

How much K is OK? – Evaluating different methods for K-concentration determination and the effect of the internal K-concentration on feldspar luminescence dating

L. Maßon, S. Riedesel, S. Opitz, A. Zander, M., A. Bell, H. Cieszynski and T. Reimann

Responses to referee comment from Anonymous Referee 1:

Anonymous Referee 1: The manuscript by Maßon et al. describes different methods for K-content estimation for feldspar dating and compares them against previously published values. They also provide a review of hundreds of dating studies that shows varied user behavior towards K-content, ranging from neglect to own measurements. Though the issue of K-content is not new, this manuscript highlights that it is by no means solved and future dating studies would do well to add one of the methods described here into their routine dose rate estimation protocols. Though the results suggest a sample-specific (or at least site-specific) K-content estimation is necessary, it was encouraging to see that the choice of estimation method does not significantly affect results. The text is clear and has very nice figures of high quality. The manuscript will be of interest to GChron readers and I had very few comments that need addressing, the rest are mostly typos.

Reply: Thank you very much for your positive and encouraging feedback on our manuscript. We are particularly pleased that you found both the clarity of the text and the quality of the figures appropriate, and that the relevance of the topic for the GChron readership was evident. We respond to your further comments individually below.

1) The data presented by the authors seems strong enough to me that a recommendations of how to refer to the sample fraction being dated could be made. Should the community move towards using the terms ‘feldspar dating’ or ‘alkali feldspar dating’ rather than ‘K-feldspar dating’?

Reply: Thank you very much for this thoughtful suggestion. We fully agree that the terminology used to describe the dated sample fraction should reflect the actual material being measured. As a result, we have added the following sentence to the manuscript in lines 648-649 in the new version to clarify our position:

“We also suggest that unless confirmed by measurements, future publications should refer to the dated mineral fraction as ‘feldspar dating’ rather than ‘K-feldspar dating’.”

2) This study rightfully works only with coarse-grain feldspar samples, but I wonder if the authors could include a few sentences in the discussion on implications for polymineral fine-grain dating. If Na-feldspars are also contributing significantly to the blue signal, then samples without density separation are even more likely to have overestimated K-contents.

Reply: Thank you for pointing out that we haven’t clarify why this is not of relevance for polymineral fine-grain dating. We added the following sentence in lines 531-534:

“An exception to this is polymineral fine-grain luminescence dating, where usually grains with a diameter of 4-11 μm are considered. According to Guérin et al. (2012), the β self-dose of the internal K-concentration is as low as 0.007, which implies that the internal K-concentration does not significantly contribute to the $\dot{D}_{\text{int}}$ in this context.”

3) I’d like to see some images of the μ -XRF and SEM analyses (example spectrum and fit) as supplementary material, since they are central to the study.

Reply: We added fitted example spectra to the supplement as Fig. S4 and referenced them in lines 306 and 320 of the new manuscript.

4) Could you provide a worked example for the stoichiometric calculations for SEM or μ -XRF estimations (using O, Na, Mg, Al, Si, K, Ca, Ti, and Fe)? Since they are not standard methods in luminescence dating, it would be helpful for readers to have a reference.

Reply: To calculate elemental concentrations stoichiometrically, we first fit the spectral peaks of each target element in the acquired spectrum (see comment 3). Next, the net counts under each fitted peak are summed per element and converted to mass concentrations (wt %). Oxygen concentrations are then derived from these oxide formulations rather than measured directly. Finally, these mass concentrations are normalised to 100 %. Table S4 in the Supplementary Material illustrates this workflow step by step. We have added both the detailed table and an explanatory note to the Supplementary Material and have cited it at the relevant points in the main text in lines 305 and 320.

Element	Σ net counts	Mass concentration (wt %)	Normalised concentrations (wt %)
O	-	8.41	50.09
Na	17	0	0
Mg	0	0	0
Al	642	1.38	8.22
Si	6513	5.81	34.6
K	3784	1.08	6.43
Ca	224	0.04	0.24
Ti	88	0.01	0.06
Fe	2298	0.06	0.36
Sum	13566	16.79	100

5) In section 5.3, the authors recommend μ -XRF measurements of single grains.

5a) Could you provide an estimate of how long it takes to measure grains for one sample with μ -XRF? I expect that drawing the polygons is quite time-consuming.

Reply: The preparation of a sample for β -counter analysis is relatively fast and straightforward, taking approximately 5 minutes, while the subsequent evaluation of the data is completed in an even shorter time. On average, we spent about 60 minutes to evaluate one disc using μ -XRF. However, this duration can be significantly reduced with increasing routine. In our case, the measurement time was extended because the grains were selected and analysed in a predefined order to match luminescence measurements. If, as we recommend, grains are measured without requiring this correlation, the overall effort can be considerably lower.

