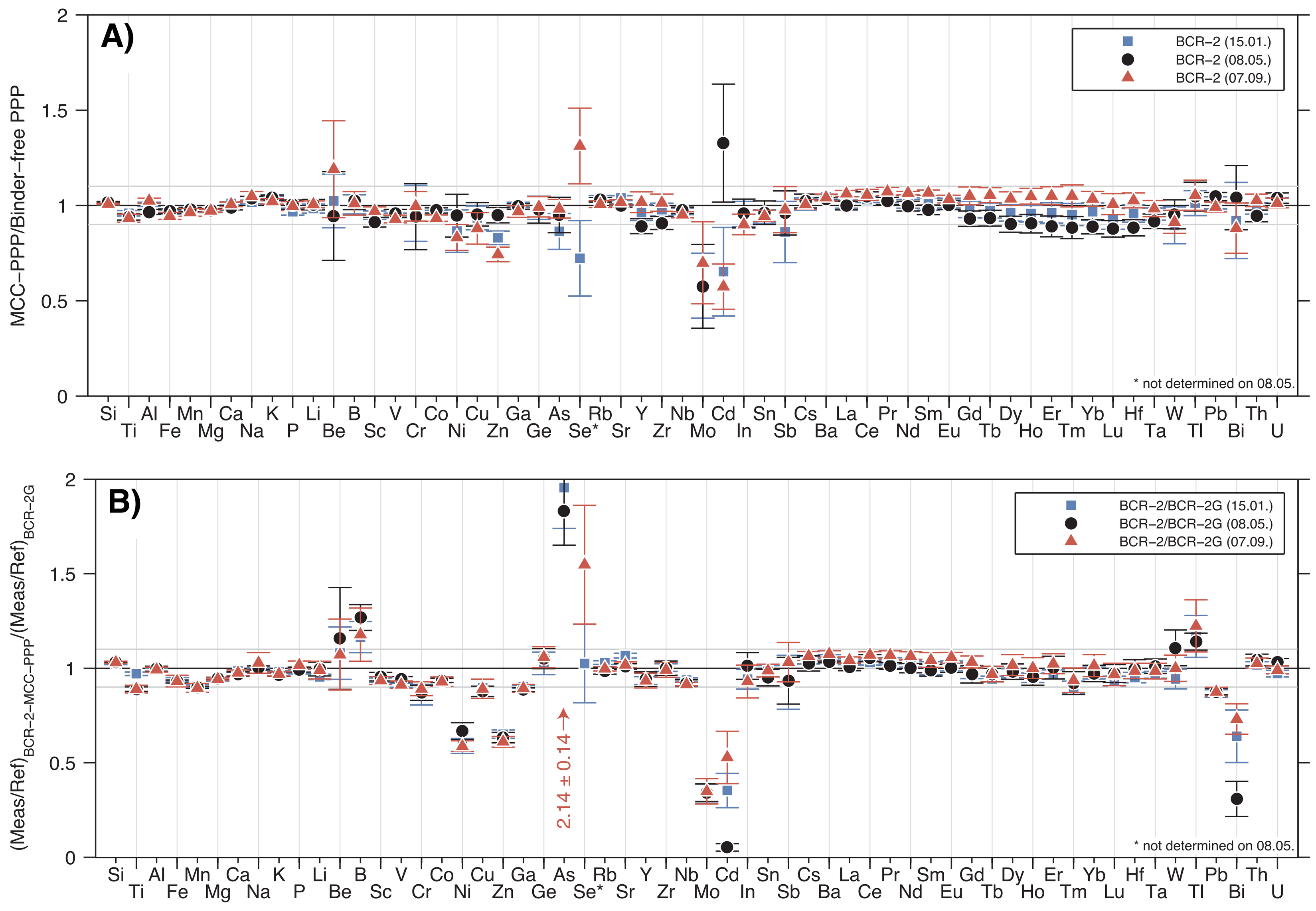

Fig. 2 Examination of matrix effects based on the GRM BCR-2, quantified using SRM 610 as the external standard and measured in three different measurement sessions (dates in brackets). **(A)** Measured concentrations of pressed powder pellet (PPP) with MCC binder normalised to binder-free PPP. **(B)** Comparison of measured concentrations for PPP and glass employing BRC-2 basalt by plotting BCR-2 MCC-PPP measured concentrations normalised to reference data $(Meas/Ref)_{BCR\text{-}2\text{-}MCC\text{-}PPP}$ divided by BCR-2G measured concentrations normalised to reference data $(Meas/Ref)_{BCR\text{-}2G}$. Error bars represent the 1 SD uncertainties on the external reproducibility of six spot analyses (n = 6).

