# Response to referee #1 for GChron-2020-15

We would like to thank anonymous referee #1 for their thorough but positive review as well as their constructive comments that have helped to greatly improve this manuscript. We agree with most comments and have added or adjusted text and figures accordingly. For the cases we did not agree with the recommendations made, we give explanations below (responses in green and italic). Also, we would especially like to thank referee #1 for their spelling and grammar suggestions. This is very much appreciated as none of the authors is a native English speaker.

# **General comments**

This manuscript presents new data based on luminescence analyses of minerogenic sediments in a palaeovalley in Switzerland. As the title indicates, most focus is on the dating and quite little is spent on the implications of the actual ages and their geological context. The luminescence analyses are thorough, though: the authors have used quartz, feldspar and polymineral fractions of several different grain sizes and carried out different tests to evaluate the luminescence properties of the sampled sediment. Both dose and dose-rate related issues are discussed. The discussions are relevant and interesting for luminescence users not only in the Alps, but also in other parts of the world.

Apart for some specific comments, generally about clarifications, and some minor technical corrections as listed below, my main objection or concern about this manuscript is its structure. Though the headings follow the normal IMRAD standard, content-wise there is a mix between Methods, Results and Discussion. When reading Results, in particular, it is like following the project and measurements as they developed. Results are presented, comparisons and references to other studies are made to evaluate data and motivate the next methodological step, which is then described, etc. In a way, this is quite nice, and likely saves some flipping back and forth between pages to check Methods for what was done and how compared to the Results etc, but it also means that, for example, some details regarding methods are presented first in Results, making it hard to find information, and it is in places not that easy to distinguish what are the new results of the authors' in the text (though obvious from tables and figures).

It is indeed hard to present this type of data in a meaningful and readily comprehensible way. However, the raised issue about mixing methods, results and discussion is certainly justified. Accordingly, we have adjusted subheadings and re-structure the content where necessary to allow for a more appropriate fit between sub-headings and text. For ease of reading, methods, results and discussion are jointly presented in aspect specific subsections. This was previously done but is now implemented in a more stringent manner. Also, we have emphasised in the text when presented results were from a different study.

1. Does the paper address relevant scientific questions within the scope of GChron? Yes

2. Does the paper present novel concepts, ideas, tools, or data? Yes, new data from a combination of existing methods and ideas

3. Are substantial conclusions reached? Yes

4. Are the scientific methods and assumptions valid and clearly outlined? Yes, valid and largely clearly outlined *See below*.

5. Are the results sufficient to support the interpretations and conclusions? Yes

6. Is the description of experiments and calculations sufficiently complete and precise to allow their reproduction by fellow scientists (traceability of results)? Overall, yes. Some minor details could be clarified. *See below*.

7. Do the authors give proper credit to related work and clearly indicate their own new/original contribution? Proper credit, yes, own results partly and slightly lost in text.

8. Does the title clearly reflect the contents of the paper? Yes, but see specific comment about proglacial below. *See below*.

9. Does the abstract provide a concise and complete summary? Yes

10. Is the overall presentation well structured and clear? Yes and no, see above for general comment. *See below*.

11. Is the language fluent and precise? Yes, largely. Some spelling or grammar mistakes, but with one or two exceptions nothing that hampers understanding. *See below.* 

12. Are mathematical formulae, symbols, abbreviations, and units correctly defined and used? Yes, overall

13. Should any parts of the paper (text, formulae, figures, tables) be clarified, reduced, combined, or eliminated? Yes. Fig. 12 needs revision (too small and/or light text). Regarding text, see general comment above. *Fig. 12 has been revised – see below*.

14. Are the number and quality of references appropriate? Yes

15. Is the amount and quality of supplementary material appropriate? There is none.

# Specific comments

• L83-86: Please provide some more detail about the sediments including interpretation. Are the sediments that you date really proglacial like the title says? It is not mentioned here. The dated sediments are only described as "sandy gravel" (no glacier indicated), "lacustrine" (no glacier indicated) and "diamicts and colluvium" (no glacier indicated).

We agree that the manuscript title might have been misleading and that details about the sediments was to scarce. Indeed, the investigated deposits are of glacial/proglacial to periglacial origin. Therefore, text has been added and the title ('Luminescence properties and dating of glacial to periglacial sediments from northern Switzerland') has been changed accordingly.

• L125: Please explain the combined recycling and IR depletion step. Do you mean that this is one set of measurement (one column (or row) in the sequence) only or is it two? Combined to me sounds as if it is one, but then you would either have a proper recycling (using blue stimulation only as after the first regen doses) or an IR depletion test (using infrared + blue stimulation), not both. And if you have two measurements, it is not combined.

Only an IR depletion test was implemented. Text has been changed accordingly.

# • L146 (and thereabouts): What was the cutheat?

Only preheats were used as is stated in 2.2 'Sample preparation and measurement': 'All performance tests [and measurements] were conducted using preheating previous to the natural, regenerative and test doses for which the aliquots were heated with 5 °C s<sup>-1</sup> to the tested temperature and held for 10 s (Q, fQ) or 60 s (F, PM).' To clarify [...] has been added.

## • L157: Against what value was the normalisation done?

Normalised was against the same fixed dose of ca. 90 Gy. Text has been added accordingly.

• L184/214: What do you mean by "rarely inherit luminescent behaviour"? That the sediments are efficiently bleached (no inherited signal/dose)? That the grains do not reflect the properties of the source rocks? Please explain.

Meant was that only few of the grains actually give a luminescence signal after artificial irradiation. Text has been changed to clarify.

• L187: The reduction of the signal to background level after 15-20 s is not shown in the figure 7, which is referred to. The Q and fQ plots there only cover 6 s. Also, the background seems to me to be reached already after 2-4 s.

## This is correct and the text has been changed accordingly.

• You touch upon the issue of incomplete bleaching in a couple of places, but you do not really evaluate/discuss it for your samples, apart from a few lines about RIN 8 and 13 (L300-). Yet, also samples RIN1-4 have significantly skewed dose distributions, and diamictons (RIN1-3) are not obviously well-bleached sediments. With easy-to-bleach quartz ages older than or similar to harder-to-bleach feldspar ages, it may not be a problem, but that is also an interesting result that could be discussed, even if only briefly.

### We agree and have added a text to briefly present these findings.

# • Will the implications of the ages be presented and discussed in some other paper? If not, I think that part should be expanded a bit here.

This is a good point and yes there will be a summary paper discussing the sedimentological context and the implications of the derived ages. This manuscript will use ages from outcrops, two cores from the Lower Aare Valley and the core presented here. The PhD student within the project (Lukas Gegg) will present this work.

# **Technical corrections**

• L30: replace "waters which both" with "waters, both of which"

# Text has been changed.

• L80: remove comma after Rinikerfeld

Text has been changed.

• L85: "transition" should be "transitions"

Text has been changed.

• L87: remove "in" after "10 cm"

# Text has been changed.

• L89: exchange "embrace" for a verb that more clearly indicates over- and underlying, rather than incorporating

Text has been changed.

• L118: "an" should be "a"

Text has been changed.

• L184: Please add something after "1%". Is it 1% of the aliquots, of the grains...?

Text has been changed.

• L184: replace "BSL" with blue light

Text has been changed.

• L186: clarify that 200 grains are for Q only, not fQ

Text has been changed.

• L186: "rains" should be "grains"

Text has been changed.

• L208: "FQ" should be "fQ"

Text has been changed.

• L215/219: Please check figure order. Fig. 10 must be referred to before Fig. 11.

Order has been changed accordingly.

• L241: Add "Of" in front of "the measured"

Text has been changed.

• L241: Replace "saturation only allowing" with "saturation, allowing only"

Text has been changed.

• L253: remove "RIN13 with" before "216.8"

# Text has been changed.

• L258 and elsewhere: It is confusing that you use the same letter symbol, D, both for dose and dose rate. A subscript (here e and total) typically indicates different versions of the same entity, not different entities. When I first read "Dtotal" I interpreted it as 'total dose', which did not make sense, and it was only after looking at table 2 that I found out that you meant 'total dose rate'. Please exchange one of the symbols.

# Nomenclature has been changed and total dose rates are now presented with DR<sub>total</sub>.

• L266: Please rephrase the last sentence. I do not understand what you mean. What is the offset less pronounced than? The difference between the Q and fQ? The offset between F (or PM) and Q (or fQ)?

# Following suggestions of referee #2, the entire subsection has been changed and this sentence became redundant.

• L280 and elsewhere: Either use ' ' for both corrections or for none. le, either "Lamothe correction" and "Kars correction", or "'Lamothe' correction" and "Kars' correction".

The use of ' ' has been adopted and was implemented in a stringent manner.

• L296: remove "according"

Text has been changed.

• L297: replace "this" with "the age"

Text has been changed.

• L311: specify which g-value is referred to (F or PM)

Text has been changed.

• L317: Should it be "1.9 % per decade" or do you mean that the values are 0.5-1.9 % lower than some other value? Please clarify.

Meant was '% per decade' and the text has been changed.

• L328: Add "the" after "However, "

Text has been changed.

• L347: Replace "were" with "have been"

Text has been changed.

• Fig. 1: check spelling of diamicts and carbonaceous

Text has been changed.

• Fig. 9 caption: add also RIN 5 fQ and PM.

Text has been changed.

• Fig. 11 caption: check spelling of luminescence on second line

Text has been changed.

• Fig. 11: the greens rings are hardly discernible from the white rings, consider using another colour

*Lines of the green circles have been dashed to help distinguish between them and the white circles. Unfortunately, other tested colours did not improve distinguishability.* 

• Fig. 12: the text inside the plots is not legible, it is way too small. The light grey colour in the four lower plots is hard to see, particularly for the text.

The colour has been changed and the text for RIN13 has been adjusted to make it easier to read.

• Fig. 13. Would it be possible to indicate the stratigraphy (e.g. unit numbers and boundaries) in these plots? It would help a reader to remember the stratigraphic context and which samples belong where.

The top part of the log have been added to the figure.

# Response to referee #2 for GChron-2020-15

We would like to thank referee #2, Sebastian Kreutzer, for his thorough review as well as his constructive comments that have helped to greatly improve this manuscript. We agree with most comments and have substantially added or adjusted text and figures accordingly. For the cases we did not agree with the recommendations made, we give explanations below (responses in green and italic).

One major point we like to address here is the suggestion to shift the scope of the manuscript towards an environmental study and to transfer it to a different journal. Sebastian argues that we are foremost presenting a luminescence-dating study and that from his perspective substantially more tests and a larger dataset are needed to justify a publication in GChron.

We disagree with this recommendation for the following reasons:

- 1. The scope of this manuscript is geochronology and how to get it 'right'.
  - The study itself is part of a large project investigating the fill of several palaeovalleys within northern Switzerland to ultimately reconstruct the environmental history for the wider region. Implications drawn for the Rinikerfeld are important for and will guide the luminescence dating approach for ten other cores and multiple outcrop samples. For this, the confidence in our results is crucial and, therefore, luminescence aspects have to be examined carefully. As both quartz and feldspar from Switzerland inherit challenging luminescence behaviour, we face a multitude of obstacles/opportunities that cannot be adequately addressed in a manuscript focusing on the reconstruction of past environments. The amount of technical details will defocus any such article and be little to non-accessible for the none-specialist.
- 2. Following the aims and scope on the webpage, GChron is defined as '[..] unified outlet for highquality basic and applied research in geochronology, independent of technique used or timescale considered. Geochronology publishes research in all aspects of geoscience that aim to determine times or rates of geologic events and processes [..]'. Our manuscript presents applied research in geochronology.
- 3. Referee #2 argues that the 'performed tests and measurements are [..] something luminescence-dating studies present all the time'. Indeed the 'standard test suit' is conducted while electron trapping probability assessment, deconvolution of quartz signals, pulse annealing experiments to assure not to measure artificial signal introduced during laboratory irradiation and quality control of reader performance go beyond the extent of most papers that are simply presenting and using luminescence ages for environmental reconstructions.

# **Contribution summary**

The manuscript presents a dating study from a palaeovalley (Rinikerfeld) from northern Switzerland. The authors retrieved eight samples from a drilling campaign for luminescence dating. Prepared were either the quartz, feldspar or the polymineral fraction using three different grain-sizes. The preferred grain-size fraction was altered with the sedimentological environment. Along with the chronology, the study aims at providing better insight into the luminescence characteristic to assess the potential for further luminescence dating studies that region.

# Recommendation

I suggest that the manuscript changes a little bit the story and becomes transferred to another Copernicus journal, e.g., E&G (<u>https://egqsj.copernicus.org</u>).

We regard journals with a focus on the reconstruction of Quaternary environments such as E&G not suitable for the present manuscript.

# Justification

The manuscript presents a concise luminescence-dating study for different minerals, including tests, such as preheat/dose recovery tests or thermal transfer tests from a drilling site in the foreland of the Swiss Alps. All tests are reasonably explained, justified, and they help to support the chronological findings.

However, the manuscript claims to target, specifically, luminescence properties of "proglacial sediments from northern Switzerland", but it remains foremost (and nothing is wrong with it!) a luminescence-dating study. The manuscript conclusion by the authors may best reflect this assessment. The performed tests and measurements are not particular, but something luminescence-dating studies present all the time to increase the confidence in the results. The study on eight samples characterises, as a byproduct, to some extent the luminescence properties of those samples. Nevertheless, it does not investigate luminescence properties in general for the area or directly compares findings from a large dataset (e.g., as a meta-study).

Hence, the exciting part of the study is the chronology itself concerning the palaeovalley. This is the story the manuscript should exploit and detail further. Currently, only a few lines (including the conclusion) wrap the setting of the site and its geomorphological and geological background. Thus, it falls short and more context would also help to understand the chronological findings better.

At the end of the introduction the authors wrote that they "asses" luminescence properties of the samples from the site, but again, it does not evolve beyond standard tests.

Therefore, I suggest that the authors alter the story a little bit towards a geoscientific focus, add some details as requested below (e.g., dose rate) keep what they have and submit the manuscript to, e.g., E&G (<u>https://egqsj.copernicus.org</u>).

I left a couple of comments below. Mainly referring to some glitches here and there, except the missing results and discussion on the dose rate (the water content is discussed though) and the sketchy discussion on the fine grain quartz age underestimation, nothing critical.

The geoscientific focus will be the subject of a separate manuscript by Gegg et al. that will use this and other cores from the area together with geomorphological and outcrop data. This is done to access the long-term erosion history of the area in context of the siting for the Swiss nuclear waste disposal site. The present contribution represents the rigorous testing program that is used to highlight the potential and limitations of luminescence dating in the region. Adding the results presented in this GChron manuscript would overload the paper with focus of reconstruction of Quaternary environments.

# **General comments**

1. The manuscript reads clear, and the preparation of figures and tables is good. Besides, the authors use many abbreviations in the figures that remain unexplained on top of rather short figure captions. The latter is not necessarily bad, but the manuscript may want to address a broader audience. Currently, the apparent target audience is readers with a background in luminescence dating working in the Swiss Alps. However, other readers may want to have a look into the article as well. Hence, figure captions and abbreviations should elaborate a little bit more, and figures should be more self-explanatory.

We agree and figures and figure caption have been adjusted according to the below mentioned specific comments.

2. The manuscript oddly seems to focus a lot on the differences between coarse and fine grain quartz results, towards an interpretation of an age underestimation of the fine-grain quartz fraction compared to the coarse grain fraction. Indeed, such an age underestimation has been reported in the literature, and I do not doubt these findings for the particular sites.

