

Global response to the reviewers – Gchron 2024-6

The manuscript Gchron 2024-6 was reviewed by four different reviewers (R2: James Metcalf, R4: Bruce Idleman and R1-3 anonymous), who are greatly thanked for the time they took to do so and for the extensive comments and recommendations they provided. These reviews contain similar or related comments, and in the following we will attempt to address and clarify those general comments.

*To supplement this overarching response, **if the manuscript is accepted for review**, individual and precise replies will be produced as responses to each review on the Gchron 2024-6.*

Our general response will initially address the scientific remarks and issues before moving on to the manuscript structure comments. We conclude with a proposal for a revised version of the manuscript.

SCIENTIFICS COMMENTS:

NOBLE GAS QUADRUPOLE MASS SPECTROMETER CALIBRATION AND USE:

R1, R2, and R4 each provide significant comments, criticisms, and inquiries regarding the approach outlined in the manuscript for calibrating and measuring the ^4He gas. We will address the scientific aspects of these comments. However, they also raise the question "why develop this approach when a better one already exists?" This legitimately challenges the relevance of the proposed manuscript and will therefore be addressed separately in the last part of the reply ("Future of the manuscript").

From our perspective, one of the key comments about the noble gas protocol concerns equation 2, particularly the parameters S and I_e . Reviewers R1 and R4 highlighted numerous aspects of this equation that may lead its misinterpretation and, consequently, its calibration and use for noble gas content. These comments rightly challenge our presentation of the protocol, and we recognize that the original manuscript is lacking detail and clarity in several places. The following paragraphs will attempt to rectify these problems.

In a revised manuscript, the following information would be added to the Introduction section (to clarify the context and purpose of the article) and to the methodological section.

➤ Technical justification for development of the protocol

The original manuscript failed to mention a practical constraint that led us to develop our protocol. During development, the laboratory did not have sufficiently accurate pressure sensors with a wide enough pressure range, so we were unable to accurately assess the different volumes in the line (up to ~20% error for some volume determinations). This then limits accurate determination of the partial pressure or amount of ^3He that will reach the quadrupole mass spectrometer using a classical approach based on the line volume derived from parameters such as pressure, volume, and number associated with the ^3He tank and pipette.

However, the experience gained by A.D. during his work at the GEOPS laboratory between 2017 and 2021, lab operating for over a decade (see Gautheron et al., 2021), suggested that it would be possible to address these limitations.

At this juncture, A.D. must acknowledge that the development presented in the article cannot solely rely on the research conducted between 2021 and 2023 at Geosciences Rennes, but also draws from the experience acquired beforehand.

➤ Missing formal hypothesis:

Consequently, we suggested that for the same "analytical set-up" (i.e., sequence of volume opening/closing/cleaning), the amount or partial pressure of the ^3He spike reaching the quadrupole mass spectrometer for analysis will be constant (except for the spike, which decreases with use). Therefore, by analyzing the ^3He signal (in amperes) together with the signal (also in amperes) of different **known** amounts of another gas (in partial pressure or moles), it is possible to calculate the amount of ^3He spike (in partial pressure or moles) in the quadrupole. The calculated value could then be used instead of one derived using line volume and retained if the "analytical set-up" remains unchanged.

Given this approach, we suggest that the obtained ^3He spike content will be determined with the same precision as the known amount of the other gas used. Below we address the relevant comment from R4

R4: *"I question the validity of using a material with a somewhat poorly constrained age like Durango apatite as a primary standard for helium measurement calibration – it can be done, but it introduces both additional complexity and uncertainty."*

While we agree with R4's comment, this will be further discussed in the last section ("Future of the manuscript"). However, the explanation below provides arguments to support this discussion.

➤ Applying the hypothesis of external volume calibration:

For spike calibration, various known amounts of gas are typically required, and the conventional approach involves using a precisely calibrated tank/pipette with a precisely known amount of ^4He gas and varying its ^4He amount thank to the different line characterized volumes. However, due to the difficulties in estimating our line volume we propose using different-sized fragments of Durango as different " ^4He tank sizes".

We acknowledge that the Durango crystal is far from perfect, it is one of the few internationally recognized standards for ^4He content (McDowell et al., 2005, Wu et al., 2021). Given this, we decided to assess its use as a calibration standard for our protocol.

The usual equation used to determine ^4He content is:

$$^4\text{He} [\text{mol}] = \frac{^4\text{He}_{\text{blanc corrected}} [\text{signal or partial pressure}]}{^3\text{He}_{\text{blanc corrected}} [\text{signal or partial pressure}]} \times ^3\text{He}_{\text{pip}} [\text{mol}] \quad (1)$$

where the $^3\text{He}_{\text{pip}}$ is determined using the classical approach (tank pressure and volume, pipette volume and number and line volumes).

