

Geochronology Manuscript Comments from Referee

2:

The study uses in situ Rb-Sr dating of shale unit fragments and thermal modelling of literature data to constrain the thermochronological evolution with depth of an Australian Proterozoic basin site. It represents an important contribution towards increased understanding of the effects of heating and diffusion from secondary events on isotopic Rb-Sr systematics of basin rock units since few natural studies occur on the matter. The study also highlights the use for coupling knowledge of the thermal evolution in multiple dimensions when interpreting the significance and meaning of geochronological data. The utilized methodology offers a route to achieve that. However, the strength of each specific method and the combination of them is not demonstrated in much detail, as outlined by the following points.

1) The modelling of literature thermometry data with time is considerate and useful with vast amounts of input data, but since secondary mechanisms causing isotopic disturbance also can move laterally, the limit of a one-well model increases the interpretation uncertainty. In the absence of a horizontal modelling dimension, the following considerations should be discussed or clarified regarding the central issue of estimating the boundary conditions isotopic disturbance:

a) How may any lateral variations in the geological setting and the processes and events affecting the rock sequence impact the conclusions drawn on the timing, spatial occurrence and sheer cause of isotopic disturbance given that conclusions are based on a modelled vertical line?

The vast majority of the thermal regime is controlled in the vertical dimension, which in this case is also the dimension in which the best geological constraints are present. Geologically, lateral transfer of heat is minimal, hence the prevalence of 1D models (e.g. Hall et al., 2020). Additionally, modelling in 2D or 3D space would require robust knowledge of the lateral geology which is not available such that it would improve reliability of our models. The only lateral thermal scenario which would significantly impact this model would be the emplacement of a (very) nearby intrusion at modelled stratigraphic levels which would increase temperatures broadly across the vertical sequence. Given there is no evidence for such a proximal intrusion, and thermometers from this well are adequately explained by the currently proposed overburden and bottom-hole intrusion (Figure 7), we suggest the current 1D model is appropriate. Furthermore, no major aquifers have been identified in the Velkerri Formation, suggesting that the potential for lateral fluid flow is unlikely.

b) Migrating fluids are inferred as cause for isotopic resetting beneath 900m depth. Can these fluids be traced by veins, mineralizations, crystal zonations or else? If so,

can direct thermometry or other geochemical characterization be applicable of such? Has this been observed and considered in any previous study of the site? Indifferent of negative or positive answers to these questions, the matter should be mentioned in the manuscript.

Migrating fluids have been observed by oil inclusions in veins cross-cutting the Derim Derim, Bessie Creek Sandstone, as reported in previous studies (Volk et al., 2005; Dutkiewicz et al., 2004). The petrographic textures of the reset shales are also more crystalline when compared to the unreset samples (now provided high-res SEM images in the revision).

2) The in situ Rb-Sr dating is the only new data collected in the study, and the technique is indeed promising and applicable for dating of diverse processes affecting shale units. In order for this analytical campaign to demonstrate the utility of the method, improvements in sample selection, presentation and discussion are due as outlined in the following points:

a) Given the spot size used and the fine-grained nature of illite, each isochron point represent a mixture of grains that may have stabilized isotopically at different times. The authors mention that each sample is predominantly composed of either unaffected or reset authigenic illite as observed by mineralogy alone. If XRD has been used to identify these in this or previous studies of the site, please provide and explain more explicitly the basis of the illite generation identifications. Is it verified that no mixtures of clay mineral phases or multiple clay growth generations (including detrital) are present in any of the samples? If so, how was this verified? If not, please comment on the implications for the age results and the interpretation of its meaning that multiple generations may exist. Please also clarify reasons for excluding grain size separation and Illite Age Analysis (Pevear, 1999) from the study.

We have now provided high resolution images to confirm lack of detrital clay minerals in samples, and elaborated on how such input can affect the resulting ages accordingly. SEM images were used to avoid coarser/more detrital-dominated bedding layers. For reset samples, all illite components (detrital and non-detrital) were assumed to be reset by the Derim Derim Dolerite intrusion. Trace element compositions (Zr, Si, Ti, REEY content) of each spot were also checked to filter detrital component.