5b) I understand the argument that the spread of K-contents provides valuable information and should be assessed. However, given the effort required to create resin mounts, measure and analyze the μ -XRF data, it might be worthwhile to discuss in detail what this additional information brings. One aspect is to get a realistic uncertainty to propagate into the final age. Looking at the variation in μ -XRF uncertainty, (± 0.1 – 0.6 K% in absolute values, or 1–13% relative to the mean) I am not sure if the effort is warranted only for this information and an educated general estimate might be sufficient. The other aspect is to understand the De distribution better, e.g., to test for partial bleaching. In this case, the spread might indeed be very relevant but this will only be necessary for a subset of samples. For example, De distributions from multi-grain aliquot measurements probably don't need this level of scrutiny. I recommend differentiating more between these two levels of analysis here and in the last

sentence of the abstract because your results show that the bulk measurements are a very good estimate of the mean K concentration that could be relatively easily implemented as routine practice for multi-grain and single-grain dating. It would be a shame if readers were discouraged from this practice by the additional effort of single-grain measurements.

Reply: While answering your second comment we added a sentence in line 531 to clarify that our results are valid for both single- and multi-grain analysis:

“The results presented here are applicable to both single-grain and multi-grain coarse-grain feldspar luminescence dating.”

The advantage of single-grain K-concentration analysis is that it allows for the assessment of the spread within a sample, which in turn enables a more accurate estimation of the uncertainty associated with the K-concentration (see 5.3 first paragraph). We added two sentences to more clearly highlight the importance of the single-grain measurements. in lines 545-549:

“This second step becomes particularly important when the bulk K-concentration is low, as single-grain measurements allow for a more informed assessment of the associated uncertainty. Low bulk values may either reflect consistently low K-concentrations across grains, requiring only a small error estimate, or they may mask a wide internal variability, in which case a larger uncertainty would be more appropriate.”

The embedding process is indeed a little time consuming. Therefore, we have added a brief discussion to the Limitations section in lines 390-409 outlining the advantages and disadvantages of resin embedding for μ -XRF measurements, since we also included answers to Referee 2 specific comment 6 on the penetration depth of the μ -XRF measurements we rephrased the entire section:

“The μ -XRF method is most suitable for polished surfaces. Thus, for best results it requires the same sampling preparation steps as the SEM-EDX measurement inducing the same limitations as mentioned above. Since the penetration depth of μ -XRF measurements exceeds the grain diameter, parts of the epoxy resin below the grains will be measured as well. The epoxy resin used consists mainly of elements that cannot be detected with the μ -XRF device (H, O, C, N). As a result, the resin does not contribute to the measured spectrum, and the relatively deep penetration depth of the method is not problematic in this context. Furthermore, for the measurements, polygons have to be drawn onto the sample area of interest, to define regions of interest for the subsequent measurement. Dependent on the shape, colour and opacity of the grain, tracing the ideal grain shape was difficult, leading to measurements of only parts of the grains in some cases, or alternatively, resulting in measuring resin in addition to the actual grain. If a measurement point within a polygon falls entirely within the epoxy resin, it will influence the average value calculated for that polygon. Particularly in the measurements of sample CSA-1-2-2, epoxy resin was included in the analysed polygons, resulting in lower total K-concentrations in grains for which resin was included in the polygon shape. In all other samples the K-concentrations determined with the μ -XRF is higher compared to the SEM-EDX (cf. section 5.2). Moreover, the embedding process is relatively time-consuming. For routine applications, a direct measurement of grains without resin embedding could be a practical alternative. This approach reduces preparation time but may decrease the measurement precision. However, it must be taken into account that the material on which the grains are mounted (e.g. a single grain disc or adhesive tape) is also included in the μ -XRF measurement due to the large penetration depth of the μ -XRF method. The potential effect of this on the measured K concentrations was not investigated in this study. Since the penetration depth of μ -XRF measurements exceeds the grain diameter, analyses of both polished, resin-embedded grains and unembedded grains can be affected by surface coatings. Nevertheless, this influence is expected to be minimal because the surface layer constitutes only a small proportion of the total irradiated volume, and the signal is largely dominated by the grain interior.”

6) My previous comment assumes that the results would also be valid for multi-grain dating. Is there any reason to think that might not be the case? I also recommend a sentence in the discussion or conclusion about multi-grain aliquots to show how relevant the results are.

Reply: While answering your second comment we added a statement concerning multi-grain analysis in line 531:

“The results presented here are applicable to both single-grain and multi-grain coarse-grain feldspar luminescence dating.”