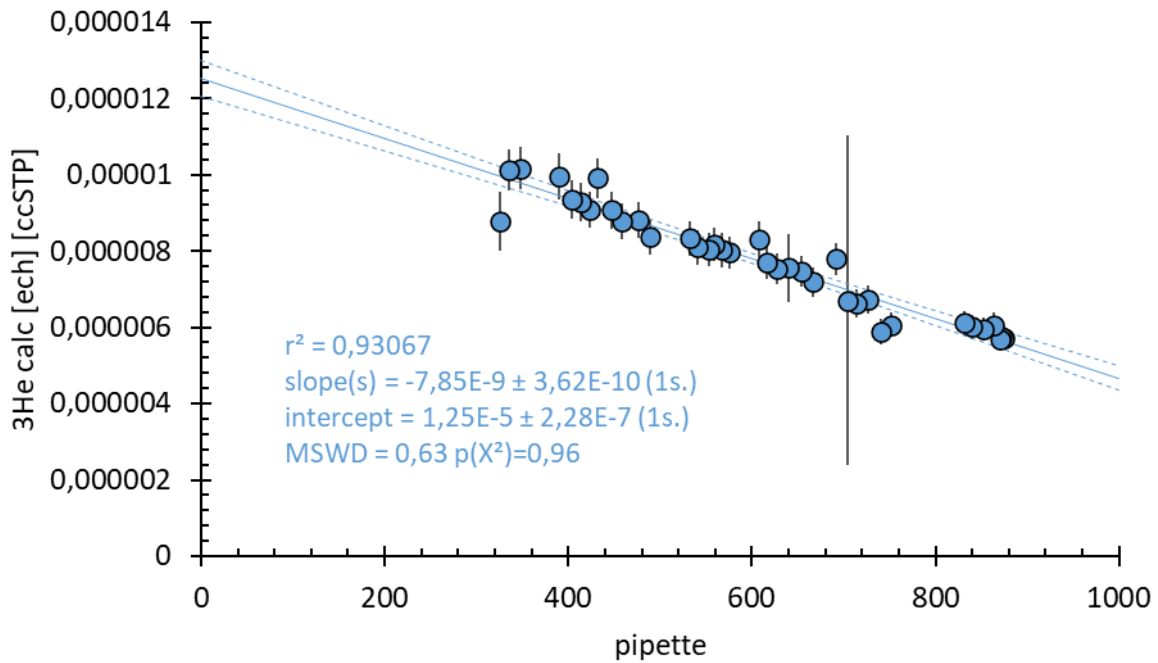
We invert the equation to give:

$${}^3\text{He}_{pip} [\text{mol}] = \frac{{}^4\text{He}_{blanc\ corrected} [\text{signal or partial pressure}]}{{}^3\text{He}_{blanc\ corrected} [\text{signal or partial pressure}]} \times {}^4\text{He} [\text{mol}] \quad (2)$$

In this case, the ${}^4\text{He}$ directly comes from a given Durango crystal and is subsequently determined using that crystal's uranium (U), thorium (Th), and samarium (Sm) content, as well as recognized ages (31.02 ± 1.01 Ma, McDowell et al., 2005). This method enables us to calculate the ${}^3\text{He}_{pip}$ content for a specific pipette and in principle allows us to implement Equation 1. However, four significant questions remain...

Question 1: Does this calibration remain valid over time?

If we repeat this operation multiple times the ${}^3\text{He}_{pip}$ spike pressure will decrease following a known equation ($\frac{\text{tank volume}}{\text{tank volume} + \text{pipette volume}} \text{ pipette nb}$) and the calculated ${}^3\text{He}_{pip}$ should similarly decrease. In our dataset we get, as is seen in our data:



This observation confirms the approach's expected behaviour over time but **does not** validate the ${}^3\text{He}_{pip}$ calculated amount in mol.

Question 2: Does this calibration remain valid for various ${}^4\text{He}$ contents and therefore variable partial pressures?

We investigated if varying the amount of ${}^4\text{He}$ gas affects the calibration result (calculated ${}^3\text{He}_{pip}$). To do this, it is necessary to obtain correct or eliminate the impact of the spike decrease. Therefore, we propose modifying Equation 1 by replacing ${}^3\text{He}_{pip}$ [mol] with ${}^3\text{He}_{pip}$ [signal], corresponding to the signal obtained for a pure ${}^3\text{He}$ spike measurement. Consequently, we obtain a similar form as Equation 1, but ${}^4\text{He}$ [mol] is substituted with a corrected ${}^4\text{He}$ [theoretical signal] adjusted for partial pressure variation and the decrease in ${}^3\text{He}_{pip}$:

$${}^4\text{He} [\text{theoretical signal}] = \frac{{}^4\text{He}_{blanc\ corrected} [\text{signal}]}{{}^3\text{He}_{blanc\ corrected} [\text{signal}]} \times {}^3\text{He}_{pip} [\text{signal}] \quad (3)$$