However, in the presented manuscript, only for one out of eight samples, a comparison of both grain size fraction is presented. Both numerical results overlap within uncertainties. All other samples report results, either for the coarse grain (top of the composite profile) or for the fine-grain quartz fraction (lower part). The author's statement on a potential age "underestimation" seems to be trigger by an age inversion in the profile. This age inversion is also present for the fading corrected fine-grain polymineral fraction. Why does this suddenly lead to the conclusion that the fine-grain quartz ages are underestimated (in comparison to the quartz coarse grain ages)? I got the impression that the authors had this idea of a fine-grain quartz underestimation in mind and then tried to see this pattern in their data. It is one possibility that deserves to be discussed, weakly supported by the data though.

We like to clarify that the authors did not go biased into the discussion with 'intent' to find an issue with fine grained quartz. This issue has not even been reported by others for this region. However, we see that the grain size discussion has become redundant and was deleted.

3. Technical detail: The chosen format to apply units is odd, e.g., the authors wrote "471.2±47.1 and 525.8±53.2 ka" instead of "471.2±47.1 ka and 525.8±53.2 ka". There is a general pattern in the manuscript and it should be corrected throughout. If I understand the author guidelines of GChron<sup>1</sup> correctly, it should even read, e.g., "(525.8±53.2) ka" because the linked SI brochure refers to the Guide to the expression of uncertainty in measurement; I might be wrong. Besides, I suggest to round values to meaningful digits. If the age uncertainty is around 10<sup>°</sup>% the number after the digit does not tell much.

<sup>1</sup> https://www.geochronology.net/for\_authors/manuscript\_preparation.html

Regarding the three points of this comment:

1. The format of units has been changed according to the referee's suggestion.

2. We have checked the guides and brochures as linked on the GChron webpage but it remains unclear whether ages and uncertainties should be presented in parentheses or not. However,

we have checked previous publications in GChron (esp. 'Highlighted articles') that not seem to have adapted to this style.

3. We agree with the suggestion of using meaningful digits and have rounded values accordingly.

# **Detailed comments**

# Abstract

1. Line 17–18: The statement that the fine-grain quartz ages are underestimated compared to the coarse grain ages does not seem to be supported by the data for three reasons: (1) The authors do not systematically compare coarse grain and fine grain quartz ages, they do this for sample RIN13 only. (2) For this particular sample, coarse and fine grain quartz ages overlap within 1. (3) Table 1 marks the coarse grain age as "underestimated due to grain size-dependent De underestimation", not the fine-grain quartz fraction.

It does not mean that the fine-grain ages are per se not underestimated (e.g., compared the general pattern observed and compared to the profile figure with corrected feldspar ages). Still, the abstract should reflect the essential outcome of the study with regard to the data.

For (1) to (3) see General Comments 2. The abstract has been changed to reflect the essential outcomes.

2. Line 19–20: That the dating reveals a rapid deposition during the (at least) MIS 6 was first mentioned in the Conclusion. Doubtlessly, it had no particular relevance to the authors, given the overall scope, but the abstract leaves the reader with the impression that this point will be detailed in the manuscript.

The context between deposits and chronology has been moved to a subsection and was elaborated on which justifies a mentioning in the abstract.

# Main text

1. Line 49: "Feldspar" ! "feldspar"

Text has been changed.

2. Line 55: Please report g-values normalised to two days or report the tc value otherwise it will be impossible for readers to compare these values with other findings from the literature.

The g-values reported in line 55 are from the literature as indicated. None of the cited studies do present tc values that might be citable. For completion, we have added the information that only one previous study does present their data normalised to 2 days (Buechi et al., 2017).

3. Line 63: I am not sure whether Thiel et al. (2011) should be mentioned as well here (for the 290 C)? *We agree and the reference has been added.* 

4. Line 73: Remove "Scientific"

By stating that a scientific drilling was conducted, it is clear that samples come from drill cores that have been taken for the purpose of scientific investigation and not as a by-product of e.g. a hydraulic rotary drilling that would disturb the integrity of the sediment and may induce further issues that are likely to impact the luminescence signal of the samples.

5. Line 101: ca 10<sup>6</sup> should suffice.

Text has been changed.

6. Line 110: Add references for the calibration quartz

Manufacturer/provider and batch number of the used calibration quartz are presented. We find this to be sufficient information at this point.

7. Line 115: Better "when comparing", because this is what people do when they double check your protocol parameters.

This is a statement regarding the work of Schmidt et al. and not a general comment. Therefore we would like to keep the text as is.

8. Line 131: "gamma-ray"

Text has been changed.

9. Lines 131–132: There is something wrong with the citation chain:

For the fine grain quartz fraction Buechi et al. (2017, p. 57) wrote: "For fine-grained quartz an a-value of 0.04±0.02 has been incorporated to account for the variability of the values reported in literature (Rees-Jones, 1995; Mauz et al., 2006; Lai et al., 2008).".

For the polymineral fraction Buechi et al. (2017, p. 57) reported: "The effect of alpha irradiation was considered with an a-value of 0.05±0.01 for PM fractions (Preusser, 1999b; Preusser et al., 2001).".

Where Preusser (1999b) is the here cited Preusser (1999). It was Preusser et al. (2001) who reported a-values for the polymineral fraction as quoted in line 132.

Contrary, Preusser (1999) reported four IRSL a-values without uncertainty with a mean of 0.05 and a standard deviation of 0.00 (all values show 0.05; their Table 2). More important is that the applied protocol is not similar to what was applied by the authors here.

In Sec. 4.2.2, the authors detail various possibilities and discuss whether the selected a-value is justified. My impression is that the here applied a-values were used, because they had been always used for samples from that region. This might be justified, but it also shows that it should be remeasured at some point. In either case, the chosen values need the proper reference.

Firstly, the citation chain and references have been fixed.

Secondly, the a-value used for fQ has been changed to  $0.04 \pm 0.02$  following Buechi et al. (2017) thereby incorporating a larger error onto the ages. This will allow to account for at least some of the uncertainty introduced by not measuring out own a-values and keep the results comparable with other studies from the region. While it is certainly favourable to measure study or sample specific a-values, this is not always feasible (i.e. our lab does not have an alpha source).

*Thirdly, the IRSL a-values go all back to Preusser (1999, Kölner Forum für Geologie und Paläontologie, ISSN 1437-3246). Here, the values are reported with uncertainties. Preusser (1999, QSR) and Preusser* 

et al. (2001, QR) use these values without providing much further details (these articles focus on reconstructions) and without uncertainties (as tables would not have fitted page width otherwise). The referee is right that the methodology used is different from the once used here. The a-values were determined using the combination of an external alpha (Am-241) and a gamma source (Co-90). While the a-values represents a physical property of a sample material that is independent of how it was determined, one could of cause argue that the lower values are due to a technical issue such as calibration or measurements procedure. However, these values have been determined the same way as several hundreds of values reported in several articles by Manfred Frechen and his team in the 1990th (mainly on loess from Eurasia). For example, Frechen and Preusser (Frankfurter Geowiss. Arb. 20D) and Preusser and Frechen (1999, Terrest. Quartärgeol.) report 25 IRSL a-values with a mean of 0.08±0.01 (hence in agreement with Schmidt et al. 2018). Since these were determined using the same equipment, calibration, procedures and operator, it is likely that the values reported from Switzerland are indeed different (for unidentified reasons).

10. Line 130: U, Th, K concentration values were deduced from gamma-ray spectrometry but only summarised values are presented. What about radioactive disequilibria?

Your environment is undoubtedly very challenging regarding the dose rate, so maybe you can present a few more results regarding the nuclide concentrations? For example, as a plot normalised to Th-232 (cf. Guibert et al., 2009), this would give a good indication. If this appears to be too much, the authors can copy and paste the data from the VKTA into a supplementing document and add one sentence to the main text addressing the possibility of radioactive disequilibria.

A sentence has been added to the main text. As a matter of fact, we regard the environment as not particularly challenging and we regularly check for radioactive disequilibrium. As this is not an issue with our samples, we do not intent to start a discussion on this topic and hence also have not added supplementary information. Upon reasonable request we are happy to hand out data to the interested reader.

11. Line 133: Gaar et al. (2013) confirm Huntley and Baril (1997); which is very reassuring. However, (1) they report  $12.9 \pm 0.4\%$  and (2) they argue for the application of the 95% confidence interval for the potassium concentration (citing Huntley and Baril 1997), means ca  $12.5 \pm 1\%$ . Since the authors cited both references, they should make elaborate why they did not follow the suggestion by Gaar et al. (2013).

## The reference of Gaar et al. has been removed.

12. Lines 140–180 (Sec. 3.1 Performance test): It should read "tests" and please add further subsections so that the results for the quartz and the feldspar/ polymineral, can be more easily separated.

Text has been changed and the manuscript was substantially restructure allowing to easily find sections for the specific minerals.

13. Line 164: It is not really a different "preheat behaviour" but a completely different design of the heating element and the thermo couple and its feedback electronic. So perhaps: "different technical design"?

Text has been changed to 'reader-specific preheat conditions'.

14. Line 198: "Chinse" -> "Chinese"

Text has been changed.

15. Line 230: The obtained overdispersion value also depends on the initial b; if set. Was it different from zero?

## No, 0 was used.

16. Line 237–238: I am not sure whether CAM is the most suitable model. The authors should doublecheck the findings by Heydari & Guérin (2018). I also suggest adding one or two dose-response curves from the lower part of the profile.

Certainly, the choice of an appropriate age model is a strongly discussed topic within the luminescence community and consensus is unlikely to be found. We have chosen the CAM out of the large number of models available as most applicable for our datasets, thereby, applying the most commonly used model.

17. Lines 257–258 ("However, if ..."): The De is not a good indication because it is a function of the dose rate and should only be used if the dose rate is homogenous over the profile. Besides, the fading corrected feldspar age appears to be also slightly younger (within 2 ok) for the Kars model. My point: If the authors want to keep that argumentation, they should extend the description of the environmental setting and the dose rate. Ages should not be disconnected from the sedimentological environment. For example, why did not a "facies change" (maybe it is not) cause all these "problems"? *The three sentences have been deleted*.

18. Lines 261: The purple density curve in RIN13 looks somehow skewed.

# All measurements of RIN13 PM are within 1 $\sigma$ of each other.

19. Lines 269–270: Preusser et al. (2014) wrote that they followed Auclair et al. (2003). Perhaps the latter one is the better reference to cite, or at least in combination with the first. Besides, it appears that Preusser et al. (2014) did not normalise their values to tc as done by Auclair et al. (2003). In that case, the g-value will be slightly different than "expected". The authors may want to add a plot showing their fading measurements; then it should become clear. Additionally, Preusser et al. (2014) measured only three points on the time axis.

The citations have been changed. More details about the fading measurements i.e. storage times, protocol and analysis have been added to the text. With already 13 figures in total, we refrain from adding this information in an illustrated way. 'As the IRSL signal commonly is subject to a loss of signal over time, fading tests were conducted following Auclair et al. (2003) for a given dose of ca. 130 Gy. Three cycles with storage times of ca. 0 s, 1 h, 2.5 h, 5 h, 10 h were implemented per aliquot while the aliquot constantly remained on the sample arm. This reduces the possibility of sample material being lost during mechanical transfer of the aliquot between sample arm and storage carousel (Preusser et al., 2014).'

With regard Kadereit et al. (GChron discussion 2020)2 the obtained g-value might be somewhat arbitrary, and so would be the following fading correction.

Nevertheless, I did not make this a major point for two reasons: (1) The manuscript by Kadereit et al. will likely be rejected and not become published (though the discussion is online and outlines the general problem). (2) Fading measurements and corrections are a tedious business. The approach chosen by the authors might be ok; it might be not. Without further age information, in particular, in the lower part of the composite profile, it is impossible to say.

Firstly, the manuscript referred to has been withdrawn by the authors and can hence not be discussed here. However, we are aware that fading measurements and corrections heavily rely on assumptions and laboratory procedures.

20. Lines 273: Unfortunately, the function the authors applied to corrected the ages after Lamothe et al. (2003) for fading has a (recently discovered) bug (https://github.com/R-Lum/Luminescence/issues/96). The consequence of the bug is that the uncertainty of the fading corrected ages is lower than it should be because the error of the g-value goes into the calculation with a weighting that does not seem to be justified. Of course, this is nothing I hold against the authors. I just wanted to mention it here.

Thank you for mentioning this. It would be good to see uncertainties without the weighting of the g-value, however, this will not change the conclusions drawn from the 'Lamothe' corrected  $D_e$  values.

21. Line 275: Please mention the tc value along with your g-value, otherwise they are not comparable. Please add throughout the manuscript.

To allow for comparison, g-values derived from the presented dataset are reported in % per decade normalised to 2 days. The '2 day#-normalisation has previously only been mentioned in the caption of Table 1. Text has been added to clarify this.

22. Lines 295–298:I was wondering whether the D0 criterion has any substance after the value became corrected for fading after Kars et al. (2008)? With the correction, the De is deduced from a new, simulated, dose-response curve. The D0 of the simulated curve should be the new reference, not the faded dose-response curve. Did I overlook something?

We agree and have reported the simulated  $2*D_0$  values here. However, this has not been properly announced. We have changed the text so that measured and simulated  $2*D_0$  values are presented and distinguishable.

23. Lines 335–336: I do not agree that based on these findings, the logical conclusion is that coarse and fine grain quartz ages are different. The profile may have some hiatus going along with the age inversion. The reason for this age inversion is not necessarily grain-size related. Do the authors have granulometric data from the core?

We apologise but this argumentation is hard to follow. How would a hiatus explain the age inversion with depth? Also, what gain do you expect from quantitatively, in comparison to qualitatively, derived grain size data? Nevertheless, the discussion about grain-size dependent ages has been removed.

24. Lines 340–341: The last point appears like an appendix in this sections and it leads to nothing further. Is this maybe some kind of leftover from a discussion the authors wanted to engage but did not?

The manuscript has substantially been restructure and we have expanded this particular point.

25. Lines 343–365: This is a helpful discussion of different scenarios and justified. However, it should engage a more general discussion on dose-rate scenarios (which does not exist yet).

We don't see the need of a more general discussion at this point; neither evidence for radioactive disequilibrium was found nor do we expect issues from the macro-dosimetry as samples were taken far enough from obvious unit boundaries.

26. Lines 366–389 (4.2.2 Alpha efficiency values and age determination): This subsection renders a potentially fascinating discussion. The problem I have with this section, in particular the first part, is

that it does not read clearly but mixes different aspects. For example, after reading the section, my conclusion was that the chosen a-value of 0.05±0.01 is the less justified value. The reasoning is that somehow all goes back to Preusser (1999) and Preusser et al. (2001) Although for Preusser et al., 2001 I am not sure whether it does not resembles values from Preusser, 1999 (?).

Means, in the worst case, the selection bases on four values with rather low a values, e.g., for the polymineral and feldspar fraction. By contrast, the majority of the other articles would favour higher values. Besides, Schmidt et al. (2018) presented an extended dataset of a-values (IRSL and pIRIR290), though the focus was pIRIR290, which was not measured here.

Of course, it does not mean that the value is wrong, but the arguments presented by the authors indicate that (as even alluded in the manuscript) that they should remeasure the value.

We have added to higher a-value spectrum to Fig. 12 and have discussed its impact on the ages. Also, see comment 9.

