

This is an interesting study that provides new data around the behaviour of the ESR signals in quartz from young (Late Pleistocene to Holocene) sediment. In particular, the authors evaluate whether the ESR signals associated to the Al and Ti centres are actually fully reset during sediment transport and prior to deposition. This is done by comparing the ESR dose estimates with those independently obtained from OSL measurements. As a matter of fact, the ESR dose values are significantly (one or two orders of magnitude) higher (between 150 and 1350 Gy depending of the centre considered) than the OSL ones (15 to 35 Gy). This indicates that the samples have not been fully bleached during transport, resulting in a massive dose (and age) overestimation if the unbleached component of the signal is not taken into account. Given these results, the authors recommend the use of modern-analogue samples to evaluate whether the ESR signals of Pleistocene samples have been fully reset.

The paper is very well written, easy to read and some of the conclusions are supported by the data displayed in this work, although the general take-home message should be in my opinion somewhat moderated (and especially the last sentence of the abstract: 'The results of this study suggest that fluvial sediments carry a significant residual dose, and therefore the subtraction of residual dose using a modern analogue is highly recommended to obtain reliable ESR ages.'). The conclusions may be valid for this specific set of samples, but should not be universally extrapolated to all samples in any sedimentary context. The present study is based on a limited number of samples, which all come from the same sedimentary environment, and supposedly have the same bleaching and deposition history. It is therefore not surprising that they all provide consistent results. The sedimentological characteristics of the sediment are known to possibly impact the level of optical bleaching achieved during transport (e.g., Voinchet et al., 2015). Moreover, there are many works in which ESR dating (using Al, Ti-Li and/or Ti-H signals) provided consistent results with independent age control based on Luminescence, Cosmogenic, Palaeomagnetism or Ar-Ar methods (see an overview in Duval et al., 2020b), for chronologies ranging from the Early Pleistocene to the Late Pleistocene. These should be considered as empirical evidence indicating that the ESR signals have indeed been fully reset (i.e., zeroed for the Ti signals, and reset to the unbleachable level for the Al signal) in frequent occasions, and that the so-called 'residual dose' is not significantly impacting the results. My point here is not to minimize the importance and relevance of the present results, but just to put them in a broader perspective. Whether the ESR signal is fully reset may depend on many factors, including the transport conditions, and nature and composition of the raw sediment. The present study is based on samples from a single environment that is perhaps simply not favourable to ensure complete bleaching of the ESR signals. Finally, there are several studies for which the D_e values derived from either Al or Ti centres are <100-150 Gy (see an overview in Bartz et al., 2020), i.e., below the dose estimates obtained in the present work. This indicate that the magnitude of this so-called unbleached (residual) component is sample- and environment-dependent. This aspect would deserve to be mentioned in the present work.

The above mentioned papers (Bartz et al., 2020 and Voinchet et al., 2015) have been referred in the introduction.

Additionally, although the 'residual dose' may be due to an incomplete bleaching of the ESR signals prior to deposition, other sources of uncertainty should also be explored, as they may impact the dose evaluation. For example, there is no mention at all of the real uncertainty associated to the ESR measurements (see point #2 in General comments), as well as of the potential bias simply induced by the way the ESR intensities are measured (see point #1). If the authors could explore and discuss these questions in the manuscript, that would surely bring added value to the study.

We have defined the residual dose in this study at the end of the introduction to avoid any confusion.

General comments

1. Identification of the ESR signals and measurement of ESR intensities.

There is still little consensus within the community around the way Ti ESR signals are identified, measured and reported. First of all, the Ti-H signal is made of a series of doublet, one of them being centred at $g = 1.915$. This one overlaps with the Ti-Li peak @ $g = 1.913$. Consequently, the peak reported as 'Ti-Li' by the authors on Figure 1B and throughout the manuscript (called option D in Duval and Guilarte, 2015) is actually resulting from the mixed contribution from Ti-H and Ti-Li signals (e.g., Toyoda et al., 2000). Therefore, this should be reported as 'Ti signal' or 'mix Ti', but not as Ti-Li (unless the authors can demonstrate that the Ti-H component has no impact on the peak). The only way to measure the single contribution of the Ti-Li centre is to measure the peak intensity @ $g = 1.979$ (Option E in Duval and Guilarte, 2015; Absorption line 1 in Beerten et al., 2020). If option E gives the same result as D, then it would be fair to consider that the Ti-H component has no significant influence on the peak intensity measured @ $g = 1.913$.

