Application Note: AN30016_E

High Precision Osmium Isotope Analyses

Negative Thermal Ionization and Static Multiple Ion Collection

Dietmar Tuttas, Thermo Electron (Bremen) GmbH, Barkhausenstrasse 2, 28197 Bremen, Germany

Key Words

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

The rhenium-osmium geochronometer (beta-decay of ^{187}Re to the stable ^{187}Os) 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 from small osmium samples.

In 1989, Heumann et al. (University of Regensburg, Germany) pioneered the possibility to measure osmium isotopic ratios with **NTI-MS** (**Negative Thermal Ionization Mass Spectrometry**). Shortly after this, the group of Wasserburg et al. (Caltech, USA) and others have improved chemistry and sample loading, with the result that this technique has evolved into an established tool for use by the geological community.

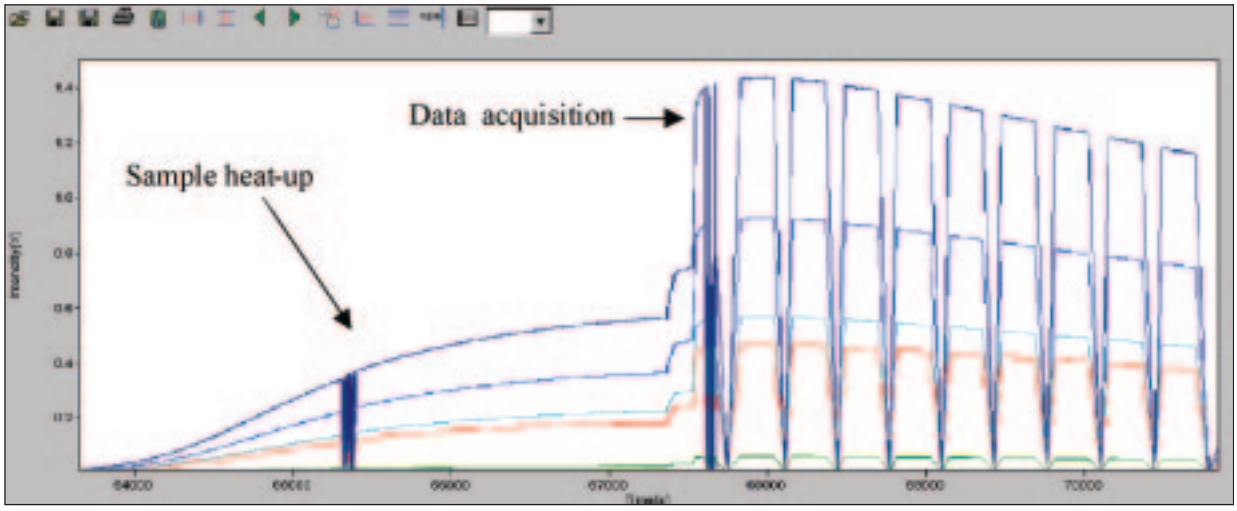
NTI is a powerful ionization technique for a broad range of elements. Many of the transition metals form negatively charged oxide ions. High ion yields can be obtained for elements with high electron affinities. The ion current is essentially influenced by the electron work function of the filament material, 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.

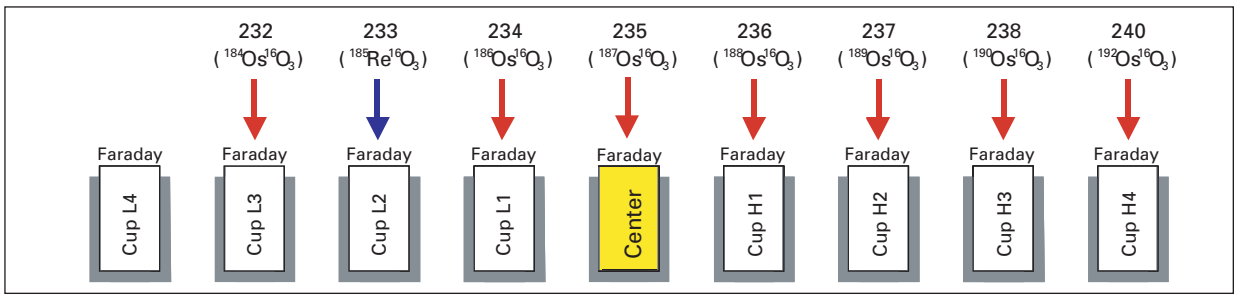
Isotopic fractionation corrections are made using the constant ratio of $^{192}Os/^{188}Os$. Because OsO_3^- -ions are formed and collected, a correction for the oxygen isotope contribution is required as well.

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.