Grain separation and illite age analysis destroys petrographic context, and can result in mixed ages. It is also time-consuming relative to the laser Rb-Sr method. This approach also cannot provide additional geochemical information (i.e. major and trace element data) which is an important deficit as it can be difficult to interpret the resulting age if mixed components do occur without these data.

b) Continuing on the illite mixing topic, estimates of the illite homogeneity can also

be provided by dissecting isotopic ratios in each LA spot signal in the absence of grain size separation. Please provide a detailed account on how the procedure of analysing spot homogeneity was carried out, on the outcomes and conclusions drawn from the observations, and mention any implications for the age results going from single downspot time frames to the combination of spots in the isochron diagram.

During processing, each spot was filtered by filtering bad signals (Si, Zr, not stable signals etc.). We like the idea of investigating single-spot isotopes variations for future investigation, but suggest that this is not required here. We calculated single-spot ages to further confirm that each spot in each sample consisted of clay phases that might not be homogeneous, but still form at the same time. This heterogeneity can actually be a positive, providing a good spread in the isochron and resulting in better errors.

c) Relating to the above points, inclusion of other minerals than illite in the LA signals is mentioned and disregarded as merely quartz in the Supplementary Material and therefore irrelevant for the Rb-Sr contents. Relating to the multimineral mineralogy maps, have the signals and spot locations been checked for mineral occurrence? If so, how was this performed? Were any spots rejected for the sake and if so, for what reasons? It was made sure that no K-bearing minerals interfered down-hole in each spot? Clarifying notes of these procedures and results should be contained at least in the supplementary information.

Similar point to previous comment. We have now elaborated further on how data processing is done (signal picking and cropping, checking major and trace elements for etc.) to clarify this.

d) the illite crystal textures and intermineralic textural relationships are qualitatively described without detailed petrographic images or accounts on variations within samples. Can such be added for the specific samples in this study and compared to previous studies describing these features at the site?

Similar point to previous comment again, we have provided more zoomed-in images and elaborate on petrographic content. And also provided additional comments and comparisons of this to Rafiei et al. (2019) and Subarkah et al., (2021) who worked on high-resolution petrography of Roper Group shales elsewhere.

e) The in situ Rb-Sr dating sample set consists of five samples over a ca. 800m depth interval. Given the discrepancy of several hundreds of meters (most shallow effect from the sill is interpreted at 600m or 800m) in the different thermal modelling predictions, please comment on how the sample interval larger than 200m below

696m depth affects the interpretation and uncertainty of the results regarding potential isotopic disturbance of fluid migration.

Four different thermal indicators (Tmax, two different aromatic hydrocarbons, bitumen reflectance, see Jarret et al., 2019) suggest that the elevated thermal gradient occurs past 900 m depths. Although the sub-sample set for the Rb-Sr analyses are sparse, the thermal data sample set are more continuous. Based on this and our thermal modelling, the isotopic disturbance should not occur prior to this depth.

f) The initial Sr values are not anchored to actual data but rather inferred from the isochrones and comes with large error ranges. Since the importance of initial Sr values for tracing crustal fluids and their sources is indeed stated in the manuscript, have any previous data source been considered for narrowing down on them in the stratigraphic sequence, or may new data collection on this be advisable? Given the spread in initial Sr values and their inference from large-error and low-Rb isochrons, it should be explicitly stated that the isochrons produce age errors ranging up to 300 Ma. Many of them overlap each other and several other dating results in the area, and yet their interpretation and meaning is provided without any note or disclaimer. The age errors and their implications for the conclusions based on the dating should be discussed. In addition, the concluding reasoning of the method as a useful discriminator of geological events in sedimentary units should regard the large age error ranges.

Good initial Sr values are limited to the availability of K(Rb)-deficient and Sr-bearing phases (e.g. carbonates) that form concurrently with the illite phases. Sometimes this is simply not available. Ideally, Sr data can be obtained from interbedded carbonates where possible. However, we haven't used the Sr initials from this study to make any interpretations. We also suggest that our method is less derivative as it calculates the initial ratio from the regression of the radiogenic Sr values and doesn't assume that other phases were cogenetic – it lets the illite data speak for itself!