Technical corrections:

Reply: Thank you also for your technical comments — we have considered most of them carefully and implemented the corresponding changes in the manuscript where appropriate. We also answered some of them in more detail below.

L. 36: Consider changing “measurements” to “estimations”. When I first read it, I thought single-grain luminescence measurements were meant rather than K-content and was confused.

Reply: Done.

L. 42: I recommend changing “external dose rate” to “external sediment dose rate”, since the cosmic dose rate is also external.

Reply: Done.

L. 72: “be not be” should read “not be” and there are two spaces before “by”.

Reply: Done.

L. 80: Suggest changing “influence” to “dominate”, but up to the authors.

Reply: We decided to keep “influence” as at this part of the manuscript our intention is to indicate that low-K grains can influence the overall K-concentration of the sample. This does not necessarily imply that all samples are dominated by low-K grains.

L. 91: No comma before “that”.

Reply: Done.

L. 94: Subscript “Tn”.

Reply: Done.

L.94: Change “effective K of the grains” to “effective K of these grains” if only the suitable ones are used.

Reply: Done.

L. 112: “i” missing in “stoichiometric”

Reply: Done.

L. 135 and 474: Recommend changing “dotted” to “dashed”.

Reply: Done.

L. 143: Change “applying” to “applied”.

Reply: Done.

L. 149: Change “did have no” to “did not have any” or “had no”.

Reply: Done.

L. 164: Change “each 21” to “each of the 21”.

Reply: Done.

L.213: I stumbled over the use of “Na-feldspar” here. Since you go on to show that several of the samples have very low K concentrations despite being in the K-feldspar density fraction, it might be better to use only “sample” here. For example, in line 255, you then refer to the samples’ “K-rich feldspar extract”, which I assume would also refer to sample KTB-383-C. Alternatively, explicitly state which of the used samples should be classified as Na-feldspar.

Reply: Thank you we implemented this.

L. 214: Change “Afterwards treated” to “Afterwards they were treated”.

Reply: Done.

L. 221: Also state briefly what the preparation difference was for MBT-I-2430.

Reply: We added the following sentences in lines 221-223 to include all differences:

“Both samples are rock samples; therefore, the light-exposed outer surface was removed prior to sample processing, and the remaining material was subsequently crushed. Neither sample was dispersed using Na₂C₂O₄.”

Table 1: I would have said “Disc hole size” rather than “Grain hole size”, but up to the authors.

Reply: We decided to retain the term "grain hole" because it is used in the official guide for the Risø Single Grain Laser OSL system (<https://luminescence.dk/docs/SGManual.pdf>)

L. 235: Define what “SGC” stands for and very briefly why the method was used here.

Reply: We rephrased the sentence.

L. 236: Change “is reported” to “are reported”.

Reply: Done.

L. 268: Change “maybe” to “may be”.

Reply: Done.

L. 275: Change “material spread” to “material was spread”.

Reply: Done.

L. 277 and 310: Use either “Rh” or “rhodium”.

Reply: Done.

L. 311: Change “discs” to “disc”.

Reply: Done.

L. 317: stoichiometry

Reply: Done.

L. 326: I would appreciate it if a rough estimate of the proportion of quartz grains was given.

Reply: Done.

L. 335: “, which” should be “that” and “greater three” should be “greater than three”

Reply: Done.

L. 336: Full stop missing.

Reply: The sentence ends with a full stop.

L. 360: Did you mean “negligible”?

Reply: Done.

L. 374 : Remove comma before “that”.

Reply: Done.

L. 387: Maybe I missed something, but why are the μ -XRF estimates usually higher than the SEM-EDX ones? Is this related to different interaction volumes (tear-shaped)?

Reply: We mentioned two reasons in our manuscript in lines 398 to 413. The definition of measurement polygons during μ -XRF analyses is less precise and the accuracy in measuring Na with the μ -XRF is limited (leading to overestimations of K).

L. 449: Change “varies” to “vary”.

Reply: Done.

L. 461: Remove comma after “grains”.

Reply: Done.

L. 462: Change “In case” to “In the case”.

Reply: Done.

L. 466: The use of “less than 20%” confused me. If I understand it right, the lower this number, the better (i.e., better agreement between single-grain and averages). In this case, I feel like “at least...” would be more appropriate. Consider rephrasing.

Reply: It seems that a "not" was mistakenly introduced during rephrasing of the sentence. We have now revised the wording and hope that the updated version in lines 488-491 is clearer and more precise.

