Application Note 30282

New Design 10^{13} Ω Amplifiers for Measurement of Small Ion Beam Currents

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Introduction

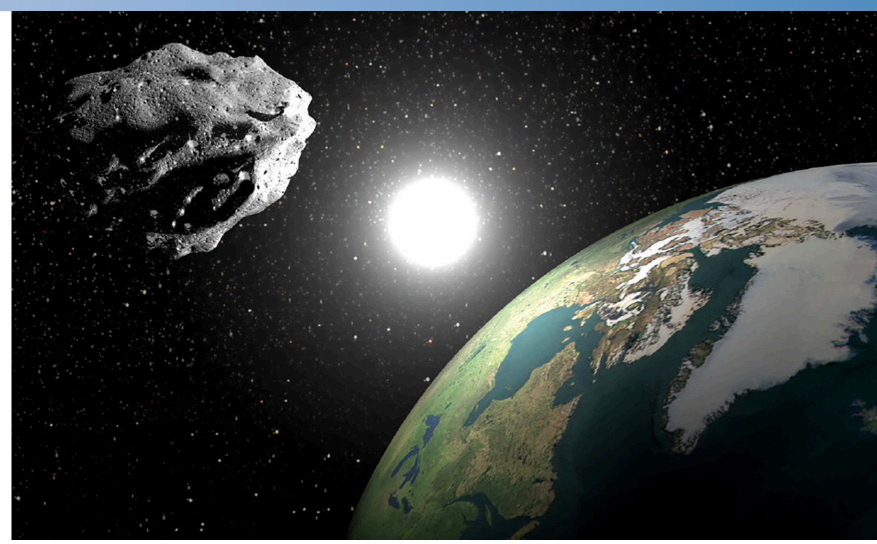
Numerous applications in Thermal Ionization MS (TIMS) and Multicollector Inductively Coupled Plasma MS (MC-ICP-MS) involve the analysis of small ion beam currents, down to the femto ampere range, which is equivalent to ion beam intensities in the range of less than 10,000 cps.

Examples of such applications are studies that focus on isotope compositions of scarce materials (e.g. dust in ice cores, inclusions in diamonds, or components of extra-terrestrial material), on materials that have ultra-low concentration of the isotopes of interest (e.g. Hf in depleted peridotite, or Re and Os in silicate rocks), low abundance isotopes such as ^{234}U, but also in studies that aim to resolve isotopic variability on a small spatial scale (e.g. growth zones in minerals, teeth or hairs).

Ultimately, the precision and accuracy of such studies are limited by the signal/noise ratio, the calibration of the detection system and the analytical blanks.

Here we present newly developed amplifiers equipped with 10^{13} Ω feedback resistors for the Thermo Scientific™ Triton Plus™ TIMS, Neptune Plus™ MC-ICP-MS and Noble Gas product line. A revolutionary new current amplifier design compensates for settling time effects observed with high-gain current amplifiers using high ohmic resistors in the feedback loop. The new design concept guarantees fast response times and very low noise characteristics.

By using a proprietary Relay Matrix, these amplifiers can be switched to any Faraday cup in the array without the need to open the amplifier housing.



Noise

The precision of analyses of small ion beam currents in multi-collection mass spectrometry is essentially limited by

(1) counting statistics, and

(2) the electrical Johnson-Nyquist noise of the resistor used in the feedback loop.

From the Johnson-Nyquist noise equation, it follows that if the resistor value R (and thus the output voltage V) is increased by a factor of 100, the noise will only increase by $\sqrt{100}$. This gives a theoretical improvement in signal/noise of 10, going from 10^{11} to 10^{13} Ω amplifiers.

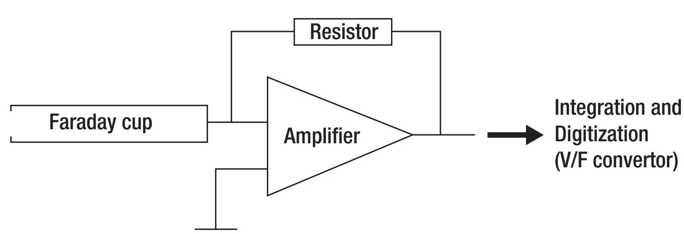


Figure 1. Schematic representation of the Faraday cup detection system. The Faraday cup is connected to electrical ground via an amplifier that is equipped with a high ohmic resistor. The amplified signal is converted to volts by a V/F converter. V = I x R, where V = voltage; I = ion current; R = resistivity. Current amplifiers can be equipped with resistors with R = 10^{10}, 10^{11}, 10^{12} or 10^{13} Ω.