Accuracy & Precision

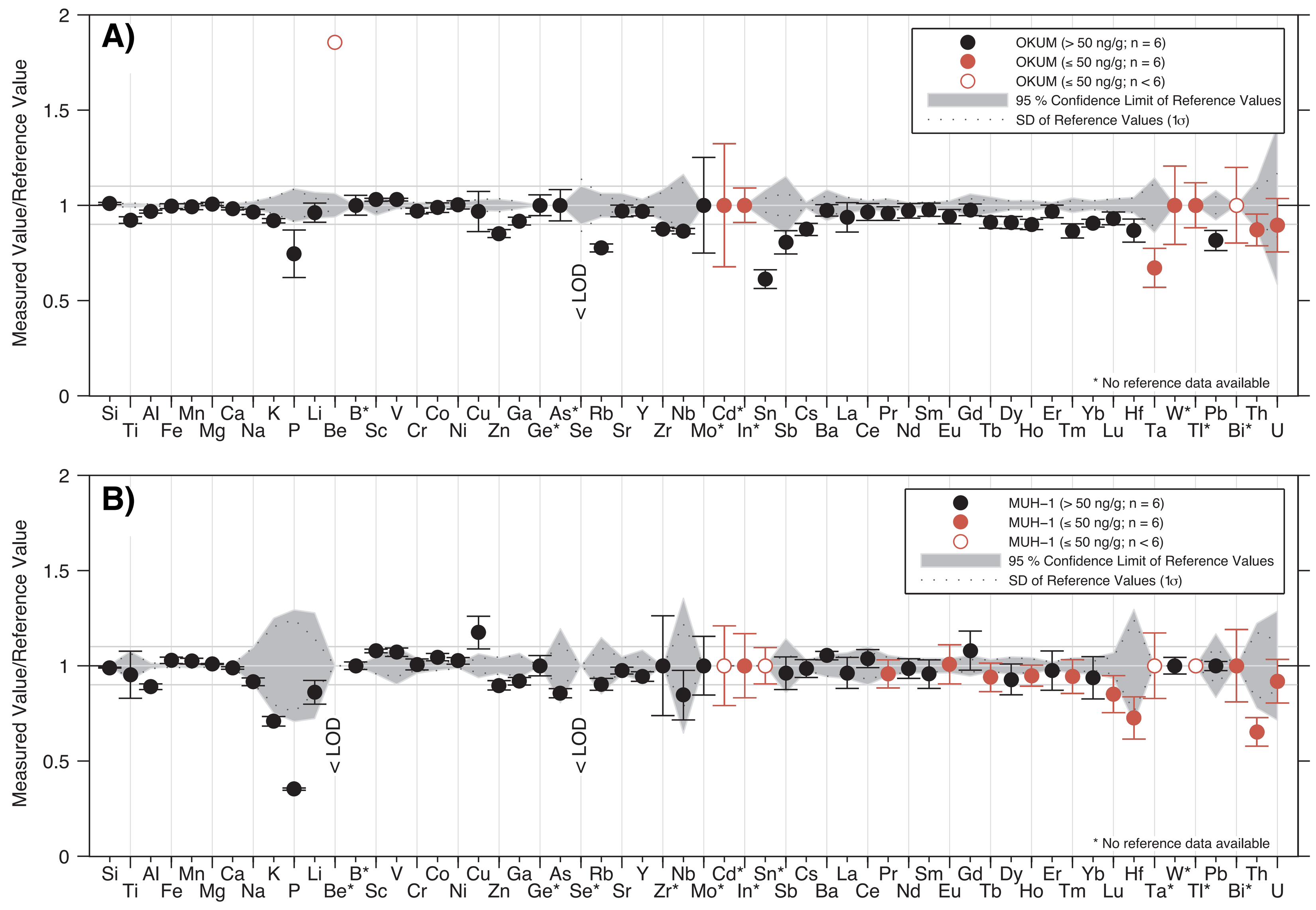

Fig. 4 Plots illustrating the analytical accuracy for International Association of Geoanalysts (IAG) ultrabasic rocks OKUM **(A)** and MUH-1 **(B)**. Data symbols are filled black for elements with concentrations above 50 ng g^{-1}, filled red for elements with concentrations below 50 ng g^{-1}, and red circles filled white for elements which did not return significant mass fractions for each spot measurement. Note that the GRMs have several elements without reference concentrations; these elements are marked with an asterisk on the x-axis and our measurement data are plotted at y = 1.0. Error bars represent the 