Supplement

A progressively elevated temperature (PET) IRSL SAR procedure – first experiments and results

Annette Kadereit[1], Mariana Sontag-González[1,2,3], Sebastian Kreutzer[1], Marco Colombo[1], Christoph Schmidt[4,] Paul R. Hanson[5]

[1]Institute of Geography, Heidelberg University, Im Neuenheimer Feld 348, 69120 Heidelberg, Germany

[2]Luminescence Dating Laboratory, Research Laboratory for Archaeology & the History of Art, School of Archaeology, University of Oxford, OX1 3QY Oxford, UK

[3]Institute of Geography, Justus Liebig University of Giessen, 35390 Giessen, Germany

[4]Institute of Earth Surface Dynamics, University of Lausanne, Bâtiment Géopolis, 1015 Lausanne, Switzerland

[5]Conservation and Survey Division, School of Natural Resources, University of Nebraska-Lincoln, Lincoln, Nebraska, USA

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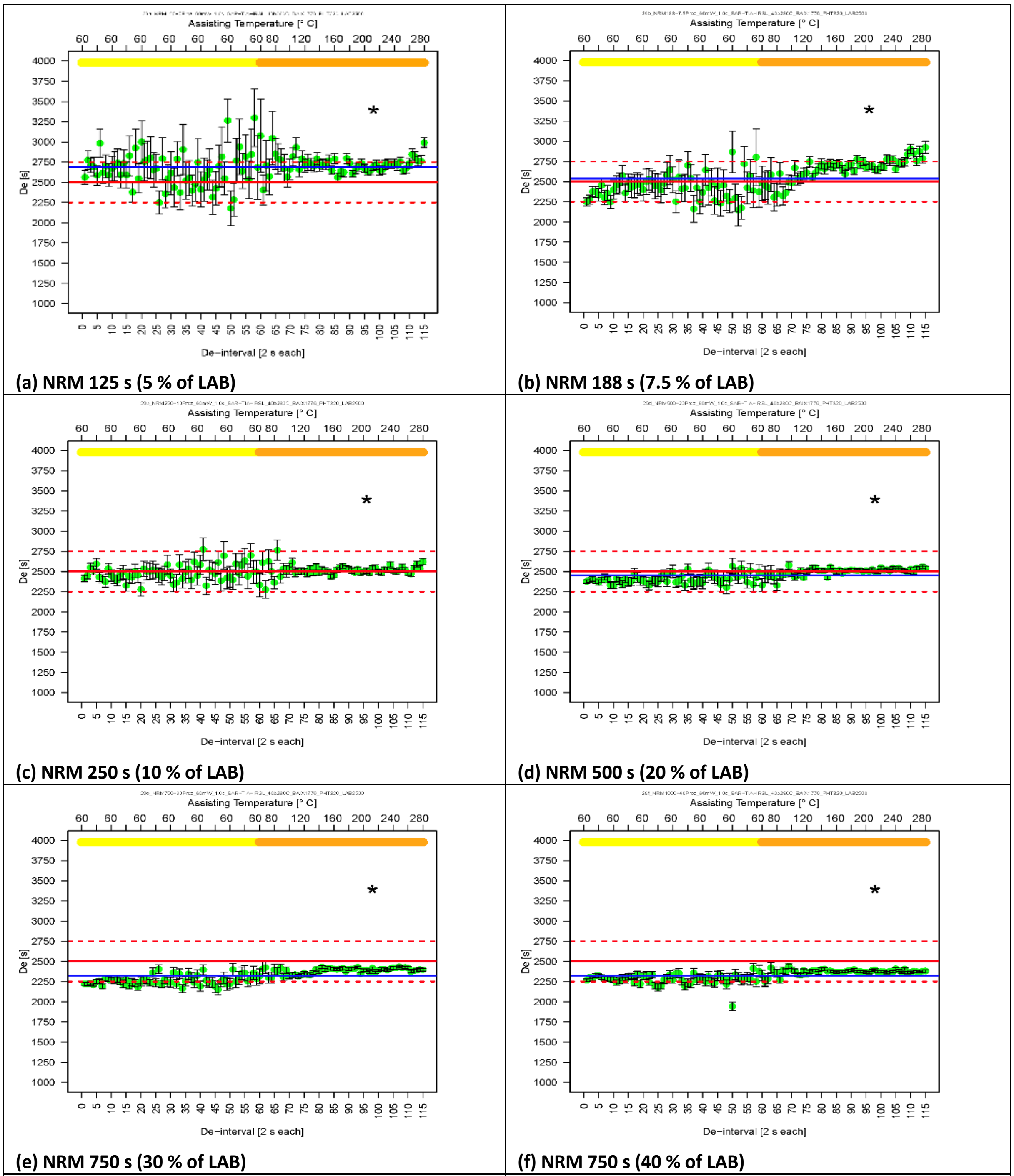
Abbreviations

1	DRC (supplement only)	dose-response curve
2	DRC-Aim (supplement only)	Expected dose reflected by REG 100 %
3	DRC 100 s (supplement only)	dose-response curve build-up around the central REG 100 % (expected dose) which here corresponds to 100 s/10.6 Gy; like (13) used in the main text
4	HBL	hotbleach
5	LAB	laboratory dose (dose administered in the luminescence reader)
6	NRM	normalisation dose (also “test dose”) used for sensitivity correction of a SAR measurement
7	PET	progressively elevated temperature; here PET-IRSL SAR
8	PET_{max}	maximum readout temperature during PET-IRSL SAR
9	PET-peak	Peak of the signal curve in the high-temperature PET-IRSL range
10	PHT	preheat temperature
11	REG	regeneration dose point
12	REGs	placed at 40 %, 70 %, 100 %, 130 %, 160 %, 0 % and 100 % of an expected dose for dose-response curve construction
13	2500 s/300 Gy dose response curve	dose-response curve build-up around the central REG 100 % (expected dose) which here corresponds to 2500 s/300 Gy

S1. Supplement 1 – LPS Baix – HDS-1776

S1.1. Results of the NRM test on sample HDS-1776 from the loess-palaeosol section (LPS) Baix



(a) NRM 125 s (5 % of LAB)

(b) NRM 188 s (7.5 % of LAB)

(c) NRM 250 s (10 % of LAB)

(d) NRM 500 s (20 % of LAB)

(e) NRM 750 s (30 % of LAB)

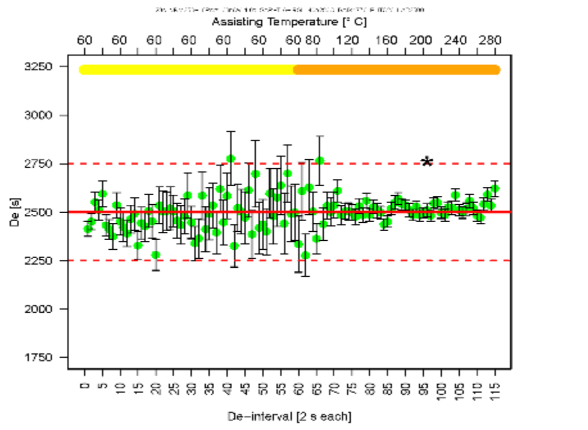
(f) NRM 750 s (40 % of LAB)

Legend:

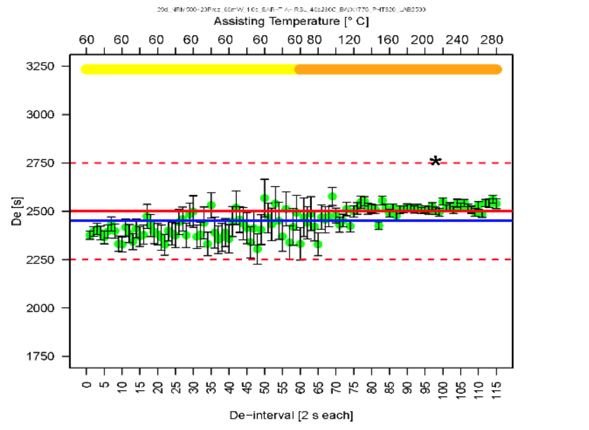
- De value
- Mean of all De values
- Expected/Administered dose
- 10 % error margin of expected value
- 60 °C temperature range
- Temperature increase 60–280 °C
- Position of PET-peak

Figure 1.1.1. Normalisation dose (NRM) test on six tiny aliquots (ø 2 mm) of feldspar coarse grains (125–212 µm) of the sample HDS-1776 from the loess section Baix analysed with the PET-IRSL SAR protocol. The administred laboratory dose (LAB) is 2500 s (ca. 303 Gy). The dose-response curve aims at 2500 s, 100 %, whith regeneration dose points (REGs) placed at 40 %, 70 %, 100 %, 130 %, 160 %, 0 % (recuperation) and 40 % (recycling ratio) of LAB. NRM was set at 5 %, 7.5 %, 10 %,

20 %, 30 % and 40 % of LAB (one aliquot each; cf. $pIR_{60}IR_{225}$ screening and dating studies by Pfaffner et al. 2024; 2025). The TL preheat was 60 s at 320 °C (280 °C in the pevious studies). PET starting with 120 s IR at 60 °C (cf. yellow line along top horizontal axis), followed by continuous temperature increase from 60 °C to 280 °C (cf. orange line along top horizontal axis) within 110 s (ramp 2 °C/s). The total IR-stimulation time is 120 s + 110 s = 230 s (2300 data channels; 0.1 s/data channel). Final green light stimulated luminescence (GRSL) hotbleach at 280 °C at the end of each SAR half-cycle (both REG and NRM half-cycle; no optical washing and or hotbleach in the $pIR_{60}IR_{225}$ studies). De-signal integral length for data analysis 2 s (20 data channels each; 115 integrals altogether; cf. bottom horizontal axis of graphs). Dose response curve fitted with a single exponential fit (*versus* exponential plus lineat fit in the $pIR_{60}IR_{225}$ studies) applying the function analyse_SAR.CWOSL of the R.Luminescence package (developer version 09.24.9000–106; Kreutzer et al. 2024). Like in the screening and dating studies NRM of 10 % and 20 % gave the best results. For NRM 10 % the mean with standard deviation for the De-signal integrals 1–60 (corresponding to 120 s IR-readout at 60 °C) is 2482.4 ± 96.4 s and for the De-signal integrals 75–115 (IR-readout 120–280 °C) 2517.3 ± 37.4 s, for NRM 20 % the corresponding values are 2407.8 ± 58.1 s and 2517.0 ± 24.4 s. The relative De errors (1 sigma) range for NRM 10 % from 1.4–6.6 % for the readout at 60 °C and 1.1–1.7 % for the increasingly hotter readout, for NRM 20 % from 0.9–4.2 % for the readout at 60 °C and 0.6–1.2 % for the hotter readout. While the central value slightly underestimates the given dose by 3.7 % for the 60 °C readout with NRM 20 %, the errors of the individual De values as well as the mean values are somewhat smaller for NRM 20 %. Although a „perfect" dose recovery for both, lower and higher temperature assistance, might be an important issue for some applications (e.g., if the post-isothermal (pIt-)IR protocol shall be applied for which the lower and higher temperature Des are the base to calculate a derived De for age calculation; Lamothe et al. 2020) this is not the case for the present study. We assume that the De values gained at a lower temperature are of a minor quality associated with instable crystal defects. Therefore, we decided for **NRM 20 % (500 s)** providing not only the smaller errors but which had also been used in the predessor studies on the Baix loess samples.



(a) NRM 250 s (10 % of LAB)



(d) NRM 500 s (20 % of LAB)

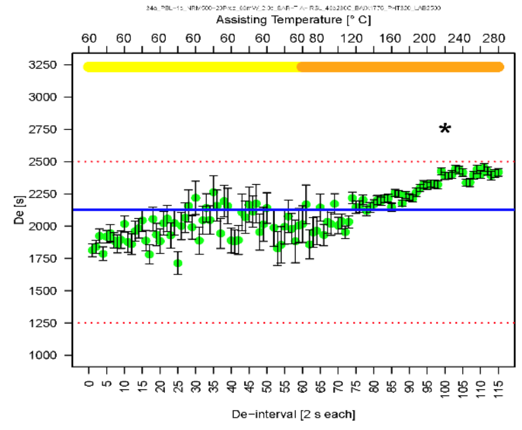
Legend:

- De value
- Mean of all De values
- Expected/Administered dose
- 10 % error margin of expected value
- 60 °C temperature range
- Temperature increase 60–280 °C
- ★ Position of PET-peak

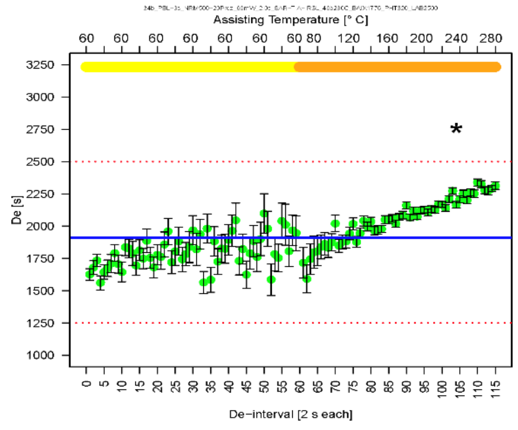
Figure 1.1.2. NRM-Tests with NRM 10 % and 20 % of LAB 2500 with enlarged y-axis. Expected value denoted by red line. NRM 20 % slightly underestimating expected value for most of the 60 °C range, but providing significantly smaller De errors and data scatter.

S1.2. PBL-tests on HDS-1776 – White-light bleaching – repeatedly used aliquots

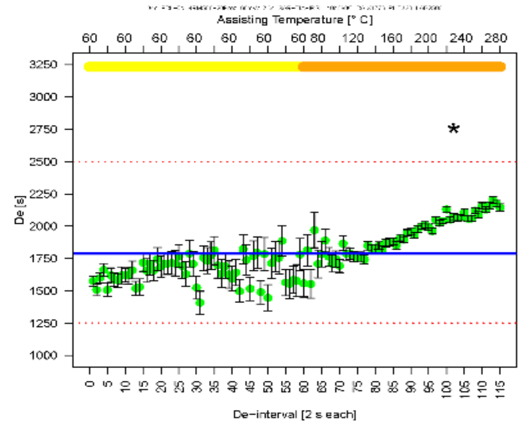
S1.2.1. Results of the partial bleach (PBL) tests on sample HDS-1776 from the loess-palaeosol section Baix – Variant IRR 1250 s + $TL_{archive}$ + PBL + IRR 1250 s + $TL_{Laboratory}$



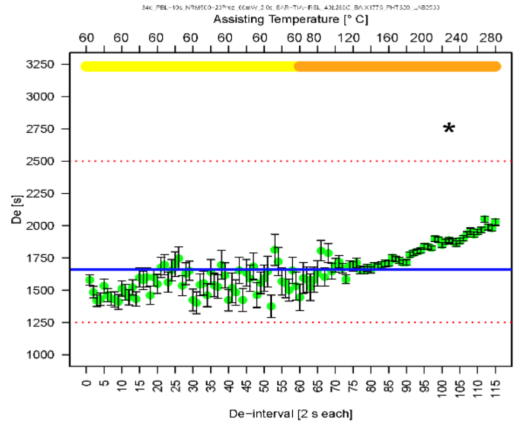
(a) PBL 1 s



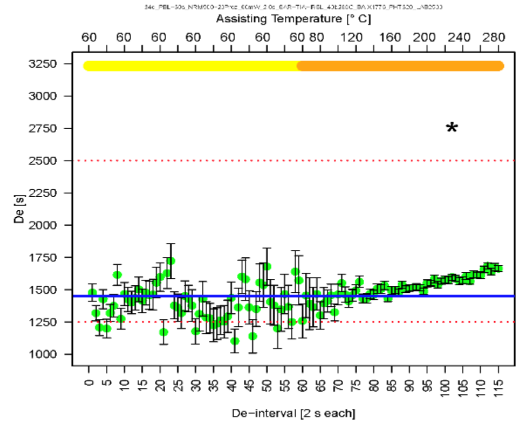
(b) PBL 3 s



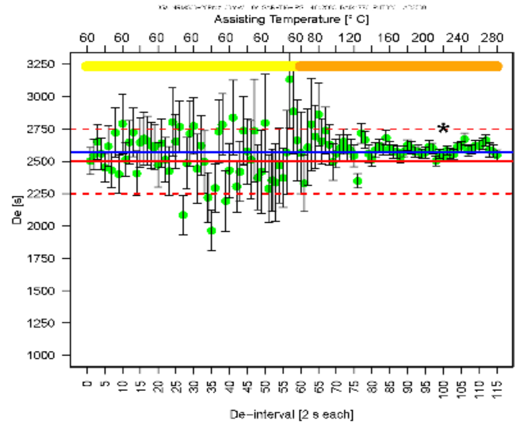
(c) PBL 5 s



(d) PBL 10 s



(e) PBL 30 s



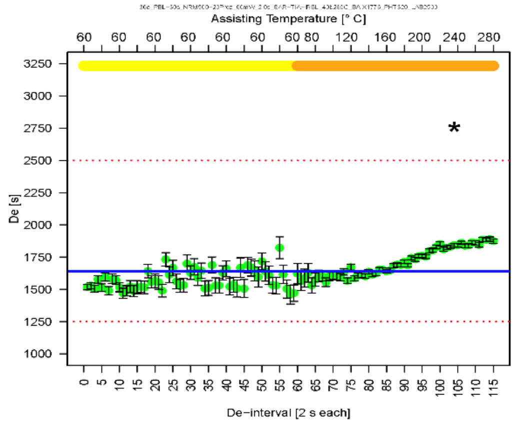
(f) DRT 2500 s – 2572.62 ± 159.01 ± 14.83 s – from PBL-TST MS_34e (PBL 30 s)

Preparation of the PBL sample – from NRM-TST MS_29d

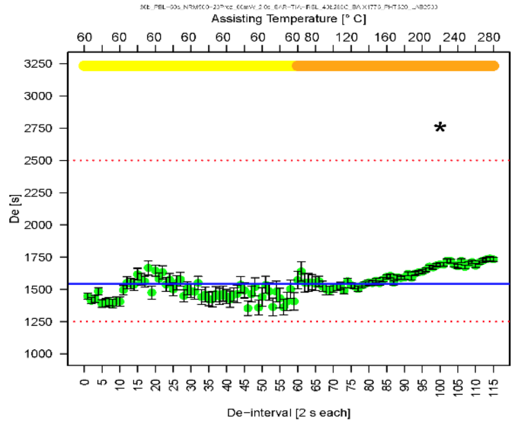
- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (1 s, 3 s, 5 s, 10 s, 30 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

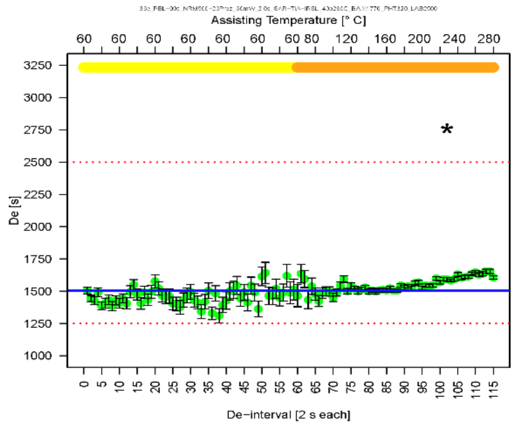
- De value
- Mean of all De values
- Sum of 1st and 2nd dose (1250 s + 1250 s)
- 2nd dose (1250 s)
- 60 °C temperature range
- Temperature increase 60–280 °C
- Position of PET-peak



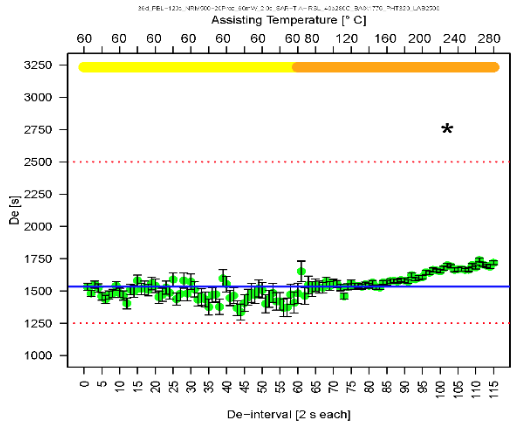
(g) PBL 30 s



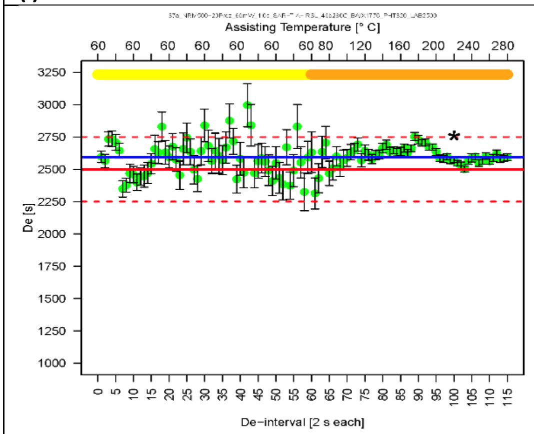
(h) PBL 60 s



(i) PBL 90 s



(j) PBL 120 s



(k) DRT 2500 s – 2592.7 ± 116.4 s – from PBL-TST MS_36d (PBL 120 s)

Preparation of the PBL sample – from De MS_28a

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (30 s, 60 s, 90 s, 120 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

- ● De value
- Mean of all De values
- Sum of 1st and 2nd dose (1250 s + 1250 s)
- 2nd dose (1250 s)
- 60 °C temperature range
- Temperature increase 60–280 °C
- ★ Position of PET-peak

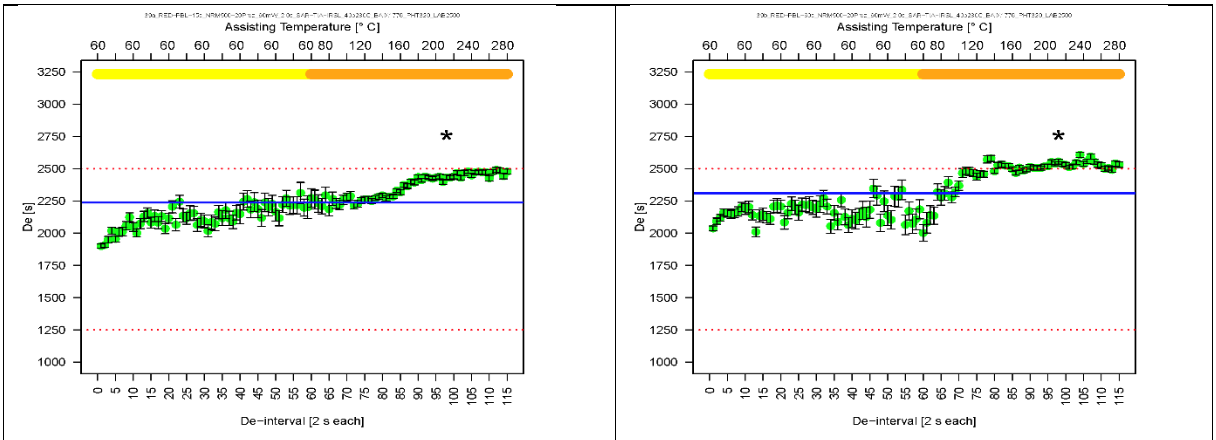
Figure 1.2.1. Tests on artificially partially bleached sample HDS-1776 repeatedly using the aliquot already used in the normalisation dose (NRM) test applying NRM 500 s (20 % of LAB 2500 s) on position **(POS) 69 (Fig. a–e) (PBL 34a–e, DRT 35a)** and in the PET-IRSL SAR measurement on **POS 64 (Fig. g–k) (PBL 36 a–d, DRT 37a)**. After the NRM test and in between the different PBL tests the aliquots were bleached in the lexsyg reader using the solar lamp (365 nm, 10 mW/cm²; 462 nm, 63 mW/cm²; 523 nm, 54 mW/cm²; 590 nm, 37 mW/cm²; 625 nm, 105 mW/cm²; and 850 nm, 90 mW/cm²) for 1 hour at room temperature. The samples received an artificial laboratory dose (LAB) of 1250 s (ca. 152 Gy) and were subsequently preheated for 60 s at 320 °C to simulate longtime storage in the sediment archive. Afterwards the samples were partially

bleached using the same wavelengths and stimulation powers as for the signal resetting in between the different tests. PBL occurred once for 1 s, 3 s, 5 s, 10 s or 30 s **(Fig. a–e) (PBL 34a–e, aliquot on POS 69)** and 30 s, 60 s, 90 or 120 s **(Fig. g–k) (PBL 36 a–d, aliquot on POS 64)**. The situation compares to samples that were only partially bleached during the last cycle of sediment reworking. Afterwards, the sample was irradiated again with 1250 s simulating a respective period of sediment storage. The samples were then measured with the usual PET-IRSL SAR protocol starting with a preheat at 320 °C for 60 s prior to the thermally increasingly assisted IRSL readout. Each PET-SAR halfcycle (both REG and NRM cycles) was finished by a green-light stimulated luminescence (GRSL) hotbleach at 280 °C. De-signal integral length for data analysis 2 s (20 data channels each; 115 integrals altogether; cf. bottom horizontal axis of graphs). Dose response curve fitted with a single exponential fit applying the function analyse_SAR.CWOSL of the R.Luminescence package (developer version 09.24.9000–106; Kreutzer et al. 2024). Each sequence of PBL tests on an aliquot was accomplished by a DRT on that aliquot. The PBL test series are accomplished by a PET-IRSL DRT **(Fig. f, k)**.

- All PBL tests show a significant increase of De values especially for the high-temperature PET-IRSL range
- The test with the shortest PBL length shows an upper plateau underestimating the expected dose by only 3.8 %
- No shift of the high-temperature PET-peak of the „natural“ signal and no strong dip of initial De values
- The aliquot on POS 64 seems not as well bleachable as the aliquot on POS 69. PBL 30 s of POS 64 starts at a higher De level as PBL 30 s of POS 69
- The less well bleachable aliquot usually shows a breach in slope of increasing De values around the PET-peak with a semi-plateau of De values beyond the peak likely pointing to a population of less bleachable traps (cf. also plateau of 34a)
- PBL 120 s on POS 64 shows higher De values than PBL 90 s on the same aliquot
- Both DRTs reproduce the given dose reasonably well, the mean of all Des overestimating the given dose by 2.9 % (POS 69) and 3.7 % (POS 64), respectively.

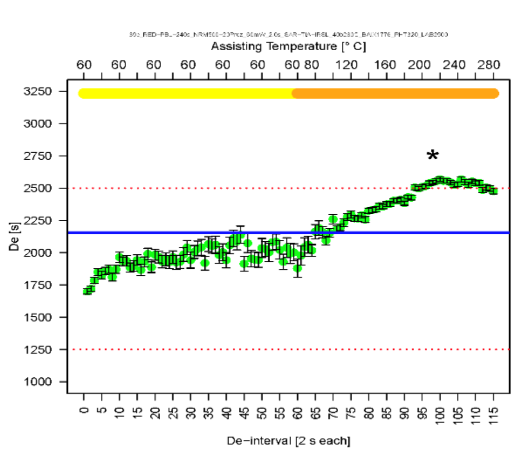
S1.3. Results of the partial bleach (PBL) tests on sample HDS-1776 from the loess-palaeosol section Baix – Variant IRR 1250 s + $TL_{archive}$ + PBL + IRR 1250 s + $TL_{Laboratory}$ – PBL RED-YELLOW-GREEN-BLUE-UV

We performed PET-IRSL SAR measurements on artificially partially bleached aliquots of the loess sample HDS-1776 using repeatedly and already measured aliquots (e.g., from NRM tests or De measurements) applying a 2500 s dose-response curve, NRM 500 s (20 % of LAB 2500 s), PHT 320 °C as well as PET_{max} and HBL 280 °C. After the preceding measurement (used aliquot) and in between the different PBL tests the aliquot was bleached in the lexsyg reader using the LED-based solar simulation unit (365 nm, 10 mW/cm²; 462 nm, 63 mW/cm²; 523 nm, 54 mW/cm²; 590 nm, 37 mW/cm²; 625 nm, 105 mW/cm²; and 850 nm, 90 mW/cm²) for 1 hour at room temperature (representing penultimate event of sediment reworking). The aliquot received an artificial laboratory dose (LAB) of 1250 s (ca. 152 Gy) and was subsequently preheated for 60 s at 320 °C to simulate long-term storage in the sediment archive. Afterwards the sample was partially bleached (representing last event of sediment reworking), using only monochromatic stimulation (same wavelength and stimulation power as for the signal resetting in between the different tests, e.g., 625 nm, 105 mW/cm² for PBL-RED). PBL occurred for 15 s, 60 s, 240 s, 480 s, 960 s and 3600 s. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g., during sunset for RED-PBL. Afterwards, the sample was irradiated again with 1250 s simulating a respective period of sediment storage. The samples were then measured with the usual PET-IRSL SAR protocol starting with a preheat at 320 °C for 60 s prior to the thermally increasingly assisted IRSL readout. Each PET-SAR half-cycle (both REG and NRM cycles) was finished by a green light stimulated luminescence (GRSL) hotbleach (HBL) at 280 °C. The De-signal integral length for data analysis was 2 s (20 data channels each; 115 integrals altogether; cf. bottom horizontal axis of graphs). The dose response curve was fitted with a single exponential fit applying the function analyse_SAR.CWOSL of the R package Luminescence version 1.0.1.9000-19 (Kreutzer et al. 2025)

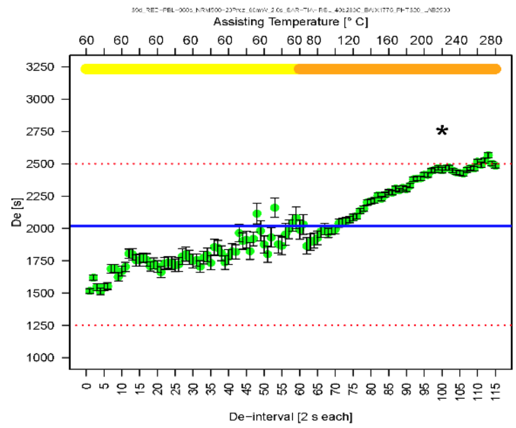


(a) PBL – Red 15 s – 21Jun2024 – no solar bleach*

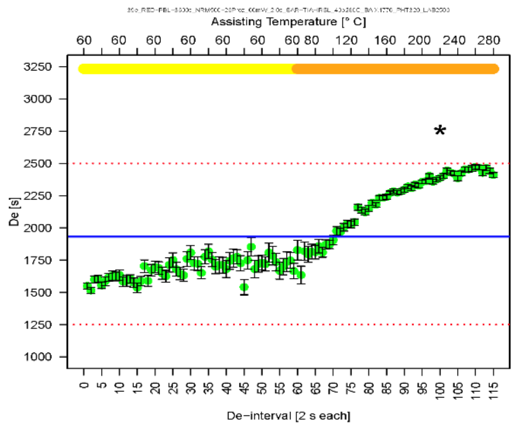
(b) PBL – Red 60 s



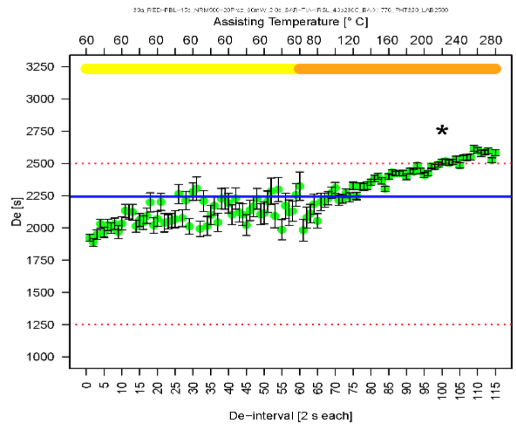
(c) PBL – Red 240 s



(d) PBL – Red 960 s



(e) PBL – Red 3600 s



(f) PBL – Red 15 s – 28Jun2024

Preparation of the PBL sample:

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (15 s, 60 s, 240 s, 960 s, 3600 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

● De value
Mean of all De values
Sum of 1st and 2nd dose (1250 s + 1250 s)
2nd dose (1250 s)
60 °C temperature range
Temperature increase 60–280 °C
★ Position of PET-peak

Figure S1.3.1. PBL-RED MS_39a–39e. Tests on artificially partially bleached sample HDS-1776 using repeatedly an already measured aliquot (here from PET-IRSL SAR De measurement MS_30g). The partial bleaching step was performed using **only the red stimulation** (**625 nm, 105 mW/cm²**) of the solar simulator. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g., during sunset. * The solar bleach step after the previous measurement and before the 15 s PBL-RED was not performed, but the latent luminescence signal had been reset by the PET-IRSL and the subsequent HBL step finalising the preceding De measurement.

- All PET-IRSL measurements show a more or less pronounced upper De-value plateau close to the value of the sum of the two single doses (1250 s + 1250 s = 2500 s)
- While the low-temperature PET-IRSL range De values read out at 60 °C generally become slightly smaller with increasing length of PBL-RED, the high-temperature PET-IRSL range around and beyond the PET-peak proofs hardly bleachable by red light as visible in an upper plateau corresponding to ca 2500 s (penultimate event) **(a–e)** which underestimates the expected value by only ca. 2 % for a PBL length of 3600 s **(e)**.
- Most of the PBL-RED tests show an initial rise of De values (downbend of De values; „nose“). As initial „noses“ were also observed for the shortest PBL tests simulationg the full solar spectrum (PBL-WHITE) with ≤ 10 s bleaching time, but not for those with a longer bleaching time, this feature seems to point to „mild“ bleaching, i.e., either very short or, if longer, with a respectively longer wavelenth.

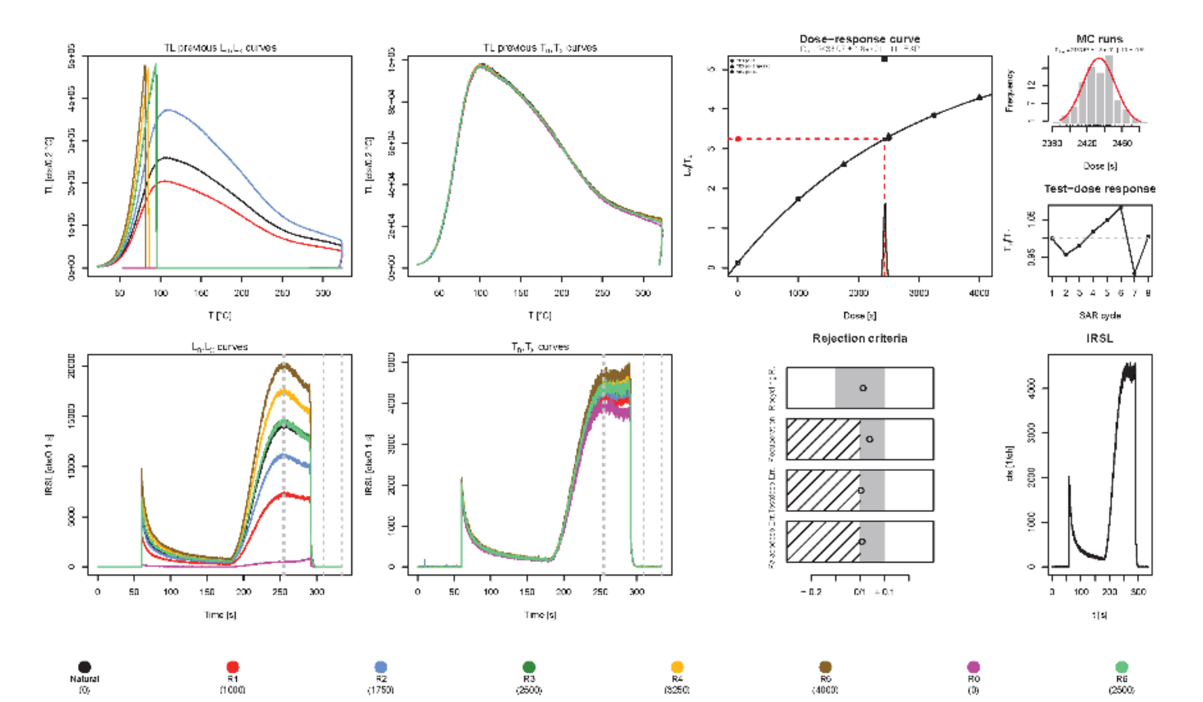
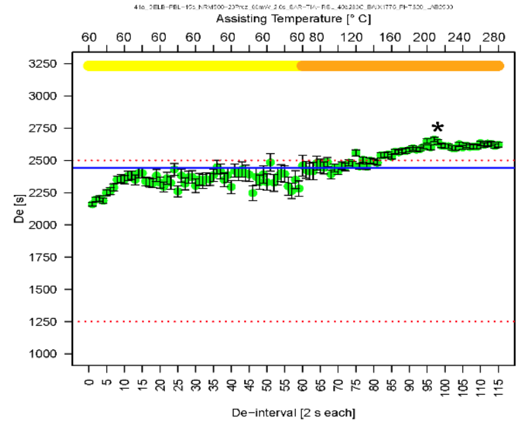
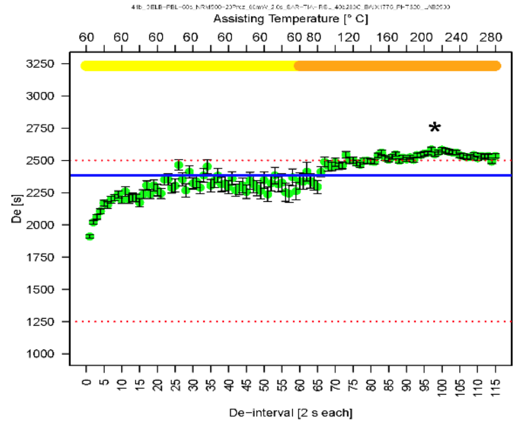


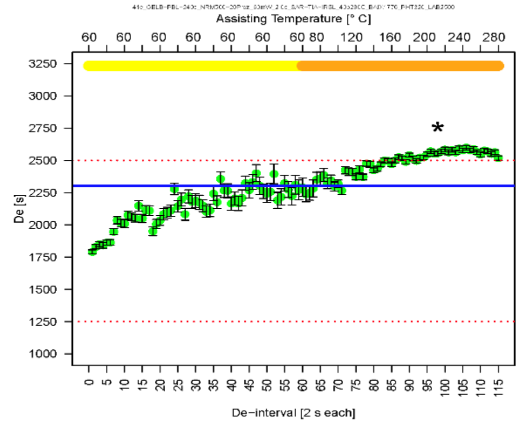
Figure S1.3.2. PBL-RED MS_39a. Graphical output summary of the PET-IRSL SAR analysis (function analyse_SAR.CWOSL of the R package Luminescence; Kreutzer et al. 2025) for De-signal integral 98.



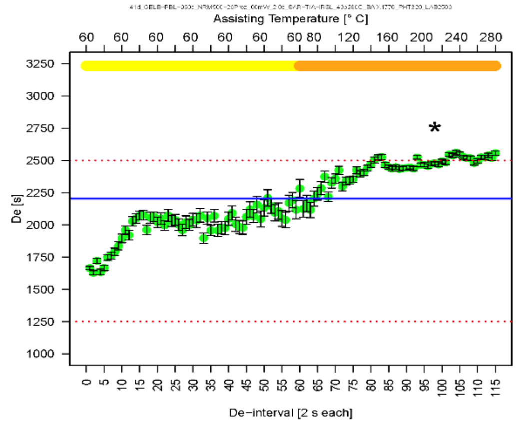
(a) PBL – YELLOW 15 s – 21Jun2024



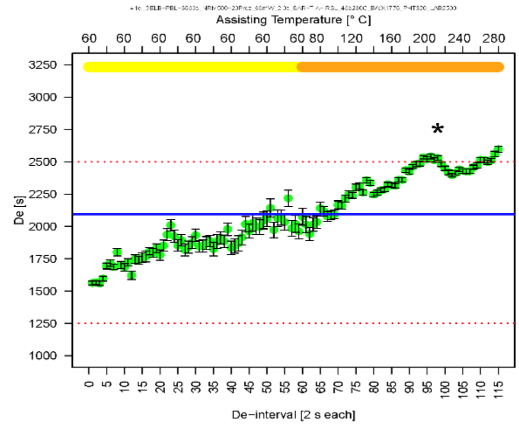
(b) PBL – YELLOW 60 s



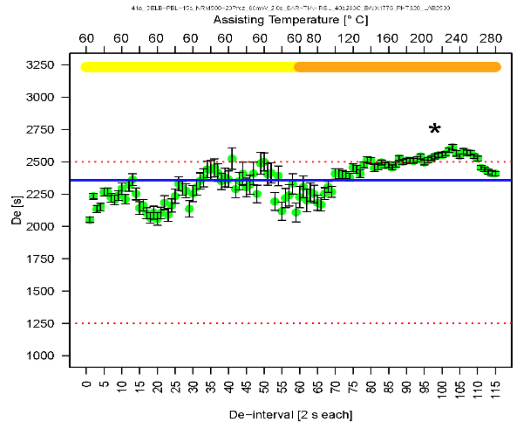
(c) PBL – YELLOW 240 s



(d) PBL – YELLOW 960 s



(e) PBL – YELLOW 3600 s



(f) PBL – YELLOW 15 s – 29Jun2024

Preparation of the PBL sample:

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (15 s, 60 s, 240 s, 960 s, 3600 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

- De value
- Mean of all De values
- Sum of 1st and 2nd dose (1250 s + 1250 s)
- 2nd dose (1250 s)
- 60 °C temperature range
- Temperature increase 60–280 °C
- Position of PET-peak

Figure S1.3.3. PBL-YELLOW MS_41a1–41e/41a2. Tests on artificially partially bleached sample HDS-1776 using repeatedly an already measured aliquot (here from PET-IRSL SAR De measurement MS_30a). The partial bleaching step was performed using **only the yellow stimulation** (**590 nm, 37 mW/cm²**) of the solar simulator. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g. in the late afternoon sunlight, in water or in a turbid suspension.

- For PBL 15 s the largest Des at the high-temperature end of the PET-SAR IRSL measurement overestimate the given dose of 1250 s + 1250 s = 2500 s by ca. 4.6 %. This may be owed to the omitted solar bleaching. However, it is also possible that the aliquot may be less well bleachable as other aliquots and/or aliquots that had not been subjected to PET-IRSL measurements before, like, e.g., the unused aliquots used for the NRM tests. PBL 15 s was repeated after PBL 3600 s. This time the upper De plateau did not show relevant overestimation. However, like the previous PBL 3600 s measurement, the course of the De values was quite wavy. It is possible that this is an effect of repeated measurements on the same aliquot, but such wavy De courses were also observed on natural aliquots.

- The aliquots show strong initial downbends of De values („nose“) before De values form a lower 60 °C plateau. Such noses were also observed for most of the PBL-RED tests and the PBL-WHITE tests with very short bleaching times ≤ 10 s pointing to mild partial bleaching.
- While the high temperature range appears hardly to not bleachable by the yellow stimulation, the 60 °C plateau areas show successive decline while still overestimating the last dose by ca. 62 % for PBL 960 s and 3600 s.

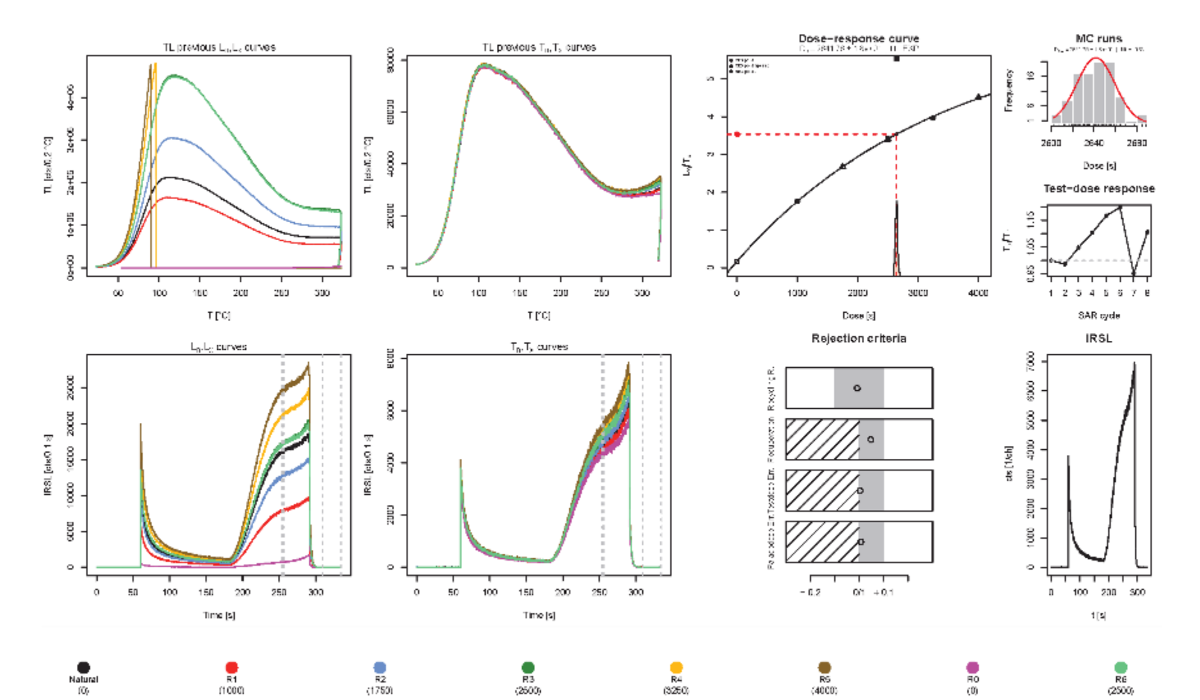
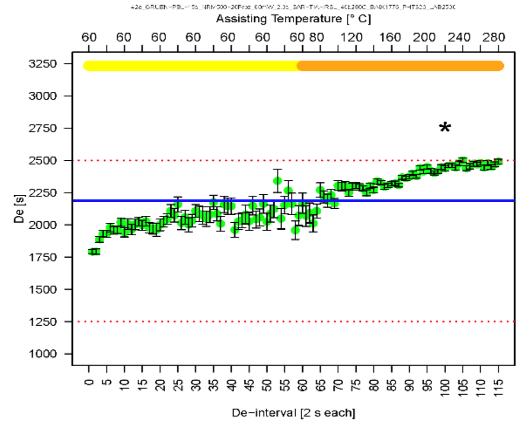
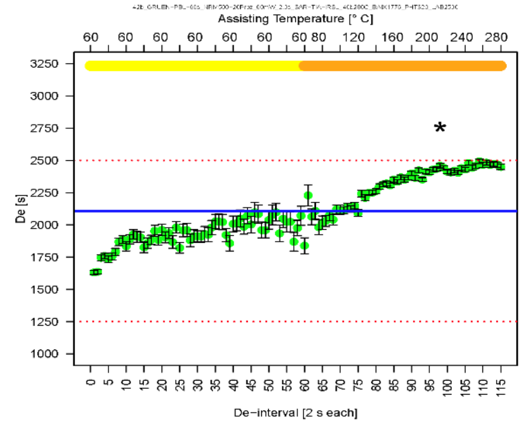


Figure S1.3.4. PBL-YELLOW MS_41a1. Graphical output summary of the PET-IRSL SAR analysis (function analyse_SAR.CWOSL of the R package Luminescence version 1.0.1.9000-19; Kreutzer et al. 2025) for De-signal integral 98.

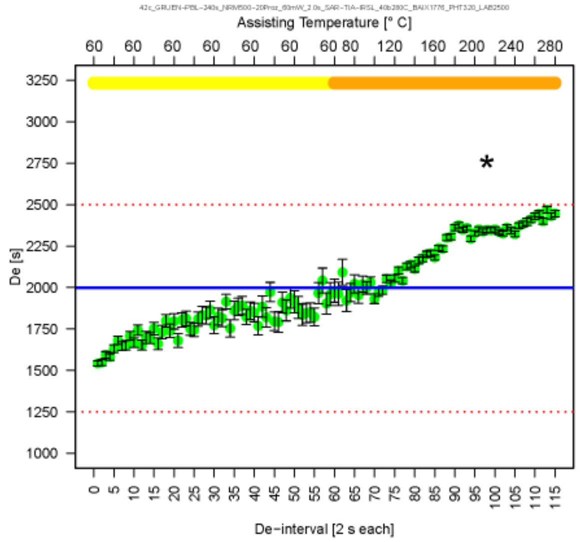
- In contrast to the aliquot used for PBL-RED (cf. **Fig PBL-RED MS_39a**) the aliquot used for PBL-YELLOW seems to possess a further PET-peak beyond 280 °C which impacts the PET-peak at ca 220 °C.



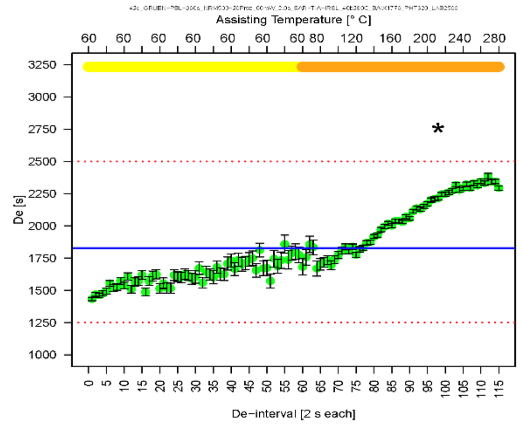
(a) PBL – GREEN 15 s



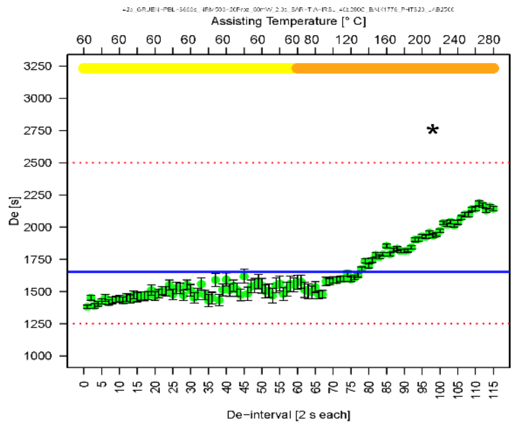
(b) PBL – GREEN 60 s



(c) PBL – GREEN 240 s



(d) PBL – GREEN 960 s



(e) PBL – GREEN 3600 s

Preparation of the PBL sample:

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (15 s, 60 s, 240 s, 960 s, 3600 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

- ● De value
- Mean of all De values
- Sum of 1st and 2nd dose (1250 s + 1250 s)
- 2nd dose (1250 s)
- 60 °C temperature range
- Temperature increase 60–280 °C
- ★ Position of PET-peak

Figure S1.3.5. PBL-GREEN MS_42a–42e. Tests on artificially partially bleached sample HDS-1776 using repeatedly an already measured aliquot (here from PET-IRSL SAR De measurement MS_30d). The partial bleaching step was performed using **only the green stimulation** (**523 nm, 54 mW/cm²**) of the solar simulator. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g. in water or in a turbid suspension.

- The uppermost part of the PET-IRSL De values point to somewhat better bleaching of the high-temperature PET-IRSL range, yet with De plateau ranges only ca. 1.2 %, 2.0 % and 2.5 % below the cumulative dose of both irradiations (1250 s + 1250 s = 2500 s) for 15 s, 60 s and 240 s PBL-GREEN.
- The 60 °C low-temperature range is better bleachable than that of PBL-RED and PBL-YELLOW.
- Nevertheless, the 60 °C plateau-like range overestimates the last dose (event) still by 26 % and 21 % for PBL-GREEN bleaching of 960 s and 3600 s, respectively.

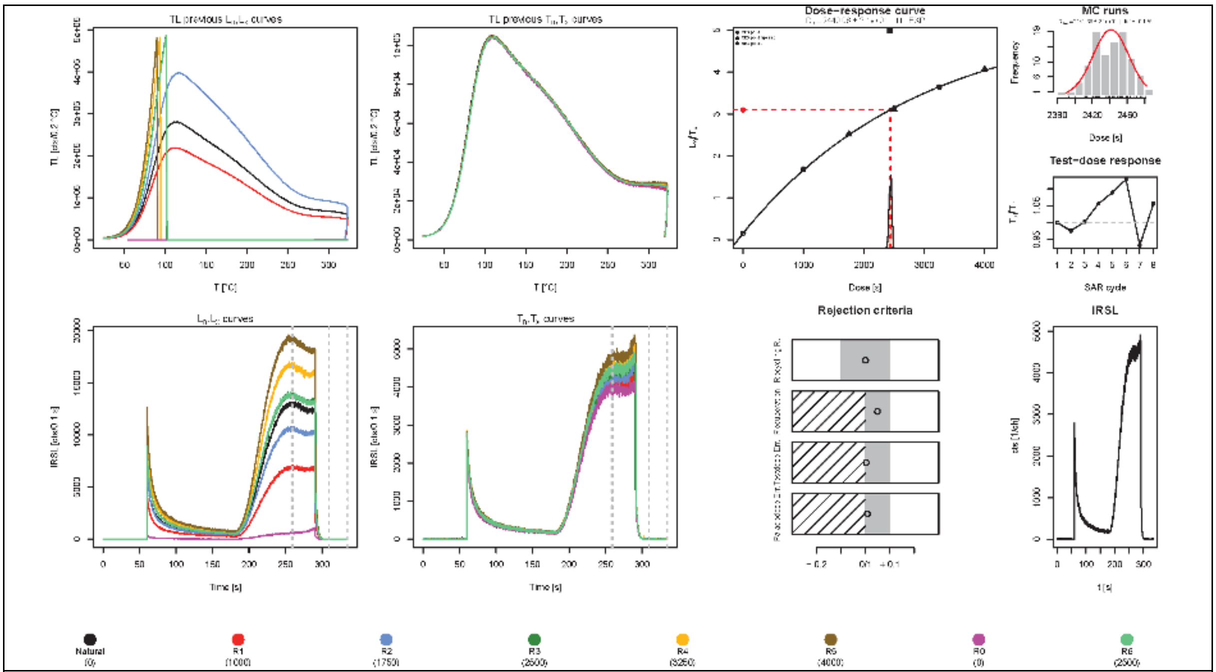
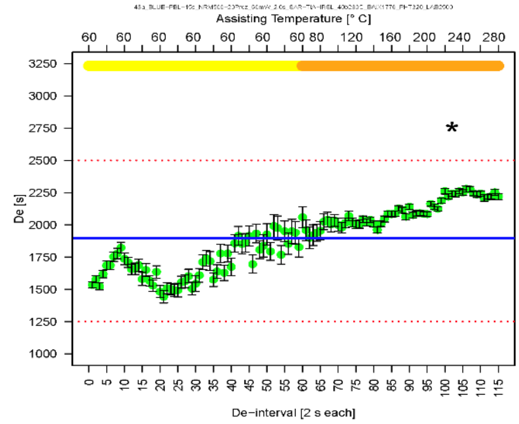
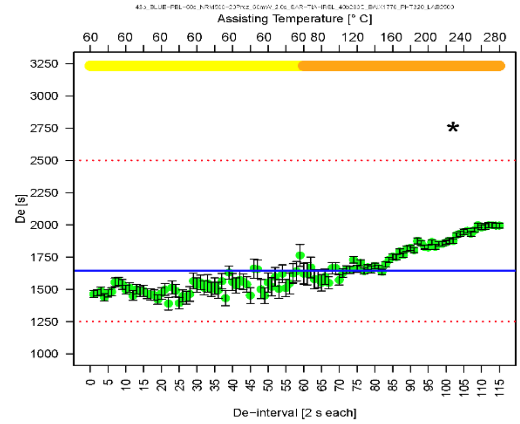


Figure S1.3.6. PBL-GREEN MS_42a. Graphical output summary of the PET-IRSL SAR analysis (function analyse_SAR.CWOSL of the R package Luminescence version 1.0.1.9000-19; Kreutzer et al. 2025) for De-signal integral 100.

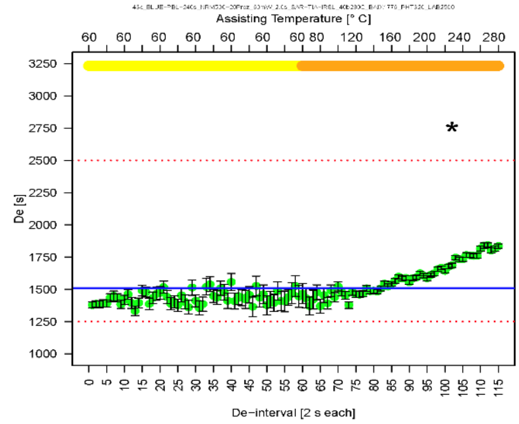
- Compared to the aliquot used for PBL-RED an impact of a further PET-peak beyond 280 °C is noticeable, which however is not as evident as in the aliquot used for PBL-YELLOW.



