

Application of $10^{13} \Omega$ Amplifiers in Low-Signal Plasma-Source Isotope Ratio Measurements by MC-ICP-MS: A Case Study with Pt Isotopes

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Recent developments in amplifier hardware enable low-noise measurements of exceedingly small ion beams in isotope ratio analysis, yielding higher precision from smaller samples than ever before. To date, these amplifiers have largely been employed in thermal ionisation instruments, with few applications using plasma-source (i.e., MC-ICP-MS) instruments. Here, we demonstrate the utility of these new generation $10^{13} \Omega$ amplifiers in MC-ICP-MS, employing Pt isotopes as a case study, a system that could greatly benefit from the promised advances. The data demonstrate that for samples with low Pt abundance, for a modest increase in uncertainties, the amount of sample required can be reduced by a factor of 50–100. This technique thereby opens up new possibilities for analysis of samples that have low Pt mass fractions or were otherwise impossible to obtain in sufficient quantities.

Keywords: MC-ICP-MS, $10^{13} \Omega$ amplifiers, double-spike, isotopes, platinum.

Received 10 Sep 19 – Accepted 07 Dec 19

Thanks to recent advances in analytical techniques and instrumentation, isotope geochemists are currently enjoying a period of great expansion in the range of isotope systems being studied and the ability to resolve small isotopic effects in high-temperature settings. Further progress in isotope studies is likely to require analysis of ever-smaller variations in smaller amounts of sample. As such, new developments in analysis of small samples are likely to be of great value.

Due to the small size of the ion beams produced by TIMS (thermal ionisation mass spectrometry) and MC-ICP-MS (multi-collector inductively coupled plasma-mass spectrometry), these instrument types commonly employ sensitive Faraday detectors connected to amplifiers with gains in the range of 10^{10} – 10^{12} , achieved using high-value feedback resistors. The precision of isotope ratio analyses is limited by counting/measurement statistics and the inherent thermal noise (Johnson-Nyquist noise) of the feedback resistors. The advantage of increasing resistor values comes from the fact that, following the Johnson-Nyquist noise equation, the gain of the amplifier increases linearly with the resistor value, while the relative noise level only increases by the square root of the resistor value (Wieser and Schwieters 2005).

The most recent generation of this amplifier hardware employs a new architecture, including feedback resistors with values of $10^{13} \Omega$ to achieve a gain of 10^{13} . Thus, it follows that a $10^{13} \Omega$ resistor will increase the gain by a factor of 100 compared with a $10^{11} \Omega$ resistor, while the noise will increase by a factor of $\sqrt{100}$. In practice, for every order of magnitude increase in the feedback resistor value, one can expect a factor of two increase in the signal-to-noise ratio (Wieser and Schwieters 2005). However, for small ion beams, and when combined for multi-collection across a number of Faraday detectors, these improvements are highly valuable.

So far, these $10^{13} \Omega$ amplifiers have been employed in a handful of isotope systems (Pb, U, Sr, Nd, Os) in TIMS (Koomneef *et al.* 2014, Klaver *et al.* 2016, Trinquier and Komander 2016, Wotzlav *et al.* 2017, Sadekov *et al.* 2019, Wang *et al.* 2019), with few applications utilising MC-ICP-MS (Pb, Ta; Kimura *et al.* 2016, Pfeifer *et al.* 2017). Given the potential advantages of these amplifiers, they are likely to see increasing use in the coming years.

In this paper, we test the use of $10^{13} \Omega$ amplifiers for measurements of exceedingly small samples with plasma-

source instruments, using Pt stable isotopes as a case study. The Pt isotope system is well suited for this comparison for several reasons. First, the mass spectrometry of Pt is well established (see Creech *et al.* 2013, 2014, 2017) and provides a suitable baseline against which to compare these measurements. Secondly, Pt has extremely low abundance in natural samples, and thus would significantly benefit from the ability to precisely measure isotope ratios in small samples and/or lowering amount of sample required for analysis. Finally, as the instrument employed in this study is equipped with five $10^{13} \Omega$ amplifiers, it is possible to employ the double-spike approach for correction of instrumental mass fractionation for Pt isotopes (Creech *et al.* 2013), which results in similar beam sizes on all masses, allowing all inversion isotopes to be measured on the same amplifiers. Furthermore, the double-spike approach reduces extraneous effects and permits the true uncertainties of analyses using these new pre-amplifiers to be characterised.