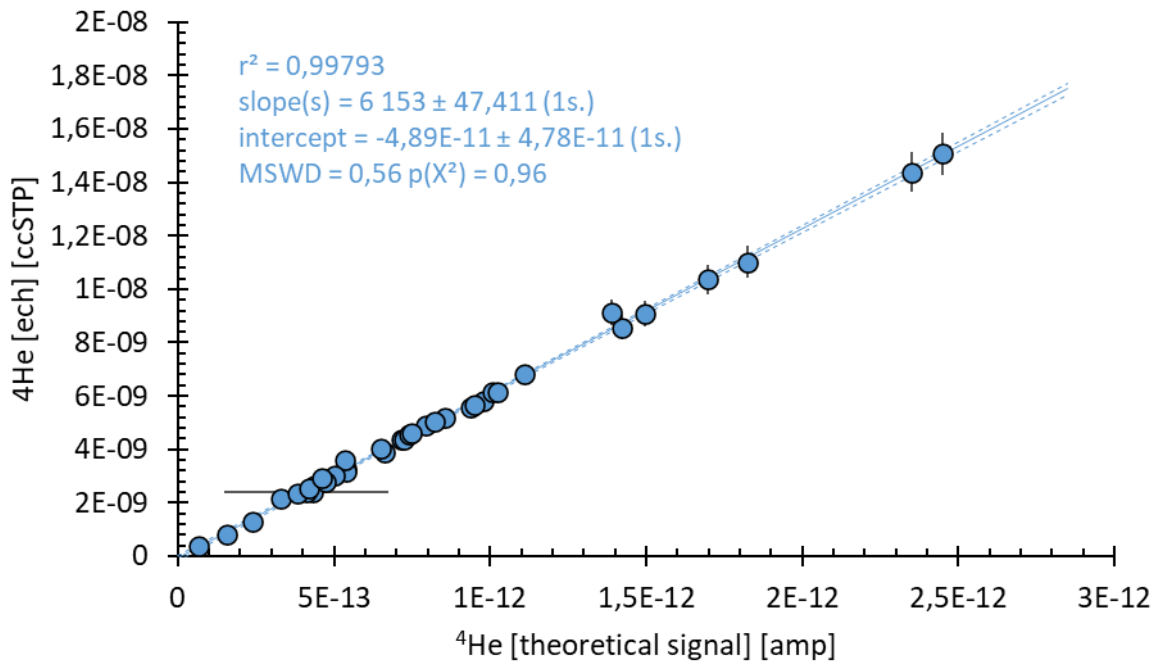
where the ${}^3\text{He}_{pip}$ [signal] is given by:

$${}^3\text{He}_{pip} [\text{signal}] = \frac{\text{tank volume}}{\text{tank volume} + \text{pipette volume}} \times {}^3\text{He}_{initial} [\text{signal}]$$

where the ${}^3\text{He}_{initial}$ [signal] corresponds to the first signal obtained for a pure spike analysis and the $\frac{\text{tank volume}}{\text{tank volume} + \text{pipette volume}}$ can be determined by:

- using the respective volumes (if known)
- monitoring the evolution of ${}^3\text{He}_{pip}$ [signal] by doing multiple pure pipettes analyses and determine the value by regression.

By implementing this modification, it becomes feasible to calculate ${}^4\text{He}$ [theoretical signal] and compare it with the actual ${}^4\text{He}$ [mol or ccSTP] derived from different-sized "Durango tanks". If the variation in ${}^4\text{He}$ content from different sources does not affect the calibration, the resulting relationship should be linear with the y-intercept should be 0.



This obtained parameters confirm that a variation of ${}^4\text{He}$ content (partial pressure) does not significantly affect our calibration and allows us to convert the ${}^4\text{He}$ [theoretical signal] in a ${}^4\text{He}$ amount [mol]. In the case the intercept =0, the slope corresponds to the parameter "S" in the original manuscript.

We propose reorganizing eq. 1 as below (eq. 2 in the original paper):

$${}^4\text{He} [\text{theoretical signal}] = \frac{{}^4\text{He}_{blanc\ corrected} [\text{signal}]}{{}^3\text{He}_{blanc\ corrected} [\text{signal}]} \times {}^3\text{He}_{initial} [\text{signal}] \times \left(\frac{\text{Vol}_{cylinder}}{\text{Vol}_{cylinder} + \text{Vol}_{pipette}} \right)^{pipette\ nb} \quad (4)$$

$${}^4\text{He} [\text{mol}] = {}^4\text{He} [\text{theoretical signal}] \times S$$

In essence, this formulation is not fundamentally different from Equation 1, but we believe that it explicitly identifies the parameters (${}^3\text{He}_{\text{initial}}[\text{signal}]$ and S) that require modification or redetermination in case of changes in the "analytical step-up" (such as adjustments in volume or spectrometer tunes).

