February 19, 2024

To the Editors and Associate Editors:

On behalf of my co-authors, I send you the revised manuscript titled "Navigating the complexity of detrital rutile provenance: Methodological insights from the Neotethys Orogen in Anatolia."

The manuscript investigates U-Pb and trace element data reduction, processing, and common Pb correction workflows using new detrital rutile U-Pb geochronology and trace element geochemistry results from the Late Cretaceous to Eocene Central Sakarya and Sarıcakaya Basins in Anatolia. We use our dataset to demonstrate how to navigate the complexities of natural datasets. We provide recommendations for common Pb correction, discordance calculation and data filtering that are applicable to detrital rutile and other common Pb-bearing detrital minerals. Additionally, to facilitate the standardization of data reporting approaches, we provide open access code as Jupyter Notebooks for data processing and analysis steps, including common Pb corrections, uncertainty filters, discordance calculations, and trace element analysis.

Reviews from the first version of this manuscript (manuscript number egusphere-2023-1293) indicated substantial revisions were needed before acceptance. Three referees and the Associate Editor provided constructive comments that enabled us to significantly clarify and strengthen our original manuscript. The three reviewers critiqued the novelty of the study, the number of U-Pb analyses discarded during data reduction, the potential bias of discarding data and the validity of interpreting discordant U-Pb analyses, and the apparent lack of novelty and complexity in geochemical data interpretations. The reviewers' comments were addressed in our previously submitted responses to reviewers. The Associate Editor's comments are addressed below. The revised manuscript includes all of the changes and revisions indicated in our responses.

The manuscript has been completely rewritten and reorganized. The major changes are summarized here. (1) All reviews questioned the large number of U-Pb data rejected. We contextualize our U-Pb data within the published literature. We demonstrate that the rejection of data during U-Pb data reduction is under-discussed, and the rejection of data during filtering (U-Pb ratio and/or discordance) is a seemingly universal limitation in detrital rutile work (Section 7.1). As far as we are aware, we are the first paper to systematically step through the data reduction process and explore the effects of various choices on resulting U-Pb date distributions. From this, we provide recommendations to the community for data treatment as well as opportunities based on current limitations. (2) Because of these new recommendations, we provide, as a companion to this manuscript, the Jupyter Notebooks used for data reduction and visualization. We have encountered many detrital geochronology users who want to use detrital rutile but are uncertain how to collect, reduce, and interpret these data. We hope that the provided open access code will be a helpful resource. (3) We added an overview and discussion of data filtering based on U-Pb ratio uncertainty and discordance. Following the Associate Editor's comments, we added an overview and discussion of various discordance metrics in U-Pb data from common Pb-bearing minerals. From the comments of Reviewer 1, all ^{207}Pb correction results were re-calculated following an iterative approach, and, based on the updated Pb-corrected results and discordance calculations, we no longer recommend using a percent discordance cutoff. Instead, we recommend a filter based on U-Pb ratio uncertainty (i.e., power law filter) and the inclusion of grains of all concordance levels. (4) Several reviews indicated that more could be done with the geochemical data but did not provide specific suggestions. The geochemical data does not show any trends by stratigraphic age, sample location, or age population. We redid the PCA analysis using compositional PCA and largely found the same results: the protolith and temperature sections capture the most important components of the trace element results. We use the geochemical data, specifically Zr-in-rutile temperature and mafic-pelitic categories (Cr, Nb), to interrogate potential bias in U-Pb data rejection and uncertainty filtering (Section 7.4). In this way, we use the geochemical data to demonstrate that the data rejection does not significantly bias the U-Pb results.

Additional changes based on reviewer comments include: (1) An updated title and text that moves away from claiming ‘new analytical workflows.’ (2) The introduction and background are updated following reviewer comments to better address rutile stability fields, U-Pb geochronology literature, and U-Pb closure temperature. (3) The common Pb correction section includes an overview of ^{204}Pb corrections and is updated to include an iterative approach for the ^{207}Pb correction. All figures and results are updated with the iterative ^{207}Pb correction approach. (4) Several reviewer comments indicated that the data reduction methods were unclear, therefore, we added additional text in the methods section (i.e., Section 4.4). Additionally, we include our data reduction workflow code as open access Jupyter notebooks. (5) The results and discussion sections have been separated. The results section now includes text on the raw U-Pb data quality with examples of accepted and rejected analyses, common Pb correction results, and a comparison of discordance metrics and uncertainty filters. The discussion section includes recommendations for U-Pb data rejection, correction, and filtering. (6) The reviewers were divided on the importance of discussing the implications of including/excluding low-U rutile grains. We significantly shortened this point. (7) We’ve expanded the supporting information text to reflect the reviewers’ comments on U-Pb and trace element standard reproducibility. (8) All of the data repository files have been updated with the U-Pb data that was newly reduced with a weighted linear spline in iolite, iterative common Pb corrections, discordance metrics, and uncertainty filters.