Materials and methods

Reagents and standard solutions

AR grade nitric (HNO_3) and hydrochloric (HCl) acids were further purified by sub-boiling distillation in Savillex[®] DST-1000 distillation systems. Acid dilutions were performed with ultrapure ($> 18 \text{ M}\Omega \text{ cm}$) water. All sample handling and preparation was performed in a class-100/ISO 5 clean laboratory at Macquarie University, Australia.

The preparation of the Pt isotope standard IRMM-010 has been described previously (Creech *et al.* 2013). The Baker Pt standard (J.T. Baker $1000 \mu\text{g ml}^{-1}$ in 20% HCl, Lot# H44N77; hereafter referred to simply as Baker), first described by Creech *et al.* (2013), was employed as a secondary standard in this study, as this solution has been previously shown to have a small but reproducible offset from IRMM-010 of $\delta^{198}\text{Pt} = 0.09 \pm 0.04\%$ ($2s$ standard deviation, $n \geq 100$; Creech *et al.* 2017).

Samples were doped with a $^{196}\text{Pt}\text{--}^{198}\text{Pt}$ DS, as described by Creech *et al.* (2013), with ^{194}Pt , ^{195}Pt , ^{196}Pt and ^{198}Pt being used in the DS inversion.

Geological materials

Two geological reference materials and one chondrite meteorite, all of which have been previously analysed for Pt stable isotopes, were selected for this study.

WPR-1 is an altered ultramafic igneous rock from the Wellgreen Complex, Yukon, Canada, which is distributed by

CANMET CCRMP. This rock powder is frequently employed in studies of platinum-group metals, owing to the relatively high concentrations of these metals. LK-NIP is a dolerite from the Nipigon diabase, Ontario, Canada, which is distributed by Geosciences Laboratories. Platinum stable isotope data for these geological reference materials have been previously reported by Creech *et al.* (2014).

In addition, the carbonaceous chondrite Allende was also analysed for this study. This widely studied meteorite has been previously analysed for Pt stable isotopes in a number of replicates by Creech *et al.* (2017).

The Carius tubes used were made of high-quality borosilicate glass, with dimensions of 33 cm in length, 2.5 cm outer diameter and 2 mm wall thickness. Prior to use, Carius tubes were cleaned by filing with dilute nitric acid and heating in a water bath for several days. This process was repeated once more, after which tubes were subsequently rinsed multiple times with ultrapure water and then dried to remove any moisture from the inside.

Samples weighing 0.5–6.5 g (depending on sample type) were weighed and then transferred to Carius tubes using a funnel made of weighing paper. The tubes were subsequently chilled in a liquid nitrogen bath, in which state 3 ml of 10.5 mol l^{-1} HCl and 9 ml of 13.5 mol l^{-1} HNO_3 were added to each tube to produce inverse *aqua regia*. Samples were double-spiked prior to digestion by adding the double-spike along with these acids. The double-spike aliquots were prepared in advance, such that 55% of the Pt in the mixture was from the double-spike, and were added to the sample in 0.5 ml of 10.5 mol l^{-1} HCl.

Carius tubes were then sealed in the flame of an oxygen-acetylene torch. After sealing, the tubes were tightly wrapped in aluminium foil, inserted into steel jackets and placed in an oven and heated at $220 \text{ }^\circ\text{C}$ for 72 h to allow for thorough dissolution and complete sample-spike equilibration. After heating, the tubes were slowly brought back to ambient temperature and removed from the jackets. Prior to opening, the tubes were once again chilled in liquid nitrogen and then opened by scoring around the top and then breaking around the score line. The contents of the tube were allowed to thaw and then transferred to clean centrifuge tubes, rinsing with dilute HCl where necessary and then centrifuged. The supernatant was then transferred to clean Savillex[®] PFA beakers and evaporated to dryness. The dried samples were dissolved in 10.5 mol l^{-1} HCl and re-evaporated, before finally taking up in 10 ml of 0.5 mol l^{-1} HCl in preparation for chemistry.

