

Reviewer 1 – Stijn Glorie

We thank the reviewer for their careful assessment of our study. Below we outline how we have incorporated their suggestions in our revised work.

1) We have modified the title to be more specific to biotite (new text is underlined):

“On the viability of detrital biotite Rb-Sr geochronology”

2) As noted by the reviewer, filtering of data is discussed and the effects of the filtering on the dataset collected and presented in this work is shown (Fig 2 – lower curves). The possibility of introducing bias through filtering is now acknowledged in various places in the study, for example (new text underlined):

“Such filtering, however, could introduce bias toward more radiogenic populations, especially in younger material that has not had time to accumulate radiogenic child product (e.g. limiting the effect of initial $^{87}\text{Sr}/^{86}\text{Sr}$ to $\sim < 5\%$ requires filtering of $^{87}\text{Rb}/^{86}\text{Sr} > 500$ @ 250 Ma and $^{87}\text{Rb}/^{86}\text{Sr} > 50$ @ 2500 Ma).”

3) We have added further information about sample preparation:

“The biotite grains were either manually picked from sediment separates and mounted in epoxy, or the sediments were poured directly into an epoxy mount. Grains were not mounted with a preferred orientation resulting in a semi-random c-axis orientation (the natural shape of a mica lath favours a c-axis normal to the mounting surface). Mixed c-axis orientations may help avoid less optimal signal stability ablating parallel to the mica c-axis (Rösel and Zack 2022) that may effect the final Rb-Sr date calculated (Lloyd et al. 2023).”

With regard to the reviewer’s comment “It is well known that mica-orientation has an influence on the apparant [sic] Rb-Sr dates.” So far as we are aware, the only published work to comment on the effect of grain orientation is Rösel and Zack (2022) wherein it is noted that mounting grains with c-axes perpendicular to the laser typically resulted in more stable signal than mounting grains with the c-axes parallel to the laser path. They do not, however, indicate that the orientation affects the final Rb-Sr date. That conclusion is mirrored by anecdotal experience in our lab. We do, however, acknowledge that a recent abstract was presented at the 2023 Goldschmidt meeting (Lyon) that does outline an affect of mica orientation on the resulting Rb-Sr date.

4) We have included additional details in the method section as suggested by the reviewer including detailed instrument settings in the supplementary materials. Contrary to the reviewer’s comment, all reference dates cited are Rb-Sr dates, not Ar-Ar, and, as in the original text, the initial $^{87}\text{Sr}/^{86}\text{Sr}$ values are listed with the appropriate reference for further information. Yes, some of the materials of known age return MSWDs that are consistent with some degree of heterogeneity. We now refer to one of these (Mica 1B) as an ‘in-house’ reference material to help avoid implying it is perfectly homogeneous. It is important to note that MSWD is only one measure of population dispersion with some significant limitations related to the effect of outliers etc. Using an alternative statistical method, such as the ‘Robust Regression’ of Powell et al. (2020 – Geochronology), which better accommodates potential geologic dispersion, yields a single population isochron for all materials of known age, including using all 20 analyses of Mica 1B from the second run (992 ± 6 with a spine (s) = 1.12; max s for a single population of 20 analyses = 1.37).

5) The SiO₂ assumption has little effect on the Ti-in-biotite temperatures calculated. Recalculating using 40 wt % SiO₂ yields Ti-in-biotite temperatures that are ~5 °C less than those calculated using 35 wt % SiO₂, well within the expected uncertainty of the method. This is now mentioned in the text (new text underlined):

“Ti-in-biotite temperatures calculated via LA-ICP-MS data generally range between ~ 650 and 725 °C for most samples (Fig. 3). The SiO₂ content used in the calculations has little effect on temperature. Increasing the SiO₂ from 35 to 40 wt. % results in only a ~ 5 °C temperature change (Fig. S2), well within the expected uncertainty of the method (Henry, Guidotti, and Thomson 2005).”

This relationship is also shown as a new supplementary figure (Fig. S2).

6) In a newly added Fig. S1 we now show the potential effect of low totals on the Ti-in-biotite temperature. Changing the wt. % from 96 to 88 (the cut-off wt. % used in this study – now noted in the methods), while maintaining Mg# results in a commensurate change in calculated Ti-in-biotite temperature from ~680 to 695 °C. In addition, we now colour the biotite electron probe data in Fig. 3A and B by the totals to further demonstrate the apparently limited effect that the total wt. % has on calculated temperature.

7) We recognize the potential unintended misuse of including the bulk regression-derived dates in the study. As suggested by both reviewers, we have removed dates calculated in this way from the study.

8) We have attempted to improve the flow of these sections (highlighted in the reviewer’s marked up PDF).

9) We have added additional annotations to help match the text in the discussion.

10) Noted, and modified throughout.

