

Geochronology Discuss., referee comment RC2  
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## Comment on gchron-2021-16

Anonymous Referee #2

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Referee comment on "Deformation recorded in polyhalite from evaporite detachments revealed by  $^{40}\text{Ar}/^{39}\text{Ar}$  dating" by Lachlan Richards et al., Geochronology Discuss., <https://doi.org/10.5194/gchron-2021-16-RC2>, 2021

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### General comments:

This manuscript reports Ar/Ar step-heating results from polyhalite in the Salt Range Formation in northern Pakistan. The authors aim to provide geochronologic age constraints for this formation (on the timing of precipitation, deformation, and/or thermal resetting), and to determine diffusion parameters for polyhalite. Polyhalite is a potassium-bearing salt [ $\text{K}_2\text{Ca}_2\text{Mg}(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$ ], formed in evaporite environments, so is an interesting target for Ar/Ar geochronology, especially as this phase has only rarely been analysed via this technique. Unfortunately, it appears that the samples chosen for this study have experienced a complicated geological history of deformation and reheating, with none of the samples yielding Ar/Ar age plateaus. The Arrhenius diffusion results are also non-linear, and likely confounded by dehydration and structural reconfiguration reactions, complicating attempts to calculate diffusion parameters for polyhalite.

The results of this study could possibly be suitable for publication in Geochronology, as even less than ideal analyses may potentially be useful when reporting results for a little-studied mineral or region. However, this usefulness would require the results and discussions to be conservative, noting the pitfalls in the data in a consistent way, and framed to highlight aspects that could lead to improvements. This manuscript has several crucial points that require resolution before it would be suitable for publication.

### Specific comments:

The most important points that need improvement are as follows:

- Line 19: states 'the established early Cambrian age of the formation'. However, Figure

2 indicates that the that the Salt Range Formation is Ediacaran-Early Cambrian. If no other geological constraints are available, the upper limit for the age of the formation is 635 Ma (the base of the Ediacaran) - not 541 Ma (the base of the Cambrian).

- The methods section currently lack sufficient details about the diffusion experiments. The methods section (around line 138) does not detail the technique used to measure the step temperature, which is crucial for diffusion experiments. The methods section must therefore be expanded to explain the methodology for the diffusion experiments and how the diffusion parameters were calculated.
- Line 150-218: the entire section for the step-heating results needs improvement. The current separation of the age spectra and results section into Figures 4 (the 'good' data) and Figure 5 ('erroneous data') is confusing, as some of the samples currently in Figure 5 (06-2.1, 06-3.2) have similar spectra to those in Figure 4. I would recommend splitting the results into 1) aliquots with high K/Ca (i.e., separates of polyhalite), and 2) aliquots with low K/Ca, imprecise step ages, and uninterpretable results. Crucially, for samples with low K/Ca, this observation indicates that the phase sampled and analysed was likely not polyhalite but is instead another mineral with lower potassium.
- Line 150-218: in a revised results section, please reduce the amount of duplicated information between paragraphs and between the main text and tables. Also ensure the results are not over interpreted - caution should be applied in attributing geological significance to the youngest and oldest steps from a disturbed heating spectrum, particularly if there is no reproducibility between aliquots. A conservative interpretation of the data presented in this manuscript indicates: 1) That the Salt Range Formation is likely older than ~500 Ma (i.e., the oldest step with good precision (Fig 6). 2) That the polyhalite Ar/Ar results yielded a broad range in ages from ~500 to 200 Ma, likely due to variable and incomplete resetting of the polyhalite via tectonic and thermal events in the region. 3) That the most recent geological event in the region that affected the polyhalite occurred less than ~200 Ma (based on the youngest step with good precision (Fig. 6)). However, if the youngest step was only partially reset by that geological event (or events), the event could be much younger than 200 Ma - especially given that tectonic activity in the Himalayas is occurring in the modern day.
- Line 153: states that 'Polyhalite single crystals, polycrystals and grain aggregates taken from larger samples underwent step-heating  $^{40}\text{Ar}/^{39}\text{Ar}$  age dating.' However, from the information provided it is not clear which samples were single crystals, polycrystals, or aggregates. This information must be provided somewhere (e.g., in Table 1) for each of the aliquots analysed. Also, please briefly explain the difference between a polycrystal and a grain aggregate.
- The results of the diffusion experiments (lines 220-226) are far too brief. This section only spans six lines, and this text is not useful, only containing a series of numbers already presented on Figure 7. The authors need to explain the choice of samples used for the diffusion experiments, outline how the diffusion parameters were calculated, and describe the features seen on the diffusion diagrams (e.g., slope, slope changes, spike at 500°C).
- As this study represents analyses of a relatively unstudied mineral (polyhalite), it could be beneficial to have a clear list of what worked, what didn't work, and what could be improved for future analyses. There are aspects of this study that were successful include 1) that some samples yielded high and consistent K/Ca values, indicating the aliquots analysed had a consistent mineralogy; and 2) that the samples had high percentage of radiogenic argon (i.e., that polyhalite is reasonably good at keeping the Earth's atmosphere out of its mineral structure). Both are basic - but non-trivial - observations.
- It may also be useful to have some recommendations for future work, including further groundwork studies on polyhalite. Presumably such groundwork studies could be more easily done in areas with a simpler geological history.
- In the documents supplied for review, I was unable to find a table containing the analytical results, which is crucial for reporting Ar/Ar data. Please ensure such a table is incorporated (likely as a supplementary dataset), and that it includes all information