Baseline

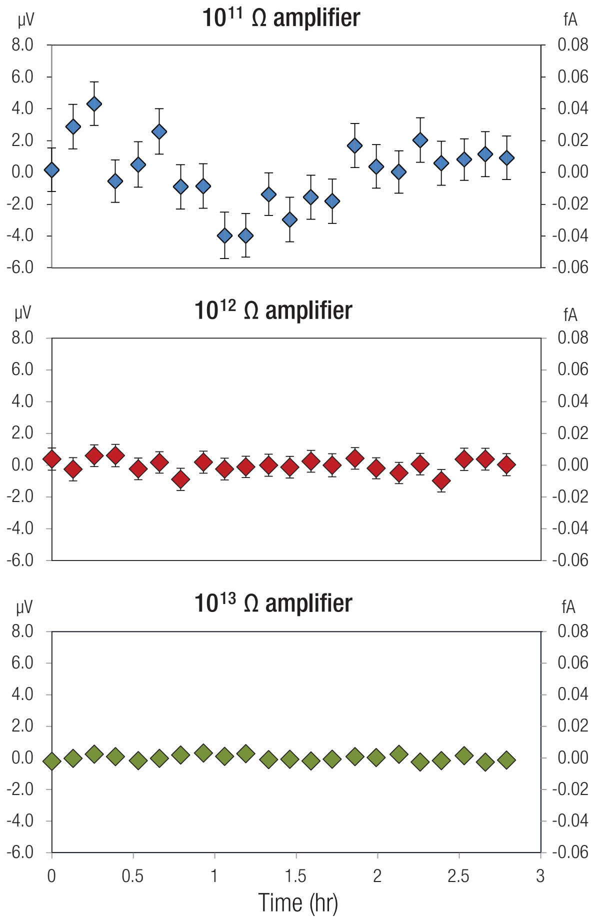


Figure 2. Reproducibility of 11 minutes baseline measurements on 10^{11}, 10^{12} and 10^{13} Ω amplifiers.

Figure 2 shows reproducibility of 11 minutes baseline measurements performed on the Triton Plus TIMS on 10^{11}, 10^{12} and 10^{13} Ω amplifiers.[1] The 1 SD (standard deviation) reproducibility for the 10^{13}, 10^{12} and 10^{11} Ω amplifiers are 0.2 µV, 0.4 µV and 2 µV (normalized to 10^{11} Ω), respectively.

Please note the two scales on the vertical axis; the left scale displays the voltage scale in µV (normalized to10^{11} Ω), whereas the right scale displays the ion current scale in pA.

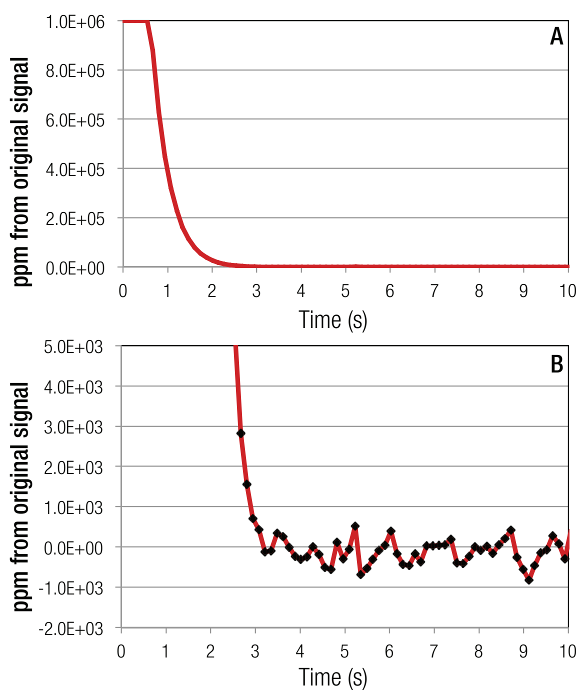


Figure 3. a) Typical signal decay for a 10^{13} Ω amplifier, b) same, but with adjusted y-axis scale (zoom-in).

Figure 3 shows a typical decay pattern for a 10^{13} Ω amplifier. The original signal was ~800 fA (i.e. 80 mV on 10^{11} Ω). Within 3 seconds, the amplifier has reached <100 ppm of the original signal.

