Nu TIMS

Nu TIMS Note **TI05**

Nu TIMS high precision osmium isotope measurements

- **Negative ion operation.**
- **Excellent reproducibility of 2 - 50 ng osmium standard DROsS.**
- **Excellent peak shape and alignment.**

Since the discovery that osmium would ionize more effectively in the negative ion mode as an oxide, the Re-Os decay scheme has been widely used as a dating tool and geochemical tracer. More recently, with improvements in instrument technology, the Pt-Os decay scheme has also been utilized to provide unique information in combination with Re-Os data.

The isotopic measurement of osmium is performed using the OsO_3^- negative ion. It is generated using a single platinum filament with a barium hydroxide coating to lower the work function of the filament and enhance the ratio of negative ions to electrons.

The TIMS instrument with Zoom Optics allows ultimate alignment of isotopes. This facilitates flexibility allowing multiple static measurement, which are averaged to minimize any effects from pre-amplifier gain or collector efficiencies. Each measurement line can be tuned for best alignment and instantly switched electronically to the next line in milliseconds during the measurement. This ensures the highest accuracy and precision without the need for movable detectors.

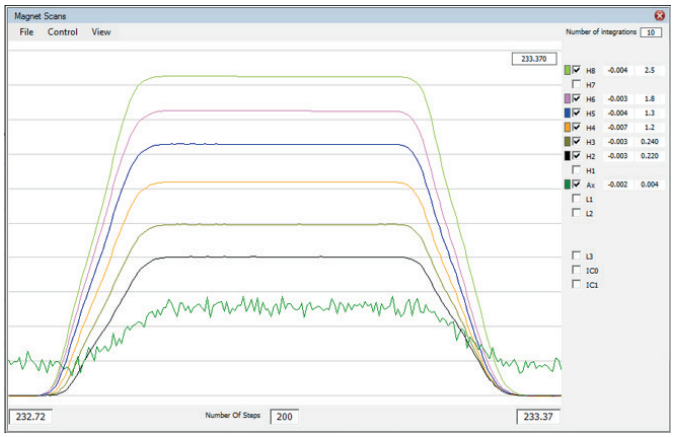

Figure 1: Alignment of osmium peaks

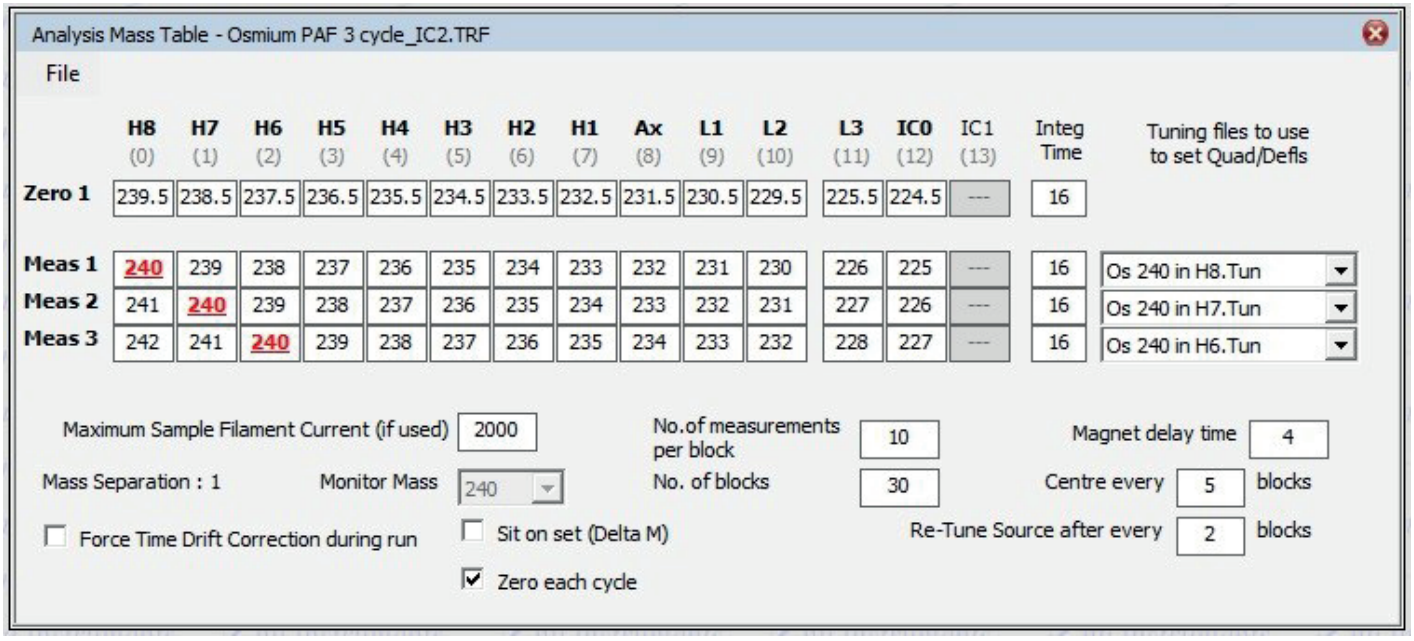

Figure 2: Detector configuration and measurement parameters for measurements of osmium

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Experiment

In order to avoid interferences from rhenium, platinum filaments were used for the analysis. The platinum ribbon was cleaned in acetone in an ultrasound bath and then rinsed 3 times with de-ionised water before being dried in an oven at 80° C. The cleaned ribbon was made into single filaments using welding tips reserved for platinum only. Before loading osmium onto the filament, they were outgassed in air for 10 minutes at dull red. The osmium standard DROsS, in HCl matrix, was syringe loaded onto the filament and left to dry in air with no current through the filament. Approximately 0.5 µl of a saturated barium hydroxide solution was loaded on top of the dried osmium and the filaments raised to a current of 0.8 Amps to dry.