The option D of Duval and Guilarte, 2015 is still dominated by the Ti-Li centre and the contribution of the Ti-H centre was considered negligible (Duval et al. 2017). The potential influence of the Ti-H centre had been already mentioned in the text.

The authors decide to use an unusual way of measuring the Ti signal (so-called option B in Duval and Guilarte, 2015; Absorption line 2 in Beerten et al., 2020). This one is not frequently employed for dating purpose (I only recall Beerten et al., 2020). Therefore, their results are not directly comparable with all previous studies that are cited in the MS, since they are mostly based on Option A (as per reported in Duval and Guilarte, 2015) (e.g., Timar-Gabor et al., 2020; Rink et al., 2007) or option D (Tissoux et al., 2007). This is why I would encourage the author to report the data from options A and E (at least in SM), so that : (i) the present work can be directly comparable with other studies, and (ii) we can evaluate whether the dose results significantly differ depending on how the signal is measured.

When we started the measurements we did not plan to compare the residual dose of the Ti centre with different charge compensators. The Al and Ti centres were measured separately, using a small sweep width for the Ti centre, and unfortunately Opt A and E were not recorded (see Figure 1b). Figure 1a was only recorded once to show a complete spectrum in the publication. The correlation of our measurement options of the Ti centre to those of Duval and Guilarte (2015) has been added in the text of section 3.

2. Measurement procedure and uncertainty associated to ESR intensities.

Unlike for tooth enamel, ESR measurements of quartz samples at low temperature are usually characterized by a non-negligible uncertainty (at least a few %) that may result from a combination of different factors, such as the stability of the ESR spectrometer, the heterogeneity of the quartz samples, the angular dependence of the signal, the position of the tubes in the cavity, etc. Although ESR measurements are usually performed under controlled conditions, measurement precision for the Al and Ti signals is typically of at least a few %. This has a non-negligible impact on the D_E values, which is why repeated measurements should be performed in order to evaluate the repeatability of the D_E estimates (e.g. Duval et al., 2017, 2020a). A variability of at least 10% may be frequently achieved (at least with the MAAD method) and it may sometimes be >30%, casting thus in this case reasonable doubts on the reliability of the ESR data obtained.

During the measurements, meticulous care was taken to ensure that the measurement temperature, sample quantity, sample tube positioning, spectrometer stability and sample rotation procedure always remained the same for all measurements.

In their measurement procedure, the authors take into account the angular dependence of the signal by rotating the tube 3 times in the cavity. So I understand that they derived a mean ESR intensity value and associated error, which was then used for DRC fitting and dose evaluation (correct? please clarify in the MS). So, I wonder:

Yes, this is the way we calculated the mean ESR intensity and associated error. This information has been added to the MS in section "ESR measurements".

- About the magnitude of the experimental error derived from the rotations in the cavity? This error may significantly vary depending on the signal considered. It would be interesting if the authors could comment on this.

Relative standard deviation (1-sigma, %) of the three measurements for each signal at different irradiation steps is shown below.

