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Interactive comment on "Technical note: On the reliability of laboratory beta-source calibration for luminescence dating" *by* Barbara Mauz et al.

Anonymous Referee #2

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General comments

This technical note presents the results of an investigation into accurate calibration of beta sources for use in luminescence dosimetry. It presents experimental and modelled data, using these to explore dose rate variations caused by grain size, aliquot diameter and source geometry. The experiments are performed using lexsyg instruments, and some of the results (notably those relating to different source geometries) will be specific to these instruments. Observations regarding grain size and aliquot diameter are likely to be more generally applicable. Beta source calibration is an important issue in luminescence dosimetry, and a number of recent publications have demonstrated that the production of accurate calibrations is not trivial. This technical note has the potential to make an important contribution to that literature. However, the

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manuscript contains a large number of loose ends, errors and unexplained assumptions. Individually none of these points are critical, but they are numerous, meaning that the manuscript will require considerable “tidying up” prior to publication.

Specific comments

1) Throughout the manuscript the authors imply that variation in dose rate due to variation in aliquot diameter is a novel observation. This isn’t true since Spooner and Allsop (2000) investigated the spatial variation of dose rate from a number of 90Sr/90Y sources, concluding that there was a measurable decrease with distance from the centre of the sample holder. They provided a table (their Table 1) listing dose-rate correction factors for different aliquot diameters, and cited two previous studies (Zimmerman, 1970; Spooner, 1987) yielding similar results. The present paper extends these studies, but the concept of a change in dose rate from the centre to the periphery of a sample holder is not in itself new.

2) When considering the experimental data regarding dose rate variation with aliquot diameter, the authors appear to assume that the activity across the face of the source is homogenous. If this isn’t the case, and the literature contains a number of studies demonstrating the existence of inhomogenous sources (e.g. Ballarini et al., 2006; Pawlyta et al., 2019), then effects attributed to aliquot diameter or irradiation geometry may actually result from non-uniform distribution of radioisotopes across the face of the source. In the present study, data were produced using only one example of each source type, meaning that it is impossible to distinguish between source inhomogeneity and other effects. As a minimum, the authors should acknowledge this potential limitation, though the best solution would be to test the homogeneity of the sources directly (e.g. Pawlyta et al., 2019) or make the same measurements on multiple examples of each source type to determine whether the pattern of change remains constant.

3) Dose rate homogeneity across the face of the sample holder (Spooner and Allsop, 2000) and the variation of dose rate with grain size (e.g. Armitage and Bailey, 2006)

are both through to vary with source-to-sample spacing, and possibly also with the dimensions of the active face of the beta source (Spooner and Allsop, 2000). It would be very helpful to the reader if this information was given for each instrument and source used in the present study, since it would allow more meaningful interrogation of the data presented. For example, does the “open ring” have a larger active face relative to the size of the sample holder than for other sources? If so, this might explain why aliquot size does not appear to be an important consideration for this source.

4) The number of aliquots used to produce each datapoint in the experimental datasets is often rather low. For example, only two 8 mm diameter aliquots of R108_4 were measured using the lexsyg SMART closed ring source, and yet all the data for that source in Figure 3 are normalised to that point. Is this really sensible? Well over half the experimental datasets are constructed from five aliquots or fewer. I accept that the calibration samples used are bright and highly reproducible, but reliance on very small numbers of aliquots makes the resulting dataset prone to other sources of uncertainty e.g. non-uniform aliquot preparation, grain(s) between the sample and hotplate, deformed sample holders (Duller et al., 2000) etc etc. The authors should at least comment on why they think that the small number of aliquots measured doesn’t pose a problem.

