Application Note: 30016

High Precision Osmium Isotope Analyses

Negative Thermal Ionization Mass Spectrometry and Static Faraday Cup Multicollection

Dietmar Tuttas, Thermo Fisher Scientific, Bremen, Germany

Key Words

- TRITON
- Faraday Mode
- Negative Ions
- Static Mode
- Osmium
- TI-MS

Introduction

The rhenium-osmium geochronometer (^{187}Re decays by beta-emission to ^{187}Os with a half-life of ca 42 Byr) is of significant interest to the geochemical community. Early on, broad use of this chronometer was hindered by the lack of sensitive techniques to precisely measure isotopic abundances in small osmium samples.

In 1989, the group of Heumann (University of Regensburg, Germany) pioneered the analytical efforts in Os isotopic analysis by **N-TI-MS (Negative Thermal Ionization Mass Spectrometry)** with ion per atom yields of ca. 25% (e.g. Voelkening et al., 1991). Shortly after, the group of Wasserburg (Caltech, USA) and others have improved chemistry and sample loading, with the result that N-TI-MS has evolved into an established tool for radiochronology (e.g. Creaser et al., 1991).

N-TI-MS is a powerful ionization technique for a broad range of elements. Many of the transition metals form negatively charged oxide ions. High ion yields can be obtained for elements with high electron affinities. The ion current is essentially influenced by the electron work function of the filament material, which can be substantially reduced by covering the filament with, for instance, barium.

For osmium analyses, platinum filaments are required. Because platinum very often contains considerable amounts of osmium, rhenium and tungsten, it is essential to use ultra pure platinum material.

Ion currents can be significantly increased by oxygen introduction during sample heat-up and data collection via a viscous flow oxygen bleed inlet system.

Because OsO_3^- -ions are formed and collected, a correction for the oxygen isotope contribution is required as well. Osmium isotopic fractionation corrections are made using the constant ratio of ^{192}Os/^{188}Os = 3.092203.

Sample amounts in the low nanogram range can be easily analyzed in multicollector Faraday cup operation mode. Using the advantages of single collector or even multiple collector ion counting, sample amounts in the picogram range can be analyzed with excellent precision.

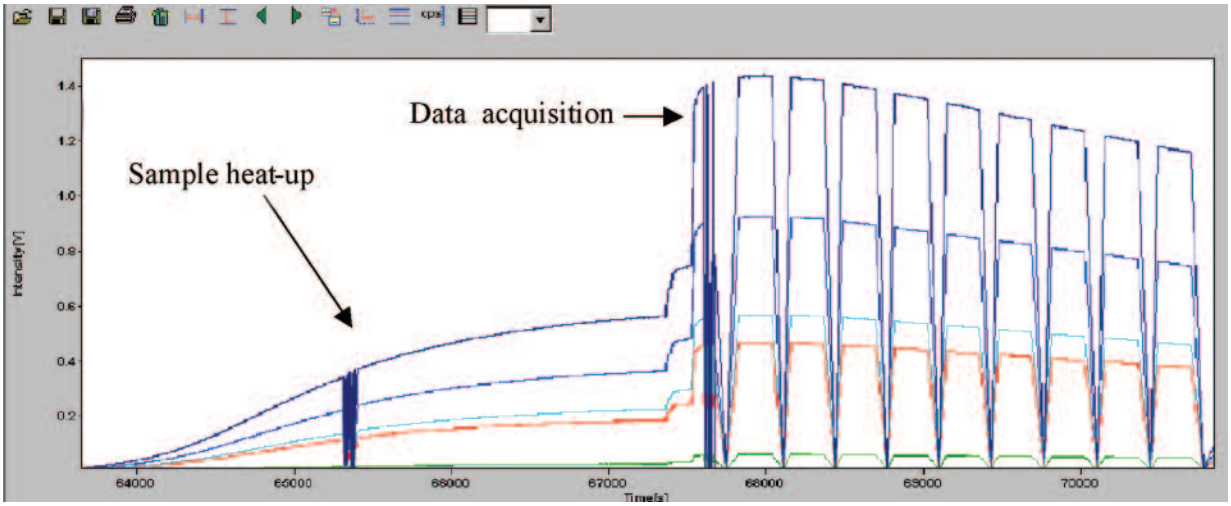
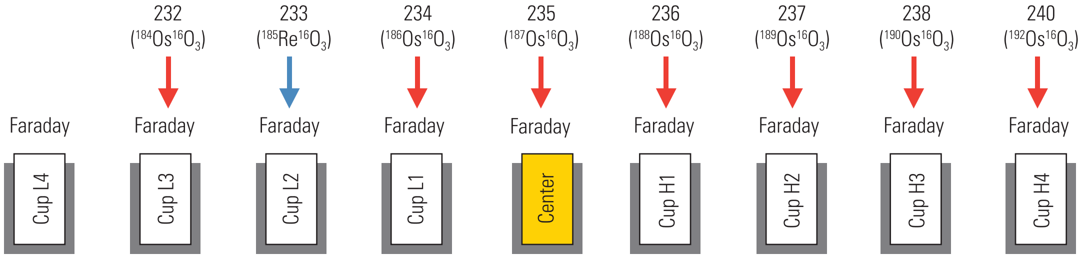


