Dear reviewer 2,

Thank you for your comments on our manuscript. Below we address each of the points raised in your review:

My first comment is that the authors should more fully recognize the previous work that has been done on laser micro-sampling for CA-TIMS U-Pb zircon geochronology. Specifically, Jim Crowley and Mark Schmitz developed and have been successfully employing laser micro-sampling at Boise State for several years. Unfortunately, they have not published the same type of detailed description of their work; however, examples of their laser micro-sampling can be found in Crowley (GSA abstract, 2018), Kovacs et al. (Engineering Geology, 2020) and Rioux et al. (JMG, 2023). The authors briefly cite the Kovaks study, but group it in with coarse mechanical micro-sampling techniques, and largely dismiss its importance:

"Over the last decades, researchers have increasingly sectioned zircon with mechanical tools such as a scalpel or using a nanosecond laser for ID-TIMS analyses, although such sampling has been coarse and largely neglected the requirement of textural homogeneity of isolated fragments (e.g., Kovacs et al., 2020; Samperton et al., 2015)."

The Boise technique uses thin (~30 microns) doubly polished zircon slabs to fully characterize the zooming patterns and chemistry of the zircon grains, before cutting the grains with a laser. Notably, the spatial resolution of the technique is similar to the current study, with the smallest micros-sampled fractions having weights of 0.1–0.5 micrograms (Crowley, 2018; Kovacs et al. appendix, 2020).

Rather than dismissing this prior work, it would strengthen the manuscript to recognize this alternate micro-sampling technique and discuss the similarities-differences and pros-cons of each method. This prior work in no way takes away from the current study, which is of high quality and very detailed. Laser and PFIB micro-sampling of zircon is a new field and it is highly beneficial to have multiple labs experimenting with different methods.

Thank you for bringing this omission to our attention. We were not aware of the GSA abstract and of the very interesting study of Rioux et al. This is certainly an interesting approach, and indeed it looks like this technique is comparable in spatial resolution to ours. We encourage the authors to document the effect of using the ns laser and the associated damage on U-Pb results.

In the revised version of the manuscript, we will cite the additional work you mentioned and recognize more fully the work on zircon cutting with the ns laser performed at Boise State. Also, we will include a sentence on the comparison of the two approaches.

Secondly, I have some general comments and questions on the origin of the scatter in the micro-sampled zircon dates. The authors did a nice job critically testing whether beam damage impacted the U-Pb systematics and working to understand the scatter in the data. The authors' conclusion that the scatter in the data is not directly related to beam damage seems reasonable, and is expected given that the damaged lattice is likely removed during the chemical abrasion step. As a clear test of this, I liked that the authors analyzed both large and small shards of mechanically broken zircon (i.e. not PFIB or laser cut). I would like to see a larger dataset of analyses of very small mechanically broken shards for both samples, which have volumes similar to the PFIB and laser micro-sampled zircon fragments. Such a data set would allow for a direct comparison between mechanically sampled grains and PFIB and laser micro-sampled grains. If the two datasets do show similar dispersion, it would more definitively rule out beam related lattice damage/Pb loss.

We fully agree with your observations. To gain more clarity on the cause of dispersion, for the revised version of the manuscript, we will analyze a few small pieces (volume similar to microsamples) of both Mud Tank and GZ7 zircon to complement our dataset.

The authors argue that the observed scatter in the micro-sampled fragments is most likely related to variable U blanks, which seems very possible. My most significant concern with this conclusion is that the percentage of impacted grains seems to be inconsistent with the U blank data reported in Fig. S14. The figure shows that over a 1.5 year period, 16 out of 20 U blanks had values of ~0.3 pg; however, if all of the scatter in the micro-sampled zircon dates is due to variable U blanks, Figure S14A suggests it would require U blanks >0.5 pg for 9 out of 13 analyses. The authors should discuss why the U blanks might be more variable in the actual analyses than in their blank measurements. It might also be informative to run an entire batch of

blanks to better understand the variability of the U blanks within a single batch. If there is significant variability, it would suggest that the authors should be using a higher U blank value with larger uncertainties.

Admittedly, we did not anticipate the U blank could cause any issue when we designed the study and until relatively late in the project. Therefore, most of the blanks reported here were measured over a different period (June 2021–January 2024) compared to our samples and might not be directly comparable. Therefore, for the completeness of the study we will also provide more U blank analyses in the revised manuscript.

I am also surprised that the U blanks are so high. U blanks are typically an order of magnitude smaller than Pb blanks, as would be expected, given the much high concentrations of Pb in the environment. The U blank data presented in Figure S14B suggest that the U blanks during the time of this study are similar to or higher than the Pb blanks. This is an unexpected result, and as the authors note, raises concerns about memory effects in the micro-capsules.

We were also surprised to find that U blanks could be as high. Since we made this discovery, we started tracking the U blank more systematically (loading, total procedural and sample beaker blanks).

In the revised version of the manuscript, we will additionally highlight that constraining the U blank and its variability should become standard practice prior to any analyses of low U zircon (especially microsamples).

Even based on the current data and text, the lab appears to be applying a U blank of 0.07 ± 0.02 pg (Fig. S14A), whereas measured U blanks have an average of 0.32 ± 0.08 pg (Fig. S14B).

In the present manuscript, we are applying a U blank mass of 0.32 ± 0.08 pg which is our best estimate of the blank applicable over the study period. The U blank of 0.07 ± 0.02 pg stated in Fig. S14A was the value used prior to our more recent measurements. We will remove this from Fig. S14A for the revised version of the manuscript to avoid confusion.

Detailed comments:

Lines 298–300: “Note that our analyses yield dates up to 20 Ma younger than the ones published in the literature (Black and Gulson, 1978; Horstwood et al., 2016; Gain et al., 2019).”

The authors should discuss this in more detail. Why are their dates 20 Ma younger?

The discrepancy between our dates and the ones from the literature was surprising for us as well. However, we note that our high-precision dataset of large crushed pieces (>1 ng U) is internally consistent, and even with very elevated U blanks, one would not reproduce the ages of Horstwood et al.

One possible explanation for the observed age discrepancies may be natural isotopic variability of Mud Tank zircon, between different crystal and possibly within a single crystal – similar to what Schaltegger et al. (2015) observed for zircon megacrysts from the Alps. We will highlight this point in the revised version of the manuscript.

Lines 462–465: “Beyond zircon, PFIB and femtosecond laser machining may substitute microdrilling as a more precise method for obtaining texturally controlled aliquots of complex samples for isotopic analyses, as well as being applied to microsampling of other U-bearing accessory minerals such as titanite, rutile, apatite and baddeleyite, and to other radiogenic or stable isotope systems.”

I recommend changing “substitute” to “replace”.

We will change “substitute” to “replace” in the revised version of the manuscript.

Figure 6: It would be useful to add labels to the concordia figure indicating which graph is Mudtank versus GZ-7.

We will add sample labels to the concordia figures (Fig. 6).

On behalf of all the authors,
Sava Markovic

References:

1. Schaltegger, U., Ulianov, A., Müntener, O., Ovtcharova, M., Peytcheva, I., Vonlanthen, P., ... & Girlanda, F. (2015). Megacrystic zircon with planar fractures in miaskite-type nepheline pegmatites formed at high pressures in the lower crust (Ivrea Zone, southern Alps, Switzerland). *American Mineralogist*, *100*(1), 83-94.