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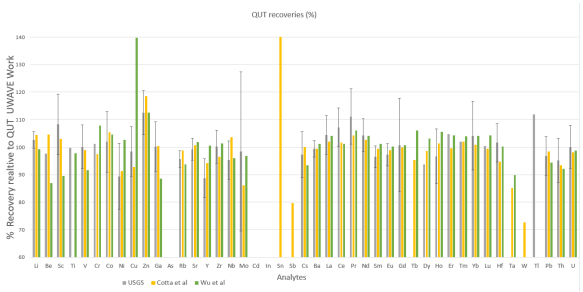
Low blank single reaction chamber microwave digestion of refractory rocks and high-matrix biological materials

OSAMA YA GHIDAN[1], BALZ S. KAMBER[1], DAVID T MURPHY[1] AND JEREMY MATTHEWS[2]

[1]Queensland University of Technology
[2]Metrohm Australia

Presenting Author: sam.ghidan@qut.edu.au



We report experimentation on digestion of problematic rocks and biological samples for direct analysis of trace element concentrations and isotope ratios by ICP-MS. Our experiments focused on low-blank digestion of rocks with refractory minerals (zircon, spinel and garnet) and matrix reducing digestion of entire insect and plant part specimens. The main instrument deployed was the single reaction chamber (SRC) microwave sample preparation system ultraWAVE 2.0 and more recently, 3.0 from Milestone srl (Bergamo, Italy). The system has a quartz bottom plate allowing temperatures exceeding 280°C at pressures of up to 200 bar. The gas flow path allows automated cleaning and improved analytical blanks while the water-cooled magnetron provides the thermal stability required for routine high-temperature operation needed for many geochemical applications.

For the rock samples and CRMs, extensive series of experiments were conducted in 7mL PFA Teflon vessels, permutating the mass of digested material, the volume of acids, duration of digestions and temperature of digestions. CRMs GSP-2 and PCC-1 were successfully digested in HF-HNO_3 mixture at 250°C at 40 bars over 90 minutes (including cooling). Critical for successful recoveries were two key insights. Firstly, the conversion of fluorides in HCl at a temperature higher than the digestion (i.e., 280°C for 20 minutes). Secondly, drying down fluorides, chlorides and later conversion to nitrates within the original Teflon tubes in a customised heat block designed to fit the dimensions of the Teflon vessels. Recoveries for GSP-2 for all elements, including those hosted in zircon and spinel and those prone to insoluble fluoride formation was 95% or better (Figure 1). The only element for which an elevated blank was found was Sn, while many metals of interest to non-traditional isotope geochemists had blanks as low as conventional beaker digests.

For the insect and plant leaf samples, we compared conventional open Teflon beaker with ultraWAVE digestions and found significant improvement in transparency and viscosity in the latter, which required a simple single digestion step with no further conversions. This allowed for direct analysis of the digestions for ultra-low level trace elements with high sensitivity ICP cones and direct Sr-isotope analysis by tandem ICP-MS.