A three-line analysis routine was used showing the flexibility of the Nu TIMS collector and minimizing any effects from pre-amplifier gain or collector efficiencies. Data was corrected for oxides and fractionation using an external measurement value of the oxygen gas obtained from an IRMS instrument and an exponential correction to $^{192}Os/^{188}Os$ = 3.083.

Parameters						
Sample	Filament Single	Analysis mode	Targer beam intensity	Warm up	Measurement duration	Baselines
2-50 ng DROsS with $Ba(OH)_2$ activator	Platinum	5-30 blocks of 10 measurements with 16 sec integration	^{192}Os at 0.5-2 V	~ 2 hours	1-6 hours	16 Seconds Each cycle

Table 1: Measurement parameters for the negative ion osmium runs

An oxygen bleed valve attached to the source chamber was used to provide a source of oxygen to assist the formation of the osmium as an oxide. The pressure in the source was maintained at approximately 1.5 x 10^{-7} mbar for the analysis.

The filament was ramped to 900 mA before optimizing to a target intensity between 0.5-2 V on Mass 240 ($^{192}Os^{16}O_3$). This was then held at the target value throughout the measurement using inter-block heating, with a peak center every 50 ratios and a source tune every 20 ratios. All isotopes were collected on Faraday detectors with 10^{11} Ohm resistors.

DROsS	$^{184}OS/^{188}OS$	$^{186}OS/^{188}OS$	$^{187}OS/^{188}OS$	$^{189}OS/^{188}OS$	$^{190}OS/^{188}OS$
N-TIMS[1] (2RSD) (n = 8)	0.001305 ± 5 (0.2%)	0.119929 ± 6 (25 ppm)	0.160924 ± 4 (12 ppm)	1.219705 ± 15 (6.1 ppm)	1.983803 ± 15 (3.8 ppm)
MC-ICP-MS[2] (2RSD) (n = 21)	0.001298 ± 2 (0.1%)	0.119920 ± 2 (8.3 ppm)	0.160924 ± 3 (9.3 ppm)	1.219722 ± 11 (4.5 ppm)	1.983788 ± 14 (3.5 ppm)
Nu TIMS 50ng (2RSD) (n = 5)	0.001303 ± 2 (0.18%)	0.119918 ± 3 (22 ppm)	0.160918 ± 4 (27 ppm)	1.219742 ± 5 (4.3 ppm)	1.983796 ± 10 (5.3 ppm)
Nu TIMS 20ng (2RSD) (n = 5)	0.001305 ± 7 (0.55%)	0.119920 ± 3 (24 ppm)	0.160918 ± 2 (12 ppm)	1.219740 ± 8 (7 ppm)	1.983800 ± 4 (2 ppm)
Nu TIMS 2ng (2RSD) (n = 10)	0.001339 ± 24 (1.9%)	0.119900 ± 28 (229 ppm)	0.160906 ± 22 (132 ppm)	1.219736 ± 40 (31 ppm)	1.983813 ± 54 (27 ppm)

Table 2: Comparison of data achieved in this study with that published in the literature. All Nu TIMS data is the static average value from the three measurement lines. All data is normalized to $^{192}Os/^{188}Os$ = 3.083. Uncertainty is reported as ±2 Σ, and 2 RSD in brackets.

