Geochronology Discuss., referee comment RC2
https://doi.org/10.5194/gchron-2021-20-RC2, 2021


# Comment on gchron-2021-20

Anonymous Referee #2

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Referee comment on "The μDose system: determination of environmental dose rates by combined alpha and beta counting – performance tests and practical experiences" by Thomas Kolb et al., Geochronology Discuss.,
https://doi.org/10.5194/gchron-2021-20-RC2, 2021

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**Review of gchron-2021-20 The mDose-system: determination of environmental dose rates by combined alpha and beta counting – performance tests and practical experiences by Kolb et al.**

In this study Kolb and colleagues present the results of performance tests and experiments undertaken on the mDose system (e.g. Tudyka et al., 2018) using IAEA calibration standards, two loess samples used by the luminescence communities as standards (Nussy and Vokegem), and a range of natural samples taken from the environment. The authors present results of experiments which seek to test the efficacy of the mDose system in measuring IAEA reference materials of known radionuclide composition, and of loess standards which have had radionuclide compositions determined at a number of different laboratories. Two further experiments are then run to i) better understand appropriate measurement times on the mDose and ii) comparing mDose measurements with results from environmental samples measured previously at different labs with different approaches.

The mDose system has only recently been developed, and offers a new means of radionuclide determination from emission counting which can be used for trapped charge dose rate calculation purposes. Whilst thorough testing has been undertaken by the manufacturers, this study offers a broader examination of how well mDose measurements compare with previously made measurements, and investigates appropriate measurement times. The manuscript is reasonably written, structured, and executed and has the potential to make positive contributions to the growing body of literature which uses the mDose system for experimentation, but also the limited literature where cross-comparison of dose rate determinations are made.

It is my view that this manuscript could be suitable for publication in Geochronology. Before I can recommend it fully, I have some comments for the authors to consider, as a well as a number minor corrections, which I hope will help the authors provide better clarity in the manuscript. I would like to urge the authors to review carefully the

paragraph at line 524 about µDose measurements potentially calculating reliable dose rates for samples showing disequilibria. I detail my reservations below, but feel strongly that this paragraph should be heavily amended or better still, deleted. You can't calculate reliable dose rates for trapped charge dating purposes where disequilibria is present without extensive measurement of the decay series (e.g. with HRGS) and with numerical modelling (even then, you can only hypothesise about disequilibria history).

**Comments/Queries**

Paragraph starting at L40: It's worth distinguishing here between emission counting and geochemical techniques, which may be relevant for considering data produced as part of the inter-laboratory comparison.

L88: You mention secular equilibrium here, can you clarify whether the mDose can be used to identify disequilibrium? I guess not because the two U series are only sampled once, and your two Th pairs are fairly close together in the decay series.

L104: Can you clarify please how repeated use of the standards will increase the accuracy of the calibration? To my mind, the accuracy (e.g. how well known the true value is) cannot be changed by repeated measurements, but precision perhaps can be monitored.

L105: Can you also clarify what you mean about the repeated measurements of the calibration? You mention later on in the manuscript that the calibration is repeated approximately every 6-8 months – is this what you mean here, or are you suggesting that the standard should be run after a fixed number of samples?

Sections 3.2 and 3.3: In these sections when reporting the various determined values, there is a switch between mg.kg-1 and Bq.kg-1. These come from the original publications, but it doesn't make it easy for the reader to navigate. It would be very helpful to convert one unit to the other, and this could be included table 4. Also, you present a number of different measurement datasets, but don't provide a rationale for why you chose the dataset that you did – is it simply a case of going for the first published values, or is there a different rationale?

Table 4: I think it would be really helpful here to provide a dose rate for these samples. I can see from the concentrations of U/Th/K that dose rates will be high, but off the top of my head, I don't know how high – this will provide context for later measurement time experiments.

Table 5: I suggest strongly to move this table to the appendix/SI. It's two pages of data

which isn't necessary for the paper and interrupts the flow. For completedness and good reporting practice, can the sampling location data for Heidelberg and Gliwice be included?

L195: Was the sieving of the samples necessary after 45 minutes of milling? If you're filtering out sand size particles, it would be preferable to extend the milling time rather than sieve away these resilient mineral grains because you may be introducing bias.

L231: do you mean for samples with average dose rates?

L248: I advise to change 'acceptable' to 'desirable' in this line – longer measurement times cannot be used as an excuse for not measuring a sample.

Section 4.3.3: this section is long and is passive in terms of not offering any value to the paper. Tabulating it would offer an easier means of digestion for the reader (e.g. columns for homogenisation techniques and determination of the radionuclides and/or dose rates). Alternatively, move this section to the appendix/SI.

L321/325/334/351 (and potentially elsewhere): please avoid the use of excellent when making reference to accuracy/precision/results/reproducibility – it's subjective, descriptive, and meaningless. E.g. what is excellent accuracy? You're better off letting the data speak for itself.

Figure 2: can you offer some further explanation for the data in figure 2 please? When you say repeated measurement, do you mean literally repeat measurements on the same sub-sample, or do you mean that you re-sample the standard and re-measure? Clearly the K is reproducible, but there's more variability in the Th and the U. Is this due to heterogeneity in the sample if you're resampling each time, or is this a reflection of your measurement uncertainty? Is there a reason that the Th shows more variability than the other two radionuclides?

