Gain Calibration Protocol for 10^{13} Ω Resistor Current Amplifiers using the Certified Neodymium Standard JNdi-1 on the TRITON *Plus*

Anne Trinquier, Thermo Fisher Scientific, Bremen, Germany

Key Words
10^{13} Ω current amplifiers, 10^{11} Ω current amplifiers, gain calibration, TRITON *Plus*, thermal ionization MS, static mode, neodymium, JNdi-1

Introduction

Newly developed 10^{13} Ω current amplifiers,[1] allow analysis of minor ion beam intensities in the range 3aA (3.10^{-18} A, equivalent to 2000 cps on a ion counter) to 0.5 fA (5.10^{-16} A) on Faraday cups. They can be used alone when all ion beam intensities are low, or in combination with 10^{11} Ω, 10^{12} Ω or 10^{10} Ω current amplifiers, for measuring the ion beams of the minor isotopes from elements with extreme isotopic ratios (e.g. Ca, U, Sr) and/or for simultaneously collecting isobaric interfering species, thus providing precise and accurate isobaric interference correction.

Selective virtual amplifier concept[2]

Following the virtual amplifier concept, during analysis, all cups are sequentially connected to all active amplifiers, all ion beams are in turn measured with the same set of amplifiers, and the stochastic calibration biases of the amplifiers can be averaged out. The virtual amplifier thus reduces the propagation of the uncertainty of the gain calibration procedure. With the introduction of high resistor current amplifiers (10^{12} and 10^{13} Ω current amplifiers), a selective virtual amplifier tool has been developed that allows sequentially connecting all active 10^{11} Ω amplifiers to all corresponding cups, while maintaining the connection between the 10^{12} and 10^{13} Ω amplifiers to their respective cups. This selective virtual amplifier protocol allows simultaneously high-precision and accurate isotopic ratios assessment (if one takes the example of neodymium, all Nd isotopes[4–5]) and precise and accurate analysis of minor isotopes of the element of interest and/or isobaric interfering species (in this example: Ce, Sm).

The present technical note presents a gain calibration protocol for 10^{13} Ω resistor current amplifiers using the certified neodymium standard JNdi-1 on the Thermo Scientific™ TRITON *Plus*™ Thermal Ionization MS (TIMS).

Choice of a Reference: Certified Neodymium Standard JNdi-1

Neodymium isotopic ratios obtained for JNdi-1 (Split 1 position 179, ref. 3) over 3 sessions of analyses serve as a reference for this study.[3] Neodymium has been chosen because up to 7 Nd isotopes can be measured simultaneously and to comparable ion beam intensities. JNdi-1 has been selected because it is a readily accessible standard with low levels of interfering contaminants (Ce, Sm). Mass bias corrected isotopic ratios $^{142}Nd/^{144}Nd$, $^{143}Nd/^{144}Nd$, $^{145}Nd/^{144}Nd$, $^{148}Nd/^{144}Nd$ and $^{150}Nd/^{144}Nd$ of JNdi-1 have been measured on 10^{11} Ω current amplifiers using the virtual amplifier, following the protocol previously described[4–5] and provide the reference isotopic ratios for this study. Three sessions of analysis[6] providing 2–5 ppm/amu external reproducibility (2RSD, n=15, 500 ng loads, 9.9 V $^{142}Nd^+$, July 2013), 1–2 ppm/amu (2RSD, n=3, 300-500 ng loads, 7.4 V $^{142}Nd^+$, January 2014) and 2–8 ppm/amu (2RSD, n=7, 200 ng loads, 4.6V $^{142}Nd^+$, February 2014) have been combined. The resulting external reproducibility of Nd isotopic ratios over the 3 sessions of analysis is 2–10 ppm/amu (2RSD, n=25, 200-500 ng loads).

ng	^{142}Nd (V)	^{142}Nd/^{144}ND	2RSD	2RSD (ppm)	^{143}Nd/^{144}ND	2RSD	2RSD (ppm)	^{145}Nd/^{144}ND	2RSD	2RSD (ppm)	^{148}Nd/^{144}ND	2RSD	2RSD (ppm)	^{150}Nd/^{144}ND	2RSD	2RSD (ppm)
400	8	1.141836	9E-06	8	0.512107	5E-06	9	0.348404	3E-06	10	0.241580	2E-06	9	0.236452	4E-06	17

Figure 1. Neodymium isotopic composition of JNdi-1.

The ratios ^{142}Nd/^{144}Nd and ^{143}Nd/^{144}Nd agree within respective uncertainty with literature: ^{142}Nd/^{144}Nd = 1.1418351 ± 42 (ref.7); ^{143}Nd/^{144}Nd = 0.512115 ± 7 (ref.3); ^{143}Nd/^{144}Nd = 0.512108 ± 6 (ref.8). All ratios agree within respective uncertainty with the data from ref. 9.