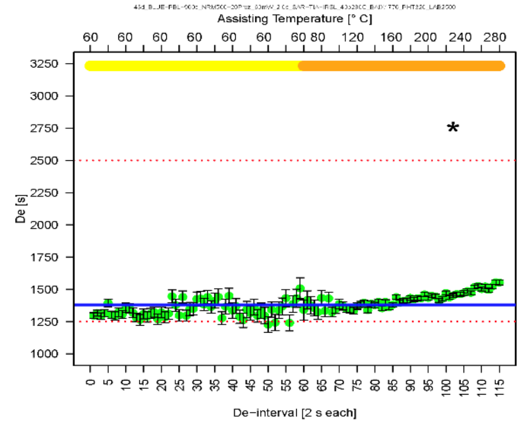
(a) PBL – BLUE 15 s – MS_43a1 – 02Jul2024



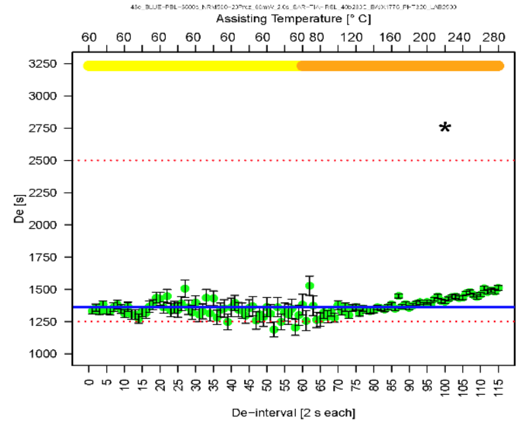
(b) PBL – BLUE 60 s



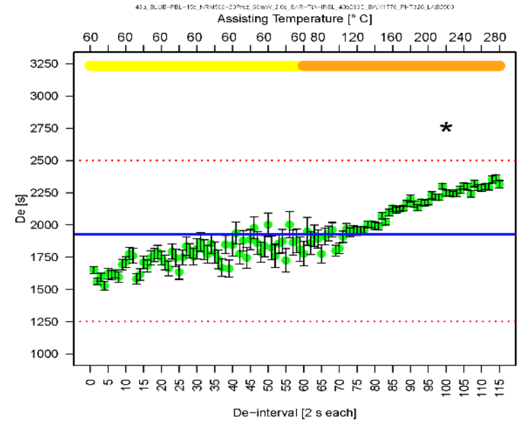
(c) PBL – BLUE 240 s



(d) PBL – BLUE 960 s



(e) PBL – BLUE 3600 s



(f) PBL – BLUE 15 s – MS_43a2 – 08Jul2024

Preparation of the PBL sample:

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (15 s, 60 s, 240 s, 960 s, 3600 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

- De value
- Mean of all De values
- Sum of 1st and 2nd dose (1250 s + 1250 s)
- 2nd dose (1250 s)
- 60 °C temperature range
- Temperature increase 60–280 °C
- ★ Position of PET-peak

Figure S1.3.7. PBL-BLUE MS_43a1–43e/43a2. Tests on artificially partially bleached sample HDS-1776 using repeatedly an already measured aliquot (here from PET-IRSL SAR De measurement MS_30f). The partial bleaching step was performed using **only the blue stimulation** (**462 nm, 63 mW/cm²**) of the solar simulator. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g. in water.

- The 15 s PBL BLUE **(a)** shows an akward bending of the 60 °C low-tempertaure range, similar to the repeat measurement of 15 s PBL-YELLOW in **Fig. S1.3.3 PBL-YELLOW MS_41a1–41e/41a2 (f)**. The PBL-BLUE repeat measurement **(f)** does not show that malign behaviour, but otherwise similar values, around 1850 s in the later 60 °C low-temperature range and with the upper plateau, though not quite as well established as in **(a)**, ca. 8 % (ca. 10 % in the first measurement) underestimation of the cumulative dose.
- An upper De value plateau is present for PBL 15 s and 60 s, but comparably short and underestimating the cumulative dose by ca. 10 % and 20 %, respectively.
- Blue light bleaches comparably efficiently, but the De values in the range 60–170 °C of the PBL 3600 s test still overestimates the last dose by ca. 7.4 %. The rising De values in the high-temperature range indicate that the last bleaching was not complete.

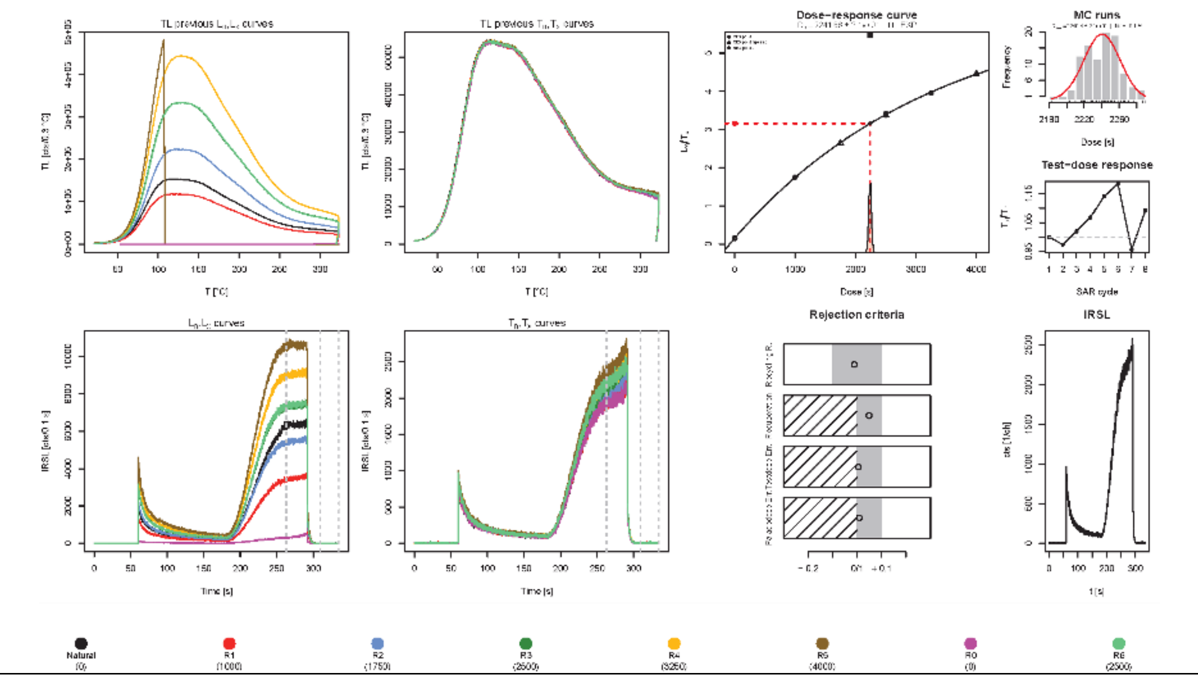
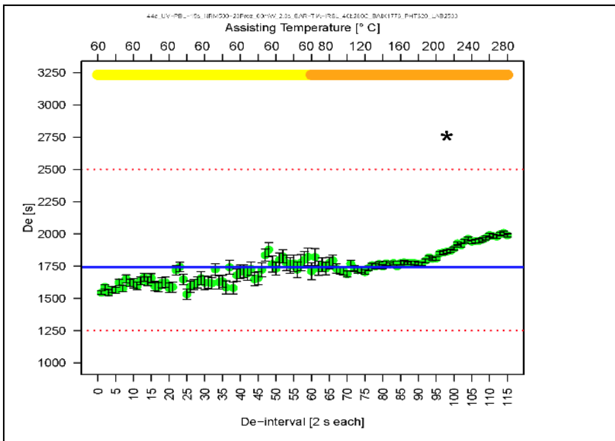
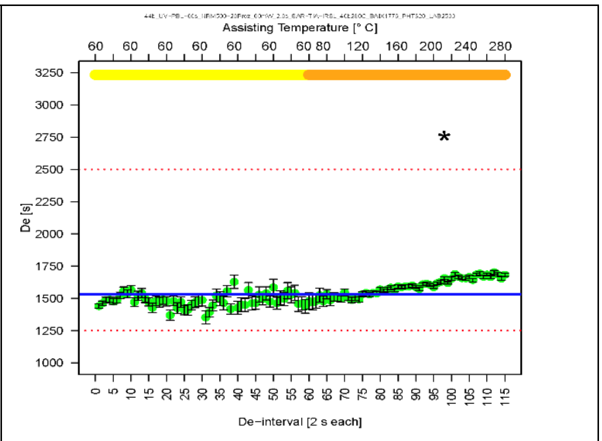


Figure S1.3.8. PBL-BLUE MS_43a1. Graphical output summary of the PET-IRSL SAR analysis (function analyse_SAR.CWOSL of the R package Luminescence version 1.0.1.9000-19; Kreutzer et al. 2025) for De-signal integral 102.

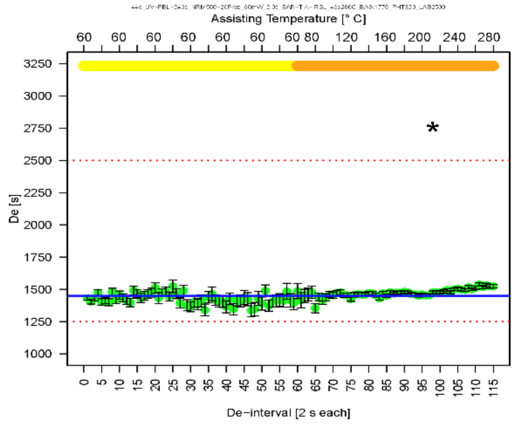
S1.3.1. PBL-tests on HDS-1776 – White-light and monochromatic-light bleaching – unused aliquots



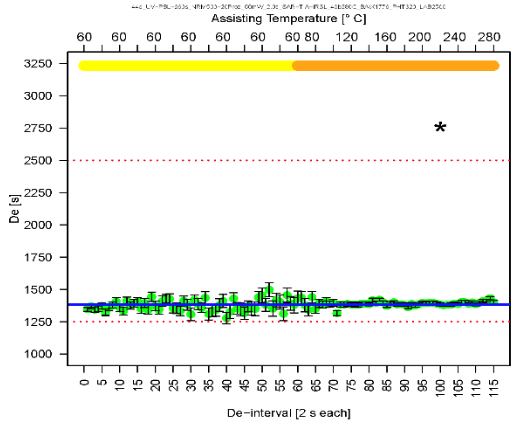
(a) PBL – UV 15 s



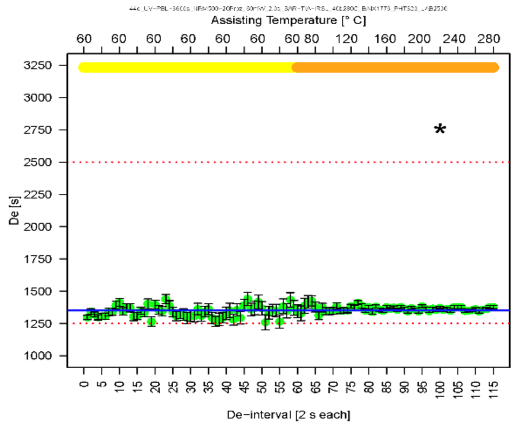
(b) PBL – UV 60 s



(c) PBL – UV 240 s



(d) PBL – UV 960 s



(e) PBL – UV 3600 s

Preparation of the PBL sample:

- IRR 1250 s (ca. 152 Gy)
- $TL_{archive}$ (60 s at 320 °C)
- PBL (15 s, 60 s, 240 s, 960 s, 3600 s)
- IRR 1250 s (ca. 152 Gy)
- $TL_{laboratory}$ (60 s at 320 °C)

Legend

● De value
— Mean of all De values
---- Sum of 1st and 2nd dose (1250 s + 1250 s)
---- 2nd dose (1250 s)
60 °C temperature range
Temperature increase 60–280 °C
★ Position of PET-peak

Figure S1.3.9. PBL-UV MS_44a–e. Tests on artificially partially bleached sample HDS-1776 using repeatedly an already measured aliquot (here from PET-IRSL SAR De measurement MS_30i). The partial bleaching step was performed using **only the blue stimulation (365 nm, 10 mW/cm²)** of the solar simulator. The situation may compare to samples that were only partially bleached during the last cycle of sediment reworking, e.g. in a high-mountain area with a strong UV component.

- UV stimulation bleaches most effectively, both the low- and high-temperature PET-IRSL ranges including the PET-IRSL peak
    - Although the De value data curves of PBL 960 s and 3600 s resemble a plateau, the mean of all 115 De-signal integrals is ca 10.6 % for PBL 960 s and 8 % for PBL 3600 s above the dose of 1250 s representing the last event. The slight overestimation may be owed to the repeated use of the aliquot

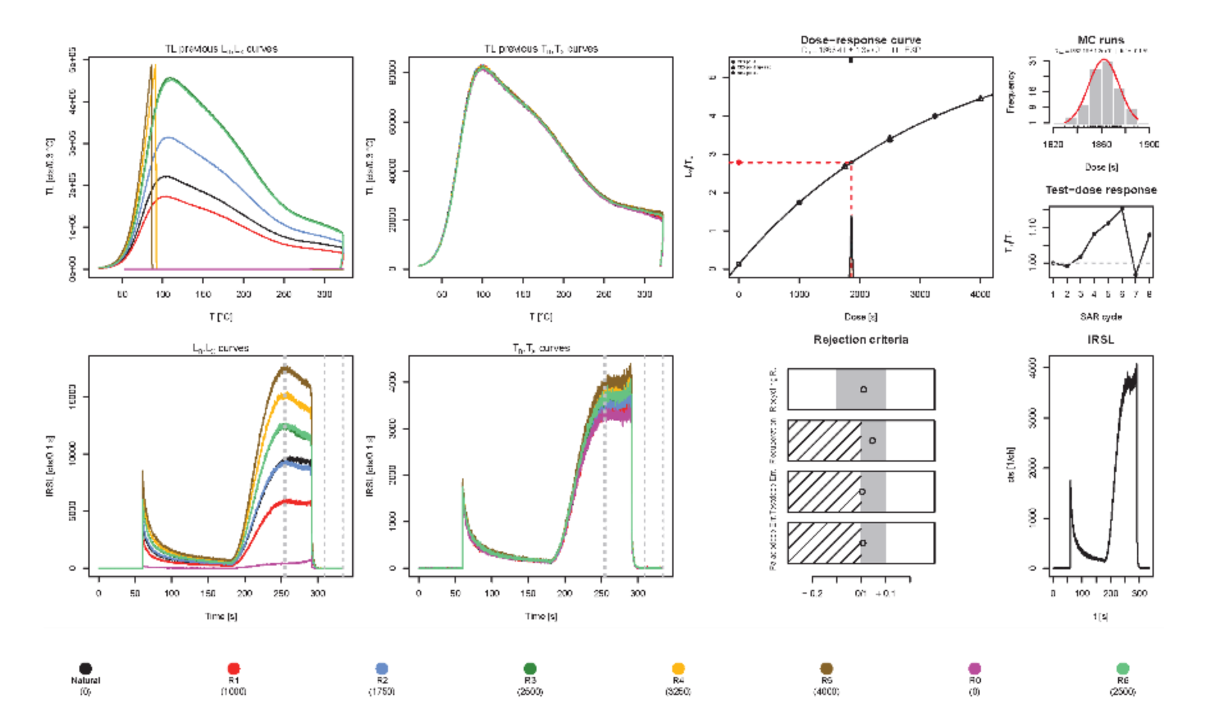
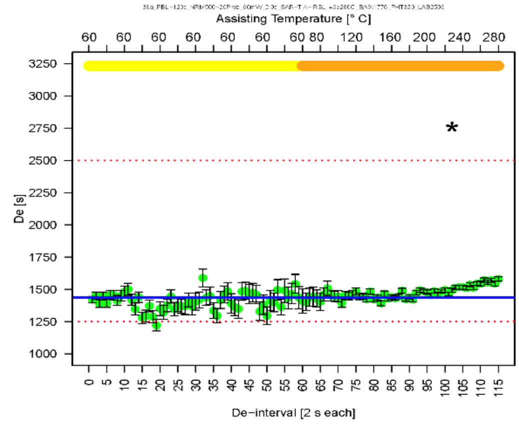
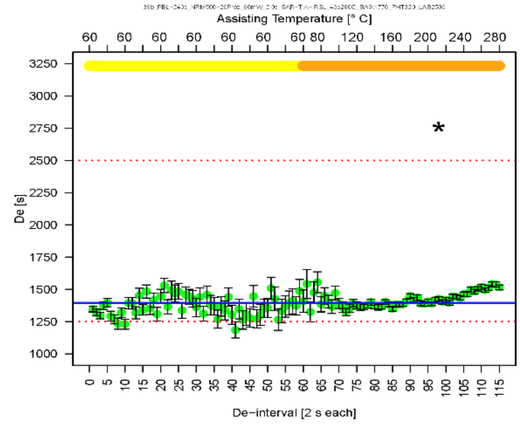


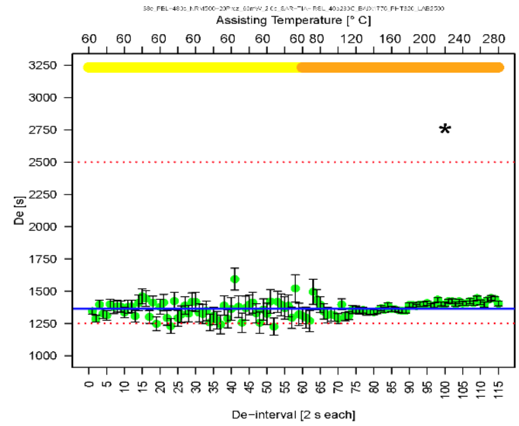
Figure S1.3.10. PBL-UV MS_44a. Graphical output summary of the PET-IRSL SAR analysis (function analyse_SAR.CWOSL of the R package Luminescence version 1.0.1.9000-19; Kreutzer et al. 2025) for De-signal integral 98.



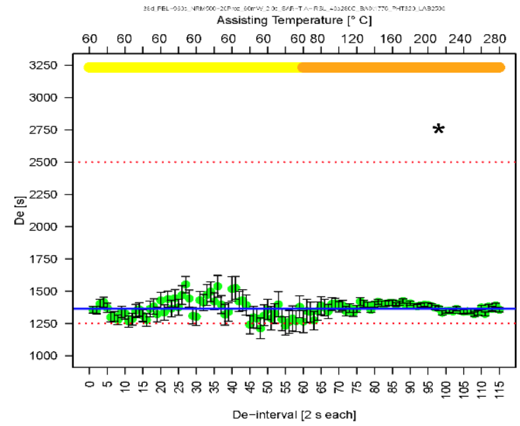
(l) PBL 120 s



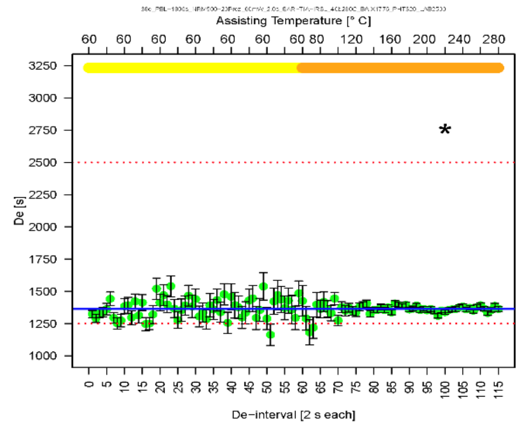
(m) PBL 240 s



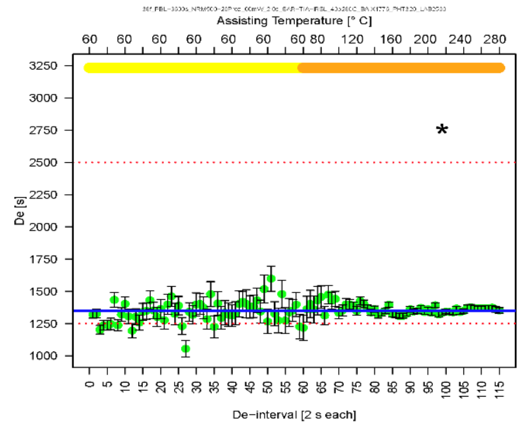
(n) PBL 480 s



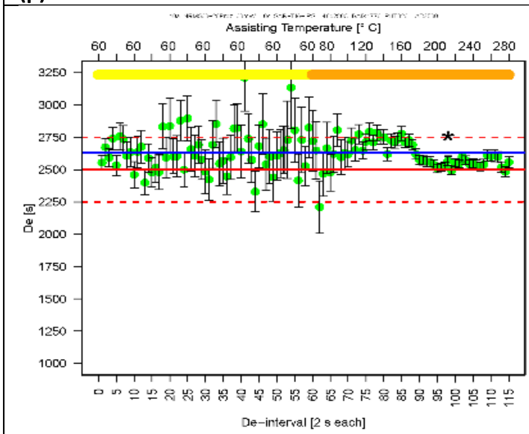
(o) PBL 960 s – 1365.9 ± 61.6 s



(p) PBL 1800 s – 1364.4 ± 64.5 s



(q) PBL 3600 s – 1349.2 ± 70.6 s



<table>
<tr><td>(r) DRT 2500 s – 2633.0 ± 141.3 s – DRT MS_40a – from PBL-TST MS_38f</td><td></td></tr>
<tr><td>Preparation of the PBL sample – from De MS_28b

• IRR 1250 s (ca. 152 Gy)
• $TL_{archive}$ (60 s at 320 °C)
• PBL (120 s, 240 s, 480 s, 960 s, 1800 s, 3600 s)
• IRR 1250 s (ca. 152 Gy)
• $TL_{laboratory}$ (60 s at 320 °C)</td><td>Legend

De value
Mean of all De values
Sum of 1st and 2nd dose (1250 s + 1250 s)
2nd dose (1250 s)
60 °C temperature range
Temperature increase 60–280 °C
★ Position of PET-peak</td></tr>
<tr><td colspan="2">Figure 1.2.2. Tests on artificially partially bleached sample HDS-1776 repeatedly using the aliquot already used in the PET-IRSL SAR measurement 28b applying NRM 500 s (20 % of LAB 2500 s) on position (POS) 65 (l–r) (PBL 38a–f). After the NRM test and in between the different PBL tests the aliquots were bleached in the lexsyg reader using the solar lamp (365 nm, 10 mW/cm²; 462 nm, 63 mW/cm²; 523 nm, 54 mW/cm²; 590 nm, 37 mW/cm²; 625 nm, 105 mW/cm²; and 850 nm, 90 mW/cm²) for 1 hour at room temperature. The samples received an artificial laboratory dose (LAB) of 1250 s (ca. 152 Gy) and were subsequently preheated for 60 s at 320 °C to simulate longtime storage in the sediment archive. Afterwards the samples were partially bleached using the same wavelengths and stimulation powers as for the signal resetting in between the different tests. PBL occurred once for 120 s, 240 s, 480 s, 960 s, 1800 s or 3600 s (PBL 38a–f). The situation compares to samples that were only partially bleached during the last cycle of sediment reworking. Afterwards, the sample was irradiated again with 1250 s simulating a respective period of sediment storage. The samples were then measured with the usual PET-IRSL SAR protocol starting with a preheat at 320 °C for 60 s prior to the thermally increasingly assisted IRSL readout. Each PET-IRSL SAR half-cycle (both REG and NRM cycles) was finished by a green light stimulated luminescence (GRSL) hotbleach at 280 °C. De-signal integral length for data analysis 2 s (20 data channels each; 115 integrals altogether; cf. bottom horizontal axis of graphs). Dose response curve fitted with a single exponential fit applying the function analyse_SAR.CWOSL of the R.Luminescence package (developer version 09.24.9000–106; Kreutzer et al. 2024). Note the magnification of the y-axis as compared to the PBL PET-tests wth PBL ≤120 s. The PBL test series is accomplished by a PET-IRSL DRT (Fig. r).</td></tr>
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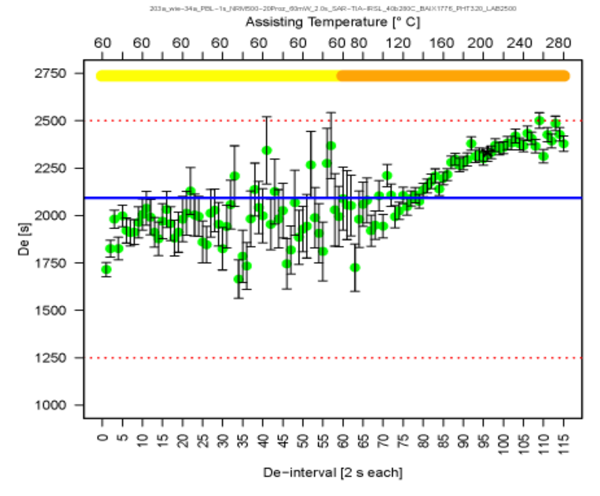
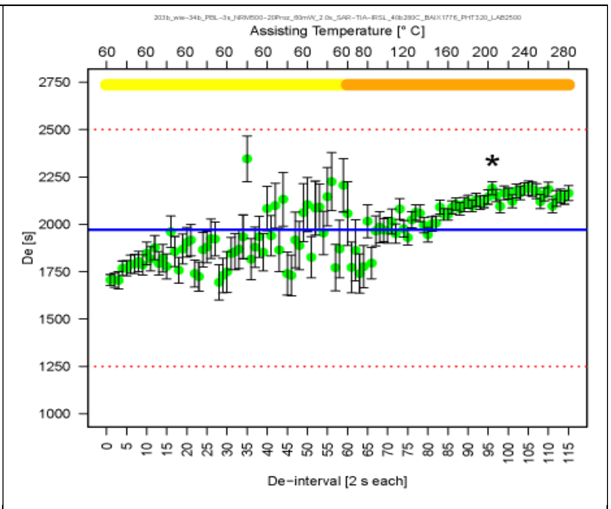
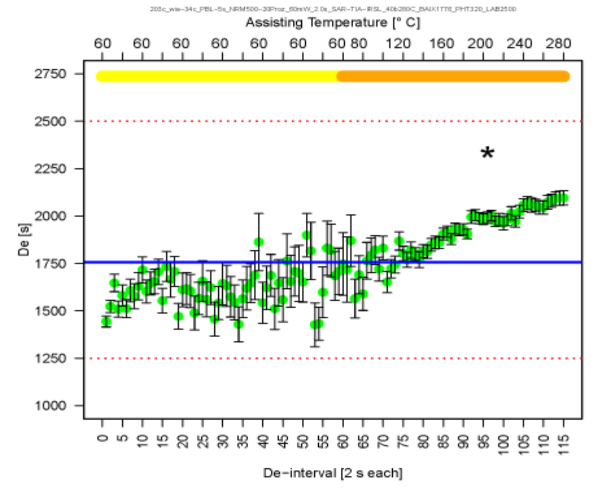
S1.4. PBL-tests on HDS-1776 – White-light and monochromatic-light bleaching – unused aliquots



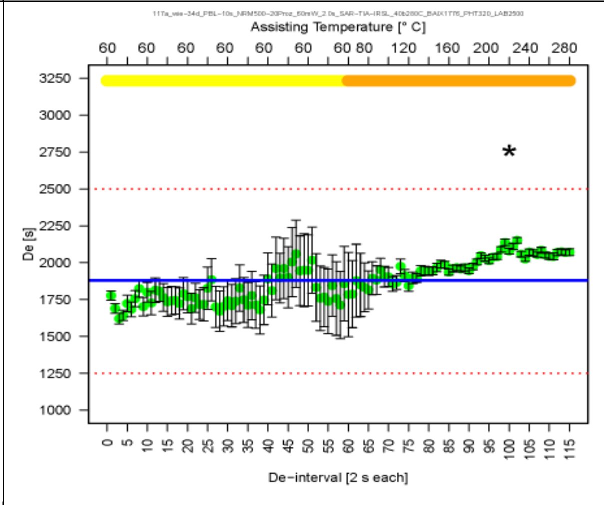
(a) HDS-1776, PBL white, 1 s



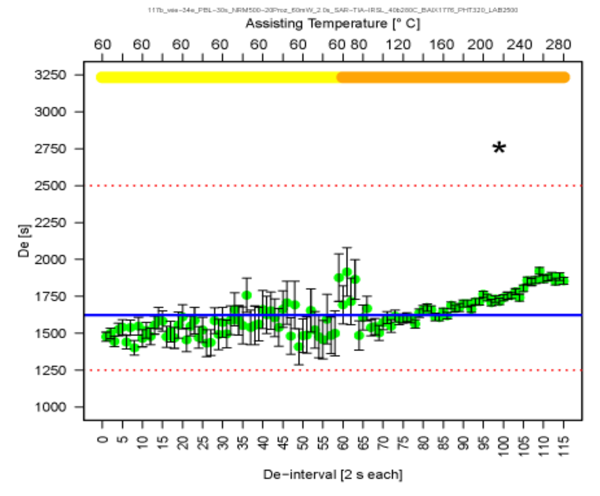
(b) HDS-1776, PBL white, 3 s



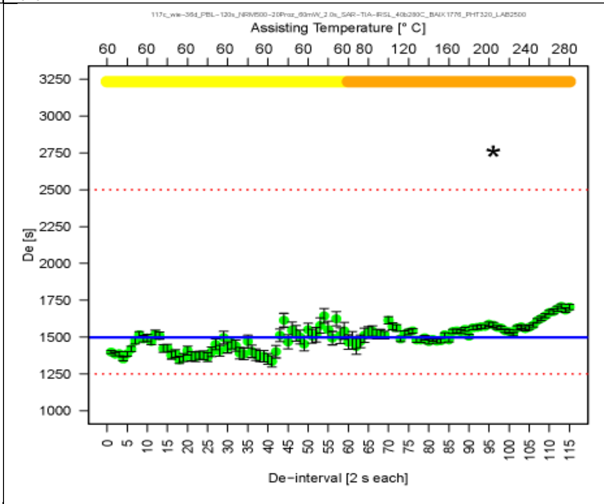
(c) HDS-1776, PBL white, 5 s



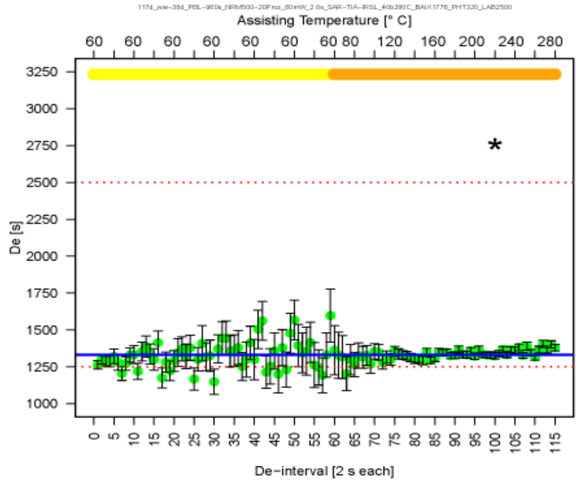
(c) HDS-1776, PBL white, 10 s



(d) HDS-1776, PBL white, 30 s



(e) HDS-1776, PBL white, 120 s



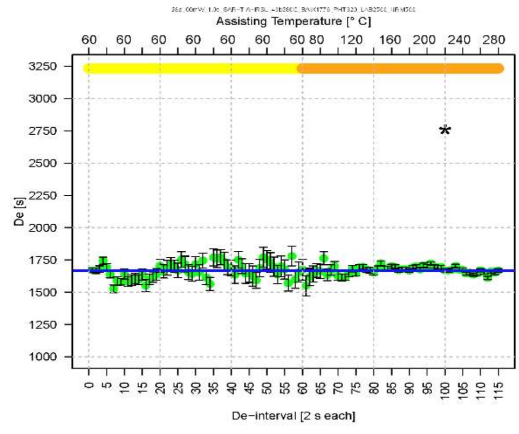
(f) HDS-1776, PBL white, 960 s – 1365.9 ± 61.6 s = 9.3 % > 1250 s

Figure S1.4.1. MS_PBL-White_203a–203c + MS_117a–117d. Results of PBL tests with white-light bleaching on unused ("fresh") aliquots of the loess sample HDS-1776 from the LPS Baix in southern France. Customary measurement parameters: IR 60 mW/cm², GRSL (HDS-1776) hotbleach in each SAR half-cycle. Same dose response curves as used for De determination aiming for HDS-1776 at 2500 s with NRM 20 %. PHT 320 °C, PET_{max} and HBL 280 °C. Blue line: Mean of all De values from integral 1–115 (2 s each). Thin red dotted lines: Administered partial doses, 1250 s after initial thorough signal resetting plus 1250 s after the partial bleaching (2500 s sum of the two partial doses). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C).

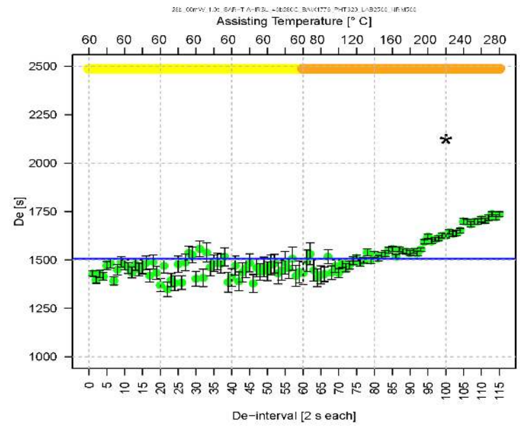
- Results similar to those on repeatedly used aliquots

S1.5. First results of PET-IRSL SAR measurements on sample HDS-1776 from the LPS Baix

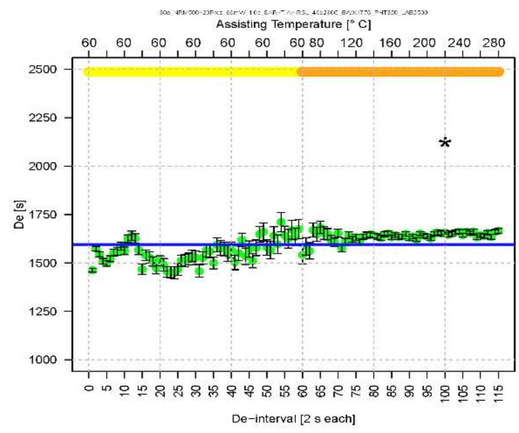
S1.5.1. Measurement series 28a-b, 30a-j and 47a-l



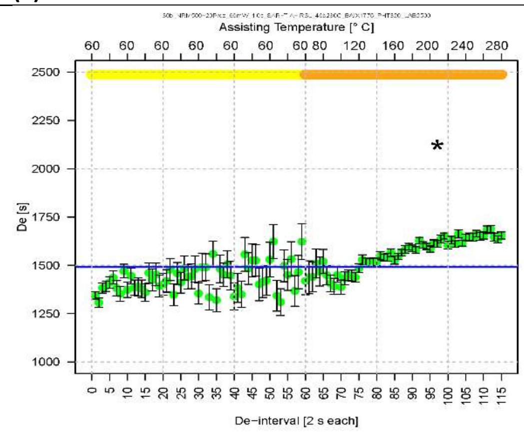
(a) 28a – Plateau ca 1668 s (200.9 ± 5.0 Gy)



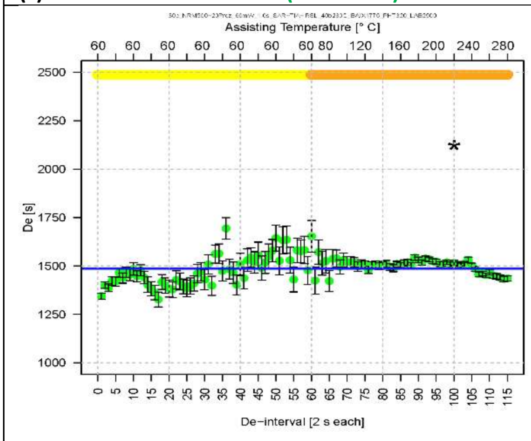
(b) 28b



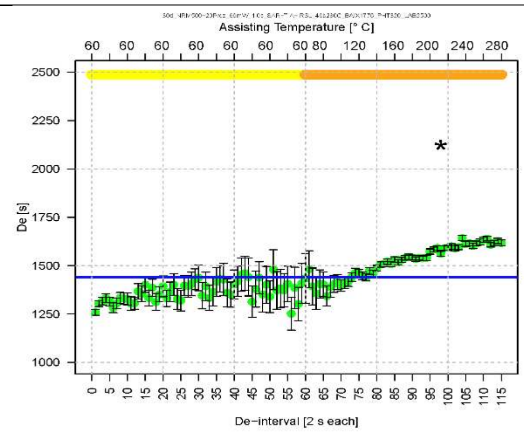
(c) 30a – Plateau ca 1641 s (60–280 °C)



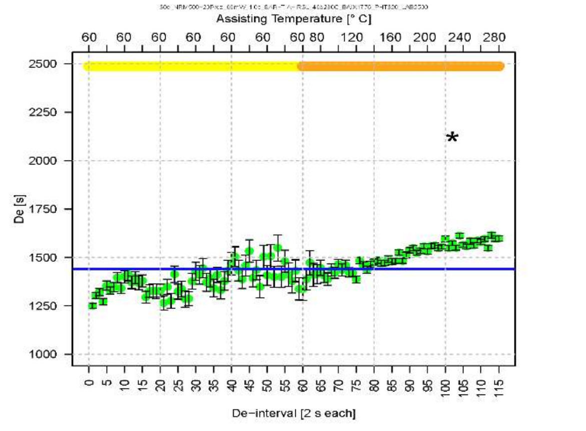
(d) 30b



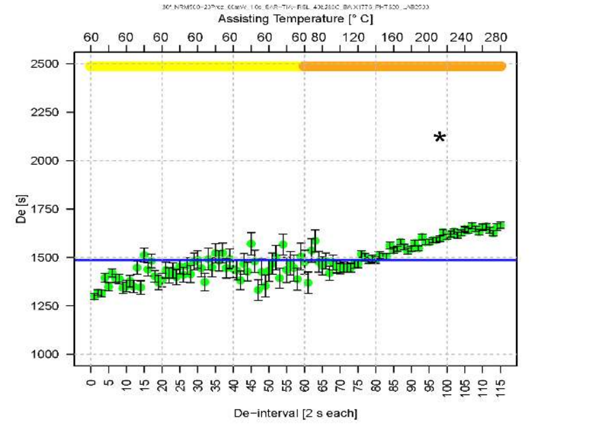
(e) 30c – Plateau ca 1488 s (179. 1 ± 4.4 Gy)



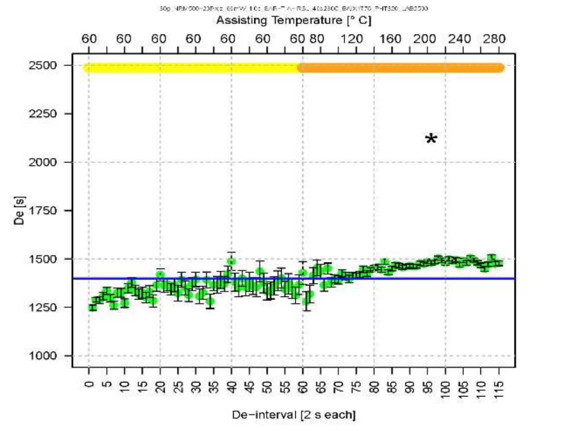
(f) 30d



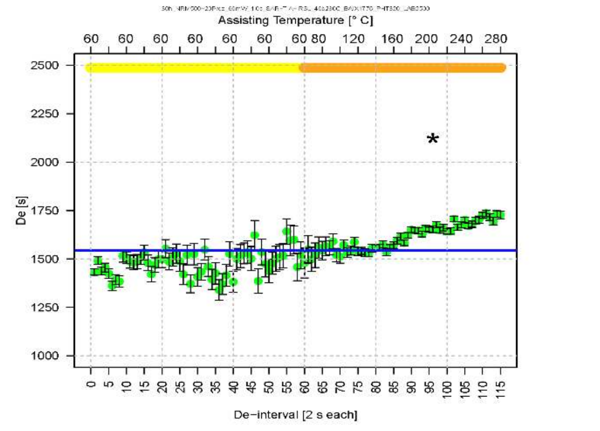
(g) 30e



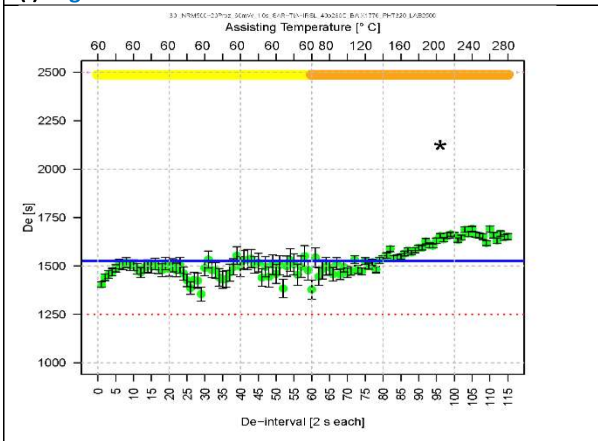
(h) 30f



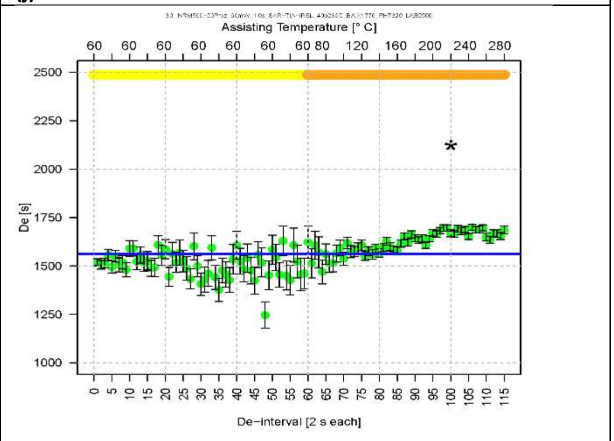
(i) 30g



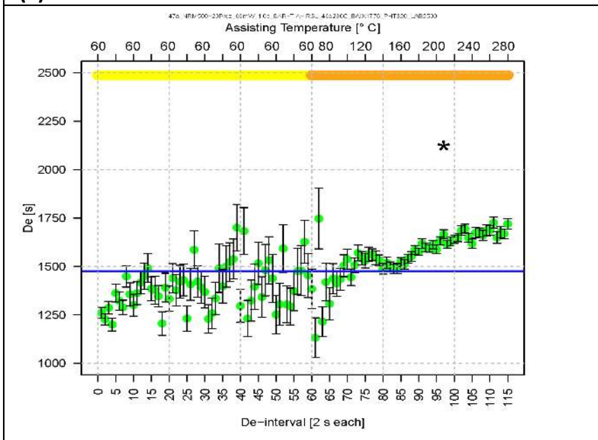
(j) 30h



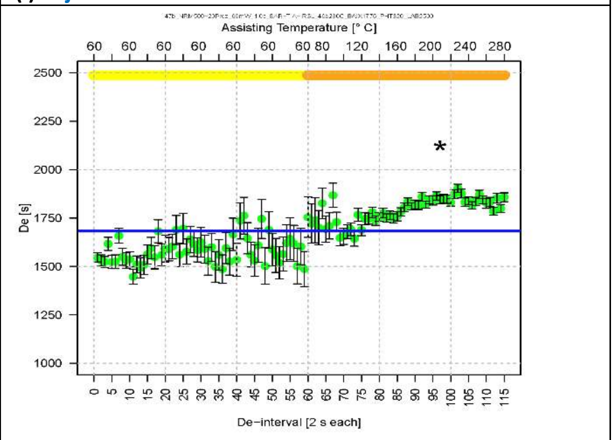
(k) 30i



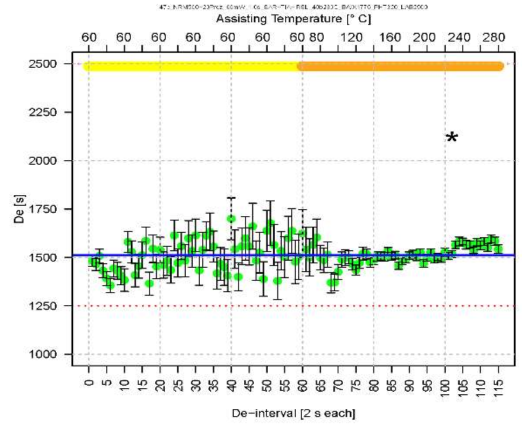
(l) 30j



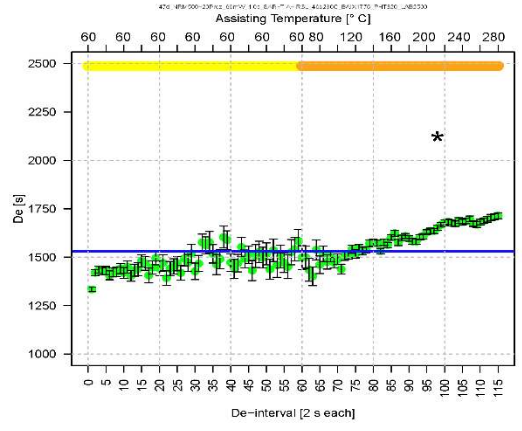
(m) 47a



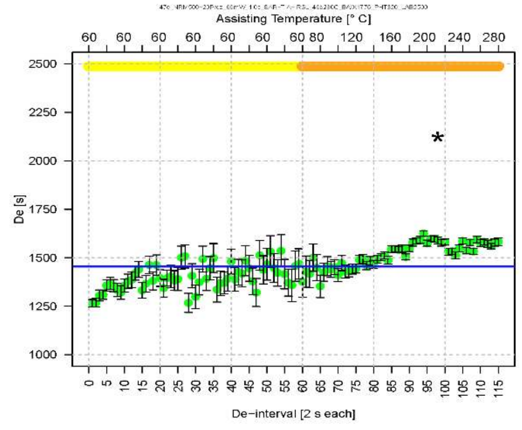
(n) 47b



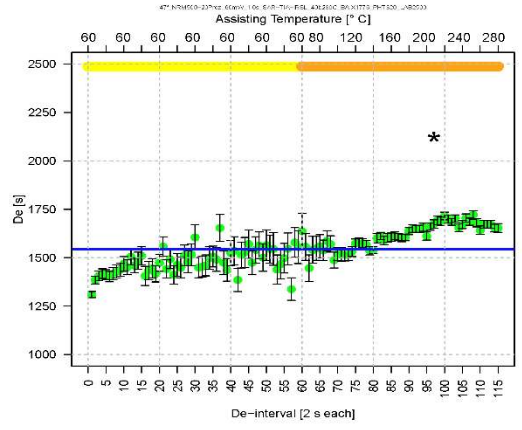
(o) 47c – Plateau ca 1511 s (181. 3 ± 4.5 Gy)



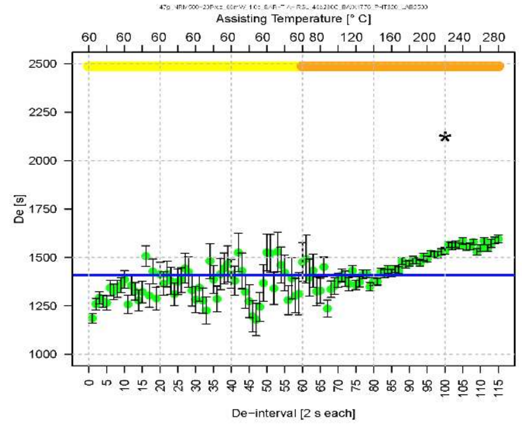
(p) 47d



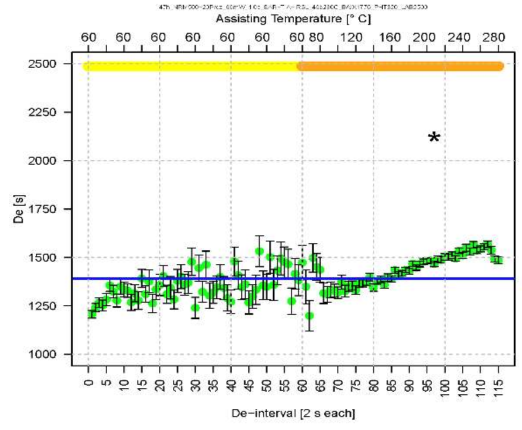
(q) 47e



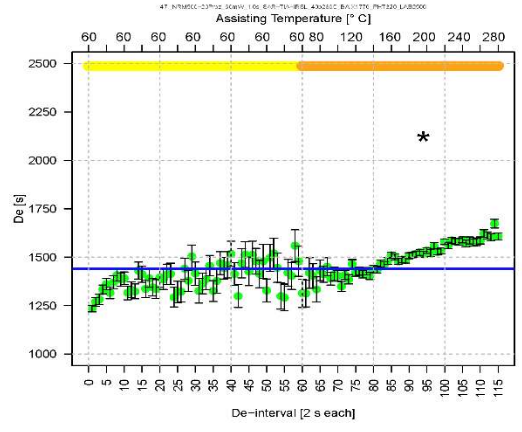
(r) 47f



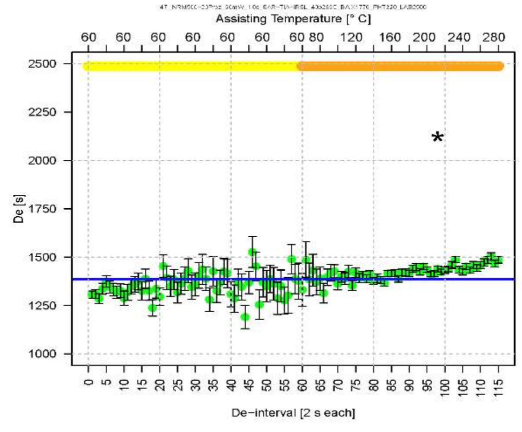
(s) 47g



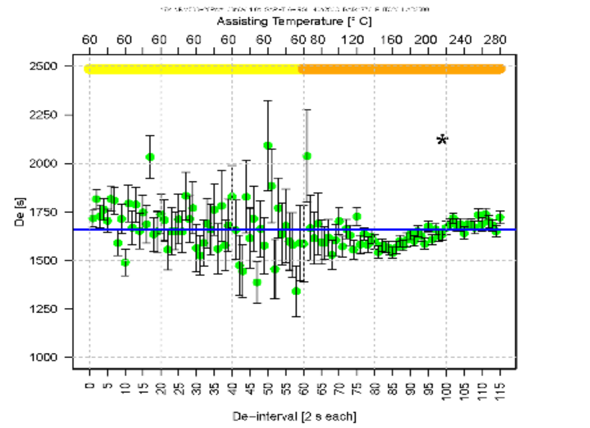
(t) 47h



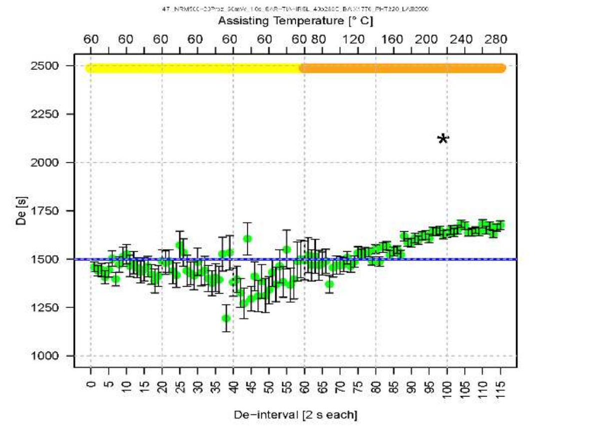
(u) 47i



(v) 47j



(w) 47k – Plateau ca 1659 s (199.1 ± 4.9 Gy)

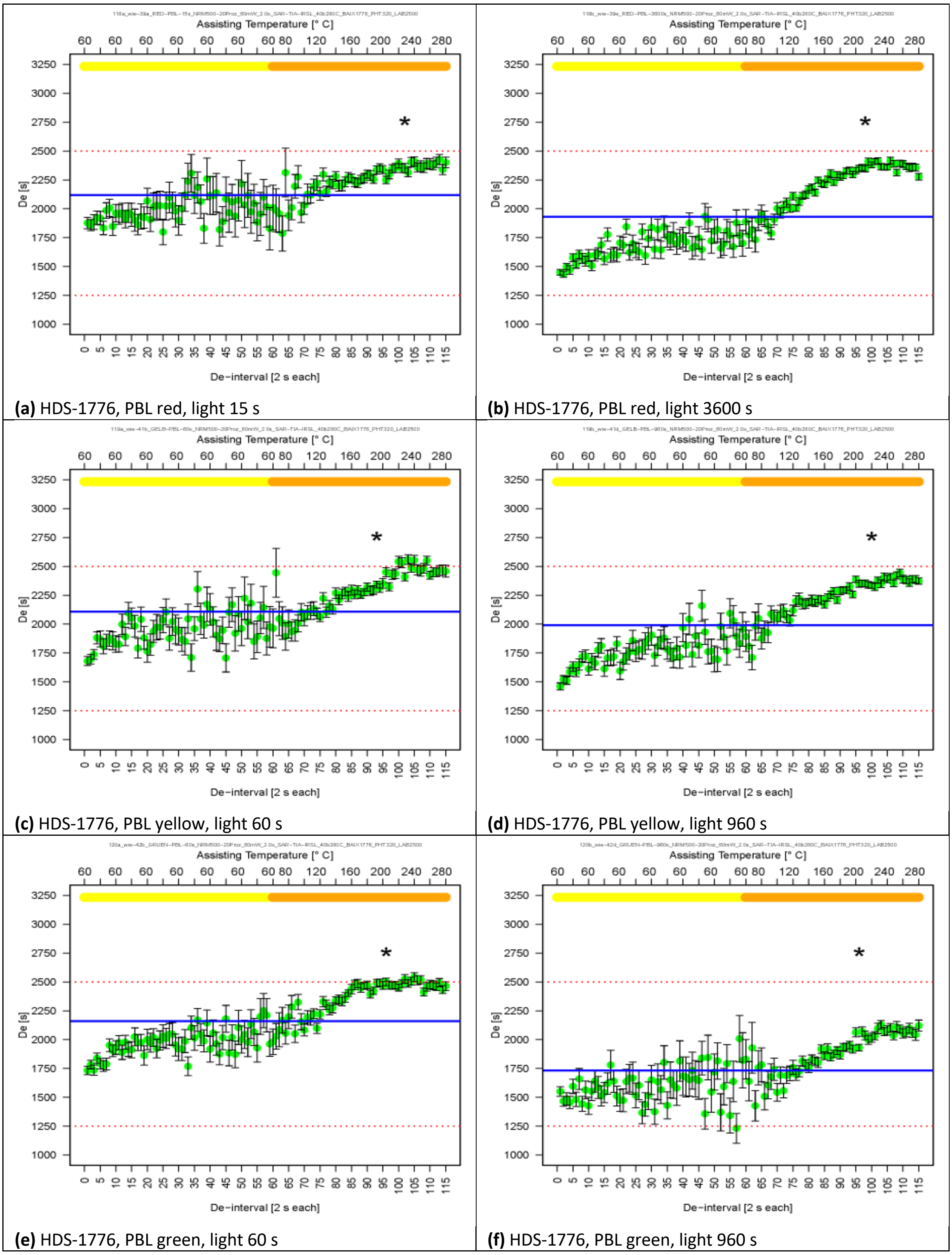


(x) 47l

Legend

- De value
- Mean of all De values
- 60 °C temperature range
- Temperature increase 60–280 °C
- ★ Position of PET-peak

Figure 1.5.1. Tiny aliquots (ø 2 mm) of feldspar coarse grains (125–212 µm) of the sample HDS-1776 from the loess section Baix analysed with the PET-IRSL SAR protocol (dose-response curve aiming at 2500 s, 100 %, ca. 303 Gy; NRM 500 s, 20 %; TL preheat 60 s at 320 °C). PET-IRSL starting with 120 s IR at 60 °C (cf. yellow line along top horizontal axis), followed by continuous temperature increase from 60 °C to 280 °C (cf. orange line along top horizontal axis) within 110 s (ramp 2 °C/s). The total IR-stimulation time is 120 s + 110 s = 230 s (2300 data channels; 0.1 s/data channel). De-signal integral length for data analysis 2 s (20 data channels each; 115 De-signal integrals altogether; cf. bottom horizontal axis of graphs).