1 SD uncertainties on the external reproducibility of six spot analyses (n = 6; except for red circles filled white).

Elemental fractionation using NIST SRM 610/612

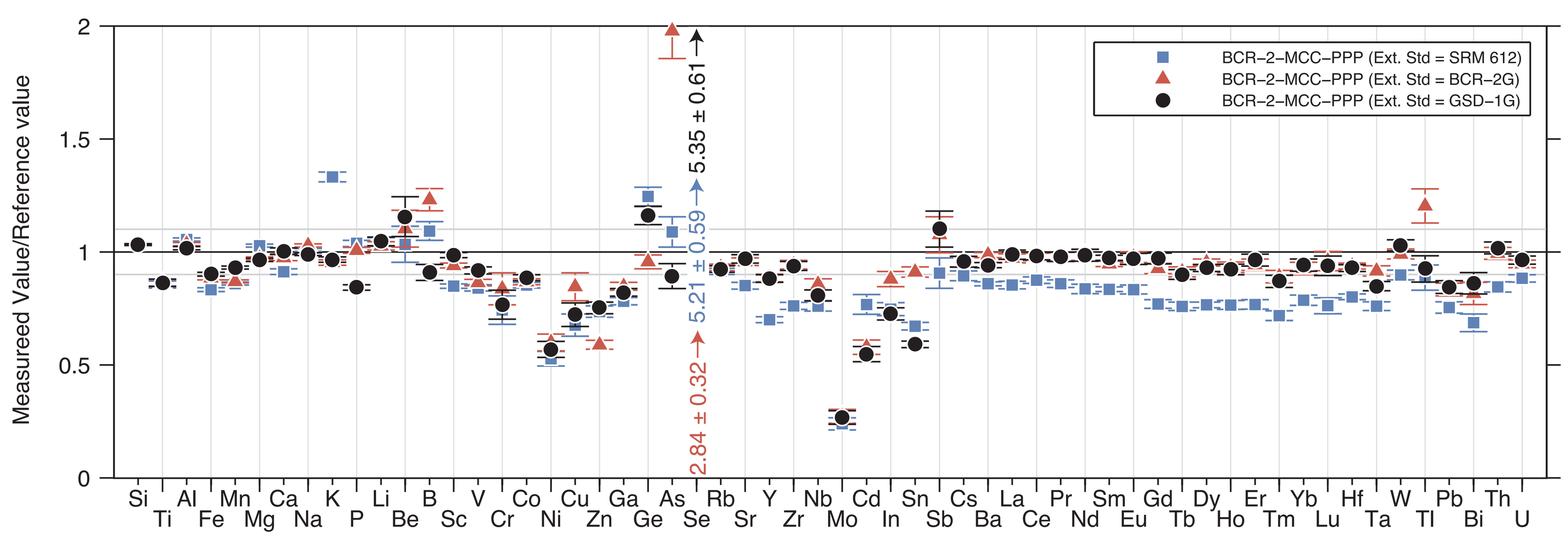

Fig. 3 Measured element concentrations of MCC-bound PPP relative to the respective reference values quantified by using different external standards: SRM 612, BCR-2G, and GSD-1G. The use of basaltic glasses as external standard minimises the apparent depletion of Y+REEs. Similar observations were made for the GRMs BCR-2G, GSP-2 and OKUM when externally standardised with NIST SRM 610/612. Error bars represent 1 SD uncertainties on the external reproducibility (n = 6).