Section starting at L335: I agree with your conclusion at the end of page 17 (L351/2) that the IAEA measurements suggest the μDose is reproducible, and don't think that at first glance there is a problem with μDose (L346). However, there is a deviation of your results for Nussy and Vokegem loess standards and the published values you've chosen for comparison, and this should be further considered. Did you resample and remeasure your standards to assess intra-sample variability? This should be the first step. Then you can consider whether the discrepancies are due to 'methodological problems' (L349) associated with other techniques, and why these discrepancies might exist. It's not enough to state that 'more than 10% are neither unusual for dosimetry measurements …' (L350), especially without any references (see works by Hossain et al., 2002, De Corte et al., 2007, Williams et al., 2010). Once you've considered the sources of these discrepancies, placing the deviations in the context of calculated dose rates would be a

positive way of reassuring the reader that you're talking about minor absolute variations in U/Th, and that these don't have a significant impact on overall environmental dose rate, and therefore, age calculation.

Section 5.2: This is a really interesting section, and likely very useful for µDose users. As a general comment, I'm a bit lost understanding how the number of counts relates to i) time and ii) dose rate. Of course, these are sample specific, but for example knowing the dose rate of the loess standards and how long it took to reach 1k, 2k, 3k counts (for example) would be very helpful to the reader.

L379: You start to explain the experiment here, but I don't quite understand how these measurement duration experiments were undertaken. Were you making only one long measurement (up to 5/7k counts) and integrating counts for the shorter count times, or were you making numerous measurements (e.g. 0-250 cts, then 0-500, 0-1000 etc)? The former is preferable in my view.

Paragraph starting L477: can you say more please about suspected disequilbirum issues, drawing from the literature about how disequilibrium might manifest in fluvial sediments. You have pretty high Th values for these samples. Olley et al.'s 1996 study finds that U238 disequilibria is more common for their modern fluvial samples than U235 or Th. For all the samples you mention, Th is higher from the µDose system than Giessen TSAC/ICPOES – why would this be? Also, can you comment on the data for sample Gi455, where the Giessen and your data diverges for all three radionuclides. K won't suffer from equilibria issues, yet your measurements is 4 times greater than the Giessen one. Is this an experimental issue? Why do you think there's such good agreement for this sample when you look at the comparison for HRGS in Figure 8b? Please offer a fuller explanation at L506 when you return to this issue

Paragraph starting at 525: I suggest most strongly that you delete this paragraph, it's not correct. Dose rates calculated in the context of disequilbria are not accurate for trapped charge dating, no matter how precisely you can determine radionuclide concentrations, because ionisation is assumed to be constant through time. You haven't discussed how COL-UGW1-4 samples have been identified as being in disequilibrium or discussed the extent to which this results in excess/loss across the U/Th chains and how this might impact final dose rate calc.

L533: "At the moment, we cannot decide whether our preliminary results are only an odd anomaly or an indicator for the µDose-system's capability to produce reliable dose rate estimates even for samples suffering from radioactive disequilibria". µDose is a measurement tool for determining radioactivity. As far as I'm aware, it cannot be used to identify disequilibria in the U/Th decay series, and even if it were, there's no way of knowing of reconstructing the radioactivity history of a sample throughout it's burial history.

L534: “In order to give a final answer, further detailed and systematic investigations are required, including the question whether the magnitude of radioactive disequilibria is a decisive factor for the µDose-system’s capability to determine correct values”. I am entirely convinced that µDose can accurately and precisely determine U/Th/K and therefore infinite matrix dose rates. However, these infinite matrix dose rates rely on two fundamental assumptions i) of equilibrium and ii) of an infinite matrix.

Table10 and the paragraph starting at L537: I personally don’t think this table or section is relevant. Why wouldn’t µDose be able to handle samples from a broad range of depositional settings? The problem only comes when the two dose rate assumptions mentioned above cannot be applied.

**Minor corrections/typos**

L23: hyphenation not required, instead “heating events, and for ESR dating, the precipitation of minerals”

L30: replace “minerals are not stimulated any more” with “minerals are no longer stimulated”

L30: replace “are still” with “remain”

L31: remove comma midway through the sentence

L32: insert “the” in front of palaeodose

L44: replace “as well as” with “additionally”

L51: remove “here”

L58: I think you mean detection rather than determination

L71: Rewrite sentence to "..an Analogue to Digital Converter (ADC) samples and transforms the …"

L74: Replace "allowing to discriminate" with "allowing discrimination"

L75: Rewrite sentence to: "..pulses, as well as the elimination of background pulses caused by interfering variables"

L103: Replace "reveal rather" with "have"

L204: Replace "might have a tampering effect on" with "may impact"

L365: Correct 'acitivity' typo

Table 7: is "1.04 ± 0.003" a typo for the Nussy loess K% on Ahnert value?

L368/9: replace 'rather fast' with 'relatively rapid'

L369/70: amend sentence to "without the need for storage for specific periods of time"

L371: do you mean precision instead of quality here?

L385: replace "view" with "a few"

L557/558: should be one paragraph

L565: these pieces of software should be referenced