(a) HDS-1776, PBL red, light 15 s

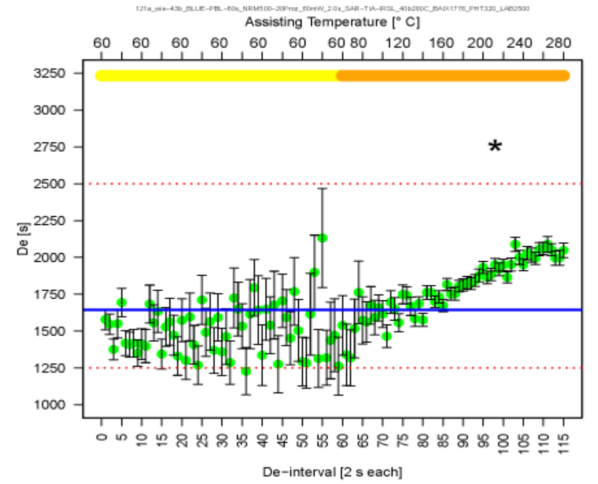
(b) HDS-1776, PBL red, light 3600 s

(c) HDS-1776, PBL yellow, light 60 s

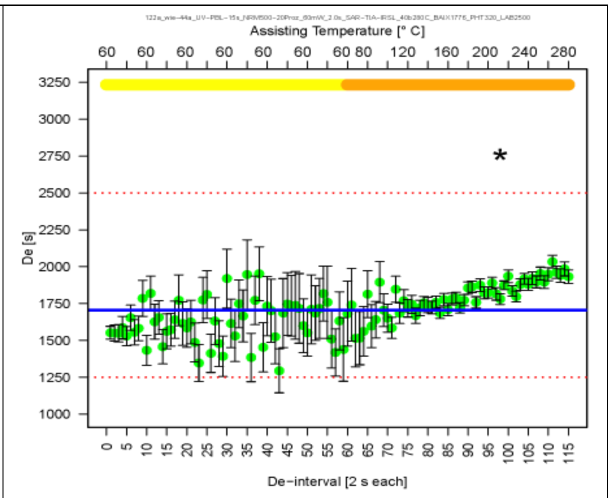
(d) HDS-1776, PBL yellow, light 960 s

(e) HDS-1776, PBL green, light 60 s

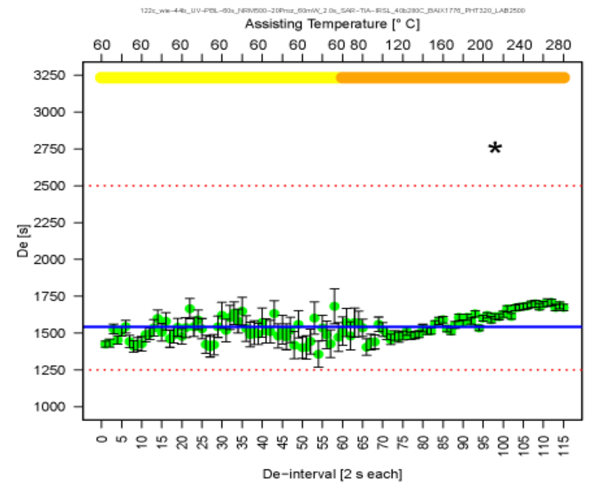
(f) HDS-1776, PBL green, light 960 s



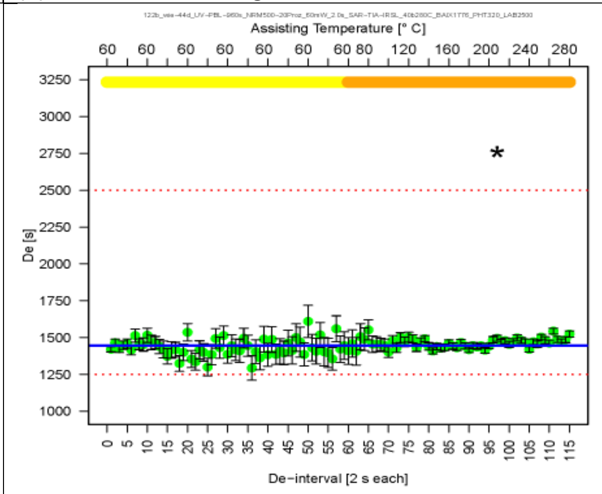
(g) HDS-1776, PBL blue, light 60 s



(h) HDS-1776, PBL UV, light 15 s



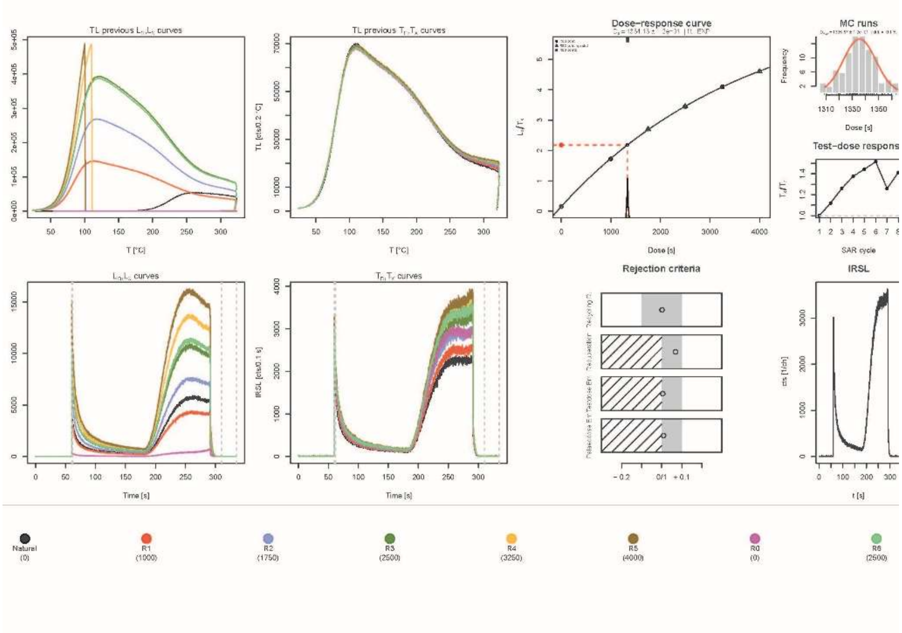
(i) HDS-1776, PBL UV, light 60 s



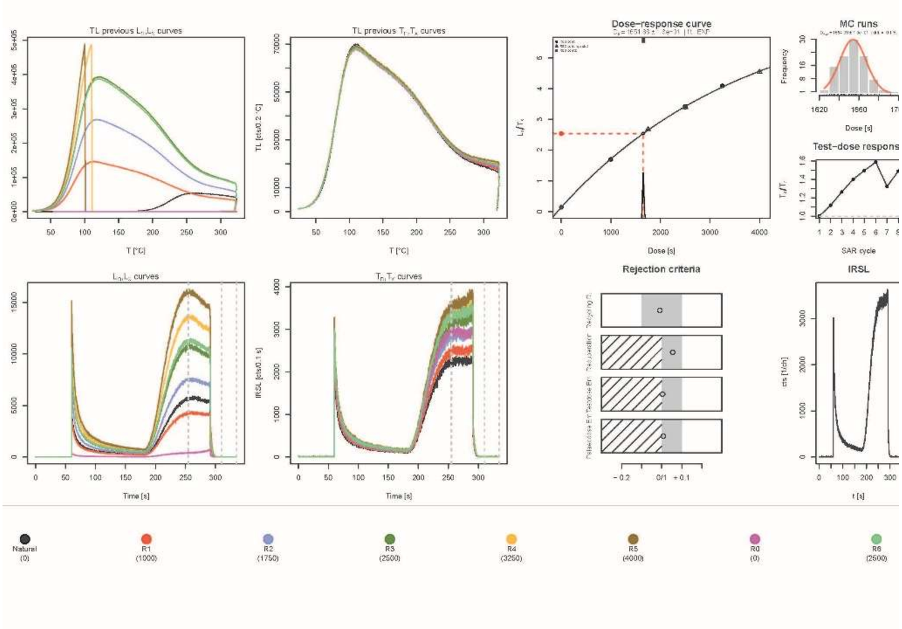
(j) HDS-1776, PBL UV, light 960 s

Figure S1.4.2. MS_PBL_RED-118a-b_Gelb-119a-b_Green-120a-b_Blue-121a_UV_122a-c-b. Results of PBL tests with monochromatic-light bleaching on unused ("fresh") aliquots of the loess sample HDS-1776 from the LPS Baix in southern France. Customary measurement parameters: IR 60 mW/cm², GRSL (HDS-1776) hotbleach in each SAR half-cycle. Same dose response curves as used for De determination aiming for HDS-1776 at 2500 s with NRM 20 %. PHT 320 °C, PET_{max} and HBL 280 °C. Blue line: Mean of all De values from integral 1–115 (2 s each). Thin red dotted lines: Administered partial doses, 1250 s after initial thorough signal resetting plus 1250 s after the partial bleaching (2500 s, sum of two doses). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C).

S1.6. De-determination on HDS-1776 – Examples of PET-IRSL signal curves



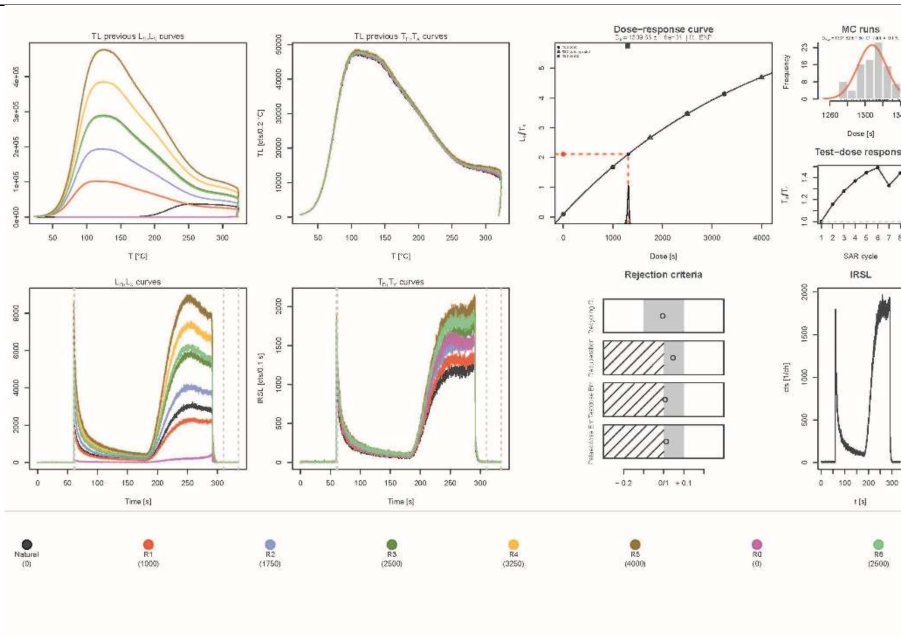
(a)



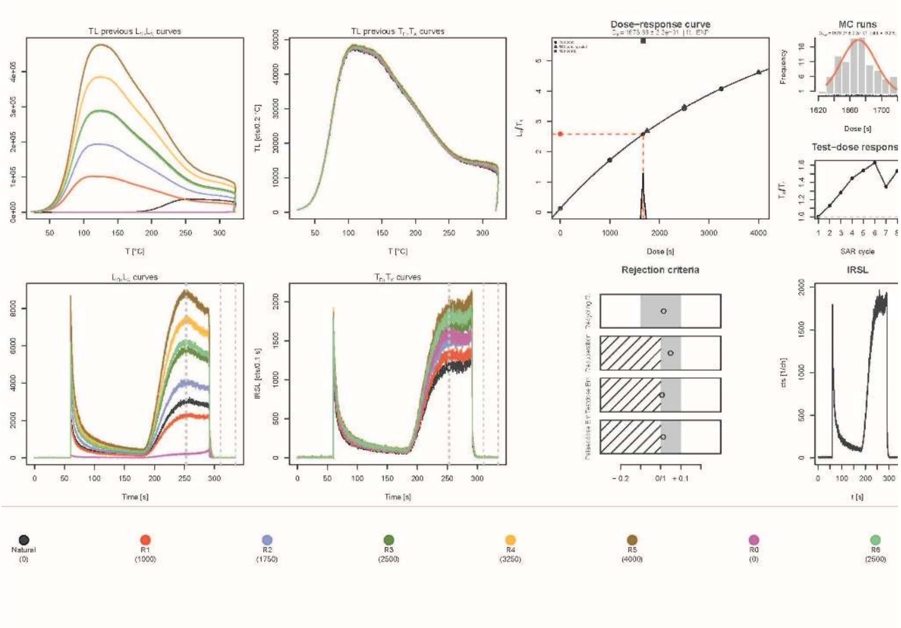
(b)

Figure S1.6.1. MS_47d. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 98 (PET-peak).

47d_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_1,98



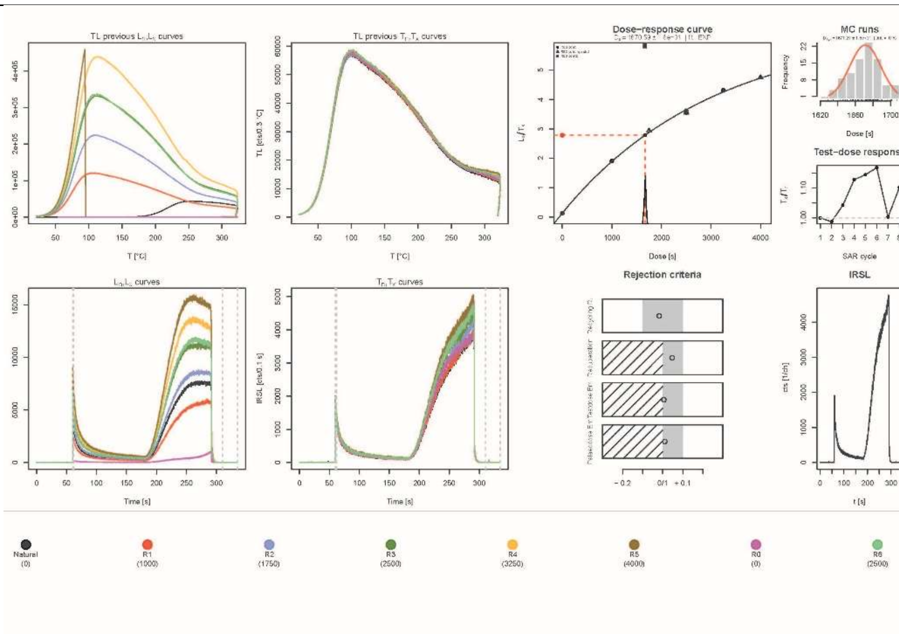
(a)



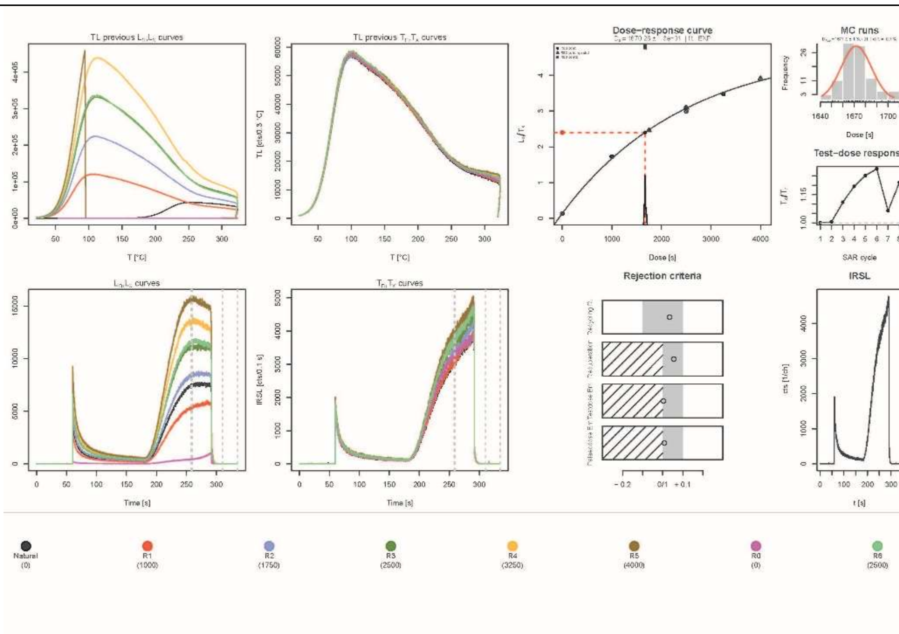
(b)

Figure S1.6.2. MS_47f. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 97 (PET-peak).

47f_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_1,97



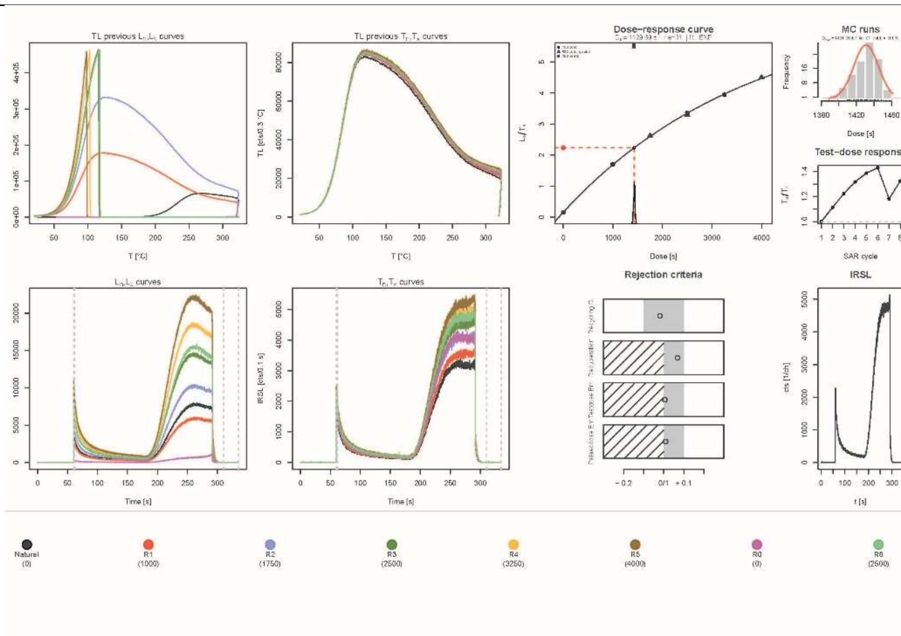
(a)



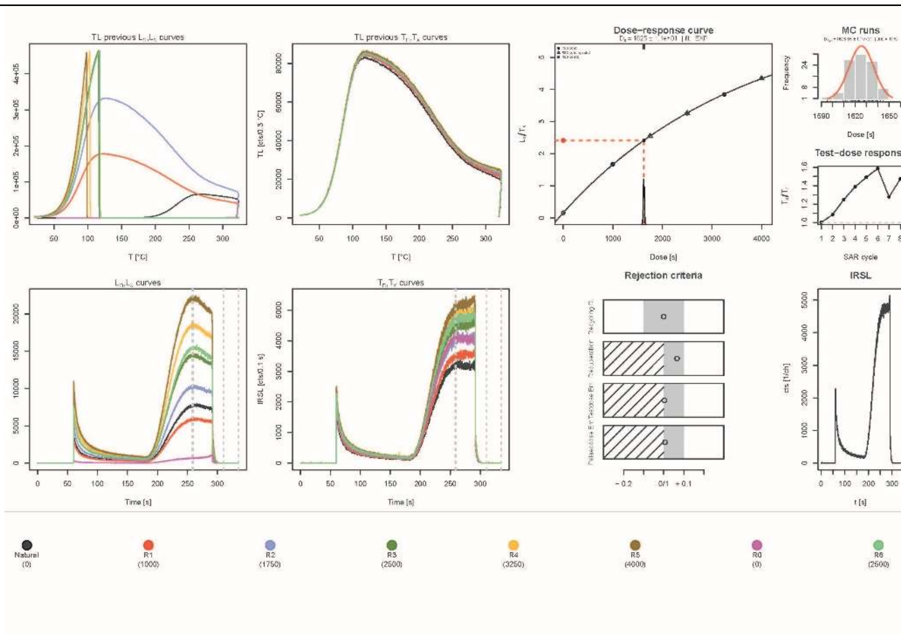
(b)

Figure S1.6.3. MS_28a. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 100 (PET-peak).

28a_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_NRM500_1,100



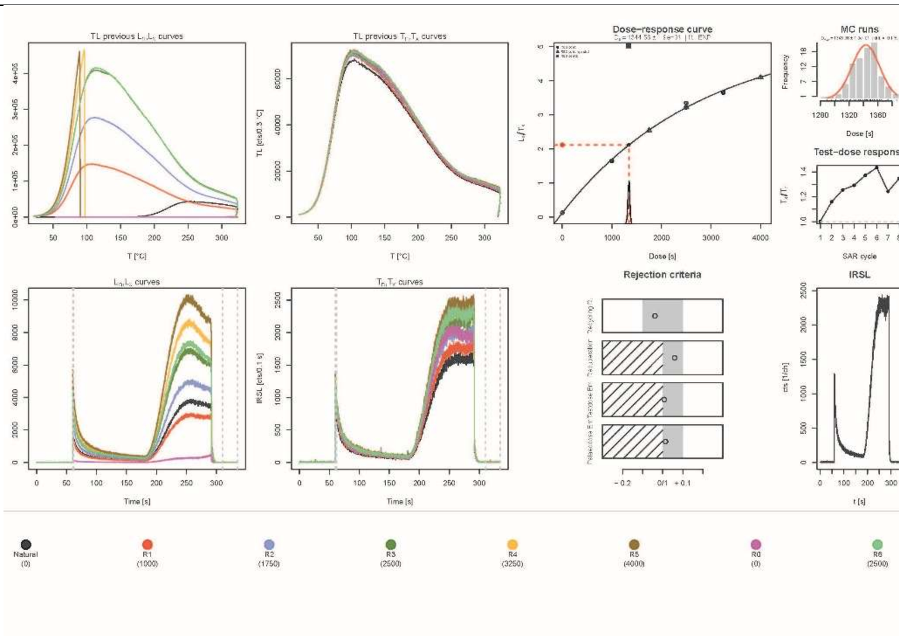
(a)



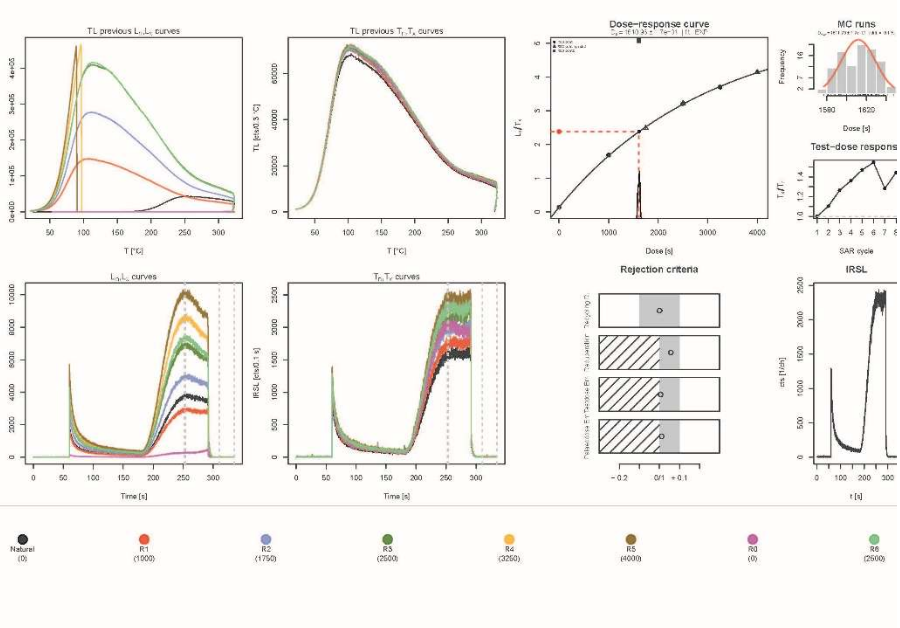
(b)

Figure S1.6.4. MS_28b. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 100 (PET-peak).

28b_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_NRM500_1,100



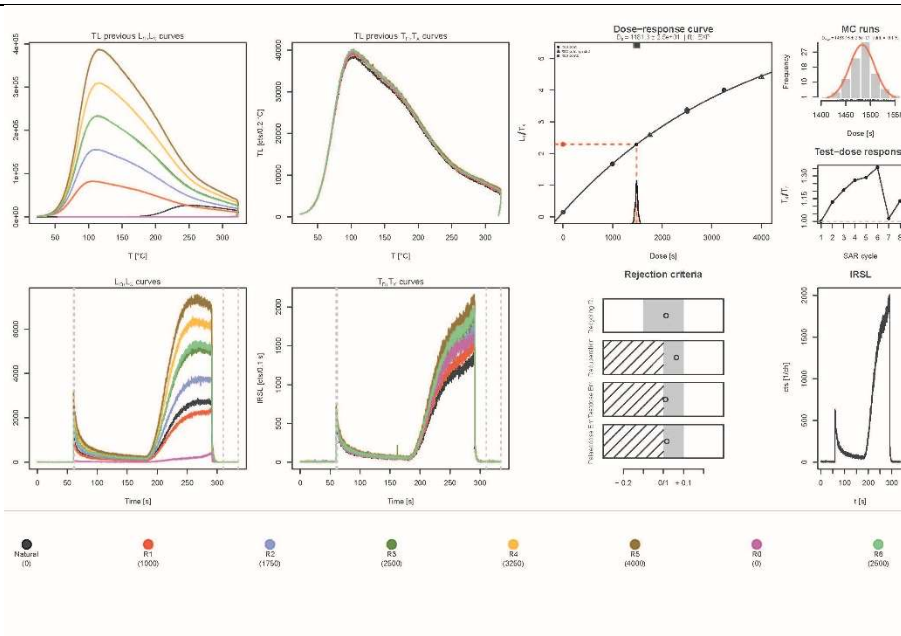
(a)



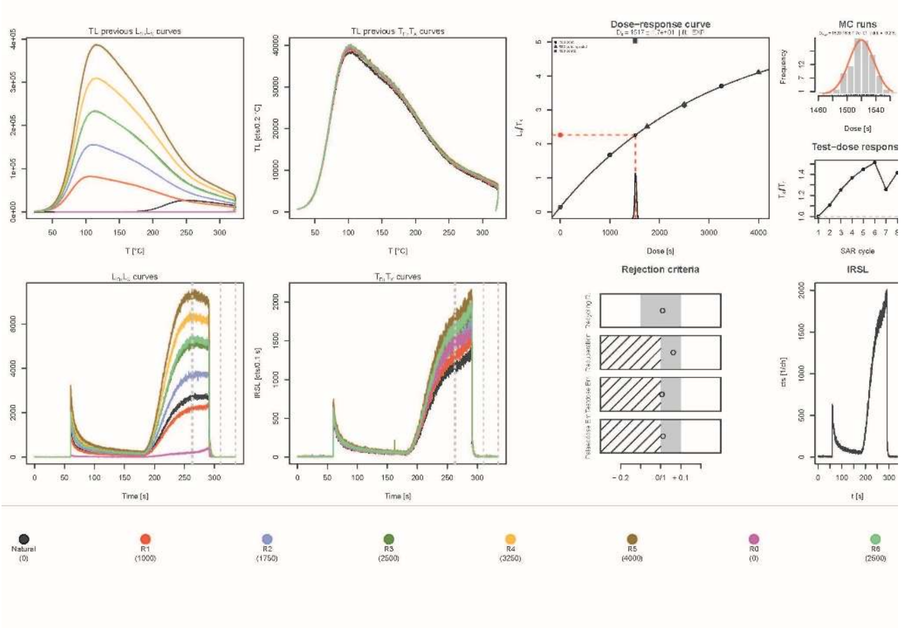
(b)

Figure S1.6.5. MS_30b. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 97 (PET-peak).

30b_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_1,97



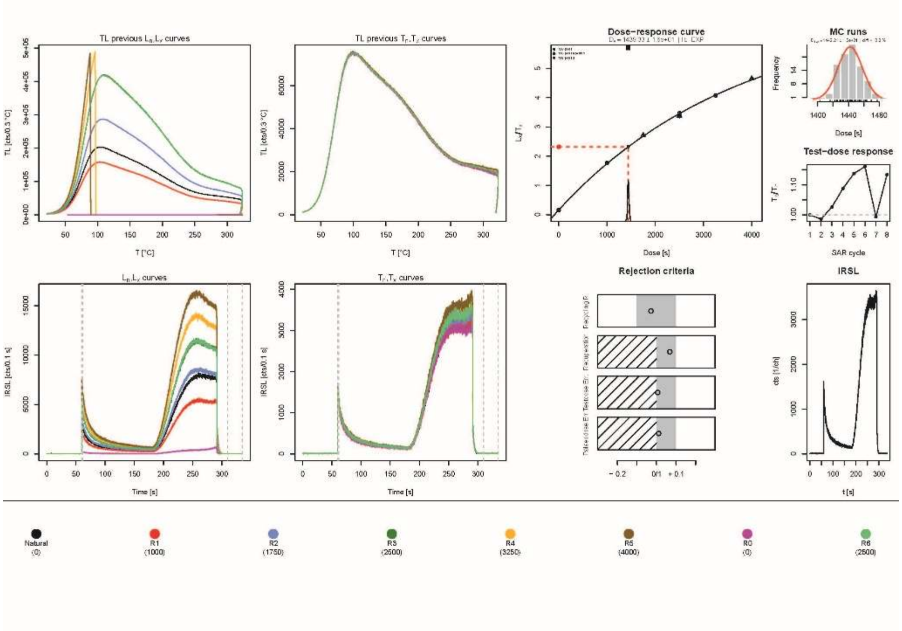
(a)



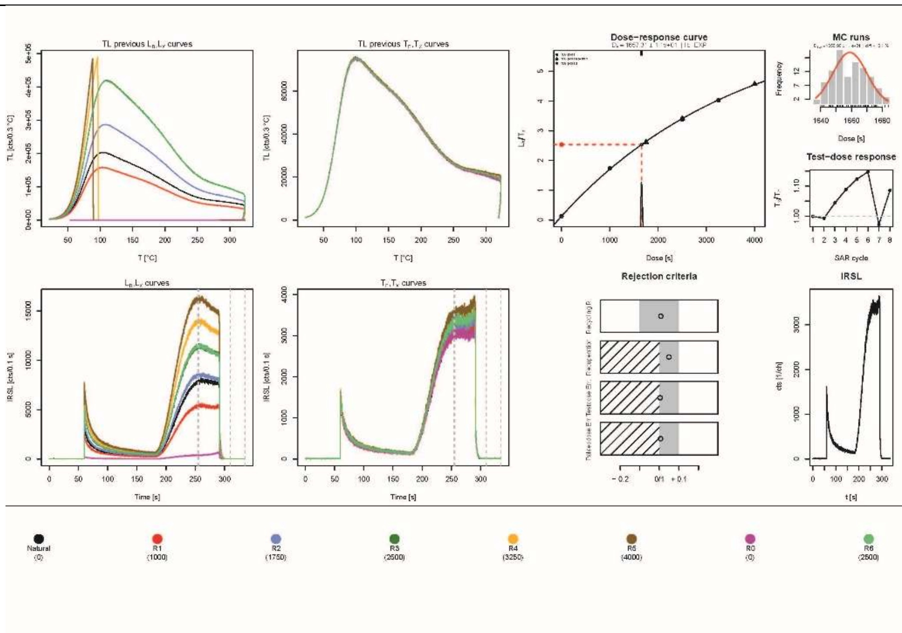
(b)

Figure S1.6.6. MS_47c. De determination on aliquot with natural luminescence of sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 102 (PET-peak).

47c_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_1,102



(a)



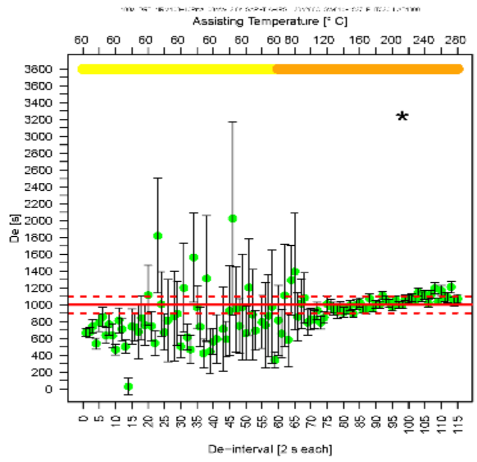
(b)

Figure S1.6.7. MS_44b. PBL test with 60 s UV bleaching on sample HDS-1776, tiny aliquot, 2500 s/300 Gy dose-response curve, NRM 500 s/60 Gy (20 %). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 98 (PET-peak).

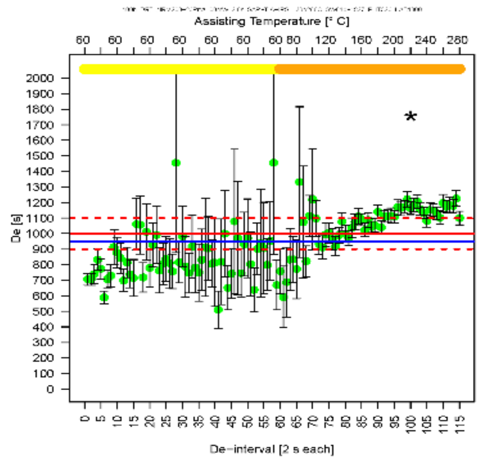
44b_UV-PBL-60s_NRM500-20Proz_60mW_2.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500_1,98

S2. Various PET-IRSL SAR tests and De measurements (MS_100a ff) on sample HDS-1827 on LR03 (PULSE)

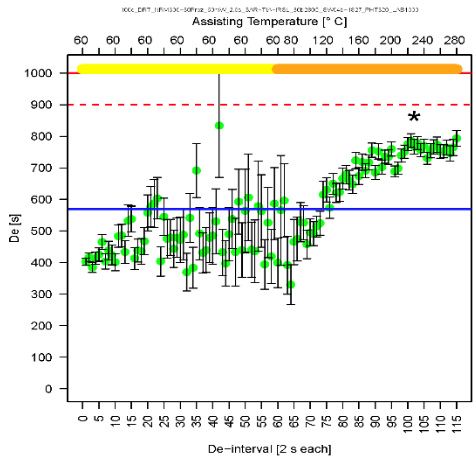
S2.1. NRM-DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – LAB 1000 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 %



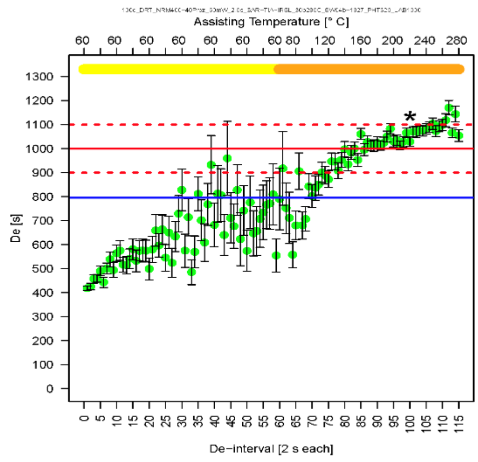
(a) NRM 10 %



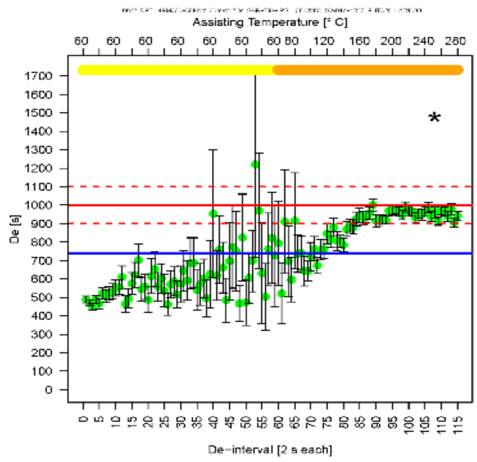
(b) NRM 20 %



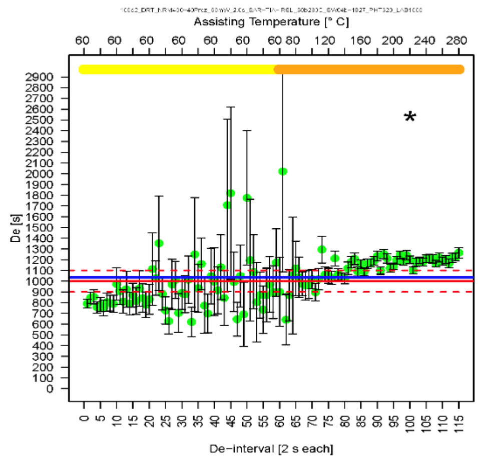
(c-1) NRM 30 %



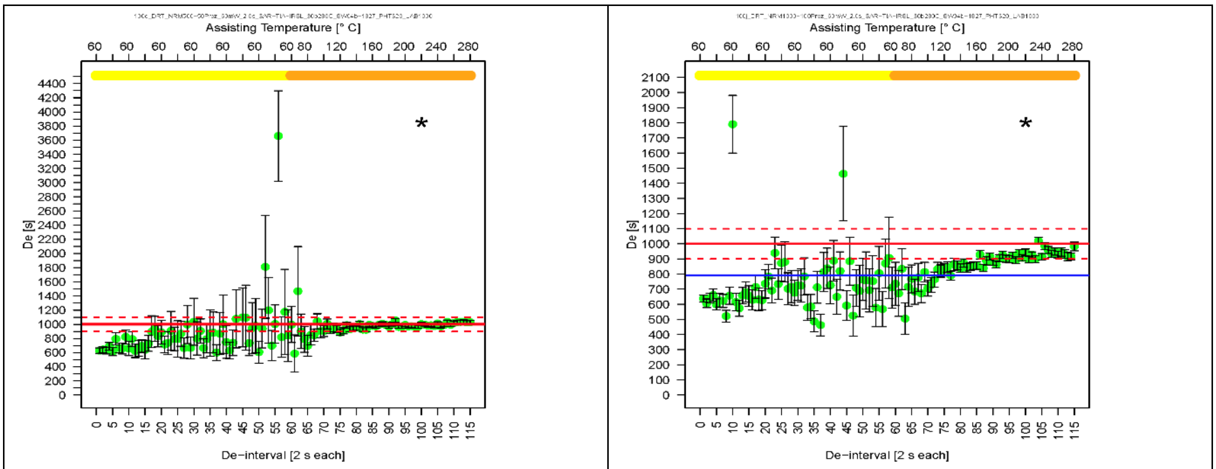
(d-2) NRM 40 %



(c-2) NRM 30 %



(d-2) NRM 40 %



(e) NRM 50 %

(j) NRM 100 %

Figure S2.1. MS_100a–100e, 100j. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), LAB 1000 s and NRM varying from 10 % to 50 % of LAB in steps of 10 %, and NRM 100 %. <u>“Classic” measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 98–102). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. “Too hot” thermal SAR parameter values not suitable for De determination.

- All NRM DRTs underestimate the given dose of 1000 s in the low-temperature PET-IRSL range
- The administered dose is recovered in the high-temperature PET-IRSL range around the PET-peak by several NRM: by 10 %, 40 %, 50 % and 100 %.
- NRM 30 % underestimates the given dose, which seems not systematic in the sequence of measurements with smaller and larger NRMs
- NRM 40 % shows a dip towards lower De values towards the earliest data values of the low-temperature PET-IRSL range
- We therefore repeated the NRM DRTs with 30 % and 40 % of LAB to investigate whether this a peculiarity of the respective NRMs
- The repeat measurement with NRM 30 % (100c2) recovers the given dose well by a plateau of De values in the high-temperature PET-IRSL range and with NRM 40 % (100d2) does not show a decline of the initial De values, but slightly overestimates the given dose in the high-temperature PET-IRSL range.

S2.2. NRM DRTS on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – LAB 400 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 %

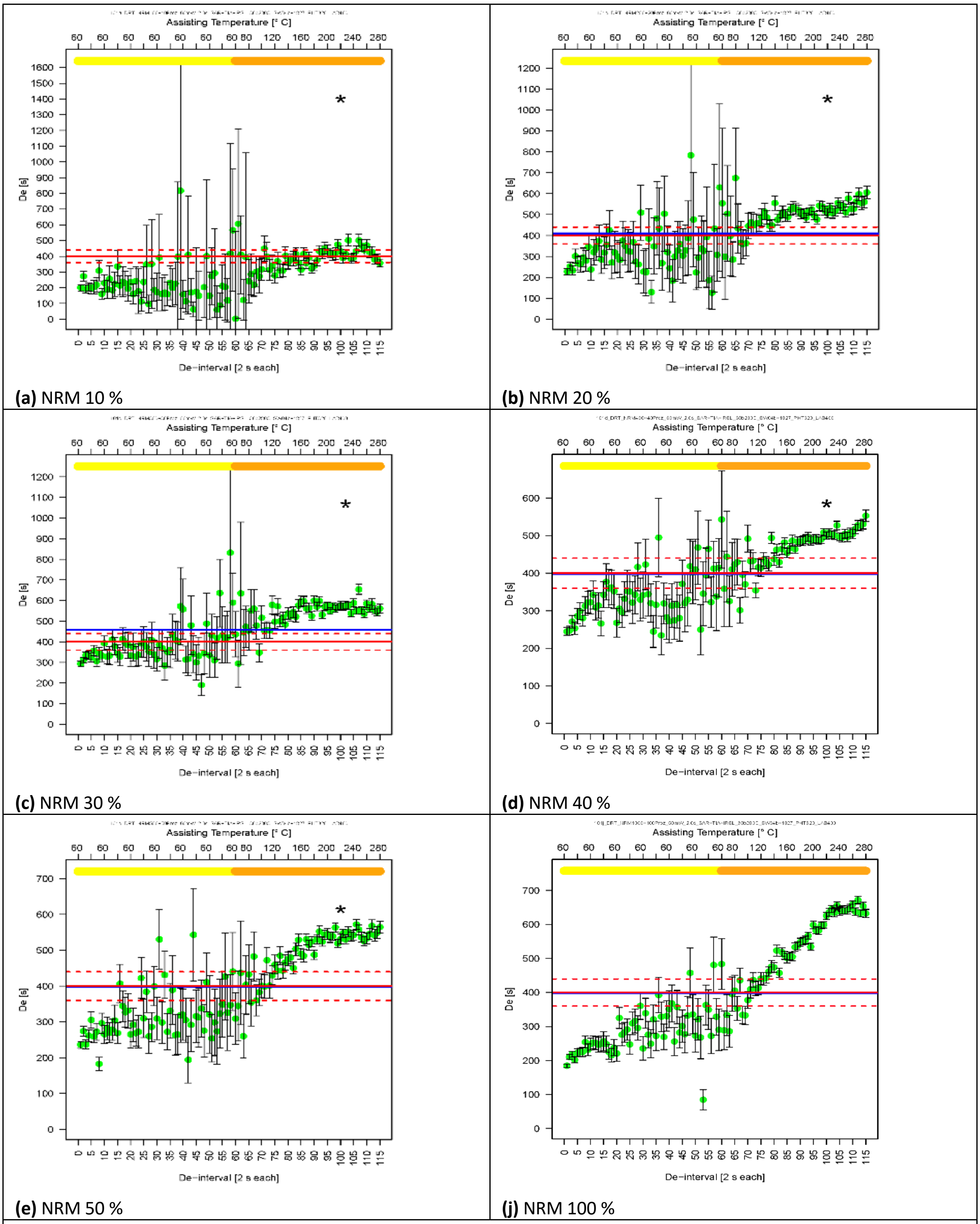
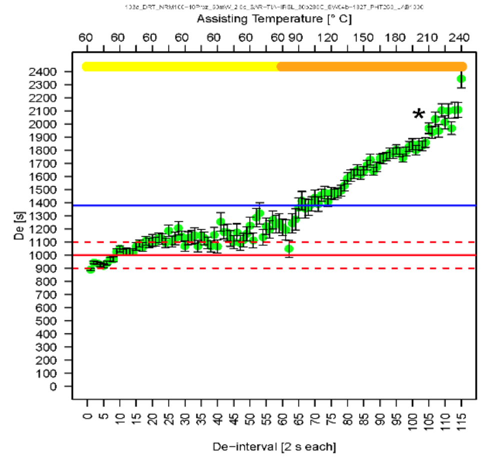


Figure S2.2. MS_101a–101e, 101j. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), LAB 400 s and NRM varying from 10 % to 50 % of LAB in steps of 10 %, and NRM 100 %. <u>"Classic" measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 100–104). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range

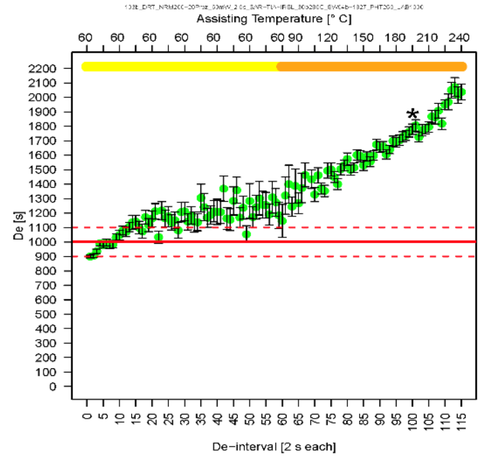
(60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. “Too hot” thermal SAR parameter values not suitable for De determination.

- The measurements of the NRM DRTs series 101a–101e and 101j differ from those in the previous series 100a–100e and 100j only by the smaller size of the given laboratory dose (LAB 400 s instead of 1000 s)
- Like for LAB 1000 s the given dose of 400 s is underestimated by all NRM in the low-temperature PET-IRSL range
- The smaller laboratory dose of 400 s is recovered in the high-temperature PET-IRSL range around the PET-peak by NRM 10 %, whereas larger NRM overestimate the given dose, the degree of overestimation increasing systematically with increasing NRM

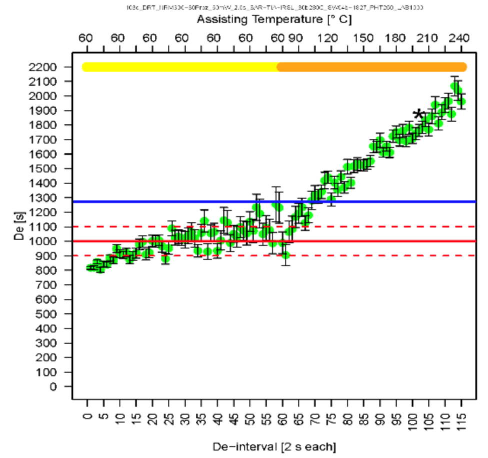
S2.3. NRM-DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle at 280 °C – LAB 1000 s – NRM 10–30 % of LAB increasing in steps of 10 % – PHT-TL at 260 °C – PET up to 240 °C – GRSL HBL likely too hot as compared to PHT-TL and maximum PET-IRSL readout temperature



(a) NRM 10 %



(b) NRM 20 %



(c) NRM 30 %

Figure S2.3. MS_103a–103c. PET-IRSL NRM DRTs with DRC aiming at 1000 s (100 %), NRM 10–30 %. “Classic” measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle at 280 °C, but PHT TL only at 260 °C and PET-IRSL readout only up to 240 °C. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 100–102). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–240 °C). All measurements up to PET_{max} 240 °C and with HBL 280 °C after PHT 260 °C. “Too hot” thermal SAR parameter values not suitable for De determination.

- Overestimation of LAB and strong increase in high-temperature PET-IRSL range with increasing readout temperature likely due to – still – too strong hotbleach exceeding PHT and maximum PET readout temperature

S2.4. NRM-DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle at 280 °C – LAB 1000 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 % of LAB – PHT-TL at 280 °C – PET-IRSL up to 280 °C – GRSL HBL also at 280 °C

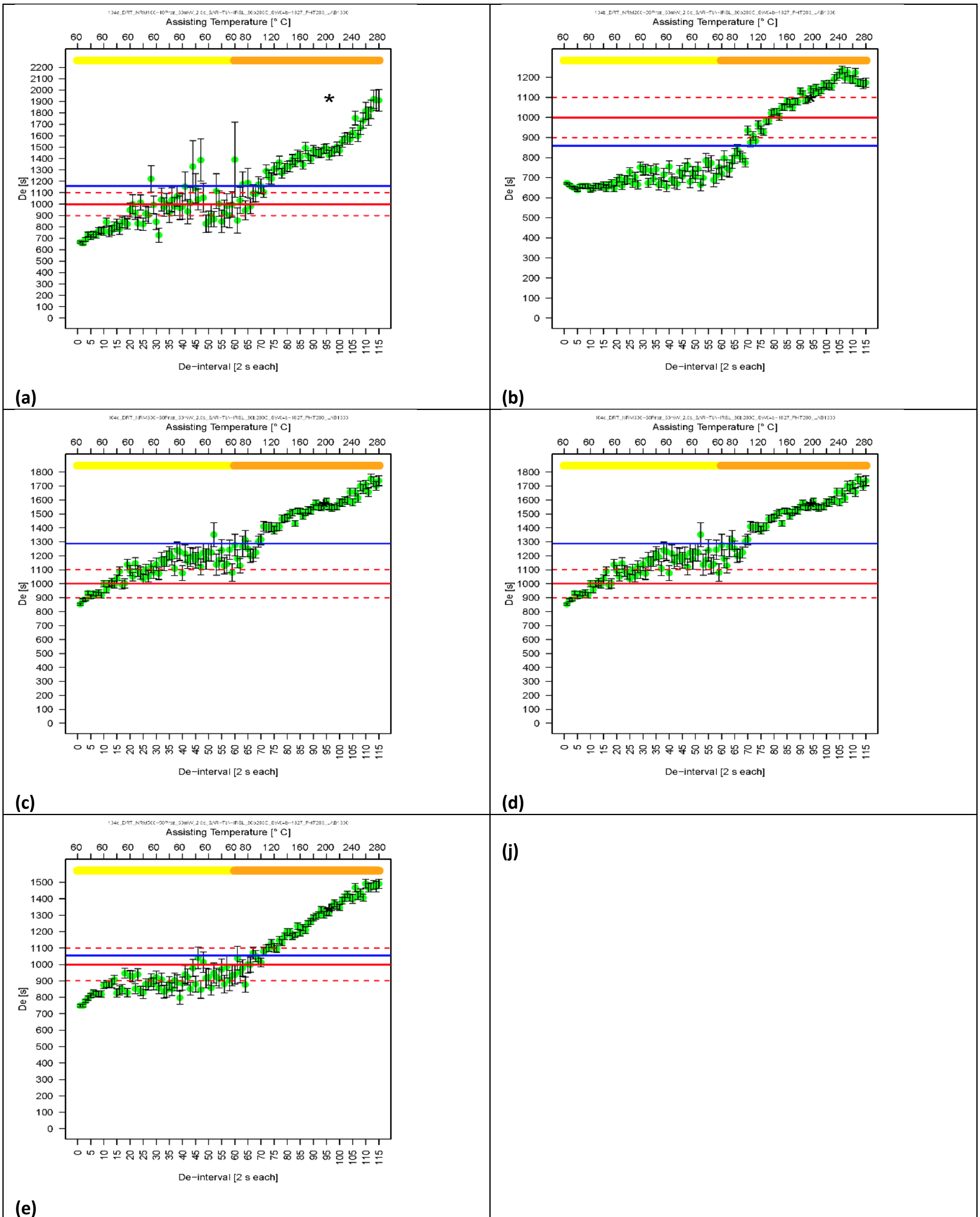
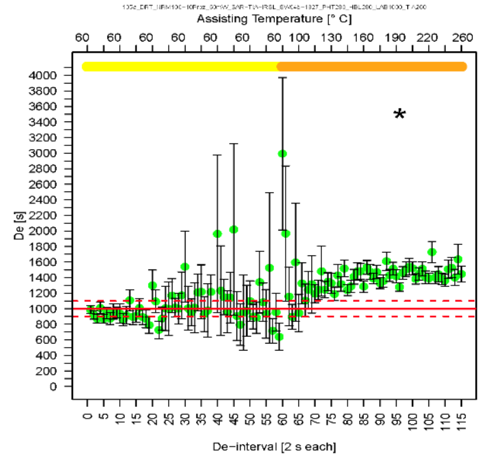


Figure S2.4. MS_104a–104e, 104j. PET-IRSL SAR NRM DRT with DRC aiming at 1000 s (100 %), NRM 10–50 % and NRM 100%. “Classic” measurement parameters: IR 60 mW/cm², PHT TL at 280 °C, PET IRSL up to 280 °C and GRSL hotbleach in each SAR half-cycle at 280 °C. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range

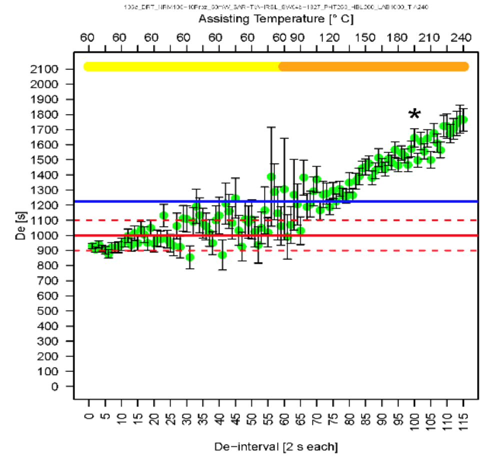
(60–280 °C). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 280 °C. "Too hot" thermal SAR parameter values not suitable for De determination.

- PHT and GRSL HBL at 280 °C and PET-IRSL readout up to 280 °C do not recover the given LAB with any of the investigated NRMs (10–50 % of LAB in steps of 10 % and 100 % of LAB)
- Test next to decrease thermal SAR parameter values PHT, PET_{max} and HBL

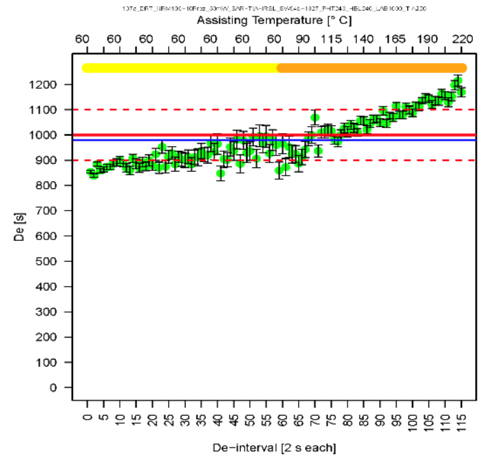
S2.5. NRM DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – LAB 1000 s – NRM 10 % of LAB – PHT-TL and GRSL HBL decreasing in steps of 20 K from 280 °C to 200 °C with corresponding PET-IRSL maximum temperature 20 K below PHT TL and GRSL HBL temperature



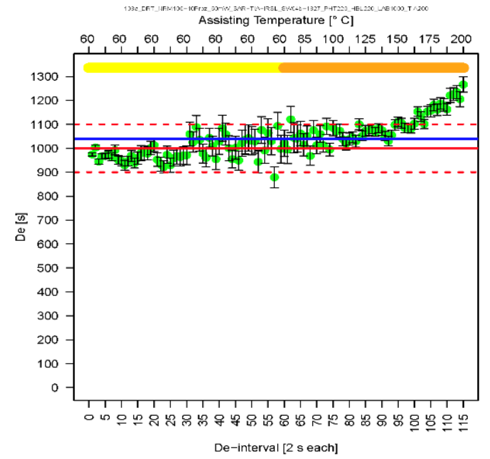
(a) PHT TL / GRSL HBL 280 °C, PET IRSL up to 260 °C



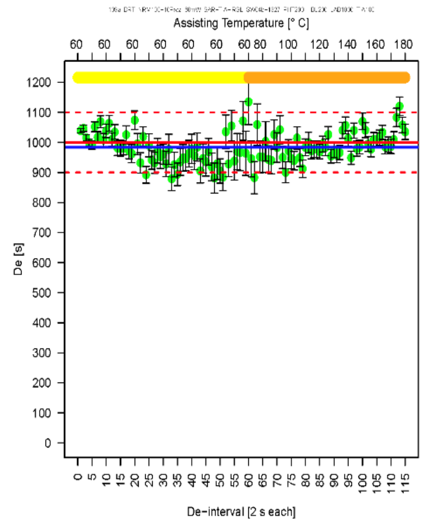
(b) PHT TL / GRSL HBL 260 °C, PET IRSL up to 240 °C



(c) PHT TL / GRSL HBL 240 °C, PET IRSL up to 220 °C



(d) PHT TL / GRSL HBL 220 °C, PET IRSL up to 200 °C



(e) PHT TL / GRSL HBL 200 °C, PET IRSL up to 180 °C

(j)

Figure S2.5. MS_105a–109a. PET-IRSL SAR DRT with DRC aiming at 1000 s (100 %), LAB 1000 s, NRM 10 % of LAB. "Classic" measurement parameters: IR 60 mW/cm². PET-IRSL up to 20 K below PHT-TL and GRSL HBL temperature. HBL in each SAR half-cycle. PHT-TL and GRSL HBL decreasing in steps of 20 K from 280 °C to 200 °C. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at

60 °C). Orange bar: High-temperature PET-IRSL range. Mostly "too hot" thermal SAR parameter values not suitable for De determination.