“Moreover, fewer than 20 % of the measured single-grains have K-concentrations that align with any of the bulk measurements, the averaged single-grain measurements, or the luminescence-weighted K estimates (cf. Fig. 3 and Fig. S5). This demonstrates that, due to the large spread of single-grain values, it is challenging to represent a sample's internal K-concentration with a single, representative value.”

L. 485: Remove comma after “samples”

Reply: Done.

L. 489: Any ideas on what causes the right-skewness?

Reply: Thank you for pointing this out. We assume that the observed right-skewness in some of the distributions is primarily caused by the presence of a few luminescent grains with higher K concentrations, while the majority of luminescent grains in these samples tend to have relatively low K concentrations. This reflects the overall distribution of K concentrations within the full grain population, which is also skewed in a similar way. Given that negative K concentrations are physically impossible, the left side of the distribution is naturally constrained, further contributing to the right-skewed shape and ruling out a normal or left-skewed distribution in these cases.

We added the following explanation to our manuscript in lines 514-517:

“We assume that the observed right-skewness in some of the distributions is primarily caused by the presence of a few luminescent grains with higher K-concentrations, while the majority of luminescent grains in these samples tend to have relatively low K-concentrations. This reflects the overall distribution of K-concentrations within the full grain population, which is also skewed in a similar way.”

L. 492: Change “Na-feldspars grains” to “Na-feldspar grains”.

Reply: Done.

L. 496 and 497: Remove comma after “recorded” and after “here”.

Reply: Done.

L. 501 and 524: Change “relative” to “relatively”.

Reply: Done.

L. 502: Change “are based” to “is based” (common practice).

Reply: Done.

L. 536, 537, 546, 565 (twice), 568: Dot missing on “Dint”.

Reply: Done.

L. 551: Remove comma after “literature”.

Reply: Done.

Fig. S1 caption: Change “with...” to “where group 1 used” (or change the tense)

Reply: Done.

Fig. S3d: I recommend using a different color for the circles. My first thought was that the red circles were the mirrored ones from panel c.

Reply: We edited the Figure and rephrased the caption: “c) grains on the sticky tape after disc removal. Red circles show locations where no grain was transferred and yellow circles show locations where only parts of a grain were transferred while still parts stuck in the SG disc. d) SG disc after removal from sticky tape. Red circles show position of grains/parts of grains still within grain holes, corresponding to the red circles in c), and yellow circles show part of grains still within grain holes, corresponding to the yellow circles in c).”

Fig. S5 and 4c: Also add in the caption that the dashed lines indicate perfect agreement, since the text in y-axis direction could be misinterpreted.

Reply: Done.

Responses to referee comment from Rachel Smedley Referee 2:

This paper uses multiple techniques to measure the internal K-contents of a good-sized sample suite of feldspar grains from a range of settings, which is rarely seen in such studies so adds value to the literature. The results are very striking, especially Fig. 2, which quite effectively shows that when we're assuming 12.5 ± 0.5 % K (as is routine for most studies), it is inappropriate, even for multi-grain aliquots of such samples (i.e. the effective K). This is something that the literature has been suggesting for some time so it's nice to see a larger dataset really emphasising this point. The paper then calculates total dose-rates and makes comment on how much they would be overestimated using the measured internal K-content, which is useful to provide some context. However, the paper is missing a few key concepts and could do more with the amazing dataset that the authors have acquired and provide additional insights into this very topical and complex area of research within luminescence dating – please see my general comments below.

Reply: Thank you very much for your positive feedback. We especially appreciate that you acknowledged the scope of our dataset — we also believe it holds even more potential besides what we were able to explore in this manuscript. We respond to your further comments individually below.

1) Although this paper is reporting on the internal dose-rates of K-feldspar, it chooses to focus entirely on the internal beta dose-rates that arise from internal K-contents and neglects to acknowledge the internal alpha and beta dose-rates that arise from internal U and Th concentrations that have been shown to be significant in grains of low internal K-contents (see Smedley and Pearce, 2016); thus, internal dose-rates (mostly internal alpha) from low K-content grains have the potential to balance out the 'total dose-rate overestimation' that is suggested by the authors. If the authors wish to make such statements, they should really have measured internal U and Th concentrations for their feldspar grains, in addition to internal K-contents, which are obviously not included. Unfortunately, the findings of Smedley and Pearce, (2016) are only considered in Lines 359-361 in the 'Limitations and Practicability' section when referring to the appropriateness of the beta-counter method, rather than the implications for single-grain internal dose-rates, which are much larger for this paper than the beta-counter method.