Reviewer 2 Hugo Olierook

We thank the reviewer for their time and efforts in helping improve our study. Below we demonstrate how we have incorporated their suggestions.

1) We appreciate the reviewers comments on filtering the data by $87\text{Rb}/86\text{Sr}$. In Larson et al. 2023 (EPSL) of the hundreds of biotite analyses reported only a single analysis was <3 . Similarly, in Qui et al. 2024 (JGSL), Camacho et al. 2020 (Geo. Cosmo. Acta), Camacho et al. 2012 (EPSL), and Rosel and Zack, 2022 (Geostandards and Geoanalytical Research) no biotite analyses had $87\text{Rb}/86\text{Sr} < 3$. As such, the analyses excluded in the present work are most likely not biotite or have significant inclusions.

Including analyses with lower ratios may not only include non-biotite material (which was not the target of the current study), but also would have very minimal statistical impact on the final interpretations. The two-point date calculated from an $87\text{Rb}/86\text{Sr} = 3 (\pm 5\%)$ and an $87\text{Sr}/86\text{Sr} = 0.7156288 (\pm 5\%)$ - as calculated from a 15 Ma isochron through 0.715) with an intercept of 0.715 ± 0.015 (as suggested by the reviewer) would be 15 ± 840 Ma. Similarly, for $87\text{Rb}/86\text{Sr} = 2 (\pm 5\%)$ and $1 (\pm 5\%)$ along a 15 Ma isochron through 0.715, the uncertainties would be 1250 and 2510 Ma, respectively. If, however, the actual initial of the analyses was 0.730, the dates calculated based on the same $87\text{Sr}/86\text{Sr}$ intercept (0.715 ± 0.015) would be 370 ± 850 Ma, 550 ± 1270 Ma and 1080 ± 2523 Ma for $87\text{Rb}/86\text{Sr} = 3, 2,$ and $1 (\pm 5\%)$, respectively.

Given the likelihood for analyses with $87\text{Rb}/86\text{Sr} < 3$ to not be biotite, and given the limited statistical effect on the age distributions, we have chosen to leave these analyses out.

With respect to filtering for highly radiogenic analyses, e.g. $87\text{Rb}/86\text{Sr} < 2500$, we now include text that recognizes the potential bias (new text underlined):

“Such filtering, however, could introduce bias toward more radiogenic populations, especially in younger material that has not had time to accumulate radiogenic child product (e.g. limiting the effect of initial $^{87}\text{Sr}/^{86}\text{Sr}$ to $\sim < 5\%$ requires filtering of $^{87}\text{Rb}/^{86}\text{Sr} > 500 @ 250$ Ma and $^{87}\text{Rb}/^{86}\text{Sr} > 50 @ 2500$ Ma).”

Moreover, as before, show the effect of filtering in Fig. 2 (lower curves).

2) As noted in the manuscript, the biotite, which were separated from samples collected in the Bay of Bengal, are affected by at least some degree of alteration/weathering leading to low totals. We now quantify the potential effect on Ti-temperatures in a new Fig. S1 and have modified Fig. 3 to be coloured by total wt% to show the net effect on our data specifically.

3) The reviewer makes a valid point about how the dates calculated using the 2nd calculation method (bulk regression) as presented in the original paper. This same issue was raised by the other reviewer and, as such, we have removed it from the revised version of the study.

All dates calculated using the two-point isochron method (as implemented in IsoplotR – Vermeesch, 2018 (GSF)) have full uncertainties propagated and presented in the supplementary information.

L7) Fixed, thank you

L34-35) We appreciate the reviewer's comments. We have added the citation suggested (new text underlined):

“The coincidence of the isochron and spot-dates derived independent of the measured initial $^{87}\text{Sr}/^{86}\text{Sr}$ indicates that detrital Rb-Sr geochronology may be a viable alternative or addition to detrital $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology (e.g. Crossingham et al. 2024), eliminating the potential time-consuming step of irradiation and permitting increased numbers of analyses.”

With regard to excess ^{40}Ar , this is discussed elsewhere in the manuscript and, as such, is not added here.

L36) Fixed

L43) Fixed

L80) The material is a phlogopite of known age. It has not been demonstrated to be fully homogeneous at the scale of analysis. It is now referred to as an ‘in-house reference material’ to avoid confusion. Moreover, the assessment of over dispersion is just in the statistical context of MSWD. If an isochron is calculated with the robust regression of Powell et al. 2020 (Geochronology) using all of the analyses from MICA 1B, it defines a single population isochron at 992 ± 6 Ma – which overlaps the expected dates of 990 ± 6 Ma (Camacho et al. 2012 – EPSL)

L180) We understand the reviewer's comment. This advantage of detrital LA-ICP-MS Rb-Sr is now noted in the introduction (new text underlined):

“The coincidence of the isochron and spot-dates derived independent of the measured initial $^{87}\text{Sr}/^{86}\text{Sr}$ indicates that detrital Rb-Sr geochronology may be a viable alternative or addition to detrital $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology (e.g. Crossingham et al. 2024), eliminating the potential time-consuming step of irradiation and facilitating increased numbers of analyses.”