Platinum was purified from the samples using the anion exchange protocol of Creech *et al.* (2014), utilising AG1-X8 (100–200 mesh) anion exchange resin. Major cationic species in geological matrices are not retained on this resin, and the platinum-group elements are sequentially eluted using increasingly strong HCl and HNO₃ acids. Pt is collected in a final step using concentrated HNO₃, with a Pt yield of ≥ 90% and purity of ≥ 95%, which is adequate for precise Pt isotope measurement results using a Pt double-spike (Creech *et al.* 2014).

Mass spectrometry and data reduction

Platinum isotope measurements were performed using a Thermo Fisher Scientific Neptune Plus MC-ICP-MS at Macquarie University, Australia. Measurements were made with two different amplifier configurations – one using standard 10¹¹ Ω amplifiers and one using the new 10¹³ Ω amplifiers – for the isotopes used in the double-spike inversion (Table 1). Aside from the pre-amplifier configuration and solution concentration, all other operating parameters were kept identical between these two configurations.

Relative amplifier gains were calibrated prior to each analytical session using a new internal current source which uses a stable reference current of 3.3 pA (i.e., 0.33 V on a 10¹¹ Ω amplifier; note: to ensure a consistent reference frame, gain corrected voltages are always reported relative to 10¹¹ Ω amplifiers; 10¹³ Ω use a relative gain of ca. 0.01, and thus for a given ion beam, the same intensity will be reported for both amplifiers). The accuracy of the calibration was monitored through repeated analyses of the secondary standard (Baker Pt std).

The MC-ICP-MS was operated in low-resolution mode, with a mass resolution of ~ 1800 ($M/\Delta M$; peak edge width

from 5 to 95% full peak height). A Cetac Aridus II desolvating nebuliser system with a 100 μl min⁻¹ PFA micro-flow nebuliser was used for introduction of the solution to the mass spectrometer. For the highest possible sensitivity, the instrument was operated using high-sensitivity Jet and X type sampler and skimmer cones, respectively. Analyses using 10¹¹ Ω amplifiers were made on solutions with 50 ng ml⁻¹ of Pt (not including the DS contribution) in 0.5 mol l⁻¹ HCl, resulting in an ion current on ¹⁹⁴Pt of ca. 1.4 × 10⁻¹⁰ A (i.e., ca. 14 V). Analyses using the 10¹³ Ω amplifiers were made using solutions with ≤ 0.5 ng ml⁻¹, also in 0.5 mol l⁻¹ HCl, with ion currents on ¹⁹⁴Pt of ca. 1.4 × 10⁻¹² A (i.e., 14 V, read as 140 mV with a gain of 0.01 relative to 10¹¹ Ω amplifiers). The sensitivity of the instrument under these conditions was ca. 640 V/ppm total Pt. Peak centring was performed at the start of each session and monitored throughout. Analyses comprised fifty × 8.389 s integrations, corresponding to ca. 7 min of data acquisition (plus 60 s uptake time). Baselines were obtained by measuring on-peak for 200 × 8.389 s integrations between each sample analysis, with the last ca. 60 s being subtracted from the subsequent analysis. Using this approach, background levels were typically ≪ 1 mV on all Pt isotopes.

All data reduction was conducted offline using the double-spike data reduction tool IsoSpike (Creech and Paul 2015), which is an add-on for the Iolite data reduction package (www.iolite.org.au). IsoSpike is open source and freely available from www.isospike.org. Pt concentrations of samples were also calculated offline using isotope dilution calculations as described by Creech *et al.* (2014).

Results are reported using δ¹⁹⁸Pt notation, representing deviation of the ¹⁹⁸Pt/¹⁹⁴Pt ratio from the same ratio for the IRMM-010 Pt isotope standard, and are expressed in parts-per-thousand (i.e., ‰). Uncertainties are expressed as the 2s of repeat measurements. The on-peak zeroes approach incorporates all uncertainties arising from analytical baselines and amplifier noise. The double-spike corrected data are normalised such that the within-session mean of the IRMM-010 standard is equal to zero. Since all unknowns are measured and reported relative to the isotope standard, uncertainties in the absolute composition of the standard are not incorporated into the uncertainty budget.