I appreciate that it is extremely difficult to measure internal U and Th concentrations and would be very large extension to this paper so do not suggest it is needed here. However, there needs to be more consideration of how internal U and Th concentrations would impact upon the internal K-contents that you have measured. This narrative needs to be included throughout the paper i.e. introduction, discussion, conclusions and abstract. Otherwise, making statements like "These differences result in overestimation of the total dose rate of up to 34.6 % compared to dose rates calculated using measured Kconcentrations" are not fully evidenced. Given that we have the literature and data for this now, it should be included.

Reply: We fully agree that this aspect has so far been underrepresented in the manuscript. Unfortunately, we currently lack the technical means to include internal U and Th concentrations in our dataset. However, we have added the following sentences to better highlight this limitation of our results, and we hope this makes it more transparent to the reader:

Introduction in lines 84-86: "However, it should be noted that if the internal U- and Th--concentrations are high, they may partially counteract the overestimation effect caused by assuming a too high K-concentration (Smedley and Pearce, 2016; Zhao and Li, 2005). Nevertheless, in this study we focus on the internal K-concentration."

Limitations in lines 354-358: "Although it has been shown that the Dint in feldspars arises from U, Th, Rb, and K (Smedley and Pearce, 2016), we focussed on the contribution of internal K concentrations to

the $\dot{D}_{int}$, thus excluding the contribution of the internal alpha $\dot{D}$ to the total $\dot{D}_{int}$. This might induce may result in an underestimations of the $\dot{D}_{int}$ in grains where the internal U- or Th-concentrations are unexpectedly high (see Smedley and Pearce, 2016; Zhao and Li, 2005)."

Discussion (section 6) in lines 560-563: "However, it should be noted that no measurements of internal U- and Th-concentrations were performed. High concentrations of U and Th could lead to an increased $\dot{D}_{int}$, which is not accounted for in our calculations (Smedley and Pearce, 2016; Zhao and Li, 2005). This may also apply to grains with elevated K concentrations (Zhao and Li, 2005)."

And also additions to two sentences in lines 601-605: "In general, we observe a systematic one directional deviation in the total $\dot{D}$ for nine out of ten samples when comparing literature value-based $\dot{D}_{int}$ to measurement-based $\dot{D}_{int}$ not incorporating $\dot{D}_{int}$ that might arise from high internal U- or Th-concentrations. Assuming that measured K-concentrations and calculated luminescence-weighted K estimates are more reliable than literature-based values, and that we do not have high internal U- or Th-concentrations, this systematic overestimation of the total $\dot{D}$ would lead to a systematic underestimation of the luminescence age."

2) Your dataset is so amazing and unique, but you could make so much more of it than you have. As you have performed SEM-EDX analysis, you will have spatially-resolved K-contents for your grains which is incredibly valuable and informative. One of the issues I have with the current narrative is that it does not consider where the K is located within the grain. We all know that feldspar grains are mostly perthitic and very rarely pure K-feldspar, Na-feldspar or Ca-feldspar. So it is important to know where the K is held within the grain, especially if these are potentially heavily weathered feldspars. Is it that there's a very small grain of K-rich feldspar (say 12 %) within a larger grain that is Na-rich (say 1 % K), which averages out to a low K-content for the grain, but in fact you are effectively measuring the luminescence emission from a very small K-rich grain. In this case, should we be applying a high internal K-content but reducing the grain size to calculate the internal dose-rate? Does this even matter if the internal beta dose-rate is effective beyond the size of the grain? These are questions that would be very useful to answer as this is where your data can really extend beyond previous studies... because you have spatially-resolved data but we don't physically see any of it, not even in the supplementary material.

Reply: We fully agree that spatially-resolved elemental data can offer important insights into the internal structure of perthitic feldspar grains and their implications for internal dose rate calculations.

However, in our current approach, we did not conduct truly spatially-resolved K analyses across individual grains. Instead, we used polygon-based integration on polished grain cross-sections to determine average K-concentrations per grain. Our primary focus was to characterize the grain-scale K variability within and between samples, rather than the intra-grain zoning or microstructures.

To achieve meaningful spatial resolution, a full 3D characterization of the grain's internal K distribution would be needed. Since our measurements are restricted to the polished 2D surface of the embedded grains, we cannot assess potential zonation or fine perthitic lamellae in depth. Importantly, a spatially-resolved interpretation of the internal dose rate would require not only elemental maps but also spatially-resolved luminescence measurements from those exact same areas—data that are currently unavailable.

Regarding the suggestion to reduce the grain size assumption in internal dose rate calculations for small high-K domains, we believe this could introduce additional complexity. According to Guérin et al. (2012), the effect of beta self-absorption in feldspars gets smaller with smaller grain sizes. Therefore, even if luminescence originates from a small high-K domain, the lower beta self-dose due to smaller effective grain size could counteract the increase in dose rate from the higher K-concentration. In our view, such compensating effects would need to be carefully modeled, ideally based on both 3D geochemical data

and high-resolution luminescence maps, before any adjustments to grain size assumptions can be justified.