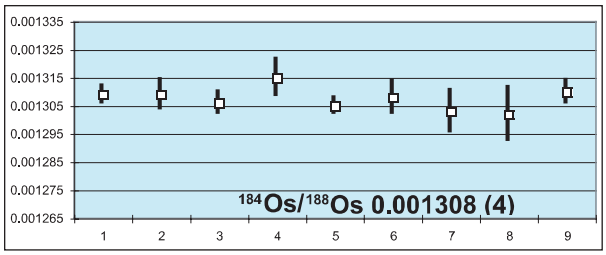


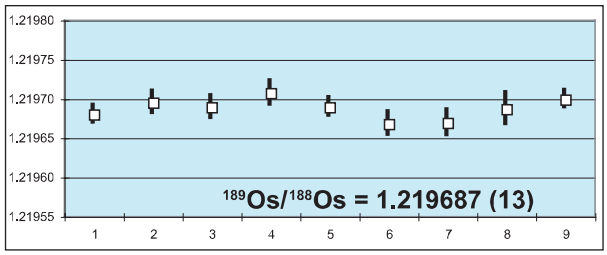
Isotope Traces of an Osmium Measurement

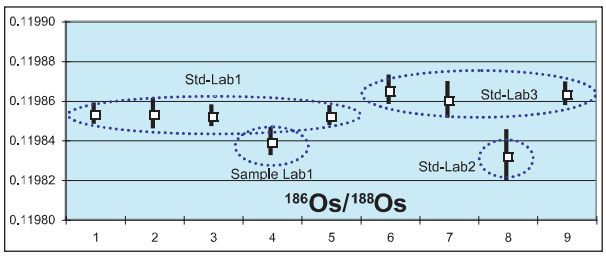
Schematic of the Multi-Collector Configuration

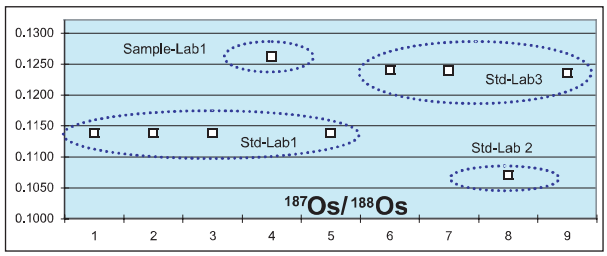


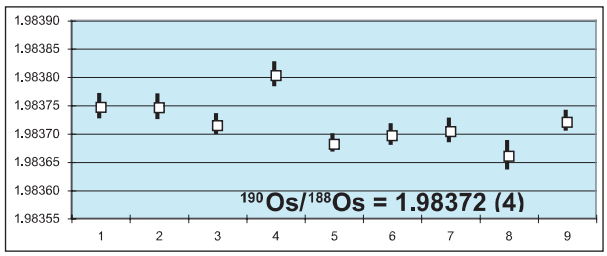
Thermo ELECTRON CORPORATION











Analysis Parameter

Samples: 1-35 ng loaded onto a platinum filament together with barium nitrate (Ref: Papanastassiou, Sharma, Brandon et al.)

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 collection mode, channel gains calibrated once. Typically collected 300 single data with integration times of 10 s each. Rhenium interference controlled by measuring ^{185}Re (m/e=233). Isotopic fractionation correction using 240/236=3.092203, followed by oxide correction.

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 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 (Lab#1) 35ng #1	0.001309	1	0.119853	2	0.113807	2	1.219680	5	1.983747	10
2	STD (Lab#1) (3-5ng) #2	0.001309	3	0.119853	3	0.113799	3	1.219695	7	1.983746	10
3	STD (Lab#1) (3-5ng) #3	0.001306	2	0.119852	2	0.113797	3	1.219689	7	1.983715	7
4	Os AP-5 Sample (Lab#1) #4	0.001315	3	0.119839	3	0.126231	3	1.219707	7	1.983803	9
5	STD (Lab#1) 35ng (2.6V) #5	0.001305	1	0.119852	2	0.113796	2	1.219689	6	1.983682	6
6	STD (DT-FMAT) #1	0.001308	3	0.119865	3	0.124075	5	1.219668	7	1.983697	8
7	STD (DT-FMAT) #2	0.001303	4	0.119860	4	0.123972	8	1.219669	8	1.983704	9
8	STD (Lab#2) #1	0.001302	5	0.119832	6	0.107010	16	1.219687	10	1.983660	11
9	STD (DT-FMAT) #3	0.001310	2	0.119863	2	0.123541	2	1.219699	5	1.983721	7
Averaged SE(int)			2.6		3.1		5.0		7.0		8.6
Mean		**0.001307**		**0.119853**		**0.113800**		**1.219687**		**1.983719**	
SD (external)		**0.000004**		**0.000001**		**0.000005**		**0.000013**		**0.000042**	
RSD (external)		**0.30 %**		**4.8 ppm**		**43.9 ppm**		**10.7 ppm**		**21.2 ppm**	
Normalization using: $^{192}OsO_3/^{188}OsO_3$ = 3.092203				Above mean values and statistics are of samples #1,2,3,5 (Laboratory #1)				Oxide correction using: $^{17}O/^{16}O$ = 0.0003708, $^{18}O/^{16}O$ = 0.002045			

Note: Different samples vary in ^{186}Os and ^{187}Os abundances. The gray shaded fields in the table indicate that the statistics have only been calculated based on the samples, which appear shaded in the table as well.

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

Australia
+61 2 9898 1244
Austria
+43 1 333 50340
Belgium
+32 2 482 30 30
Canada
+1 800 532 4752
China
+86 10 5850 3588
France
+33 1 60 92 48 00
Germany
+49 6103 4080
Italy
+39 02 950 591
Japan
+81 45 453 9100
Netherlands
+31 76 587 98 88
Nordic
+46 8 556 468 00
South Africa
+27 11 570 1840
Spain
+34 91 657 4930
Switzerland
+41 61 48784 00
UK
+44 1442 233555
USA
+1 800 532 4752

www.thermo.com

Thermo Electron (Bremen) GmbH is certified DIN EN ISO 9001:2000

AN30016_E 12/03C

Thermo
ELECTRON CORPORATION