27. Line 373–374: Please correct the reference or the a-value (see above)

## References have been corrected.

28. Line 391: Something is missing in the section title. Perhaps: "Quartz age grainsize dependency" or "Grain-size dependency of the quartz ages"

### We agree, however, the section was removed.

29. Lines 391–403 (Sec. 4.2.3): The section is very brief and, in my opinion, does not add to the understanding of the "age discrepancies" (if there is any, see comments above) and it does not discuss the quartz grain-size dependency as announced in the section title. Instead, it provides a brief, selective review of other findings, and it concludes that the lowermost two samples should be regarded as minimum ages. I would support the conclusion, but not the reasoning.

### The section has become redundant and was removed.

### 30. Line 400: Timar-Gabor et al. (2017) wrote:

On the other hand, the age discrepancy of SAR-OSL ages previously reported for Romanian and Serbian loess for ages beyond 40 ka (equivalent doses >100 Gy) was also found to be characteristic of Chinese loess. It is thus believed that this is potentially a global phenomenon, affecting previously-obtained chronologies worldwide, and further increasing concerns for the accuracy of silt-sized SAR-OSL ages in this high dose range. (Timar-Gabor et al., 2017, p. 470).

Timar-Gabor et al. (2017) expressed a guess or hypothesis as part of the conclusion. This conclusion, however, should not be become some statement on the "pattern around the globe'. At least the cited study does not provide the data to it.

Furthermore, Timar-Gabor et al. (2017) refer, first of all, to own observations from Romanian and Serbian loess comparing 4–11  $\mu$ m (fine grain) and 63–90  $\mu$ m (their coarse grain). This is not similar to what is presented in the manuscript for GChron.

### The section has become redundant and was removed.

31. Lines 411: The conclusion should reflect the results and discussion of the manuscript. The depositional history was not discussed in the manuscript and came here by surprise.

We agree and have changed the conclusion to only report what has been discussed previously.

# **Figures and tables**

## 1. Figure 1

• Do the authors have other ice extent data to show? Perhaps the LGM ice extent is a nice to have, but of limited relevance given the age results.

The LGM ice extent is presented to emphasis the fact that no glacial overprint has occurred during 'more recent' times. The only other extents that might be reasonable to be shown are the ones of the Möhlin and Beringen glaciations. The maximum extent of these glaciations are situated much further to the North within Germany and would thereby cover the entire extent of the area presented in Fig. 1. We do not believe that adding this extra information to Fig. 1 will contribute to the understanding of the here presented study. However, text has been added to subheading '2 Site setting and samples'.

• "A." and "B." is part of the figure, consequently those lettering should be part of the figure caption.

The figure caption has been changed accordingly.

- 2. Figure 2
  - An y-axis unit (core depth) is missing.

The unit (m) has been added.

• I would be good to have some photos showing the core log to better understand the setting. Also, the authors may want to indicate where one core ends and the next starts.

This goes beyond the scope of this figure and this study. The full core log and photos are available online. The reference is given in subheading 2 (i.e. Gegg et al., 2018), however, a link to the webpage has been added to the references (https://www.nagra.ch/de/cat/publikationen/arbeitsberichte-nabs/nabs-2018/downloadcenter.htm).

• What does the upper x-axis ("C, Si, Sa, Gr, Co, Bo" on top the core log) labels? Probably it is obvious, but it is not to me and maybe other readers are not familiar with it as well.

The upper x-axis presents the dominant grain size as presented in the log units. For explanation, text has been added to the caption.

• The age comparison should be based on 95% confidence intervals. However, I guess the graph will not scale very nicely given the two lower polymineral ages. This can be fixed by using a non-continuous scale.

*This would be one way of presenting the data, we have chosen not to. By introducing discontinuous x-axes, the figure will become messy and gets less easily understandable.* 

## 3. Figure 3

• The figure would benefit from more details in the figure caption. Readers not familiar with luminescence dating may struggle to understand the figures. For example: "M/G" probably means measured to given dose, "PH" means preheat etc.

Text has been added to the caption to explain 'M/G' while the x-axes labels have been adjusted to 'Preheat temperature' to avoid unnecessary abbreviations.

• The inset legend in all the figures in the right column is unnecessary because only one type of data is shown in all figures. It would suffice if the figure title (or subtitle) says "thermal transfer test". The way the figures are presented are consistent and will allow the reader to easily comprehend the different tests presented here.

• It is not clear what the data points are displaying. A single measurement with uncertainties? An average with the error bars showing the standard deviation (of the mean)?

Presented are CAM  $D_e$  values with 1  $\sigma$  uncertainties. Text has been added to the caption and the number of measurements are mentioned for clarification.

## 4. Figure 4

• The mineral fraction is missing in the figure caption

The mineral fraction 'F' has been added to the caption.

### 5. Figure 5

• Same as above, the mineral fraction is missing in the figure caption

The mineral fraction 'F' has been added to the caption.

### 6. Figure 6

Assuming this is referring to Fig. 7:

• Same problem as Fig. 3. The figure captions should explain used abbreviations (e.g., "DR")

*Explanation for used abbreviations has been added to the caption.* 

• The solid line the curve is not really showing the "given dose decay" but the "luminescence signal decay" or shine-down curve of the natural signal. It is a proxy for the "given dose decay", but is not a "dose decay".

# Text has been changed to 'luminescence signals of the natural or first given dose' in the caption and the text in the figure has been adjusted accordingly.

• "Natural decay" might lead to a wrong understanding by others who work with dating methods relying on the "radioactive decay" of isotopes. Perhaps: "natural shine-down curve" or something similar.

#### See above.

• De(t) plot was used by Bailey et al. (2003) to identify the partial resetting of the luminescence signal. They shifted and extended the signal integrals slightly at the end. Probably it was not done here, but the figure caption should detailed what was done so that the figure becomes immediately understandable.

*Text has been added to the caption to clarify that 0.4 s intervals (Q, fQ) and 1.5 s (F, PM) intervals were used.* 

• Y-axis labelling should be added to the figure in the right column.

We believe this is redundant and will make the figure messy as not only y-axis labels would have to be added to the primary y-axis of the right column but also to the secondary y-axis of the left column.

7. Figure 12

• Proper x-axis labelling is missing or figures should align more closely.

Labels have been added to all x-axes.

• Was a similar bandwidth used for all kernel density curves?

Bandwidth was determined for each *D*<sub>e</sub> distribution individually.

• RIN2: Table 1 reads 158.4±4.4 Gy instead of 158.4±4.3 Gy in the figure (minor detail, since I argue for meaningful rounding above).

Text has been adjusted in Fig. 12 and numbers are now presented as integers.

8. Figure 13

• 95% confidence intervals should be used for the age comparison.

Confidence intervals are one way of presenting an age comparison. We have decided to take another way and only show the central age values to keep the figure clear and understandable.

• There is somehow a typo in the figure caption: It reads "Accepted ages are presented with 1 uncertainty as point symbol.", however, there is no "point symbol" in the figures.

Text has been changed to clarify that this referring to signature styles.

9. Table 2

• What meant is the internal K-concentration, it should be written.

Text has been changed accordingly.

• "De" should read "D<sub>e</sub>" (subscript "e")

Text has been changed.

# Personal note to the authors

Dear Müller et al.,

I can imagine that you do not agree with my suggestion to transfer the manuscript. To avoid the impression that I am "against" your manuscript, I may add that I sincerely believe that every luminescence-dating study deserves to be published; given that it is free of significant mistakes. Luminescence dating is way too costly to ignore the data or let them disappear in a drawer. Your study indeed, should be published. However, I think for GChron it would need way more tests (e.g., TR-OSL, TL with trap parameters etc.) and a larger dataset. This is nothing I can ask of you. To the contrary, you probably have the perfect for, e.g., E & G with a ready to go chronological part if you extent the geomorphological/geological part.

Nonetheless, to ease the minds and to avoid a heated discussion: If the editor believes that the manuscript is most suitable for GChron, I will certainly not further argue against a publication in GChron. Moreover, I consider my suggestions as a piece to an open discussion, which is not carved in stone.

Thank you for clarifying your intentions and your thorough review of the manuscript. For a detailed response to the raised concerns regarding the manuscripts suitability for GChron and the suggestion to transfer the manuscript to a different journal see above.

# **Conflict of interest**

I have no conflict of interest to declare. I am not a beneficiary of the suggested references to be cited. Naturally, the authors are free to reject my reference suggestions.

# **Response to Associate Editor Decision for GChron-2020-15**

We would like to thank the Associate Editor, Julie Durcan, for her decision to publish our manuscript in Geochronology after minor revisions and for the constructive comments she made. We agree with most comments and have added or adjusted text accordingly. Below we respond to her comments in detail.

# **Comments to the Author**

In this paper by Mueller and colleagues, the authors address the dating of glacial and periglacial sediments in the Alps. As is the case in many glacial contexts, the dating of peri-/glacial sediments has been reported as problematic in terms of luminescence dating. This paper provides the detailed testing of coarse grain quartz and feldspar and fine grain quartz and polymineral luminescence signals in an attempt to resolve phases of glacial advance in the northern foreland of the Swiss Alps. Whilst the paper identifies valley fill in the area at least back to MIS6, discrepancies between the various dating signals are identified and as yet remain unresolved.

I am satisfied that this paper goes beyond a 'routine' dating study (and I echo Sebastian Kreutzer's comments that there is nothing at all wrong with routine dating studies), and therefore I believe it suitable for publication in Geochronology. This paper demonstrates that luminescence dating is more than an 'off-the-shelf' dating technique, and that efforts to interrogate the signals being dated are key in developing chronologies in geomorphic contexts which traditionally have been a challenge for luminescence daters.

I thank both reviewers for their detailed reviewers, and believe that the authors have provided a satisfactory response to provide an improved manuscript. There are a couple of comments in Sebastian Kreutzer's review which I agree are still relevant, although the authors haven't modified significantly their manuscript in response to these. These are i) the difficulties in undertaking fading measurements and analysing the subsequent data – this is where we are with luminescence dating currently, but it is important to acknowledge this; ii) the use of the central age model – and I highly recommend the paper of Heydari and Guerin (2018) to the authors which explores the use of the average dose model (Guerin et al., 2017). Whilst CAM is one of the most (if not the most) commonly used age models in luminescence dating, it shouldn't be immune from supersedure; iii) acknowledgement of the complexities of dose rate calculation, in particular issues arising from disequilibrium if present and undetected. I highlight these in order that the authors may consider these issues in future research.

Thank you for highlighting these points, we certainly will consider them in our future research. Also, we would like to make some finally comments to these points:

i) Fading measurements come with a suite of uncertainties and issues themselves. To acknowledge this, we have added the following sentence under the fading implication subsection (6.2): [..] however, it is recognizing that fading measurements and corrections come with a suite of uncertainties.

ii) We agree, the CAM is not the 'ultimate' age model that is immune to supersedure. In case of the here presented data, application of e.g. the Average Dose Model (ADM) will not make a significant difference. We have plotted (uncorrected) CAM D<sub>e</sub> values against those of the ADM to emphasise this argument (see below). Except for one sample, all results are well within 1 σ. The exception is sample RIN13 Q that gives D<sub>e</sub> values just within 1 σ. For this samples and OD of 56 % was reported and only a minimum age estimate was obtained.



iii) Dose rate determination is indeed a complex endeavour and we are aware that the detection and monitoring of disequilibria are an essential part for the establishment of accurate chronologies. Therefore, all our samples are measured routinely using high-resolution gamma spectrometry and results are checked for disequilibria. We have added a remark regarding this to the manuscript.

Further to the reviewer's comments, I have a few minor comments:

Abstract: Please expand the abstract to summarise the suite of tests you undertook on quartz and feldspar in order to provide a chronology. At present, the abstract reads like a 'regular' dating paper where you used a number of different signals, and your paper goes beyond this.

We agree and have emphasised this point accordingly.

L12: in order to overcome what? E.g. to provide reliable chronologies

We agree and the text has been changed accordingly.

L98: 'deposited' is probably more appropriate for 'emplaced' – assuming that's what you mean.

# We agree and the text has been changed.

L204: Please can you clarify whether your EMCCD measurements made after lab irradiation. As a comment only, I'm not surprised your detection of signals is so low, given the low sensitivity of the EMCCD system (for Riso systems at least- see Thomsen et al, 2015 DOI: 10.1016/j.radmeas.2015.02.015)

EMCCD measurements were conducted on the natural signals which has been clarified in the text. In addition, the EMCCD system was specified under subsection 3. (Sample preparation, equipment and dose rate determination). We also want to highlight that the observed low quartz seinsitivty is typical for the region (e.g. Trauerstein et al., 2017) and not necessarily solely explained by the use an EMCCD system.

L258: Please can you provide a short sentence to summarise the key differences between the two fading procedures.

The following sentence has been added: The first approach is based on the extrapolation of luminescence signal loss to a decadal percentage (g-value) that is used to correct the natural dose and the dose response points while the latter approach utilises the sample specific density of recombination centres ( $\rho'$ -value) to account to correct the dose response curves.

L444: For me, that not all minerals/fractions/samples are suitable for dating demonstrates the strength of your approach of systematically and testing in detail a range of signals. This is worth commenting on in your conclusion

We agree and have emphasised this point in the conclusion by adjusting the text: Not all minerals, fractions or samples are suitable to be dated using the here presented luminescence techniques, nonetheless, this systematic investigation of different luminescence signals allows for a decisive chronology to be established.

Fig 10 caption. Can you add whether these are signals from natural or lab-irradiated signals (if so, what dose please)? Also, the size of the ROI?

Text has been added to the caption to clarify that EMCCD measurements were conducted on the natural signals and that holes (ROI) are 300  $\mu$ m in diameter.

# Luminescence properties and dating of **pro**glacial to periglacial sediments from northern Switzerland

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- 10 Abstract. Luminescence dating has become a pillar of the understanding of Pleistocene glacial advances in the northern foreland of the Swiss Alps. However, both quartz and feldspar from the region are equally challenging as dosimeters with anomalous fading and, partial bleaching and unstable components being some of the obstacles to overcome for thete establishment of decisive-reliable chronologies. In this study, luminescence properties of coarse- and fine-grained quartz, feldspar and polymineral fractions of eight samples from a palaeovalley, Rinikerfeld, in northern Switzerland are
- 15 systematically assessed. Standard performance tests are conducted on all four fractions. Deconvolution of luminescence signals of the quartz fractions is implemented and shows the dominance of stable fast components. Reader specific low preheat temperatures are investigate on the infrared stimulated luminescence (IRSL) signal of feldspar. Thermal stability of this signal is found for low preheats and thermal quenching could be excluded for higher preheats. -and found appropriate for dating. WhileHowever, anomalous fading is observed of the IRSL signal of in the feldspar and polymineral fractioninfraIRSL red
- 20 signals and two correction approaches are applied. -is observed, fFor one approach, fading corrected coarse-grained feldspar ages are consistent with those derived from quartz. In general, coarse-grained quartz and feldspar as well as the fine-grained polymineral fractions of one sample are in chrono-stratigraphic agreement and present negligible evidence for partial bleaching. However, ages derived from fine-grained quartz are found to underestimate those of the coarse-grained quartz fractions. Hence, the impact of alpha efficiency and water content onto the dose rate and therefore the ages are assessed. A
- 25 finite explanation for the observed discrepancies remains lacking but this systematic investigation of different luminescence signals allows for the establishment of Impact of total dose rate on finite ages was assessed but age underestimation is likely due to grain size dependent luminescence properties. The top six samples indicate sedimentation of at least 16.6 m during Marine Isotope Stage 6 with a rapid transition from a lacustrine environment to a landscape dominated by colluvial deposits. For the two lowest samples, no finite ages are derived, a chronology for the palaeovalley fill dating back to at least Marine 30 Isotope Stage 6 (MIS 6).