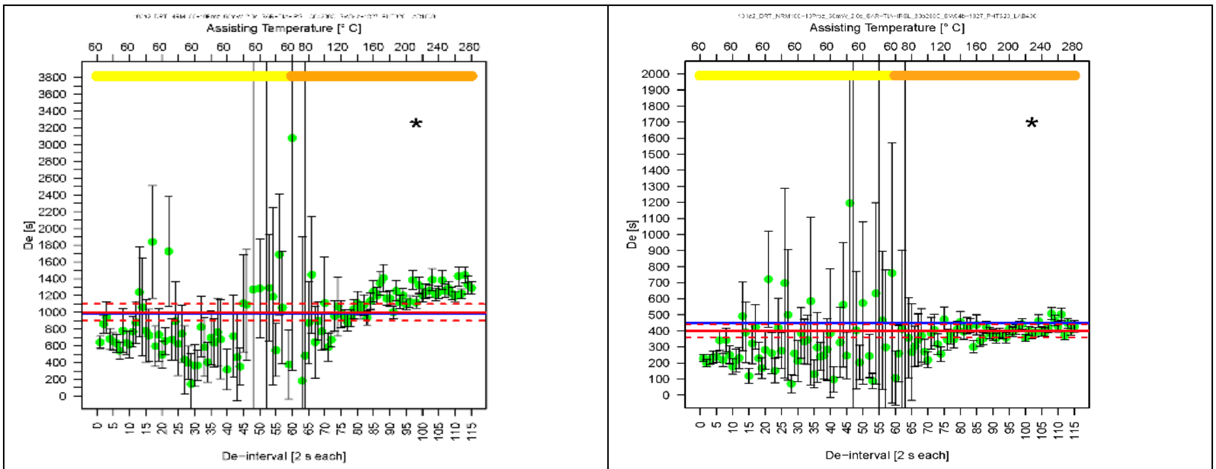
- PHT and GRSL HBL at 280 °C, 260 °C, 240 °C, 220 °C and 200 °C and corresponding PET-IRSL up to 260 °C, 240 °C, 220 °C, 200 °C and 180 °C. DRC 1000 s, LAB 1000 s, NRM 100 s.
- The weakest thermal treatment recovers LAB over the whole PET-IRSL range 60–180 °C within a 10 % error margin

Change of photomultiplier tube (PMT): ET Enterprises Ltd., Type B2F/RFI(C679AFN2)

  - Serial 39659, PMT-Type 9235
  - Serial 40462, PMT-Type 9235QB04
- Repeat measurement series 100a, 101a and 105a–109a

- After that
    - get away from HBL = PHT and substitute by HBL = PET_{max}

S2.6. NRM-DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm2 – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of GC-AIM – LAB 1000 s and 400 s – Repeat measurements with PMT-2

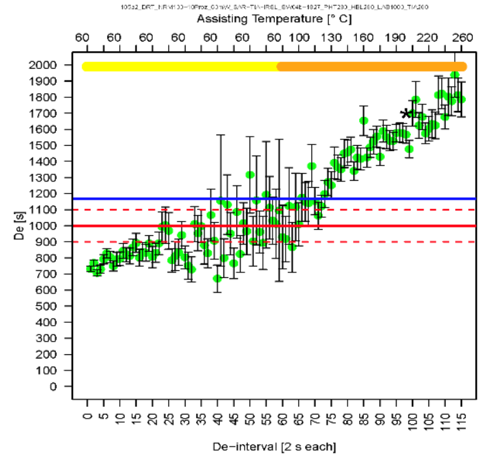


(a) NRM 10 %, LAB 1000 s

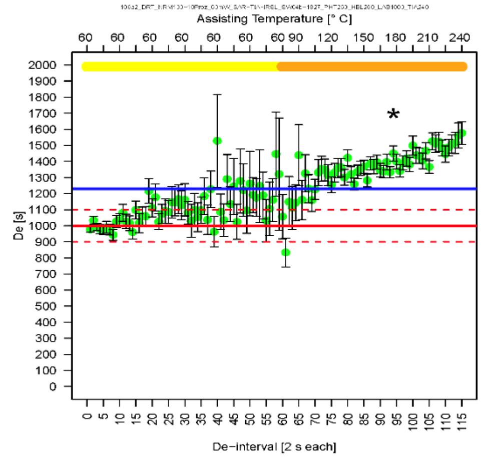
(b) NRM 10 %, LAB 400 s

Figure 2.6. MS_100a2, 101a2. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 320 °C, PET_{max} and HBL 280 °C, NRM 10 %. <u>“Classic” measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 98–102). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). “Too hot” thermal SAR parameter values not suitable for De determination.

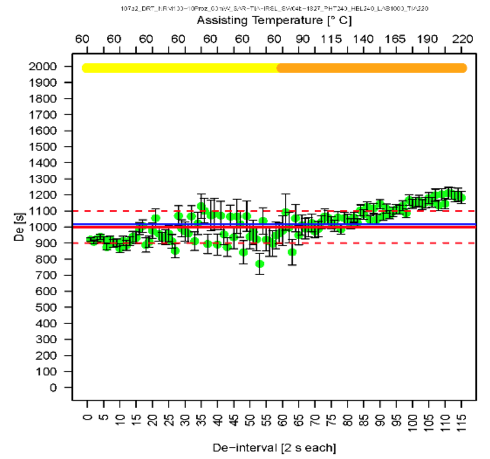
S2.7. DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm² – GRSL hotbleach every SAR subcycle – LAB 1000 s – NRM 10 % of LAB – PHT-TL and GRSL HBL decreasing in steps of 20 K from 280 °C to 200 °C with corresponding PET IRSL maximum temperature 20 K below PHT TL and GRSL HBL temperature – Repeat measurements with PMT-2



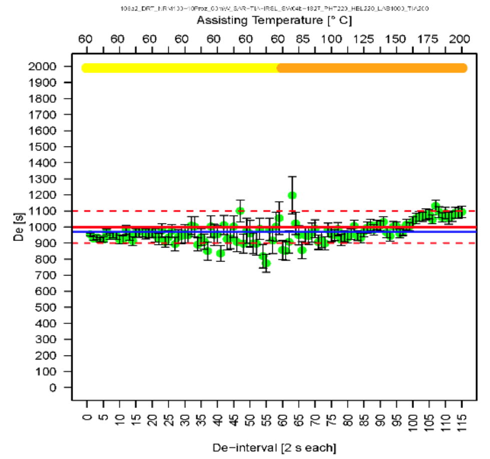
(a) PHT TL / GRSL HBL 280 °C, PET IRSL up to 260 °C



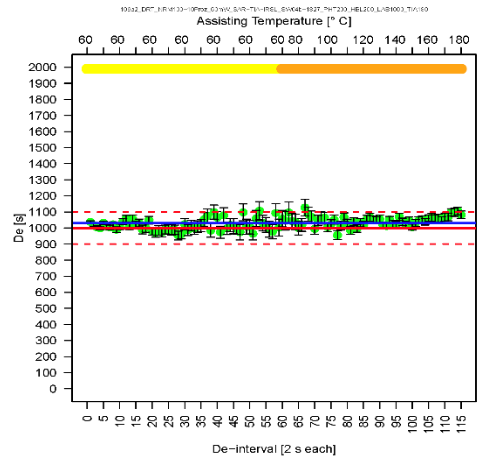
(b) PHT TL / GRSL HBL 260 °C, PET IRSL up to 240 °C



(c) PHT TL / GRSL HBL 240 °C, PET IRSL up to 220 °C



(d) PHT TL / GRSL HBL 220 °C, PET IRSL up to 200 °C



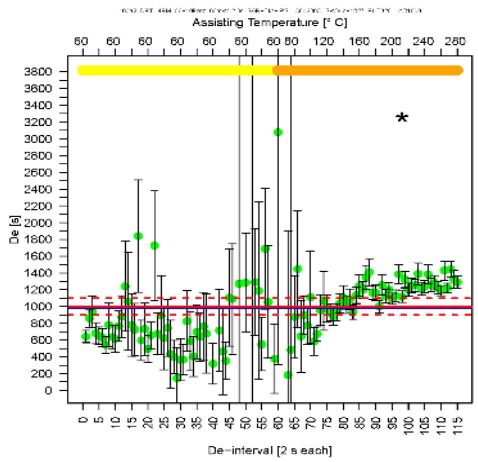
(e) PHT TL / GRSL HBL 200 °C, PET IRSL up to 180 °C

(j)

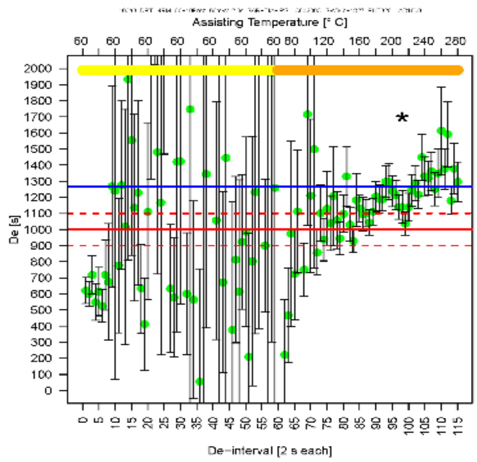
Figure S2.7. MS_105a2–109a2. PET-IRSL SAR DRT with DRC aiming at 1000 s (100 %), LAB 1000 s, NRM 10 % of LAB. “Classic” measurement parameters: IR 60 mW/cm². PET-IRSL up to 20 K below PHT-TL and GRSL HBL temperature. HBL in each SAR half-cycle. PHT-TL and GRSL HBL decreasing in steps of 20 K from 280 °C to 200 °C. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (only for aliquots read out up to ≥ 240 °C).

Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range. Mostly "too hot" thermal SAR parameter values not suitable for De determination.

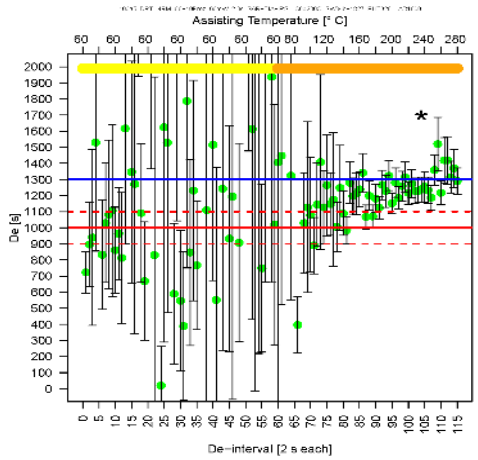
S2.8. DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR subcycle – GC 1000 s – NRM 10 % of GC-AIM – LAB 1000s – Repeat measurements – Include MS_100a2 in the graph



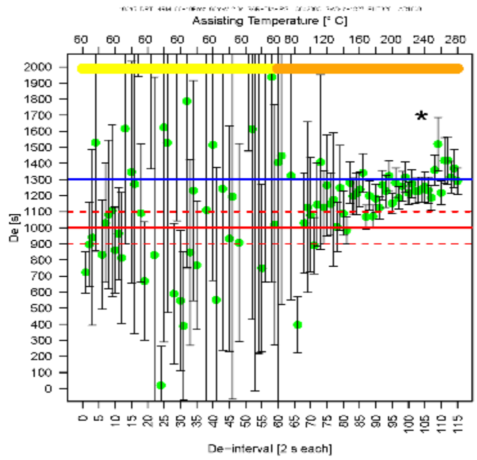
(a) NRM 10 %, LAB 1000 s, PMT-2



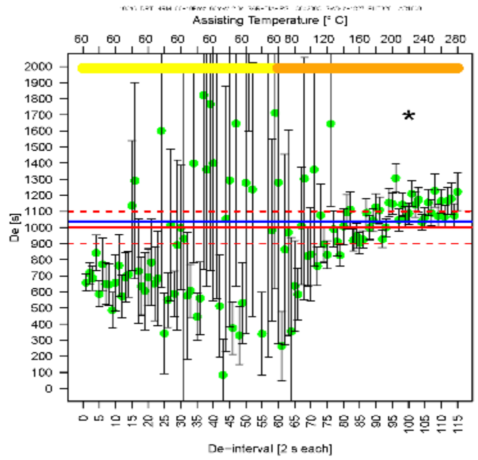
(b) NRM 10 %, LAB 1000 s, PMT-2



(c) NRM 10 %, LAB 1000 s, PMT-2



(d) NRM 10 %, LAB 1000 s, PMT-2

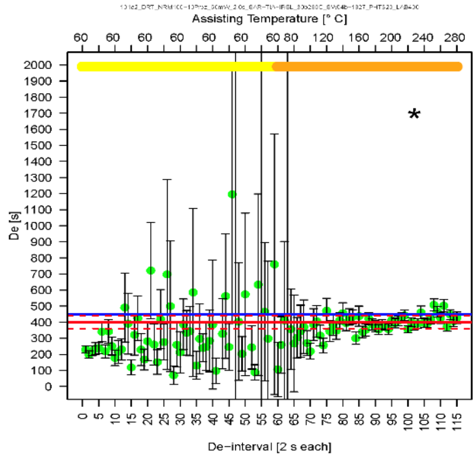


(e) NRM 10 %, LAB 1000 s, PMT-2

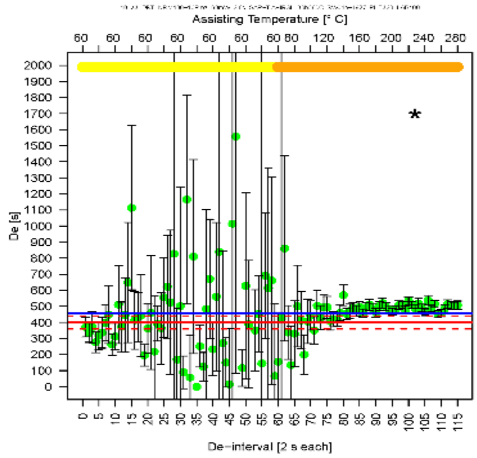
Figure S2.8. MS_100a2–MS_100a6. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, LAB 1000 s. <u>"Classic" measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET peak (here: De-signal integral 100–104). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements with PMT-2. "Too hot" thermal SAR parameter values not suitable for De determination.

• Low PET-IRSL signal leading to large data scatter; earlier prepared aliquots had lost too many grains

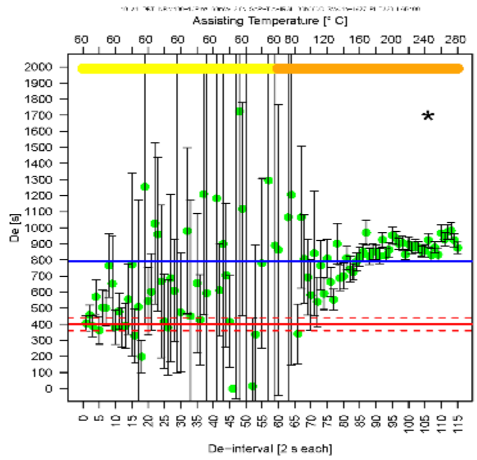
S2.9. DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 400 s – NRM 10 % of DRC-AIM – LAB 1000 s – Repeat measurements



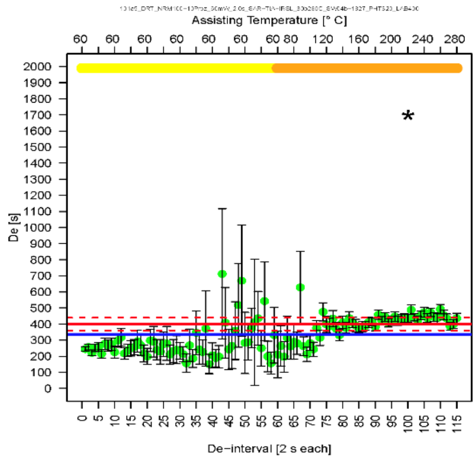
(a) NRM 10 %, LAB 400 s, PMT-2



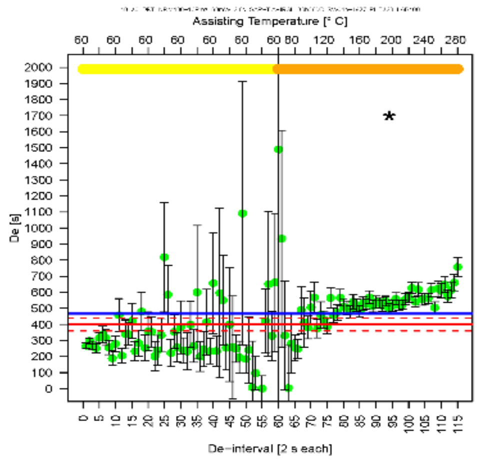
(b) NRM 10 %, LAB 400 s, PMT-2



(c) NRM 10 %, LAB 400 s, PMT-2



(d) NRM 10 %, LAB 400 s, PMT-2

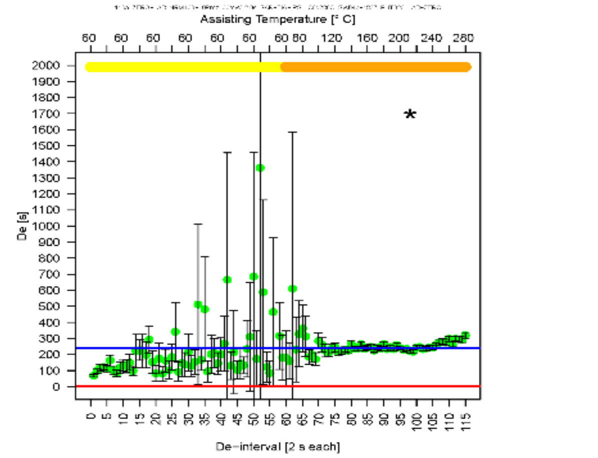


(e) NRM 10 %, LAB 400 s, PMT-2

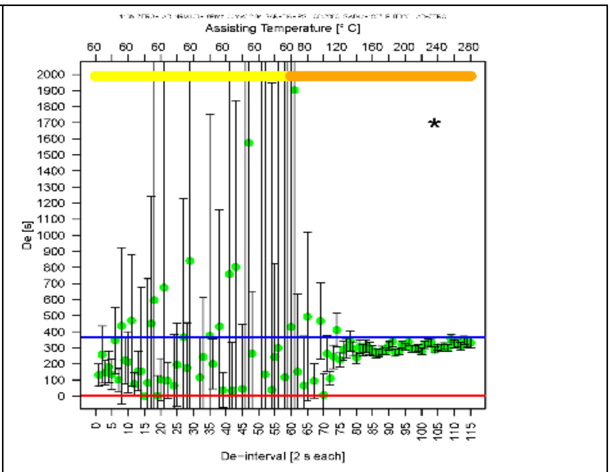
Figure S2.9. MS_101a2–MS_101a6. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, LAB 400 s. "Classic" measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR subcycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET peak (here: De-signal integral 94–106). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements with PMT-2. "Too hot" thermal SAR parameter values not suitable for De determination.

- Like for the DRT with LAB 1000 s, all recovered doses underestimate the given dose in the low-temperature PET-IRSL range and – with the exception of **MS_101a2 (a)** – overestimate it to various degrees (up to > 100 %, ca 500 s) supporting the assumption that the aliquots contain a residual dose unbleachable by the here applied bleaching procedure

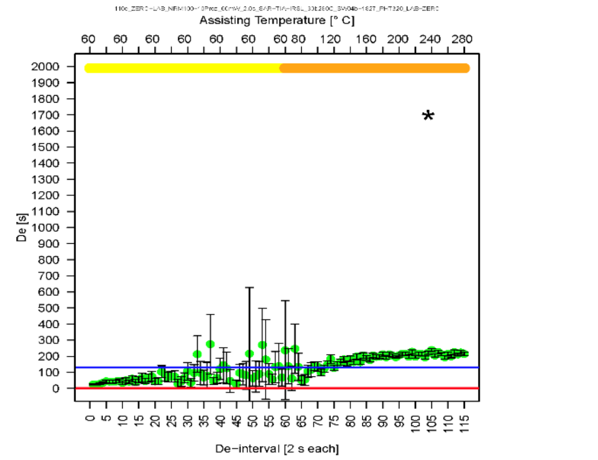
S2.10. ZERO-LAB DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – Repeat measurements



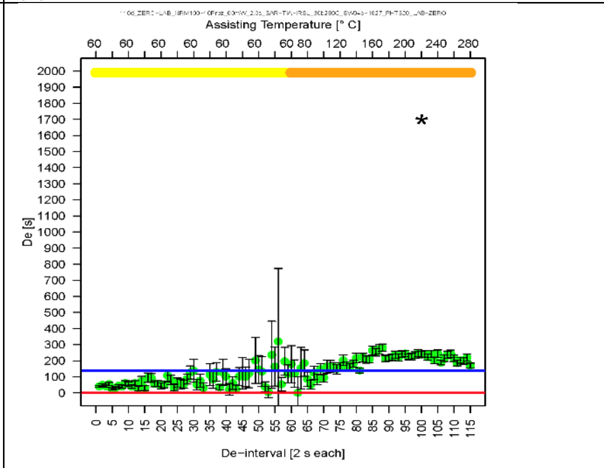
(a) NRM 10 %, LAB 1000 s, PMT-2



(b) NRM 10 %, LAB 1000 s, PMT-2



(c) NRM 10 %, LAB 1000 s, PMT-2



(d) NRM 10 %, LAB 1000 s, PMT-2

Figure S2.10. MS_110a–MS_110d. PET-IRSL SAR dose recovery test with PHT 320 °C, PET_{max} and HBL 280 °C, GC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, but zero dose. "Classic" measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR subcycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 98–104). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements with PMT-2. "Too hot" thermal SAR parameter values not suitable for De determination.

- PHT 320 °C produces a residual dose of ca 250–320 s (ca 30–38 Gy), which, when assuming a dose rate of 3.3 Gy/ka and allowing a residual dose of 10 % of the palaeodose would allow do date samples with a minimum age of 90 – 115 ka. As, however, the dose rate of the fluvial deposits in the low-lying channel position is probably considerably lower, the minimum ages would also be respectively larger, i.e., at lesat of Eemian or Rissian times. Therefore, hot PHT temperatures are not an option for the BSN samples, for which other studies suggest deposition in late-glacial times to early Holocene times

- PHT 320 °C is not an option for the BSN sample
- Next steps:
  - Zero-Dose DRTs
  - Decrease PHT temperature from 300 °C to 200 °C with corresponding PET_{max} and HBL 30 K lower → change of philosophy with respect to MS_105a–109a/MS_105a2–109a2 in which HBL was equal PHT (not equal PET_{max})

- PHT 320 – PET_{max} 280 – HBL 280 ← „classic" ← done already

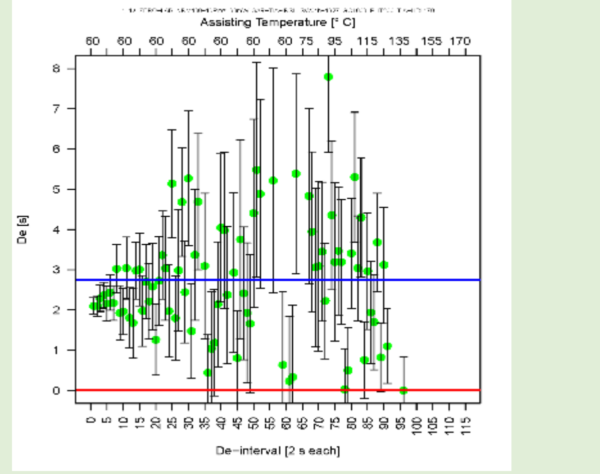
- Swap to Δ 30 K instead of Δ 40 K PET_{max}/HBL below PHT

- PHT 320 – PET_{max} 290 – HBL 290 → omit measurement, not worth it

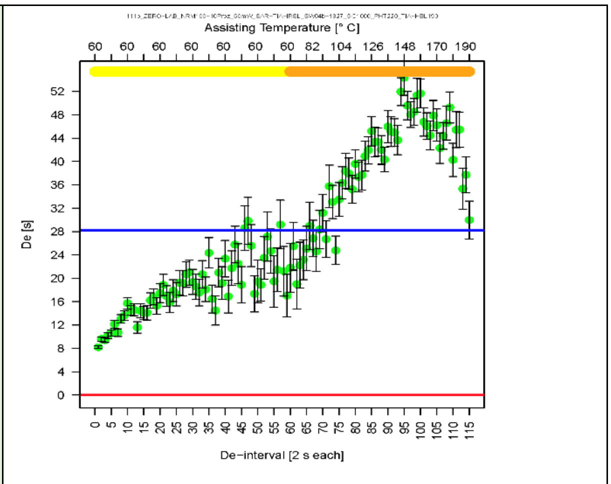
- PHT 300 – PET_{max} 270 – HBL 270
- PHT 280 – PET_{max} 250 – HBL 250
- PHT 260 – PET_{max} 230 – HBL 230
- PHT 240 – PET_{max} 210 – HBL 210
- PHT 220 – PET_{max} 190 – HBL 190
- PHT 200 – PET_{max} 170 – HBL 170

Select the one which works with the strongest thermal treatment and increase PET/HBL by 10–20 K

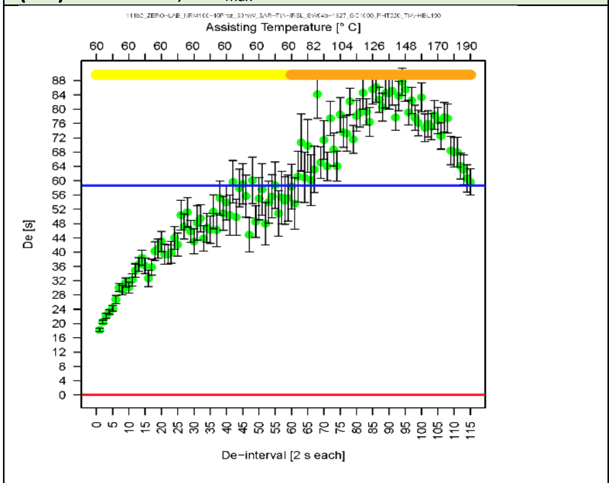
S2.11. ZERO-LAB DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – Repeat measurements



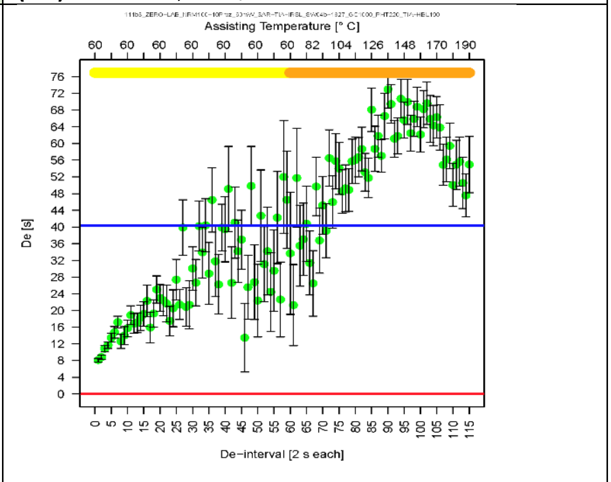
(a-1) PHT 200 °C, PET$_{max}$ and HBL 170 °C



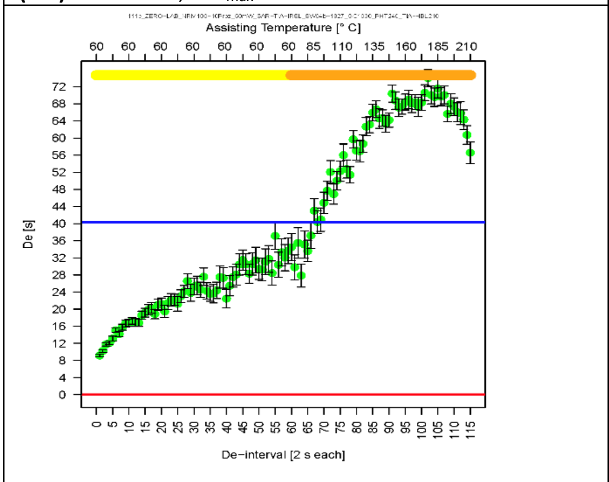
(b-1) PHT 220 °C, PET$_{max}$ and HBL 190 °C



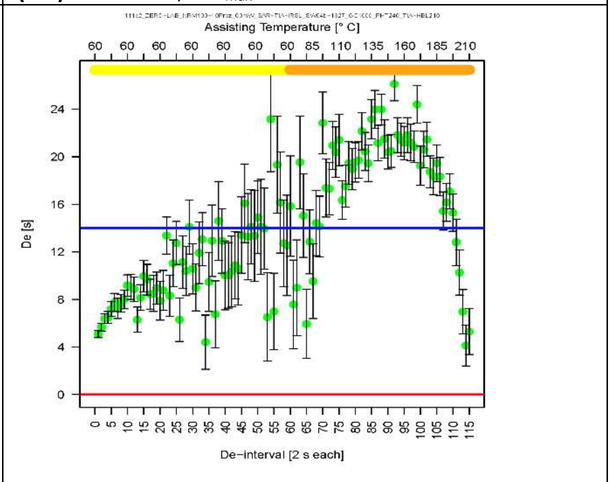
(b-2) PHT 220 °C, PET$_{max}$ and HBL 190 °C



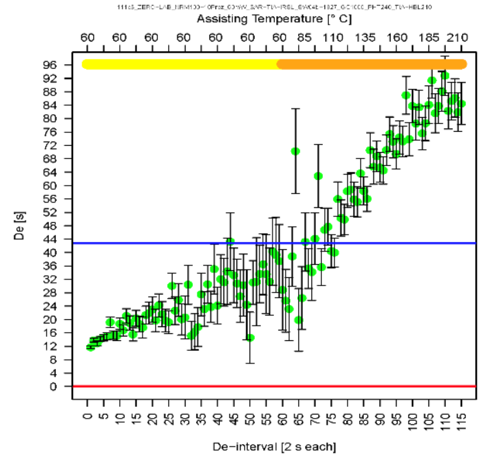
(b-3) PHT 220 °C, PET$_{max}$ and HBL 190 °C



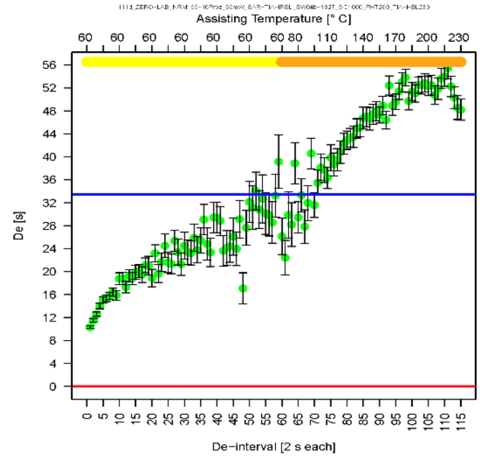
(c-1) PHT 240 °C, PET$_{max}$ and HBL 210 °C



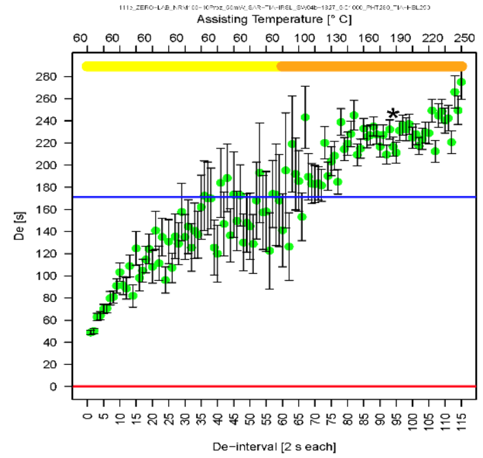
(c-2) PHT 240 °C, PET$_{max}$ and HBL 210 °C



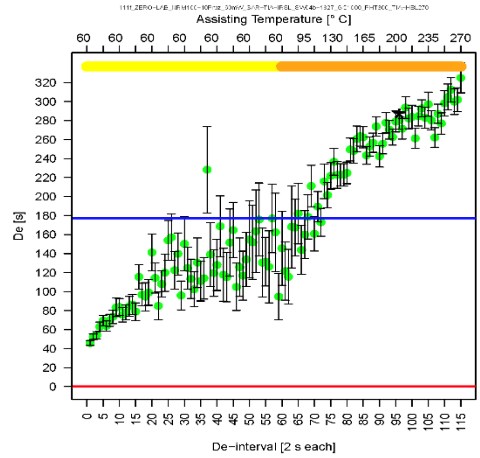
(c-3) PHT 240 °C, PET_{max} and HBL 210 °C



(d) PHT 260 °C, PET_{max} and HBL 230 °C



(e) PHT 280 °C, PET_{max} and HBL 250 °C



(f) PHT 300 °C, PET_{max} and HBL 270 °C

Figure S2.11. MS_111a–MS_111f. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, but zero dose. <u>“Classic” measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (only for higher PHT > 240 °C). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C). All measurements with PMT-2. **(a)** Giving for De-signal integral ≥ 90 mainly values < 0 s, for which the function “analyse_SAR.CWOSL” of the R Luminescence package produces mainly “NA”, which can not be graphically displayed. Mostly “too hot” thermal SAR parameter values not suitable for De determination.

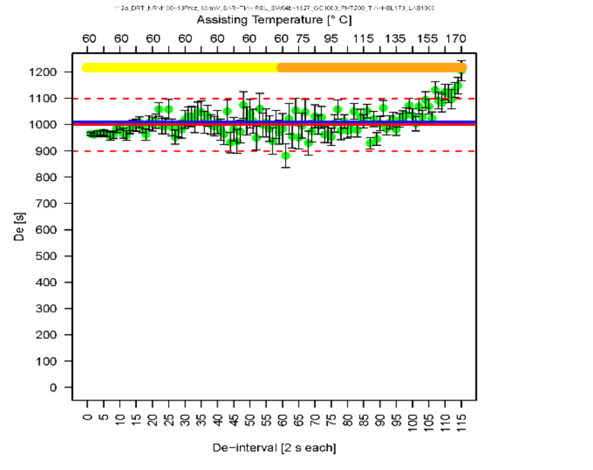
- Each measurement produces a residual dose, which – with the exception of **(a)** – starts at ca 8 s (0.944 Gy) and increases with rising PET-IRSL readout temperature, which, however, is likely associated with a lower fading rate. For dating one may accept a certain maximum residual dose of, e.g., 5 % or 10 % of a samples palaeodose. Assuming an environmental dose rate of ca 3 Gy/ka this allows to assess the approximate minimum age a sample should have to be submitted to a PHT temperature and associated PET_{max} and HBL temperature.
- **(a)** PHT 200 °C with PET_{max} and HBL 170 °C produces a residual dose of < 3 s (0.354 Gy). This would allow to date samples ≥ ca 1 ka (10 % remnant dose) or ca 2 ka (20 %), respectively.
- With higher PHT temperatures the residual dose of the distal high-temperature PET-IRSL range rises strongly to **(b)** ca 50 s (5.9 Gy) to 86 s (10 Gy), **(c)** 70 s (8.3 Gy) to 88 s (10.3 Gy), which allows to date pleniglacial samples with a minimum age of ca **(a)** 20–33 ka, **(b)** 28–34 ka (bith 10 %).
- **(c)** However, we repeated PHT 220 °C with PET_{max} and HBL 190 °C and PHT 240 °C with PET_{max} and HBL 210 °C each three times to investigate the potential variability of the residual dose, and found for the stronger thermal treatment also an aliquot (**MS_111c2**) which delivereed a much smaller residual dose of ca 22 s (2.595 Gy), which would allow to date samples with a minimum age of ca 8.7 ka (10 %) or 17.3 ka (5 %), which would conform to the expected late-glacial depositional age of the SW04b sample.
- With a residual dose of 53 s (6.3 Gy) PHT 260 °C with PET_{max} and HBL 230 °C similar to **MS_111b** does not show a systematic increase with increasing thermal treatment.
- **(e)** PHT 280 °C with PET_{max} and HBL 250 °C, however, shows a significant rise of the residual dose to ca 230 s (27 Gy) which, by range, conforms to the ZERO-LAB DRTs with PHT 320 °C with PET_{max} and HBL 280 °C

(**MS_110a–110d**). Similar to **MS_110a–110d MS_111e** shows a pronounced PET-peak (position at De-signal integral 94) and exhibits a longer high-temperature plateau of elevated De values.

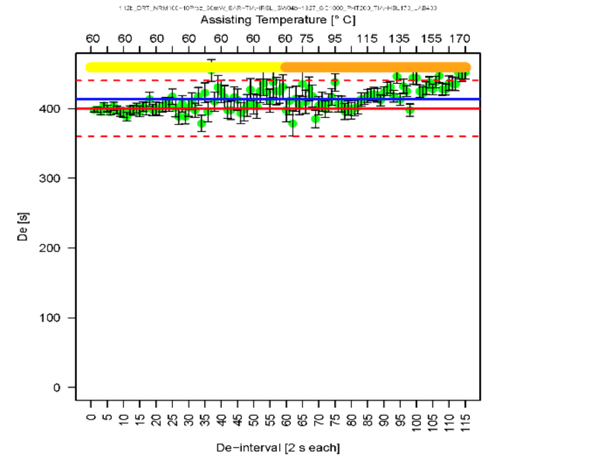
- With a residual dose of 290 s in the high-temperature PET-IRSL range, a pronounced PET-peak (here at De-signal integral 96) and a high-temperature PET-IRSL plateau of elevated De values **(f)** fits the series of measurements with PHT ≥ 260 °C. It is possible that measurements with PET_{max} and HBL 40 K below PHT (cf. **MS_110a–110d**) show a neater plateau than measurements with PET_{max} and HBL 30 K below PHT.

- Next steps:
    - DRT with DRC 1000 s, NRM 10 % and LAB 1000 s and 400 s with:
        - PHT 200 °C, PET_{max} and HBL 170 °C
        - PHT 260 °C, PET_{max} and HBL 230 °C

S2.12. NRM-DRTs on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – LAB 1000s and 400 s – PHT 200 °C – PET_{max} and HBL 170 °C



(a) NRM 10 %, LAB 1000 s

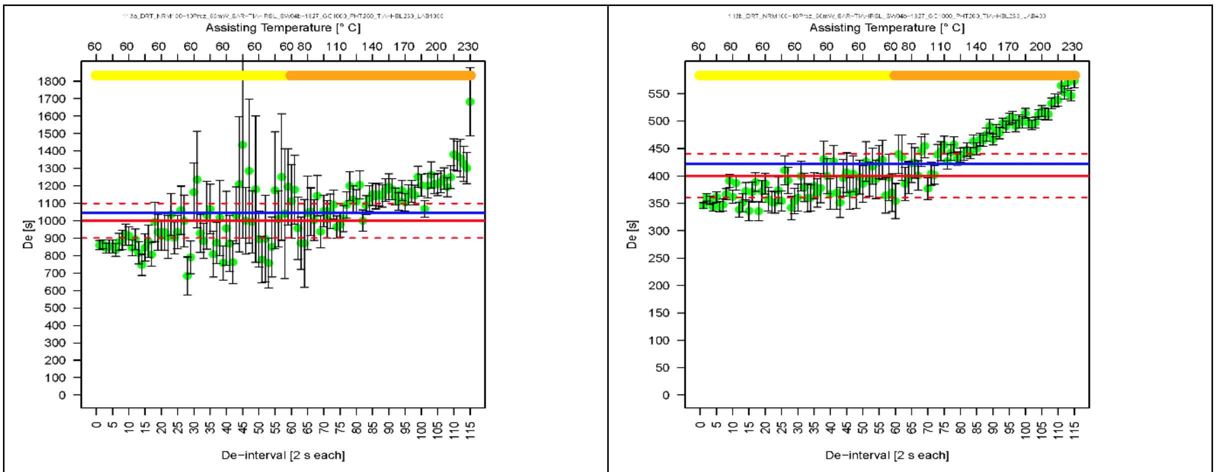


(b) NRM 10 %, LAB 400 s

Figure S2.12. MS_112a–112b. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. "Classic" measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. **(a)** LAB 1000 s. **(b)** LAB 400 s. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. No PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). Modest thermal SAR parameter values suitable for de determination.

- Both LABs are recovered reasonably well within error margins
- Use SAR parameter values for De determination

S2.13. NRM-DRTs on SW04b-1827 – PET “classic” – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – LAB 1000 s and 400 s – PHT 260 °C – PET$_{max}$ and HBL 230 °C

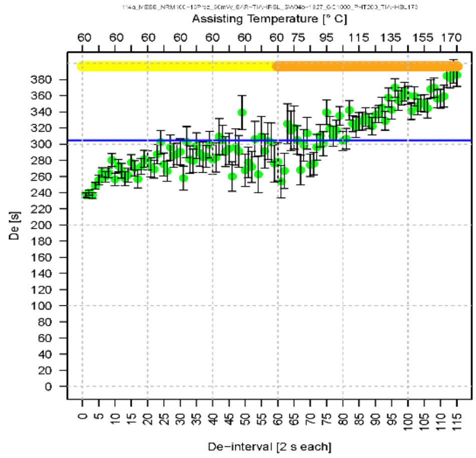


(a) NRM 10 %, LAB 1000 s

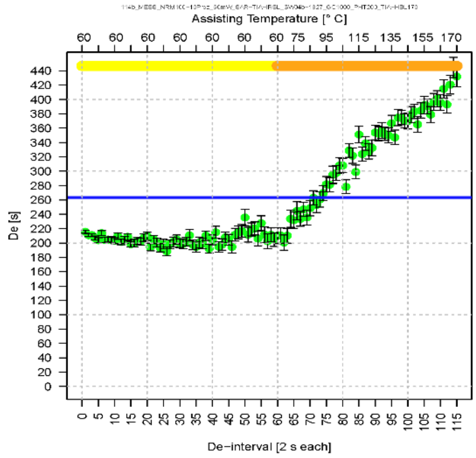
(b) NRM 10 %, LAB 400 s

Figure S2.13. MS_113a – 113b. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 260 °C, PET$_{max}$ and HBL 230 °C. <u>“Classic” measurement parameters:</u> IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. **(a)** LAB 1000 s. **(b)** LAB 400 s. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. No clear PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). “Too hot” thermal SAR parameter values not suitable for De determination.

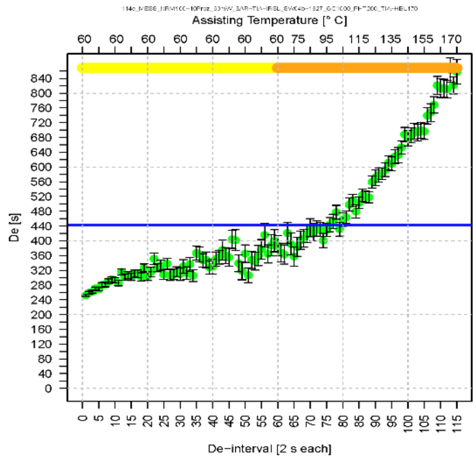
S2.14. De determination on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – GC 1000 s – NRM 10 % of DRC-AIM – PHT 200 °C – PET_{max} and HBL 170 °C



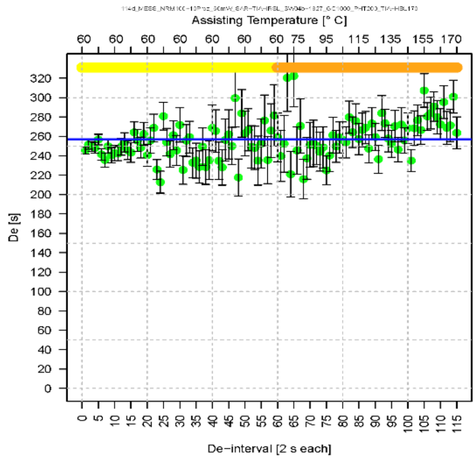
(a) Mean De-integral 96–111: ca 357 s (42.098 Gy; 14.0 Ka)



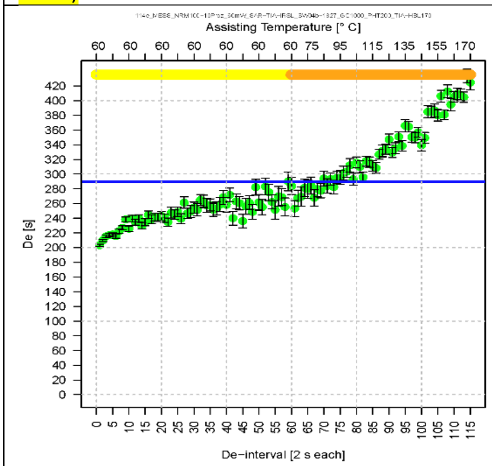
(b) Steady increase to ca 430 s (50.7 Gy; 16.9 Ka)



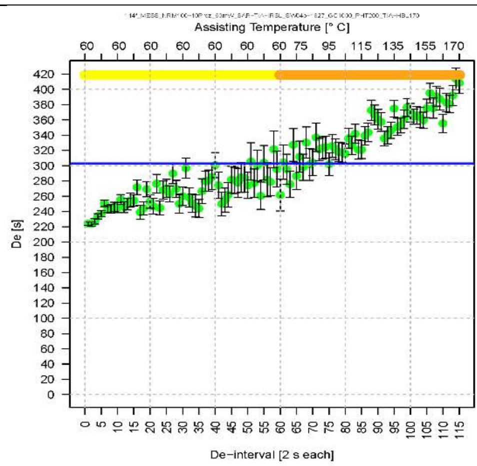
(c) Up to ca 814.5 s (mean De-integral 109–112) (96 Gy; 32 ka)



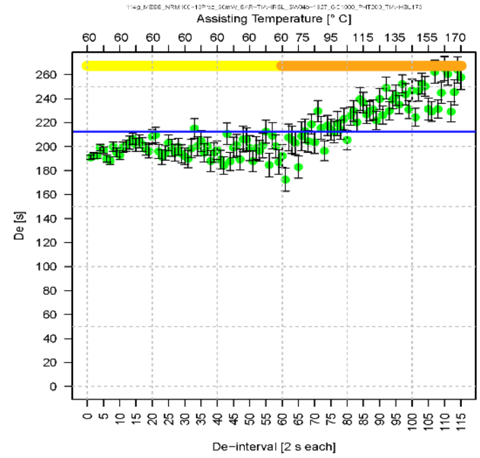
(d) Mean De-integral 105–115: ca 283 s (33.4 Gy; 11.1 Ka)



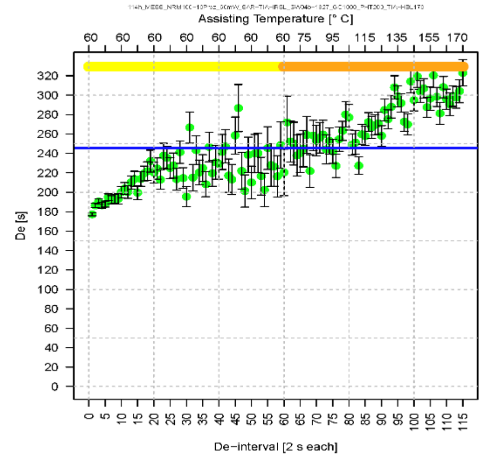
(e) Up to ca 405.7 s (mean De-integral 108–113) (47.8 Gy; 16 ka)



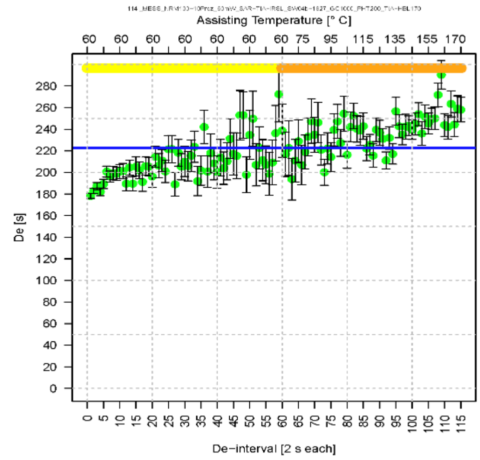
(f) Up to ca 381.8 s (mean De-integral 105–113) (45 Gy; 15 ka)



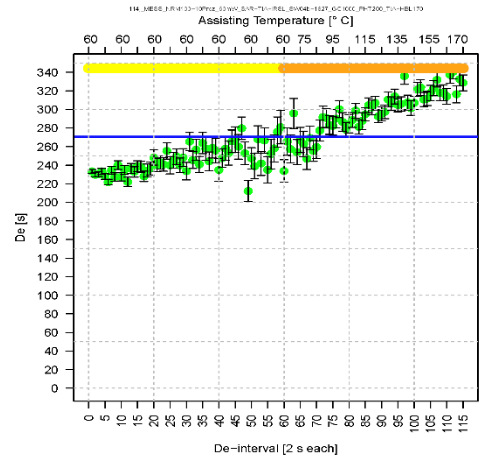
(g) Mean De-integral 97–115: ca 245.7 s (28.97 Gy; 9.7 ka)



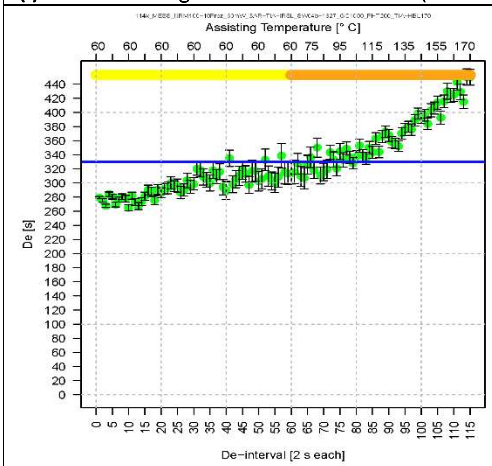
(h) Mean De-integral 99–115: ca 302.4 s (35.4 Gy; 11.9 ka)



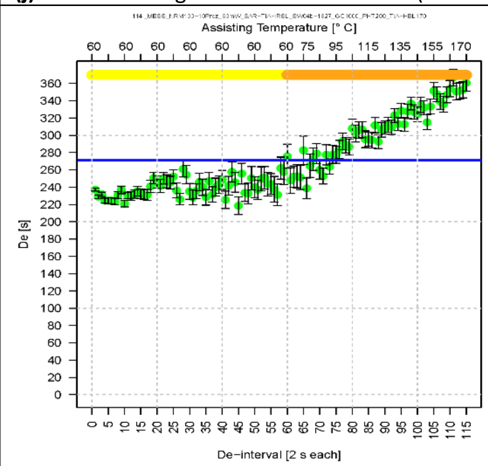
(i) Mean De-integral 102–115: ca 254.7 s (29.98 Gy; 10 ka)



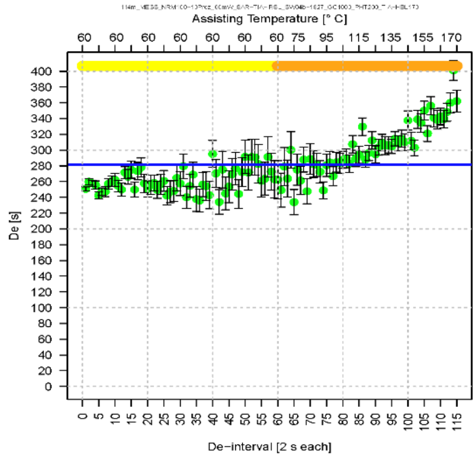
(j) Mean De-integral 101–115: ca 323.1 s (38.1 Gy; 12.7 ka)



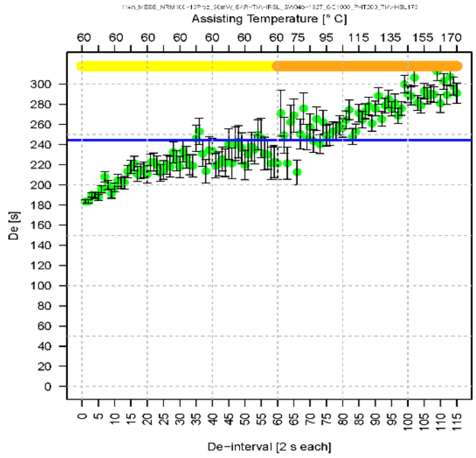
(k) Steady increase up to ca 450 s (53 Gy; 17.7 ka)



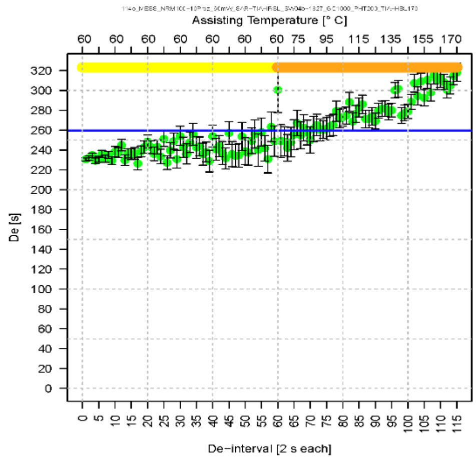
(l) Mean De-integral 105–115: ca 350.1 s (41.3 Gy; 13.8 ka)



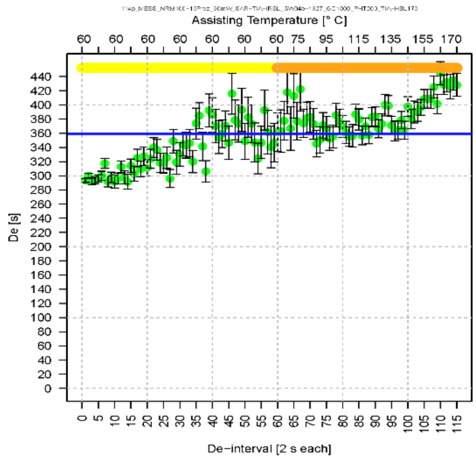
(m) Mean De-integral 105–115: ca 351 s (41.4 Gy; 13.8 ka)



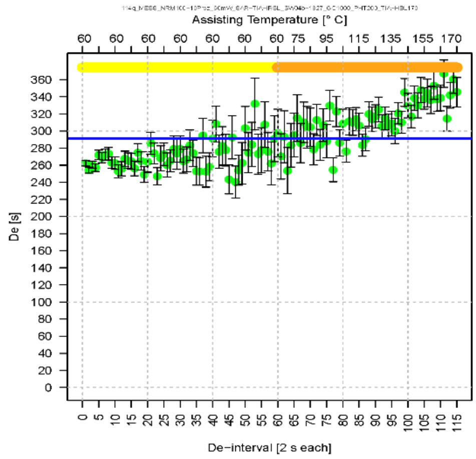
(n) Mean De-integral 99–115: ca 293.5 s (34.6 Gy; 12.7 ka)



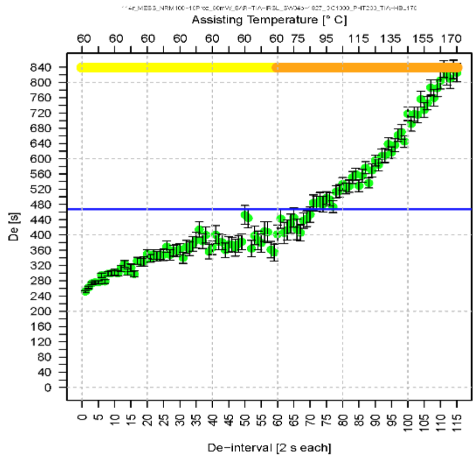
(o) Mean De-integral 102–115: ca 307 s (36.2 Gy; 12.1 ka)



(p) Mean De-integral 108–115: ca 427.7 s (50.4 Gy; 16.8 ka)



(q) Mean De-integral 104–115: ca 343.7 s (40.5 Gy; 13.5 ka)



(r) Up to ca 821 s (mean De-integral 110–115; 96.8 Gy; 32.3 ka)

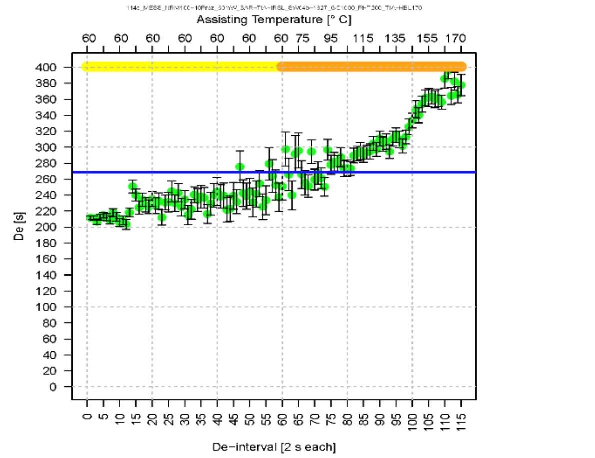
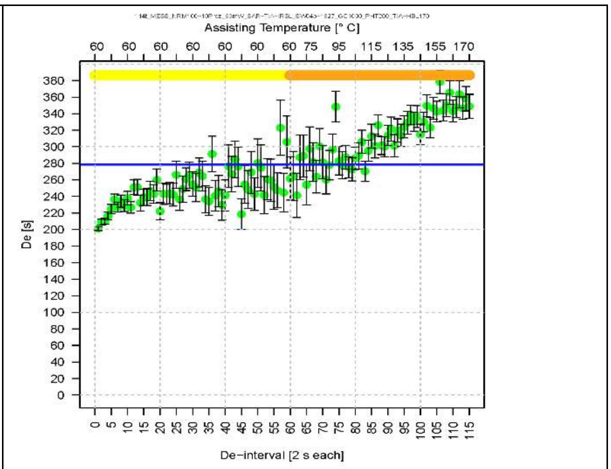
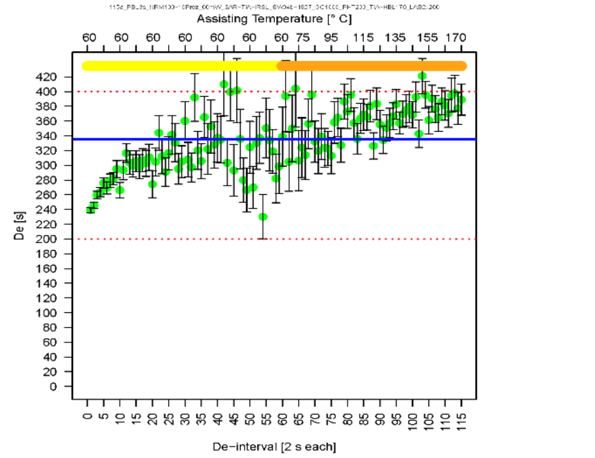
	
(s) Mean De-integral 110–115: ca 378.6 s (44.6 Gy; 14.9 ka)	**(t)** Mean De-integral 104–115: ca 352.9 s (41.6 Gy; 13.9 ka)

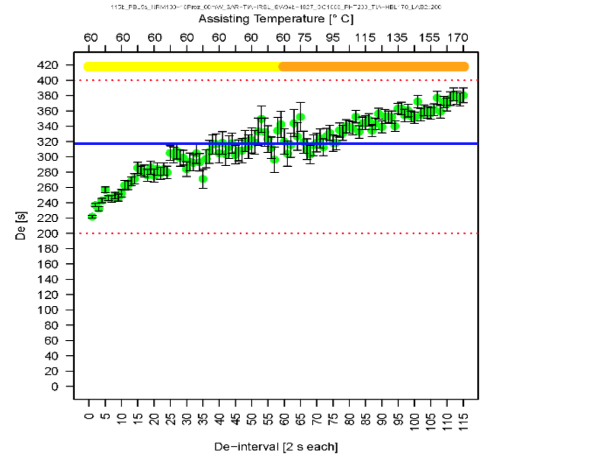
Figure S2.14. MS_114a–114t. PET-IRSL SAR De determination with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. "Classic" measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. Blue line: Mean of all De values. No PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). Aliquots highlighted in yellow chosen for fading tests.