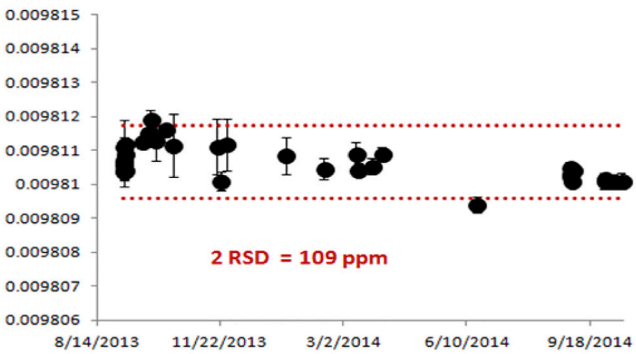


Figure 4. Long-term gain stability of a 10^{13} Ω amplifier.

Figure 4 shows the gain stability of a 10^{13} Ω amplifier over more than 1 year. Gains were assessed by measuring the Nd isotope ratio standard JNdi-1 on the Triton Plus (for more detail see Technical Note 30285).

Internal Precision

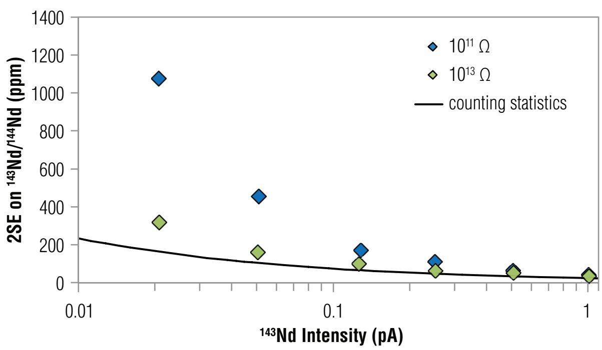


Figure 5. Internal precision on $^{143}Nd/^{144}Nd$ as function of ion beam intensity using 10^{11} and 10^{13} Ω amplifiers.

Figure 5 shows internal precision (2 SE) as a function of beam intensity on measured $^{143}Nd/^{144}Nd$ using 10^{11} Ω and 10^{13} Ω amplifiers compared to the error predicted from counting statistics (black line). These measurements were done on the Triton Plus TIMS.

External Precision

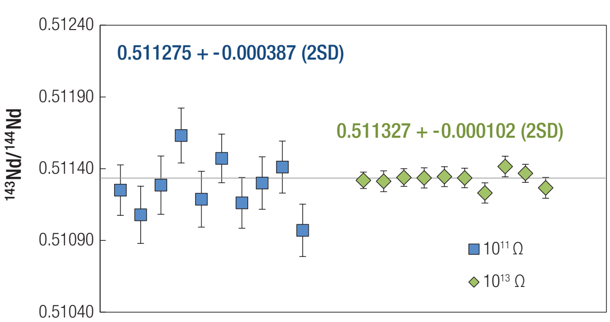


Figure 6. External reproducibility for $^{143}Nd/^{144}Nd$ isotope ratios measured on 10^{11} and 10^{13} Ω amplifiers.

Figure 6 shows external reproducibility of multiple analyses of $^{143}Nd/^{144}Nd$ ratios at 50 ftA ^{143}Nd beam intensity measured on the Triton Plus TIMS using 10^{11} Ω and 10^{13} amplifiers. The average and 2 SD of the data for each set of amplifiers are shown on the graph. The grey horizontal lines represent the long-term average of large (~100 ng) aliquots of the in-house standard CIGO ($^{143}Nd/^{144}Nd$ = 0.511334 +/– 0.000010, n=28) at the VU University in Amsterdam.

100 pg Nd Load

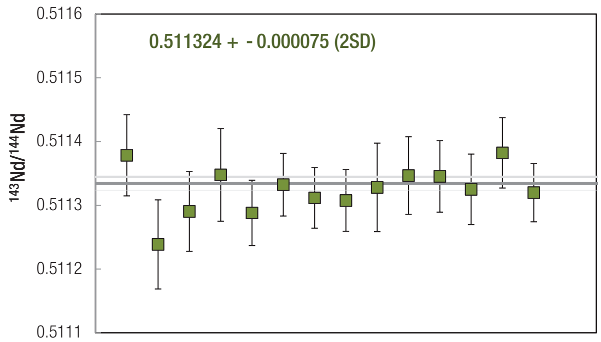


Figure 7. External reproducibility of $^{143}Nd/^{144}Nd$ on individual 100 pg Nd loads using 10^{13} Ω amplifiers.

Figure 7 shows external reproducibility of $^{143}Nd/^{144}Nd$ ratios measured on the Triton Plus TIMS on individual 100 pg loads of an in-house Nd isotope ratio standard ("CIGO") using 10^{13} Ω amplifiers. The average and 2 SD for the 100 pg samples are shown on the graph. Intensities varied around 200 fA (i.e. 20 µV relative to 10^{11} Ω). The dark horizontal lines represent the long-term average of large (~100 ng) aliquots of CIGO ($^{143}Nd/^{144}Nd$ = 0.511334 ± 10, n=28) at the VU University in Amsterdam.