1

#### **1** Introduction

Pleistocene glaciations in the northern foreland of the Swiss Alps have been studied since the early 19th century (summarised 35 in Preusser et al., 2011). Whereas it appears that a minimum of eight glacial advances have shaped the lowlands, consensus on the exact number and timing is still lacking. In the last three decades, numerical dating turned into has become a crucial component in the reconstruction of the environmental past of northern Switzerland (e.g. Preusser, 1999a; Graf et al., 2007; Kock et al., 2009; Dehnert et al., 2012). In particular, luminescence dating has become a pillar of chrono-stratigraphy and understanding of glacial advances (Preusser et al., 2011). However, luminescence dating of glacial and pro-glacial deposits 40 can be complex (e.g. Duller, 1994, 2006; Spencer and Owen, 2004). For one, in such environments sediment sources and sinks are often within short distance of each other and transport may occur in turbid waters, both of which both which reduces the chance for grains to experience sufficient sunlight exposure to reset or bleach any pre-existing luminescence signals. This effect will consequently lead to age overestimations and requires consideration in such deposits. The measurement of single grains or small aliquots has been advised to allow for monitoring of luminescence signal commonly presented as equivalent 45 dose (D<sub>e</sub>) distributions and the isolation of a proportion of the D<sub>e</sub> distributions that is considered well bleached (e.g. Ollev etal., 2004; Duller, 2006; Trauerstein et al., 2017). The luminescence signal of quartz is more readily bleachable than thatis of feldspar (Godfrey-Smith et al., 1988; Murray et al., 2012), and is hence is favoured as dosimeter (e.g. Lowick et al., 2015).

But, to asse<u>s</u>s partial bleaching of samples from northern Switzerland, Trauerstein *et al.* (2017) have recommended to<u>he</u> compare<u>ison of</u> luminescence signals obtained from both dosimeters. However, quartz and feldspar from the region have found to be equally challenging for dating.

For quartz from the wider region, unstable components were reported for some (e.g. Klasen *et al.*, 2016) but not all samples (e.g. Gaar *et al.*, 2013). Moreover, laboratory dose response curves for quartz were often best fitted with a double saturating exponential function that accounts for high dose responses beyond single exponential behaviour (Lowick *et al.*, 2010; Dehnert *et al.*, 2012). This phenomenon is also known for samples from other regions where luminescence ages are either in agreement

55 (e.g. Murray et al., 2008; Pawley et al., 2010) or disagreement (e.g. Lai, 2010; Timar et al., 2010; Timar-Gabor et al. 2011) with independent age control. However, the physical reason for this behaviour has yet to be identified (Wintle, 2008). In northern Switzerland, quartz luminescence ages of up to ca. 250 ka have been found reliable (Anselmetti et al., 2010; Dehnert et al., 2012; Lowick et al., 2015; Buechi et al., 2017), but not beyond. Hence, this approach is not suitable to establish an independent chronology for the recently modified Mid\_Pleistocene glaciation history (Graf, 2009; Preusser et al., 2011).

60 significantly limiting the use of quartz as a dosimeter for the region. While [Feldspar requires longer for bleachingmore time to reset the signal prior to deposition, it is known to saturate at higher doses and therefore often allows to date much older deposits than quartz (cf. Duller, 1997). Yet, the infrared stimulated luminescence signal of feldspar measured at 50 °C (IRSL) often suffers from anomalous fading contributing to age underestimation (Wintle, 1973; Spooner, 1994). Different approaches to determine and to account for the loss of signal over

65 time have been proposed (Huntley and Lamothe, 2001; Auclaire et al., 2003; Lamothe et al., 2003; Huntley, 2006; Kars et al.,

2008). These approaches rely on observing signal loss over hours to days within laboratory experiments and deducing from this the signal loss that has occurred over geological time scales. In such storage tests, fading has been observed for most samples from northern Switzerland with (g-values between 1 % and 3 % per decade (Dehnert et al., 2012; Lowick et al., 2012, 2015; Gaar and Preusser, 2012) and 2.7 ± 0.3 % per decade (normalised to 2 days; Buechi et al., 2017). + However, uncorrected

- 70 IRSL D<sub>e</sub> values were either (1) beyond the linear part of the dose response curve and therefore unsuited for most fading correction approaches, (2) corrected IRSL D<sub>e</sub> values were close to saturation and consequently rejected for age determination or (3) uncorrected feldspar ages were in better agreement with quartz ages and therefore favoured (Dehnert et al., 2012; Lowick et al., 2012, 2015; Gaar and Preusser, 2012; Buechi et al., 2017). Only Gaar et al. (2013) found correcting for fading appropriate and necessary for samples <100 ka. For dating of older samples, Lowick et al. (2012) tested alternative
- measurement protocols that target signals that are more stable than the IRSL signal, the post infrared infrared stimulated 75 luminescence signals (pIRIR). The pIRIR signals are measured at higher temperatures after an initial readout of IRSL signals at 50 °C (Thomsen et al., 2008; Buylaert et al., 2011, Thiel et al., 2011). While fading is expected to decrease or become negligible for the more stable pIRIR signals, these take longer to bleach by sunlight during transport. For the used-selected pIRIR<sub>225</sub> and pIRIR<sub>290</sub> signals, fading was still observed and age overestimations led to the conclusion that these approaches
- 80 are not beneficial for the investigated waterlain-sediments from northern Switzerland (Lowick et al., 2012). Here, luminescence properties of samples from the Rinikerfeld in the northern foreland of the Swiss Alps are assessed. Scientific drilling was conducted at a stratigraphic key site within the former glacier forefield as part of a larger campaign. The campaign is aimed to acquire further insights into the long-term glacial and fluvial landscape evolution of nNorthern Switzerland. Modelling of future erosion scenarios will be based on the gained knowledge and thereby assist in the assessment
- of the safest and most suitable location for a prospective Swiss nuclear waste depository. For this, the establishment of a 85 chrono-stratigraphy is essential and, hence, dating is a crucial component. Therefore, feldspar, polymineral as well as fine- and coarse-grained quartz are investigated to exploit the dating potential of the samples from this site. Performance tests, signal intensity and composition of quartz as well as signal and fading properties of feldspar are discussed and two approaches to correct for fading are applied. Eventually ages are derived and discussed with regard to problems found for other studies of the region. For ease of reading, methods, results and discussion are jointly presented in aspect specific subsections.

#### **2** Material and method

#### 2.1 Site setting and samples

The study site (Fig. 1) is located within the Rinikerfeld, at the eastern tail of the Jura Mountains, about 30 km NW of Zurich. Rinikerfeld, is part of an extensive palaeovalley structure and situated in an elevated position, ca. 50 m above the nearby 95 present-day Lower Aare Valley. At the site, the palaeovalley is carved into Mesozoic bedrock (Bitterli Dreher et al., 2007) Formatiert: Schriftart: Kursiv

and presumed to be of Mid-Pleistocene age (Bitterli-Dreher et al., 2007; Graf, 2009). It was spared from glacial overprint during the Last Glacial Maximum (Bini et al., 2009), however, the outermost ice margins of preceding glaciations-were were found to the North of the study area, (cf. Preusser et al., 2011), suggesting a glacial or, proglacial to periglacial sedimentary nature of the palaeovalley fill-(cf. Preusser et al., 2011).

- and aAbout 40 m of Quaternary sediments, are overlying the bedrock (carbonaceous marl) and were recovered in cores a composite core (Fig. 2) within a scientific drilling campaign (Gegg et al., 2018). At its base, the composite core consists of about 4 m of glacial diamicton overlain by ca. 2 m of glacial or glacier-proximal sandy gravel that contains frequent angular, faceted, and, in the lowest part, striated clasts. This unit gradually transitions into ca. 24 m of lacustrine clay and silt.
- 105 The interbedded gravelly sand and silt at the bottom of the lacustrine unit suggest an initial dominance of deposition by traction currents, possibly in a glaciolacustrine or deltaic setting. Relatively homogeneous laminated sediments in the rest of the lacustrine unit indicate suspension settling and low-energy currents. Towards the top, the lacustrine unit progressively coarsens and is capped by ca. 4.6 m of interbedded gravelly/sandy diamictons, rich in locally-derived angular clasts. This heterogeneous unit is likely to represent a mix of fluvial and colluvial deposits possibly deposited in a periglacial setting (Raab et al., 2007).
- 110 The upper lacustrine unit progressively coarsens and is capped by ca. 7.5 m of diamicts and colluvium (Fig. 2). For luminescence dating, samples were obtained from ca. 10 cm in diameter cores recovered in plastic liners (Table 1); three samples were taken from the gravelly to sandy diamictondiamicts unit (RIN1, 2, 3), one sample of lacustrine sands (RIN4) and one of gravelly sand and silt (RIN13). The two latter embrace-bracket the fine-grained, lacustrine unit whereof another three samples were taken (RIN5, 6, 8). Sample material was collected in ca. 10 cm segments whereby the outer rind and the
- 115 surface, exposed to daylight during core cutting and splitting, were allocated for dose rate determination and the inner core was used for De determination.

#### 32.2 Sample preparation, equipment- and dose rate determinationmeasurement

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To determine De values, samples were wet-sieved and treated with HCl and H<sub>2</sub>O<sub>2</sub> to remove carbonates and organic matter, respectively. Of five samples (RIN1 to RIN4, RIN13), coarse grained (150-200 and 200-to-250 µm) potassium-rich feldspar (F) and quartz (Q) were separated using sodium polytungstate at densities of 2.58 g cm<sup>-3</sup> and 2.7 g cm<sup>-3</sup>. The outer rind of the quartz grains was etched in 40 % HF for 1 h followed by a 32 % HCl treatment for another hour to eliminate any fluoride precipitates. For the remaining three samples (RIN5, 6, 8) and RIN13, the fine-grained fraction (4-11 µm) was separated using settling under Stokes' Law. One half of the sample remained pristine as polymineral fraction (PM) while the other half was 125 treated with hexafluorosilicic acid for seven days in order to obtain purified quartz (fQ). The fine-grained fractions were suspended in acetone and settled onto cups of 9.8 mm in diameter (>1.1\*10<sup>6</sup> grains). For the coarse-grained fractions, sample material was mounted onto cups using silicon oil stamps with diameters of 1 mm for feldspar and 4 mm for quartz (ca. 13 and 200 grains, respectively).

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D<sub>e</sub> values were obtained with a Freiberg Instruments *Lexsyg Research* using LEDs with peak emission at 458 nm (BSL) for 130 quartz and at 850 nm (IRSL) for feldspar and polymineral. BSL and IRSL signals were detected by an ET9235QB photomultiplier tube filtered through a 2.5 mm Hoya U-340 glass combined with a 5 mm <u>Delta-BP 365/50 DeltaEX-</u> interference filter or a 3 mm BG 39 Schott glass and a 3.5 mm HC 414/46-1 AHF Brightline interference filter (referred to as <u>"4410 nm-filter combination"</u>), respectively. Additionally, a cardboard barrier was mounted into the filter wheel for IRSL measurements (for details see 3.34.2). Laboratory irradiation was given by a <sup>90</sup>Sr/<sup>90</sup>Y beta source mounted into the reader. The 135 beta source was calibrated using *Risø* calibration quartz batches 108 (4-11 µm) and 118 (180-250 µm) to -ca. 0.10 Gy s<sup>-1</sup> and

135 beta source was calibrated using *Risø* calibration quartz batches 108 (4-11 μm) and 118 (180-250 μm) to ~ca. 0.10 Gy s<sup>-1</sup> and 0.11 Gy s<sup>-1</sup>, respectively. <u>Measurements with the electron multi-plying charge-coupled device (EMCCD) were conducted using an *Andor jXon Ultra 897* camera mounted to the *Lexsyg Research*.</u>

Notable is the preheat behaviour of the used *Lexsyg Research* reader (Lexsyg ID 09-0020); at a heating rate of 5 °C s<sup>-1</sup>, the socalled 110 °C TL peak emerges at much lower temperatures than expected. For example, for *Risø* calibration quartz (batch

140 118) TL counts are peaking at 87 ± 3 °C during preheat. This is in agreement to observations made by Schmidt *et al.* (2018b) who compared the preheat behaviour of various OSL readers and discovered the appearance of the 110 °C TL peak within a range of 60 °C. This has to be considered when assessing appropriate measurement protocols (see section 3.14).
Performance tests were conducted on both the coarse and fine grained fractions of two representative samples (RIN2O).

RIN2F, RIN5FQ, RIN5PM). Preheat plateau tests were carried out on the natural dose while for thermal transfer and dose recovery tests, sample material was exposed to an daylight lamp for 16 h (Q, fQ) to 30 h (F, PM). All performance tests were

145 recovery tests, sample material was exposed to an daylight lamp for 16 h (Q, fQ) to 30 h (F, PM). All performance tests were conducted using preheating previous to the natural, regenerative and test doses for which the aliquots were heated with 5 °C s<sup>-1</sup> to the tested temperature and held for 10 s (Q, fQ) or 60 s (F, PM). Both BSL and IRSL measurements were conducted for D<sub>a</sub> determination following the SAR protocol (Murray and Wintle,

2000) with test doses of ~46 Gy and ~23 Gy administered, respectively. The initial signal was derived from the first 0.4 s of

150 the BSL and the first 15 s of the IRSL signals. A late background subtraction using the last 40 s (BSL) or 50 s (IRSL) was applied. D<sub>e</sub> values were calculated using the numOSL package for R (Peng *et al.*, 2018).

For Q and fQ, a combined recycling and IR depletion ratio (>20 %) step was implemented at the end of each sequence to check the adequacy of the sensitivity correction and for feldspar contamination. A maximum of four aliquots per sample failed this rejection criterion. None of the measured Q, fQ, F and PM aliquots presented dim (<3\*BG level) or imprecise (>20 %) test

155 dose signals and were hence accepted. For RIN3 F, one aliquot was rejected as it failed the recycling ratio test (>20 %) and for RIN13 Q, three aliquots were rejected as their D<sub>a</sub> values were in saturation.

For dose rate determination (Table 2), sample material was dried and radio-nuclide content was determined via high-resolution gamma-ray spectrometry at VKTA (Dresden, Germany). In absence of evidence for radioactive disequilibrium (cf. Degering and Degering, 2020), only contents used for dose rate determination are presented in Table 2. A mean alpha efficiency factor

160 of  $0.054 \pm 0.024$  (a-value) was assumed for all fQ following (Buechi *et al.*; (2017). as well as An a-value of  $0.05 \pm 0.01$  was applied for the F and PM fractions (Preusser, 1999a, 1999b; Preusser *et al.*, 19992001). For the two latter an internal potassium content of 12.5  $\frac{6}{5} \pm 0.5$  % was used (Huntley and Baril, 1997; Gaar *et al.*, 2013). The cosmic contribution was determined for

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present day depths following Prescott and Hutton (1994). Water content relative to the dry weight of the sample and capacity of water absorption (DIN 18132:2012-04, 2016) were determined in the laboratory. Representative long-term water content estimates of 20 to  $25 \frac{\%}{2} \pm 5 \%$  were used for total dose rate (DR<sub>total</sub>) determination. Age calculations were conducted with ADELE 2017 software (www.add-ideas.com; Degering and Degering, 2020).

