

## Major to ultra trace element bulk rock analysis of nanoparticulate pressed powder pellets by LA-ICP-MS

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An efficient, clean procedure for bulk rock major to trace element analysis by 193 nm Excimer LA-ICP-MS analysis of nanoparticulate pressed powder pellets (PPPs) employing a binder is presented. Sample powders are milled in water suspension in a planetary ball mill, reducing average grain size by about one order of magnitude compared to common dry milling protocols. Microcrystalline cellulose (MCC) is employed as a binder, improving the mechanical strength of the PPP and the ablation behaviour, because MCC absorbs 193 nm laser light well. Use of MCC binder allows for producing cohesive pellets of materials that cannot be pelletized in their pure forms, such as quartz powder. Rigorous blank quantification was performed on synthetic quartz treated like rock samples, demonstrating that procedural blanks are irrelevant except for a few elements at the 10 ng  $g^{-1}$  concentration level. The LA-ICP-MS PPP analytical procedure was optimised and evaluated using six different SRM powders (JP-1, UB-N, BCR-2, GSP-2, OKUM, and MUH-1). Calibration based on external standardization using SRM 610, SRM 612, BCR-2G, and GSD-1G glasses allows for evaluation of possible matrix effects during LA-ICP-MS analysis.

The data accuracy of the PPP LA-ICP-MS analytical procedure compares well to that achieved for liquid ICP-MS and LA-ICP-MS glass analysis, except for element concentrations below  $\sim 30 \text{ ng g}^{-1}$ , where liquid ICP-MS offers more precise data and in part lower limits of detection. Uncertainties on the external reproducibility of LA-ICP-MS PPP element concentrations are of the order of 0.5 to 2 % (1 $\sigma$  standard deviation) for concentrations exceeding  $\sim 1 \ \mu \text{g g}^{-1}$ . For lower element concentrations these uncertainties increase to 5-10% or higher when analyte-depending limits of detection (LOD) are approached, and LODs do not significantly differ from glass analysis. Sample homogeneity is demonstrated by the high analytical precision, except for very few elements where grain size effects can rarely still be resolved analytically.

Matrix effects are demonstrated for PPP analysis of diverse rock compositions and basalt glass analysis when externally calibrated based on SRM 610 and SRM 612 glasses; employing basalt glass GSD-1G or BCR-2G for external standardisation basically eliminates these problems. Perhaps the most prominent progress of the LA-ICP-MS PPP analytical procedure presented here is the fact that trace elements not commonly analysed, i.e. new, unconventional geochemical tracers, can be measured straightforwardly, including volatile elements, the flux elements Li and B, the chalcophile elements As, Sb, Tl, Bi, and elements that alloy with metal containers employed in conventional glass production approaches. The method presented here thus overcomes many common problems and limitations in analytical geochemistry and is shown to be an efficient alternative for bulk rock trace elements analysis.