required (see Renne et al. 2009 Data reporting norms for  $^{40}\text{Ar}/^{39}\text{Ar}$  geochronology. Quaternary Geochronology v4 p346-352).

- Several areas of text are poorly written or confusing. Please go over the full manuscript and ensure clarity for both general writing and scientific concepts. Some particularly notable examples include lines 14-18, 24, 62-63, 130, 303-304, 315-318, 326-327 (which has quite a jump in context between line 326 (talking about processes in the Permian) and 327 (talking about modern day processes), 359-366, 371.

### **Technical corrections:**

Line 30: write the chemical formula for halite – all the other minerals in this portion of the text have formulae.

Line 35: start a new paragraph.

Line 59: ‘...the deformation history’.

Figure 1: label the x and y axes as ‘Longitude ( $^{\circ}\text{E}$ )’ and ‘Latitude ( $^{\circ}\text{N}$ )’.

Figure 1: in the caption, mention that the study site is the Kewera mine. On the figure, can the authors write ‘Kewera’ in a different font (e.g., red)? This will be useful to draw the reader to the site.

Lines 88-102: this text is essentially a duplication of information in Figure 2. The main text would be streamlined and improved by removing this text or moving it to the figure caption.

Lines 106-111: the minerals described are not identifiable in the hand-sample images of Figure 3A and 3B. Could the authors also supply other images that identify these minerals if available e.g., close-up photos/thin section photos/SEM.

Figure 3 caption: extra text is required to explain what can be seen in these images. Also, please specify the scale.

Lines 115-138 (and elsewhere in the manuscript): please ensure correct use of superscripts and subscripts.

Line 115: what was the grain size of the crushed aliquots?

Line 118: replace 'alteration' with 'dissolve'.

Line 124: J factors are reported with too many digits and should be limited to significant figures only.

Line 124: which J factors correspond to what samples?

Line 126: what was the frequency of air pipettes? How many air pipette analyses were included in the discrimination calculation?

Line 129: write the abbreviation 'IR' out in full.

Line 155-157: delete the following text as it is poorly written and unnecessary in the main text. 'Apparent age spectra plots display the apparent ages for each step of the experiment and are calculated representing a percentage of cumulative  $^{39}\text{Ar}$  released with the last step resulting in 100%  $^{39}\text{Ar}$  released from the sample. Stacked below each age plot are the K/Ca ratios.'

Line 162-164: provide a reference for this definition of a plateau.

Lines 175-179: this information should be in the figure caption.

Line 188-198: suggest changing to 'Sample 06-1.2 produced very little gas, with low K/Ca values and analyses barely above blank levels, yielding very imprecise ages; the results from this sample have therefore been discarded.'

Table 1: as none of the samples yielded useable plateau or isochron ages, the following columns should be deleted: Plateau Age,  $\pm$ , MSWD, % $^{39}\text{Ar}$ , Inverse Isochron, MSWD. Deleting these columns will free up space in the table.

Line 248: delete 'closed'. If the system has been partially or wholly reset, it is not closed.

Table 2: numbers are reported with too many digits – limit to significant figures only.

Line 277: what are the 'various analogues'?

Line 289: what is meant by 'with variations to pressure'?

Line 300: please explain why that approach was not possible in this study.

Line 315: instead of 'magmatic' do the authors mean 'sedimentary'?

Line 356: here and elsewhere: this should be 'minimum age'.