Table 1.
Neptune amplifier configurations used in Pt isotope measurements with 10¹¹ Ω and 10¹³ Ω amplifiers

Cup	Mass	Amplifier	
		10 ¹¹ Ω configuration	10 ¹³ Ω configuration
L4			
L3	¹⁹⁰ Pt	10 ¹³	10 ¹¹
L2	¹⁹¹ Ir	10 ¹³	10 ¹¹
L1	¹⁹² Pt	10 ¹³	10 ¹¹
C	¹⁹⁴ Pt	10 ¹¹	10 ¹³
H1	¹⁹⁵ Pt	10 ¹¹	10 ¹³
H2	¹⁹⁶ Pt	10 ¹¹	10 ¹³
H3	¹⁹⁸ Pt	10 ¹¹	10 ¹³
H4	²⁰⁰ Hg	10 ¹³	10 ¹³

Results and discussion

10¹¹ Ω amplifier configuration

A baseline for comparison was first established by analysing samples using the conventional 10¹¹ Ω amplifier

set-up. The results, shown in Table 2 and Figure 1, are in good agreement with previously published results.

The Baker Pt isotope standard yielded $\delta^{198}\text{Pt} = 0.081 \pm 0.036\text{‰}$ ($2s$, $n = 23$), which is in excellent agreement with previous measurements of this solution (0.090 ± 0.040 ; $2s$, $n \geq 100$; Creech *et al.* 2017). Similarly, the single analysis of Allende in the $10^{11} \Omega$ configuration gave $\delta^{198}\text{Pt} = -0.124 \pm 0.060\text{‰}$, which is in excellent agreement with the value of Creech *et al.* (2017) of -0.120 ± 0.060 ($2s$, $n = 18$).

Small offsets from the previously published values are apparent in the data for the reference materials WPR-1 and LK-NIP. In the case of WPR-1, this discrepancy is likely due to sample heterogeneities. In processing a number of replicates of WPR-1, Creech *et al.* (2014) noted variability that exceeded the measurement repeatability determined by repeated analysis of other reference materials, which was attributed to heterogeneity of the sample powder. The heterogeneity was also apparent in the Pt mass fractions of the various replicates. While further analysis is required to prove this hypothesis, the offset in the WPR-1 sample is

Table 2.
Summary of Pt isotope data for $10^{11} \Omega$ and $10^{13} \Omega$ amplifier configurations compared with reference values

Sample	Configuration	$\delta^{198}\text{Pt}$ (‰)	\pm (‰)	n	Pt mass fraction ($\mu\text{g g}^{-1}$)
Baker	Creech <i>et al.</i> (2017)	0.090	0.040	≥ 100	
	$10^{11} \Omega$	0.081	0.036	23	
	$10^{11} \Omega$ low-int	0.213	0.365	3	
	$10^{13} \Omega$	0.006	0.237	34	
WPR-1	Creech <i>et al.</i> (2014)	-0.075	0.076	48	0.258
	$10^{11} \Omega$	0.009	0.042	2	0.273
	$10^{13} \Omega$	-0.018	0.297	10	0.273
LK-NIP	Creech <i>et al.</i> (2014)	-0.216	0.028	2	0.0123
	$10^{11} \Omega$	-0.108	0.066	2	0.0123
	$10^{13} \Omega$	-0.184	0.235	7	0.011
Allende	Creech <i>et al.</i> (2017)	-0.120	0.060	18	1.240
	$10^{11} \Omega$	-0.124	0.060	1	1.274
	$10^{13} \Omega$	-0.165	0.070	2	1.273

n is the number of observations.
Uncertainties are $2s$, except where $n < 2$, in which case it represents the 'internal' $2SE$ of the analysis.

consistent with sample heterogeneity, and the isotope composition determined here overlaps within uncertainties with the value determined by Creech *et al.* (2014; $\delta^{198}\text{Pt} = 0.009 \pm 0.042\text{‰}$ compared with $-0.075 \pm 0.076\text{‰}$, respectively).

In the case of LK-NIP, this material was less comprehensively characterised previously, with a reference composition of $\delta^{198}\text{Pt} = -0.216 \pm 0.028\text{‰}$. The data presented here double the data set for this material, and thus give a more realistic value for the uncertainty in the composition of LK-NIP. Combining the analyses of Creech *et al.* (2014) with those here gives a weighted mean of $\delta^{198}\text{Pt} = -0.15 \pm 0.07\text{‰}$.

In any case, as the solutions analysed in both amplifier configurations are aliquots of the same solution at different dilutions, the data obtained permit direct comparison of measurement results between the two amplifier configurations.