R1 and R4 noted that the choice of "S" as a constant name for the parameters calculating the ${}^4\text{He}$ [mol] from the ${}^4\text{He}$ [theoretical signal] is not ideal, as it may cause confusion with sensitivity parameters used for magnetic sector spectrometers. We agree with this observation, and for any future manuscript submissions, we will consider changing its name to "T" for theoretical calibration.

Finally, we emphasise that by selecting crystal fragments of different sizes, we cover a wide range of total ${}^4\text{He}$ content and therefore a wide range of partial pressures. This makes it possible to validate a wide range of calibrations using only "Durango tanks".

Question 3: Is the inferred ${}^3\text{He}$ content accurate?

Given eq. 4, this parameter is not used (or at least not directly) to quantify the ${}^4\text{He}$ content of a sample. It is replaced by other parameters: "S", ${}^3\text{He}_{\text{initial}}$ and the evolution of calculated ${}^3\text{He}_{\text{pip}}$ (pipette function) and for all these parameters it is possible to define their uncertainty:

- "S": calculated using the regression results from ${}^4\text{He}$ [theoretical] error (gas analytical uncertainty) and ${}^4\text{He}$ content error (Durango ages uncertainty + chemical analytical uncertainty)
- ${}^3\text{He}_{\text{initial}}[\text{signal}]$: correspond to the analytical uncertainty of the first pure ${}^3\text{He}$ spike analysis
- Pipette function $\left(\frac{\text{Vol}_{\text{cylinder}}}{\text{Vol}_{\text{cylinder}} + \text{Vol}_{\text{pipette}}}\right)^{\text{pipette nb}}$: in our case this is calculated from the tank and pipette volumes (only one that could have been determine externally using a membrane pressure gauge, MKS902, an know volume). Note that, this function, and associated error, can also be derived from ${}^3\text{He}$ spike signal evolution.

Following the comments of R1 and R4, the sum of these errors is the one that should be compared to the ${}^3\text{He}_{\text{pip}}$ [mol] in case of a conventional analysis. In a new submission of the manuscript, this will appear in the discussion.

Question 4: Is the obtained ${}^4\text{He}$ content for a conventional sample reliable?

To address this question, we want to analyze crystals of other standards. Assuminf that the U, Th, and Sm content are well characterised, any discrepancies in the characterization of ${}^4\text{He}$ would be manifested as a deviation from the expected age. Due to the absence of international standards, we only conducted this analysis on the MK1 standard (Wu et al. 2021), which yielded successful results. However, we argue that since each fragment of Durango varies in total ${}^4\text{He}$ content, each one could be seen as a "new blind" ${}^4\text{He}$ standard each time.

➤ Routine use of the method:

Using this approach on a day-to-day basis with a consistent analytical setup revealed that the calibration performance (represented by the "S" parameter) may become inaccurate after unexpected events (such as power failures or changes in atmospheric conditions) or after prolonged periods of analysis or inactivity (several months). This phenomenon is rarely addressed

in publications, one notable exception is Gautheron et al. 2021, which proposes incorporating this phenomenon using a parameter "D" (for "Drift") in Equation 1 and manually calibrating it using regular Durango analyses. For the approach presented in our manuscript, there are two ways to address this calibration inaccuracy (drift):

