

Geochronology Discuss., referee comment RC2  
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## Comment on gchron-2021-30

Anonymous Referee #2

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Referee comment on "Technical note: Accelerator mass spectrometry of  $^{10}\text{Be}$  and  $^{26}\text{Al}$  at low nuclide concentrations" by Klaus M. Wilcken et al., Geochronology Discuss., <https://doi.org/10.5194/gchron-2021-30-RC2>, 2022

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### General Comments

Difficulties in the measurement of  $^{10}\text{Be}$  and  $^{26}\text{Al}$  by accelerator mass spectrometry (AMS) are covered in this paper, as well as a comparison of background correction and isotope ratio calculation methods. These are important topics and deserve further attention in the literature. However, I am of two minds with the manuscript as it has been presented for review.

The content of Figures 5 – 8 and sections 1, 2.3 and 3.2 to the end are worthy of publication with minor editing. However, unless I have very much misunderstood the content, I find the remainder of the manuscript very sloppy. I have provided detailed comments below covering both minor points and also what I feel to be major issues. In particular, Figures 1, 2, 4, the associated discussions, and Equations 1 – 6 require major attention. If the paper stood on this content alone, I doubt it would pass review. While lines 46-47 state that these data are particular to ANSTO, the authors also state the the data are generally valid. I do not feel they have provided sufficient grounds for the latter. However, the content that was well fleshed out deserves publication and will be of benefit to those involved in the routine measurement of  $^{10}\text{Be}$  and  $^{26}\text{Al}$ .

### Specific Comments

Line 49: I do not believe that references are required for this line because AMS is an established technique by now. If references are to be added then I think some justification is needed for the inclusion of two references to the exclusion of all the other good reviews of the AMS process, or at minimum some acknowledgement that these are but two among many. While Finkel, Suter and Fifield are quite prominent in AMS and the cited texts are useful, there are similarly comprehensive reviews written by the original founders of the technique from the 1980s through 2010s that are available online (ex. at Mass Spectrometry Reviews). In particular, as Finkel and Suter (1993) is a book chapter, it is less accessible to many readers than accounts given in journal articles that can be downloaded online.

Lines 51 – 54: the wording here is less precise than I think it should be. Magnets select for the momentum to charge ratio, electric analyzers for the kinetic energy to charge ratio, and there may be additional elements, such as a gas filled magnet or secondary stripper, to further help separate isobaric interferences. Statements such as, "... one mass with a given charge state ..." are not adequately descriptive. Secondly, neutrals can also be formed during electron stripping and molecular ions can survive the stripping process in low charge states if conditions are correct. Determining conditions to "kill" molecular interferences at low stripping energy for low charge states was key to the success of small AMS instruments.

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"Section 2.1: Measurement of  $^{10}\text{Be}$ " needs major editing.

Figure 1: These data are confusing to me and I feel that they should not be accepted for publication as presented in the current form. They could be included as a guide for others if more clarification is provided, at least as outlined in 1 – 2 below.

1) Are these data being presented as equilibrium charge state yields?

1a) If not, then what these data represent needs to be more explicitly stated. If so, much

more detail about the measurement process is needed, such as yield versus gas pressure and where the current was monitored. Although I suspect this not to be a major issue, what effort was taken to ensure that the beam composition measured at the accelerator injection was, in fact, nearly 100% that of the ion being investigated? For example,  ${}^9\text{Be}^{16}\text{O}$  and  ${}^{12}\text{C}^{13}\text{C}$  are molecular isobars. Was any effort made to look for such isobars at the high energy end? Or, was a target composed of the binding agent (Nb?) with no BeO inserted to see the injection current drop to 0?

1b) Transmissions are given as %, but no indication of the range of injection currents is given. I assume micro-Amperes, but some indication of a range would be useful.

2) The paragraph following the figure states that Be-, BeO- and BeO<sub>2</sub>- were used to “cover a wide energy range”.

2a) What do you mean by, “Ion energy at the accelerator terminal”, then? Is this calculated as  $q/\text{original ion mass} \times \text{terminal voltage}$ ? This is what line 100 appears to imply but I feel that methods to estimate ion energy should be more explicitly given in the text.

2b) How did you confirm that stripping yields are equivalent for molecular ions versus atomic ions? Which energy and charge state combination was used to normalize each of the data sets for molecular and atomic stripping yields? Why does the figure not indicate which data are for which ions? It is not clear to me that molecular ion stripping gives the same yield as atomic stripping, and no references are provided to back up this implied assumption. This would imply that any “coulomb explosion” associated with molecular break-up has no effect. It is known that there is a difference in the energy distribution of stripped molecular and atomic ions when stripping in foils.

