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Hydrofluoric acid is essential for accurate Re-Os isotopic analysis

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Complete digestion and dissolution, as well as complete recovery of elements of interest, are essential for accurate measurements; in particular when parent and daughter isotopes amount ratios are to be determined. In the beginning of Re and Os analysis, in the mid to end of the 1950's, alkali fusion was the dominant means of liberating and dissolving all Os containing mineral phases without oxidising Os to a volatile species. After utilising acid digestion under reducing conditions (HCl, HF and ethanol) in the 1980's, the rediscovery of Carius tubes (used in the 1940's for platinum-group element dissolution) permitted the use of concentrated HCl and HNO3 only, and was thought to be a major step forward towards complete digestion and spike – analyte equilibration for Re as well as for Os in the 1990's, due to its oxidising nature. Later, it was observed that Carius tube (CT) digestion at ca. 230 °C was not always sufficient for complete Os dissolution in ultramafic samples, particularly harzburgites. By the end of the 1990's it was demonstrated that a minimum temperature of 300 °C was essential in the case of harzburgites. These conditions were achieved either with a High Pressure Asher (320 °C) or with high temperature Carius tubes (350 °C). This digestion method was validated with various kinds of peridotites and complete recovery of Re and Os was demonstrated by also applying fusion as an independent method of sample digestion. Thus, acid digestions under high pressure and temperature are currently assumed to be the state of the art for isotope analysis within Re-Os isotope system.
However, this is not necessarily the case for more silica-rich crustal samples. An inter laboratory study in the early 2000's on MORB (EN026-10D3) already demonstrated that digestion with CT and HPA-S without hydrofluoric acid led to a lower recovery of Re and Pd. Bottle to bottle variation was thought to be the problem at that time. Recent more systematic studies on basalts shed further doubts on the ability of hydrochloric and nitric acid to liberate all the Re from, in particular, fresh silicates e.g. pyroxenes. It can be demonstrated that the use of HF is essential to completely recover all Re from pyroxene-rich rock matrices. Thus, the sole application of CT or HPA-S without the use of HF cannot assure complete digestion for Re and Os. Currently there is an attempt to certify peridotites for all PGE, Re and Au according to ISO guideline, but certification of a basalt is currently out of reach. Reference material TDB-1 is not certified for Re and Os, but is currently the best alternative for method development.