L217) We appreciate the reviewer's interest in this. As noted previously in response to R1 we have now modified our Figure 3, which shows Ti-in-biotite temperatures, to include total weight % information. In the new figure, it is apparent that the majority of the lower T outliers have low total weight %.

Fig 1) Done

Fig 3) The 22O refers to the number of oxygen in the normalized biotite mineral formula, not an isotope. Changed to Mg# and box and whisker plot. We have not added the uncertainties in panel D as suggested. The calculated mean Euclidean distance is essentially a measure of the average distance away from the 1:1 line and, as noted in the text, it is suggested that such an uncertainty should be added to the temperature estimate.

Associate Editor Axel Schmitt

Additional private note (visible to authors and reviewers only):

Line 9: Consider mentioning that these are IODP cores.

Changed

Line 15 (and elsewhere): Consider spelling out “@”

Changed

Line 55: Somewhere, the full sample name (according to IODP convention) should be stated, also in the supplementary tables.

The full sample names are noted in the text and have now been added to the supplementary materials.

Line 63: “sediment separates” is unclear. What was separated? Was the sample washed/sieved? Any magnetic/density separation? What was the diameter/thickness of the biotite flakes picked/analysed? Please also consider stating the amount of available material, and/or the type of IODP sampling (wedge, cylinder, quarter round, etc.).

Reference to Najman et al. 2019 is now provided.

Line 70: Here and elsewhere: used SI unit “ μm ”. Please also state depth of analysis crater, not only diameter.

Changed

Line 72 (and elsewhere): The official name is: NIST SRM610; unit is missing after diameters.

Changed

Line 75: Somewhere in this section, please specify which uncertainties were propagated, and how error correlations were calculated. Please also define “rho” in the supplementary tables.

Additional details have been provided including further citations that detail the methods utilized.

Line 90: subscript TiO_2

Changed

Line 96: CAMECA is an acronym (Compagnie des Applications Mécaniques et Electroniques au Cinéma et à l'Atomistique), and the company itself uses capitalization.

Changed

Line 97: add space between number and unit

Changed

Line 110: kaolinite (?)

Changed

Line 115: lower case “One”

The ‘One’ is starting a sentence, so it has been retained.

Line 117: Please state MSWD with a consistent number of significant figures.

Changed

Line 131: radiogenic child product = tautological; just say radiogenic isotope, or radiogenic ^{87}Sr
Changed

Fig. 3 D: Please add “°C” to Euclidean distance, as in text

We have updated the figure and caption to be consistent with the text throughout.

Line 162: “the effect of initial intercept” consider adding “selection”. The sentence overall is difficult to follow. Consider breaking up and explaining more clearly how the model was set up.

Changed and sentence broken up.

Line 181: presented

Changed

Line 197: As formal GTS subdivisions, Early and Middle should be capitalized.

Changed

Editor Klaus Mezger

l 14 dependent (adjective) not dependant (noun)

Changed

l 37 a non-radiogenic intercept: awkward wording, do you mean: initial isotope composition?

Changed

l 39: can be regressed through : do you mean: can be regressed to define...

Changed

l 40: an initial, non radiogenic intercept. Tautology, the intercept is by definition unradiogenic. (non radiogenic: not a proper term)

Changed

54 Ti, like in the rest of the text

Changed

l 56 test the viability of detrital Rb-Sr, : word missing after Rb-Sr, there is not such thing as detrital Rb-Sr, may be the Rb-Sr systematics of detrital biotite...

Changed

167 and Hogmalm et al. (Hogmalm et al. 2017) : should be and Hogmalm et al. 2017)

Changed

l 86: granulite grade : there is no such thing as granulite grade, you mean granulite facies.

Changed

l 87 .. to molar weight per cation TiO_2 equivalent : TiO_2 is not a cation, Ti is, what do you mean here?

l 88 MTiO_2 = molar weight per cation: TiO_2 is not a cation

Changed

l 110 kaolinite, you mean the mineral not the rock.

Changed

l 136 ashed grey lines : they are black

They are actually dark grey; have modified the text to simply say 'dashed'

l 160 Tautology, value of the initial intercept can : either: value of the initial isotope composition or value of the intercept can

Changed

1 184 Larson, Button, et al. 2023) : correct reference

Changed

1 189 (Jager, Niggli, and Wenk 1967; Armstrong, Jäger, and Eberhardt 1966: correct references, only one author and then et al!

Changed

1 191: this variability or the variability

Changed

1 210 the temperature is not measured in, it is derived from or estimated from.

Changed