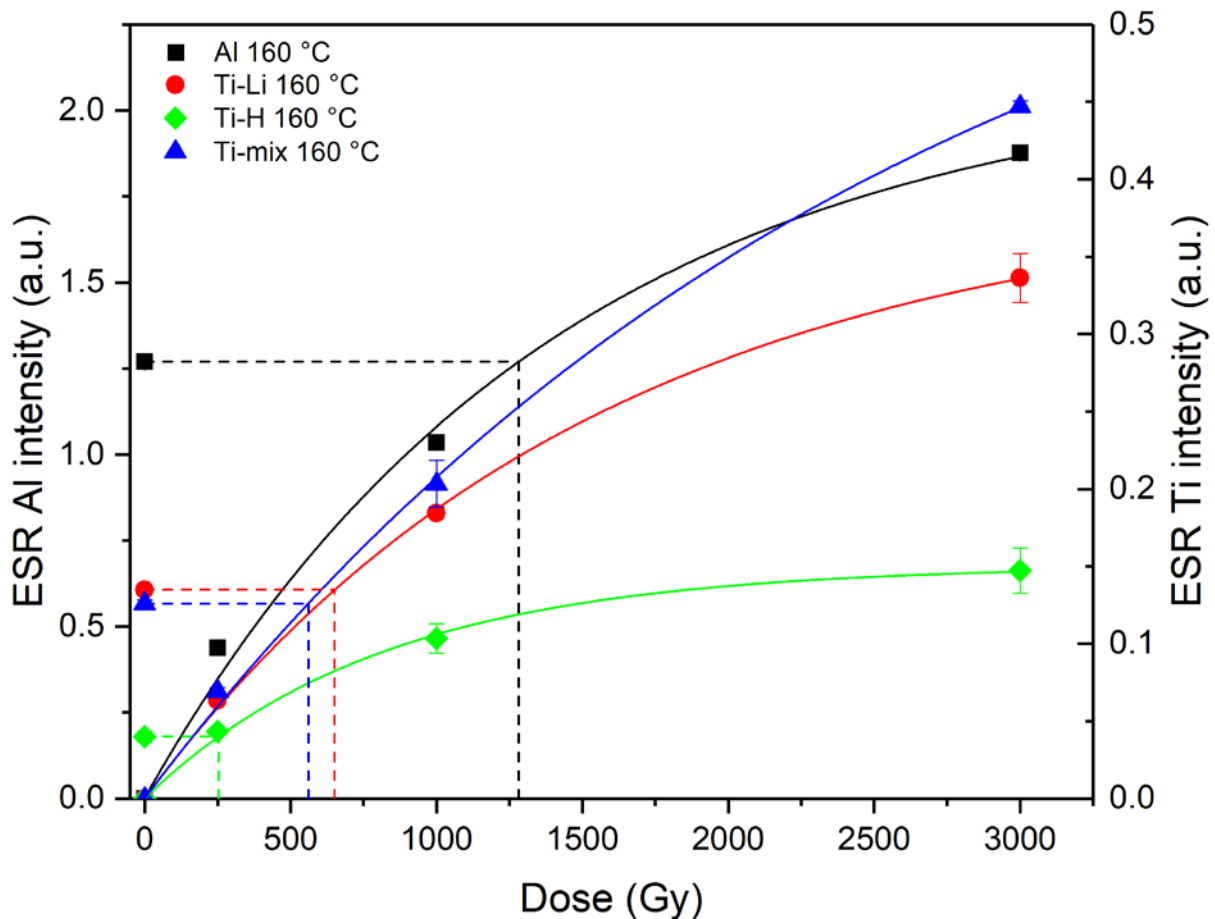
In % of mean intensity	Al centre	(Al after subtraction of peroxy)	Ti-Li	Ti-mix	Ti-H
Natural	0.6	0.9	2.4	8.4	16.4
Dose 1	0.9	1.3	2.8	2.6	4.2
Dose 2	0.8	0.9	2.3	5.3	5.3
Dose 3	1.0	1.1	3.0	4.2	9.4

- In which extent this experimental error may impact the De value? This should be discussed.

The cause is probably the turning of the sample within the cavity. In the case of Ti-H the size of the experimental error is largest for the natural sample probably due to the relatively small natural signal intensity. The fitting of the dose response curves went well for all samples, a $R^2 > 0.99$ was generally achieved. Therefore, we can assume that the majority of the experimental error arises from the turning of the sample within the cavity.

Moreover, there is no Dose Response Curves (DRC) displayed. Can the authors provide some examples in the MS for the different signals measured?

This Figure shows the DRCs for sample RB-II for the four used ESR signals at 160 °C preheat. This figure has been added (is Figure 2B).



Apart from the angular dependence of the signal, there is no mention of any repeated ESR measurements, while it is known that the variability of the ESR intensities over successive days of measurements may be significant. And this variability may be even higher when using a HS cavity (compared with a ST cavity). This may be especially crucial for the Ti-H signal, whose ESR intensity is the weakest of all signals, resulting in significant experimental errors that may sometimes exceed 10%. In other words, a poor De repeatability could possibly partly explain the dose results obtained in this work, which is why this should be evaluated for the present set of samples.

Finally, only 1 single aliquot has been measured per sample, but again, we have no idea of how representative is this aliquot of the whole sample. It seems essential to measure a few aliquots per sample in order to evaluate the dispersion of the results. I imagine this would be a standard procedure in OSL dating, and I would recommend to do it here in order to evaluate in which extent the homogeneity of the sample may impact dose estimates.

The robustness of our measurement procedure had been tested by the dose recovery test and the result was already discussed. Regarding intra-aliquot scatter, one aliquot (60 mg of quartz) contains a few thousand grains of quartz. So any grain to grain variabilities in pre-burial bleaching must have been well averaged. Moreover, the purpose of this study is to investigate the range of residual doses from a set of fluvial samples, not to date individual samples. We do not think measuring more aliquots would add significant more values in this paper.