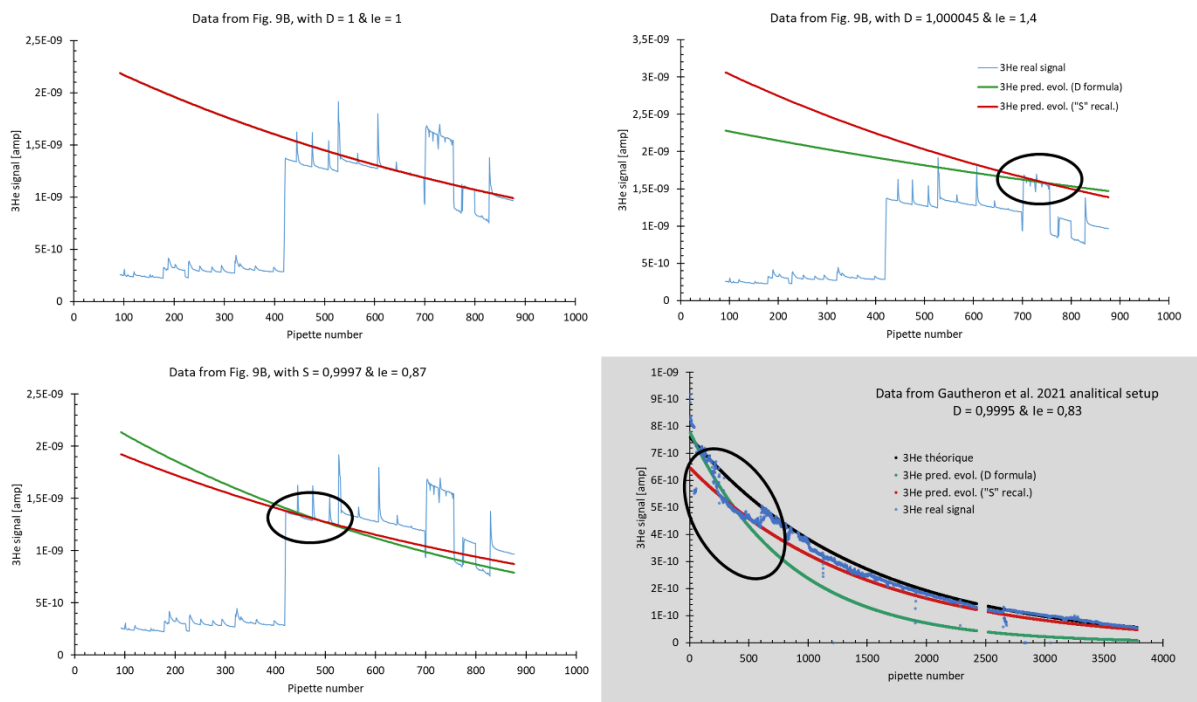
1. Recalibrate the "S" parameter regularly (e.g. every month)
2. Incorporate the "D" parameter from Gautheron et al. (2021) into Equation 4 and only recalculate the "S" parameter in case of changes in the analytical setup (such as a change in purification process).

Equation 4 modification: $\dots \times \left(\frac{Vol_{cylinder}}{Vol_{cylinder} + Vol_{pipette}} \times D \right)^{pipette\ nb}$

After comparing the two approaches for the same dataset over ~2-3 months, it appears to us that recalibrating S is preferable: the "S" parameters changed one time and it was stable, whereas "D" was modified 2-3 times and seem to be unstable. However, this is just a feeling and to obtain statistically indicative data it would be necessary to carry out this work over a longer period.

This feeling is consistent with how the "D" parameter functions within the equation by playing inside the pipette decrease. Indeed, the "D" construction seems incompatible with the evolution of the signal (in amps) of the ³He pipette analysis over time, which demonstrates good predictability using the equation.

To clarify this, the four following charts represent the ³He signal evolution against the pipette number using the same approach as for Fig. 9B in the original manuscript. It presents the real ³He signal (in blue), the ³He predicted evolution using the equation including D (in green) and using the equation without D but a "S" modification (in red and simulates a le parameter change). Additionally, the last chart (in grey) displays the same approach but with the ³He signal related to the data and line from Gautheron et al. 2021. It appears that playing on the D parameter (green curve) changes significantly the predicted ³He evolution, but not in a "linear" way. This aspect of the D parameter implies a frequent recalibration in case of any small variation or derive from the initial calibration.



However, the simplicity of using the "D" parameter during daily analysis is appealing. Therefore, we adapted it to our approach (case 1) by incorporating a multiplicative factor to the "S" parameter. This is the "le" parameter in our original equation 2. It can be calibrated using Durango similarly to the calibration process for the "D" parameter. From our perspective, this offers a simple way to incorporate the "Drift" phenomenon but linking it more closely to variations in intra-quadrupole mass spectrometer parameters (such as partial pressure, voltage stability, and filament life), than to variations in ^3He tank and pipette volumes.