- Ca ages assessed on the assumption of ca 3 Gy/ka palaeoenvironmental dose rate → likely smaller, also due to water content → wait for dose-rate data
- One measurement **(d)** shows a De-value plateau → If dose rate ca 2.5 Gy/ka -> age ca 13.3 ka
- Some measurements **(a, g–j, l–q, s–t)** show a kind of short De-value plateau at the far end of the high-temperature PET-IRSL range (here up to 170 °C)
- Other measurements **(b, c, e, f, k, r)** show a more or less steady increase in the high-temperature PET-IRSL range up to 170 °C
- Generally, apparently insufficient bleaching → as would be expected for sediments from the river bed of the late-glacial (to early-Holocene) Bergstraßenneckar
- What do the conspicuous "noses" at the beginning of the low temperature PET-IRSL range mean? Partial bleaching, fading?
- Perform PBL tests with very modest white-light bleaching

S2.15. PBL test on SW04b-1827 – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR subcycle – GC 1000 s – NRM 10 % of GC-AIM – LAB 2 x 200 s – PHT 200 °C – PET_{max} and HBL 170 °C



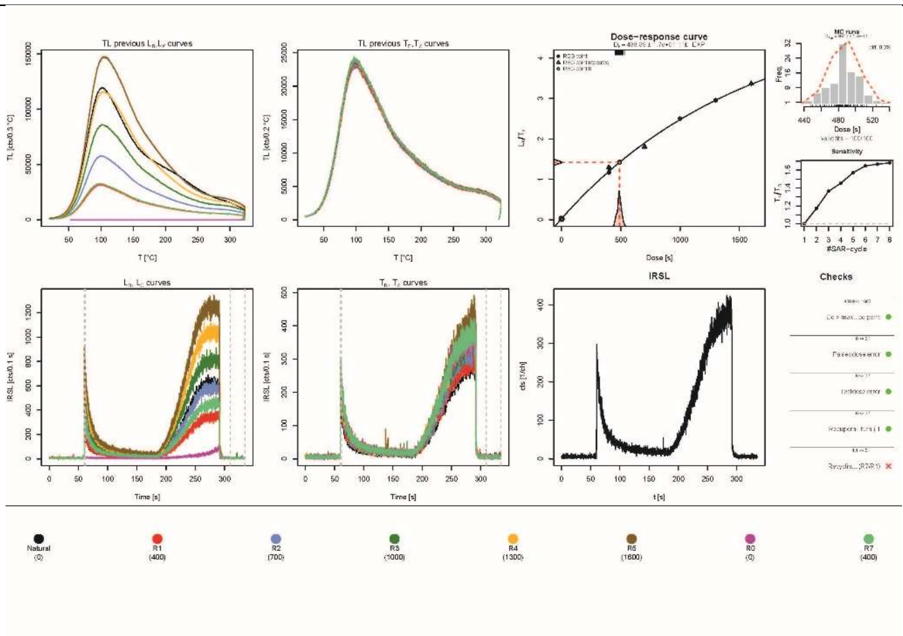
(a) NRM 10 %, LAB 2 x 200 s, PBL 3 s



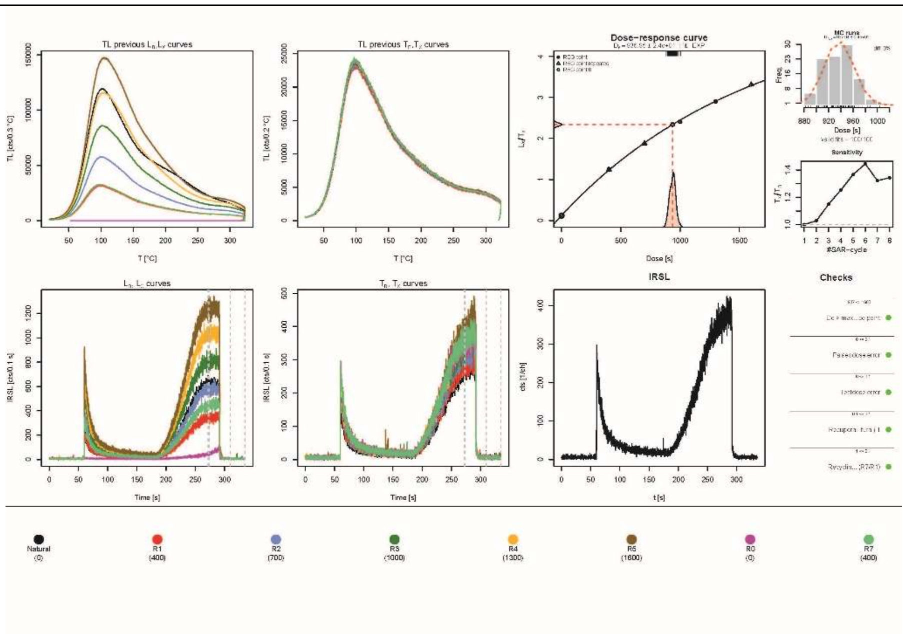
(b) NRM 10 %, LAB 2 x 200 s, PBL 5 s

Figure S2.15. MS_115a–115b. PET-IRSL SAR dose recovery test with DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. "Classic" measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. LAB 2 x 200 s. Partial bleach (PBL) subsequent to first LAB 200 s and TL-PHT at 200 °C **(a)** 3 s solar simulator, **(b)** 5 s solar simulator. Partial doses: Dotted red lines. Blue line: Mean of all De values. No clear PET-peak. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C).

- **(a, b)** Both measurements show the conspicuous "noses" as also observed in most of the measurements on the aliquots with the natural palaeodose. The earliest Des of both measurements slightly overestimate the second applied dose by ca **(a)** 25 %, **(b)** 10 %.
- **(a)** The "hottest" De values (≥ De-integral ca 100) cover the sum of the two applied doses within error margins of the individual De values. **(b)** The De values of the high-temperature PET-IRSL range into an acceptable 10 % error margin of the sum of the two applied doses.
- We tentatively interpret the results that the lowest and highest De values give a minimum age for the last but one and a maximum age for the last sediment reworking admitting that this hypothesis has to be investigated in more detail in future studies. As, however, the easiest bleachable component is usually also the one to fade most strongly, we would also consider it possible that the PET-IRSL De-data curve exhibits more strongly faded (early part) and less strongly faded sections (later part).



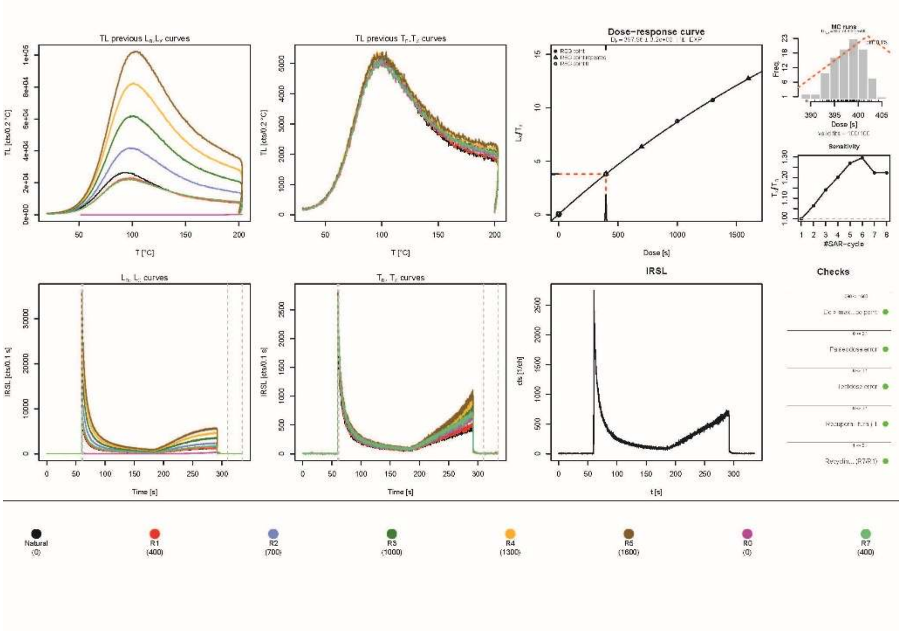
(a)



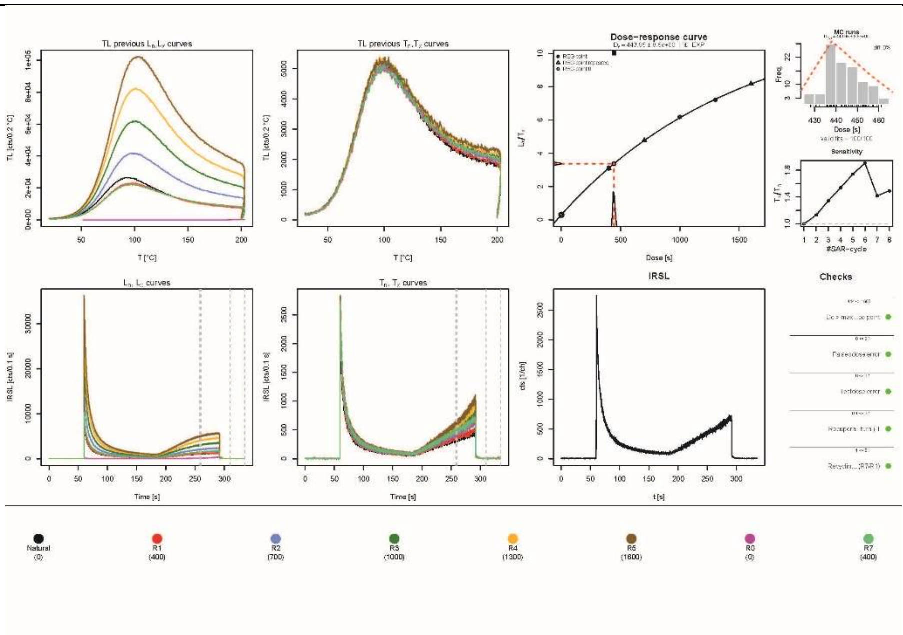
(b)

Figure S2.16. MS_100c2. Dose-recovrey test of sample HDS-1827, small aliquot, 1000 s/120 Gy dose-response curve, NRM 300 s/36 Gy (30 %) (cf. **Fig. S2.1(c-2)**). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 107 (PET-peak). “Too hot” thermal SAR parameter values.

100c2_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_SW04b-1827_PHT320_LAB1000_Peak-I107



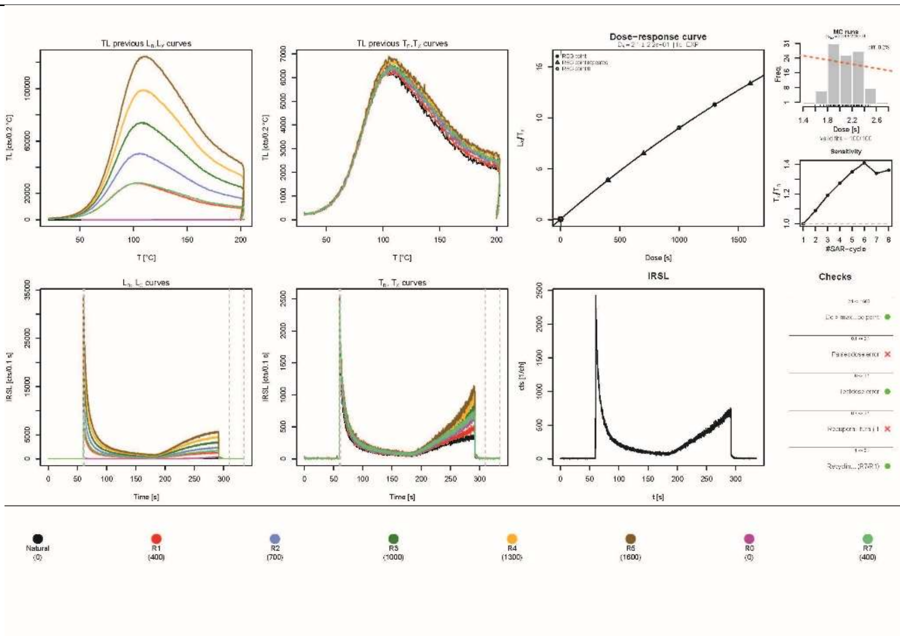
(a)



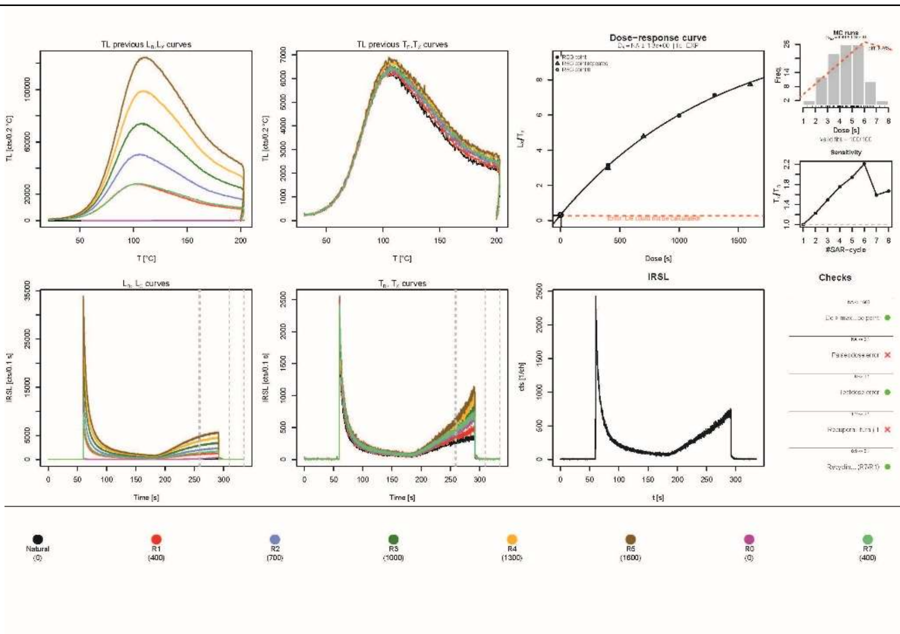
(b)

Figure S2.17. MS_112b. Dose-recoverey test of sample HDS-1827, small aliquot, 1000 s/120 Gy dose-response curve, NRM 100 s/12 Gy (10 %) (cf. **Fig. S2.12b**). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 100 (no PET-peak). Modest thermal SAR parameter values, appropriate for De determination.

112b_DRT_NRM100-10Proz_60mW_SAR-TIA-IRSL_SW04b-1827_GC1000_PHT200_TIA-HBL170_LAB400_No-Peak



(a)



(b)

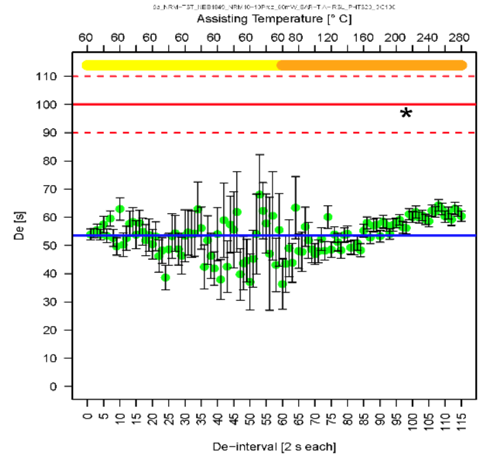
Figure S2.18. MS_111a. Zero-dose test of sample HDS-1827, small aliquot, 1000 s/120 Gy dose-response curve, NRM 300 s/36 Gy (30 %) (cf. **Fig. S2.11(a-1)**). **(a)** Dose determination for De-signal integral 1. **(b)** Dose determination for De-signal integral 100 (no PET-peak). Modest thermal SAR parameter values, appropriate for De determination.

111a_ZERO-LAB_NRM100-10Proz_60mW_SAR-TIA-IRSL_SW04b-1827_GC1000_PHT200_TIA-HBL170_No-Peak

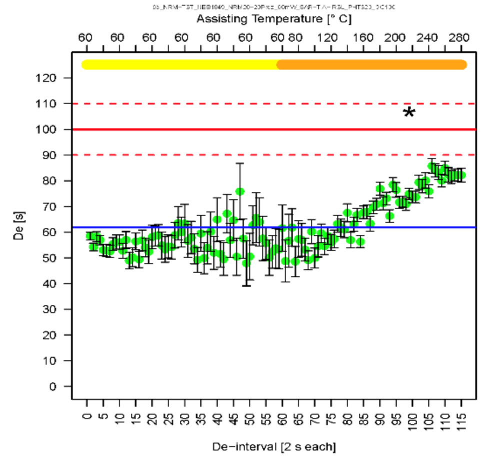
S3. Various PET-IRSL SAR tests and De measurements (MS_9a ff) on sample HDS-1849 on LR04 (COLOUR)

S3.1. NRM DRTs NEB-1849 – PET "classic" – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – LAB 100 s (9a–9j2) and LAB 50 s (10a–10j2), respectively – NRM 10–100 % of LAB increasing in steps of 10 % – for LAB 50 s additionally NRM 7.5 % and 5 % of LAB (10k, l).

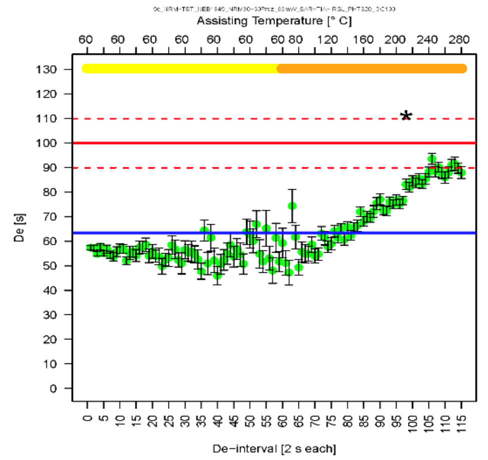
S3.1.1. DRC 100 s – LAB 100 s (9a–9j2)



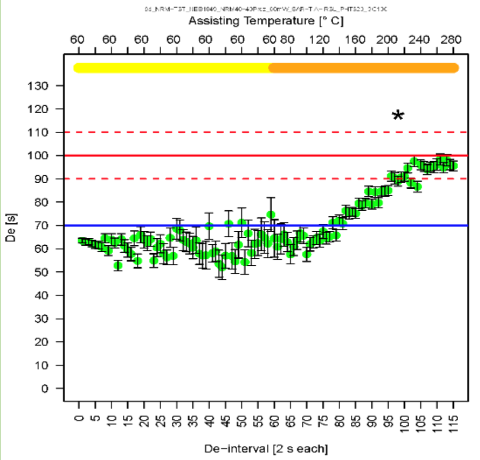
(a) NRM 10 %



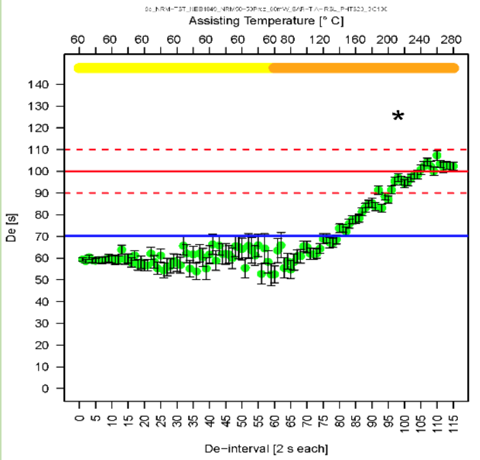
(b) NRM 20 %



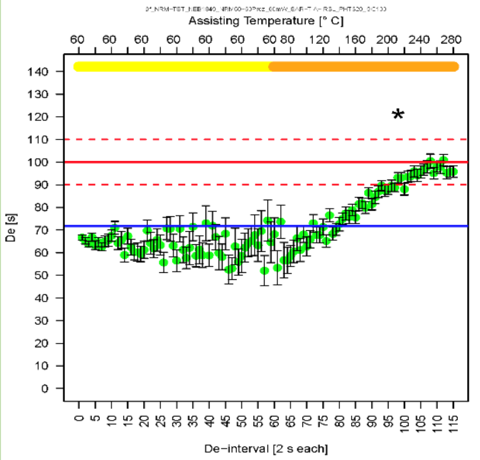
(c) NRM 30 %



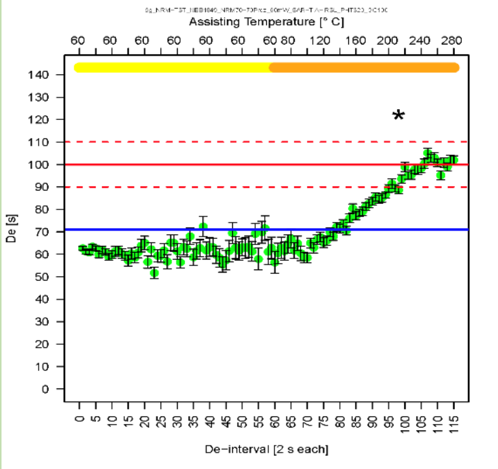
(d) NRM 40 %



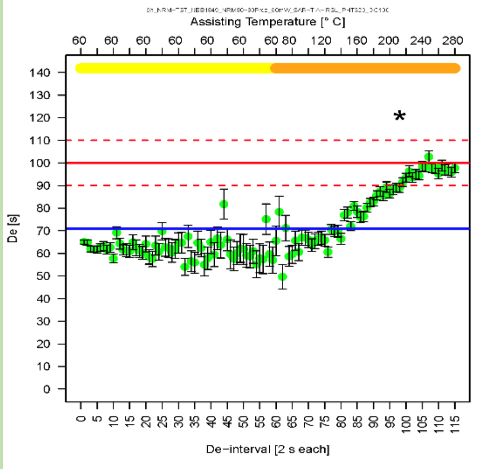
(e) NRM 50 %



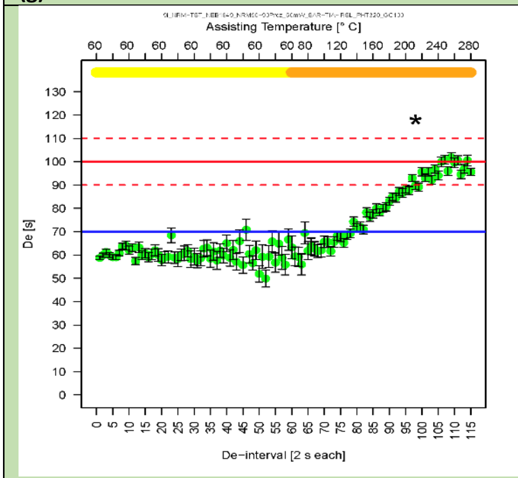
(f) NRM 60 %



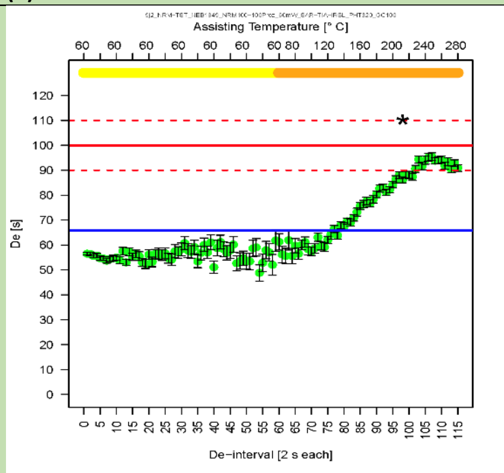
(g) NRM 70 %



(h) NRM 80 %



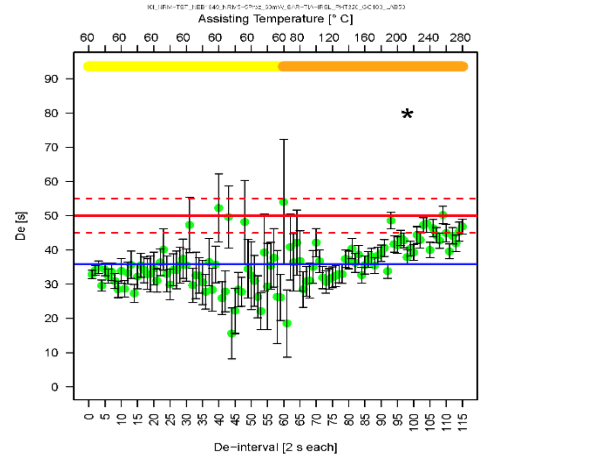
(i) NRM 90 %



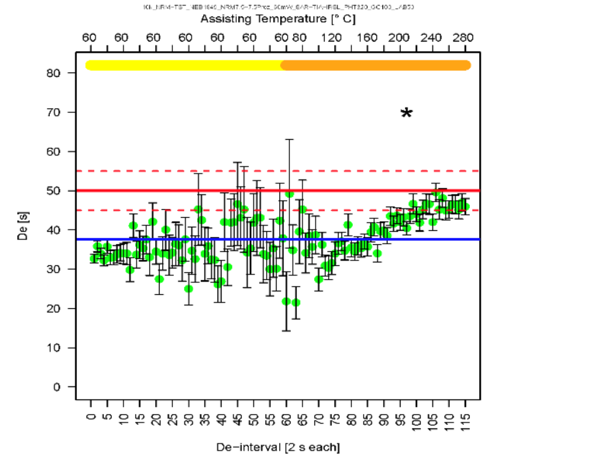
(j) NRM 100 % - Meas 9j2

Figure S3.1. MS_9a–9j2. PET-IRSL SAR NRM DRTs with DRC aiming at 100 s (100 %), LAB 100 s and NRM varying from 10 % to 100 % of LAB. “Classic” measurement parameters: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 98–99). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. “Too hot” thermal SAR parameter values not suitable for De determination.

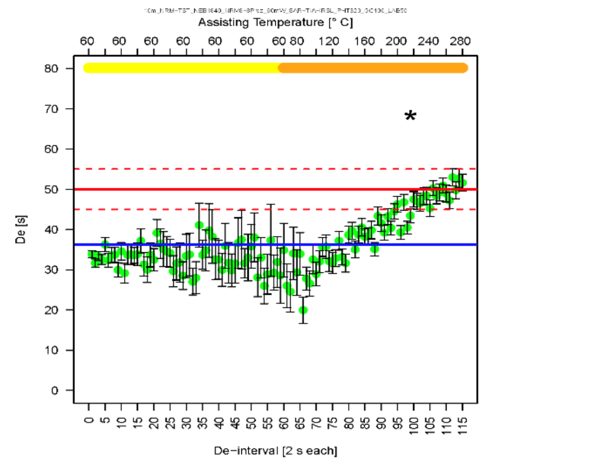
S3.2. DRC 100 s – LAB 50 s (10k–10n + 10a–10j2)



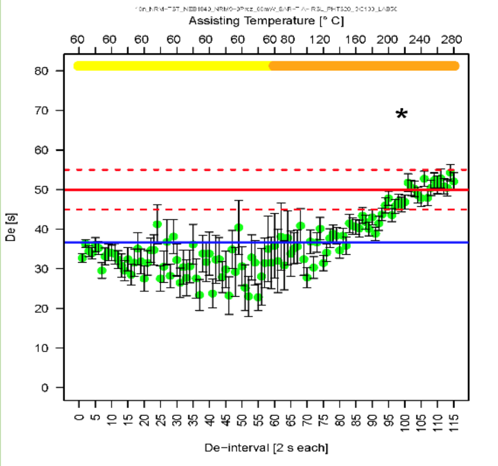
(l) NRM 5 %



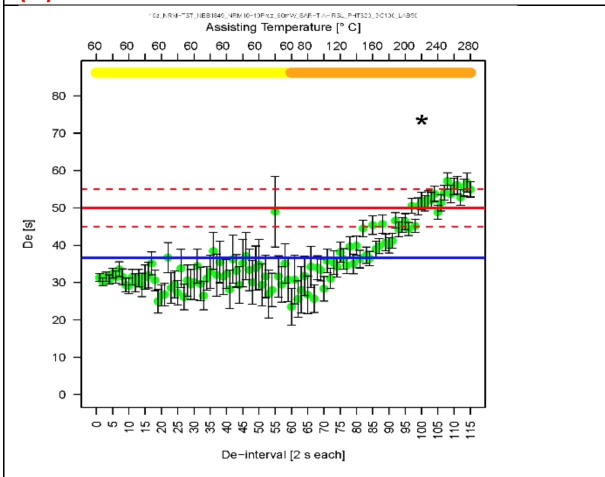
(k) NRM 7.5 %



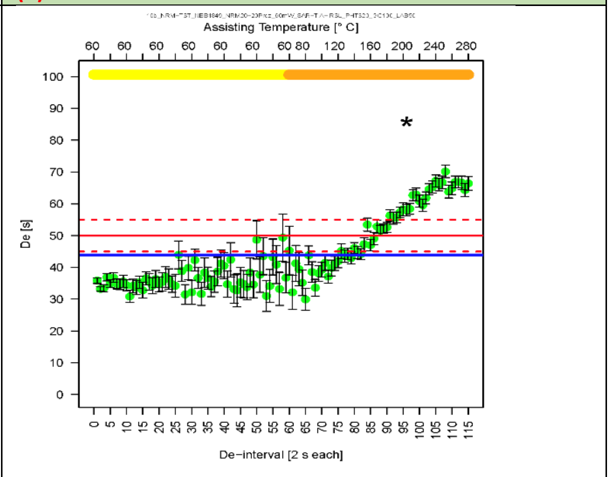
(m) NRM 8 %



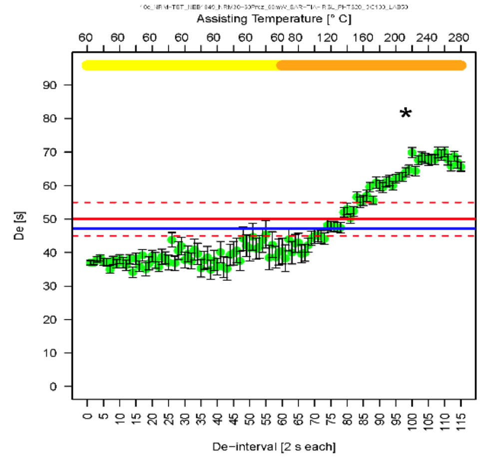
(n) NRM 9 %



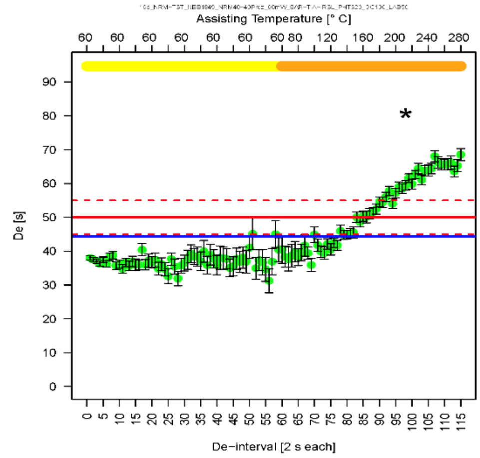
(a) NRM 10 %



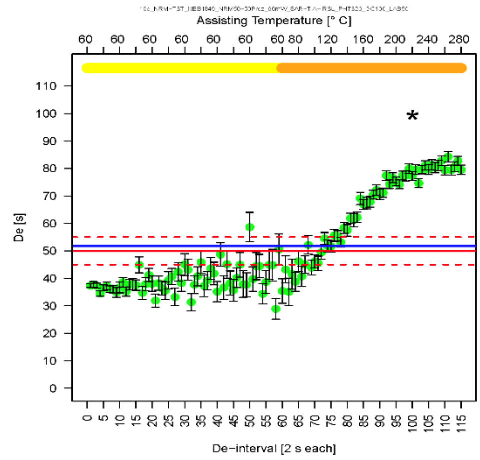
(b) NRM 20 %



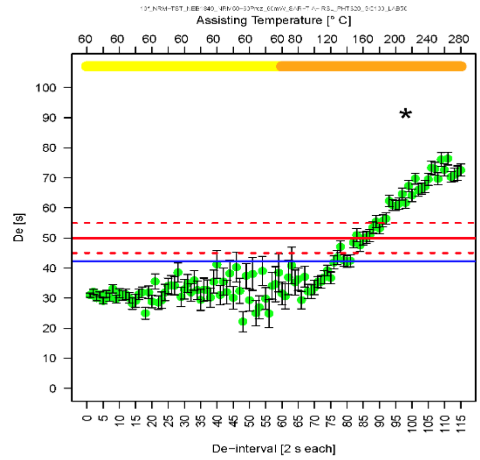
(c) NRM 30 %



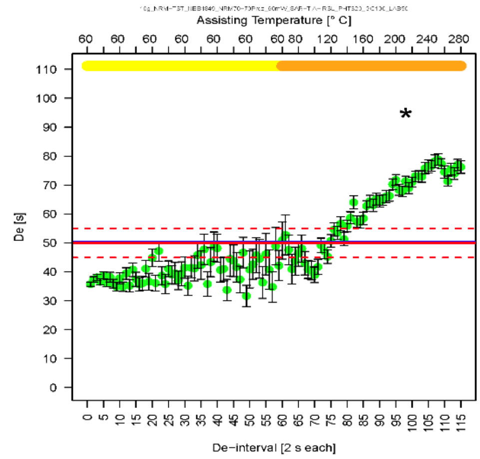
(d) NRM 40 %



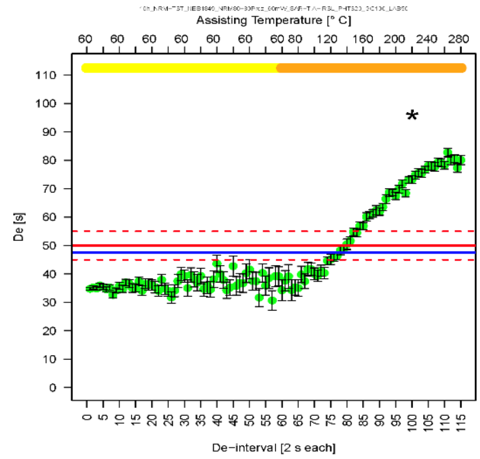
(e) NRM 50 %



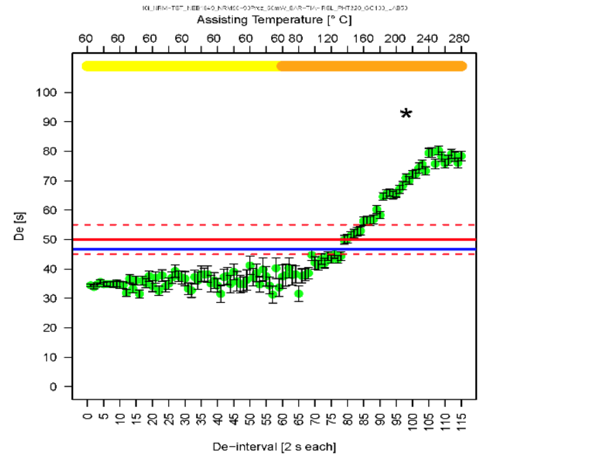
(f) NRM 60 %



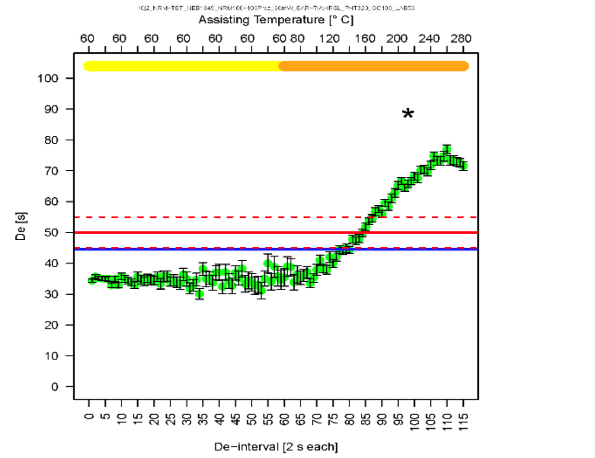
(g) NRM 70 %



(h) NRM 80 %



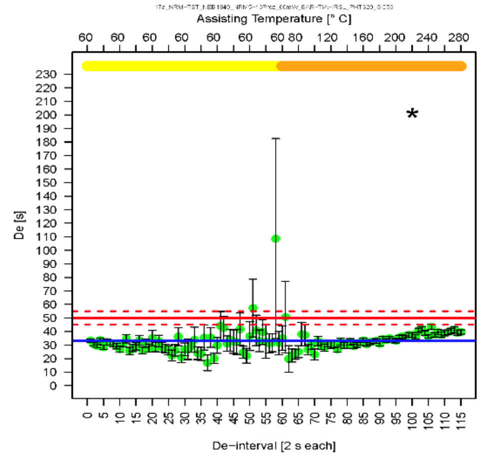
(i) NRM 90 %



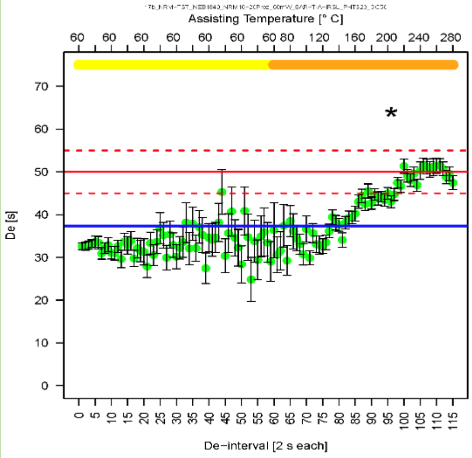
(j) NRM 100 % - Meas 10j2

Fig S3.2. MS_10a-10j, 10k-l, 10m-n. PET-IRSL SAR NRM DRTs with DRC aiming at 100 s (100 %). LAB only 50 s, i.e., half of DRC-AIM, and NRM varying from 10 % to 100 % of DRC-AIM or 20 % to 200 % of LAB, respectively. “Classic” measurement parameters: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. Red line with 10 % error magin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 96–100). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. “Too hot” thermal SAR parameter values not suitable for De determination.

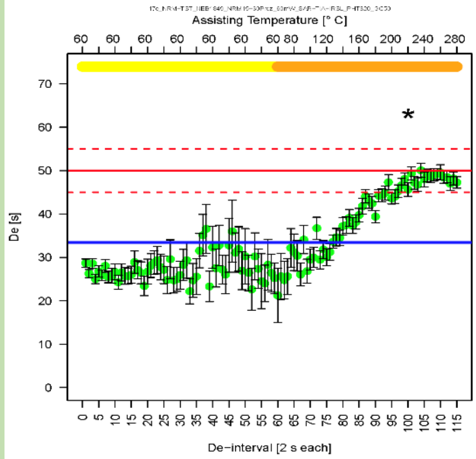
S3.3. NRM DRTs NEB-1849 – PET “classic” – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 50 s – LAB 50 s – NRM 10–50 % of LAB, plus 32 % and 35 % of LAB



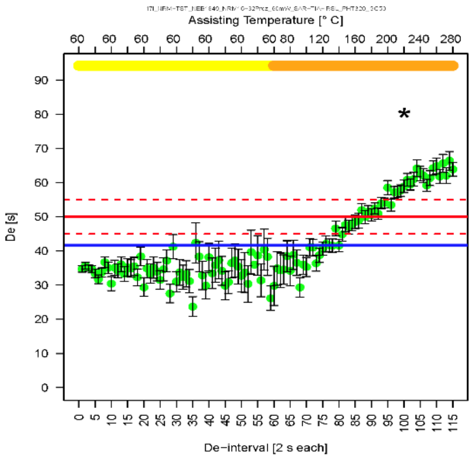
(a) NRM 10 %



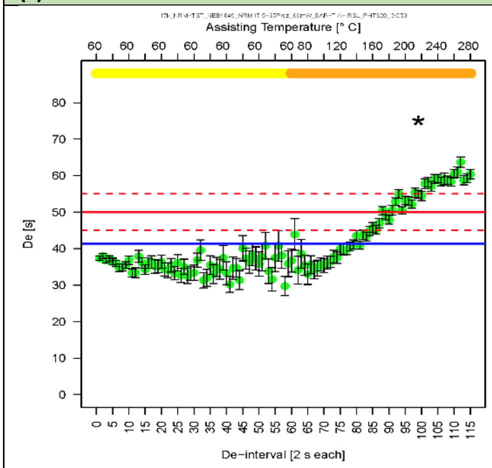
(b) NRM 20 %



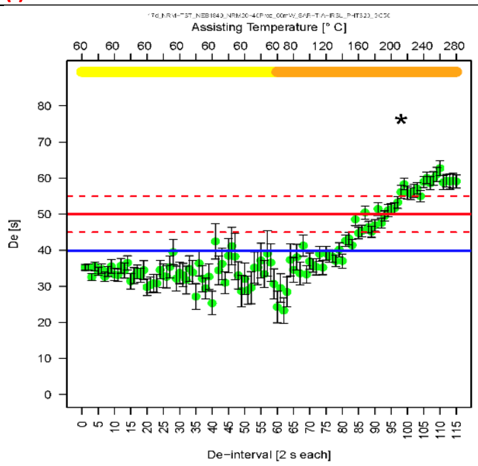
(c) NRM 30 %



(l) NRM 32 %



(k) NRM 35 %



(d) NRM 40 %

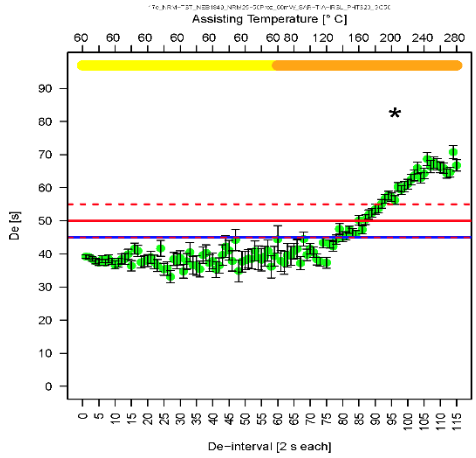
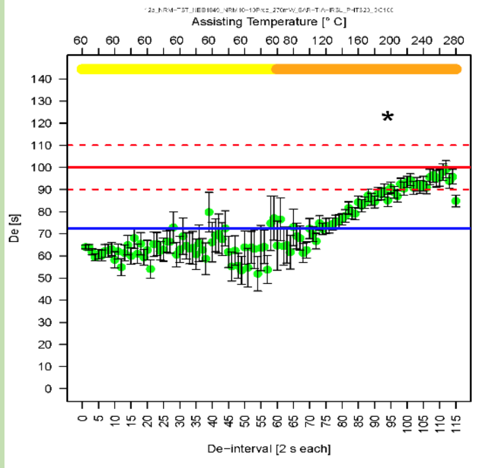
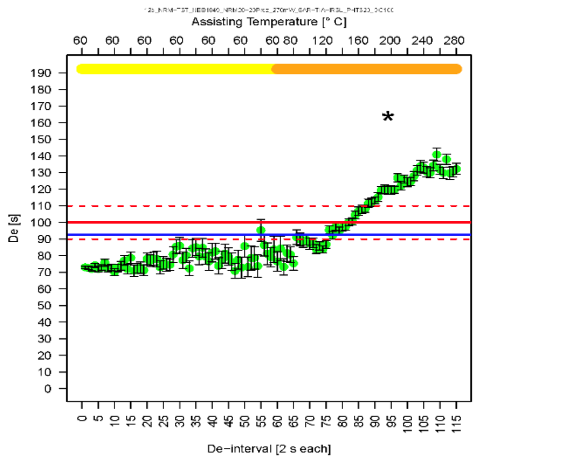
 **(e)** NRM 50 %	

Figure S3.3. MS_17a-17e, plus 17k-l PET-IRSL SAR dose recovery test with DRC aiming at 50 s (100 %), LAB 50 s and NRM varying from 10 % to 50 % of LAB. "Classic" measurement parameters: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: mostly De-signal integral 100). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. "Too hot" thermal SAR parameter values not suitable for De determination.

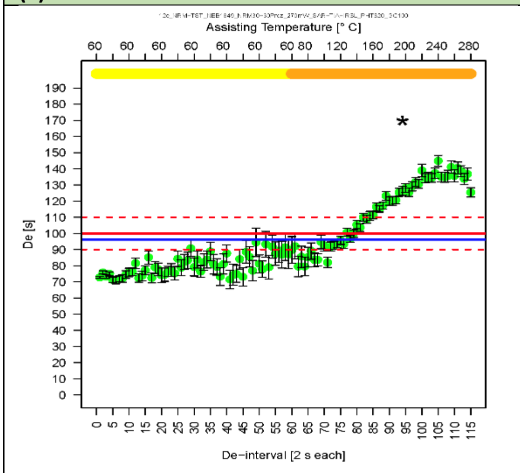
S3.4. NRM DRTs NEB-1849 – PET-IRSL with increased IR diode power – IR 270 mW/cm² – “usual” BLSL hotbleach every SAR half-cycle – DRC 100 s – LAB 100 s – NRM 10–100 % of LAB increasing in steps of 10 %.



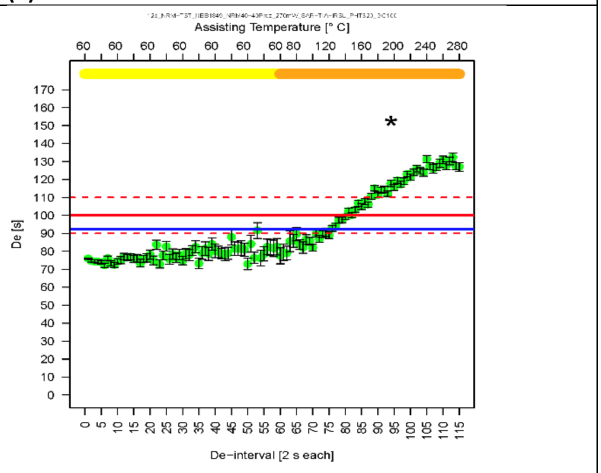
(a) NRM 10 %



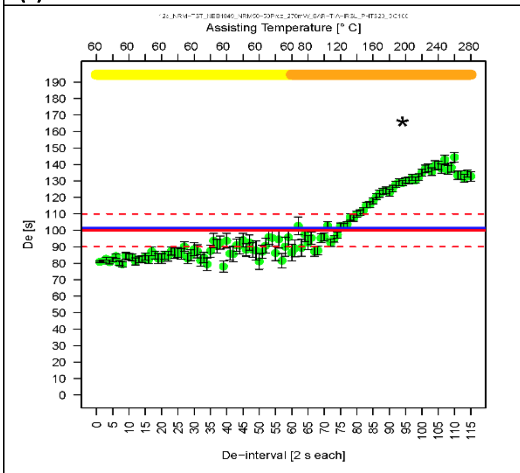
(b) NRM 20 %



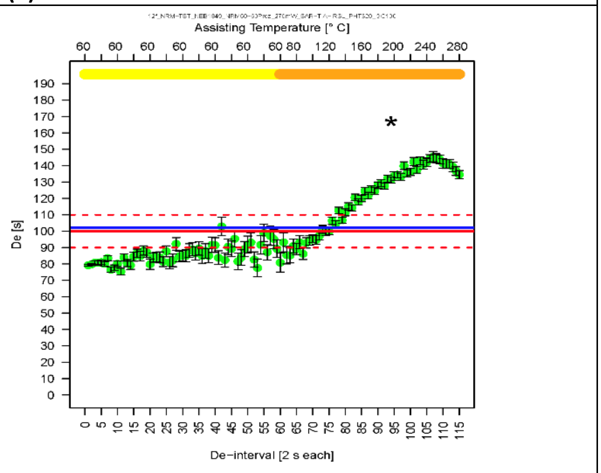
(c) NRM 30 %



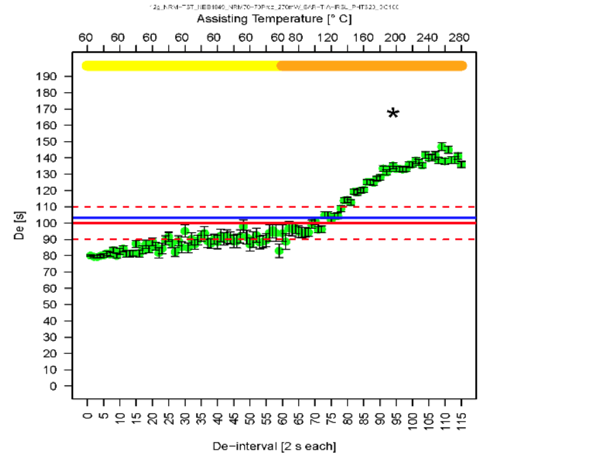
(d) NRM 40 %



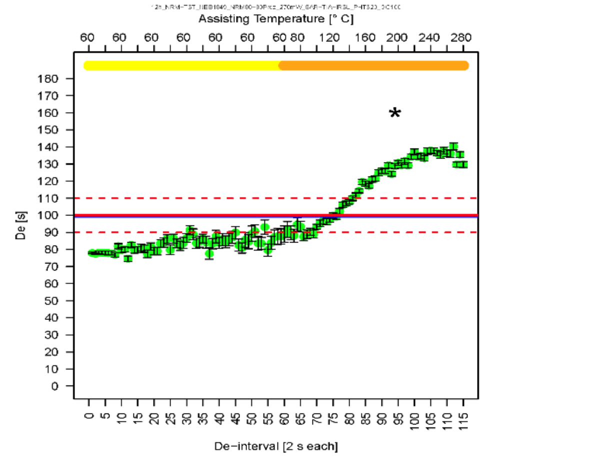
(e) NRM 50 %



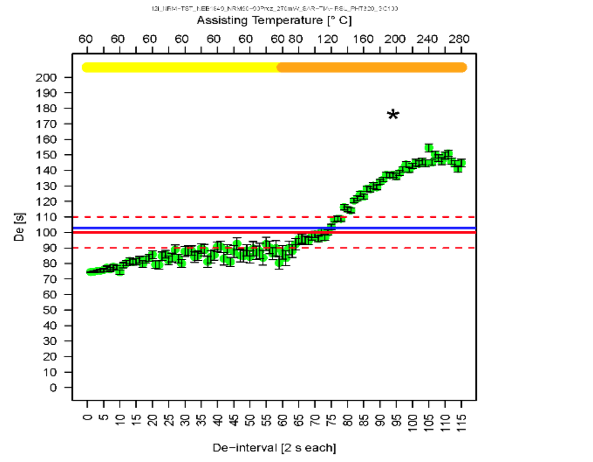
(f) NRM 60 %



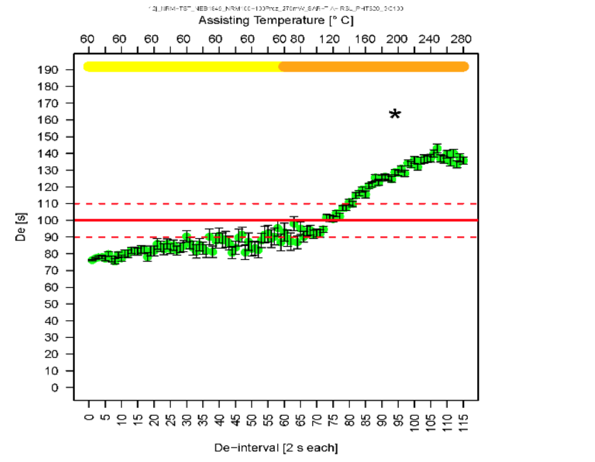
(g) NRM 70 %



(h) NRM 80 %



(i) NRM 90 %



(j) NRM 100 %

Figure S3.4. MS_12a-12j. PET-IRSL SAR dose recovery test with DRC aiming at 100 s (100 %), LAB 100 s and NRM varying from 10 % to 100 % of LAB in steps of 10 %. Measurement parameters differing from the "classic" ones by an <u>increased IR diode power for the PET-IRSL readout.</u> IR 270 mW/cm², BLSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 94). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. "Too hot" thermal SAR parameter values not suitable for De determination.

- The PET-IRSL signal strength at IR 270 mW/cm² is generally larger than for IR 60 mW/cm² and the ratio of the signal strength in the low temperature PET-IRSL range to the signal strength in the high-temperature range is larger
- The position of the PET-IRSL peak is slightly earlier (2–6 De-signal integrals, mostly 4 De-signal integrals) for the stronger IR LED power than for the PET measurements in the "classic" mode
- The administered De is underestimated for all NRMs for the low-temperature PET-IRSL range (by ca 20–30 %), as already observed for the "classic" PET-IRSL measurement and the pIRIR measurement.
- The given dose is recovered for the distal part of the high-temperature PET-peak and beyond for NRM 10 %, whereas it is strongly overestimated in that measurement range for all other NRMs; basically, the pattern compares to that of the "classic" PET-IRSL measurement with DRC-AIM 100 s and LAB 50 %
- For NRM ≥ 20 % the early Des values of the low-temperature PET-IRSL range show a tendency to be smaller than those of the later part of the low-temperature PET-IRSL range, the trend increasing with increasing NRM. This picture of a "sloping" De-value trend is not observed for the DRTs with IR 60 mW/cm²

S3.5. NRM DRTs NEB-1849 – PET with increased IR diode power and IR hotbleach – IR 270 mW/cm² – IRSL hotbleach every SAR half-cycle – DRC 100 s – LAB 100 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 %.