#### 3 Results

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#### 3.14 Performance tests

- 170 Performance tests were conducted on both the coarse- and fine-grained fractions of two representative samples (RIN2Q, RIN2F, RIN5fQ, RIN5PM). Preheat plateau tests were carried out on the natural dose while for thermal transfer and dose recovery tests, sample material was exposed to a daylight lamp for 16 h (Q, fQ) to 30 h (F, PM). All performance tests and measurements were conducted using preheating previous to the natural, regenerative and test doses for which the aliquots were heated with 5 °C s<sup>-1</sup> to the tested temperature and held for 10 s (Q, fQ) or 60 s (F, PM).
- 175 Both BSL and IRSL measurements were conducted following the SAR protocol (Murray and Wintle, 2000) with test doses of ca. 46 Gy and ca. 23 Gy administered, respectively. The initial signal was derived from the first 0.4 s of the BSL and the first 15 s of the IRSL signals. A late background subtraction using the last 40 s (BSL) or 50 s (IRSL) was applied. D<sub>e</sub> values were calculated using the numOSL package for R (Peng *et al.*, 2018).

For Q and fQ, an IR depletion ratio step (>20 %) was implemented at the end of each sequence to check the adequacy of the

180 sensitivity correction and for feldspar contamination. A maximum of four aliquots per sample failed this rejection criterion. None of the measured Q, fQ, F and PM aliquots presented dim (<3\*background level) or imprecise (>20 %) test dose signals and were hence accepted. For RIN3 F, one aliquot was rejected as it failed the recycling ratio test (>20 %) and for RIN13 Q, three aliquots were rejected as their D<sub>e</sub> values were in saturation.

#### 4.1 Quartz OSL

- For preheat plateau tests on the natural signal of Q (RIN2), statistically consistent (1 σ) D<sub>e</sub> values were obtained on the larger grain size fraction for preheats between 200 and 240 °C (Fig. 3). D<sub>e</sub> values of fQ (RIN5) for this preheat temperature range are consistent at 2 σ (Fig. 3). A given dose of ~ca. 130 Gy was fully recovered from both tested samples (RIN2 Q and RIN5 fQ; Fig. 3) at 240 °C with measured-to-given-dose ratios (M/G-ratios) of 1.01 ± 0.03 and 1.05 ± 0.04, respectively. Maximal thermal transfer was detected with <4 Gy which equals to less than 3.5 % of the natural D<sub>e</sub> and is therefore considered negligible. Hence, a preheat temperature of 240 °C was chosen for both grain size fractions of quartz (Q, fQ) for all investigated
  - samples.

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#### 4.2 Feldspar and polymineral IRSL

For F, poor recovery was obtained for preheats above 190 °C. Considering the poor performance at high temperatures and the early emergence of the 110 °C TL peak on the used *Lexsyg Research* reader (see section 2.23), lower temperatures were tested and a full recovery of the given dose (~ca. 130 Gy) was possible for F and PM with a preheat of 170 °C. No plateau but increasing trends of De values from the natural signal were observed between 150 and 210 °C for F and between 170 and 210 °C for PM. Natural De values decrease rapidly for preheats above 210 °C while thermal transfer behaves inversely. However, thermal transfer is within <2 Gy (ca. ~1.5 % of the natural De), and is therefore negligible. To investigate whether thermal quenching induces an underestimation of De values at high preheat temperatures, preheat measurements were conducted on aliquots of RIN2 F following Wallinga *et al.* (2000). Therefore, the natural signal was bleached with IR LEDs, a fixed dose of ca. 90 Gy was administered and each aliquot was preheated to 50 °C for 10 s followed by IRSL at 50 °C for

- 0.1 s. This was repeated for preheat temperatures between 50 and 300 °C in 25 °C steps. After the highest preheat <u>the aliquots</u> are expected to be sensitised and can be used for normalisation<sub>s</sub>, therefore, the entire measurement sequence was repeated using the same fixed dose of ca. 90 Gyto allow for normalisation (Fig. 4). An increase in normalised IRSL sensitivity would be expected if the electron trapping probability changes with temperature (Wallinga *et al.*, 2000). Yet, a decrease was observed
- 205 be expected if the electron trapping probability changes with temperature (<u>Wallinga et al.</u>, 2000). Yet, a decrease was observed for temperatures above 200 °C suggesting that it is not a change in trapping probability causing the lower natural  $D_e$  values and dose recovery ratios. Rather the high preheat temperature may cause a removal of signal commonly used for  $D_e$ determination. Therefore, a preheat temperature of below 200 °C is most advisable. Best performance of the SAR protocol was obtained at a preheat of 170 °C (Fig. 3) which suggests a much lower preheat than conventionally used. Buylaert *et al.*
- 210 (2011) advice against the use of preheats below 200 °C due to thermally unstable signals induced during laboratory irradiation. However, their study was conducted on a *Risø* reader with different, <u>reader-specific</u> preheat <u>conditionstemperature behaviour</u>. It is stressed that temperature parameters of *Risø* and *Lexsyg* readers are not readily comparable. Nevertheless, to investigate a potential contribution from artificially filled unstable traps, short shine pulse annealing tests were conducted on aliquots of RIN2 F with naturally and laboratory irradiated material following Wallinga *et al.* (2000). Each aliquot was preheated for 10 s
- 215 respectively and then stimulated with IR LEDs at 50 °C for 0.1 s. Preheats were conducted consecutive in 10 °C steps from 130 to 400 °C. The signal was normalised with a test dose measurement of the same aliquot and renormalized to the first measurement (Fig. 5). Signal distribution of both the natural and laboratory induced irradiation are indistinguishable and it is unlikely that a thermally unstable component from artificially filled traps contributes to the IRSL signal even at low preheat temperatures. Thermal stability of IRSL signals from the here investigated samples is given and thermal quenching was not
- 220 detected, therefore, preheats of 170 °C are considered appropriate. In absence of preheat plateaus of the natural signals, the dose recovery test results are indicative (RIN2 F 0.97 ± 0.03; RIN5 PM 0.98 ± 0.03) and hence, preheats of 170 °C were used all for measurements.

Yet, the differences in temperature parameters make it necessary to test whether the configuration of the *Lexsyg Research* reader <u>a</u>effects the temperature during stimulation as for preheating. Therefore, De values of the natural signal were obtained

225 at stimulation temperatures of 30, 50 and 70 °C for one representative sample (RIN2 F) (Fig. 6). While the scatter in the natural  $D_e$  values decreases with stimulation temperature, mean  $D_e$  values of all stimulation temperatures are statistically consistent with each other, implying that differences between the reader systems may not be as pronounced in the low temperature range as they are at temperatures above 150 °C.

#### 230 3.25 SQuartz OSL signal properties

#### 5.1 Quartz OSL

Quartz grains from the investigated site are relatively dim and rarely <u>inherit-present</u> luminescent <u>behaviourproperties</u>. Tests with an EMCCD camera showed that <1 % <u>of the coarse-grained grains</u> emitted a <u>natural luminescence</u> signal when stimulated with <u>BSLblue light</u>. This is common for quartz from Switzerland (Trauerstein *et al.*, 2017) and to allow for bright enough signals during measurement, an aliquot size of 4 mm in diameter was chosen. While <u>this allows for</u>-ca. 200 grains to <u>beare</u> measured at once <u>using this approach</u>, only <u>a</u> few grains are expected to significantly contribute to the emitted luminescence signal and, thereby, these are considered as small aliquots.

The signals emitted by Q and fQ are dominated by the fast component and reduce to background after about <u>15-2</u> to <u>204</u> s of stimulation (Fig. 7). To further investigate the BSL signals, signal decomposition was conducted (Fig. 8) using the numOSL

- 240 package in R (Peng *et al.*, 2018) that applies the 'Levenberg-Marquardt algorithm' suggested by Bluszcz and Adamiec (2006). The initial signals consist of 95 % for Q and 90 % for fQ of the fast component while the relative contribution of the medium components to the natural and regenerative doses are almost identical. The latter is considered an indicator for a stable medium component (Steffen *et al.*, 2009). Further, when assessing D<sub>e</sub>(t)-plots with 0.4 s intervals of Q from RIN2 (Fig. 7), mainly consistent D<sub>e</sub> values are obtained for the measurement of the natural dose as well as for the dose recovery experiment which
- indicates uniformity of signal contribution. For RIN5 fQ, De values decrease with the shift of integration intervals between 0 and 2 s stimulation time. However, this phenomenon is only present in the natural signal but not in those of dose recovery tests. Li and Li (2006a) observed a similar decrease of De values within the first 3.6 s for their DGF-1 sample (coarse-grained, Chinese, aeolian deposit) and found an explanation in a thermally unstable medium component. They proposed the use of De(t)-plots to derive De values separately from the fast and medium component by fitting their Equation (3). For RIN5 fQ,
- 250 negligible differences were found for  $D_e$  values derived using either component assuming photo-ionising cross sections as proposed by Jain *et al.* (2003) and Li and Li (2006b). However, Steffen *et al.* (2009) found that the equation is highly dependent on the photo-ionising cross section of the individual components, the determination of which introduces an array of uncertainties and was therefore deemed unpractical.
- $D_e$  values derived using early background subtraction (0.4 to 1.0 s; cf. Cunningham and Wallinga, 2010) are statistically 255 consistent with those obtained using late background subtraction (2  $\sigma$ ). This indicates an unproblematic slow component. For

subtraction using the last 40 s was applied. An example of an extended dose response curve is shown for one aliquot Q from RIN 2 in Fig. 9. Laboratory saturation levels exceed 600 Gy (Q) and 800 Gy (FQ) and 2\*D<sub>0</sub> values are below 400 Gy (Q) and 580 Gy (fQ; Table 1). Dose response curves of both Q and fQ are well fitted with a double saturating exponential as well as a single saturating exponential function. The latter implies that a single type of trap is responsible for the signal (Aitken, 1998).

#### 3.35.2 Feldspar and polymineral IRSL signal properties

In contrast to Q, the natural signal of 1 mm F aliquots (ca. 13 grains) is very bright and cause saturation of the photomultiplier tube, in particular at high doses. Noteworthy is that only few grains inherit an effective, natural luminescence signal with even 265 less being particularly bright as shown by tests with an EMCCD camera (Fig. 10+). A Schott NG-11 neutral density filter was tested together with the 410 nm-filter combination but failed in protecting the photomultiplier tube from saturation. A cardboard disc with a hole in its centre was mounted into the filter wheel, reducing the photon passage to 4 mm in diameter which allowed to retrieve a detectable signal. A dose recovery test (given dose of ca. 130 Gy) was conducted with and without using the cardboard disc (Fig. 110). Results of both setups are statistically consistent with each other and suggest that the use 270 of the cardboard is unlikely to impact the reliability of the De determination. Hence, the cardboard was used in all IRSL

measurements within this study.

D<sub>e</sub>(t)-plots with 1.5 s intervals show consistent results over the first 200 s of the IRSL signal as presented for both the natural signal and the artificial signal induced during dose recovery tests of RIN2 F and RIN5 PM (Fig. 7). For age determination, De values were derived using the first 1.5 s of the signal and a late background subtraction of the last 50 s. An example of an

extended dose response curve is shown for one aliquot from RIN2 F in Fig. 9. Laboratory saturation levels exceed 1500 Gy 275 (F) and 1800 Gy (PM) and 2\*D<sub>0</sub> values are about 600 Gy (F) and above 425 Gy (PM; Table 1). For both F and PM dose response curves are well fitted with both a double saturating exponential and a single saturating exponential function.

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#### 3.46 De distributions and ages

- 280 The D<sub>e</sub> distributions of the four top Q samples show the tendency towards being positively skewed and have overdispersion (OD) values between 21 ± 3 and 25 ± 3 % (Fig. 12). Arnold et al. (2007) recommend the application of a MAM3 (Galbraith et al., 1999) for De distributions of partially bleached samples that are significantly positively skewed and/or show OD values of >40 %. Following this, skewness of RIN1, RIN2 and RIN4 require for the use of a MAM3. However, the decision tree of Arnold et al. (2007) is based on fluvial samples with divers bleaching histories that are comparably young (<20 ka). For older

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285 samples, it is suggested that distributions may vary as time dependent factors are expected to contribute to the data spread (Galbraith and Roberts, 2012). For example, the impact of beta microdosimetry (Mayva et al., 2006) will increase with the age of the sample. Therefore, the application of a MAM3 was refrained from and derived CAM De values of between

 $144.2 \pm \underline{Gy}6.8$  and  $194.7 \pm 7.8$  Gy were used for the age determination of the top four samples (Table 1). Q ages for these samples range between  $163.5 \pm 9$  ka.1 (RIN4) and  $179.6 \pm 11.0$  ka (RIN3) and are statistically consistent with each other (1  $\sigma$ ). The D<sub>e</sub> distributions of RIN13 Q is also positively skewed but presents a much higher OD value,  $56 \pm 10$  %, in comparison to

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the four top Q samples. the measured aliquots, 12 % are in saturation only allowing for the assessment of a truncated distribution. In this case, the application of the MAM3 is also recommended (Arnold *et al.*, 2007). Besides an expected impact of beta microdosimetry, fitting uncertainties are likely accountable for the skewness and spread of the distribution. For RIN13 Q, the natural doses are projected on the high proportion of the dose response curves emphasising any fitting uncertainty.

- 295 Therefore, a MAM3 D<sub>e</sub> value is unlikely to represent the distribution most appropriately and the CAM D<sub>e</sub> value is considered to represent a more conclusive lower limit of the distribution. The CAM D<sub>e</sub> value of 255.4 ± 34.2 Gy is consistent with the 2\*D<sub>e</sub> limit of 274.1 ± 16.3 Gy and it is arguable whether an obtained age at this range is still reliable. However, the minimum CAM age of 185.8 ± 25.6 ka is consistent with ages of the top four samples and from this and the stratigraphy it can be deduced that RIN13 is of similar age or older than RIN1 to RIN4.
- 300 The four fQ samples (RIN5, RIN6, RIN8, RIN13), representing depths of 10 to 36 m, show normally distributed D<sub>e</sub> values (Fig. 2, Fig. 12, Table 1). This is to be expected for fine-grained samples as the number of grains on each aliquot will have an averaging effect that will mask any skewness of the D<sub>e</sub> distributions. CAM D<sub>e</sub> values of 195.0 ± 4 Gy.4 to 388.0 ± 12.0 Gy were derived which are all well below 2\*D<sub>0</sub> (Table 1). For the lowest samples (RIN13), the obtained age RIN13 with 216.8 ± 12.8 ka is close to the upper limit of reliable quartz ages in the region (Lowick *et al.*, 2015). Obtained fQ ages for the
- 305 two top most lacustrine samples are 108.0 ± 5.6 ka (RIN5) and 113.5 ± 5.9 ka (RIN6). These ages are between 30 and 40 % lower than ages derived for the Q fractions of RIN1 to RIN4. This is from a chrono-stratigraphy perspective unreasonable and can be ascribed to either issues with the Devalue or the dose rate determination. The latter will be discussed in detail in section 4.2. However, if fQ ages are equal or older than Q ages higher Devalues are expected for fQ. This is due to higher Devalues of RIN4 and RIN5 are almost equal (ratio 1.00 ± 0.05) resulting in a much lower for fine-grained sediments. But, Devalues of RIN4 and RIN5 are almost equal (ratio 1.00 ± 0.05) resulting in a much lower 310
- None of the F and PM D<sub>e</sub> distributions show a trend towards being skewed and OD values are between 0 and 17 % (Fig. 12). This is to be expected for the fine-grain PM aliquots that each represent a D<sub>e</sub> value derived from over one million grains. For F, each aliquot consists of only about 13 grains and the D<sub>e</sub> distributions are not affected by skewness. The CAM was applied for all F and PM samples following the decision tree of Arnold *et al.* (2007). CAM D<sub>e</sub> values of between 140.9 ± 3 <u>Gy</u>.1 (RIN1
- 315 F) and 412.8 ± 14.1 Gy (RIN13 F) were derived for the F fraction and between 148.2 ± 1.6 Gy and 332.7 ± 7.2 Gy for the PM fraction (Table 1). The offset between RIN4 F and RIN5 PM is with 19 ± 3 Gy less pronounced than in the quartz fractions.