I realise that these extra measurements might be time consuming, but they can actually be carried out much faster with the SAR method than the MAAD with given the limited number of dose steps and irradiation times. My opinion is that all these issues should be thoroughly explored to see in which extent they may significantly (or not) impact the dose estimates.

We do not think that the laboratory working time for SAR is shorter than MAAD, because we do X-ray irradiation, preheats and ESR measurements sequentially in the laboratory. Since SAR estimates D_e by an interpolation of a dose response curve, it does not need many number of dose points. Only the significant issue, which can affect the accuracy of the dose measurement is the potential sensitivity change by the annealing step, which was tested by the dose recovery test.

3. Residual dose

This might go beyond the scope of the present paper, and would perhaps deserve a proper consensus within the ESR dating community, but I am personally struggling with the use of the term 'residual dose', for two reasons. First, I am not sure whether the term is actually correct: technically, it should rather be a 'residual signal', which can then be converted into a dose value. Second, I find the term 'residual dose' misleading, as it may correspond to either (i) the unbleachable component of the Al signal, or (ii) the remaining component of the Al and Ti signals due to incomplete reset prior to deposition, (iii) or sometimes even both.

Moreover, the comparison of residual dose values from the various signals may be biased by the unbleachable component of the Al signal (see specific comments #8 & #9). This one should be subtracted to the so-called 'residual dose' in order to make it directly comparable with those from the Ti centres. Actually, I am not sure why the magnitude of this residual unbleachable component of the Al signal has not been evaluated in the present study using a sunlight simulator, so that it can be subtracted from the signal of the Natural and Gamma-irradiated aliquots. And I wonder in which extent this unbleachable component of the Al signal may impact the dose recovery experiment (and the poor ratio obtained), since this component cannot be optically reset (but thermally yes).

We have clarified the definition of the residual dose for the Al centre in this paper always includes the unbleachable part of the centre in the introduction. The reason we did not evaluate the unbleachable part of the Al centre was that there is no need if there are appropriate modern analogue samples are analysed and the residual dose from such samples were subtracted.

Specific comments

1. Abstract:

- I.4: does the value of 1350 Gy obtained for the Al centre include the unbleachable component of the signal? if so, this should be mentioned.

Yes, the unbleachable signal part is included. This information has been added to the abstract.

- I.8 to 10: I would suggest to add a sentence indicating that the dose recovery ratios obtained for Al and Ti-H signals suggest dose estimates may be significantly overestimated and underestimated, respectively.

This information has been added to the abstract.

- I.9 to 11: Again, I would suggest some moderation in the final sentence: the results obtained by the authors indicate that the fluvial sediment samples **that have been investigated in this work** carry a significant residual dose, but this conclusion should not be extrapolated to all samples in any context. And especially for the Al and Ti-H signals given their dose recovery ratios.

The reviewer is right. This fact has been clarified in the text now.

2. I.20-22: there is also significant variability among the samples for the bleaching kinetics of the Ti-H and Ti-Li signals (see Tissoux et al., 2007; Duval et al., 2017). This may be mentioned.

This information has been added to the text.

3. I.22-23: The two parts of the sentence are not incompatible. The signal may be fully bleachable and incompletely bleached due to insufficient exposure to sunlight. Please rephrase without the 'although'.

The sentence has been split and 'although' has been removed.

4. I.24: studied or reported? All the publications from the 'French' group systematically provide the so-called 'bleaching coefficient' for the Al centre, which is the relative intensity of the unbleachable component (e.g., Voinchet et al., 2020; Duval et al., 2017, 2020a).

We have mentioned Voinchet et al. 2015 about the bleaching index and residual doses after subtracting the unbleachable residual of the Al center now in the MS (section 1).

5. I.24: use 'residual signal' instead of 'residual dose'

The aim is to quantify the residual and to make it comparable to other studies (Tsukamoto et al., 2017 and Timar-Gabor et al., 2020). Therefore, dose is the chosen unit.

6. I.24, 'So far very few studies have reported residual doses of the quartz ESR signals from sediments': just to be clear, you mean residual component due to the incomplete reset of the ESR signal prior to sediment deposition?

Yes, this is what we are referring to. The sentence has been rephrased to, "So far very few studies have reported residual doses of the quartz ESR signals from young or modern analogue samples, which could be directly comparable with the quartz OSL D_e values."