We fully acknowledge the potential of spatially-resolved analyses and agree that this is a promising direction for future studies—particularly those aiming to link geochemical heterogeneity to luminescence emission domains. Therefore, we added a remark at the end of our conclusion in lines 645-647 since we think this should be investigated: “Furthermore, spatially resolved luminescence measurements would be desirable to assess whether luminescence originates exclusively from K-rich domains within individual grains or from the entire grain, regardless of its K content.”

3) Based on your samples, and that which has been reported in the literature, it looks like the beta counter bulk K-content measurements are quite effective at indicating whether single-grain measurements are required. If you have high internal K-contents measured with the beta counter, then the sample is dominated by K-rich feldspar grains. If the beta counter values are lower (i.e. <8 % K), then the sample is probably perthitic and is not dominated by K-rich grains; thus requires single-grain measurements. This is a useful screening approach worth emphasising a bit more in your paper for the community. You do touch upon it in Lines 513-517, but I think you could make more of the screening approach to assess K-rich feldspar vs non-K-rich feldspar, which is essentially what we’re seeing here. If the beta counter shows that it’s K-rich feldspar, then further single-grain measurements are probably not necessary but if the beta counter shows that it’s not, then they are necessary.

Reply: To emphasize the importance of routine K-concentration measurements, we have added a corresponding statement to the conclusion in lines 641-644:

“Based on our findings, we strongly recommend routinely measuring the bulk K-concentration for each sample as a standard procedure of the $\bar{D}$ determination protocol. If the bulk measurements indicate low K-concentrations, thus potentially suggesting either heterogeneous K-concentrations or overall low K-concentrations, this routine should be complemented by targeted single-grain K-concentration analyses to assess intra-sample variability.”

4) I’d be interested to see the overdispersion caused by the internal K-contents you measure as done in Smedley and Pearce (2016). Basically, you can use the age of your sample and the single-grain dose-rates you measure to calculate what the D_e values would have been for each grain (based on the variability in internal K that you measure). Then calculate the overdispersion on these D_e . It’s quite quick to do as you already have grain-specific dose-rates and would give us an idea of the amount of scatter we might expect in a single-grain D_e distribution for a well-bleached sample that arises solely from the internal K-contents. It would be interesting to know whether different samples would need different σ_b values for the minimum age model (MAM) based on the variability caused by the internal K-content. It would also provide more insights into the comparison of scatter in quartz (lower overdispersion generally) and feldspar (lower overdispersion generally) D_e distributions, which is still unresolved definitively. I think that this would add great value to your paper and be key point of reference for citations of all single-grain dating studies.

Reply: Thank you for this suggestion. To evaluate it, we used sample-wise CAM ages (calculated with a fixed K concentration of $10 \pm 2\%$ and D_e values from our dose recovery test) together with each grain’s individual dose rate to generate hypothetical single-grain D_e values. We then calculated the resulting overdispersion for each sample following the approach of Smedley and Pearce (2016). In all cases, the computed overdispersion was effectively zero. The reason for this is that, unlike Smedley and Pearce (2016), we did not have access to an independent reference age and therefore had to rely solely on our CAM-based ages to model the D_e . The uncertainties associated with the CAM ages directly influence

the modelled D_e values, resulting in relatively large errors. As a consequence, the modelled D_e values are statistically indistinguishable within their error margins, which prevents the calculation of a meaningful overdispersion. In our case, the uncertainty of the CAM ages has a greater impact on the modelled D_e than the scatter in the total $\dot{D}$.

Because these results indicate that internal K-content variability does not by itself produce meaningful scatter in a well-bleached sample's D_e distribution, we believe that including this analysis would not add substantive value to our manuscript. Accordingly, we have not implemented this calculation in the revised manuscript.

Specific comments:

1. Title – The first part of the title (“How much K is OK?”) does not add anything to the manuscript and I would suggest removing it.

Reply: We preferred to keep it.

2. Line 17-18 in the abstract I would recommend use the specific citation for each of the values suggested as this has quite important implications.

Reply: Thank you very much for pointing this out. We changed it to avoid any confusion.

3. Line 56-57 – Durcan et al. (2015) did not discover that the internal dose-rate in a grain arises from the U, Th, K and Rb within the grain. You should cite original papers rather than review papers.

Reply: We changed the reference to Mejdahl, 1987.

