

## A highly efficient method to reduce nitric acid Os content to femtogram levels by hydrogen peroxide

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The Re-Os system has been used to constrain the age of ore deposits, meteorites, organic-rich shales and hydrocarbon systems, and to provide insights on paleo-climate change and mantle processes [1]. However, as smaller geological samples with lower Re and Os concentrations present new analytical challenges, extremely low total Re and Os levels require intense scrutiny of blanks. Accurate characterization of Re and Os blanks is critical to working with low level samples.

Here we present an investigation in reducing nitric acid's Os concentration as this is the main contributor to Os blank. The treatment of nitric acid proceeds in three steps: bubbling, volatilization and heating. The duration of the bubbling step is a useful proxy to estimate the Os liberated from nitric acid. Longer reaction of the bubbling step correlates with lower Os blanks in nitric acid. A series of key variables controlling reaction time of the bubbling step including nitric to peroxide ratios, the effect of wet versus dry glassware, and a dark versus lit reaction environment were studied. The ratio of H<sub>2</sub>O<sub>2</sub>:HNO<sub>3</sub> exerts the dominant control on reaction time and final Os content based on a well-defined exponential relationship, whereas the use of a dry reaction beaker or the absence of fluorescent light during the bubbling step may further extend the duration of bubbling and hence reduce Os blanks. At a H<sub>2</sub>O<sub>2</sub>:HNO<sub>3</sub> ratio of 1:4, the total procedural Os blank is reduced >99% from 6.5 pg (no H<sub>2</sub>O<sub>2</sub>) to 0.043 pg (based on GFS Veritas double distilled 69% nitric acid feedstock). The <sup>187</sup>Os/<sup>188</sup>Os of the Os blank ranges from 0.18 to 0.36, consistent with Os blank compositions obtained by the AIRIE Program and other Re-Os labs worldwide and is uncorrelated with all experimental variables.

In contrast to Os, pre-treatment with hydrogen peroxide did not improve the Re blank of nitric acid; reducing Re background requires conventional methods such as sub-boiling distillation.

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[1] Shirey and Walker, (1998) *J. Annu. Rev. Earth Planet. Sci.* **26**, 423–500.