5) There are a large number of errors in the figures and tables. a) Three aliquot diameters were measured for sample R113_180 using the lexsyg SMART closed ring source, yet only data for the 5 mm diameter aliquots are provided in Table 2. b) In Figure 3a, the caption states that all data are for the 8 mm diameter aliquots, yet this size wasn’t measured for sample F14_90 using the lexsyg SMART closed ring source, and the data presented appear to be for the 5 mm aliquots. Similarly, the data in Figure 3b are normalised to the 8 mm diameter aliquots, but I suspect that the 5 mm datapoint for F14_90 closed ring source is the 5 mm data normalised to itself, which is misleading. c) Figure 4 is the same as Figure 3A – I suspect that Figure S1 was actually intended to be Figure 4, in which case what is Figure S1? Whatever the answer, the correct

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Figure 4 needs to be inserted. d) The data in Figure 7 claim to be normalised to the 10 m grain size and 7.95 mm (reported as 8 mm elsewhere in the paper) aliquot diameter, whereas I suspect they are normalised to the 10 m, 5 mm aliquot. Also, why (and how?) did you perform the GEANT4 simulation without the sample cup? e) I suspect that the data in Figure 8 are normalised to the 10 μm 2.5 mm point in the 7.95 mm dataset, though the caption states that data are normalised to the "10 μm grain size and large aliquot", which I think is the entire starred dataset. Please clarify.

6) I don't really understand the paragraph starting on line 234, which is problematic because it appears to be critical to your explanation of grain size effects with small aliquots. Please expand and clarify.

Technical points

Line 65: The phrase "ring shaped source closed to the top" is awkward. As I understand it there is an "open ring" where the active face is a donut shape constructed of 17 "mini-sources" whereas the "closed ring" is an approximately circular active face constructed from 23 "mini sources". Diagrams in the SI would help clarify this point and are probably required to answer point 3 above anyway. Then, the terminology "open" and "closed" ring would be easier to understand.

Figure 1. Please make the small text larger.

Line 99: Please explain the rationale for using different stimulation powers for different sized aliquots and show which power was used for which size.

Line 109: Here and elsewhere I think the sample cylinder layers are 5 or 10 μm thick not 5 or 10 mm.

Line 110: Why was a density of 1.8 g/cm3 used for the SiO2? This is a good approximation for the density of a homogenous quartz sand with no matrix i.e. an idealised natural sediment from which a luminescence sample could be taken. However, if I understand the modelling work correctly, the sample is represented as stacked discs

of pure SiO2 i.e. it should have a 2.65 g/cm3. If the reduced density is a modeller's approximation of sand deposited as a monolayer on a sample holder, please explain the logic – I won't be the only non-modeller to read this paper.

Figure 2: Please add A and B to the figures. Please also provide a clearer explanation of exactly what Figure 2B is simulating – possibly your revision of the paragraph beginning on line 234 will help with this explanation (see point 6 above). Caption line 119 "plan-view".

Line 153: Here and throughout the paper the terms "fine" and "coarse" grains are used. I accept that this is standard laboratory slang for different grain sizes, but it would be better to be specific e.g. 4-11 μm, particularly given that you use two different sizes of "coarse grain" e.g. line 164. Even if you don't make this change, please move the "(fg=fine grain)" statement somewhere else since it is currently out of place.

Table 3 and 4: Dashed and solid horizontal lines appear more or less at random. Please standardise.

Line 179: You state that for 8 mm aliquots the effect of grain size is insignificant. By what criterion? If by your own stated in Line 171, please say so. I assume that this is the case since the percentage difference for the "4:90" ratio is 6.144$\pm$0.006 i.e. a ratio of 1.06144$\pm$0.00006, which must be statistically significantly different from unity?

Line 181: "6-26%" should read "0.6-26%".

Line 182: "...the magnitude of the difference is controlled by the shape of the source". This sentence requires more explanation.

Sentence starting on Line 182: Assuming this sentence is actually referring to Figure S1, this statement isn't true for the 180-250 μm data measured using the closed ring source.

Line 192: I'm not convinced that the Armitage and Bailey (2006) data "jump" between 50 and 100 μm – it appears that the dose rate increases fairly linearly with grain size

from 0-50 μm, after which it plateaus. As the authors suggest (line 191), the experimental dataset is actually rather similar to the simulated data.

Line 224: I think this should read “>5%” not “<5%”.

Line 239: The figure reference should probably be to Fig. 9, in which case a reduction in the absorbed dose of $\sim$3% looks more appropriate by eye.

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