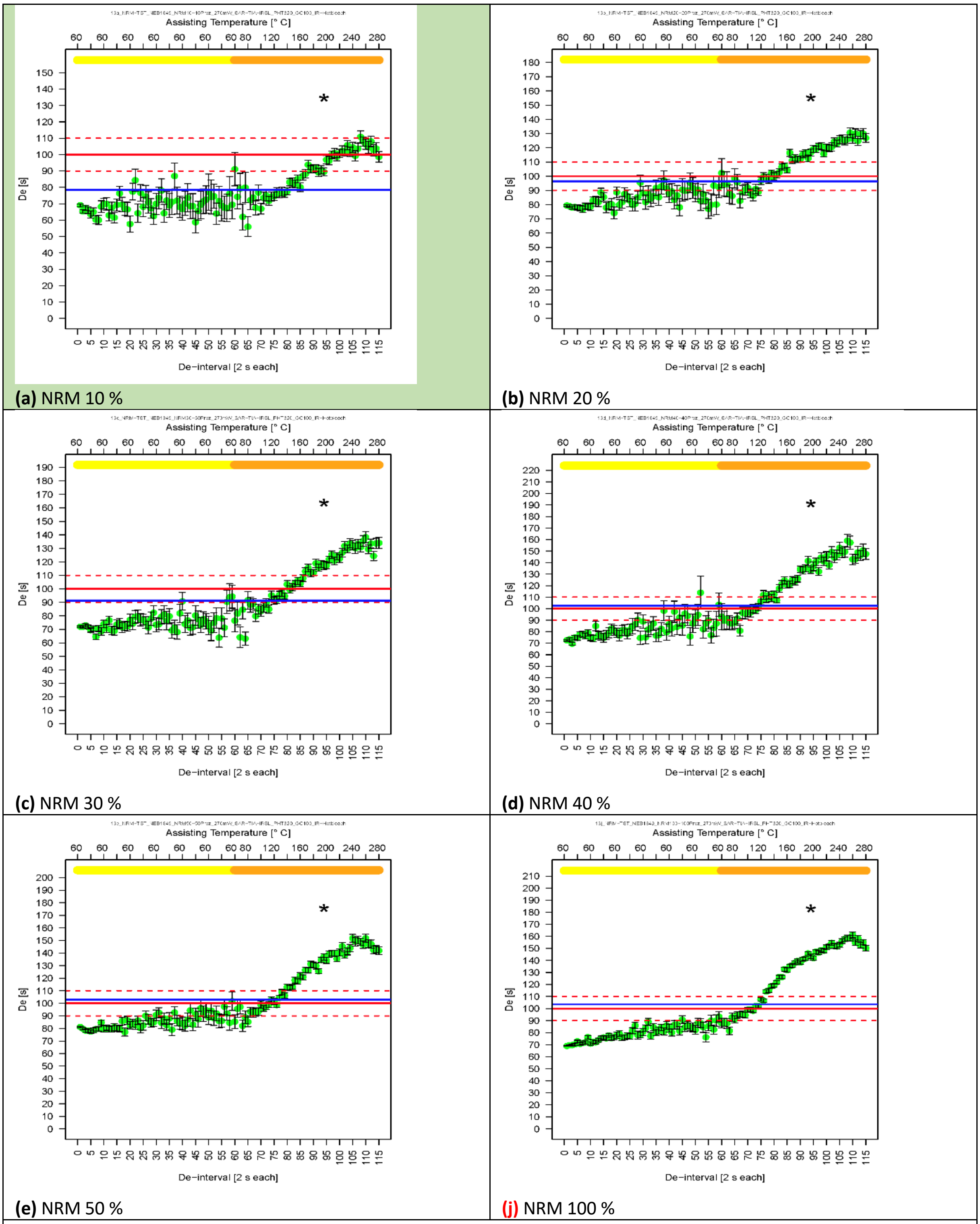
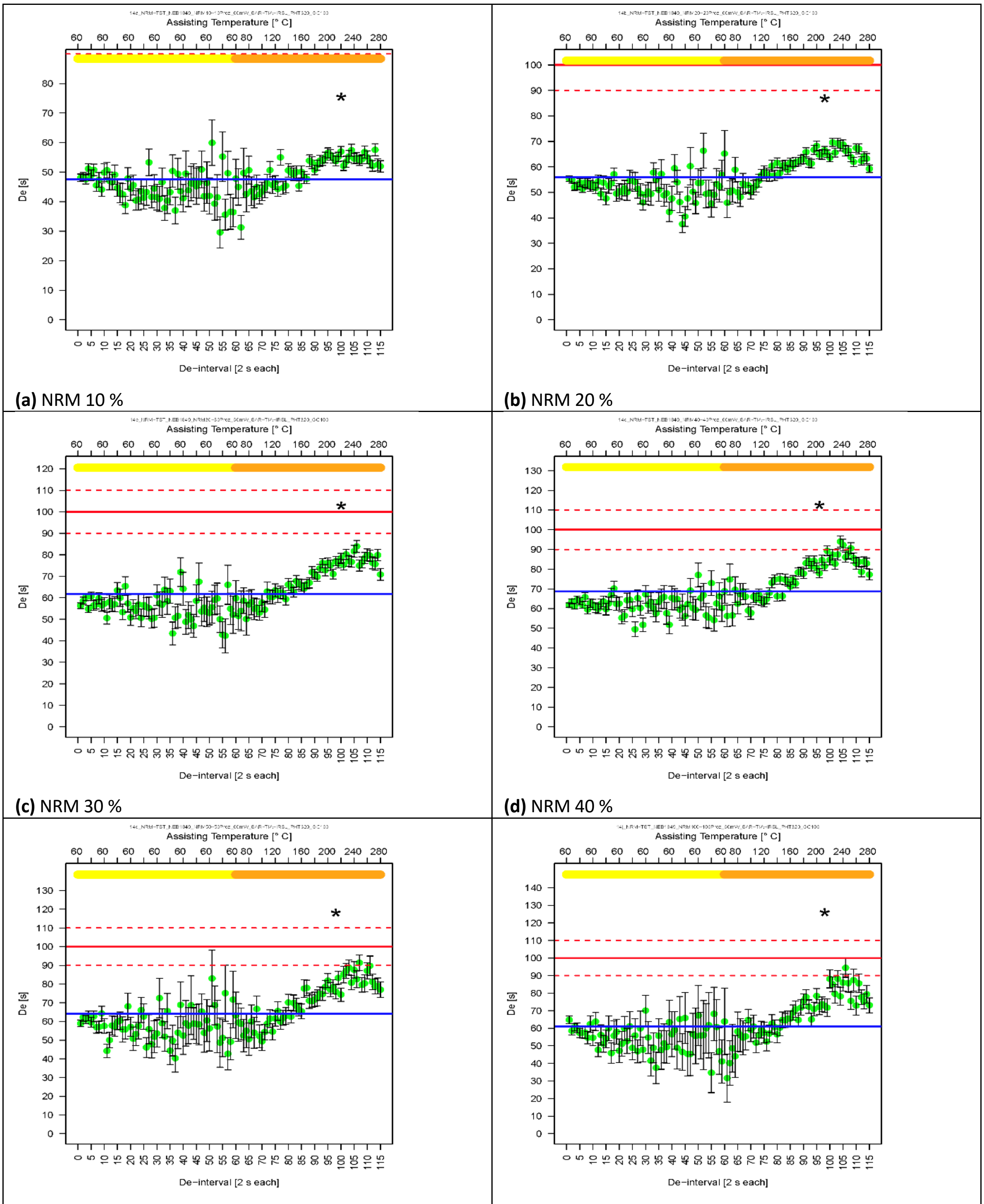


Figure S3.5. MS_13a-13e, 13j. PET-IRSL SAR dose recovery test with DRC aiming at 100 s (100 %), LAB 100 s and NRM varying from 10 % to 50 % of LAB in steps of 10 %, and NRM 100 %. Measurement parameters differing from the "classic" one by an <u>increased IR diode power for the PET-IRSL readout and IRSL hotbleach</u> (instead of BLSL hotbleach): IR 270 mW/cm², IRSL hotbleach in each SAR half-cycle. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 94). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. "Too hot" thermal SAR parameter values not suitable for De determination.

- The results are similar to those of the measurements with LED power 270 mW/cm² and a BLSL hotbleach: Same earlier position of the PET-IRSL peak, underestimation of the given dose in the low temperature PET-IRSL range, correctly recovered De for the distal part of the PET-peak and beyond for NRM 10 %, here, however, towards the upper end of the 10 % error margin whereas towards the lower end of the 10 % error margin for LED power 270 mW/cm² with a BLSL hotbleach. The slight differences may yet be due to inter-aliquot variation.

S3.6. Investigating further variations of hotbleaches / omitting hotbleaches

S3.6.1. NRM DRTs NEB-1849 – PET with "usual" IR diode power and BLSL hotbleach – IR 60 mW/cm² – BLSL hotbleach only after a complete SAR cycle – LAB 100 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 %.



(a) NRM 10 %

(b) NRM 20 %

(c) NRM 30 %

(d) NRM 40 %

(e) NRM 50 %

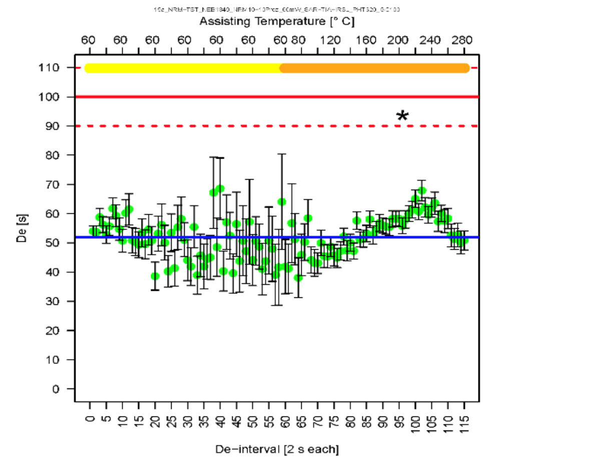
(j) NRM 100 %

Figure S3.6. MS_14a–14e, 14j. PET-IRSL SAR dose recovery test with DRC aiming at 100 s (100 %), LAB 100 s and NRM varying from 10 % to 50 % of LAB in steps of 10 %, and NRM 100 %. Measurement parameters differing from the "classic" ones by omitting the BLSL hotbleach in the first SAR half-cycle: IR 60 mW/cm², <u>BLSL hotbleach only after each completed SAR cycle</u>. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values.

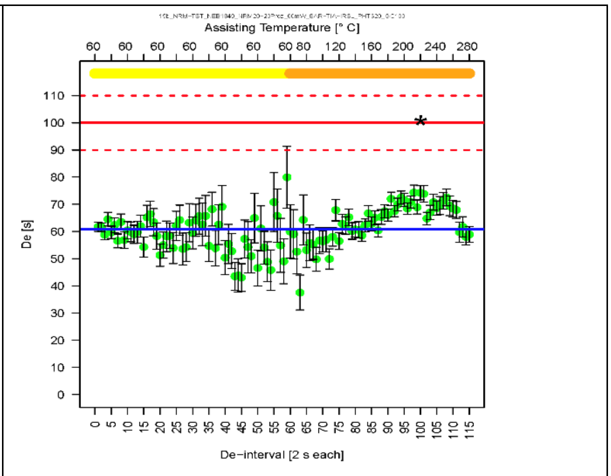
Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 96–100). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. “Too hot” thermal SAR parameter values and omission of hotbleaches in every second half-cycle not suitable for De determination.

- The PET-peak in the hot temperature range occurs again in later positions, as expected for PET-IRSL readout with decreased LED power.
- The omission of the hotbleach in the first SAR half-cycle leads to stronger curved SAR dose response curves (with smaller D0), stronger De underestimation in the low-temperature PET-IRSL range and the underestimation of the expected dose also over the whole range of the high-temperature PET-IRSL readout, including the PET-peak and the distal part beyond.

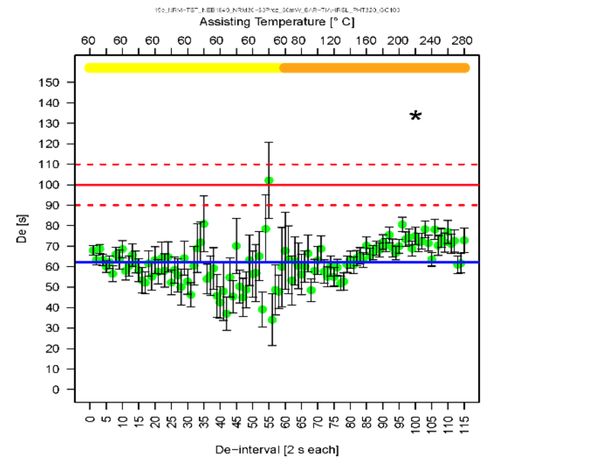
S3.6.2. NRM DRTs NEB-1849 – PET with “usual” IR diode power but no hotbleach – IR 60 mW/cm² – LAB 100 s – NRM 10–50 % of LAB increasing in steps of 10 % and NRM 100 %.



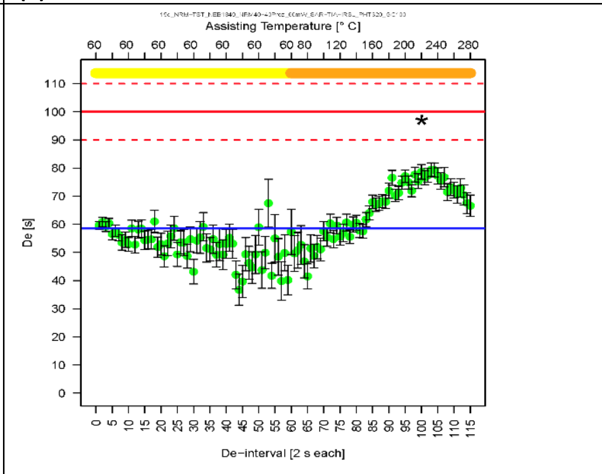
(a) NRM 10 %



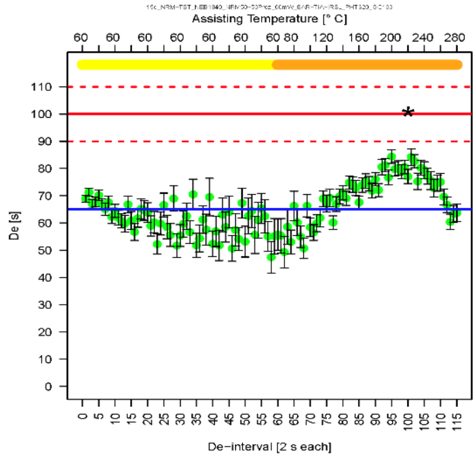
(b) NRM 20 %



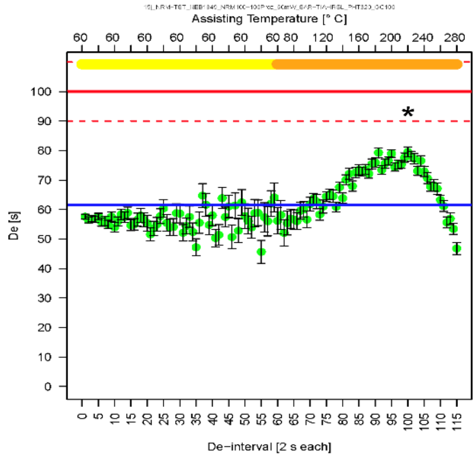
(c) NRM 30 %



(d) NRM 40 %



(e) NRM 50 %



(j) NRM 100 %

Figure S3.7 MS_15a–15e, 15j. PET-IRSL SAR dose recovery test with DRC aiming at 100 s (100 %), LAB 100 s and NRM varying from 10 % to 50 % of LAB in steps of 10 %, and NRM 100 %. Measurement parameters differing from the "classic" ones by omitting the BLSL hotbleach in both SAR half-cycles: IR 60 mW/cm², no hotbleach. Red line with 10 % error margin (dashed red lines): Expected De-value range. Blue line: Mean of all De values. Star signature: Approximate position of high-temperature PET-peak (here: De-signal integral 96–100). Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–280 °C, ramp 2 K/s). All measurements up to PET_{max} 280 °C and with HBL 280 °C after PHT 320 °C. "Too hot" thermal SAR parameter values and omission of hotbleaches in every half-cycle not suitable for De determination.

- The PET-peak in the high-temperature range occurs again in later positions, as expected for PET-IRSL readout with decreased LED power.
- The omission of both BLSL hotbleaches leads to stronger curved SAR dose response curves (with smaller D0), stronger De underestimation in the low-temperature PET-IRSL range and the underestimation of the expected dose also over the whole range of the high-temperature PET-IRSL readout, including the PET-peak and the distal part beyond
- In addition, this variant of PET-IRSL measurement without any hotbleaches leads to unwanted, increased recuperation and, in most cases, to a decline of De values beyond the high-temperature PET-IRSL signal peak.

S3.7. Extra table

Table S3.1 Overview of the NRM DRTs on sample NEB-1849 with "classical", increased and decreased energy of OSL readout/electron washout.

Measurement series	GC-AIM [s]	LAB [s]	TIA	Hotbleach				Mode	Relative power level*
			IR LED power [mW]	LED type	LED power [mW]	each SAR subcycle	end of SAR cycle		
9a - 9j2	100	100	60	BLSL	90	x		"classic"	0
10a - 10j2	100	50	60	BLSL	90	x		"classic"	0
12a - 12j	100	100	270	BLSL	90	x		increased TIA readout	+ 1
13 a - 13e, 13j	100	100	270	IRSL	270	x		increased TIA readout + IRSL hotbleach	+ 2
14a - 14e, 14j	100	100	60	BLSL	90		x	only 1 hotbleach per SAR cycle	- 1
15a - 15e, 15j	100	100	60	NA	NA	NA	NA	no hotbleach	- 2

* with reference to the electron washout

S3.8. NRM DRTs on NEB-1849 – PET "classic" – IR 60 mW/cm² – BLSL hotbleach at 280 °C every SAR half-cycle – DRC 100 s – LAB 100 s – NRM 10–50 % of LAB and 100 % of LAB – PHT only 60 s at 200 °C (cf. Buckland et al. 2019) and PET-IRSL readout up to 170 °C (therefore no PET-peak)

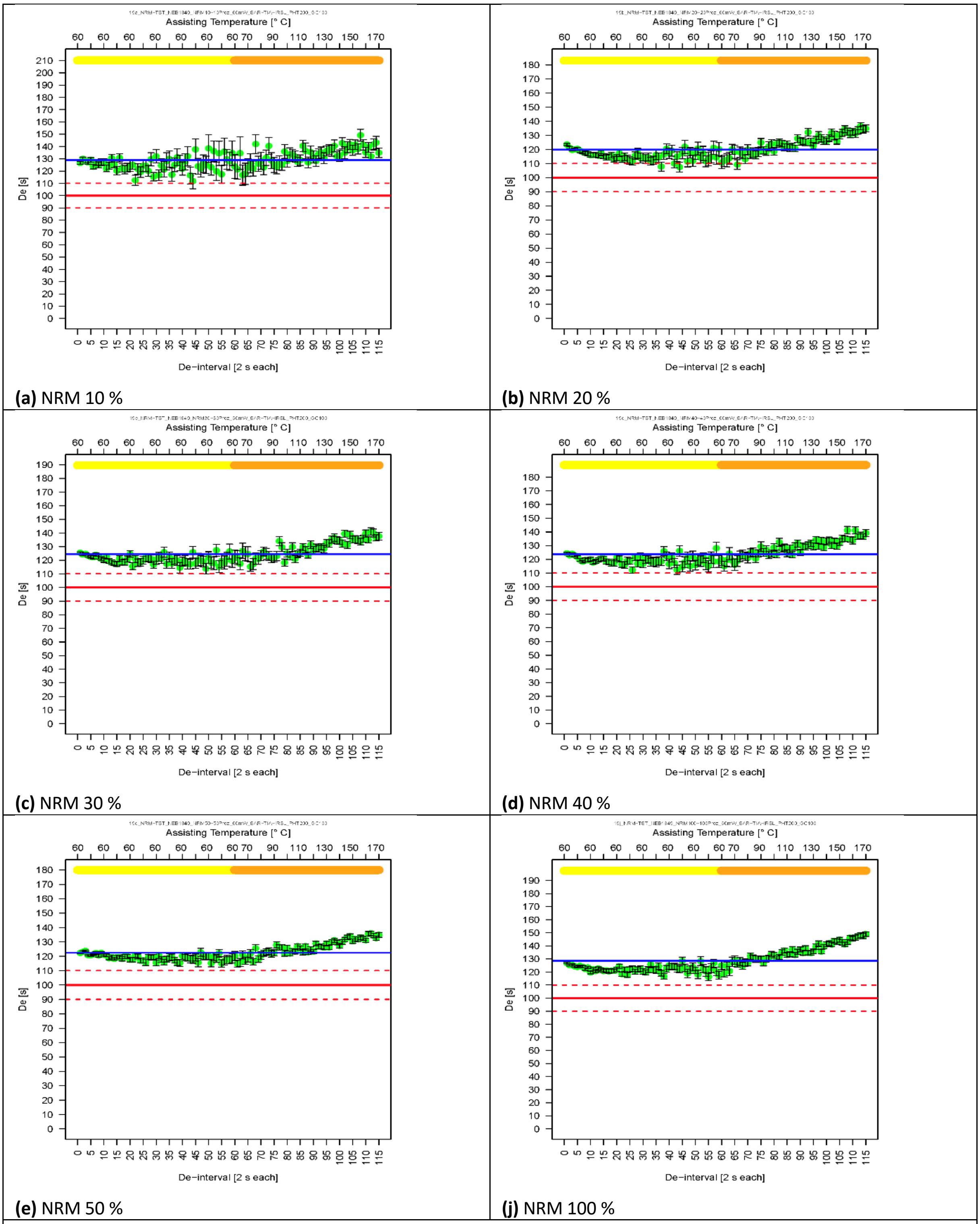
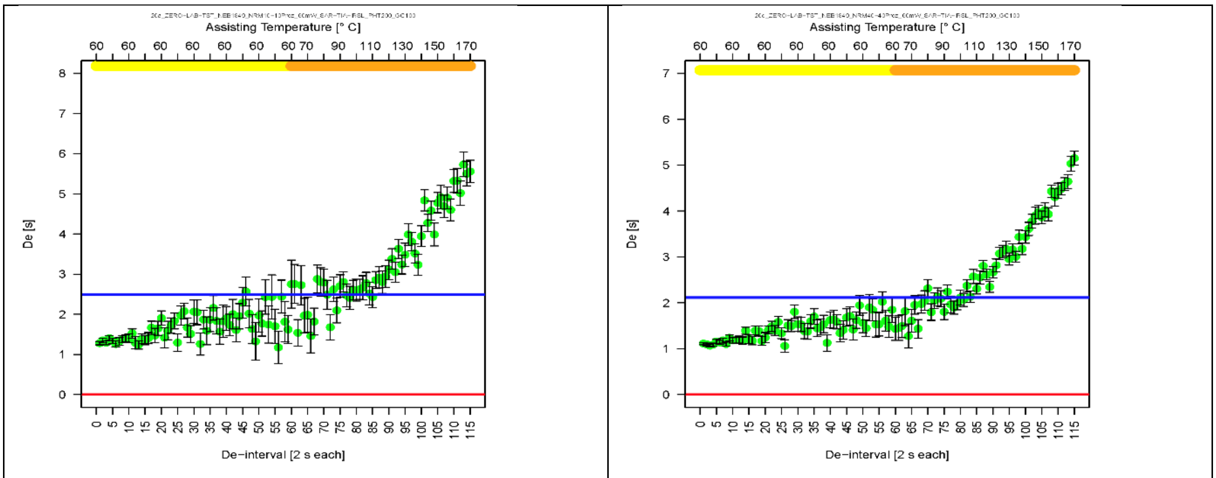


Figure S3.8. MS_19a – 19e, 19j. PET-IRSL SAR dose recovery test with DRC aiming at 100 s (100 %), LAB 100 s and NRM increasing 10– 50 % of LAB in steps of 10 %, and 100 % of LAB. IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle (conforming to "classic" PET). But preheat only 60 s at 200 °C (following Buckland et al., 2019) and PET-IRSL readout only up to 170 °C (therefore no PET-peak observed). Please note that the PHT is at 280 °C as suggested by Buckland et al. (2019). "Too hot" SAR parameter value for the hotbleach not suitable for De determination.

- LAB is overestimated by ca 20 % in the low-temperature PET-IRSL range and by up to 40 % at the upper end of the high-temperature PET-IRSL range
- Next step: ZERO-LAB DRT with NRM 10 % and NRM 40 % to investigate whether unbleachable remnant dose may explain the overestimation and may possibly be subtracted

S3.9. ZERO-LAB-DRTs on NEB-1849 – PET "classic" – IR 60 mW/cm² – BLSL hotbleach at 280 °C every SAR half-cycle – GC 100 s – LAB 0 s – NRM 10 % and 40 % of LAB – PHT only 60 s at 200 °C (cf. Buckland et al. 2019) and PET-IRSL readout up to 170 °C (therefore no PET-peak)

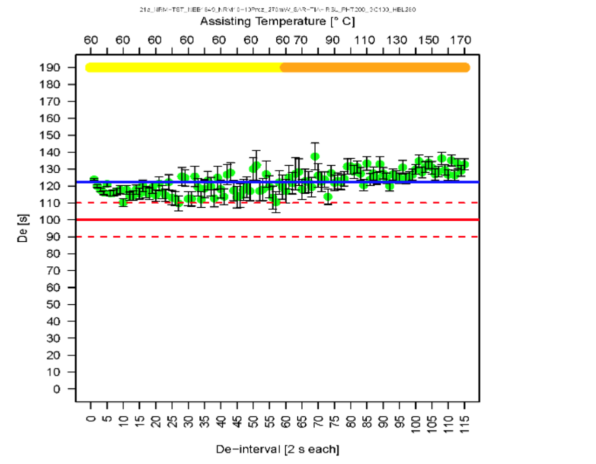


(a) NRM 10 % **(d)** NRM 40 %

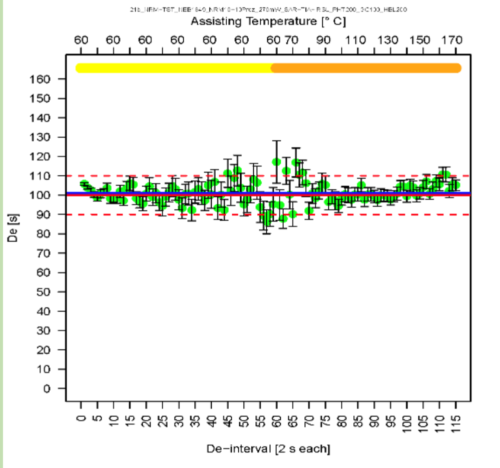
Figure S3.9. MS_20a, 20d. PET-IRSL SAR ZERO-LAB dose recovery test with GC aiming at 100 s (100 %), LAB 0 s and NRM 10 % and 40 % of LAB. IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle (conforming to "classic" PET). But preheat only 60 s at 200 °C (following Buckland et al. 2019) and PET-IRSL readout only up to 170 °C (therefore no PET-peak observed). "Too hot" thermal SAR parameter value for the hotbleach not suitable for De determination.

- The remnant dose of up to ca 5–5.5 s at the upper end of the high-temperature PET-IRSL range (here up to 170 °C) corresponding to 0.53 – 0.59 Gy conforms by size to the remnant dose observed by Buckland et al. 2019 after 1.5 hours of bleaching and applying a pIRIR SAR protocol
- Yet a residual of such a small size cannot explain the overestimation of LAB in the tests 19a–19j, neither in the low-temperature nor in the high-temperature PET-IRSL range

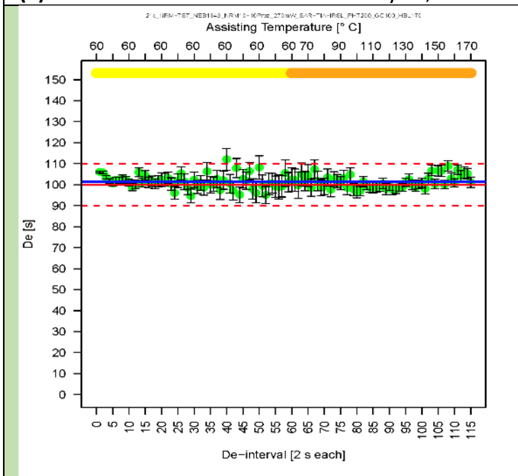
S3.10. DRTs on NEB-1849 – GC 100 s – LAB 100 s NRM 10 % of GC/LAB – PET “classic” with IR 60 mW/cm² and PET with increased IR power of 270 mW/cm² – BLSL hotbleach at varying temperatures every SAR half-cycle – PHT 60 s at only 200 °C (cf. Buckland et al. 2019) and PET-IRSL readout up to 170 °C (therefore no PET-peak) – BLSL HBL either at 280 °C (cf. Buckland et al. 2019), 200 °C (like PHT) or 170 °C (maximum PET-IRSL readout temperature)



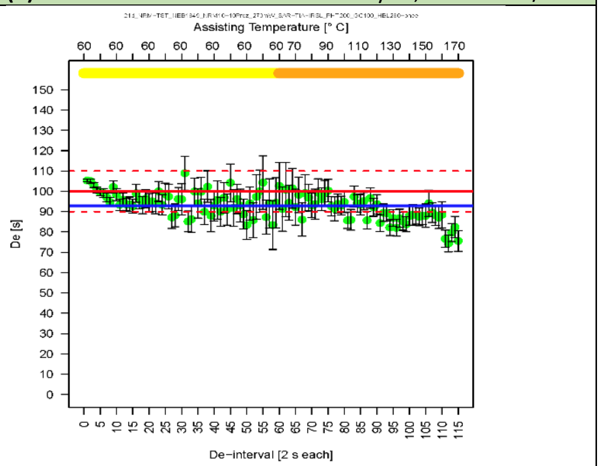
(a) BLSL HBL at 280 °C each SAR half-cycle, IR 270 mW/cm²



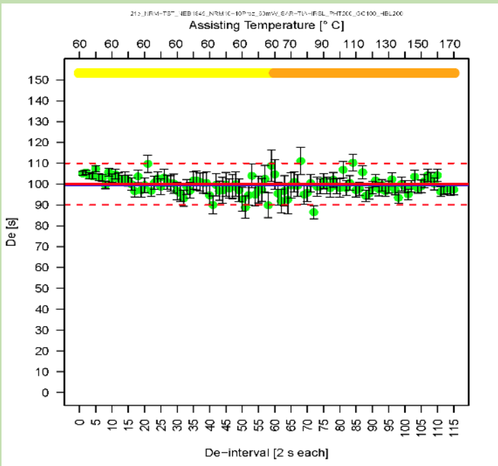
(b) BLSL HBL at 200 °C each SAR half-cycle, IR 270 mW/cm²



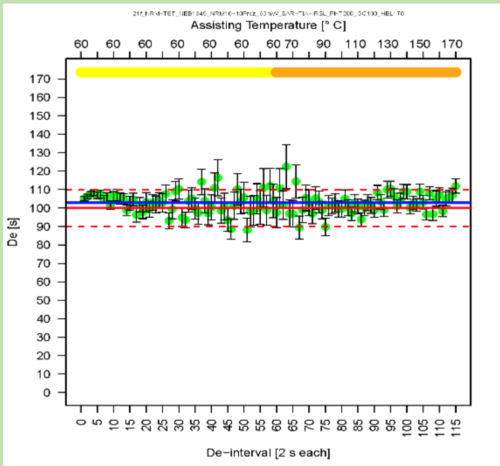
(c) BLSL HBL at 170 °C each SAR half-cycle, IR 270 mW/cm²



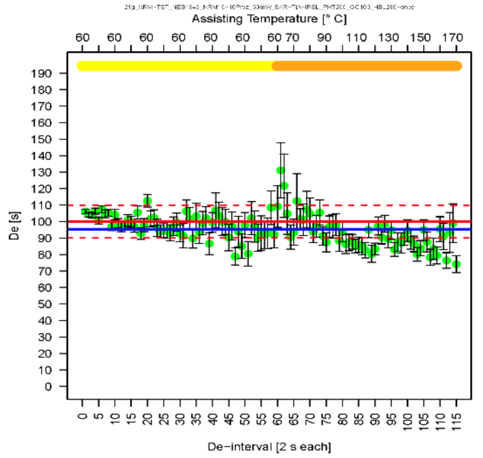
(d) BLSL HBL at 280 °C end of each SAR cycle, IR 270 mW/cm²



(e) Like **(b)** but IR 60 mW/cm²



(f) Like **(c)** but IR 60 mW/cm² – Fig. 6d in main text



(g) Like **(d)** but IR 60 mW/cm²

Figure S3.10. MS_21a–21g. PET-IRSL SAR dose recovery tests with DRC aiming at 100 s (100 %), LAB 100 s and NRM 10 % of LAB. Preheat only at 200 °C (following Buckland et al. 2019) but for 60 s (instead of 10 s, like Buckland et al. 2019) and PET-IRSL readout only up to 170 °C (therefore no PET-peak observed). **(a–d**) High IR-energy density IR 270 mW/cm². **(e–g)** "Classic" PET-IRSL readout with IR 60 mW/cm².

Varying BLSL hotbleach modes:

(a) In each SAR half-cycle at 280 °C (cf. "classic" PET), IR 270 mW/cm²

(b) In each SAR half-cycle at 200 °C (comparing to the PHT TL temperature), IR 270 mW/cm²

(c) In each SAR half-cycle at 170 °C (comparing to the maximum PET-IRSL readout temperature), IR 270 mW/cm²

(d) At the end of a SAR cycle at 280 °C (almost like Buckland et al. 2019, who performed an IR HBL at 290 °C), IR 270 mW/cm²

(e) In each SAR half-cycle at 200 °C (comparing to the PHT TL temperature), IR 60 mW/cm²

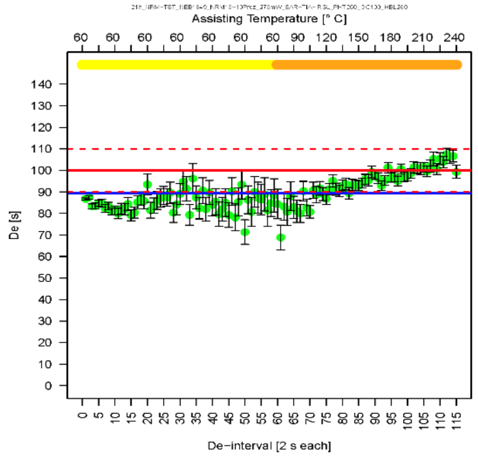
(f) In each SAR half-cycle at 170 °C (comparing to the maximum PET-IRSL readout temperature), IR 60 mW/cm²

(g) At the end of a SAR cycle at 280 °C (almost like Buckland et al. 2019, who performed an IR HBL at 290 °C), IR 60 mW/cm².

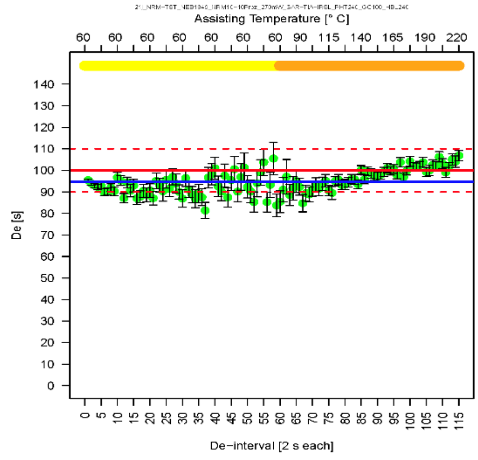
- **(a)** Like for IR 60 mW/cm², with a strong BLSL hotbleach at 280 °C, LAB is overestimated – though to a slightly lower degree – by ca 15 % in the low-temperature PET-IRSL range and by up to ca 30 % at the upper end of the high-temperature PET-IRSL range
- **(b, c)** LAB is perfectly recovered over the complete PET-IRSL temperature range (here up to 170 °C) for both BLSL HBLs at 200 °C comparing to the PHT TL and 170 °C comparing to the highest PET-IRSL temperature
- **(d)** The strong BLSL HBL at the end of a complete SAR cycle leads to a slight underestimation when considering the mean De, and a stronger underestimation especially in the high-temperature PET-IRSL range above ca 120 °C. In that temperature range the dose-response curves show – comparatively – strong curvature with corresponding low D0 values
- **(e, f)** Like **(b)** and **(c)**, both with IR 270 mW/cm², the IR 60 mW/cm² PET-IRSL SAR DRTs with a BLSL hotbleach at the end of each SAR half-cycle at 200 °C, comparing to the PHT temperature, and at 170 °C, comparing to the highest PET-IRSL readout temperature, recover LAB over the whole range of the PET-IRSL readout
- **(g)** Like **(d)** unwanted strong bending of the dose-response curve especially for the high temperature PET-IRSL range, with correspondingly small D0-values. Also like **(d)** tendency to underestimate LAB in the high temperature PET-IRSL range, starting at ca 100 °C.

S3.11. DRTs on NEB-1849 – PET with increased IR power – varying PHT/HBL temperatures and corresponding maximum PET temperatures – IR 270 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – LAB 100 s – NRM 10 % of LAB. PHT and BLSL HBL temperature 260 °C, 240 °C and 220 °C, PET IRSL readout temperature in each case 20 K lower.

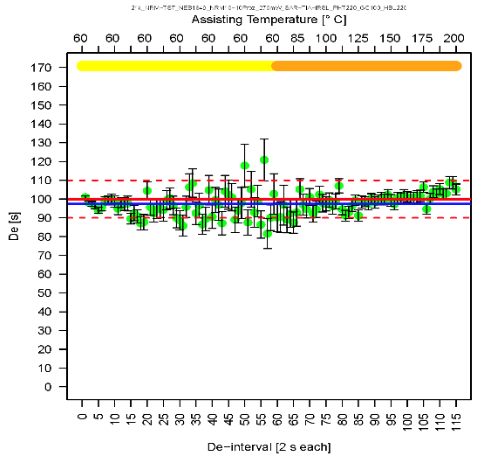
S3.11.1. Testing whether the PHT temperature may possibly be increased



(h) PHT and BLSL HBL at 260 °C, PET to 240 °C, IR 270 mW/cm²



(j) PHT and BLSL HBL at 240 °C, PET to 220 °C, IR 270 mW/cm²



(k) PHT and BLSL HBL at 220 °C, PET to 200 °C, IR 270 mW/cm²

Figure S3.11. MS_21h-21j-21k. PET-IRSL SAR dose recovery tests with DRC aiming at 100 s (100 %), LAB 100 s and NRM 10 % of LAB. Preheat and BLSL HBL temperature varying: 220 °C, 240 °C and 260 °C (PHT for 60 s, BLSL HBL for 250 s). PET-IRSL readout 20 K below PHT resp. BLSL HBL temperature. **(h, j, k)** High IR-energy density IR 270 mW/cm².

Varying combined PHT, PET-IRSL readout and BLSL-hotbleach modes with BLSL HBL at the end of each SAR half-cycle:

(h) PHT and BLSL HBL at 260 °C, PET-IRSL up to 240 °C.

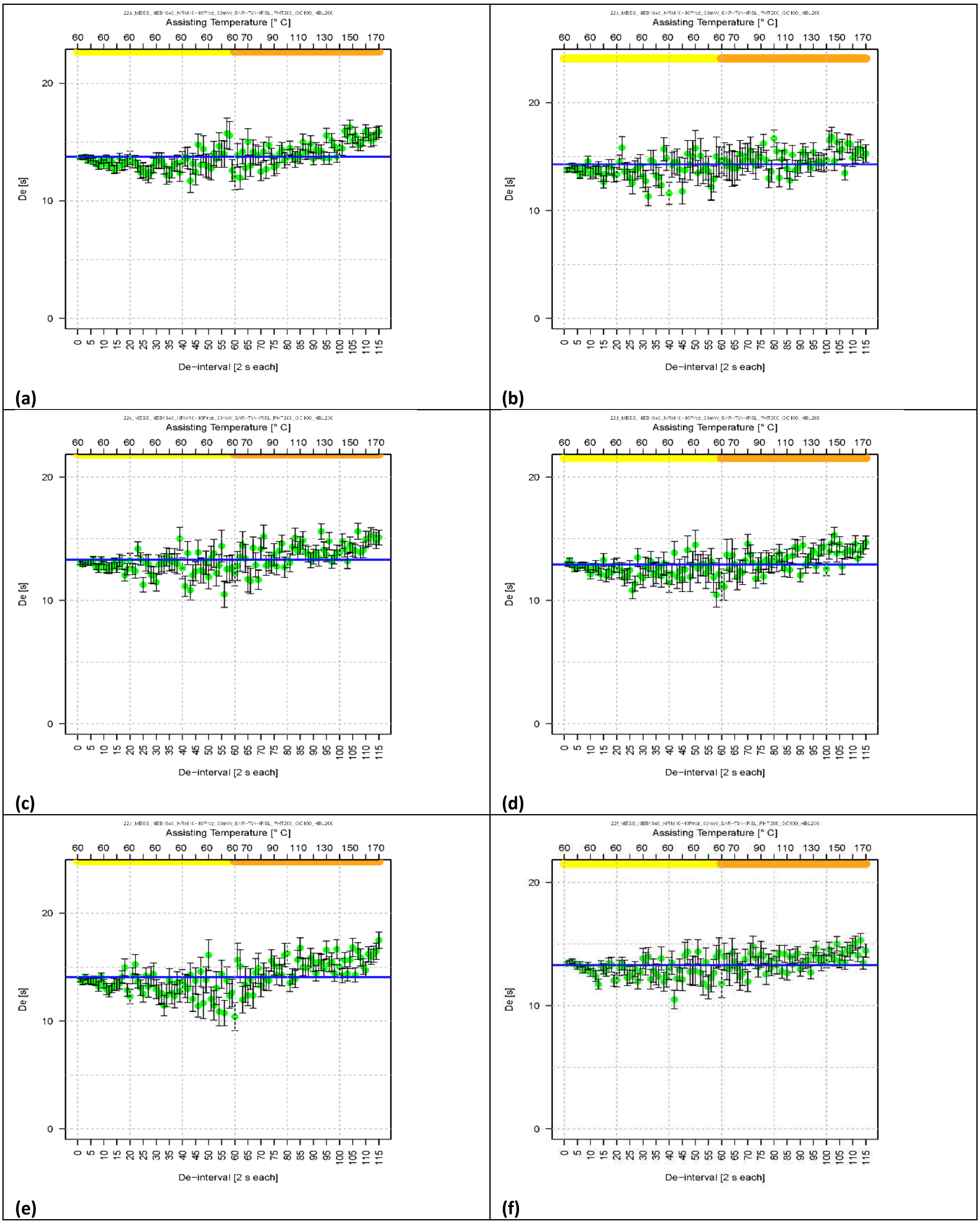
(j) PHT and BLSL HBL at 240 °C, PET-IRSL up to 220 °C.

(k) PHT and BLSL HBL at 220 °C, PET-IRSL up to 200 °C.

Measurements 21h, 21j, 21k (Messung mit Bezeichnung 21i wurde vergessen bzw. der Buchstabe übersprungen)

- **(h)** Slight underestimation of LAB in the low-temperature PET-IRSL range; De values within 10 % error margin of expected De, but increasing from the lower 10 % margin to the upper 10 % margin. PET-peak position already at channel 90/or rather not really identifiable?
- **(j)** Still slight underestimation of LAB in the low-temperature PET-IRSL range, though slightly less than **(a)**; De values within 10 % error margin of expected De, but increasing from the lower 10 margin to the upper 10 % margin, similar **(a)**. PET-peak position already at channel 90/or rather not really identifiable?
- **(k)** De values both in the-low temperature and in the high-temperature PET-IRSL range are well recovered, though in the latter still with a slight slope/trend of increasing values towards the highest PET-IRSL readout temperature (here 200 °C)
- Not worth doing repeat measurements at IR 60 mW/cm²
- We go back to 20b, c and 20e, f which delivered perfectly recovered Des – though with a lower PHT temperature which might involve stronger fading

S3.12. De determination on NEB-1849 – PET-IRSL – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % – PHT and BLSL HBL at 200 °C – PET-IRSL up to 170 °C



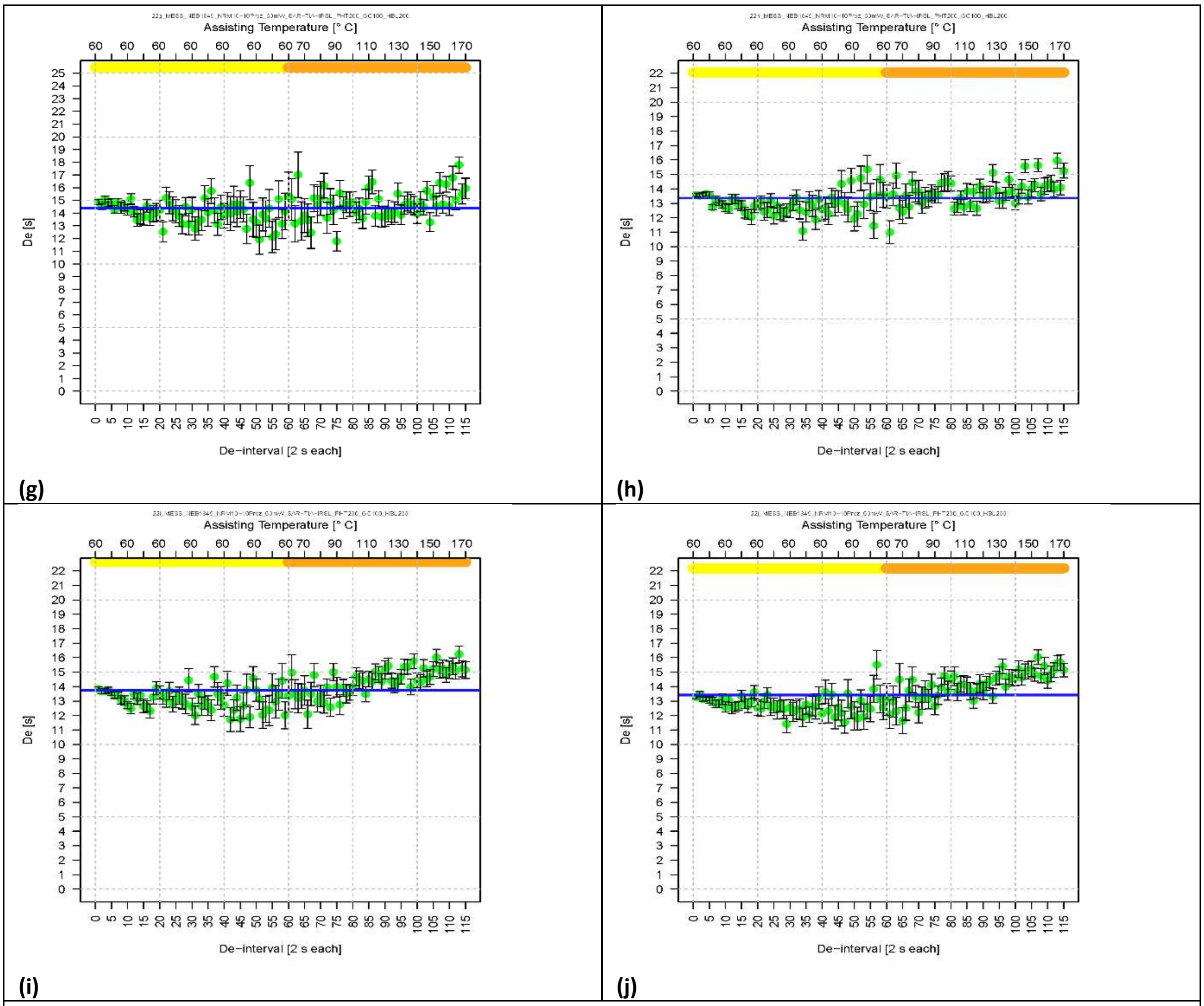
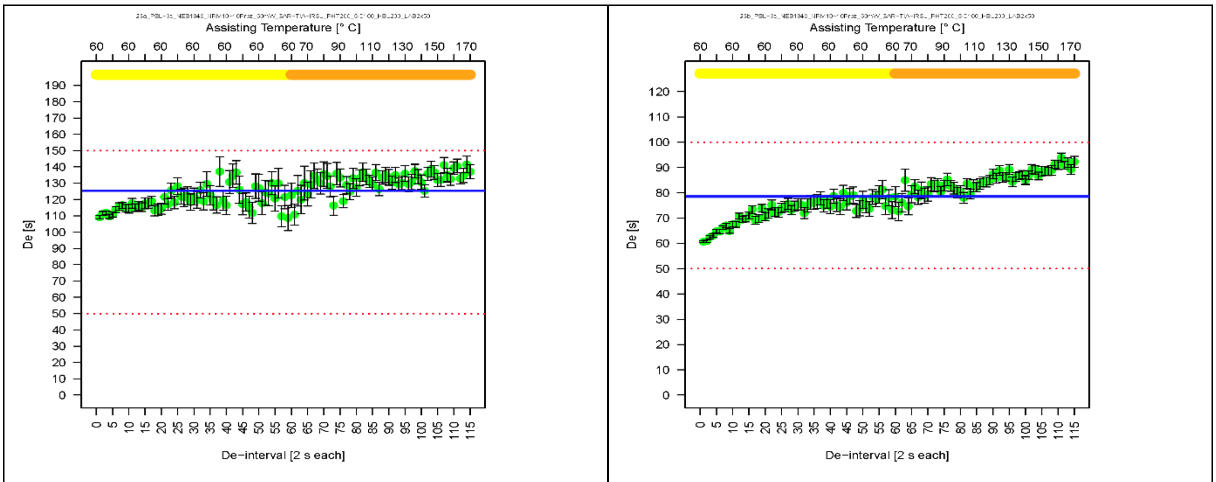


Figure S3.12. MS_22a –22j. PET-IRSL SAR De determination with: IR 60 mW/cm², GC 100 s, NRM 10 %, BLSL hotbleach in each SAR half-cycle. PHT and BLSL at 200 °C, PET-IRSL up to 170 °C

- Distal part of high-temperature PET-IRSL range ca 15 s → underestimating quartz age of 1000 a

S3.13. PBL DRT on NEB-1849 – PET-IRSL – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – GC 100 s – NRM 10 % – PHT and BLSL HBL at 200 °C – PET-IRSL up to 170 °C – PBL 3s

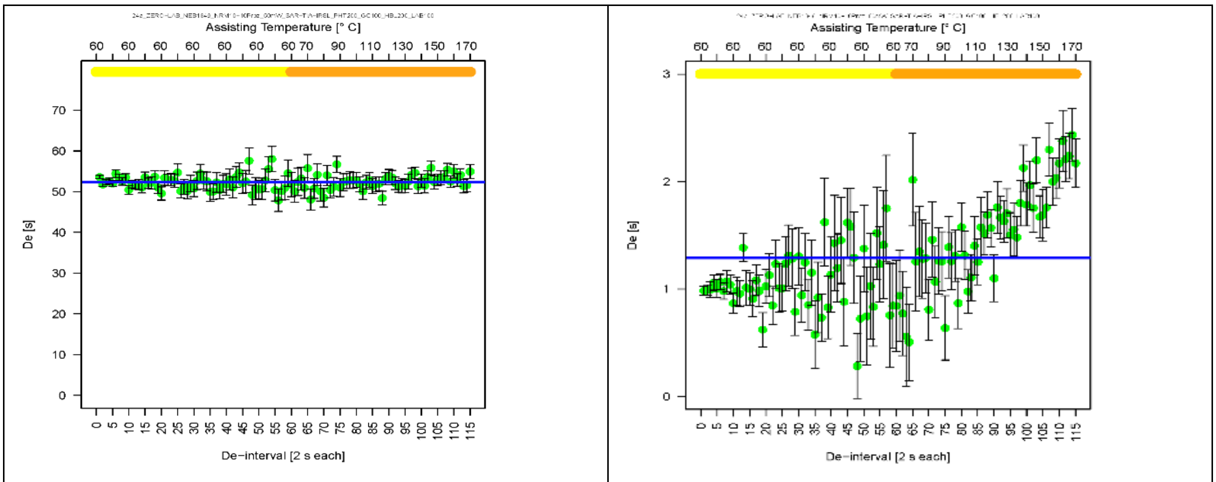


(a) Mishap – But partial bleaching discernable **(b)**

Figure S3.13. MS_23a–23b. PET-IRSL SAR De determination with: IR 60 mW/cm², GC 100 s, NRM 10 %, BLSL hotbleach in each SAR half-cycle. PHT and BLSL at 200 °C, PET-IRSL up to 170 °C

- 23a mishap → IRR 50 s + PHT TL + PBL 3 s + IRR 50 s + PHT TL + IRR 50 s + PHT TL → despite wrong measurement → partial bleaching discernible → repeat (23b)
- 23b → downbending in the earliest part of the low-temperature PET-IRSL range and no upper plateau but only uploping De values in the high-temperature PET-IRSL range (here up to 170 °C) up to the highest readout temperature of 170 °C
- None of the PET-IRSL De-value curves in Fig. S3.12 shows such a conspicuous initial downbending pointing to partial bleaching of the sample

S3.14. ZERO-LAB DRT on NEB-1849 – PET-IRSL – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % – PHT and BLSL HBL at 200 °C – PET-IRSL up to 170 °C



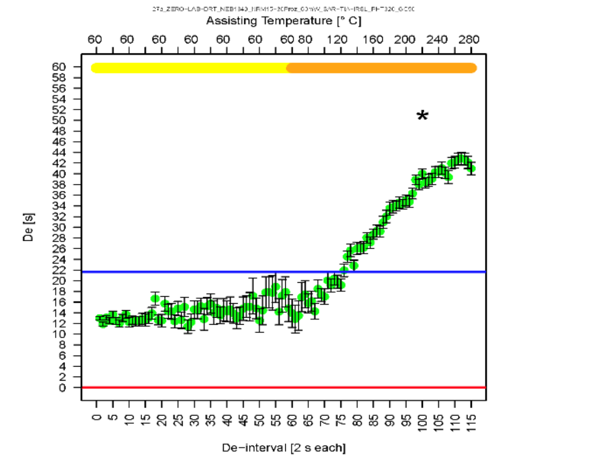
(a) Mishap → 50 s dose

(b)

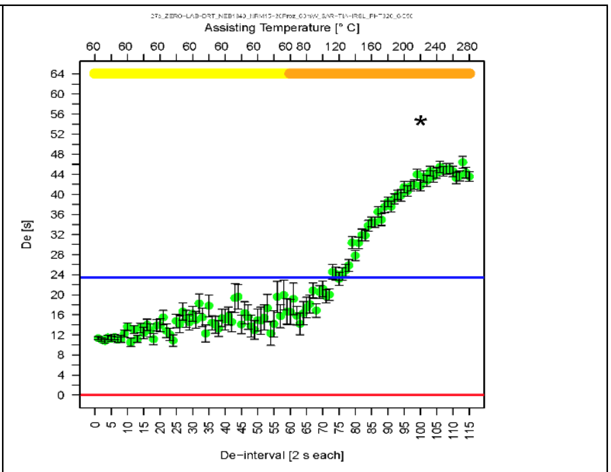
Figure S3.14. MS_(24a–)24b. ZERO-LAB DRT with the PET-IRSL SAR protocol with: IR 60 mW/cm², GC 100 s, NRM 10 %, BLSL hotbleach in each SAR half-cycle. PHT and BLSL at 200 °C, PET-IRSL up to 170 °C

- 24a = mishap → ZERO-LAB + PHT TL + IRR 50 s + PHT-TL → slight overestimation (cf. blue line denoting mean) → due to double PHT? → Or because ca 1 s is the unbleachable remnant dose in the low-temperature PET-IRSL range and up to ca 2.5 s in the high-temperature PET-range → cf. **(b)** DRT with DRC 100, but LAB 50
- 24b → ca 1 s unbleachable residual dose in the low-temperature PET-IRSL range and up to ca 2.5 s residual dose for the highest PET-IRSL readout

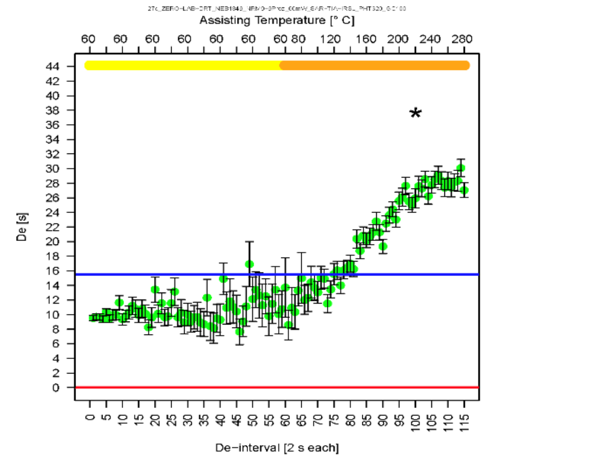
S3.15. ZERO-LAB DRTS on NEB-1849 – Use GC 100 s – LAB 50 s – NRM 9 % (like 10n) and use GC 50 s – LAB 50 s – NRM 30 % (like 17c)



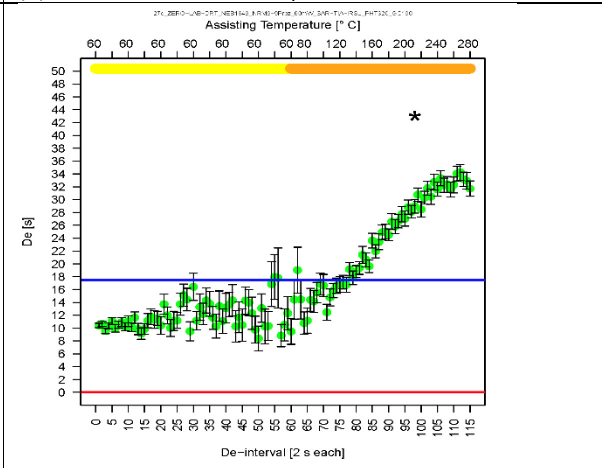
(a) DRC 50 s, NRM 30 %, aliquot #1



(b) DRC 50 s, NRM 30 %, aliquot #2



(c) DRC 100 s, NRM 9 %, aliquot #1

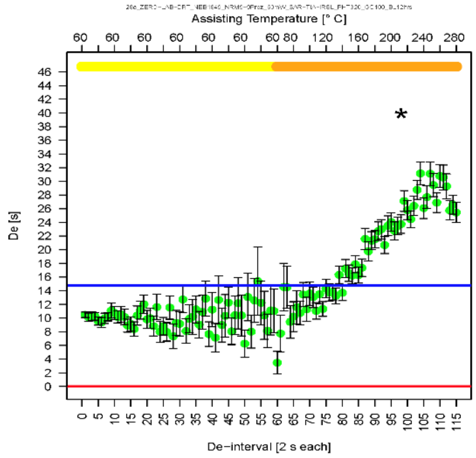


(d) DRC 100 s, NRM 9 %, aliquot #2

Figure S3.15. MS_27a–27d. ZERO-LAB DRT with the PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. PHT 320 °C, PET-IRSL up to 280 °C, BLSL at 280 °C. "Too hot" thermal SAR parameter value not suitable for De determination.