Geochemical application

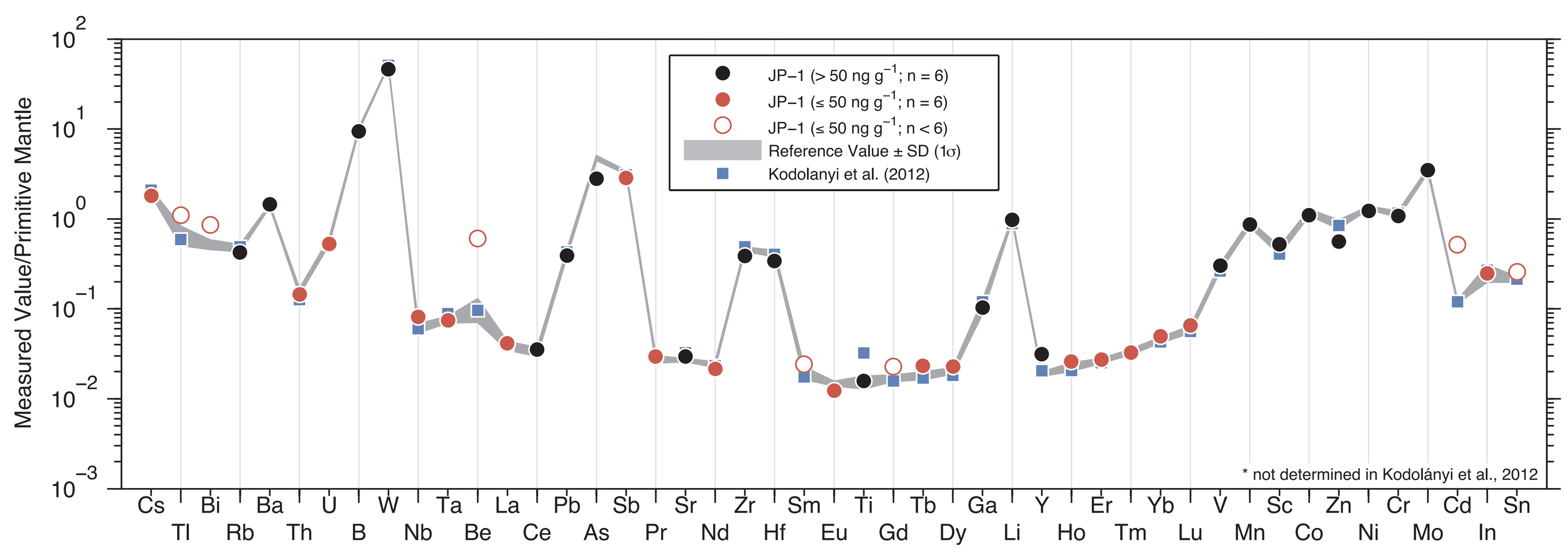

Fig. 5 Comparison of MCC-PPP data from this study vs. liquid ICP-MS data ([4]; bomb digestion), based on JP-1, plotted as a primitive mantle (PM)-normalised spider diagram (PM values [5]). Most analytical uncertainties are smaller than the symbol size.

Selected Conclusions

- ✧ **LA-ICP-MS analysis of MCC-bound nanoparticulate PPPs is an accurate and efficient alternative for the analysis of bulk major to trace element concentrations**
- ✧ **Unconventional geochemical tracers, such as Li, B, As, Sb, as well as highly refractory elements (e.g., Cr, HFSE) are accurately quantified**
- ✧ **Elemental fractionation for analysis of intermediate to ultrabasic bulk rock compositions (PPP and glass samples) is minimised/eliminated when basalt glasses are employed as external standard**
- ✧ **Employing MCC as a pellet binder allows (i) for the analysis of all solid materials, and (ii) has great potential for standard addition approaches**

[1] Peters & Pettke (2016). Geostand. Geoanalyt. Res. in press. **[2]** Garbe-Schönberg & Müller (2014). JAAS 29(6), 990-1000. **[3]** O'Connor et al. (2007). JAAS 22(3), 273-282. **[4]** Kodolanyi et al. (2012). J Petrology 53(2), 235-270. **[5]** Palme and O'Neill (2004). In: Treatise on Geochemistry, 2 ed. (3), 1-38.

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