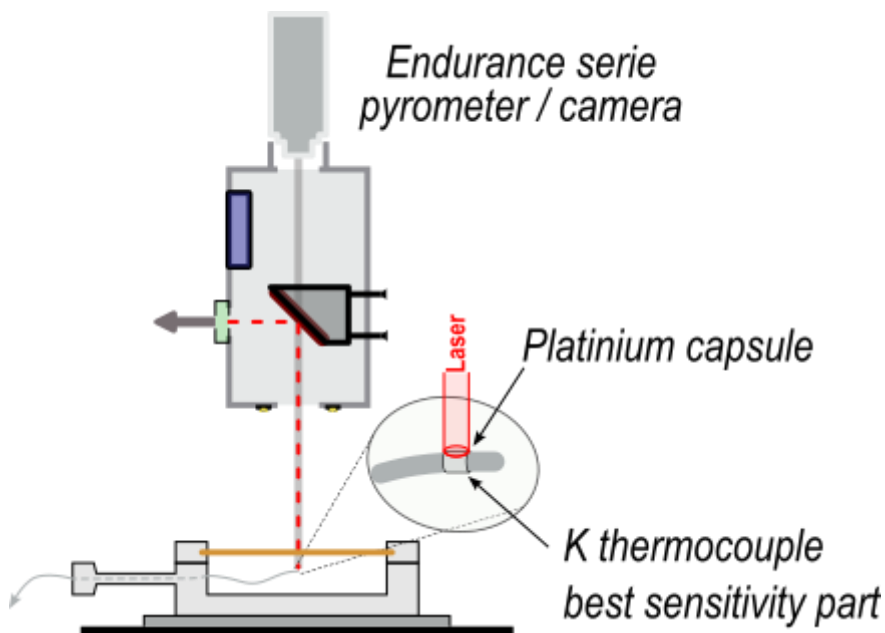


To conclude the “**Noble gas quadrupole mass spectrometer calibration and use**” authors would like to highlight that at the end, the obtained equations are not so different from the classical one. Irrespective, our method circumvents the need for line volume calibration by relying solely on the quadrupole mass spectrometer signal and Durango fragments. Therefore, it may be useful to researchers who have similar technical limitations to ours. However, demonstrating how to successfully apply the approach requires more discussion which we return to later ("Future of this manuscript").

RAMPED HEATING PROTOCOL:

Reviewer R1 and R4 provided detailed comments about the ramped heating methodology, and the following paragraph aims to address them. They expressed concern about the quality of our temperature monitoring calibration. We use a two-wavelength industrial pyrometer (Endurance® Series – E2RL-V0-0-0) sold by Fluke Process Instrument™ to monitor the temperature of the packet during heating. This pyrometer is certified to measure the temperature of metallic surfaces with a precision of $\pm 2^\circ\text{C}$. While this industrial certification provides an initial guarantee, the details of the calibration process are proprietary, and therefore inaccessible to the authors. Consequently, we conducted our own test, as presented in our original manuscript.

The test involved using a thermocouple K type - class 2 with an extremity diameter of 1mm, threaded inside the packet of 1mm diameter (and 1mm height). Given the packet and fibre diameter, we estimate that the temperature coupling between the packet and the pyrometer was nearly perfect.



Consequently, we argue that the results presented in the original manuscript are reliable within the discussed limits in the original manuscript ($\pm 6^\circ\text{C}$ above 300°C), that is far above what's needed for conventional analysis but not for ramped heating. In response to R4's recommendation, we will replace Fig. 11.B with a chart presenting the temperature difference vs temperature. R1 proposed adding a third calibration by conducting a CRH analysis on a Durango crystal and then using published diffusion parameters of Durango (D_0 and E_a) to reassess the measured temperatures. While an interesting idea, we believe this solution may be inefficient, as the Durango diffusion parameters are not easily reproducible, as noted by R4, and it would introduce additional uncertainty from the mass-spectrometer analysis and the kinetic parameter uncertainties.

R4 also highlighted the limitation of our current pyrometer that does not allow precise temperature determination below 350°C . This is a crucial range for kinetic parameter determination, especially for apatite where temperatures between 25 - 250°C are most significant to study crystal damaging/annealing effects. As in the original manuscript, we acknowledge this limitation as a significant issue, which is one of the main reasons why CRH analyses are not more developed in this publication, and **why we did not present kinetic parameters**. However, we believe that the technology presented in the manuscript holds promise, particularly with the introduction of the new E3ML pyrometer (Fluke Process Instrument™) with a range of 50 - 1000°C , though we have not had the opportunity to test it. To our knowledge, these methodological perspectives have not been published to date, therefore, the results presented in the manuscript offer limited information but provide a first insight into the reliability of this technology.

In case of re-submission, a paragraph will be added to the discussion to clarify and discuss this aspect.

“CHEMICAL PROTOCOL” PART (SECTION 3.3 IN THE ORIGINAL MANUSCRIPT):

This section in the initial manuscript was unclear for Reviewers 1 through 4, with Reviewer 3 specifically requesting additional clarifications. Therefore, for a revised manuscript, we will clarify and add details on this part of the protocol.

However, we believe that it is not necessary to present the chemical protocol in great detail, as it is already published (for instance Tharaud et al. and reference therein) and routinely performed in many laboratories (for instance the SARM at CRPG, Carignan et al. 2001). Nevertheless, we will add some details as in the following paragraph (initial text in black and the added text in red).