- Both ZERO-LAB DRTs show erroneously high residual doses in the high-temperature PET-IRSL range beyond the apex of the PET-peak of
    - Ca 42–44 s for DRC 50 s
    - Ca 28–32 s for DRC 100 s
    - These come in both cases close to the range of the gained palaeodoses
        - Try SUPERBLEACH → if this does not help → hot thermal treatment is no good for comparatively young samples like NEB-1849

S3.16. ZERO-LAB DRTs on NEB-1849 – Use DRC 100 s – LAB 50 s – NRM 9 % (like 10n) but do SUPERBLEACH (12 hrs solar simulator) prior to ZERO-LAB DRT



(a) GC 100 s, NRM 9 %

Figure S3.16. MS_28a. ZERO-LAB DRT with the PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. PHT 320 °C, PET-IRSL up to 280 °C, BLSL at 280 °C. Like MS_27c and 27d, but after a “superbleach” of 12 x 1 hour solar simulator

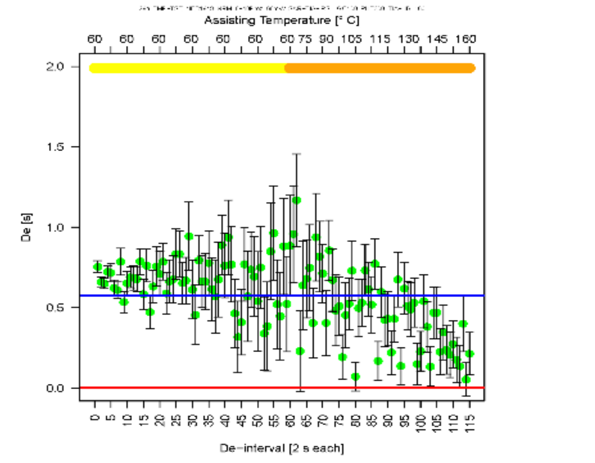
- Similar result like MS_27c and MS_27d: ca 28–32 s for GC 100 s
  - Unbleachable residual dose which is probably transferred by the thermal (pre-)treatment into the IRSL trap
- Test again systematically increase of thermal SAR parameters with Zero-Dose-DRTs

Next step: Increase PHT and corresponding PET_{max} and HBL

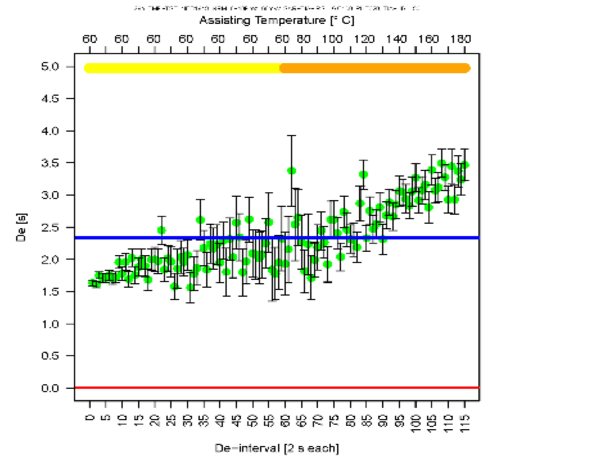
- PHT 320 – PET_{max} 280 – HBL 280 ← „classic“
- PHT 300 – PET_{max} 260 – HBL 260
- PHT 280 – PET_{max} 240 – HBL 240
- PHT 260 – PET_{max} 220 – HBL 220
- PHT 240 – PET_{max} 200 – HBL 200
- PHT 220 – PET_{max} 180 – HBL 180
- PHT 200 – PET_{max} 160 – HBL 160 ← PET_{max} / HBL 10 K below Buckland et al. (2019)

Select the one which works with the strongest thermal treatment; increase PET_{max}/HBL by 10–20 K

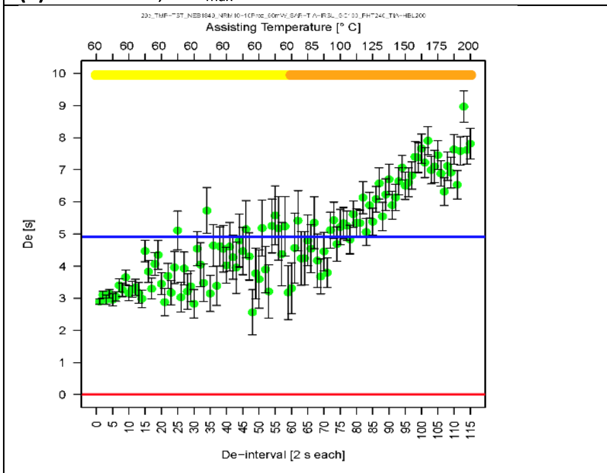
S3.17. ZERO-LAB DRTS on NEB-1849 – DRC 100 s – NRM 10 % – Increase PHT in steps of 20 K from 200 °C to 320 °C – PET_{max} and HBL 40 K below PHT



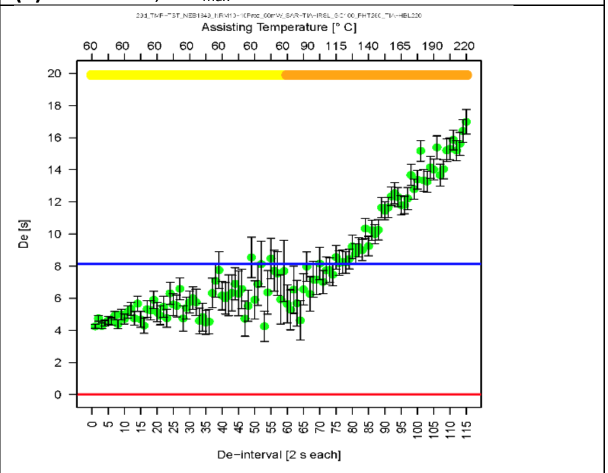
(a) PHT 200 °C, PET_{max} and HBL 160 °C



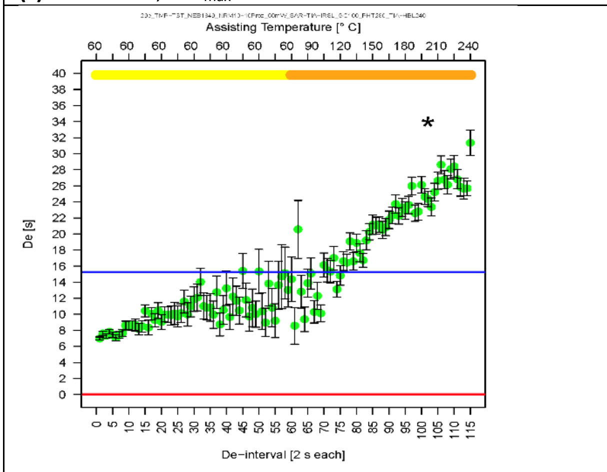
(b) PHT 220 °C, PET_{max} and HBL 180 °C



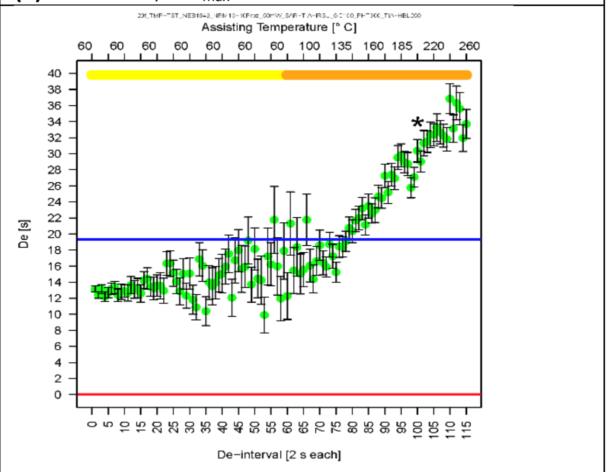
(c) PHT 240 °C, PET_{max} and HBL 200 °C



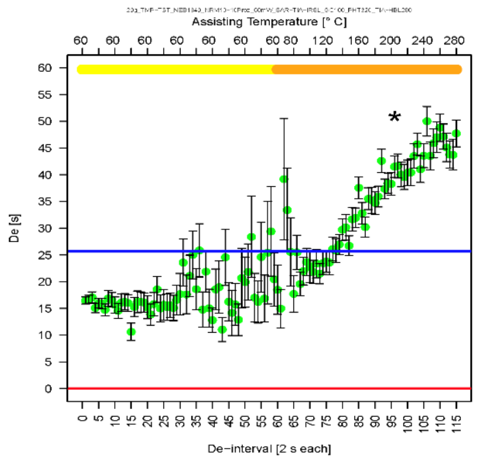
(d) PHT 260 °C, PET_{max} and HBL 220 °C



(e) PHT 280 °C, PET_{max} and HBL 240 °C



(f) PHT 300 °C, PET_{max} and HBL 260 °C



(g) PHT 320 °C, PET_{max} and HBL 280 °C

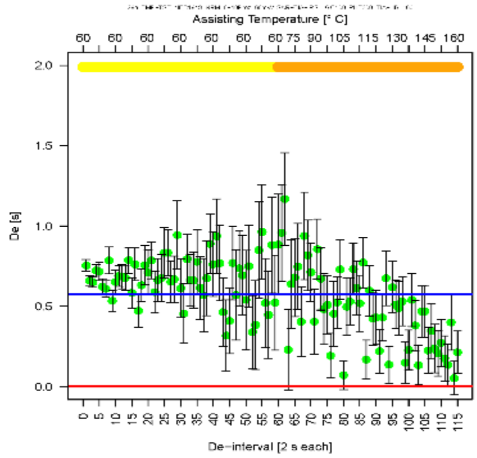
Figure S3.17. MS_29a–29g. ZERO-LAB DRTs with the PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. Varying PHT and corresponding PET_{max} and HBL temperatures. PET_{max} and HBL 40 K below PHT. PET-peaks only for PHT ≥ 280 °C.

The measurement series 20a–29g shows systematic increase of recuperation with increasing PHT and accompanying PET_{max} and HBL temperatures. The distal part of the high temperature PET-IRSL range (here varying from 160–280 °C) probably shows the comparably least fading. Therefore, one would tend to extract a De from that section. However, with the exception of **(a)** all measurements exhibit for that likely most stable part the highest recuperation. Accepting for dating a residual dose of ca 10 % or 5 % of the palaeodose, this would respectively constrain the upper age limit for samples to being submitted to PET-IRSL SAR dating. Assuming an effective environmental dose rate for the here compiled examples this would mean:

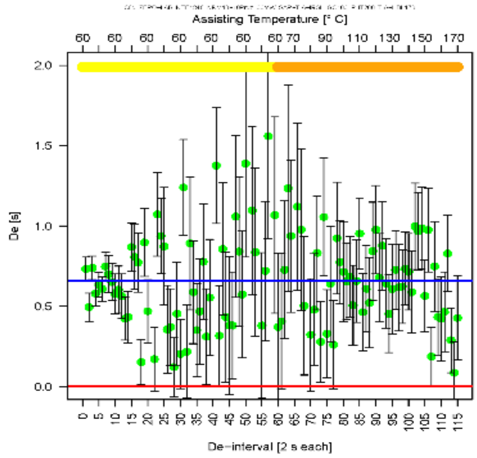
- **(a)** The observed residual dose of ≤ 1s (ca 0.107 Gy) would allow to date samples with a minimum palaeodose of ca 1 Gy (10 % residuum allowed) or 2 Gy (5 % residuum allowed) corresponding for the here assumed dose rate of ca 3 Gy/ka to minimum ages of ca 350–700 a
- **(b)** The residual dose of ca 3.5 s (ca 0.374 Gy) would allow to date at least ca 1.2 ka (10 % acceptable residual dose) or 2.4 ka (5 %) old samples. For sample HDS-1849 with an expected dose of ca 1 ka this measurement setup would therefore already be borderline.
- **(c)** The residual dose of ca 8 s (ca 0.854 Gy) would allow to date samples with minimum ages of ca 2.8 ka (10 %) or 5.6 ka (5 %).
- **(d)** The observed maximum residual dose of ca 18 s (1.921 Gy) would allow to date mid to early Holocene samples ≥ 6.4 ka (10 %) or ≥ 12.8 ka (5 %) under the given environmental dose rate conditions.
- **(e)** The gained residual dose of 28–32 s (ca 3–3.4 Gy) would allow to date samples of at least ca 10 ka (10 %; earliest Holocene) or 20 ka (5 %; LGM). This is the same order of magnitude as gained with DRC 100s, NRM 9 %, PHT 320 °C and PET_{max} and HBL 280 °C (cf. **MS_27c-27d**, **MS_28a**).
- **(f)** The measured residual dose of 32–34 s (ca 3.6 Gy) and up to individual values of 36–38 s (ca 4 Gy) would allow to date samples of at least 12 ka (10 %) or 24 ka (5 ka).
- **(g)** The residual dose of up to 50 s (5.337 Gy) would allow to date samples of at least ca 18 ka (10 % remnant dosed allowed; LGM) or 36 ka (5 %; late MIS 3). The observed residual dose is ca 80 % larger than the one gained with the similar PET-IRSL SAR protocol (cf. **MS_27c-27d**, **MS_28a**) differing only in the size of NRM (here 10 % vs 9 %). However, here it can not be decided whether the size of NRM or possible inter-aliquot variability are the reason for the differing results. In case of a dating study several aliquots should be tested for borderline samples.

- Further investigate PHT 200 °C → with PET_{max}/HBL 170 °C and 180 °C

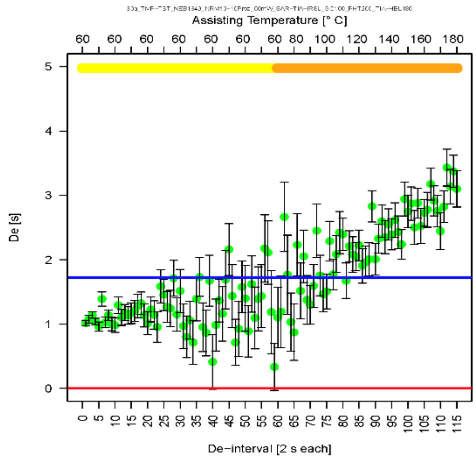
S3.18. ZERO-LAB DRTS on NEB-1849 – GC 100 s – NRM 10 % – PHT 200 °C – PET_{max} and HBL increasing from 160 °C (copied from MS_29a), to 170 °C (MS_31b) to 180 °C (MS_30a)



(a) PHT 200 °C, PET_{max} and HBL 160 °C (from **29a**)



(b) PHT 200 °C, PET_{max} and HBL 170 °C (**MS_30b**)



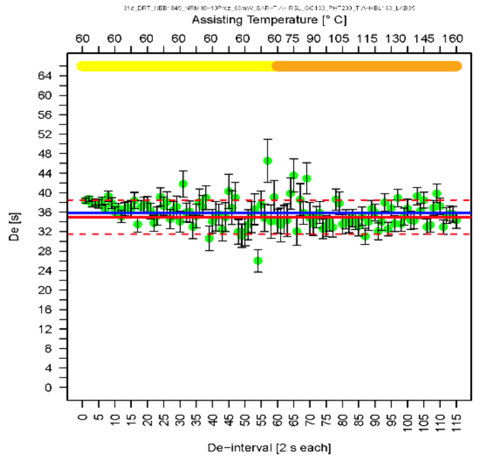
(c) PHT 200 °C, PET_{max} and HBL 180 °C (**MS_30a**)

Figure S.3.18. MS_30a and MS-30b, together with MS_29a. ZERO-LAB DRTs with the PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. PHT at 200 °C and PET_{max} and HBL increasing in steps of 10 K from 160 °C to 180 °C. Compilation of new measurements **30b and 30a** together with **MS_29a**.

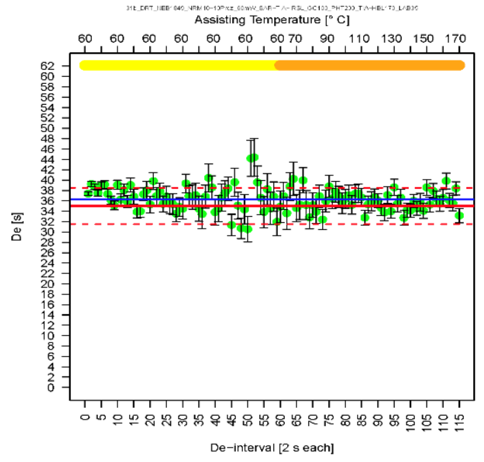
- Use PET_{max}/HBL 170 °C for measurements → different from 22a–22j ← PHT and HBL 200 °C, PET_{max} 170 °C→ age underestimation → measure ca 15 s → expected ca 30–35 s (3.2–3.7 Gy)

Measure DRTs with LAB 35 s following MS_29a, MS_30b and 30a

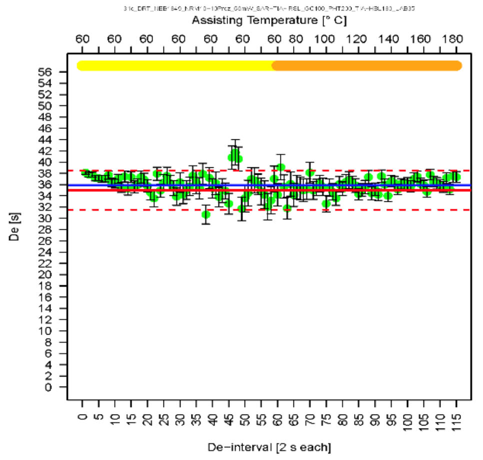
S3.19. DRTS on NEB-1849 – DRC 100 s – NRM 10 % – LAB 35 s – PHT 200 °C – PET$_{max}$ and HBL increasing from 160 °C to 180 °C in steps of 10 K



(a) PHT 200 °C, PET$_{max}$ and HBL 160 °C



(b) PHT 200 °C, PET$_{max}$ and HBL 170 °C

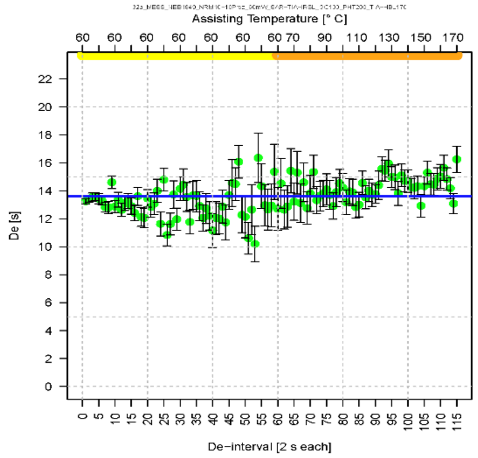


(c) PHT 200 °C, PET$_{max}$ and HBL 180 °C

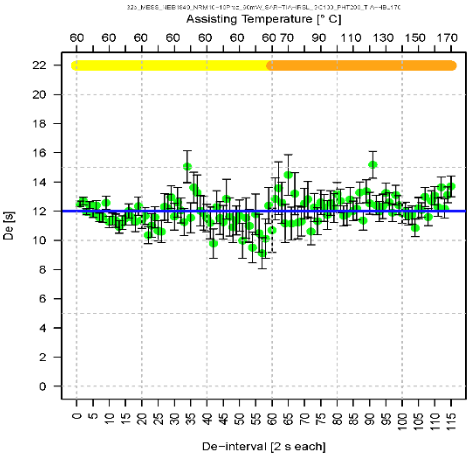
Figure S3.19. MS_31a–31c. DRTs with LAB 35 s following the PET-IRSL SAR protocols in previous figure (MS_30a–30 c). PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. PHT at 200 °C and PET$_{max}$ and HBL increasing in steps of 10 K from 160 °C to 180 °C.

- All three DRTs recovered LAB 35 s within error margins
- For De determination we choose the variant with PET$_{max}$ and HBL 170 °C

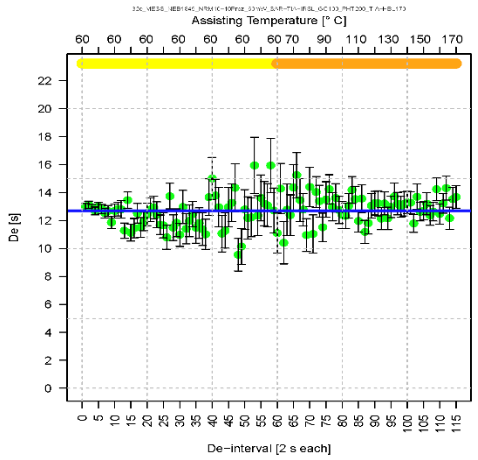
S3.20. De determination on NEB-1849 – PET-IRSL – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % – PHT at 200 °C – PET_{max} 170 °C – BLSL HBL at 170 °C → cf. DRT 31b



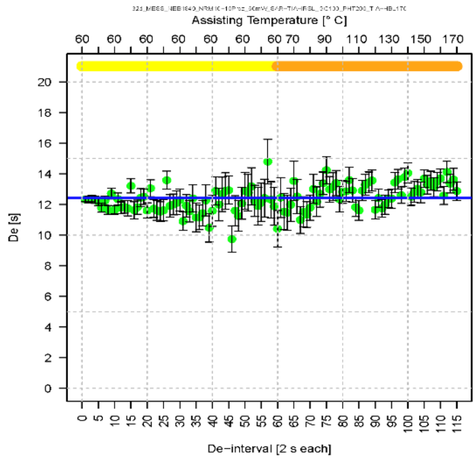
(a) Mean De-integral 100-115: 14.7 s (1.57 Gy; 0.52 ka)



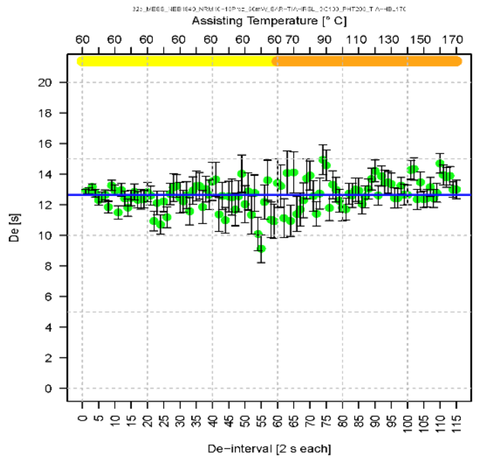
(b) Mean De-integral 100-115: 12.5 s (1.33 Gy; 0.44 ka)



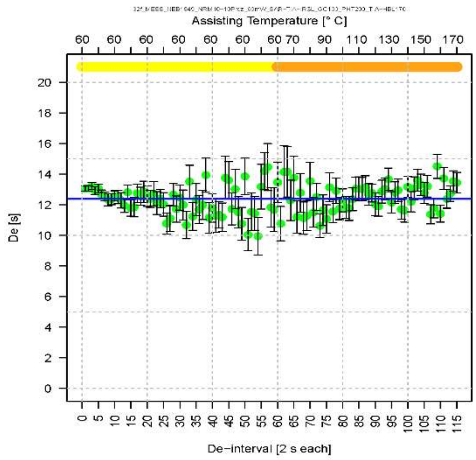
(c) Mean De-integral 100-115: 13.05 s (1.39 Gy; 0.46 ka)



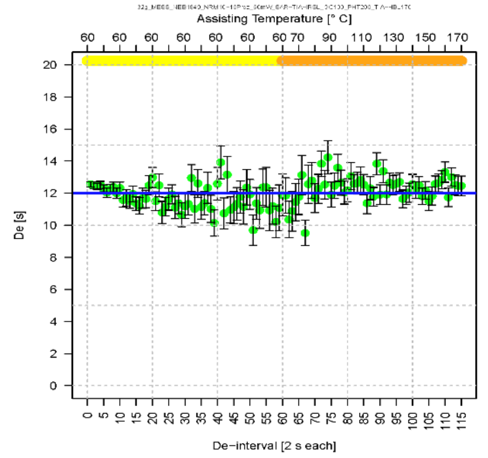
(d) Mean De-integral 100-115: 13.03 s (1.39 Gy; 0.46 ka)



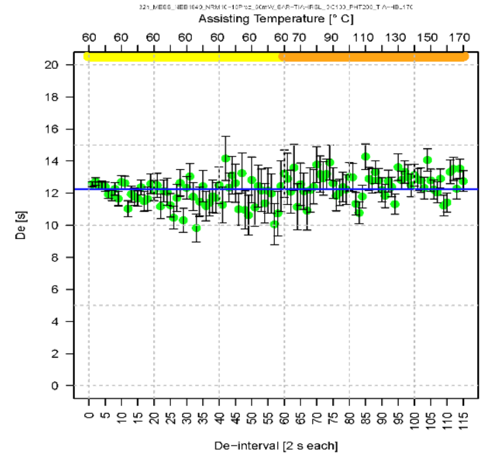
(e) Mean De-integral 100-115: 13.2 s (1.4 Gy; 0.47 ka)



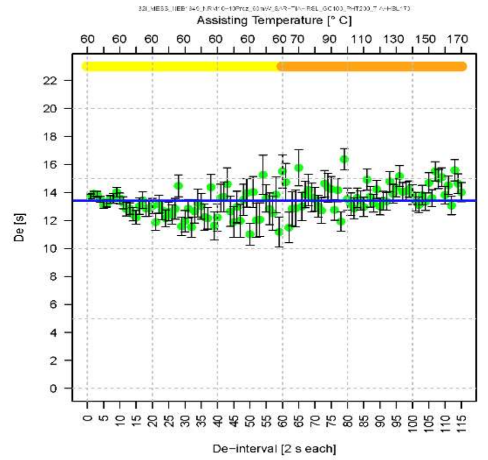
(f) Mean De-integral 100-115: 12.7 s (1.36 Gy; 0.45 ka)



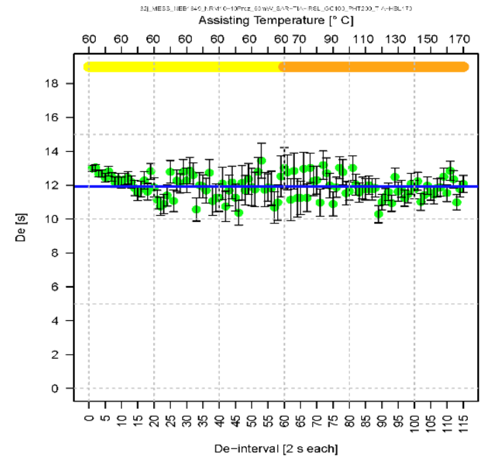
(g) Mean De-integral 100-115: 12.4 s (1.32 Gy; 0.44 ka)



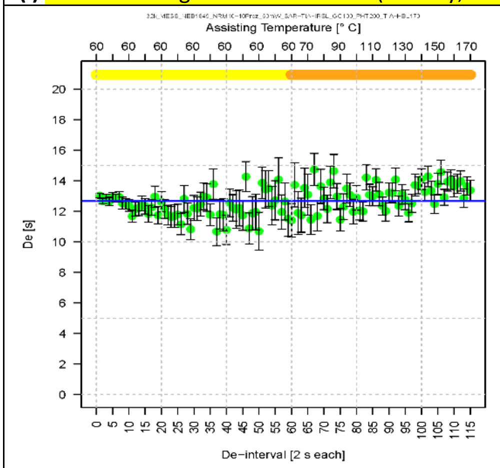
(h) Mean De-integral 100-115: 12.7 s (1.35 Gy; 0.45 ka)



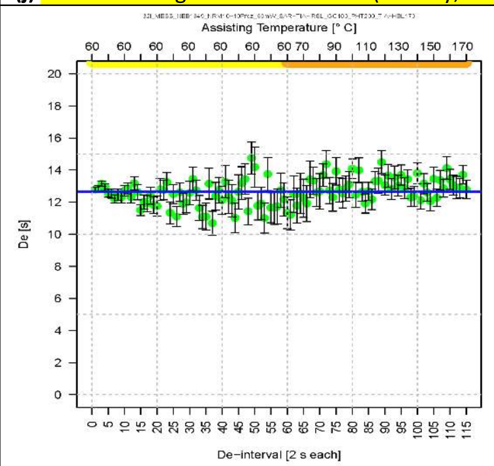
(i) Mean De-integral 100-115: 14.1 s (1.51 Gy; 0.50 ka)



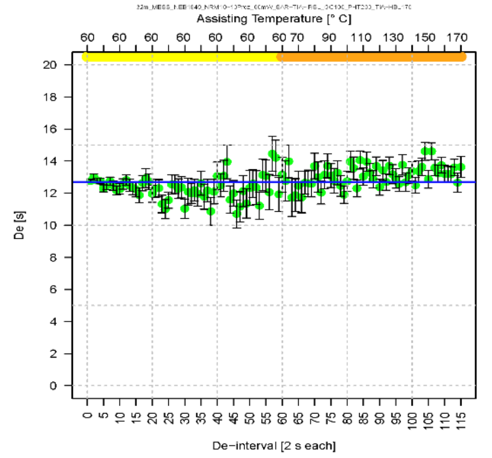
(j) Mean De-integral 100-115: 11.7 s (1.25 Gy; 0.42 ka)



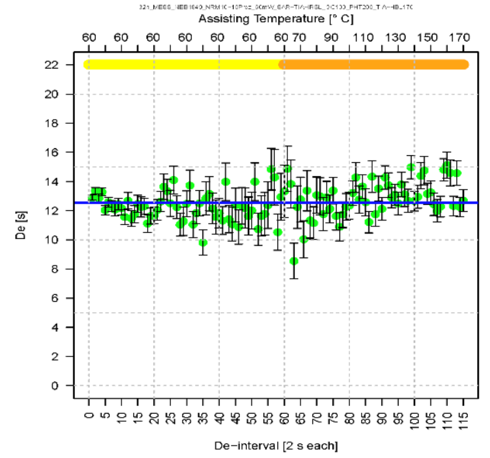
(k) Mean De-integral 100-115: 13.4 s (1.43 Gy; 0.48 ka)



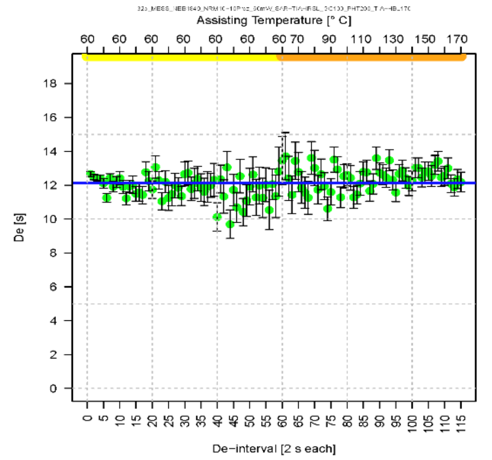
(l) Mean De-integral 100-115: 13.05 s (1.39 Gy; 0.46 ka)



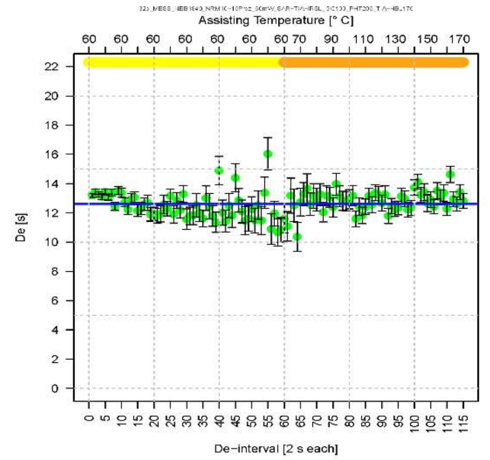
(m) Mean De-integral 100-115: 13.3 s (1.42 Gy; 0.47 ka)



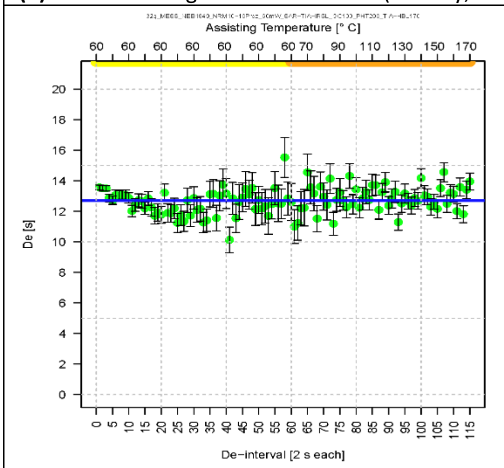
(n) Mean De-integral 100-115: 13.4 s (1.43 Gy; 0.48 ka)



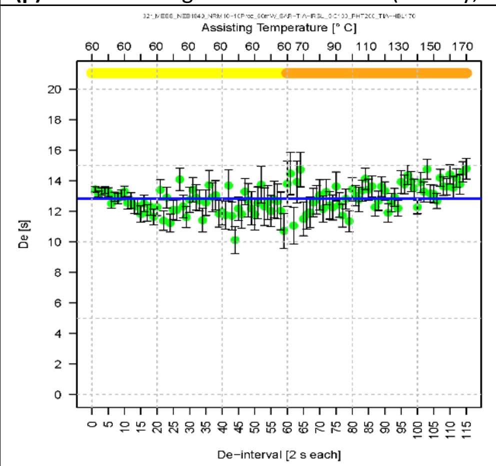
(o) Mean De-integral 100-115: 12.6 s (1.34 Gy; 0.45 ka)



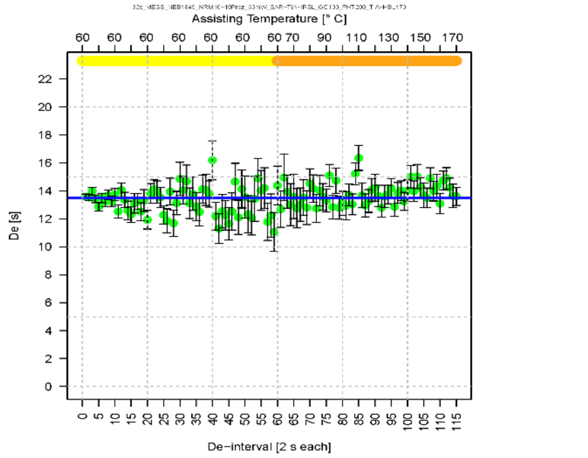
(p) Mean De-integral 100-115: 12.95 s (1.38 Gy; 0.46 ka)



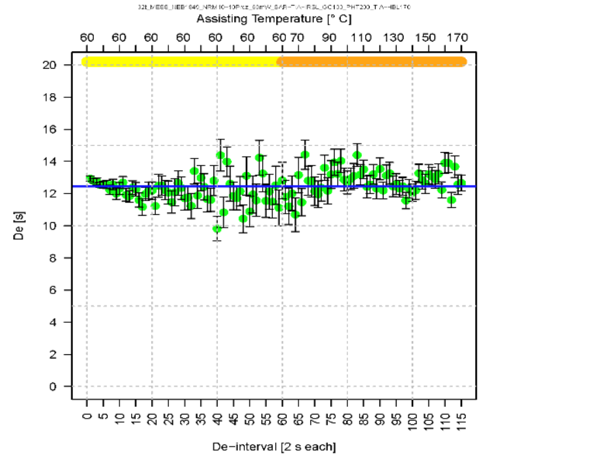
(q) Mean De-integral 100-115: 12.9 s (1.38 Gy; 0.46 ka)



(r) Mean De-integral 100-115: 13.6 s (1.45 Gy; 0.48 ka)



(s) Mean De-integral 100-115: 13.96 s (1.49 Gy; 0.50 ka)



(t) Mean De-integral 100-115: 12.76 s (1.36 Gy; 0.45 ka)

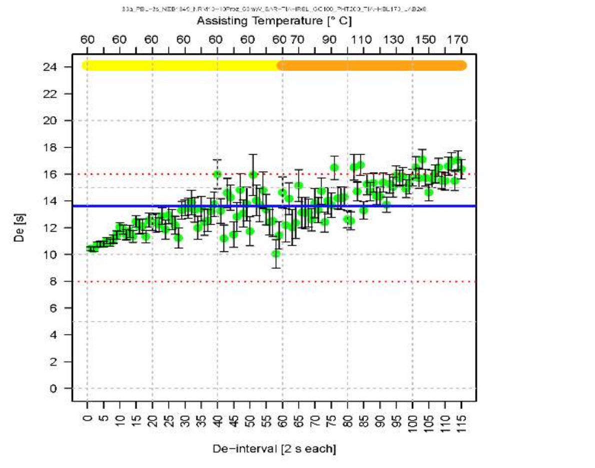
Figure S3.20. MS_32a–32t. PET-IRSL SAR De determination with: IR 60 mW/cm², DRC 100 s, NRM 10 %, BLSL hotbleach in each SAR half-cycle. PHT at 200 °C, PET-IRSL up to 170 °C, BLSL HBL at 170 °C. Preliminary Age assessments in **(a)**–**(t)** based on an assumed dose rate of 3 Gy/ka.

- De values around ca 12–15 s (1.281–1.601 Gy) would provide for an effective dose rate of 3–3.3 Gy an age of 0.4–0.5 ka, which is significantly younger than the quartz age of ca 1 ka.
- Mean of De-signal integral 1–5 = 12.9 [0.4] s; Mean of De-signal integral 71–115 = 13.0 [0.6] s; De-signal integrals corresponding to integrals with higher and lower g-values, respectively
- Aliquots marked in yellow used for g-value determination
- Next: perform PBL tests

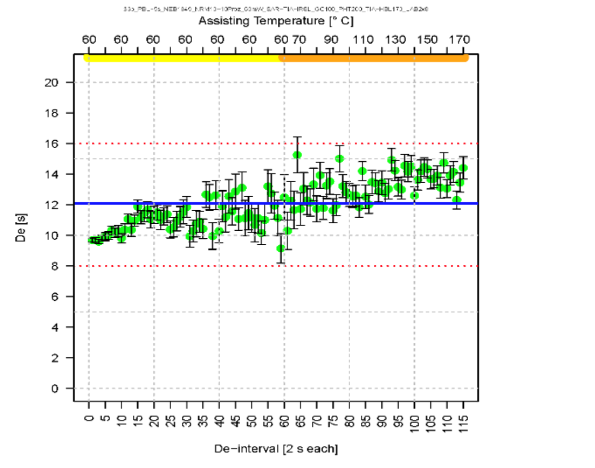
Choose 5 aliquots for fading measurements from MS_32a–32t: largest De, smallest De, intermediate Des (MS_35)

- **(d)** Mean De-signal integral 100-115: 13.03 s (1.39 Gy; 0.46 ka) → POS-57
- **(g)** Mean De-signal integral 100-115: 12.4 s (1.32 Gy; 0.44 ka) → POS-60
- **(i)** Mean De-signal integral 100-115: 14.1 s (1.51 Gy; 0.50 ka) → POS-62
- **(j)** Mean De-signal integral 100-115: 11.7 s (1.25 Gy; 0.42 ka) → POS-63
- **(s)** Mean De-signal integral 100-115: 13.96 s (1.49 Gy; 0.50 ka) → POS-72
- **(d)** Mean De-signal integral 100-115: 13.03 s (1.39 Gy; 0.46 ka) → POS-57
- **(g)** Mean De-signal integral 100-115: 12.4 s (1.32 Gy; 0.44 ka) → POS-60
- **(i)** Mean De-signal integral 100-115: 14.1 s (1.51 Gy; 0.50 ka) → POS-62
- **(j)** Mean De-signal integral 100-115: 11.7 s (1.25 Gy; 0.42 ka) → POS-63
- **(s)** Mean De-signal integral 100-115: 13.96 s (1.49 Gy; 0.50 ka) → POS-72

S3.21. PBL DRT on NEB-1849 – PET-IRSL – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % – PHT and BLSL HBL at 200 °C – PET-IRSL up to 170 °C – PBL 3 s and 5 s



(a) PBL 3 s white light



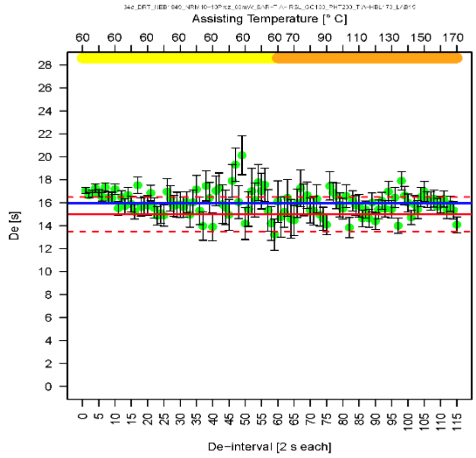
(b) PBL 5 s white light

Figure S3.21. MS_33a–33b. PET-IRSL SAR De determination with: IR 60 mW/cm², DRC 100 s, NRM 10 %, BLSL hotbleach in each SAR half-cycle. LAB 2 x 8 s, PHT and BLSL at 200 °C, PET-IRSL up to 170 °C.

- Measurements 33a–33b similar MS_23b, but lower first and second LAB: 2 x 8 s (instead of 2 x 50 s) – the sum corresponding by size ca to the Des gained in MS_32–32t
- In both measurements the De values of the early part of the low-temperature PET-IRSL range are depressed overestimating the second applied dose of 8 s by **(a)** slightly more than ca 25 % for PBL 3 s and **(b)** slightly less than 25 % for PBL 5 s.
- While the distal part of the high temperature PET-IRSL range **(a)** meets the sum of both doses for PBL 3 s, **(b)** slightly underestimates it for PBL 5 s.
- None of the De measurements shows such a De-value curve pointing to partial bleaching comparable in intensity to 3 s or 5 s bleaching with the solar simulator.

Perform also a DRT with LAB 15 s – like MS_31b, but with LAB 15 s, which compares to the size of the natural Des

S3.22. DRT on NEB-1849 – DRC 100 s – NRM 10 % – LAB 15 s – PHT 200 °C – PET_{max} and HBL 170 °C



(a) PHT 200 °C, PET_{max} and HBL 170 °C

Figure S3.22. MS_34a. DRT with LAB 15 s following the PET-IRSL SAR protocol as applied for the De determination **(MS_32a–30t)**. PET-IRSL SAR protocol with: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. PHT at 200 °C and PET_{max} and HBL 170 °C.

- The expected dose (red line) is met within an acceptable error margin of 10 % (dotted upper and lower lines)

S3.23. Example of TL-PHT and PET-IRSL signal curves and data analysis with the function “analyse_SAR.CWOSL()” – MS_32a

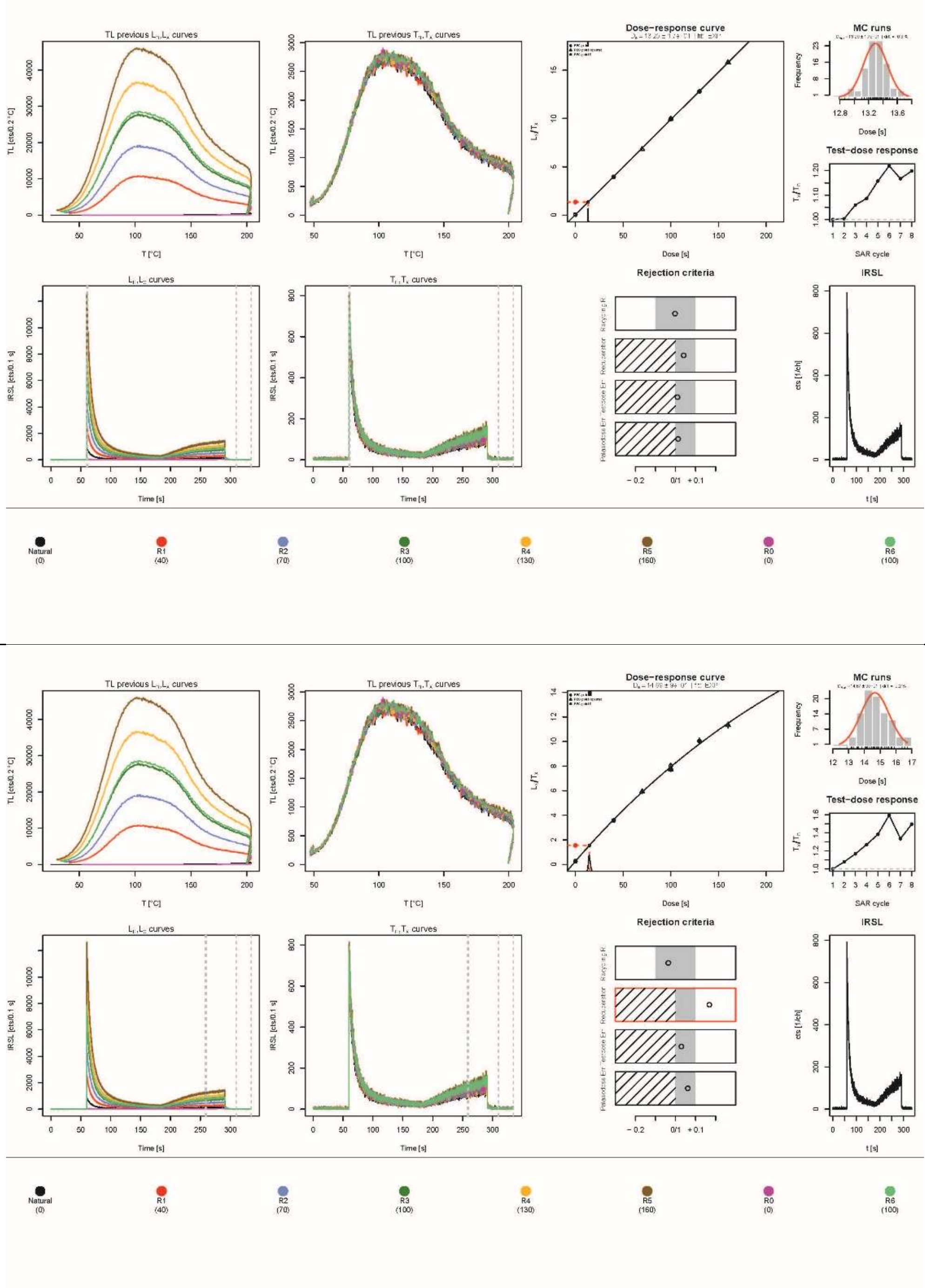
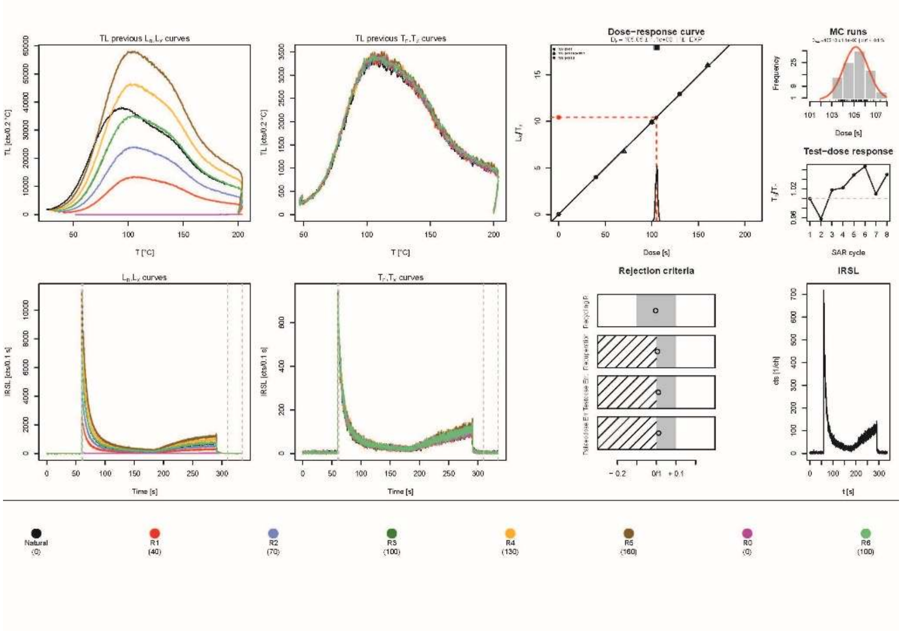
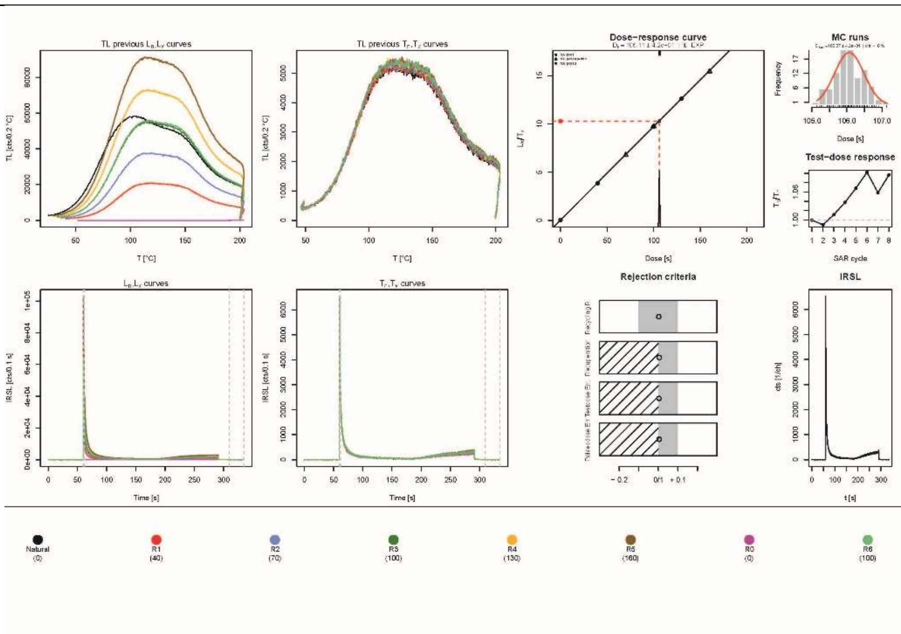


Figure S3.23. MS_32a. Analysis of MS_32a (cf. **Fig. S3.20**). “Classic” – PET-IRSL with IR 60 mW/cm² – BLSL hotbleach each SAR half-cycle – DRC-AIM 100 s – NRM 10 % – PHT 200 °C – PET_{max} and HBL 170 °C. Examples of De determination for De-signal integral 1 and 100. No position of PET-peak determinable for measurements with the modest thermal SAR parameters.

S3.24. Example of PET-IRSL signal curves measured with 60 mW/cm² (MS_21f) vs 270 mW/cm² (MS_21c)



(a)

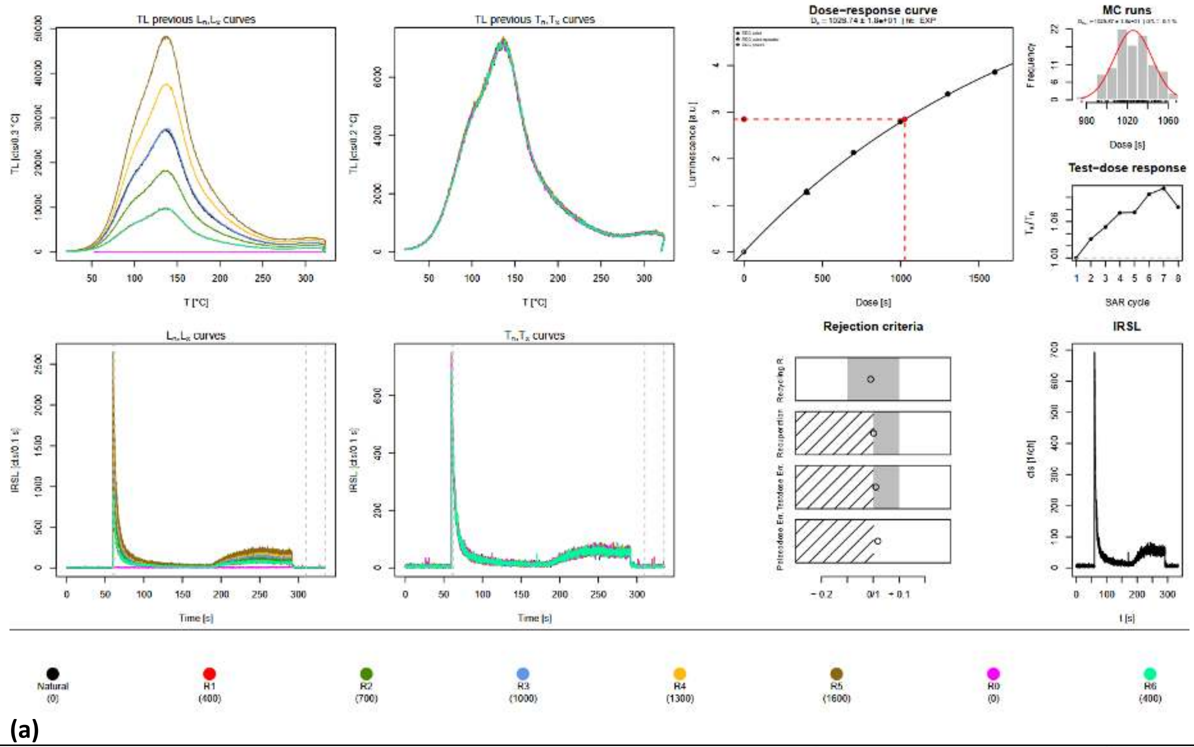


(b)

Figure S3.24. MS_21c, MS_21f. Analysis of MS_21c (cf. **Fig. S3.10c**) measured with **(a)** the usual IR 60 mW/cm² and MS_21f (cf. **Fig. 3.10f**) measured with **(b)** increased IR 270 mW/cm². Other "classic" parameters: BLSL hotbleach each SAR half-cycle – DRC-AIM 100 s – NRM 10 % – PHT 200 °C – PET_{max} and HBL 170 °C. Examples of De determination for De-signal integral 1. No position of PET-peak determinable for measurements with the modest thermal SAR parameters.

