

Geochronology Discuss., referee comment RC2  
<https://doi.org/10.5194/gchron-2021-1-RC2>, 2021  
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## **Comment on gchron-2021-1**

Anonymous Referee #2

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Referee comment on "Technical note: Analytical protocols and performance for apatite and zircon (U-Th)/He analysis on quadrupole and magnetic sector mass spectrometer systems between 2007 and 2020" by Cécile Gautheron et al., Geochronology Discuss., <https://doi.org/10.5194/gchron-2021-1-RC2>, 2021

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### **General comments:**

This paper presents a very detailed, start-to-finish (i.e. whole rock to publishable dataset) methodology for the determination of (U-Th)/He ages from single grain apatite and zircon crystals. While much of this information is available to the community through the plethora of papers utilizing these methods over the last two plus decades, this work provides a single, succinct source for researchers to consult and cite. Additionally, this paper provides many additional details not commonly published, which, among other uses, is a significant resource for those interested in developing the ability to make (U-Th)/He age determinations in their own laboratories. No major revisions or changes to the paper structure are required. However, I believe minor additional details or clarifications would be beneficial, which are outlined in the line-by-line comments below. Most importantly, clarification to how He tank standards are calibrated for use on either the quadrupole or magnetic sector instruments is required.

### **Line-by-line comments:**

Line 13: Would "we detail the complete protocols in use for more than a decade" be more clear?

Line 15 and line 20: Sensitivity instead of "sensibility"

Line 33: "homemade" is used a number of times in this manuscript but "housemade" or "built-in-house" may better describe the laboratory quality of this equipment.

Line 33 to 34: "noble gas extraction-purification lines, one coupled to a quadrupole mass spectrometer and the other to a magnetic sector mass spectrometer" may clarify to the reader these are completely separate setups.

Line 41: All methods in this manuscript described by the authors are suitable for the described purposes but it may be helpful to show that these methods are not the sole way to accomplish (U-Th)/He age determinations. Here, offering LST as a less toxic alternative to the already mentioned heavy liquids may be useful.

Line 61 to 62: The possibility of Nb packets influencing the measured [U] and [Th] is certainly of concern. If this issue is going to be mentioned in section 2.1 then it may be worthwhile to tell the reader this point will be discussed at length later in the manuscript and the current texts leaves the reader feeling like this issue remains completely unresolved.

Line 66: Since both extraction lines described in the manuscript are being presented for the purposes of measuring He, a name such as "Quad line" instead of "He line" may help the reader more easily identify the two.

Line 74: Figure 1 has many details not discussed in this paper. May be worth a short comment about features such as the He diffusion cell or the exclusion of those portions from the schematic.

Line 84 to 86: Since only the planchet and line before first valve would see atmosphere, is the described heating targeted at just the planchet or the entire extraction and cleanup line? How is the heat applied?

Line 93: It is worth noting here that a shorter but hotter zircon degassing conditions are also possible.

Line 94 to 95: What threshold (percent?) is used to identify an acceptable return to background level signal?

Line 103 to 106: The method to measure temperature is calibrated to 1150° C but the zircon setpoint is stated to be >1250° C. How is the temperature monitored during

degassing of zircon?

Line 126: At what temperature are each SAES getter operated at?

Line 136: C+++ may be a concern for low  $^4\text{He}$  abundance measurements. Is mass 6 (C++) monitored for this concern?

Line 137: Longer gettering time could be offered as a suitable alternative to reduce the  $\text{H}_2$  influence on  $^4\text{He}$  peak if one does not wish to add a getter after mass spec inlet valve.

Line 146: It is unclear to me if " $^3\text{He}_c$  is the  $^3\text{He}$  content value adapted for each calibration" means each  $^3\text{He}$  spike tank is calibrated at the start of that tank's use or if this is a determination made during each batch of analyses. Is  $^3\text{He}_c$  is calibrated manometrically (or against another known tank) or is calibration solely done with Durango standards?

Line 164 to 165: Again, it is unclear if the tank shot is initially calibrated manometrically then tweaked from Durango standards that are specific to the analysis batch.

Line 183 to 184: It is unclear to me here if the authors are describing a test to verify this outlined cleaning procedure is adequate or if they are describing a procedural blank carried out with each batch of analyses.

Line 225: The  $^{235}\text{U}/^{238}\text{U}$  value of 0.00725 is equivalent to  $^{238}\text{U}/^{235}\text{U}=137.93$  (different from the more commonly used 137.88 referenced on line 288). For consistency, I suggest only referencing  $^{238}\text{U}/^{235}\text{U}$  with a value of 137.88?

Line 289 to 290: Another word besides "important" may better describe the linearity effect below 150 Ma. How is data processed for samples with ages >150 Ma?

Line 310: Do the author's have an explanation regarding the two  $\text{H}_2$  spikes observed (nearly two orders of magnitude increase in signal size)? Considering the  $\text{H}_2$  tail can influence the  $^4\text{He}$  signal, a comment regarding whether spikes such as these should be used to alert the user to be suspect of the  $^4\text{He}$  measurement would be useful.

Line 322: Deleting "to mostly" may make this sentence read more clearly.

Line 315 to 326: It is difficult for me to follow the details of the quad line standardization and I think this section could be improved upon with some clarification. Specifically, when exactly is  $^3\text{He}_c$  and D determined? Is it done for every batch of analyses? The comment about 1 to 2 months is confusing. Is that just the timeframe that variation is typically observed over? Figure 4 shows some Durango ages (panel C and D) that considerably differ from the acceptable (U-Th)/He age. How is data like this handled? Is some sort of outlier detection utilized? Are  $^4\text{He}$  measurements of unknowns run in batch sequence proximal to these outliers being processed (or rejected) using these outlier Durango ages, such as seen on panel C and D? If so, how?

Line 324: Sensitivity instead of sensibility?

Line 340: Does "To better calibrate the  $^4\text{He}$  cylinder" imply the cylinder is calibrated in another fashion (e.g. manometrically or against another known tank)?

Line 343 to 359: As with the quad line section above, it is unclear exactly when sensitivity is determined for the VG instrument. The text reads as if the instrument sensitivity has only been determined twice. Is there a "D" type factor used to tweak the instrument sensitivity for each run? Currently the text reads to me as if sensitivity is not determined for each batch of analyses, which I would consider necessary for the determination of absolute quantities of ions such as is required for (U-Th)/He age determinations.

Line 402 to 405: Using the phrase "needs to be considered" in this sentence could be misinterpreted as a correction is not currently being made (although my interpretation of the text is that a correction is made using the measured parent isotope ratios of the different blank).

Line 440: I assume the Ma is not needed here.

Line 545: say "in use for more than ten years" or "developed over the last ten years" instead of "developed for over more than ten years"?