#### 3.5 6 Fading attributes and correction

#### aFading attributes

- 320 As the IRSL signal commonly is subject to a loss of signal over time, fading tests were conducted following Auclair et al. (2003) Preusser et al. (2014) for a given dose of ca. ~130 Gy. with s Three cycles with storage times of up to 10 hca. 0 s, 1 h, 2.5 h, 5 h and 10 h were implemented per aliquot while theon aliquot constantly remained on the sample arm. This reduces the possibility of sample material being lost during mechanical transfer of the aliquot between sample arm and storage carousel (Preusser et al., 2014). For all fading tests, aliquots previously used for De determination were utilised. Two fading correction
- 325 procedures were conducted, following Lamothe et al. (2003) and Kars et al. (2008), respectively, using the R Luminescence package (Kreutzer and Mercier, 2019; King and Burow, 2019). The first approach is based on the extrapolation of luminescence signal loss to a decadal percentage (g-value) that is used to correct the natural dose and the dose response points while the latter approach utilises the sample specific density of recombination centres ( $\rho$ '-value) to account to correct the dose response curves. For this To apply both approaches,  $\rho'$ - and g-values were derived, whereby the latter was normalised to 2 days.
- Fading properties ( $\rho$ '-value of 2.21\*10<sup>-6</sup>, g-value of 3.1 % ± 0.6 % per decade) were obtained for RIN2 as a representative 330 sample for the F fractions and fading correction was applied to the F measurements, providing corrected De values and ages (Table 1). Following Kars et al. (2008), the corrected F De values, except for RIN13 F, are about 16 % higher than those corrected using Lamothe et al. (2003). 'Kars' corrected De values are between  $247.2 \pm 76.5$  Gy (RIN1) and  $297.3 \pm 65.5$  Gy (RIN4) and well below  $2*D_0$  (RIN1 measured  $57760 \pm 620$  Gy, simulated  $560 \pm 7$  Gy; RIN2 measured  $60326 \pm 8-22$  Gy,
- 335 simulated  $602 \pm 9$  Gy; RIN3 measured  $560990 \pm 18$  Gy, simulated  $590 \pm 9$  Gy; RIN4 measured  $61241 \pm 246$  Gy, simulated 612 ± 24 Gy). For RIN13 F, a significant number of aliquots 12 ('Lamothe' correction) and 19 ('Kars' correction) out of 30 measured samples aliquots are in saturation. Minimum CAM De values of >694.5 Gy and >973.74 Gy are obtained, respectively, which are beyond the  $2*D_0$  limit (measured  $6510.5 \pm 321.8$  Gy; 'Kars' simulated  $666 \pm 4$  Gy). However, for all other samples the 'Kars' corrected F ages (for age determination see section 7) are in good agreement with those of the Q
- 340 fraction (statistically consistent at  $14 \sigma$ , Table 1). It cannot be excluded that this effect is due to an overestimation of both 'Kars' corrected F and Q ages. Due to the lack of evidence for the prior, the Kars et al. (2008) fading correction approach is considered most appropriate for samples of this study,

For PM, fading properties ( $\rho$ '-value of 1.89\*10<sup>-6</sup>, g-value of 2.4-  $\pm$  0.6 % per decade) were obtained from one representative polymineral sample (RIN5). Following Lamothe et al. (2003), the corrected ages are 20 % to 100 % lower than those corrected 345 using Kars et al. (2008). As the latter have shown good agreement for F and Q ages, ages calculated using 'Kars' corrected PM D<sub>e</sub> values are presented in the following. Corrected PM ages of RIN5 and RIN6 are, with  $1388.4 \pm 7$  ka6.6 and  $169.0 \pm \frac{8}{2}$  % and  $\frac{4450}{9}$  % higher than the associated fQ ages. While RIN5 is younger than to be expected from the Q and fading corrected F ages of the overlaying samples (RIN1 to RIN4), the derived fading corrected age is still consistent at  $2\sigma$  with the latter. The corrected PM age of RIN6 is statistically consistent (at  $1\sigma$ ) with the ages of the overlaying samples.

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Consequently, an age offset as observed between Q and fQ cannot be reported for F and PM and confidence of the chosen 'Kars' fading correction is given.

For RIN8 PM and RIN13 PM, corrected ages of  $471.2 \pm 47$  ka.-1 and  $5265.8 \pm 53.2$  ka were calculated, respectively. Fading corrected D<sub>e</sub> values are with  $11010.5 \pm 96.7$  Gy (RIN8) and  $955.4 \pm 85.6$  Gy (RIN13) far beyond the according  $2*D_0$  values (RIN8 measured  $480.1 \pm 4039.5$  Gy, simulated  $536 \pm 4$  Gy; RIN13 measured  $514.5 \pm 398.6$  Gy, simulated  $534 \pm 3$  Gy).

Both g-values, obtained in this study, are similar to those previously found for northern Switzerland (see section 1.). However, while most studies have abstained from applying fading corrections, the here presents 'Kars' fading corrected F and Q ages are in good agreement, thereby justifying the application of this fading correction.

For RIN13 PM, this is in agreement with the minimum age of >436.6 ka obtained for the F fraction of the same sample. There

- 360 are three possible explanations for the high D<sub>e</sub> values of RIN8 and RIN13; 1) the sediment was deposited about half a million years ago, 2) the applied fading correction overestimates for doses derived from the high dose range of the response curve or 3) these D<sub>e</sub> values are the result of an averaged signal with a greater contribution of partially bleached, high residual signals. The latter is an effect that is to be expected for glacier proximal deposits that are likely to have had limited light exposure during short transport times. Samples RIN8 and RIN13 are possibly from such an environment. However, no drastic increase
- 365 in D<sub>e</sub> values was observed for the fQ fraction, but the OSL signal of quartz bleaches faster than feldspar. Nevertheless, corrected D<sub>e</sub> values are higher than the 2\*D<sub>0</sub> values. This measure has been introduced as a dating limit for quartz (Wintle and Murray, 2006) but was found to be as well an appropriate measure for adequately dating feldspars (Zhang and Li, 2020). Therefore, it has been refrained from using the presented results for finite age determination of samples RIN8 and RIN13.

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#### 370 4 Discussion

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#### 4.1 Fading correction

One fading property, the g-value, determined for this study is in good agreement with those obtained for comparable studies in the investigated region (2-3 % per decade; Lowick *et al.*, 2012, 2015; Gaar *et al.*, 2013; Buechi *et al.*, 2017). However, most studies have abstained from relying on fading correction for several reasons. Buechi *et al.* (2017) concluded that already uncorrected PM IRSL ages exceeded the reliable range for dating. Lowick *et al.* (2012) found that corrected IRSL ages overestimated what was to be expected from independent age controls while uncorrected IRSL ages were in good agreement. In Lowick *et al.* (2015), fading correction was rejected as most corrected IRSL ages were higher than quartz ages obtained for the same samples. Gaar and Preusser (2012) found lower g-values of between 0.5 and 1.9 % and argued that corrected PM ages lead to overestimation and much higher ages than those derived for fQ. In contrast, Gaar *et al.* (2013) recommended fading correction for one well bleached sample as corrected F and Q ages were consistent with each other. However, those studies used a fading correction following Huntley and Lamothe (2001), a correction based on the loss of luminescence signal

measured on artificially irradiated aliquots and extrapolated to a decadal percentage (g value) and designed for natural values that are projected onto the linear part of the dose response curve. For most investigated samples, this is not the case and two different approaches to account for anomalous loss of signal over time were applied here. Lamothe *et al.* (2003) found a dose rate correction equation that incorporates differences of irradiation in nature and those of the laboratory irradiation source. This equation is also applicable for geologically old samples. Kars *et al.* (2008) published an approach that requires the estimation of the sample specific density of recombination centres (p' value). This approach may allow correction beyond the linear part. Therefore, these two fading correction measures were found most appropriate for the here investigated samples and were tested, however, it is recognizing that fading measurements and corrections come with a suite of uncertainties.

390 However<u>Nonetheless</u>, 'Kars' corrected F ages have been found to be in good agreement with those of the Q fraction (see 3.5, Table 1) and are therefore considered most appropriate for samples of this study.

#### 7 De distributions and ages

#### 7.1 Quartz OSL

- 395 The D<sub>e</sub> distributions of the four top Q samples show the tendency towards being positively skewed and have overdispersion (OD) values between 21 % ± 3 % and 25 % ± 3 % (Fig. 12). Arnold *et al.* (2007) recommend the application of a MAM3 (Galbraith *et al.*, 1999) for D<sub>e</sub> distributions of partially bleached samples that are significantly positively skewed and/or show OD values of >40 %. Following this, skewness of RIN1, RIN2 and RIN4 require for the use of a MAM3. However, the decision tree of Arnold et al. (2007) is based on fluvial samples with diverse bleaching histories that are comparably young (<20 ka).</p>
- For older samples, it is suggested that distributions may vary as time-dependent factors are expected to contribute to the data spread (Galbraith and Roberts, 2012). For example, the impact of beta microdosimetry (Mayya *et al.*, 2006) will increase with the age of the sample. Therefore, the application of a MAM3 was refrained from and derived CAM  $D_e$  values of between 144 ± 7 Gy and 195 ± 8 Gy were used for the age determination of the top four samples (Table 1). Q ages for these samples range between 164 ± 9 ka (RIN4) and 180 ± 11 ka (RIN3) and are statistically consistent with each other (1  $\sigma$ ).
- 405 The D<sub>e</sub> distributions of RIN13 Q is also positively skewed but presents a much higher OD value, 56 ± 10 %, in comparison to the four top Q samples. Of the measured aliquots, 12 % are in saturation, allowing only for the assessment of a truncated distribution. In this case, the application of the MAM3 is also recommended (Arnold *et al.*, 2007). Besides an expected impact of beta microdosimetry, fitting uncertainties are likely accountable for the skewness and spread of the distribution. For RIN13 Q, the natural doses are projected on the high proportion of the dose response curves emphasising any fitting uncertainty.
- 410 Therefore, a MAM3  $D_e$  value is unlikely to represent the distribution most appropriately and the CAM  $D_e$  value is considered to represent a more conclusive lower limit of the distribution. The CAM  $D_e$  value of 255 ± 34 Gy is consistent with the 2\* $D_0$ limit of 274 ± 16 Gy and it is arguable whether an obtained age at this range is still reliable. However, the minimum CAM age

of  $183 \pm 26$  ka is consistent with ages of the top four samples and from this and the stratigraphy it can be deduced that RIN13 is of similar age or older than RIN1 to RIN4.

- 415 The four fQ samples (RIN5, RIN6, RIN8, RIN13), representing depths of 10 to 36 m, show normally distributed D<sub>e</sub> values (Fig. 2, Fig. 12, Table 1). This is to be expected for fine-grained samples as the number of grains on each aliquot will have an averaging effect that will mask any skewness of the D<sub>e</sub> distributions. CAM D<sub>e</sub> values of 195 ± 4 Gy to 388 ± 12 Gy were derived which are all well below 2\*D<sub>0</sub> (Table 1). For the lowest samples (RIN13), the obtained age of 224 ± 18 ka is close to the upper limit of reliable quartz ages in the region (Lowick *et al.*, 2015). Obtained fQ ages for the two topmost lacustrine
- 420 samples are  $113 \pm 9$  ka (RIN5) and  $117 \pm 9$  ka (RIN6). These ages are between 20 % to almost 50 % lower than ages derived for the Q fractions of RIN1 to RIN4. This, is from a chrono-stratigraphy perspective, is unreasonable and can be ascribed to either issues with the  $D_e$  value or the dose rate determination and will be discussed in detail in section 8.

#### 7.2 Feldspar and polymineral IRSL

None of the F and PM D<sub>e</sub> distributions show a trend towards being skewed and OD values are between 0 % and 17 % (Fig.

- 425 12). This is to be expected for the fine-grain PM aliquots that each represent an averaged  $D_e$  value derived from over one million grains. For F, each aliquot consists of only about 13 grains, whereof few give a luminescence signal (see 5.2), and the  $D_e$  distributions are not affected by skewness. Therefore, as no evidence for partial bleaching is observed, the CAM was applied for all F and PM samples (Table 1). For the upper four samples, 'Kars' corrected CAM  $D_e$  values of between 247 ± 7 Gy (RIN1 F) and 297 ± 6 Gy (RIN4 F) were derived and which equates to ages of 160 ± 7 ka (RIN4) to 167 ± 8 ka (RIN2). These
- **430** ages are statistically consistent at 1  $\sigma$  with those of the Q fraction (Table 1). For RIN5 PM and RIN6 PM 'Kars' corrected CAM D<sub>e</sub> values of 252 ± 3 Gy and 343 ± 9 Gy or ages of 138 ± 7 ka and 169 ± 9 ka were obtained respectively. RIN5 PM presents an age inversion (-22 ± 10 ka) in comparison with the overlying samples RIN1 to RIN4. A similar trend is found for both RIN5 and RIN6 fQ ages. This, is from a chrono-stratigraphy perspective, is unreasonable and can be ascribed to either issues with the D<sub>e</sub> value or the dose rate determination and will be discussed in detail in section 8. However, the PM age of
- RIN6 is statistically consistent at 1 σ with overlying F ages.
   For RIN13 PM, the age is in agreement with the minimum age of >437 ka obtained for the F fraction of the same sample.
   There are three possible explanations for the high D<sub>e</sub> values of RIN8 and RIN13; (1) the sediment was deposited about half a million years ago, (2) the applied fading correction overestimates for doses derived from the higher dose range of the response curve or (3) these D<sub>e</sub> values are the result of an averaged signal with a greater contribution of partially bleached, high residual
- 440 signals. The latter is an effect that is to be expected for glacier-proximal deposits that are likely to have had limited light exposure during short transport times. From the sedimentological context, samples RIN8 and RIN13 are likely from such an environment. However, no drastic increase in  $D_e$  values was observed for the fQ fraction, but it has to be considered that the OSL signal of quartz bleaches faster than feldspar. Nevertheless, corrected  $D_e$  values are higher than the 2\*D<sub>0</sub> values. This measure has been introduced as a dating limit for quartz (Wintle and Murray, 2006) but was found to be as well an appropriate

445 measure for adequately dating feldspars (Zhang and Li, 2020). Therefore, D<sub>e</sub> values of RIN8 PM, RIN13 PM and RIN13 F are unsuitable for the determination of finite ages.