Figure 1. Osmium isotope signals during heat-up procedure and acquisition.

Schematic of the Multi-Collector Configuration

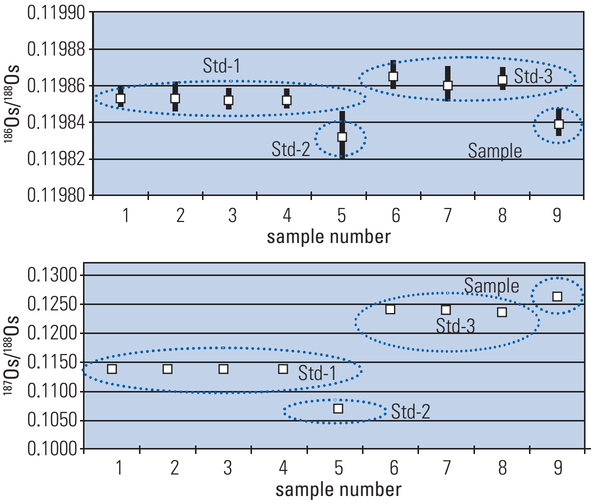


Analysis Parameter

Samples: 1-35 ng of Os loaded onto a platinum filament together with barium nitrate (e.g. Creaser et al, 1991).

Sample Warm-up: performed very slowly while introducing oxygen via bleed system, filament currents around 2000 mA, intensities 500-2500 mV.

Data Acquisition: Negative ion mode at 10 kV, static Faraday ion multicollection mode, channel gains calibrated once. Typically 300 single data collected with an integration time of 10 s. Rhenium interference controlled by measuring ^{185}Re (m/e = 233). Isotopic fractionation correction using ^{192}Os/^{188}Os = 3.092203, followed by oxide correction.



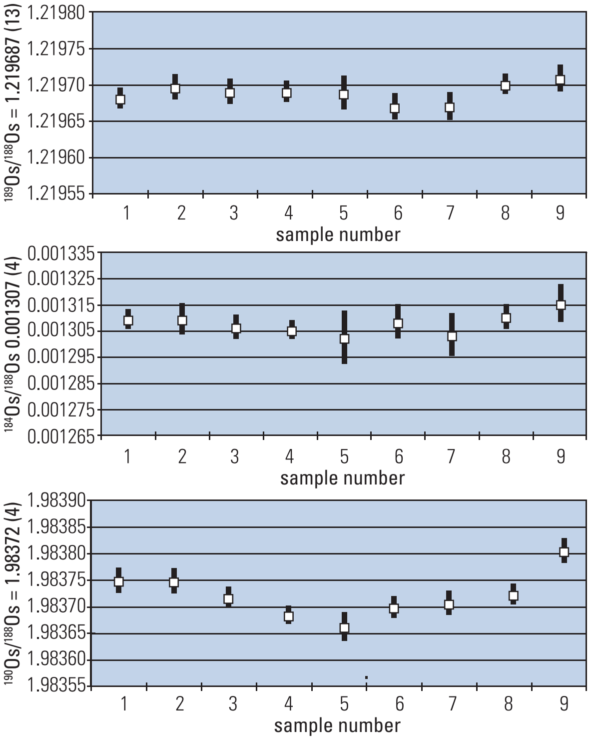


Figure 2. Os isotopic ratios in 3 standards and 1 sample. Errors are 2SE. See Table for details.