4. I felt that the literature review should be integrated into the introduction as is normal practise for scientific papers, and I don't think that Fig. 1 is necessary.

Reply: We believe that the systematic literature review warrants its own dedicated section. Literature reviews that are embedded within the introduction are typically brief and often lack a systematic analysis of existing studies.

Furthermore, we consider Fig. 1 to be a meaningful addition, as it provides an immediate visual overview showing that approximately 50% of the studies rely on the K-concentration suggested by Huntley and Baril (1997). It also highlights a critical issue: almost 20 % of the reviewed publications lack reproducibility because they do not clearly state which K-concentration was used and why.

5. Section 3.3.3 – do you have an idea of how much of the grain you are analysing with the SEM-EDX? Please could you state whether you are only analysing the surface of the grains, or managing to penetrate to depths. This is quite important as previous studies have shown that the surface of grains can often have a surface coating that would give anomalously low K-contents if the measurements are only from the surface. It's also been shown that very different K-contents can be measured for different parts of the grain (even with large spot sizes) due to the perthitic nature of feldspars

Reply: We are aware that grain surfaces might exhibit different K-concentrations compared to the grain interiors. However, since the grains were embedded in resin and subsequently polished, the original outer surface was removed. Therefore, we expect this potential surface effect to be negligible in our measurements.

We added two sentences to section 3.3.3 in lines 302-304 to clarify how the measurement works:

“The penetration depth of the measurements depends not only on the measurement settings but also on the material being analysed and can therefore can vary. However, it is generally in the order of a

few micrometres (Kanaya and Okayama, 1972)."

We also added a section to the limitations to point out why polished surfaces would yield more reliable results in lines 381-386:

"Nevertheless, previous studies have shown that the outer surface of feldspar grains can differ in their elemental concentration compared to the interior of the grain (e.g. Smedley et al., 2012) as a result of weathering processes (Parish, 1994). Therefore, polishing offers the advantage of avoiding a bias from potential surface coatings. It should also be noted that, due to the shallow penetration depth of SEM-EDX (on the order of a few micrometres), only elemental concentrations of the polished surface are captured, and any heterogeneity deeper within the grain remains undetected."

6. Section 3.3.4 – same comment as above. Do you know how much of the grain you are analysing?

Reply: The penetration depth of μ -XRF measurements is sample-specific and depends on the matrix composition. However, for silicate materials such as feldspars, the average penetration depth is generally greater than 1 mm. Since all grains analysed in this study are smaller than 1 mm in size, the entire grain volume is effectively irradiated during μ -XRF analysis.

As a result, surface coatings at the not-polished downfacing site (if present) may influence the measured signal. Nevertheless, this influence is expected to be minimal because the surface layer constitutes only a small proportion of the total irradiated volume, and the signal is largely dominated by the grain interior.

It is also important to note, however, that the focal point of the μ -XRF measurement lies at the grain surface.

We added a sentence to section 3.3.4 in lines 316-317:

"The penetration depth depends on the material analysed but is generally greater than 1 mm, thus the collected signal originates from the entire grain, rather than just from its polished surface."

We also rephrased a section of the limitations concerning the penetration depth and potential problems with surface coatings in lines 390-409, since we also incorporated answers to Referee 1 comment 5b, we rephrased the entire section:

"Since the penetration depth of μ -XRF measurements exceeds the grain diameter, parts of the epoxy resin below the grains will be measured as well. The epoxy resin used consists mainly of elements that cannot be detected with the μ -XRF device (H, O, C, N). As a result, the resin does not contribute to the measured spectrum, and the relatively deep penetration depth of the method is not problematic in this context. Furthermore, for the measurements, polygons have to be drawn onto the sample area of interest, to define regions of interest for the subsequent measurement. Dependent on the shape, colour and opacity of the grain, tracing the ideal grain shape was difficult, leading to measurements of only parts of the grains in some cases, or alternatively, resulting in measuring resin in addition to the actual grain. If a measurement point within a polygon falls entirely within the epoxy resin, it will influence the average value calculated for that polygon. Particularly in the measurements of sample CSA-1-2-2, epoxy resin was included in the analysed polygons, resulting in lower total K-concentrations in grains for which resin was included in the polygon shape. In all other samples the K-concentrations determined with the μ -XRF is higher compared to the SEM-EDX (cf. section 5.2). Moreover, the embedding process is relatively time-consuming. For routine applications, a direct measurement of grains without resin embedding could be a practical alternative. This approach reduces preparation time but may decrease the measurement precision. However, it must be taken into account that the material on which the grains are mounted (e.g. a single grain disc or adhesive tape) is also included in the μ -XRF measurement due to the large penetration depth of the μ -XRF method. The potential effect of this on the measured K concentrations was not investigated in this study. Since the penetration depth of μ -XRF measurements exceeds the grain diameter, analyses of both polished, resin-embedded grains and unembedded grains can be affected by surface coatings. Nevertheless, this influence is expected to be minimal because the

surface layer constitutes only a small proportion of the total irradiated volume, and the signal is largely dominated by the grain interior.”

