Optimal extraction and purification of Re from geological samples: the role of coarse anion resin beads

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The rhenium-osmium (¹⁸⁷Re-¹⁸⁷Os) geochronometer and geochemical tracer is now widely applied to geological samples. However, samples with low ppt levels of Re and Os remain challenging to analyze. Natural rhenium has only two isotopes, ¹⁸⁵Re and ¹⁸⁷Re, precluding the use of an on-line mass fractionation correction. Using the N-TIMS technique, Re is therefore measured only on static Faraday cups where high intensity signals are of paramont importance. Therefore, optimizing chemical recovery and emission efficiency is essential to obtain sufficiently high Re signal intensities needed for measuring low-level Re samples.

Coarse anion resin bead "clean-up" follows Re isolation by anion resin column chemisty or acetone-NaOH solvent extraction, and is the final step in the Re purification process before loading for N-TIMS measurement. The clean-up consists of four steps: (1) preparing resin beads; (2) equilibration between Re-bearing solution and the resin beads; (3) rinsing impurities from Re-loaded resin beads; and (4) elution of Re from resin beads. To evaluate the effect of each step on Re yield and purity, solution aliquots were collected and tested during routine Re-Os analyses. Based on coupled ICP-MS and N-TIMS data, the optimized protocol calls for two back-to-back 30 min bead preparing steps using 0.8 mol/L HNO3 (step 1); 60 min of shaking for loading Re onto resin beads in 0.05 mol/L HNO3 (step 2); 5 min shaking with 0.8 mol/L HNO₃ for rinsing (step 3) and 60 min shaking to elute Re with 6 mol/L HNO₃ (step 4). Ultimately, the net Re signal intensity was improved ~15 times compared to our previous protocols - 2.5 times improvement in the chemical recovery (yield) and 6 times improvement in emission efficiency (purity). Measured Re isotopic ratios do not show any fractionation and are within the external long-term reproducibility of AIRIE's 1407 Re in-house standard solution.

Along with consistently low femtogram level Os blanks¹, improved Re chemistry permits Re-Os investigation of geological samples and media (e.g., light oils) with very low ppt-level Re and Os contents.

[1] Yang et al. (2015) Anal. Chem. 87, 7017–7021.