1: Luguet et al. Chemical Geology 248 (2008) 342-362, 2: Nowell et al. Chemical Geology 248 (2008) 363-393.

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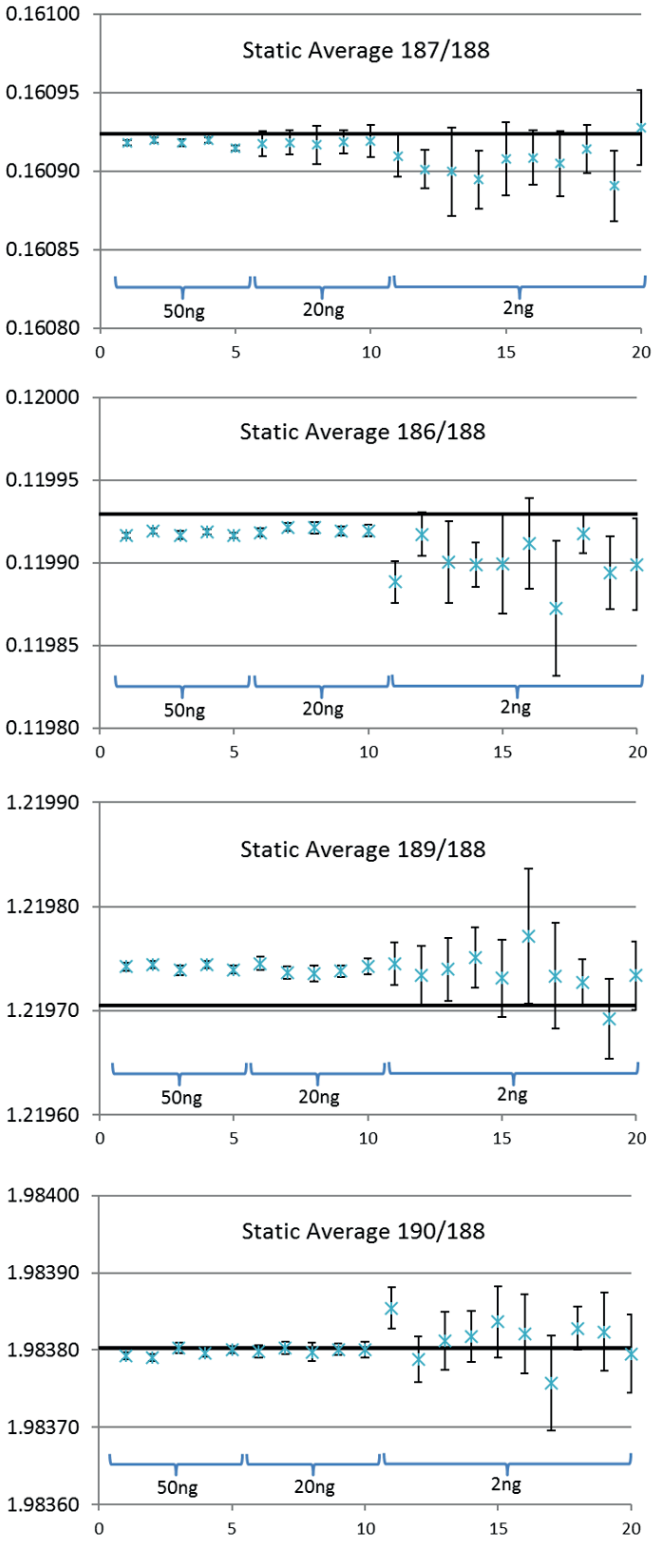

Figure 3: A combination of the data for the different size loads for each ratio. The black line represents the values from Luguet et al. Chemical Geology 248 (2008) 342-362. Error bars are reported as ±2 Σ.

Conclusion

Isotopic measurements of osmium have been performed in the negative ion mode using a Nu TIMS to produce results that are comparable in precision to osmium isotopic data in the literature for the DROsS standard.

The values measured in this application note were not corrected for platinum oxide interferences.