*“After the degassing, capsules are retrieved and put in individual 10mL vials (INFO) for chemical digestion and elemental quantification. This quantification is done by signal comparison **between dissolved apatite solutions and a range of standard solutions with adapted concentration**. In addition to U, Th and Sm **concentrations** used for helium age calculation, all REE elements are routinely **quantified** to provide complementary information for interpretation on each grain (inclusion, sample homogeneity, source for detrital samples). Ca is also systematically analyzed to determine apatite weight, considering Ca is stoichiometric and a fluorapatite composition (Eq. 3, Gautheron et al., 2021).*

$$Apatite_{weight} = \frac{Ca_{weight}}{0.3974} \quad (3)$$

The range of standard solutions is prepared using mothers’ solutions (Inorganic Ventures®, CMS1 and CGCA) characterized by precisely known element concentrations (uranium (U), thorium (Th), samarium (Sm), rare earth elements (REEs), and calcium (Ca)) with ISO 17034 and ISO 17025. Those mothers’ solutions are combine and diluted using a 0.5N HNO₃ solution (distilled from HNO₃ 65N – Normapur® VWR®) to obtain 10 to 13 daughter solutions with varying concentration to cover the expected range in dissolved apatite. Specifically, we target concentrations between 0.1 ng.l⁻¹ (or ppt) and 10,000 µg. l⁻¹ (or ppb) based on a calculation of dissolving a spherical apatite grain with a radius of 50µm (typical size of analyzed apatite) in 2 to 10 mL (required analytical volume), considering U, Th, Sm, REE concentrations ranging between 1 to 500 ppm and a stoichiometric Ca input (~1%).

The apatite digestion protocol is adapted from Farley (2000) and Gautheron et al. (2021) and is performed by 100µl acid attack (HNO₃ at 5N or 27%, – distilled from HNO₃ 65N – Normapur® VWR®) on a hot plate set to 65°C during 3 hours. After 30 minutes of cooling, solutions are then completed for QQQ-ICP-MS analysis with a HNO₃– 0.5N or 2% solution (distilled from HNO₃ 65N – Normapur® VWR®) to 2 to 10 ml depending on the required concentration and analytical protocol. The micropipette used for the acid attack and dilution come from Eppendorf Research Plus® respectively 100-1000µl and 0.5-5 ml. After the analysis the platinum capsules are retrieved and returned to the supplier for recycling.

*The elemental characterization in solution is made on an Agilent 8900 QQQ-ICP-MS. **During analysis, the full standard solutions are analyzed alongside sample apatite solutions at regular intervals, typically all 10 for every five apatite solutions. The obtained raw signals are then used to determine the concentrations of apatite solutions using regression (e.g. Tharaud et al., 2015). To calculate the element content for each individual apatite, obtained apatite solutions***

concentrations are then multiply by the dilution factor. The dilution factor of *each digestion session is determined using* the micropipette volumes and uncertainties. For each chemical session, the micropipettes volumes are checked by randomly weighing 10 *apatite solutions* after dilution. *The reliability of our element quantification has been assessed by analyzing fragments of homogeneous apatite crystals previously characterized by the same method in a certified lab (SARM, ISO 5 and 6), and by another method (LA-ICP-MS) at Géosciences Rennes (see Section X.X for the results). Results of this test will be discussed in Section 4.4.*

To assess the stability/reproducibility of these protocols, we analyze Durango and MK1 crystals as if they were unknowns, and then use it to control the obtain results. This choice was logic as they are already routinely measured for (U-Th)/He age calculations.”

FORM COMMENTS:

MANUSCRIPT ORGANIZATION:

All reviewers emphasize that the manuscript is poorly organized and lacking details, leading to difficulties in following and understanding it:

Reviewer 1: *"The manuscript is poorly written and organized. The structure of the paper is confusing, lacking clear sections for methods, results, or discussions. The text is difficult to follow."*

Reviewer 2: *"Areas where the contribution could be greatly improved with additional clarifications, discussions, and modifications. A re-organization would be beneficial."*

Reviewer 3: *"I cannot support the publication in its current form. My main concerns relate to the organization of the manuscript and missing details in some descriptions. Sections 2-3 could be combined under a Methods section, and separating Results and Discussion would be helpful."*

Reviewer 4: *"A lack of organization makes the discussions difficult to follow. I suggest considering a major reorganization of the text. Additionally, the description of instrumentation and basic analytical methods should not be mixed with that addressing data treatment and analytical complications."*

To address this, we propose restructuring the manuscript, primarily based on the suggestions from Reviewer 3:

- Expand the Introduction (Section 1) to briefly outline the current "classical" analytical protocol and the associated aspects/challenges that will be addressed in the paper leading to our proposed approach. This addition will also facilitate a discussion about the benefits and drawbacks of our protocol, as suggested by Reviewer 4. Additionally, we will contextualize the development and objectives of this protocol to include details provided in the Scientific reply
- Merge Sections 2 and 3 into a single "Methods" section, comprising two main subsections: a "Noble Gas Protocol" and a "Chemical Protocol," as recommended by Reviewer 4:

"I suggest that the authors consider a major reorganization of the text, at a minimum separating the sections dealing with the He mass spectrometry and those describing the LA-ICP-MS measurements for U, Th, and Sm.”