#### 4.28 Age comparison

- For the top four samples, Q and fading corrected F ages range between 1<u>6059.6 ± 6.67 ka</u> (RIN4 F) and 1<u>8079.6 ± 11.6</u> ka
  (RIN3\_Q) and are, thereby, statistically consistent with each other at 21 σ. For the two samples (RIN5, RIN6) below, a discrepancy between fQ and PM ages (2<u>102 %</u> to 30<u>44</u> %) was reported between. Also, fQ ages of these two samples are up to <u>almost</u> -<u>5</u>40 % lower than those of the overlying samples RIN1 to RIN4. This leads to an age offset of ca. <u>35-50 ka</u> between Q and fQ that is unaccountable from a chrono-stratigraphic perspective. The observed age offset equals 2<u>30</u>% to 540 % difference and is likely to be emphasised through the actual age calculation which in terms relies onto derived D<sub>e</sub> and
- 455 DR<sub>total</sub> values. The used D<sub>total</sub> values are latter are all based on the assumptions that firstly, (1) the long-term average estimates of the water content are accurate, and that secondly, (2) used alpha efficiency values are appropriate in this context. However, in contrast to the top four coarse grained samples (RIN 1 to RIN4), D<sub>e</sub> values of RIN5 and RIN6 were determined on the fine-fraction. Therefore, (3) the effect of grain size dependent luminescence characteristics has to be assessed as well.

#### 460 84.2.1 Water content and age determination

A water content of 20 % ± 5 % was assumed for all but two samples which is within 5 % uncertainty of the measured field water content (Table 2). RIN5 and RIN6 have a slightly higher field water content (23 %3 to 24 %) due to their argillaceous character and hence, an average long-term water content of 25 % was found more appropriate for these samples. For comparable similar sedimentary facies, values between 20 % and 30 % were have been used (Anselmetti et al., 2010; Dehnert 465 et al., 2012; Lowick et al., 2015; Buechi et al., 2017). However, the assessment of an appropriate long-term water content is complex but important to avoid age over- or underestimations. It is well known that moisture has an attenuation effect of beta and gamma radiation (Zimmermann, 1971) which may lead to drastic changes in DRtotal and thereby age determination (Nathan and Mauz, 2008). Between 34 % and 49 % of water, in relation to the samples dry weights, were absorbed over a 24 h period by unconsolidated sample material in this study. The highest amounts of water were absorbed by RIN5 and RIN6 which also 470 presented the highest field water contents. For the purpose of comparison, an absolutethe water content maximum can be set to 50 % which is similar toto represent the highest observed absorption capacity for unconsolidated sample material (Table 2). The DR<sub>total</sub> values are calculated for water contents between 10 % and 50 %, in 10 % steps. In addition, a step with 25 % water content, as assumed appropriate for age determination of RIN5 and RIN6, is included in the comparison. Re-calculated ages (Fig. 13 A. and B.) show a difference in ages for the different water contents are up to 40 % for Q, 15 ½ to 25 % for F and 40 % for PM and fQ. A water content of 10 % for Q and F (RIN1 to RIN4) of 10 % results in statistically consistent ages at Formatiert: Überschrift 1

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$2 \sigma$  with fQ and PM (RIN5, RIN6) at 50 %. However, a 10 % moisture for the coarse-grained samples is unlikely as under current conditions ground water penetrates these layers and oxidation features are present. Also, 50 % of moisture for fQ and PM are at the limit of maximum water absorption capacity for unconsolidated material of these samples. It is unlikely that for the consolidated deposits, as found in nature, the maximum absorption capacity will be equally high and that these samples

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were saturated to a maximum for over ca. 150 ka. Therefore, neither 50 % nor 10 % water content are considered representative as a long-term average. Consequently, assumptions of the water content may well have an effect onto the age discrepancy between RIN5 and RIN6 and the coarse-grained samples from above (RIN1 to RIN4) but they are not the sole cause for this offset.

### 485 4.28.2 Alpha efficiency values and age determination

A further cause for the observed discrepancy may be found in the chosen alpha efficiency values (a-values) that account for the efficiency of alpha particles to produce luminescence which are grain size dependent (cf. Mauz et al, 2006). For the coarse Q fraction, the need to consider alpha radiation is circumvented by HF etching the outer rind of the grains and thereby removing the sphere penetrated by alpha particles. However, fQ grains cannot be etched due to the possibility of total dissolution and therefore an appropriate a-value has to be chosen. In this study, an a-value of  $0.05\pm \pm 0.012$  was used for fQ following following. Buechi *et al.* (2017). In the literature, a-values between  $0.02 \pm 0.01$  and  $0.05 \pm 0.01$  have been presented for fine-grained quartz from several continents (e.g. Rees-Jones, 1995; Mauz *et al.*, 2006; Lai *et al.*, 2008) and a-values of  $0.03 \pm 0.02$ 

to 0.05 ± 0.01 were used in studies of northern Switzerland (e.g. Gaar and Preusser, 2012; Lowick *et al.*, 2015; Buechi *et al.*, 2017). For the polymineral fractions, a-values of up to 0.10 ± 0.01 and 0.11 ± 0.01 (e.g. Rees-Jones, 1995; Lang *et al.*, 2003,
Schmidt *et al.*, 2018a) are presented in the literature. However, for two study sites in northern Switzerland, a-values with a

- mean of  $0.05 \pm 0.01$  have been determined while a values of between  $0.05 \pm 0.01$  and  $0.07 \pm 0.02$  (Preusser, 1999a, 1999b; Preusser *et al.*, 2001; Gaar and Preusser, 2012; Gaar *et al.*, 2013; Lowick *et al.*, 2015). Considering that these two studies represent the nearest approximation of geographical position and provenance, their a-value was used here, are commonly used for polymineral and coarse-grained feldspar in the studied region. Here, an a value of  $0.05 \pm 0.01$  is used.
- 500 The impact of a-value estimates onto the age calculation, can be observed for ages of theis investigated samples when by recalculatinged ages for different a-values (0.01 to 0.05 for fQ and 0.01 to 0.11 for PM, Fig. 13 C. and D.). For fQ and PM, a reduction of the a-value leads to a 11 %3 to 16 % age increase, while for corrected F the a-value has an insignificant impact onto the finite ages (-ca. 2 ka). Re-calculated ages are consistent with each other at 2 σ over the a-value spectrum between 0.01 and 0.05. Between corrected F and PM samples, lower a-values (e.g. 0.01) will decrease the age differences. However,
- 505 the chosen a-value of 0.05 is <u>already</u> at the lower limit of values proposed in the literature. <u>Increasing the a-value only</u> emphasises the age difference (Fig. 13 C. and D.). Furthermore, using the lowest possible a-value of 0.01 for fQ, an offset of ca. 20 ka to the Q ages of the top four samples is still apparent. A sample specific determination of a-values <u>would be time</u> consuming and is not straight forward regarding calibration issues, is needed to justify the use of a lower a value. However, in

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absence of sample specific a values and <u>Considering the</u> limited gain of age precision, the initially chosen a-value will be are retained as these appear to be the most reasonable estimates.

Also, with the lowest possible a-value of 0.01 for fQ, an offset to the Q ages of the top four samples and RIN5 and RIN6 is with ca. 20 ka still apparent. A further increase of the used a values will amplify the age offset. Consequently, neither the chosen a value nor water content are the sole causes for the observed age offset.

#### 515 4.28.3 Quartz grain-size dependency

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A further component, capable of causing discrepancies in ages is the effect of grain-size dependent luminescence characteristics. Here, two different grain size ranges (200-250 and 4-11 µm) were used for age determination and the offset is seemingly marked by the two grain size groups.

A comparison of fine and coarse grained quartz of waterlain sediments from the studied region reported ages in agreement for both fractions or older ages for the fine-grained fractions (Lowick *et al.*, 2015). Similarly, in several studies older ages are presented for the fine-grained fractions of alluvial samples, even though, bleaching conditions were expected to be preferential for the fine fractions (e.g. Olley *et al.*, 1998; Colls *et al.*, 2001). Nevertheless, a pattern has been observed for numerous sites from around the globe where fine-grained quartz ages of ~40 ka underestimate ages derived using coarse grained quartz and/or independent age controls (cf. Timar-Gabor *et al.*, 2017). A dose dependency (>100 Gy) was linked to this phenomenon and

525 thermal instability was rejected as a potential cause, though, a finite explanation is still lacking. Given the internal consistency of Q ages for RIN1 to RIN4 and findings summarised by Timar-Gabor *et al.* (2017), fQ ages of RIN5 and RIN6 should be regarded as minimum age estimates. This is also applicable for fQ of RIN8 and RIN13.

#### 8.3 Implications

Neither the chosen water contents nor the a-values are the sole cause An obvious cause for the age offset between RIN5 and

530 RIN6 fQ and RIN1 to RIN4 Q-has not been identified. Given the internal consistency of Q ages for RIN1 to RIN4 and the chrono-stratigrahic context findings summarised by Timar-Gabor et al. (2017), fQ ages of this study should be regarded as minimum age estimates. RIN5 and RIN6 should be regarded as minimum age estimates. Unlike for other studies in the area (e.g. Gaar and Preusser, 2012), corrected F ages are in agreement with Q ages while uncorrected ages are suspect to significant age underestimate. However, obtained Q ages remain under the reliable quartz luminescence age limit of ca. 250 ka (Anselmetti

535 et al., 2010; Dehnert et al., 2012; Lowick et al., 2015; Buechi et al., 2017). With only minimum age estimates of the two lowest samples (RIN8 fQ >163 ka, RIN13 Q >183 ka, RIN13 fQ >224 ka) it remains unclear whether the transition from glacial/proglacial influenced environments to lacustrine environment was of rapid nature or occurred much earlier than the deposition of the entire lacustrine sequence. For the lacustrine sequence, ages of 169 ka (RIN6 PM) and 138 ka (RIN5 PM) were obtained. The sandy top of the lacustrine unit is with ca. 160 ka (RIN4 F, Q) Formatiert: Überschrift 2

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540 statistically consistent with ages from the sandy diamicton (RIN1 to RIN3; 160 to 180 ka F and Q). This is also applicable for fQ of RIN8 and RIN13.

This indicates a deposition of at least 16.6 m during Marine Isotope Stage 6 (MIS 6) with a rapid transition from lacustrine to colluvial-dominated, periglacial environments.

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# 95 Conclusions

The investigated IL uninescence properties of coarse- and fine-grained quartz, feldspar and polymineral fractions of eight samples from a palaeovalley in northern Switzerland were assessed, and six were found appropriate for finite datingQuartz components have been found to be stable and the occurrence of a second exponential function in the dose response curves is

- 550 missing. Noteworthy is the presence of positively skewed De distributions for coarse-grained quartz, but the lack of a comparable pattern for the slower bleaching feldspar suggests that partial bleaching is unlikely to influence these distributions. However, an age discrepancy between fine- and coarse-grained quartz is present. Different scenarios for dose rate components (a-value, water content) are assessed, but do not explain the age discrepancy. From the two applied fading corrections, the 'Kars' fading corrected feldspar ages are in good agreement with each other, with the quartz ages and with one fading corrected
- 555 polymineral age from the unit below. Noteworthy is the presence of positively skewed  $D_{e}$  distributions for coarse-grained quartz while the slower bleaching feldspar lacks of a comparable pattern suggesting that partial bleaching is unlikely to influence these distributions. Not all minerals, fractions or samples are suitable to be dated using the here presented luminescence techniques, nonetheless, this systematic investigation of different luminescence signals allows for a decisive chronology to established. For the chosen dose rates (a value of 0.05, water content of 20 to 25 %), derived age estimates are
- 560 in good agreement with each other and the chrono-stratigraphic context (Fig. 2). For the upper four samples (RIN1 to RIN4), Q and 'Kars' fading corrected F ages are in good agreement with each other and with fading corrected PM ages of two samples directly underneath (RIN5, RIN6). Minimum fQ for the two latter are no contradiction. This indicates For the study site, Rinikerfeld, a deposition of at least 16.6 m during MIS 6 with a rapid transition from lacustrine to colluvial-dominated, periglacial environments is reconstructed. For the the-two lowest lowest samples, only minimum age estimates can be derived
- 565 from the chrono-stratigraphic context indicating that those glacial to proglacial diamictons, F and PM measurements exceeded the dating limit at 2\*D<sub>0</sub>-but minimum fQ and Q ages indicate that these samples are are of similar age or older than the top units.

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## Author contributions

570 Manuscript conceptualisation and experiment design was conducted by DM with support by FP. DM obtained and analysed the presented data and prepared the manuscript with contributions from all co-authors. Sample context and material was provided by MB and LG. LG contributed Fig. 1 and parts of Fig. 2 and Fig. 13 while GD was instrumental for funding acquisition.

#### 575 Competing interests

The authors declare that they have no conflict of interest.

# Data availability

Data is available upon request.

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### **Figures and Tables**

Fig. 1 <u>A. Location of the study area within Switzerland. B.</u> Overview of the study area and location of drill site.
Fig. 2 Core log and age-depth model of the investigated site. Ages for all minerals and fractions are presented with 1 σ uncertainty. Fading corrected ages are calculated following Kars et al. (2008). Grain size fractions are subdivided into clay (C), silt (Si), sand (Sa), gravel (Gr) and a combined fraction of cobbles and boulders (Co, Bo).
Fig. 3 Preheat plateau, dose recovery and thermal transfer test sresults for Q and F of RIN2 and fQ and PM of RIN5. Presented are CAM D<sub>e</sub> values with 1 σ uncertainties for 3 (F, M) or 5 (Q, fQ) aliquots per preheat temperature. Dose recovery results are

**Fig. 4** To determine thermal quenching, repeated short shine measurements of Lx/Tx for a given dose of 90 Gy after preheats at different temperatures were conducted on one aliquot of RIN2 F.

785 Fig. 5 Short shine pulse annealing experiment on naturally and laboratory induced doses of two aliquots from RIN2 F. The IRSL signal is normalised to a test dose and re-normalised to the first measurement.

Fig. 6 De determination of the natural signal using different stimulation temperatures for each three aliquots of RIN2 F.

- Fig. 7 D<sub>e</sub>(t)-plots of the natural dose determination and dose recovery (DR) tests for Q and F of RIN2 and fQ and PM of RIN5.
  D<sub>e</sub>(t)-plots were calculated for 0.4 s intervals (Q, fQ) and 1.5 s (F, PM). The normalised luminescence signals of the natural or first given (DR) dose are presented Decay and D<sub>e</sub>(t) plots of the natural doses and dose recovery tests for Q and F of RIN2 and fQ and PM of RIN5...
- 795 Fig. 8 Component deconvolution of the natural and regenerative OSL response of RIN2 Q and RIN5 fQ.

Fig. 9 Extended dose response curves for RIN2 Q and F and RIN5 fQ and PM.

presented as measured-to-given-dose-ratios (M/G-ratio).

Fig. 10 <u>Coloured EMCCD image obtained from the natural IRSL signal of 100 F grains of RIN2. The grains were placed on</u>
 a <u>Risø single grain disc with a 10\*10 grid of holes</u> (300 μm in diameter-). A. Holes containing F grains emitting luminescence

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are presented in with dashed green circles while those without emission are shown in white. **B.** The 3D surface plot emphasises that some grains inherit very bright signals while others are rather dim.

Dose recovery test (RIN2 F, 130 Gy given dose) at different preheat temperatures with and without a mounted cardboard barrier to reduce photon passage.

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Fig. 11 Dose recovery test (RIN2 F, 130 Gy given dose) at different preheat temperatures with and without a mounted cardboard barrier to reduce photon passage.

Coloured EMCCD image obtained from 100 F grains of RIN2. The grains were placed on a Risø single grain dise with a 10\*10 grid of holes. A, Holes containing F grains emitting luminesce are presented in green while those without emission are shown in white. B. The 3D surface plot emphasises that some grains inherit very bright signals while others are rather dim.

Fig. 12  $D_e$  distributions of all measured samples and minerals. CAM  $D_e$  values are given with 1  $\sigma$  uncertainty. F and PM  $D_e$  values are uncorrected.