$10^{11} \Omega$ amplifier low-intensity test

For direct comparison with analyses made on $10^{13} \Omega$ amplifiers, the dilute Baker Pt standard solution prepared for analyses using the $10^{13} \Omega$ amplifiers – that is, diluted by a factor of 100 to 0.5 ng ml^{-1} – was also analysed in the $10^{11} \Omega$ amplifier configuration. Given that all other parameters remained the same between the two different amplifier configurations, this permits a direct determination of the improvement in uncertainties arising from the lower noise $10^{13} \Omega$ amplifiers.

The results of this test using the 0.5 ng ml^{-1} solution showed that for the $10^{11} \Omega$ configuration, analyses at low intensities (ca. 0.1–0.40 V on the four inversion isotopes, compared with ca. 10–35 V in the 50 ng ml^{-1} solution) had predictably poorer reproducibility, giving $\delta^{198}\text{Pt} = 0.21 \pm 0.37\text{‰}$ ($2s$, $n = 3$). Analyses of the same solution using the $10^{13} \Omega$ configuration compare favourably, yielding $\delta^{198}\text{Pt} = 0.01 \pm 0.24\text{‰}$ ($2s$, $n = 34$). While the $2s$ of measurements using the $10^{11} \Omega$ configuration may improve (i.e., decrease) somewhat with a greater number of analyses, these data show that the $10^{13} \Omega$ amplifier configuration leads to a significant improvement in the reproducibility for these measurements of small ion beams.

$10^{13} \Omega$ amplifier configuration

Analyses of all of the solutions described above were repeated on aliquots diluted to ca. 0.5 ng ml^{-1} , with all four isotopes for the double-spike inversion all on $10^{13} \Omega$ amplifiers. The results are shown in Table 2 and Figure 1.

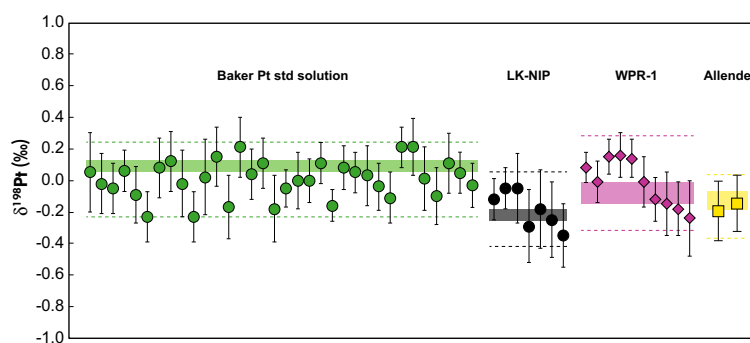


Figure 1. Platinum isotope data acquired using the $10^{13} \Omega$ amplifier configuration for the Baker Pt standard solution, two geological reference materials and the carbonaceous chondrite Allende. Dashed lines represent the 2s of the analyses they enclose. Shaded boxes represent the reference value and 2s thereof for each sample from references mentioned in the text. [Colour figure can be viewed at wileyonlinelibrary.com]

Analyses of the solutions in the $10^{13} \Omega$ configuration give values of $\delta^{198}\text{Pt} = 0.006 \pm 0.237$ (2s, $n = 34$) for the Baker Pt standard, $\delta^{198}\text{Pt} = -0.018 \pm 0.297\text{‰}$ (2s, $n = 10$) for WPR-1, $\delta^{198}\text{Pt} = -0.184 \pm 0.235$ (2s, $n = 7$) for LK-NIP, and $-0.165 \pm 0.070\text{‰}$ (2s, $n = 2$) for Allende. In all cases, these values are in good agreement with reference values (Table 2; Figure 1), albeit with much larger uncertainties, as expected for these low-intensity measurements.

While the measurements at low intensities lead to a concomitant decrease in measurement repeatability, the amount of Pt consumed per analysis is decreased by a factor of 50–100, while the resulting uncertainties are larger by only a factor of ca. 3–6 (Table 2; Figure 1). Consequently, while this approach may not be advantageous for abundant natural rocks where we are not sample-limited, this approach has the potential to be extremely advantageous for certain sample types, particularly those with low natural abundances of the element of interest, or where sample availability is limited, as illustrated below.