In addition to the first round reviewers, the following reviewers are suggested:

Sarah George, University of Oklahoma, sarahgeorge@ou.edu
Emily Finzel, University of Iowa, emily-finzel@uiowa.edu
Will Jackson, University of Memphis, wtjckson@memphis.edu
Gary O’Sullivan, Trinity College Dublin, osullig3@tcd.ie
Andrew Kylander-Clark, University of California Santa Barbara, kylander@geol.ucsb.edu

Thank you for your guidance during the submission and review process and for consideration of the revised manuscript.

Sincerely,
Megan Mueller

Response to the Associate Editor's comments on manuscript egusphere-2023-1293

1. You use the percentage of common Pb (using the Stacey-Kramers model) as a discordance filter. As mentioned on line 195 of your manuscript, this filter skews the age distribution towards younger ages. Have you tried the logratio definition of discordance? You may find that this has less of an effect on the shape of the age distributions.

Thank you for this suggestion. In the revised manuscript we compared the Stacey-Kramers discordance model to the Aitchison (logratio) model (Section 2.3.5 and Section 5.3 and Figure 6). In addition to this study, many detrital rutile U-Pb datasets include analyses that are highly discordant with some analyses plotting close to common Pb compositions (i.e., Govin et al., 2018 Geology). The logratio distances are smallest for analyses closest to the concordia at both the upper and lower intercepts; therefore, a discordance filter based on this metric would exclude the 'middle space' analyses. The Aitchison distance metric does not seem to be applicable to datasets with high discordance analyses, but may be better suited for datasets with little discordance. On the other hand, the Stacey-Kramers distance seems to be a more representative metric of U-Pb systematics in common Pb bearing minerals, where grains closest to the common Pb composition have the highest discordance.

2. Please remove the use of similarity, likeness and cross-correlation from your study. As a rule of thumb, it is best to avoid dissimilarity measures that were invented by geologists, not statisticians.

We have removed this section.

3. Please make a cleaner separation between results and interpretation.

The updated manuscript has separated the text into results (Sections 5 and 6) and discussion (Section 7).

4. I agree with the reviewers that PCA is potentially powerful, but hasn't been used to its full potential in your study. However, there is another issue that needs to be fixed first. According to line 372, you used OriginPro to do these calculations. I haven't used Origin before, but I understand that it is a general purpose statistics package. Therefore, I suspect that it ignores the fact that trace element concentrations are compositional data. The important difference between 'regular' PCA and 'compositional' PCA is explained by Aitchison (1983, doi:10.1093/biomet/70.1.57). There are lots of computer programs that implement compositional PCA (using logratio statistcs), including CoDaPack (in Excel) and the compositions, robCompositions and provenance packages in R. The interpretation of compositional biplots is succinctly described in Raimon Tolosana-Delgado's "CoDa in a nutshell" document: http://www.sediment.uni-goettingen.de/staff/tolosana/extra/CoDaNutshell.pdf.

Thank you for this comment. We were not aware of this limitation in OriginPro and changed our PCA approach to use the robCompositions package in R. The results are broadly similar, where the variance in detrital rutile trace element chemistry is best explained by both protolith and metamorphic grade.

5. Your response to reviewer 2 attributes the poor performance of the Kragero secondary standard to a pulse-analog conversion issue. Does the summary table of Figure A2 represent all the data or only the 'good' data?

Following the comments of Reviewer 2, we now show the U-Pb reference material results in concordia diagrams in the supplement which include all of the 'good' and 'bad' data. We have added an extensive discussion of standard reproducibility in the supplemental text, following our response to reviewers.

6. I have never used DetritalPy, but the cumulative distributions of Figure 5 look wrong. Cumulative distributions should not be smooth but should consist of discrete steps.

The figures with cumulative distributions look smooth because they are cumulative KDE distributions. The figure captions have been updated to explain this.

7. Lines 261-262 of your paper claims that "Detrital rutile U-Pb raw data are given in the data repository". However, when I go to the data repository, I get a spreadsheet with isotopic ratios and concentrations. This is not what I understand as raw data. It would be great if you could share your raw mass spectrometer data. Your *response to Reviewer 2 contains some interesting plots showing the raw mass spectrometer data to illustrate what you mean with 'spiky' data. I would be interested to see some of this detail incorporated in the paper.*

This is a good point and we have modified Figure 3 to include representative signal intensity patterns. We show examples of acceptable analyses and rejected analyses. We acknowledge that a large number of analyses were rejected and this modified figure helps illustrate and justify the situation. We are unaware of any published papers with raw data to compare our results with, so this serves as an important first step toward a more standardized community understanding of detrital rutile U-Pb data reduction workflows. Additionally, the supplemental material includes the plot from our response to Reviewer 2 within our discussion of standard reproducibility. The raw U-Pb and trace element data are now included in the data repository (https://doi.org/10.17605/OSF.IO/A4YE5).