All final data corrected for outliers by 2-sigma-criterion. Total sample analysis time 2-3 hours including sample warm-up.

Summary of Different Osmium Isotopic Analyses

RUN #	SAMPLE	^{184}Os/^{188}Os	1SE	^{186}Os/^{188}Os	1SE	^{187}Os/^{188}Os	1SE	^{189}Os/^{188}Os	1SE	^{190}Os/^{188}Os	1SE
1	Std-1 #1 (35ng)	0.001309	1	0.119853	2	0.113807	2	1.219680	5	1.983747	10
2	Std-1 #2 (35ng)	0.001309	3	0.119853	3	0.113799	3	1.219695	7	1.983746	10
3	Std-1 #3 (35ng)	0.001306	2	0.119852	2	0.113797	3	1.219689	7	1.983715	7
4	Std-1 #4 (35ng)	0.001305	1	0.119852	2	0.113796	2	1.219689	6	1.983682	6
5	Std-2	0.001302	5	0.119832	6	0.107010	16	1.219687	10	1.983660	11
6	Std-3 #1	0.001308	3	0.119865	3	0.124075	5	1.219668	7	1.983697	8
7	Std-3 #2	0.001303	4	0.119860	4	0.123972	8	1.219669	8	1.983704	9
8	Std-3 #3	0.001310	2	0.119863	2	0.123541	2	1.219699	5	1.983721	7
9	Os AP-5 Sample	0.001315	3	0.119839	3	0.126231	3	1.219707	7	1.983803	9
Mean of 1SE (internal)			2.7		3.0		4,9		6,9		8.6
Mean of runs **1SD (external)**		**0.001307 (n=9)** **0.000004**		**0.119853 (n=4)** **0.000001**		**0.113800 (n=4)** **0.000005**		**1.219687 (n=9)** **0.000013**		**1.983719 (n=9)** **0.000042**	
1RSD (external)		**0.30%**		**4.8 ppm**		**43.9 ppm**		**10.7 ppm**		**21.2 ppm**	

Notes: Different samples vary in ^{186}Os and ^{187}Os abundances. The gray shaded fields in the table indicate the statistics calculated based solely on the samples, which appear shaded. Normalization using: $^{192}OsO_3/^{188}OsO_3$ = 3.092203. Oxide correction using: $^{17}O/^{16}O$ = 0.0003708, $^{18}O/^{16}O$ = 0.002045

References

Creaser R.A., Papanastassiou D.A. and Wasserburg G.J. (1991) Geochimica et Cosmochimica Acta, 55, 397-401. Voelkening J., Walczyk T. and Heumann K.G. (1991) International Journal of Mass Spectrometry and Ion Processes, 105, 147-159.

www.thermoscientific.com/IRMS

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

Africa-Other	+27 11 570 1840
Australia	+61 3 9757 4300
Austria	+43 1 333 50 34 0
Belgium	+32 53 73 42 41
Canada	+1 800 530 8447
China	+86 10 8419 3588
Denmark	+45 70 23 62 60
Europe-Other	+43 1 333 50 34 0
Finland/Norway/Sweden	+46 8 556 468 00
France	+33 1 60 92 48 00
Germany	+49 6103 408 1014
India	+91 22 6742 9434
Italy	+39 02 950 591
Japan	+81 45 453 9100
Latin America	+1 561 688 8700
Middle East	+43 1 333 50 34 0
Netherlands	+31 76 579 55 55
New Zealand	+64 9 980 6700
Russia/CIS	+43 1 333 50 34 0
South Africa	+27 11 570 1840
Spain	+34 914 845 965
Switzerland	+41 61 716 77 00
UK	+44 1442 233555
USA	+1 800 532 4752

Thermo Fisher Scientific (Bremen) GmbH Management System Registered to ISO 9001:2008
Thermo Fisher Scientific, San Jose, CA USA is ISO Certified.

AN30016_E 01/12G