Line No.	Mass Set	L4	L3	L2	L1	RPQ/ IC1 C	H1	H2	H3	H4	Integration Time(s)	Number of Integrations	Idle Time(s)	Control Cup Peakcenter	Control Cup Focus
1	Main	^{140}Ce	^{142}Nd	^{143}Nd	^{144}Nd	^{145}Nd	^{146}Nd	^{147}Sm	^{148}Nd	^{150}Nd	16.77	1	18.000	^{144}Nd	^{144}Nd

Figure 2. Neodymium Cup configuration.

Gain Calibration Protocol for 10^{13} Ω Current Amplifiers

In the present study, 10^{11} Ω current amplifiers are connected to the cups collecting ^{144}Nd and ^{146}Nd. ^{144}Nd is the denominator of all Nd isotopic ratios and ^{144}Nd/^{146}Nd is the reference ratio used for instrumental mass bias correction, using the exponential law. Up to five 10^{13} Ω current amplifiers can be connected to masses ^{142}Nd, ^{143}Nd, ^{145}Nd, ^{148}Nd, ^{150}Nd, allowing up to five 10^{13} Ω current amplifiers gains to be assessed simultaneously. JNdi-1 loads of 200–500 ng are run at ion beam intensities <500 mV on ^{142}Nd$^+$ for 80 cycles, 17 s integration times and an analysis time of ca 40 min. The ratio between certified and mass bias corrected ^{i}Nd/^{144}Nd values multiplied by 0.01 (to account for the 2 order of magnitude difference between the 10^{11} and the 10^{13} Ω amplifiers) yields the amplifier gains.

Note: Ion beams measurable with 10^{13} Ω current amplifiers are highlighted in red, ion beams measured with 10^{11} Ω current amplifiers are highlighted in blue. In this study, up to four 10^{13} Ω current amplifiers gains have been assessed simultaneously using masses ^{142}Nd (cup L3), ^{143}Nd (cup L2), ^{145}Nd (cup C), ^{148}Nd (cup H3).

In this campaign of analyses, the gains of two 10^{13} Ω current amplifiers were assessed from the period September 2013 to February 2014 and the gains of two additional 10^{13} Ω current amplifiers were assessed from the period September to November 2013. Internal precision is on average better than 50 ppm (2RSE) and external reproducibility over 6 months is better than 100 ppm (2RSD).

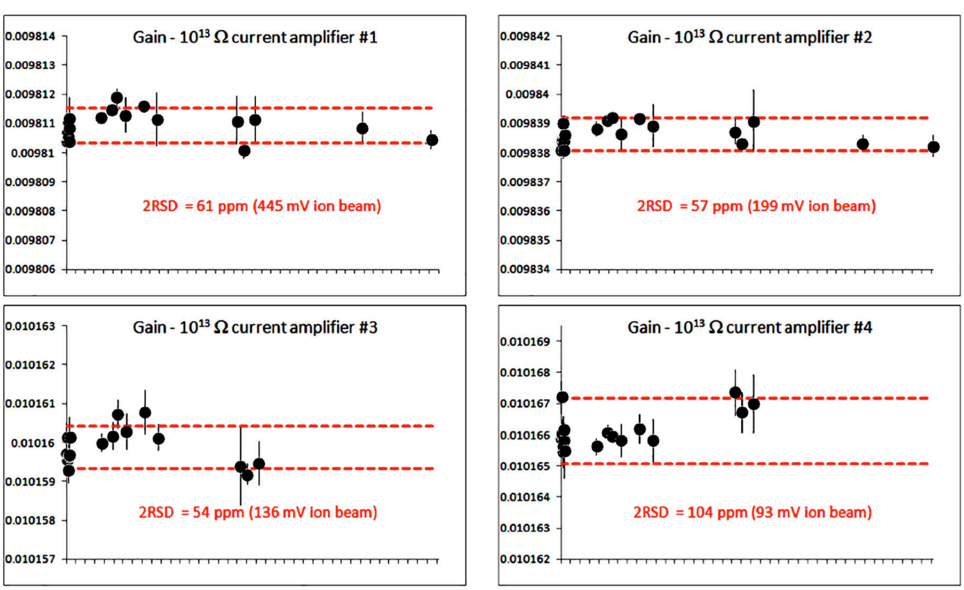

Figure 3. Gain calibration external reproducibility (2RSD) over 6 months (#1-2) and over 3 months (#3-4).

References

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