The large errors for some samples is a fair point and hard to avoid in this technique. However data are separable and show meaningful and interpretable effects. This technique strives for accuracy over precision and we suggest that we have demonstrated the efficacy of it here. In addition, we will also attempt to provide the single spot ages for each sample. The population of single spot age results from the unaltered and altered samples should be statistically different from each other.

3) The combination of the methods have been shown to provide thermochronological constraints, but since the authors repeatedly emphasize its

utility, may it be described what actually makes this particular combination so powerful and how it distinguishes from other thermochronological methodological schemes?

Yes, we have now elaborated and compared this with other thermochronological methods. Primarily, other thermochron like K-Ar, Ar-Ar, fission tract (zircon, apatite) that date the surrounding sediments and make inferences on if this applies to the shales (or organic-rich units they want to constrain). They are also destructive to the petrographic context of each sample, time consuming, and expensive. On the other hand, traditional temperature constraints in petroleum systems (Tmax, vitrinite/bitumen reflectance, aromatic hydrocarbons) don't provide age data. This method allows for direct dating of these shales, and couple them with the thermal proxies previously mentioned. This is unique, fast and an affordable way to collect considerable useful data.

Specific comments

L161 Are there any tectono-thermal perturbation that would be expected to have affected the area, and if so, when and of what type? Any orogeny that may have disturbed radiogenic isotopic system?

Recent AFT thermochronology data presented by Nixon et al. (2022) across the McArthur Basin does indicate slow regional cooling during the Devonian-Ordovician Alice Springs Orogeny, attributed to minor regional uplift concurrent with this event. Crucially, there is no observed major structural reactivation within the basin associated with this event. This study does not find evidence for any other thermal perturbation within the basin following the Cambrian. Furthermore, no orogenic reworking is preserved in the McArthur Basin or regional basement in the form of metamorphism, igneous intrusion, large scale folding or angular unconformities, suggesting this region has not experienced major orogenesis following the Proterozoic

L167 Word missing after terminated? Otherwise the sentence does not make sense.

Will reword for clarity.

L280, L365 Avoid subjective adjectives such as good, here and on later places in the text.

Will reword for clarity.

L382-384 How can such a specific statement be motivated considering the large age errors?

Will reword for clarity, but also note that the data are distinguishable despite the errors

L456-458 Generally, chapter 5-8 contains multiple repetitions which can be slimmed. The sentence starting with "This event.." is one of those that includes statements already appearing repeatedly up to this point in the manuscript.

Will reword for clarity.

L488-501 Contains statements repeated from previous sections, but if this has the function of a concluding section it should work.

Will reword for clarity.

L493 Ages are in the text not seldom referred as comparative and relative, e.g. here in mentioning "younger ages". Precise age ranges would have made the text more concise and apt to follow in instances such as this.

Will reword for clarity.

Figure text Figure 6. Avoid the use of "better" and possibly the whole last sentence that can be deemed obvious and irrelevant.

Will reword for clarity.

Figure 5. The color scheme indeed needs adjustment, too many undistinguishable green colours.

Done.

Supplementary Material

L21 What is the last sentence supposed to mean? The signal interval selected in the data reduction procedure? Please clarify, and it is not helpful to put an explanatory word in apostrophes and then not explain it.

Done.

L45 Ideal is an interesting word of choice, would not more ideal for in situ spot-based LA dating at least be that individual grains can be targeted?

Will reword for clarity.

L56 Expressing that the textures do not look detrital should be replaced with a description and/or a detailed, high magnification petrographic photograph forming the basis of these genetic interpretations, which is also a general remark for the mineral texture descriptions (see comment 2d above).

Will reword for clarity.

References

Pevear, D.R., 1999. Illite and hydrocarbon exploration. *Proc. Natl. Acad. Sci. USA* 96, 3440–3446. <http://dx.doi.org/10.1073/pnas.96.7.3440>.