Consequences for required sample masses with the example of Pt

Platinum was chosen as a case study in part due to its low natural abundance in most rocks. These low abundances translate to small amounts of Pt despite processing relatively large samples, and thus can necessitate low-intensity analyses in certain sample types. As shown above, a typical analysis using the conventional $10^{11} \Omega$ amplifier configuration consumes on the order of 50 ng Pt per analysis. While in some rock types this amount is readily attainable, this frequently involves processing relatively large

amounts ($\gg 10$ g) of material, and in some cases sufficient material is not available.

For example, in terrestrial mantle rocks (i.e., peridotites), Pt mass fractions are typically on the order of ca. $5\text{--}10 \text{ ng g}^{-1}$, and thus minimum of 10 g of sample would be required for the typical analysis using $10^{11} \Omega$ amplifiers outlined above (Table 3). In the case of terrestrial samples, this amount of material is typically available, and thus such analyses are possible.

In certain cases of high interest, Pt concentrations in natural samples are lower, and/or sufficient masses of sample are not readily available. Take now, for example, a lunar rock with a Pt mass fraction of ca. 0.5 ng g^{-1} (based on data from Walker *et al.* 2004). The typical analysis using

Table 3. Comparison of $10^{11} \Omega$ and $10^{13} \Omega$ amplifier configurations in the context of Pt isotope measurements in various sample types

Amplifier configuration	$10^{11} \Omega$	$10^{13} \Omega$	
Pt required for analysis (μg)	0.05	≤ 0.001	
Typical uncertainty (%)	± 0.06	± 0.25	
Sample type	Pt mass fraction ($\mu\text{g g}^{-1}$)	Required test portion mass (g)	Required test portion mass (g)
Peridotite	0.005	10	0.2
Chondrite	0.5–5	0.1	0.002
Iron meteorite	5–20	< 0.1	0.002
Martian	0.004	10	0.2
Differentiated achondrite	≤ 0.0005	≥ 100	≥ 2
Lunar rocks	≤ 0.0005	≥ 100	≥ 2

50 ng of Pt as described above would thus require on the order of 100 g of material, which is likely impossible to obtain for this purpose. Now consider the same sample, but employing the $10^{13} \Omega$ configuration described here. Now each analysis will consume on the order of 0.5 ng of Pt, and thus the minimum mass of sample required is reduced to ca. 2 g, which may well be possible to obtain.

As described above, the uncertainties of analyses using the $10^{13} \Omega$ configuration are somewhat larger than when analysed at higher intensities using $10^{11} \Omega$ amplifiers, which must be taken into account when applying to such samples. In the case of Pt, however, it is likely that the natural samples with the lowest abundance of Pt will also be most isotopically fractionated, and thus natural variations would still be expected to be readily resolved despite the larger uncertainties. Thus, the application of $10^{13} \Omega$ amplifiers in MC-ICP-MS is likely to result in various new types of sample being available for isotopic analysis.

Future

Further advances in resistors and amplifier technology are likely to continue, resulting in increasingly sensitive amplifiers. As demonstrated here, these advances can lead to increases in analytical precision of small ion beam, and/or dramatically reduce required sample masses, in exchange for a relatively modest increase in analytical uncertainty. However, while similar advances in the future are desirable, these may no longer be applicable to plasma-source instruments, whereas with TIMS, the noise on small ion beams is dominated by the Johnson-Nyquist noise, an additional noise source is present in MC-ICP-MS data: that is, the plasma sources itself. Even in the data presented here for $10^{13} \Omega$ amplifiers, a significant portion of the noise likely reflects the noise/stability of the plasma ion source itself, rather than the amplifiers. If future advances in amplifier technology are to be embraced by MC-ICP-MS instruments, some similar advance will be required in the ion source, which, given the inherent noise of the plasma, may not be possible.

Summary

We show that new developments of $10^{13} \Omega$ amplifier hardware can be applied to plasma-source mass spectrometry. In the example of Pt stable isotopes, required sample masses can be reduced by a factor of ~ 50 , with a concomitant increase in measurement uncertainties of just a factor of ca. 3–6. Therefore, the use of these amplifiers may enable the analysis of samples that were heretofore unavailable or impractical samples, such as lunar rocks and other rare materials.

Acknowledgements

The authors thank two anonymous reviewers for their thoughtful comments on the manuscript, and Thomas Meisel for his helpful comments and efficient editorial handling.

Data availability statement

All data are available within the paper or are available from the authors on request.

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