Faraday vs SEM

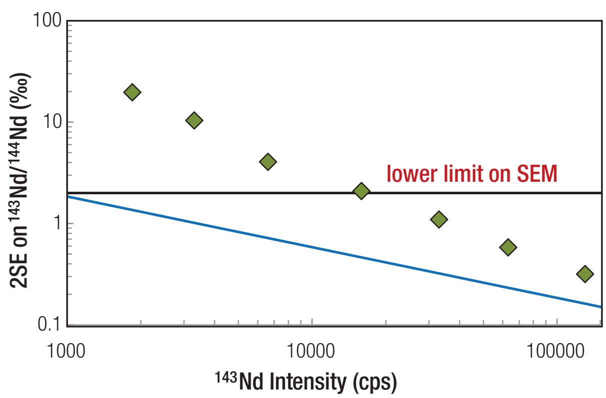


Figure 8. Comparison of the internal precision of $^{143}Nd/^{144}Nd$ on SEM vs 10^{13} Ω amplifiers.

Figure 8 shows a comparison of the internal precision (2 SE) of $^{143}Nd/^{144}Nd$ measurements on 10^{13} Ω resistors over the ^{143}Nd intensity range usually covered by secondary electron multiplier SEMs (1000 cps to 20k cps) (1000 cps to 20 kcps, i.e. 0.16–3.2 fA). The green squares show the precision of the 10^{13} Ω amplifier Nd measurement at different ion beam intensities. The horizontal black line describes the ion counter/Faraday cup cross calibration uncertainty, which is estimated to be 0.2%. The blue line describes the shot noise limit of precision calculated from counting statistics. Compared to counting statistics, the analyzed errors are somewhat larger due to the inherent electronic noise of the 10^{13} Ω amplifiers. For beams sizes higher than 20 kcps, the high ohmic amplifiers yield more precise data compared to SEMs suggesting a potential benefit in using the 10^{13} Ω amplifiers at higher count rates.

Dynamic Range

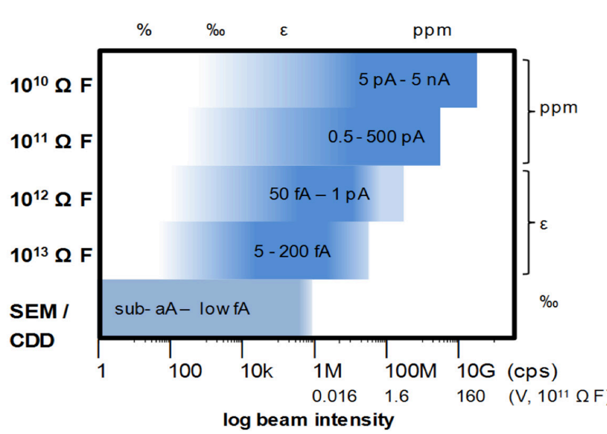


Figure 9. Dynamic range of different detector types (F = Faradays Cup).

Figure 9 shows the dynamic range of different detector types (F = Faradays Cup). The upper dynamic range of 10^{11}, 10^{12} and 10^{13} Ω amplifiers are 50 V, 5 V and 0.5 V respectively. As can be seen from Figure 2 though, the 10^{13} Ω amplifiers have a real benefit at intensities up to 500 fA, which is 50 mV on 10^{11} Ω . The lower limit of the amplifiers are determined by the noise limit of the amplifiers. The recommended detector(s) available for a given intensity are indicated by the intensity of shading. The counting statistic limit on precision for a 10 minute acquisition is indicated at the top, with typical limits for detector type on the right.

References

More details can be found in *Koornneef et al*. (2014):

J.M. Koornneef, C. Bouman, J.B. Schwieters, G.R. Davies 2014. Measurement of small ion beams by Thermal Ionisation Mass Spectrometry using new 10^{13} Ohm resistors, *Analytica Chimica Acta*, Volume 819, p49–55.

Summary and Conclusion

- The new 10^{13} Ω amplifiers have up to 10x times improved signal to noise in comparison to standard 10^{11} Ω amplifiers.
- Faraday cups can now resolve signal intensities down to 0.5 µV (ca. 2 times RSD of noise) which corresponds to about 30 cps.
- All Faraday cup measurements overcome the cross calibration limit which usually limit the attainable accuracy in combined multicollector and ion counting measurements.

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