Fig. 13 Ages of A. Q and fQ as well as B. F and PM (fading corrected after Kars et al., 2008) are shown for water contents between 10% and 50% and plotted against depth (line signaturesymbols). C. Ages of fQ are shown for alpha efficiency values between 0.01 and 0.05 and plotted against depth (line signature). No alpha component was considered for age determination of HF etched Q, but accepted ages are presented for completeness. D. F and PM (fading corrected after Kars et al., 2008) are shown for alpha efficiency values between 0.01 and 0.11. Ages of C. fQ as well as D. F and PM (fading corrected after Kars et al., 2008) are shown for alpha efficiency values between 0.01 and 0.11. Ages of C. fQ as well as D. F and PM (fading corrected after Kars et al., 2008) are shown for alpha efficiency values between 0.01 and 0.11 and 0.05 and plotted against depth (line symbols). No alpha component was considered for age determination of HF etched Q, but accepted ages are presented for the sake of completeness. Accepted ages are presented with 1 σ uncertainties as point symbols signature. The upper part of the core log is given on the right for comparison (see Fig. 2 for full log).

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**Table 1**. De values and derived ages for all samples,  $1 \sigma$  uncertainties are given. The number of accepted/measured aliquots is provided by n. Ages to be considered for chrono-stratigraphic interpretation are bold.

Table 2. Dosimetric data and total dose rates as used for age determination (all values are given with 1  $\sigma$  uncertainties).

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Fig. 1 A. Location of the study area within Switzerland. B. Overview of the study area and drill site Fig. 1 Overview of the study area and location of drill site (data source: Swisstopo, 2013).





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Fig. 4 To determine thermal quenching, repeated short shine measurements of Lx/Tx for a given dose of 90 Gy after preheats at different temperatures were conducted on one aliquot of RIN2.



Fig. 5 Short shine pulse annealing experiment on naturally and laboratory induced doses of two aliquots from RIN2. The IRSL signal is normalised to a test dose and re-normalised to the first measurement.



Fig. 6 De determination of the natural signal using different stimulation temperatures for each three aliquots of RIN2 F.



RIN2 – Q











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0.2

0.0

50

0 }

40 80









200

150

100

50

250

200

150

100

50

• 🕂

40 80 120 160







• D<sub>e</sub> \_\_\_\_\_ ca. 130 Gy

€ . . . . 1.0

0.8

0.6

0.4

0.2

000

s

200 0.0 g











**Fig.** 7  $D_{e}(t)$ -plots of the natural dose determination and dose recovery (DR) tests for Q and F of RIN2 and fQ and PM of RIN5.  $D_{e}(t)$ -plots were calculated for 0.4 s intervals (Q, fQ) and 1.5 s (F, PM). The normalised luminescence signals of the natural or first given (DR) dose are presented. Decay and  $D_{v}(t)$ -plots of the natural doses and dose recovery tests for Q and F of RIN2 and fQ and PM of RIN5.

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Fig. 8 Component deconvolution of the natural and regenerative OSL response of RIN2 Q and RIN5 fQ.



870 Fig. 9 Extended dose response curves for <u>RIN2 RIN2</u> Q and F and <u>RIN5 fQ and PM</u>.



Fig. 10 Dose recovery test (RIN2 F, 130 Gy given dose) at different preheat temperatures with and without a mounted cardboard barrier to reduce photon passage.



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Sample	М	Grain	n	OD <sup>A</sup>	2D <sub>0_CAM</sub>	D <sub>e_CAM</sub>	D <sub>e_Lamothe</sub> <sup>B</sup>	D <sub>e_Kars</sub> C	Age_ <sub>CAM</sub> D	Age <sub>Lamothe</sub> <sup>B</sup>	Age <sub>Kars</sub> <sup>C</sup>	
code		size (µm)		(%)	(Gy)	(Gy)	(Gy)	(Gy)	(ka)	(ka)	(ka)	
RIN1	Q	200-250	30/31	24 ± 3	31 <del>5<u>6</u>.5</del> ± 1 <del>6.6</del> 7	1 <u>49.50</u> ± <u>7</u> 6.9	-	-	177-4 ± 1 <u>10-5</u>	-	-	Formatiert: Schriftart: Fett
	F	150-200	30/30	11 ± 2	577 <del>.2</del> ± 20 <del>19.5</del>	14 <u>10.9</u> ± 3 <del>.1</del>	21 <u>4.85</u> ± 5 <del>.4</del>	247 <del>.2</del> ± <u>76.5</u>	94 <del>.0</del> ± 4 <del>.0</del>	143 <del>.4</del> ± 6 <del>.3</del>	165 <del>.0</del> ± 7 <del>.3</del>	Formatiert: Schriftart: Fett
RIN2	Q	200-250	30/31	25 ± 3	 34 <u>32.5</u> ± 1 <del>2.7</del> 3	144 <del>.2</del> ± <u>76.8</u>	-	·	_168.9 ± 10 <del>.2</del>	-	-	Formatiert: Schriftart: Fett
	F	200-250	30/30	14 ± 2	626 <del>.2</del> ± 2 <u>21.7</u>	158 <del>.4</del> ± 4 <del>.4</del>	24 <u>21.9</u> ± <u>87.5</u>	280 <del>.2</del> ± 9.4	9 <u>54.6</u> ± 4.4	14 <del>4.</del> 5 ± 7 <del>.0</del>	167 <del>.4</del> ± 8 <del>.3</del>	Formatiert: Schriftart: Fett
RIN3	Q	200-250	30/30	25 ± 3	317 <del>.4</del> ± 15.4	146 <del>.3</del> ± <u>76.9</u>	-	-	1 <u>8079.6</u> ± 11 <del>.0</del>	-	-	Formatiert: Schriftart: Fett
DINIA	F	200-250	30/31	14 ± 2	609 <del>.3</del> ± 18 <del>.3</del>	151 <del>.0</del> ± 4 <del>.0</del>	22 <u>87.7</u> ± <u>76.6</u>	265 <del>.0</del> ± 8 <del>.2</del>	92 <del>.4</del> ± 4 <del>.2</del>	139 <del>.3</del> ± <u>7</u> 6.6	162 <del>.1 ± <u>8</u>7.9</del>	Formatiert: Schriftart: Fett
RIN4	Q F	200-250 150-200	30/33 30/30	21 ± 3 6 ± 1	365 <del>.2</del> ±1 <u>65.8</u> 64 <u>10.5</u> ± 25.6	19 <u>54.7</u> ± 7.8 16 <u>76.8</u> ± 2 <del>.3</del>	- 25 <u>65.5</u> ± 4.1	- 297 <del>.3</del> ± <u>6</u> 5.5	$1643.5 \pm 9.1$ 9089.5 ± 43.5	- 137 <del>.1 ± 5.</del> 6	- 1 <u>60</u> 59.6 ±	Formatiert: Schriftart: Fett
											<u>7</u> 6.6	Formatiert: Schriftart: Fett
RIN5	fQ	4-11	7/7	-	43 <u>10.7</u> ± 2 <u>8</u> 7.7	195 <del>.0</del> ± <del>4.</del> 4 <sup>F</sup>	-	-	>1 <u>1308.0</u> ± <del>5.6</del> 9 <sup>F</sup>	-		
	PM	4-11	30/30	3 ± 1	445 <mark>.2</mark> ± 19 <mark>.0</mark>	148 <del>.2</del> ± <u>2</u> 1.6	20 <u>9</u> 8.6 ± 2.4	25 <u>2</u> 1.6 ± 3.1	8 <u>2</u> 1.5 ± <u>4</u> 3.9	11 <u>5</u> 4.8 ± <u>6</u> 5.5	138-4 ± <u>76-6</u>	Formatiert: Schriftart: Fett
RIN6	fQ	4-11	7/7	-	459 <del>.0</del> ± 31 <del>.4</del>	227 <del>.3</del> ± 5 <del>.3</del> F	-	-	>11 <u>7<del>3.5</del> ± 95.9<sup>F</sup></u>	-	-	
	PM	4-11	7/7	-	45 <u>87.5</u> ± 44.0	18 <u>87.5</u> ± <u>43.7</u>	27 <u>2</u> 1.7 ± <u>6</u> 5.9	343 <del>.0</del> ± <u>98.6</u>	9 <u>22.4 ± 54.7</u>	13 <u>43.9</u> ± <u>76.9</u>	169 <del>.0</del> ± 8.9	Formatiert: Schriftart: Fett
RIN8	fQ	4-11	7/7	-	577 <del>.1</del> ± 2 <u>76.8</u>	365 <del>.0</del> ± 10 <del>.4</del> <sup>F</sup>	-	-	≥1 <u>6358.2</u> ± <u>13</u> 8.8 <sup>F</sup>	-	-	Formatiert: Schriftart: Fett
	PM	4-11	7/7	-	480 <del>.1</del> ±	31 <u>34</u> .7 ± <u>8</u> 7.6	5 <u>60</u> 59.7 ±	110 <u>1</u> <del>0.5</del> ±	13 <u>4</u> 4.3 ± 7 <sup>∺</sup> .2	2 <u>40</u> 39.6 ±	471 <del>.2</del> ±	
					<u>40</u> 39.5		19 <del>.3</del>	9 <del>6.</del> 7		14 <sup>H</sup> .1	47 <sup>⊞</sup> .1	
RIN13	Q	200-250	19/26	56 ± 10	274 <del>.1</del> ± 16 <del>.3</del>	255 <del>.4</del> ± 34.2 <sup>6</sup>	-	-	>18 <u>32.8</u> ± 25.6 <sup>FG</sup>	-	-	Formatiert: Schriftart: Fett
	fQ	4-11	7/7	-	545 <del>.2</del> ± 28 <del>.4</del>	388 <del>.0</del> ± 12 <del>.0</del> <sup>F</sup>	-	-	>22416.8 ±	-	-	Formatiert: Schriftart: Fett, Hervorheben
	F	200-250	30/30	17 ± 3	65 <u>10.5</u> ± 3 <u>2</u> 1.8	41 <u>3</u> 2.8 ± 14.1	>69 <del>4.</del> 5 <sup>E</sup>	>97 <u>3.74</u> <sup>E</sup> ⊞	1 <u>8</u> 2.3 185.4 ± <u>10</u> 9.5	>311 <del>.4<sup>E∐</sup></del>	>43 <u>6.67</u> <sup>E</sup> <u>H</u>	Formatiert: Hervorheben
	PM	4-11	7/7	-	514.5 ± 3 <u>8.69</u>	33 <u>32.7</u> ± 8 <u>.2</u>	542 <del>.4</del> ± 17 <del>.3</del>	9 <u>5</u> 55.4 ± 8 85.6 <sup>H</sup>	183 <del>.1</del> ± <del>9.7<u>10</u></del>	29 <u>9</u> 8.5 ± 17 <del>.0</del>	52 <u>6</u> 5.8 ±	

<sup>A</sup> Overdispersion was calculated using the CAM (Galbraith et al., 1999).

<sup>B</sup> A g-value (normalised to 2 days) of 3.1 ± 0.6 was used for F of RIN1 to RIN4 and RIN13 and of 2.4 ± 0.6 for PM of RIN5 to RIN13.

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	920	<sup>C</sup> A ρ'-value of 2.21*10 <sup>6</sup> was used for F of RIN1 to RIN4 and RIN13 and of 1.89*10 <sup>6</sup> for PM of RIN5 to RIN13.		Formatiert: Hochgestel
		$^{\rm D}$ Ages were calculated using the weighted mean of the uncorrected D <sub>e</sub> values derived with the CAM (Galbraith et al., 1999).		Formatiert: Hochgestel
		<sup>E</sup> Out of 30 measured aliquots, 12 (Lamothe') and 19 (Kars') aliquots are in saturation following fading correction and, therefore, the values shown are derived using a CAM on a		
		truncated distribution.		
925		<sup>F</sup> Regarded to be minimum estimates due to grain size dependent D <sub>e</sub> underestimation <u>chrono-stratigraphic reason</u> .		
	<sup>6</sup> Derived CAM is based on a truncated distribution and results should be considered as minimums.			
		<sup>H</sup> Based on D <sub>a</sub> values beyond 2*D <sub>a</sub> .	-(	Formatiert: Tiefgestellt
				Formatiert: Tiefgestellt

## Table 2. Dosimetric data and total dose rates as used for age determination (all values are given with 1 $\sigma$ uncertainties).

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Sample	Core	WC	WC	WC	Radio	nuclide concenti	rations	DRcosmic	DRtotal	DRtotal
code	depth	field <sup>A</sup>	max <sup>A</sup>	used <sup>A</sup>	U	Th	к	(Gy ka <sup>-1</sup> )	Q and fQ <sup>B</sup>	F and PM <sup>C</sup>
	(m)	(%)	(%)	(%)	(ppm)	(ppm)	(%)		(Gy ka <sup>-1</sup> )	(Gy ka <sup>-1</sup> )
RIN1	4.12	16	39	20 ± 5	0.81 ± 0.17	$3.52 \pm 0.24$	$0.44 \pm 0.03$	0.135 ± 0.014	$0.84 \pm 0.05$	1.50 ± 0.06
RIN2	6.12	17	46	20 ± 5	0.69 ± 0.15	3.31 ± 0.22	$0.53 \pm 0.04$	0.108 ± 0.011	$0.85 \pm 0.05$	1.67 ± 0.08
RIN3	6.34	19	34	20 ± 5	0.93 ± 0.18	$3.20 \pm 0.22$	$0.49 \pm 0.04$	0.105 ± 0.011	0.81 ± 0.05	$1.63 \pm 0.08$
RIN4	9.04	18	40	20 ± 5	1.44 ± 0.26	4.90 ± 0.30	0.77 ± 0.05	0.081 ± 0.008	$1.19 \pm 0.07$	1.86 ± 0.07
RIN5	10.25	24	48	25 ± 5	$1.54 \pm 0.27$	$6.00 \pm 0.40$	0.96 ± 0.07	$0.073 \pm 0.007$	1.7 <del>9</del> <u>3</u> ± 0. <del>09</del> <u>14</u>	$1.82 \pm 0.09$
RIN6	16.55	23	49	25 ± 5	1.75 ± 0.29	6.70 ± 0.40	1.18 ± 0.08	$0.044 \pm 0.004$	<u>12.9400 ± 0.150</u>	2.03 ± 0.10
RIN8	29.63	19	42	20 ± 5	$2.26 \pm 0.35$	$6.80 \pm 0.50$	$1.28 \pm 0.09$	0.020 ± 0.002	2. <del>31<u>23</u> ± 0.1<u>8</u>3</del>	2.34 ± 0.13
RIN13	35.37	15	45	20 ± 5	1.53 ± 0.24	$5.50 \pm 0.40$	0.99 ± 0.06	0.015 ± 0.002	1.40 ± 0.06 (Q)	2.23 ± 0.11 (F)
									1.7 <u>3</u> 9 ± 0.1 <u>40</u>	1.91 ± 0.10 (PM)
									(fQ)	

<sup>A</sup> Water content as measured from the samples (field), maximum absorption capacity as measured in laboratory tests (max) and as used for D<sub>g</sub> determination (used).

 $^{B}$  Alpha efficiency of 0.054  $\pm$  0.042 was assumed for fQ.

<sup>C</sup> Alpha efficiency of 0.05  $\pm$  0.01 and <u>internal</u> potassium content of 12.5 <u>%</u>  $\pm$  0.5 % were assumed.

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Formatiert: Tiefgestellt

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