I.26: again, technically, these are not residual doses' but rather 'residual signal that correspond to dose values of XXX'.

The aim is to quantify the ESR residue, to make it comparable, and not just to report a dimensionless unit of intensity. Regardless, since there is no clear definition in the scientific community regarding the term "residual dose" it should be specified in the text, as the reviewer stated above, which of the three possibilities is meant. Here, the definition is given in the context of the paper itself and the papers, which we refer to.

7. I.27-29: Question: does the so-called 'residual dose' reported by Tsukamoto et al. (2017) for the Al centre includes both unbleachable and unbleached (due to incomplete reset) components ? This should be clarified, otherwise these large numbers provided for the Al centre may be misleading.

The residual doses of the Al centre reported by Tsukamoto et al. (2017) include the unbleachable signal part. This information has been added to the text now to clarify.

8. I.28-32: I believe the same comment applies to the study by Timar-Gabor et al (2020): in order to be discuss values that are directly comparable for the Al and Ti centres, the author should report the so-called residual dose for the Al signal without the unbleachable component. Otherwise, this may be again misleading: the huge apparent residual dose for

the Al centre is actually made of 2 components, with one of them that cannot be optically reset. The standard procedure in the dose evaluation associated with the Al centre does include the subtraction of the unbleachable component of the signal (e.g. Voinchet et al., 2020; Duval et al., 2017, 2020a)

The determination of the unbleachable signal part requires extra ESR spectrometer time and extensive solar simulator time. Therefore, it may not always be possible to determine the unbleachable signal part of the Al centre. If it is noted that the information given is the total dose/total signal, this should be regarded as sufficient.

9. I.34: again, what does residual dose mean here? The magnitude of the unbleachable component is systematically reported in all the studies mentioned (cf., 'bleaching coefficient' in the tables).

This has already been answered to in 5.-7.

10. I.35: Toyoda and al do not report ESR any age estimates in that paper.

This has been corrected to ESR intensities.

11. I.45: why for the first time? The authors report many previous studies with a similar approach (Beerten et al. 2006; Tsukamoto et al., 2017; Timar-Gabor et al., 2020

We report ESR residual doses for the first time for a fluvial sedimentary environment. Tsukamoto et al., 2017 and Timar-Gabor et al., 2020 report residual doses for aeolian environments. Beerten et al., 2006 QG in fact state for sample MS9 with total Ti-Li De of 53+-5 Gy "The existence of a significant nonzero Ti-Li equivalent dose for the youngest sample (MS9) strongly indicates the presence of an unbleached or unbleachable residual dose in this sample." But again, aeolian deposits.

12. I.48, section 2. Samples:

- Just checking, the samples analysed in the present study are the same that have been analysed earlier by Lauer et al (2011): i.e., these are the exact same prepared quartz ? or do they come from a another sample preparation? This should be clarified in the MS.

Yes, the exact same samples as used by Lauer et al. (2011) have been used. No additional preparation has been done. This has been clarified in the text now (section "Samples" of the MS).

- I.48: The authors should provide a basic description of the sedimentary environment (facies, grain size) for each sample: these parameters are known to impact the reset of the ESR signals (e.g., Voinchet et al., 2015)

The information of the grain size has been added to the MS (see below). We added a table with a brief description of the samples (Table 1) and a cross reference to the Lauer et al. 2011 paper to section "Samples" of the MS.

- I.48: gain size of the samples dated ? (this can be added to Table 2)

The grain size for all the samples were 100-250 microns (same as for the luminescence dating carried out by Lauer et al., 2011). This information has been added to section "Samples" of the MS.

- I.50-55: Unfortunately I could not access the paper by Lauer et al. (2011). Would be good if the authors could provide some additional info about how the OSL D_e values were calculated (single grain? Single aliquot? How many aliquots/grains measured to get the mean D_e values? etc.)

The SAR protocol has been used by Lauer et al. (2011) to obtain D_e . This information has been added to the section “samples” of the MS. Lauer et al. (2011) do not provide any information of how many aliquots were used to get the mean D_e values from OSL measurements, but from their diagrams, it must have been ~50 small aliquots per sample were measured.

14. I.59, Section 3. ESR measurements:

- I.65: ‘modulation amplification’ or ‘modulation amplitude’?

The text has been corrected to “modulation amplitude”.

- I.66: remove ‘of the spectra’

‘of the spectra’ has been removed in this sentence.

