Re-evaluation of digestion methods for accurate Re-Os isotope and highly siderophile element analyses

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The current database on highly siderophile elements (HSEs: Re, Au, Ir, Os, Ru, Rh, Pt and Pd) and Os isotopes in geological samples is dominated by isotope dilution methods, coupled with high-temperature sample digestion using inverse aqua regia in closed glass vessels such as Carius tubes (CT) or a high-pressure asher system (HPA). These acid digestion techniques are preferred over traditional flux fusion techniques, such as NiS fire assay, largely due to the ability to measure Re and Os on the same sample aliquot. By contrast, two major limitations - the 'nugget effect' and incomplete digestion - have often been associated with these acid digestion techniques. Recent data suggest that an additional HF step is essential to release HSEs hosted in the silicate portions of certain basaltic materials, necessitating modification of typical digesting procedures.

To address this issue, we systematically conducted analytical tests for CANMET reference material TDB-1 with varying digestion apparatus (microwave, CT, HPA), conditions (temperature, duration, sample size) and protocol (with or without HF desilicification, either before or after aqua regia attack). We found the optimum method for simultaneous determination of $^{187}Os/^{188}Os$ and HSE concentrations used inverse aqua regia to attack 1-2 g of powder over long durations, such as a Carius tube heated to 240˚C for 72 hours, followed by an HF desilicification step after CCl_4 solvent extraction of Os. It is anticipated that extended HPA digestions will also achieve the same effect.

The method provides strong linear correlations on Os versus Ir-Ru-Pt concentration diagrams for repeat dissolutions of TDB-1, reflecting an effect of powder heterogeneity. This is further supported by a linear array on the $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ diagram, yielding a meaningful age of 1230 ± 47 Ma (MSWD=2.1), consistent with the Mesoproterozoic (~1265 Ma) formation of diabase TDB-1. In comparison, excellent reproducibilites for all HSEs were obtained from USGS reference material BIR-1: RSDs for 1-2 g aliquots were 7.4% Os, 4.4% Ir, 1.5% Ru, 5.2% Pt, 1.6% Pd and 0.7% Re (n=8). Thus, BIR-1 might be suitable material for narrowing the confidence intervals of HSE certified values.

Metasomatism recorded in the peridotite overlying metamorphic sole of the the Oman ophiolite: an analog of mantle-wedge events

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Mantle peridotites have been modified to various extents at slab-mantle interface within the mantle wedge. To know the details of the event, e.g., which elements and how much amounts?, the materials from the mantle wedge are needed. Ophiolite is a place where we can investigate the slab-mantle interaction, because it has the metamorphic sole, formed at the ophiolite obduction. Its formation is interpreted as an analog of an incipient arc system transformed from the ocean floor.

The Oman ophiolite is quite famous for good exposure and preservation of the oceanic stratigraphy with widespread formation of metamorphic sole. We did systematic sampling at the southern Oman ophiolite from the sole garnet amphibolite to the peridotite up to 110 m above the amphibolite/peridotite boundary, i.e., an analog of slab-mantle boundary. The peridotite is deformed and serpentinized to various degree, and occasionally has mylonitized bands. The peridotite protolith is fertile lherzolite: the Fo content of olivine and Cr# [= Cr/ (Cr + Al) cation ratio] of chromian spinel is 90-93 and 0.11-0.35 (except for the spinels in highly serpentinized peridotite), respectively. The Al_2O_3 contents of clinopyroxenes and orthpyroxenes are high (up to 5.3 and 6.3 wt.%, respectively). Fine pyroxene grains show a low Al_2O_3 content, < 1 wt.%. The peridotites contain amphiboles irrespective of the distance from the amphibolite/peridotite boundary, and the amphiboles are not only tremolite after clinopyroxene but also hornblendes. The incompatible trace-element patterns of clinopyroxene are enriched in LREE and some LILE (Rb, Ba and Sr), and those of hornblendes show similar patterns although some show higher abundances than clinopyroxene.

The peridotites just above the metamophic sole have been cooled as well as extensively hydrated (at least 110 m above the boundary) and enriched in fluid-mobile elements as mantle-wedge peridotites.

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