The new "Chemical Protocol" sub-section will be expanded based on the details provided in the Scientific reply.

- Divide Section 4 into two separate sections, "Results" and "Discussion," structured similarly to the Method section, in accordance with the advice from Reviewer 3: "

“Furthermore, it would be helpful to have the same (similar) subsection headings in Methods and Results so that one knows where to find the results (like you have it for Sections 3.2.2, 3.2.3 and 4.3 concerning ramped heating).”

- Introduce a subsection in the "Discussion" where the advantages and disadvantages of the presented protocol will be evaluated in comparison to existing protocols, as recommended by Reviewer 4.

Given these recommendations, we will restructure these parts of the manuscript accordingly

Language and style:

All reviewers have pointed out the presence of French words in figures and tables, which will be thoroughly reviewed and corrected for any subsequent manuscript submission.

Inconsistency between the supplementary material data, figures and main text:

The main author (A.D.) extends sincere apologies for the inconsistency identified, which solely resulted from an oversight on his part. The supplementary data underwent re-editing after the initial submission, as detailed in the discussions on GChron, to incorporate additional data originally intended for "upon request" availability. During this process, the main author transitioned to another laboratory, and the overly rapid retrieval of the raw data compounded by human error, led to mixing of conventional, ramped heating, and development/test data.

For any revised manuscript, we commit to thoroughly reviewing and ensuring consistency between the manuscript and the supplementary material.

R1: several mention to this aspect in it “Th/U vs. Sm/Th relationship:” section

R3: several mention to this aspect in it “Inconsistency of results and data presentation:” section

FUTURE OF THE MANUSCRIPT

The different reviews highlighted scientific errors and structural ambiguities in the original manuscript. We acknowledge and largely agree with these criticisms and have aimed to address them in this response. However, the general question remains regarding the purpose and scientific significance of the article, essentially asking, "Why develop this approach ⁴He calibration when a good one already exists?" To address this question, we wish to provide context for our work.

The (U-Th)/He method has become a routine technique (for approximately two decades) but there is ongoing developments to enhance its resolution and introduce new perspectives. Despite this extended period of use and the presence of well-established labs, there seems to be a lack of openly available publications describing a complete analytical protocol. This observation, not intended as criticism, suggests perhaps a potential blockage when establishing new laboratories and the prospect of losing acquired knowledge and cumulative feedback from different labs. While of course there are forums for exchange (e.g. the Noble Gas network) we view the GChron – Technical Notes as a more formal route to make analytical protocols publicly available. We note that reviewer 2 made a similar observation:

“As a (U-Th)/He lab manager I found this paper very interesting, and am very supportive of it, and other technical descriptions of analytical set ups, appearing as technical notes in EGU Geochronology.”

In this context, the study presents the results of a first (U-Th)/He analytical development in a new laboratory at Rennes University. However, after two years of development, A.D. who developed the protocol moved on and the laboratory underwent renovations that halted its analytical capabilities. The two-year period was sufficient to test and establish an initial version of the protocol and conduct some routine analyses, but not enough to refine. We believe that valuable information and knowledge was accumulated during this two-year development process, such as the possibility to perform the U, Th, Sm and Ca in a simpler way than isotopic dilution, the potential of using multiwavelength pyrometer for temperature determination rather than a thermocouple and the possibility to avoid volume calibration.

The authors acknowledged that all this development (especially regarding volume calibration) does not allow for obtaining the same “quality” as the conventional approach and therefore led to a “step back” in precision. However, from a practical perspective, the lab has been running for less than a year, and this short period of time leads the authors to assume that with more time, it may be possible to significantly improve precision and reaching the conventional “quality”. To our knowledge, these methodological perspectives have not been published to date, and as the analytical laboratory was turn down for an undetermined time, authors believed that it would contribute to the community to share those developments and there is the reason of this article.

Finally, we did not write the manuscript to prescribe our specific approach in preference over others but rather to present its development and our initial results. Over time, as is normal, the scientific community will determine its usefulness. We recognize that a published article must adhere to ethical and scientific standards, including a reproducible protocol, and that the peer review process is in place to evaluate this. Therefore, they will understand if reviewers and editors conclude that the study does not meet these standards and decide to reject the original

manuscript. In any case, authors express gratitude to the reviewers for their valuable feedback and help in organizing their thoughts, hoping that the dialogue remains open.