- I.68: what does ‘homogenous value’ mean? What about rephrasing as ‘to take into account the angular dependence of the signal’ instead ?

Yes, that is what it was supposed to say. For clarification, the text has been changed to the reviewer’s suggestion.

- I.69: a microwave power of 10mW for the Ti signals seems too much. Have the authors performed a microwave saturation curve ? Our data show that the Ti signals saturate at such power, which is why we usually measure using 5 mW.

Previously, we measured the microwave power dependency on a sample of Chinese loess and did not observe any saturation up to 20 mW for the Ti centre.

- I.74: that is correct, and therefore the peak @ $g=1.915$ does not result from the single contribution of the Ti-Li signal, but from a mixture of both centres. This should be reported in this way throughout the manuscript.

Other authors also used the term “Ti-Li” (e.g. Tissoux et al. 2008). In the MS, we clarify that “Ti-Li” this signal is also affected by Ti-H. We added a reference to the Duval and Guilarte 2015 paper labelling of the Ti centres.

- I.79: What is this reference ‘Tsukamoto, 2019, unpublished’? Is this a technical report? Not sure this should be mentioned here as it cannot be accessed.

A paper containing this information has been already submitted to RM and is in review stage at the moment. Therefore we changed the reference to “Tsukamoto et al, submitted”.

18. I. 99, Subsection Equivalent doses, residual doses and ESR ages:

- I.109: I am not sure in which extent the OSL dose rate can be directly used for ESR. Maybe they should be adjusted. There are some specificities, like the alpha efficiency of 0.07 (Bartz

et al., 2019b), that should probably be taken into account for a small (but non-negligible) internal dose rate and external alpha dose rate components.

Dose rate is the dose absorbed by the host material. We think it is logical to use the same dose rate as the quartz OSL.

- I.111: 'These residual ages show how significant the effect of the residual dose **may be** in ESR dating of fluvial sediments.' This is valid for the present set of samples. Not all samples behave the same.

The text has been changed to the reviewer's suggestion.

19. Subsection Dose recovery test, I.121: in which extent the unbleachable component of the Al signal may impact the dose recovery ratio ?

This is a good question, but we do not know yet.

20. I.127: 'The results clearly show that the ESR D_e for all samples are significantly larger than the OSL D_e of Lauer et al. (2011) and therefore residual subtraction is highly recommended'. 2 comments here: (1) if this residual component resulting from incomplete bleaching of the signal is not subtracted, then the ESR age results should be regarded as maximum possible burial ages; (2) residual subtraction would require the use of modern-analogue samples, but this should not be regarded as the perfect solution: it is based on the assumption that modern-analogues and Pleistocene samples that are being dated have experienced similar transport and bleaching conditions, which can not always be verified. Therefore, subtracting the 'residual' signal derived from modern-analogue samples may introduce another source of uncertainty in the equation, and does not ensure that the ESR signal of the dated sample has actually been fully reset.

We also do not think that the residual subtraction using modern analogue would be a perfect solution. However, we obtained relatively uniform residual doses at least from from the Ti centre (Ti-Li and mix) and therefore this value can be used for instance, the higher terrace sediments. This is at least a better approach than not subtracting.

21. Figure 1: I think there is an issue with the peaks reported for the Ti-H signal. There should be doublets for each g value, and there is actually an overlap on the Ti-Li peak @1.913 (e.g. Toyoda et al., 2000).

The indicator for peak g_3 for the Ti-H signal has been corrected and the indicators for the doublets have been added to Figure 1.

22. Figures 3 and 5: does the Al residual D_e values include the unbleachable component ? I believe it should be subtracted, in order to make the all data really comparable.

Yes, the D_e values for the Al centre in Figure 3 and 5 include the unbleachable component. We did not estimate the size of the residual signal by a bleaching test, because if we subtract a residual dose, preferably obtained from a same set of sedimentary sequence, there is no need to determine the size of bleachable/unbleachable components of the Al centre. We consider it is a huge advantage of this approach, considering the fact that we needs to change a lamp of the solar simulator every time we do a bleaching experiment (for 1000 hours).

Table 1, footnote: *including unbleachable signal component

The footnote in Table 1 (and 2) has been changed to the reviewer's suggestion.

References

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Mathieu Duval, Burgos, 14/05/2021.

PS: not sure why, but the numbering of the sections and bullet points changes from the Edit to the Preview versions. Hope this can be fixed.

We also do not know why, we hope this will not occur again.