7. It is probably better to integrate the limitations of each technique from Section 4 into the methods description for that technique (e.g. Section 3.3.3) for each approach. It would read more efficiently & more similar to a scientific paper.

Reply: We understand your perspective, but we believe that summarizing all limitations in one dedicated section improves clarity and readability. Especially as some of the limitations apply to more than one of the methods discussed.

8. Line 399 – “This chapter...”. I think you mean “This section...”. This is a scientific paper not a thesis.

Reply: It now reads sections.

9. Some of the paragraph structuring needs revisiting as some are a little short. There are several ‘paragraphs’ that only contain one sentence in the discussion, which really impacts the quality of argument constructed.

Reply: We revised the paragraphing.

Responses to community comments from Sebastian Kreutzer:

Sebastian Kreutzer #1: Thank you for this very interesting and thorough study. I enjoyed browsing through it.

In particular, I like the condensed presentation of the different methods of estimating the potassium concentration and their comparison.

I am confident that this will prove to be of practical benefit to the community. I have two remarks and one question to share.

Reply: Thank you very much for your positive feedback. We respond to your further comments individually below.

1) The variation in potassium concentration among different host rock types is not surprising and is documented in standard textbooks. To enhance your analysis, perhaps you want to consider adding a few lines outlining your general expectations of potassium concentrations based on the origin of the samples (i.e., the host rock). This will allow you to assess the extent to which your assumptions deviate from the general expectation. If this is too much for all studies, you could randomly select approximately 30 to 50 studies.

Reply: The heterogeneity of K-concentrations in the host rocks is indeed not surprising. However, our study aims at the heterogeneity of K-concentrations within potentially K-rich feldspar separates. It is known that our laboratory methods to separate K-rich feldspars from other minerals within a sediment sample are not yielding perfect results (Huntley and Baril, 1987; Woor et al., 2022). Yet, it is generally assumed that only K-rich feldspars emit usable bright luminescence signals when a blue filter combination is used.

We agree that including information on the host rock for each study in Table S1 could be a valuable addition. However, since the majority of the 432 reviewed studies were conducted on sediment samples, we cannot assume a single host rock type. Instead, it is likely that the feldspars derive from a heterogeneous mixture of source rocks, further complicating assumptions about internal element concentrations.

2) In the XLSX table (supplement), it would be beneficial to include a column that represents the signal being investigated. For instance, if the study measured IR-RF on K-feldspar, I would not anticipate any signal interference from other minerals that could potentially impact the results (assuming that K-rich grains contribute to the dose rate heterogeneity in general).

Reply: We agree that including information on the specific luminescence signals investigated in each study (e.g., IR-RF on K-feldspar) would be a valuable addition to Table S1 and could improve the interpretability of signal-related aspects, such as potential interference from other mineral phases. We appreciate the suggestion and will certainly consider including this in future database updates or follow-up studies where a more targeted analysis is feasible.

3) What caught my attention was the result for your sample KTB-383-C. Do you believe that the measured low potassium concentrations may be attributed to the leaching of potassium during the etching process? It appears that this was the only sample subjected to this treatment. You mentioned this treatment; however, it did not appear to be further explored in the rest of the manuscript; please correct me if I am mistaken. If HF was to play a significant role, the sample should be excluded from the general comparison, with general explanation.

Reply: Since we do not have an un-etched feldspar separate, we can not rule out, that the HF etching might influence the K-concentration. Nevertheless, we do not think that the low K-concentration results can be solely attributed to leaching during sample preparation. The sample originates from a

Sillimanite-muscovite-biotite-gneiss and only ~18% of the feldspars can be considered as K-feldspars (Guralnik et al. 2015).

References:

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Huntley, D. J. and Baril, M. R.: The K content of the K-feldspars being measured in optical dating or in thermoluminescence dating, Ancient TL, 15, 3, 1997.

Woor, S., Durcan, J. A., Burrough, S. L., Parton, A., and Thomas, D. S. G.: Evaluating the effectiveness of heavy liquid density separation in isolating K-feldspar grains using alluvial sediments from the Hajar Mountains, Oman, Quaternary Geochronology, 72, 101368, <https://doi.org/10.1016/j.quageo.2022.101368>, 2022.