

Low-level $^{187}\text{Os}/^{188}\text{Os}$ analysis by laser ablation, multi-ion-counting ICPMS

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Precise $^{187}\text{Os}/^{188}\text{Os}$ analysis of <0.1 ng Os traditionally has been achieved by negative thermal ionization mass spectrometry (N-TIMS). Though N-TIMS remains the most precise method for Os isotope analyses, its time-consuming nature makes generating large numbers of precise Os isotope data at low concentration levels expensive and time-consuming. Some applications, particularly in low-temperature geochemistry, require large quantities of high-quality data. For instance, the short marine residence time of Os causes variations in $^{187}\text{Os}/^{188}\text{Os}$ on time-scales of a few thousand years. Consequently, >10,000 data points are required to fully characterize the marine Os isotope record of the Cenozoic alone.

We present a new method for analyzing small quantities (>5 pg) of Os by multiple ion counting ICPMS. In a multi-dynamic measurement procedure Os isotopes are simultaneously detected using continuous dynode electron multipliers on a Finnigan NEPTUNE multicollector ICPMS. Osmium is separated and purified by conventional methods (sparging or micro-distillation) and loaded with a micro-pipette in small (sub-mm) depressions onto a carrier substrate. After drying on a hotplate at low temperature, the substrate is placed in a laser ablation cell and ablated with a 213 nm laser beam operated in pre-programmed raster mode. Each analyte spot is ablated in less than one minute, causing a transient signal in the ion counters of several hundred cps on isotope ^{187}Os . The use of a transient signal is advantageous for very small amounts of analyte as it improves the signal to noise ratio and allows for an internal precision in $^{187}\text{Os}/^{188}\text{Os}$ of 1-2%, even at total analyte amounts of ~5 pg. This is more than an order of magnitude better than with conventional sparging using a single-collector Finnigan ELEMENT2. The method also does not suffer from washout problems. We are currently working towards refining this method for the routine analysis of low-level samples (e.g., natural waters).

In-situ analysis of Os and Pb isotope ratios using laser ablation and collision-cell quadrupole ICP-MS

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Introduction

The use of a conventional (quadrupole) ICP-MS with laser ablation for precise, *in-situ* Os and Pb isotope measurements have been investigated. A hexapole collision cell, with a small gas flow (<2ml/min) has been used in order to improve accuracy and precision. The collision cell produces effects known as collisional focusing, which improves sensitivity, and collisional damping, which improves isotope ratio precision. By using both collisional focusing and damping, *in-situ* variations in Os and Pb isotope ratios in magmatic minerals have been measured.

Method Description

By introducing a low gas flow into the hexapole collision cell of an ICP-MS, ion energy spread is reduced. More ions are focused through the quadrupole leading to higher transmission and thus, improved sensitivity (collisional focusing). Collisions between ions and the collision cell gas reduces the kinetic energy of the ions. Each ion packet spends longer in transmission (collisional damping). Adjusting the hexapole and quadrupole settings to approximately match the rate of ion transmission leads to improved isotope ratio precision. Laser conditions were optimized by adjusting laser frequency, power, spot size and using various raster patterns or static spots. Sulfides were calibrated using the NiS reference material PGE-A for Os-isotopes. NiS beads, spiked with NIST SRM 981 and 982 Pb-isotope reference materials, were used to calibrate for Pb-isotopes. For silicates, the Pb-isotope ratios of NIST 610 and 612 glasses were used for calibration.

Results and Applications

Precision for $^{187}\text{Os}/^{188}\text{Os}$ and Pb-isotope ratios in sulfides varies with Os- and Pb-contents. Errors range from 0.25%-2% (2σ). Errors for Pb-isotopes in silicates are also related to Pb-contents, ranging from 0.3%-2.5% (2σ). Mineral-scale records of crustal contamination and assimilation in sulfide and silicate phases were examined from the Duluth, Bushveld and Muskox Intrusive complexes. Sulfides from the Duluth Complex show a wide range of Os- and Pb-isotope ratios. The variations recorded are consistent with a model of sulfur sequestration from crustal sources during magma emplacement.