2c) The figure caption states that, “The gap in the transmission data represents an energy region where ion optical losses through the accelerator have greater impact on the measured charge states and so these data have been excluded”. That sentence, to me, seems insufficient. How did you evaluate “optical losses” and how are these accounted for in the data that are presented?

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Lines 135 – 142: The phenomenon of current fall-off has been observed by others, but it is not fully understood and if these data are presented then clarification is needed. Is this

a regular occurrence for you or was the 6.5kV run a "one-off"? Are you suggesting that all targets that are idle for 60 hours after initial sputtering at 6.5kV will under-perform as yours did? Klein and Mous (2017) NIMB 406, 210-213 suggest using > 11kV, and Southon and Roberts (2000) NIMB 172, 257-261 also suggest higher target voltages. The standard sputter voltage for the HVE SO110b is 7kV for Al<sub>2</sub>O<sub>3</sub> + Ag targets. While this current drop-off effect has been observed by others, are you suggesting that it is solely due to the sputter voltage and could not have been due to other factors? The 6.5kV targets had a slow rise and the largest peak current output from that "grey" set was less than the least peak current output from the 4.5kV "blue" set. For example, are you certain that your Al<sub>2</sub>O<sub>3</sub> : Ag ratios were correct and that the pins were all firmly pressed? Was the ion source cleaned prior to each of the 6.5kV and 4.5kV runs? The authors need to clearly state how certain they are that this current drop was indeed due only to the sputter voltage and how they arrived at that conclusion. Otherwise, they should re-word this to avoid further confounding an already confusing issue.

Lines 148 – 155: Lines 151-2 were a little unclear: Were all the targets mixed with Ag and pressed into cathodes at ANSTO or at the labs where the Al<sub>2</sub>O<sub>3</sub> was prepared? Were the Al<sub>2</sub>O<sub>3</sub> powders analyzed for actual %[Al] content by any methods other than AMS? Did all the Al<sub>2</sub>O<sub>3</sub> powders have a pristine color? Does each lab have a similar [Al] yield from the sample preparation procedures?

Figure 4: As was the case for Figure 1, I do not feel that these data should be accepted for publication as currently presented. Similar clarifications as those outlined for Figure 1 listed above should be given.

Equation 1, line 220: I disagree with this equation. First, the denominator does not include <sup>9</sup>Be from the sample material itself. Is the sample material assumed to be quartz? Or is the "dissolved quartz" in line 224 assumed to be contamination from the quartz vessels used to process the original material from which Be is to be extracted? If quartz is the assumed sample material then the denominator in equation 1 should read exactly as the numerator reads, but with subscript "10" replaced by "9". An explicit statement should then be added that, very reasonably, the assumption is made that  $N_{9,sp}^s + N_{9,AMS}^s \ll N_{9,q}^s + N_{9,c}^s$  so that those terms are ignored. However, in present form, exclusion of the  $N_{9,q}^s$  term from equation 1 affects the following equations 3 – 6. This section should be almost identical to that for <sup>26</sup>Al, as far as I can tell. For example, in the current form, the <sup>10</sup>Be/<sup>9</sup>Be ratio approaches infinite as the amount of carrier added to the sample approaches zero. This does not appear to be a typo as equation 3 follows from equations 1 and 2 in their current forms. This, then, renders the remainder of the section in error.

Lines 253 – 254: The sentence needs to be edited. As it is written, the sentence needs to be split at line 254 “when” and then the two sentences edited accordingly. However, it is a good idea to state the implicit assumption that if the carrier masses are roughly equal then the two “N9,c” rare isotope count terms are roughly equal for “s” and “b”. You have stated above that Al- current yields from different labs are not the same so the reader should be alerted to the possible pit-fall of this assumption.

Lines 350 – 355: I have also been thinking about this issue for some time. If we “sum the total counts and charge before calculating the final ratio” and we assume instrumental drift is cyclical over a period of time comparable to the full measurement time of a target then we are actually better off just burning through each target in sequence rather than separating the analyses into shorter intervals.

#### Technical Corrections

Line 87: This sentence needs editing for grammar. For example, “Practical methods to optimize sample consumption and negative ionization include: ... ii) what binding material is used....” should be reworded.

Lines 210 – 214: For clarity, this sentence should be split into smaller sentences rather than using semicolons to separate ideas.

Line 222: “where sub and superscripts ...” >> “where sub- and superscripts...” or “where subscripts and superscripts...”

Line 226: Poor grammar > "The impact of AMS measurement on the recorded 10Be events is accounted with N10,AMS and again can be different between sample and blank."

Lines 237 - 240: Grammar > "... close in time for similar duration when small source memory contribution can be approximated equal...". I also suggest splitting the sentence into two sentences.

Lines 346 - 347: The grammar needs editing.