S4. Supplement 4 – Feldspar specimens – PET-IRSL SAR measurements

S4.1. G-40 Feldspar (Feldspar Corporation)



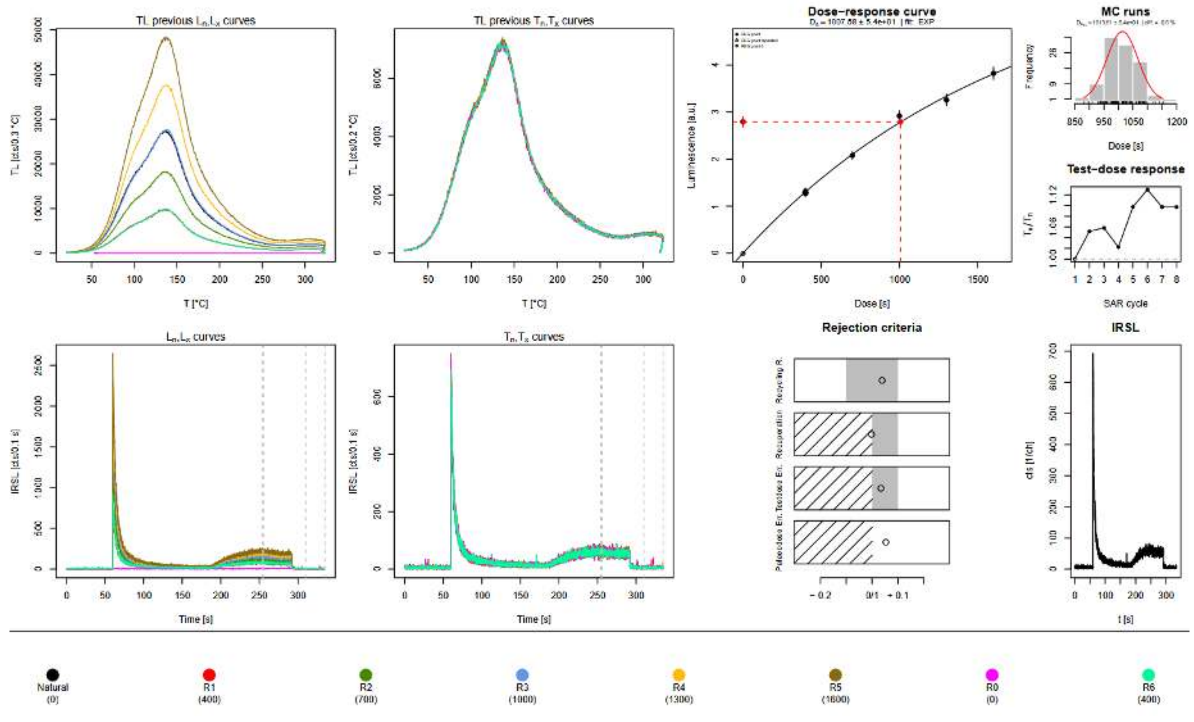
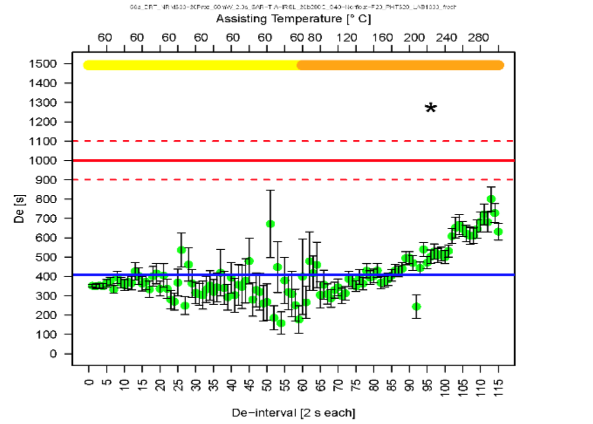
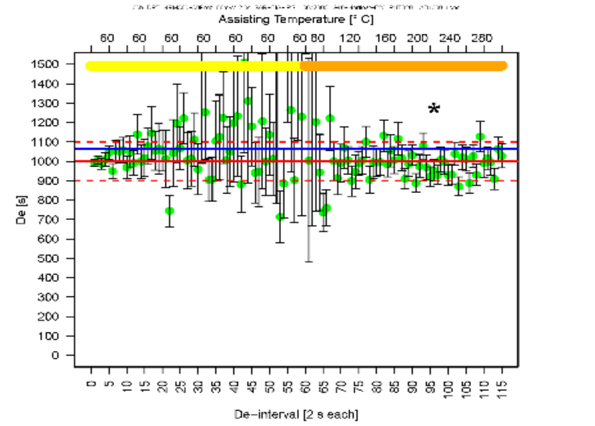


Figure S4.1.1. Dose recovery test on used aliquot of **G40** coarse-grain feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Recovered dose for De-signal integral 1. **(b)** Recovered dose for De-signal integral 98 (PET-peak).

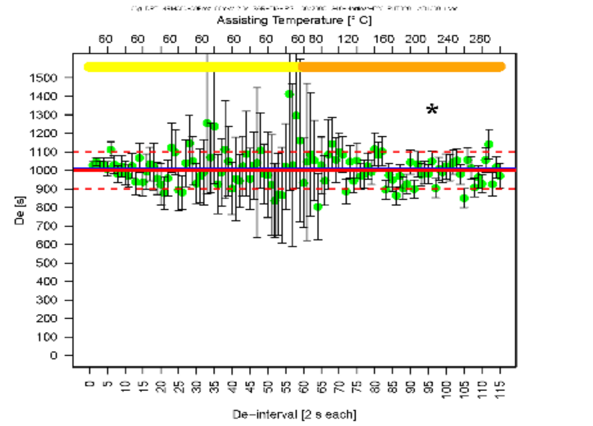
66g_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used



(a)



(b)



(c)

Figure S4.1.2. Dose recovery test on **G40** feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Fresh aliquot underestimating the administered dose. **(b–c)** Used aliquot recovering the administered dose well within an error range of 10 % over the complete PET-IRSL readout. PET-peak at De-signal integral 96 **(a)** and De-signal integral 98 **(b, c)**, respectively.

66a_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_fresh
66b_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used
66g_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used

S4.2. Norfloat Potash Feldspar 371214 (Cookson Matthey)

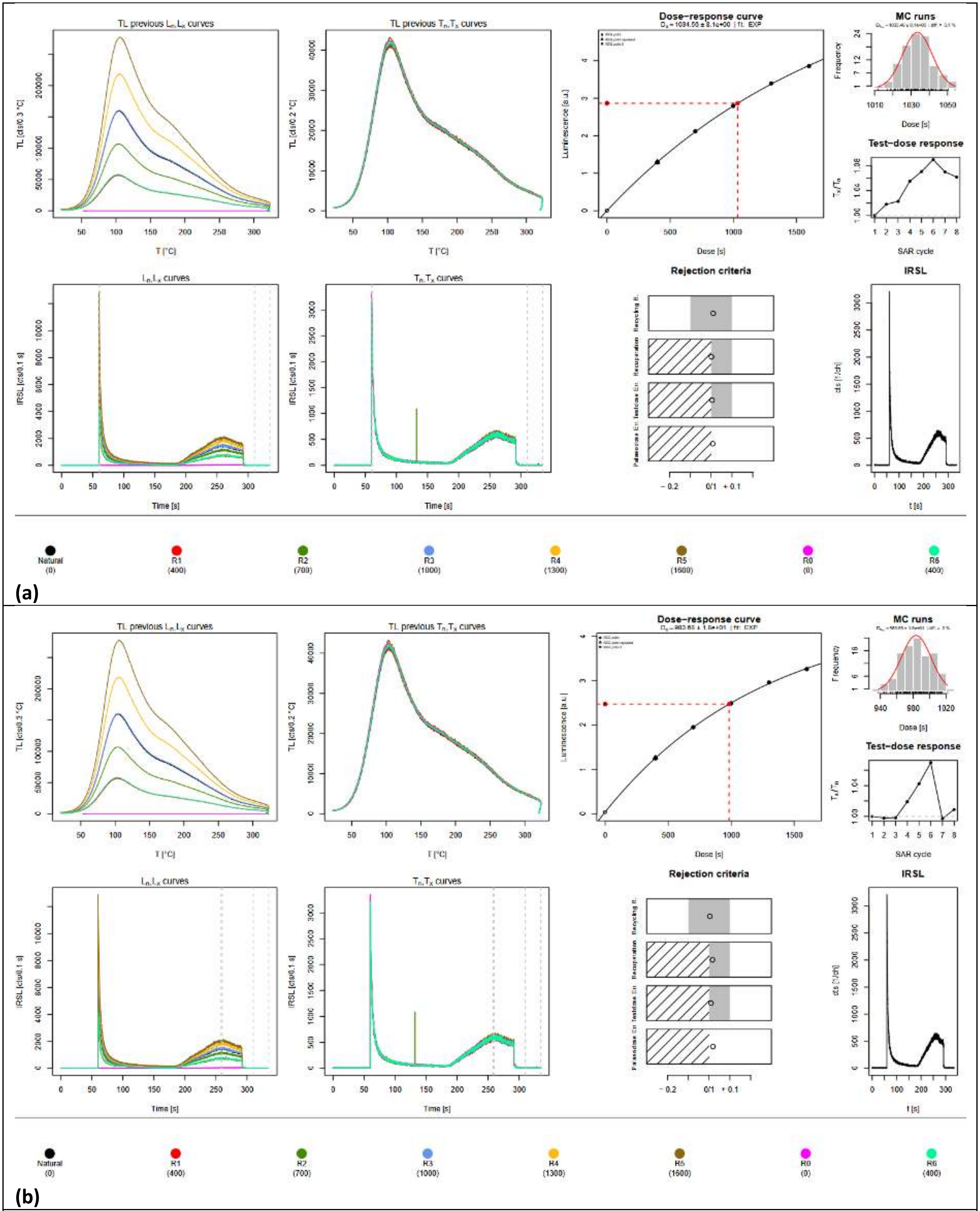
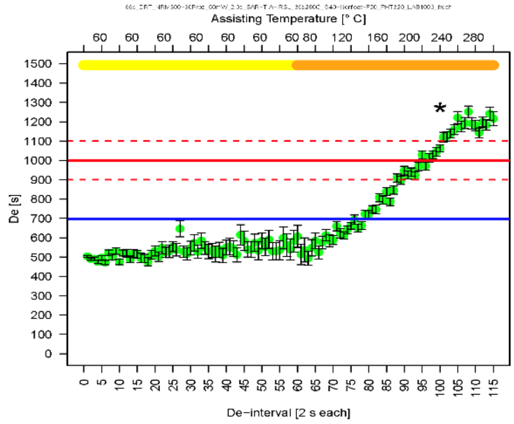
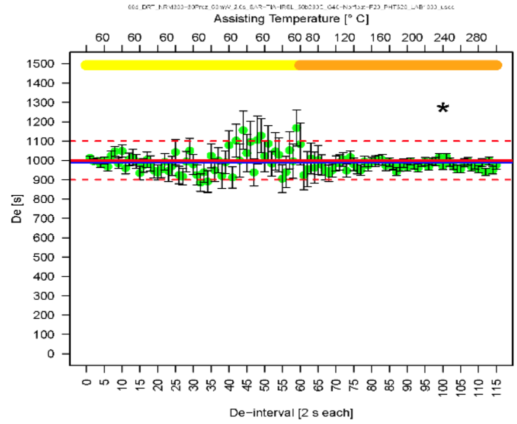


Figure S4.2.1. Dose recovery test on used aliquot of **Norfloat (potash)** coarse-grain feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Recovered dose for De-signal integral 1. **(b)** Recovered dose for De-signal integral 100 (PET-peak).

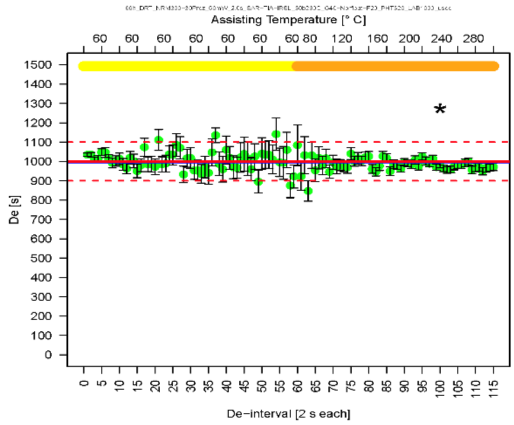
66h_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used



(a)



(b)



(c)

Figure S4.2.2. Dose recovery test on **Norfloat (potash)** feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Fresh aliquot underestimating the administered dose below the PET-peak and overestimating the administered dose above the PET-peak. **(b –c)** Used aliquot recovering the administered dose well within an error range of 10 % over the complete PET-IRSL readout. PET-peak at De-signal integral 100.

66c_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_fresh
66d_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used
66h_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used

S4.3. F-20 Feldspar (Feldspar Corporation)

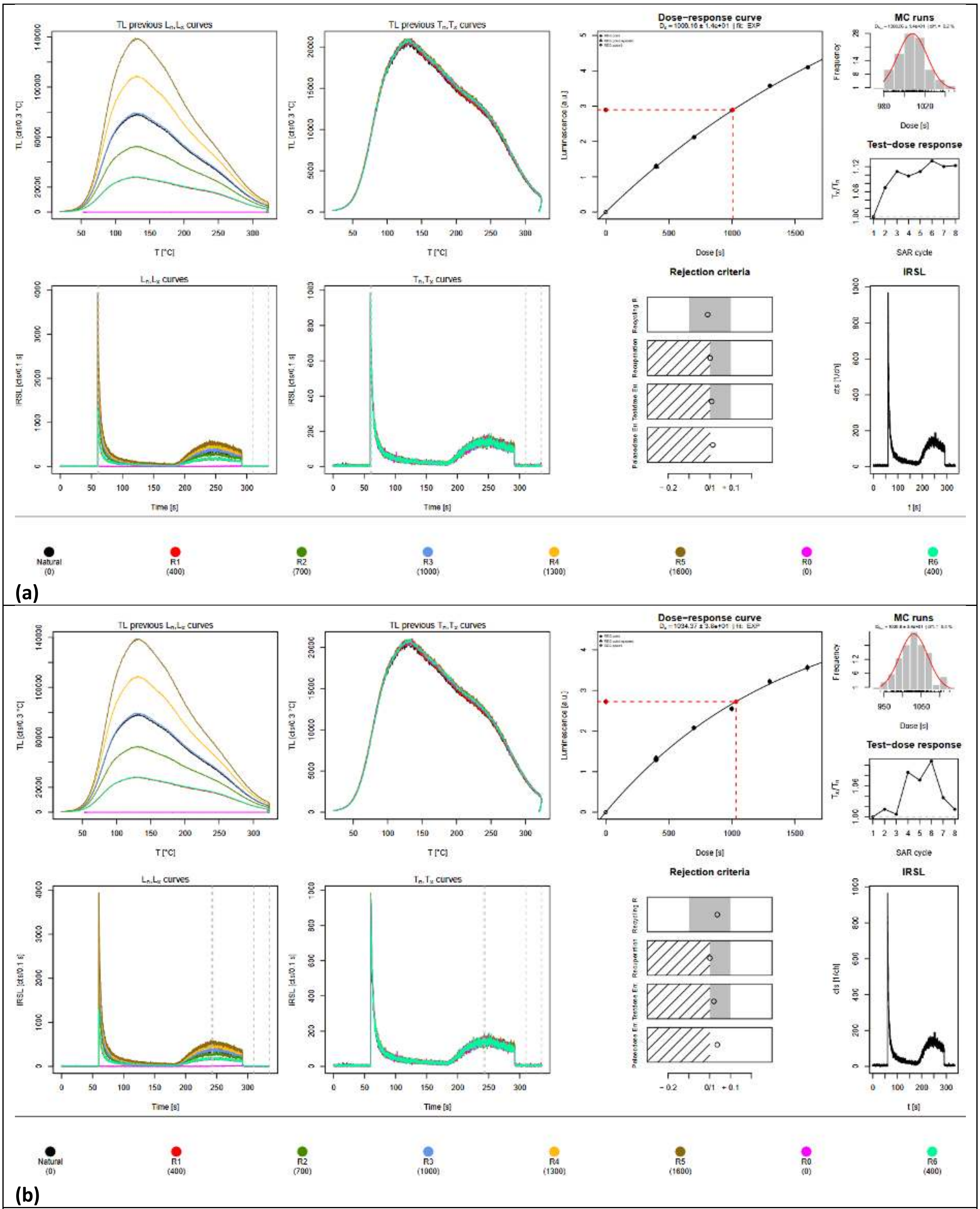


Figure S4.3.1. Dose recovery test on used aliquot of **F20** coarse-grain feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Recovered dose for De-signal integral 1. **(b)** Recovered dose for De-signal integral 100 (PET-peak).

66i_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used

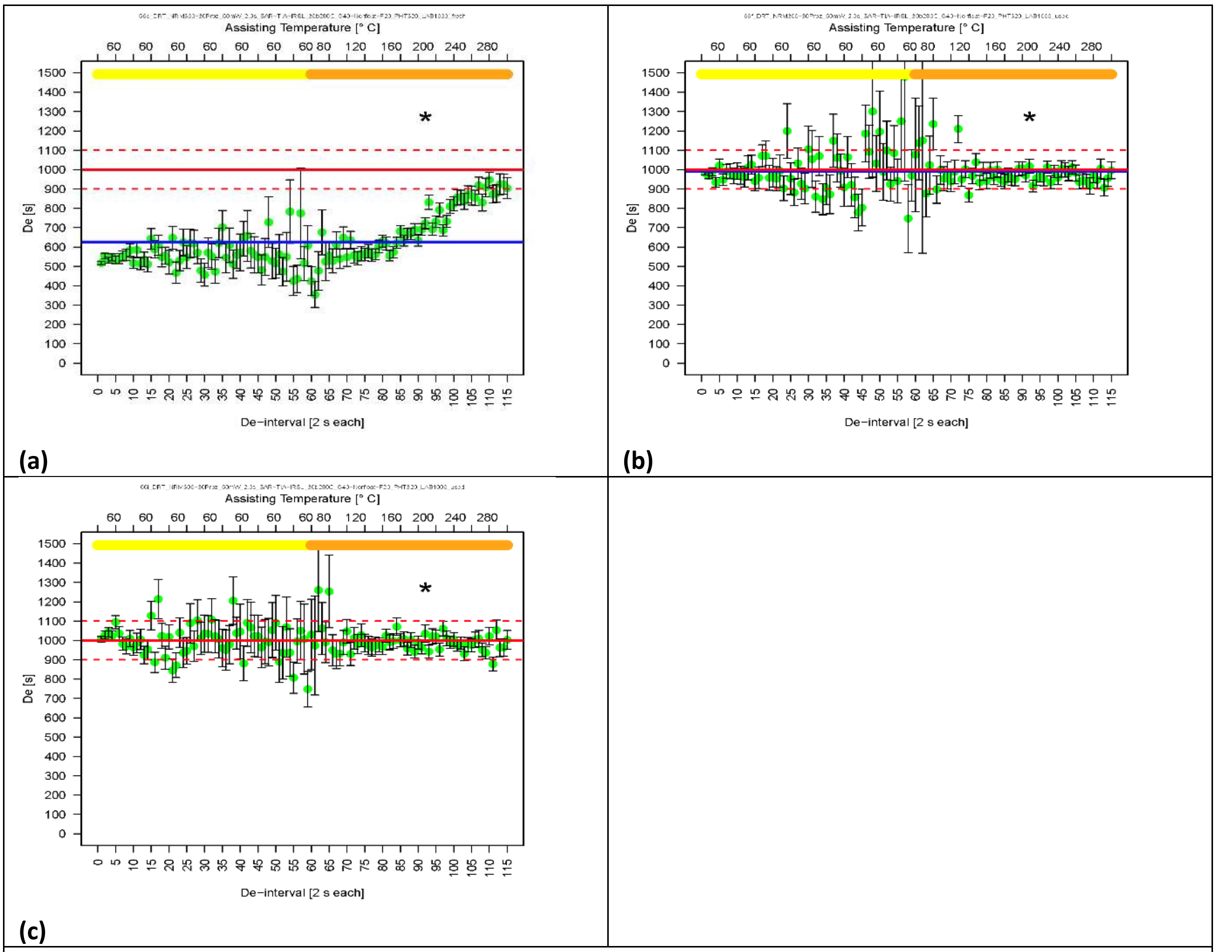


Figure S4.3.2. Dose recovery test on **F20** feldspar, tiny aliquot, LAB 1000 s, NRM 300 s. **(a)** Fresh aliquot underestimating the administered dose. **(b –c)** Used aliquot recovering the administered dose well within an error range of 10 % over the complete PET-IRSL readout. PET-peak at De-signal integral 92.

66e_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_fresh
66f_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used
66i_DRT_NRM300-30Proz_60mW_2.0s_SAR-TIA-IRSL_30b280C_G40-Norfloat-F20_PHT320_LAB1000_used

Comment

Fig. S4.1.1–S4.3.2 illustrate DRT measurements on tiny aliquots of the three commercially available feldspars G-40 (10.4 wt.% KO_2), Norfloat Potash (12.0 wt.% KO_2) and F-20 (4.1 wt.% KO_2). The samples at hand have been exposed to light in an unknown manner, but still contain remnants of a geologic dose. One aliquot per sample was dosed and measured repeatedly, firstly after bleaching in the *lexsygresearch* reader with the solar lamp facility for one hour ("fresh" aliquot), secondly and thirdly subsequently to a previous PET-IRSL SAR measurement and subsequent bleaching by the solar facility (used aliquot). The DRTs were measured with PHT 320 °C, a 1000 s dose-response curve, LAB 1000 s and NRM 30 % (300 s). With these parameter values the administered dose could be recovered well for the used aliquots over the complete low- and high-temperature PET-IRSL range, but was underestimated for the "fresh" aliquots. This may be owed to a not optimized preheat temperature and/or normalisation dose (NRM). The size of NRM was an arbitrary choice and further NRM tests were not carried out, as the samples are not relevant for sediment dating, but serve as chemically well characterized specimens. As such they show a comparable PET-IRSL signal curve as the sediment samples subjected to the same parameter values, with a shine-down along the low-temperature PET-

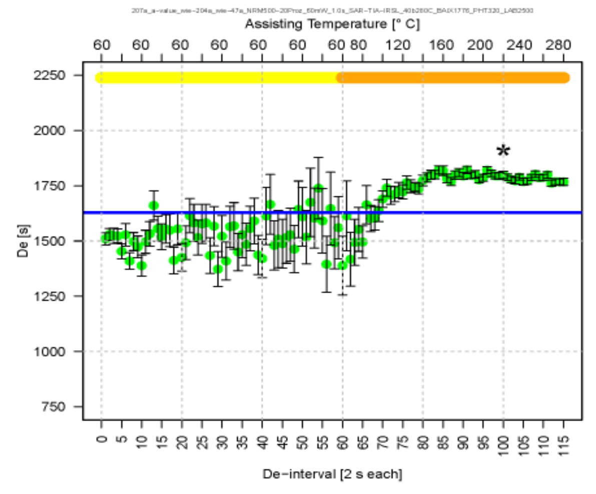
IRSL range and a signal increase starting with the beginning of the high-temperature PET-IRSL range and a PET-peak somewhere in the De-signal integral range 90–102 (180–230 °C). The position of the PET-peak varies between samples, but is rather constant for repeat measurements of the same aliquot. PET-peak positions, as roughly determined by optical inspection, are at De-signal integral 96 (ca 204 °C; G-40), De-signal integral 100 (ca 220 °C; Potash Norfloat) and De-signal intergral 92 (ca 188 °C; F-20). Thus, the PET-peak position shifts to a somewhat higher temperature with increasing potassium content. The most pronounced PET-peak is exhibited by Potash Norfloat which possesses the highest potassium content. The least pronounced and broadest PET-peak is observed for G-40, while the specimen with the lowest potassium content F-20 shows a slightly more developed PET-peak.

The coarse-grain feldspars are documented in the DigitalFire.com Reference Library – G40, Norfloat (potash) and F20 (https://digitalfire.com/).

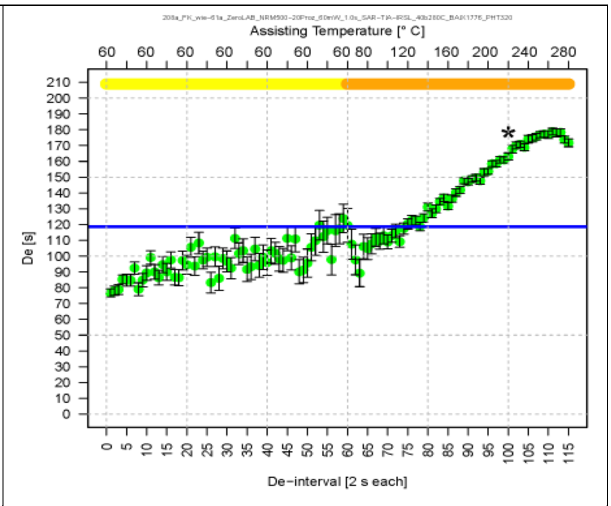
S5. Supplement 5 – a-value and g-value determination

S5.1. a-value and g-value-determination - A

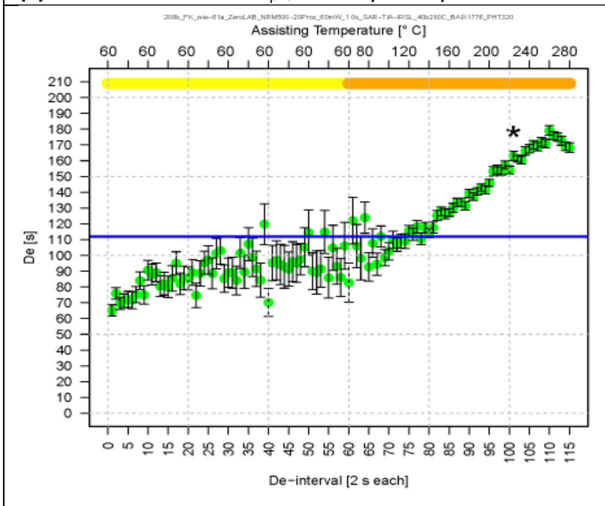
- **a-value determination on HDS-1776 – Fig. S5.1.1 – S5.1.2**
- **a-value determination on HDS-1849 – Fig. S5.2.1 – S5.2.4**
- **g-value determination on HDS-1776 – Fig. S5.3.1 – S5.3.3**
- **g-value determination on HDS-1827 – Fig. S5.4.1 – S5.4.3**
- **g-value determination on HDS-1849 – Fig. S5.5.1 – S5.5.3**



(a) HDS-1776 – FG – LAB$_{Alpha}$ 1563 Gy – Aliquot 1



(b) HDS-1776 – FG – zero-dose test – Aliquot 2

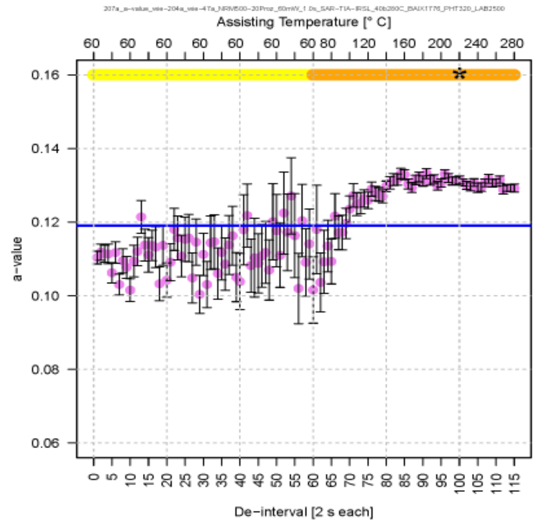


(c) HDS-1776 – FG – zero-dose test – Aliquot 3

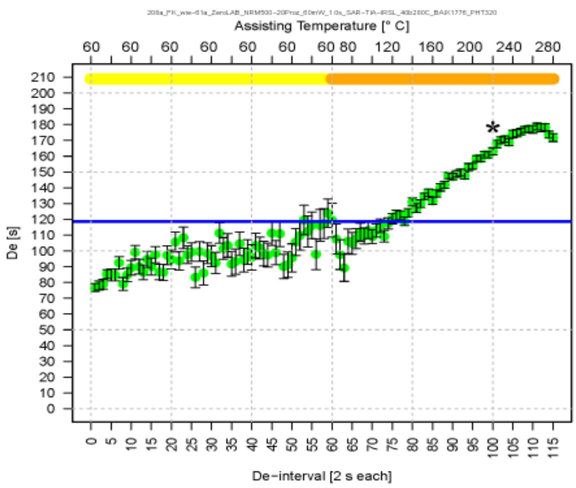
Figure S5.1.1. Beta-equivalent dose of LAB$_{alpha}$ 1563 Gy (6 h 25 min alpha irradiation) on aliquot 1, which prior to the administration of LAB$_{alpha}$ had been bleached for one hour in the solar simulator of reader LR03 (PULSE) (for a-value calculation and presentation cf. **Fig. S5.1.2b**. **(b, c)** Zero-dose tests on aliquot 2 and aliquot 3 (for subsequent a-value determination cf. **Fig. S5.1.2d, f)**. The residual dose conforms to ca 7 % (low-temperature PET-IRSL range) to ca 10 % (late high-temperature PET-IRSL range) of the beta-equivalent dose in **Fig. S5.1.2a.** Beta-equivalent doses represented in s beta-irradiation time on reader LR03 (PULSE; 0.111 Gy/s).

- 207a_a-value_wie-204a_wie-47a_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500 – POS-31
- 208a_FK_wie-61a_ZeroLAB_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320 – POS-32
- 208b_FK_wie-61a_ZeroLAB_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320 – POS-33

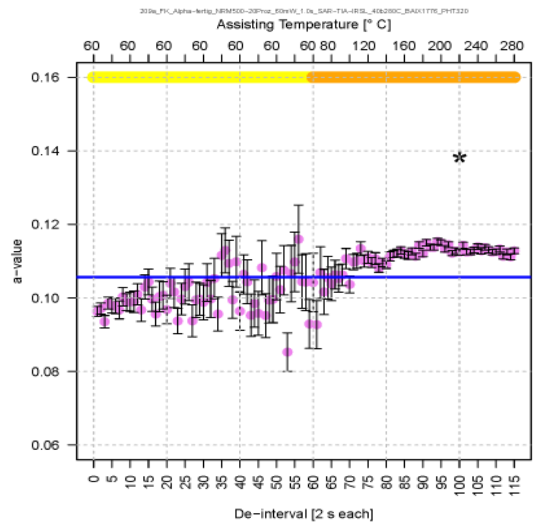
(a) HDS-1776 – FG – **no** zero-dose test – Aliquot 1



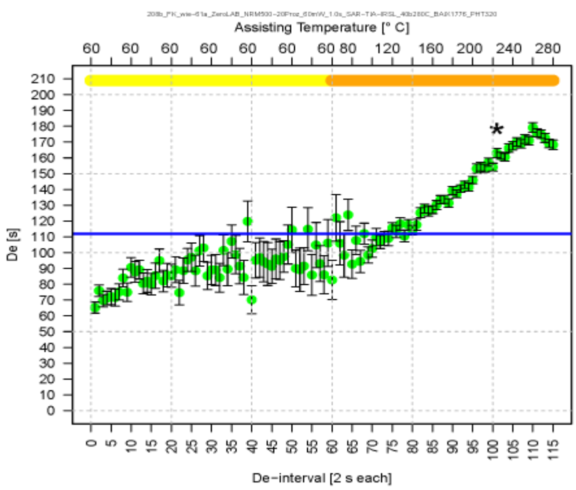
(b) HDS-1776 – FG – LAB$_{Alpha}$ 1563 Gy – Aliquot 1



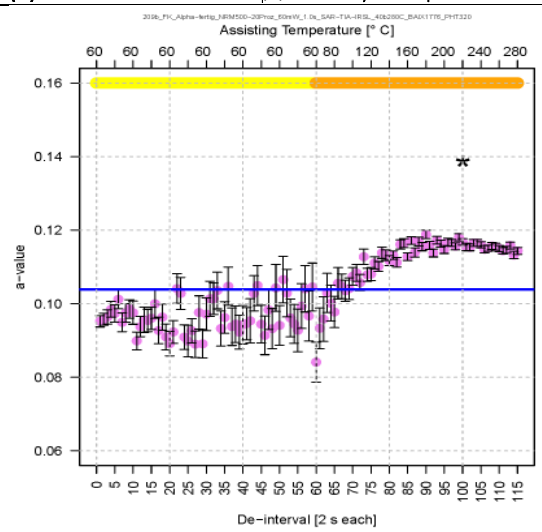
(c) HDS-1776 – FG – zero-dose test – Aliquot 2



(d) HDS-1776 – FG – LAB$_{Alpha}$ 1563 Gy – Aliquot 2



(e) HDS-1776 – FG – zero-dose test – Aliquot 3



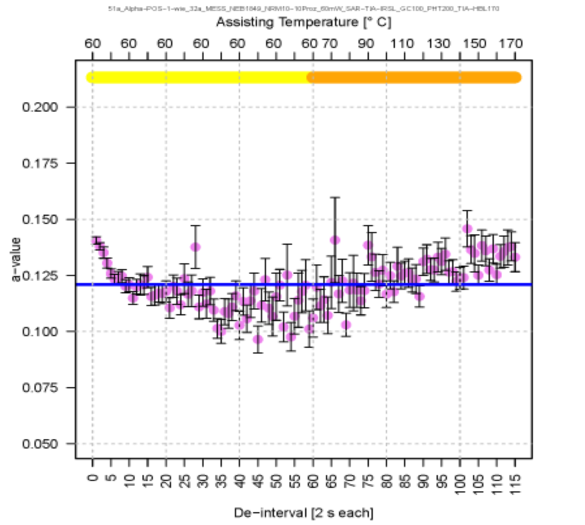
(f) HDS-1776 – FG – LAB$_{Alpha}$ 1563 Gy – Aliquot 3

Figure S5.1.2. a-value determination on three aliquots of powdered feldspar extracts from HDS-1776. **(a)** No zero-dose test. The aliquot was bleached in the solar simulator before receiving LAB$_{alpha}$ 1563 Gy (6 h 25 min alpha irradiation). **(c, e)** Zero-dose measurements on aliquot 2 and aliquot 3. These may represent by size the residual dose also present in **(b)**. Beta-equivalent doses represented in s beta-irradiation time on reader LR03 (PULSE; 0.111 Gy/s). **(d, f)** a-value determination on the aliquots in **(c, e)** after the zero-dose test. Like aliquot 1 with LAB$_{alpha}$ 1563 Gy (6 h 25 min alpha irradiation). The a-values of **(d, f)** are slightly lower than the a-values of **(b)**: ca 0.11 versus ca 0.10 in the low-temperature

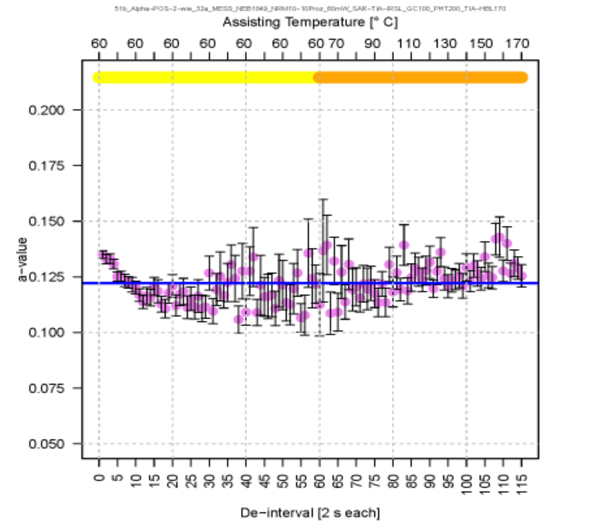
PET-IRSL range and ca 0.115–0.12 versus 0.13 in the high-temperature PET-IRSL range. The difference is likely owed to the small residual dose in **(b)**. The graphs in **(c, e)** are identical to those in **Fig. S5.1.1 (b, c)**.

- 207a_a-value_wie-204a_wie-47a_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320_LAB2500 – POS-31
- 208a_FK_wie-61a_ZeroLAB_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320 – POS-32 mit 209a_Alpha_...
- 208b_FK_wie-61a_ZeroLAB_NRM500-20Proz_60mW_1.0s_SAR-TIA-IRSL_40b280C_BAIX1776_PHT320 – POS-33 mit 209b_Alpha_...

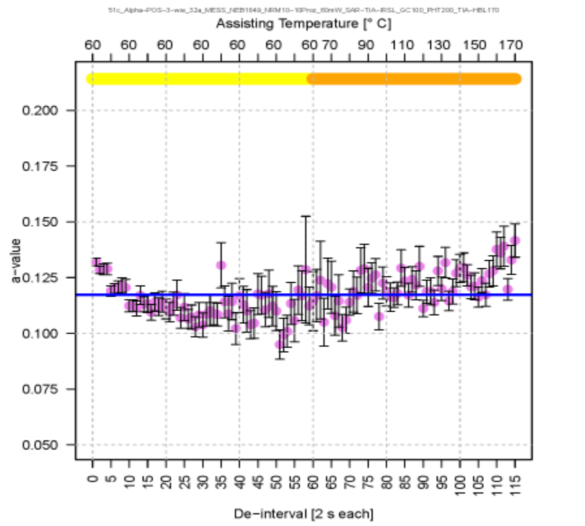
S5.2. a-value and g-value-determination - B



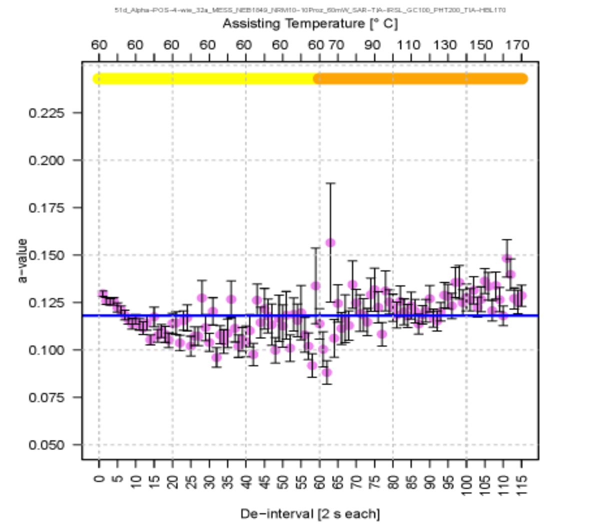
(a) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 1



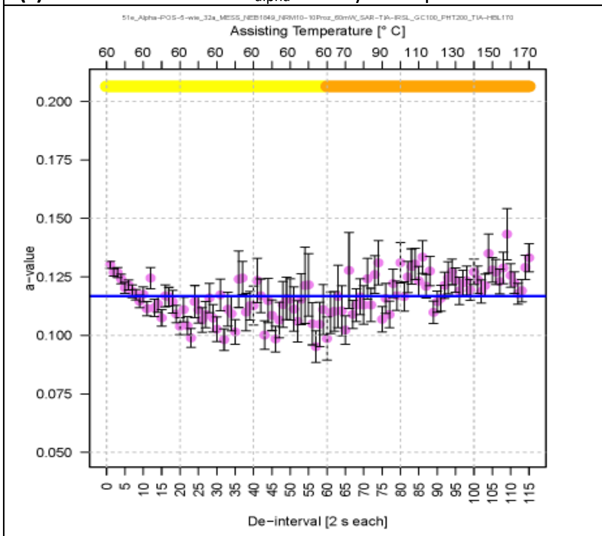
(b) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 2



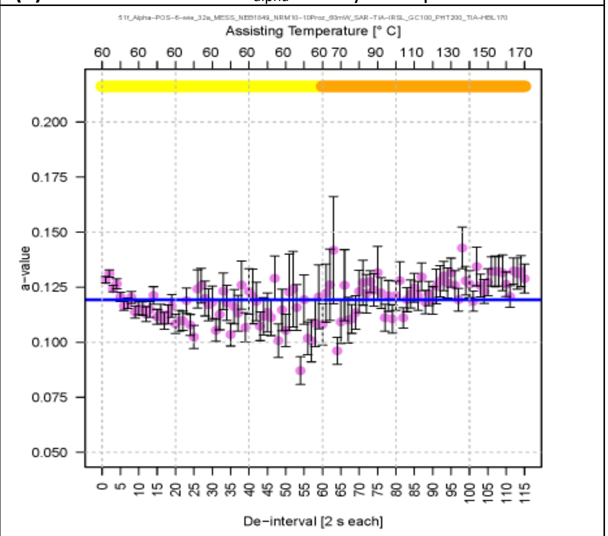
(c) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 3



(d) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 4



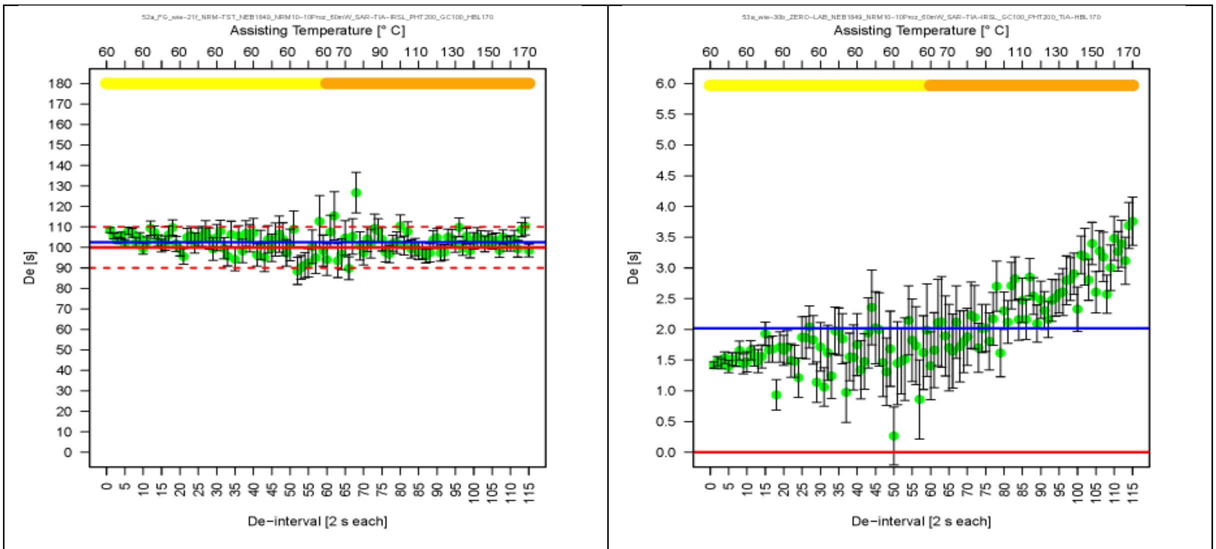
(e) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 5



(f) HDS-1849 – FG – LAB_{alpha} 146 Gy – Aliquot 6

Figure S5.2.1. a-value determination on six aliquots of powdered feldspar extracts from HDS-1849. The aliquots were bleached for one hour under the solar simulator before receiving LAB_{alpha} 146 Gy (36 min alpha irradiation) under vacuum. The aliquots were then measured with the same PET-IRSL SAR protocol as the aliquots subjected to De determination. As the a-value was slightly larger than expected, LAB_{alpha} 146 Gy had to be extrapolated on the beta-dose response curve slightly beyond REG_{max}. Yet, the results are reproducible. Nonetheless, the a-value measurements were repeated with a lower LAB_{alpha} (cf. **Fig S5.2.3** and **Fig S5.2.4**).

- 51a_Alpha-POS-1-wie_32a_MESS_NEB1849_NRM10-10Proz_60mW_SAR-TIA-IRSL_GC100_PHT200_TIA-HBL170 … MS_51a–MS_51f



(a) HDS-1849 – FG – LAB 100 s

(b) HDS-1849 – FG – no laboratory dose

Figure S5.2.2. DRT and zero-dose test on aliquots of powdered feldspar extracts from HDS-1849. The aliquots were bleached for one hour under the solar simulator before receiving **(a)** LAB 100 s on reader LR04 (COLOUR) or **(b)** no laboratory dose. The aliquots were then measured with the same PET-IRSL SAR protocol as the aliquots subjected to De determination. The tests serve to investigate whether the aliquots possess a residual dose that might increase the a-value. However, both tests show that no significant residual dose exists that might lead to an erroneously enlarged a-value.

- 52a_FG_wie-21f_NRM-TST_NEB1849_NRM10-10Proz_60mW_SAR-TIA-IRSL_PHT200_GC100_HBL170
- 53a_wie-30b_ZERO-LAB_NEB1849_NRM10-10Proz_60mW_SAR-TIA-IRSL_GC100_PHT200_TIA-HBL170

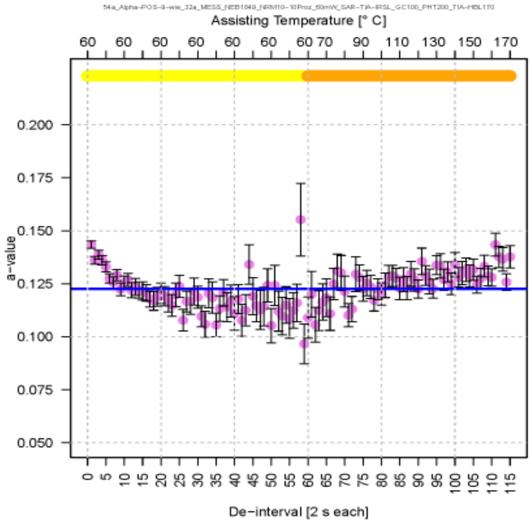
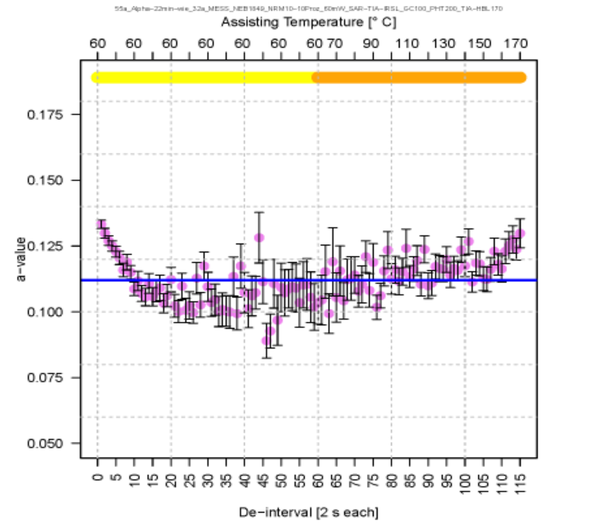
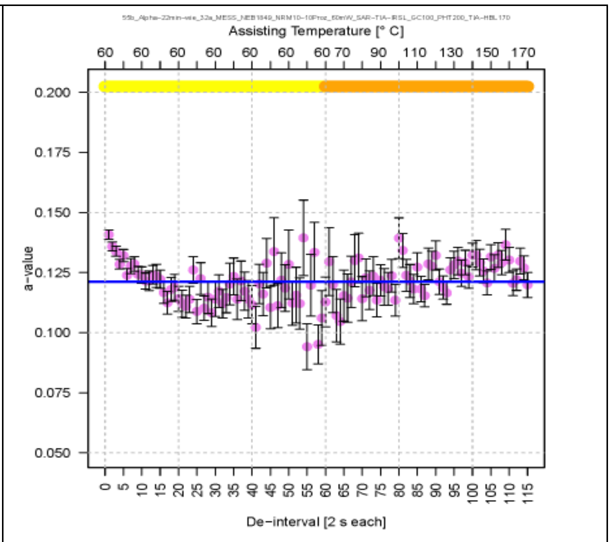
 **(a)** HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 1	

Figure S5.2.3. a-value determination on one aliquot of powdered feldspar extracts from HDS-1849. The aliquot was bleached for one hour under the solar simulator before receiving LAB_{alpha} 89 Gy (22 min alpha irradiation) under vacuum. The aliquot was then measured with the same PET-IRSL SAR protocol as the aliquots subjected to De determination. This time, the beta-equivalent of LAB_{alpha} 89 Gy was in the range of DRC-AIM of the 100 s dose-response curve. The result conforms to the measurements with LAB_{alpha} 146 Gy.

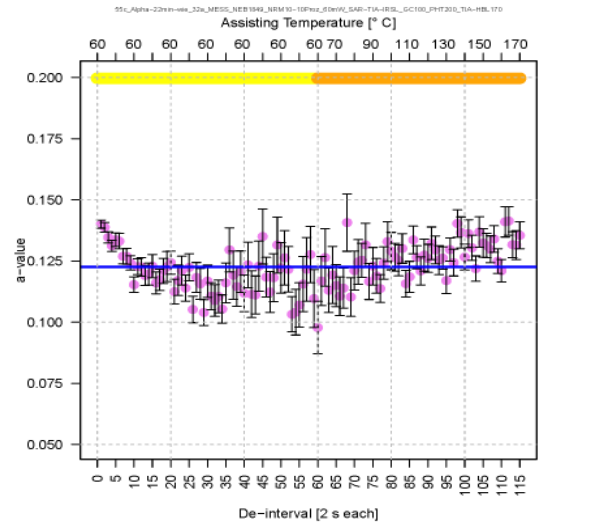
- 54a_Alpha-POS-9-wie_32a_MESS_NEB1849_NRM10-10Proz_60mW_SAR-TIA-IRSL_GC100_PHT200_TIA-HBL170



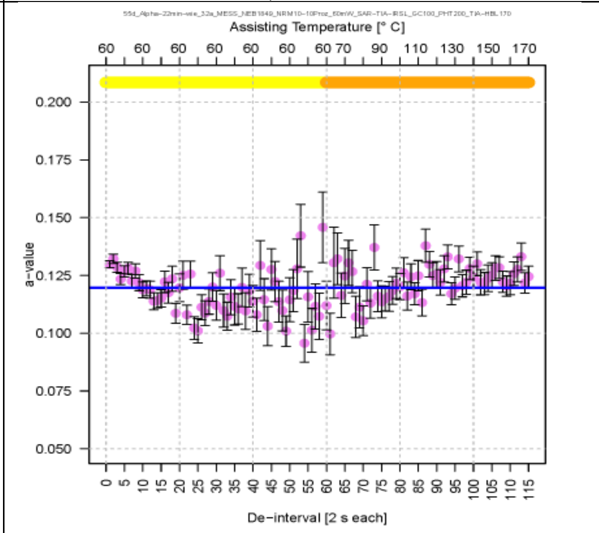
(a) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 1



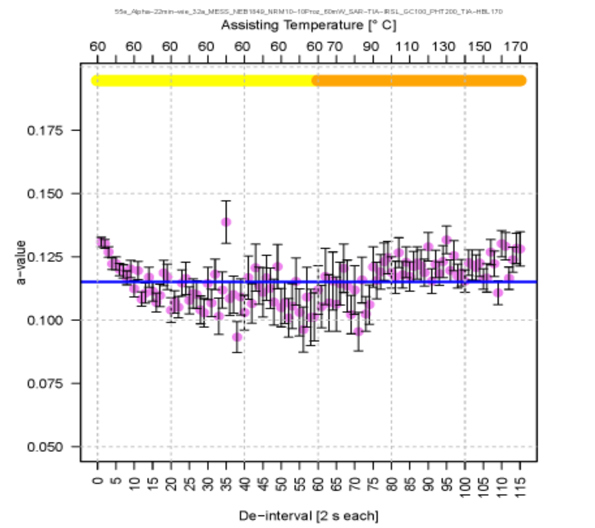
(b) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 2



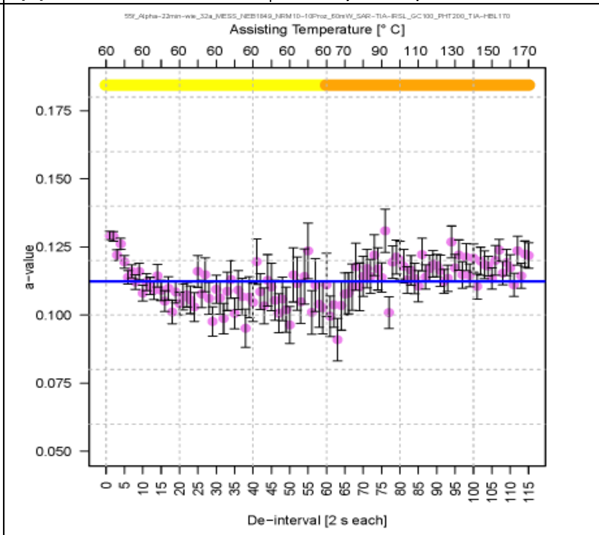
(c) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 3



(d) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 4



(e) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 5

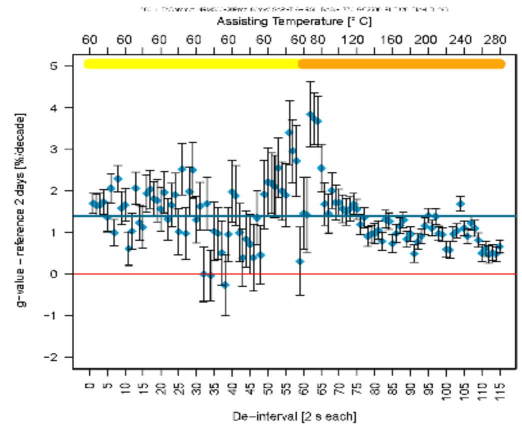


(f) HDS-1849 – FG – LAB_{alpha} 89 Gy – Aliquot 6

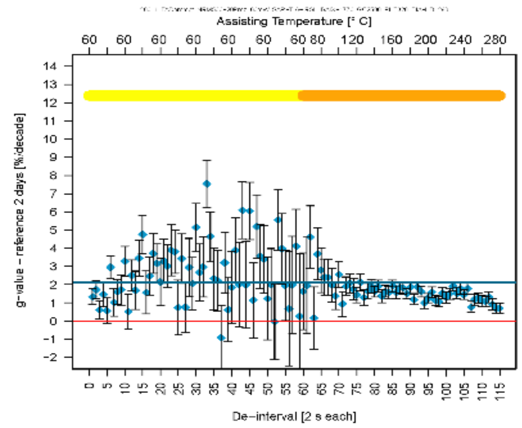
Figure S5.2.4. a-value determination on six aliquots of powdered feldspar extracts from HDS-1849. The aliquots were bleached for one hour under the solar simulator before receiving LAB_{alpha} 89 Gy (22 min alpha irradiation) under vacuum. The aliquots were then measured with the same PET-IRSL SAR protocol as the aliquots subjected to De determination. The results conform to those of the other a-value determinations both with LAB_{alpha} 146 Gy (**Fig. S5.2.1**) and LAB_{alpha} 89 Gy (**Fig. S5.2.3**).

- 55a_Alpha-22min-wie_32a_MESS_NEB1849_NRM10-10Proz_60mW_SAR-TIA-IRSL_GC100_PHT200_TIA-HBL170
- 55a_ … 55f_

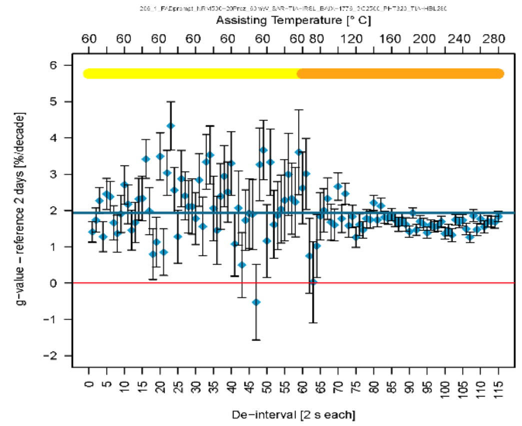
S5.3. g-value determination on 5 aliquots of BAIX-1776 (cf. MS_205a–205b) – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – GC 2500 s – NRM 20 % of DRC-AIM – LAB 1750 s – PHT 320 °C – PET$_{max}$ and HBL 280 °C



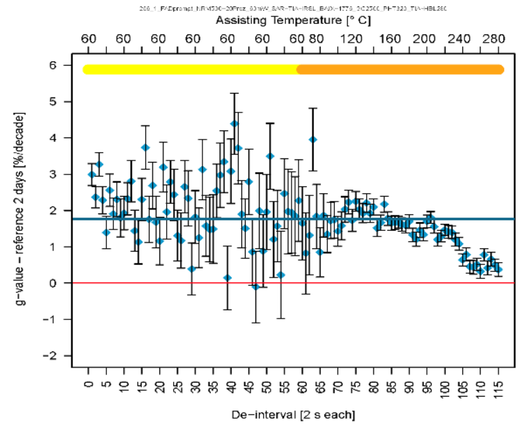
(a) Aliquot 1 – from MS_205a



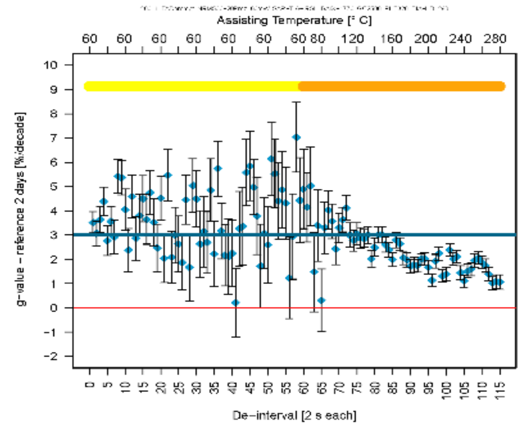
(b) Aliquot 2 – from MS_205b



(c) Aliquot 3 – from MS_205c



(d) Aliquot 4 – from MS_205d



(e) Aliquot 5 – from MS_205e

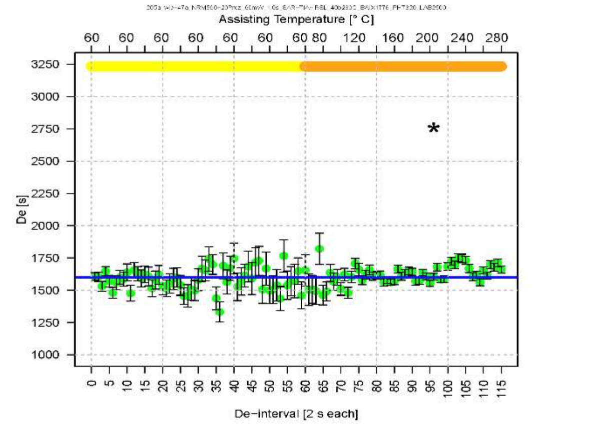
g-values first 15 vs last 15 De integrals:

- Aliquot-1: 1.52 vs 0.83
- Aliquot-2: 1.94 vs 1.26
- Aliquot-3: 1.94 vs 1.60
- Aliquot-4: 2.18 vs 0.78
- Aliquot-5: 3.79 vs 1.6

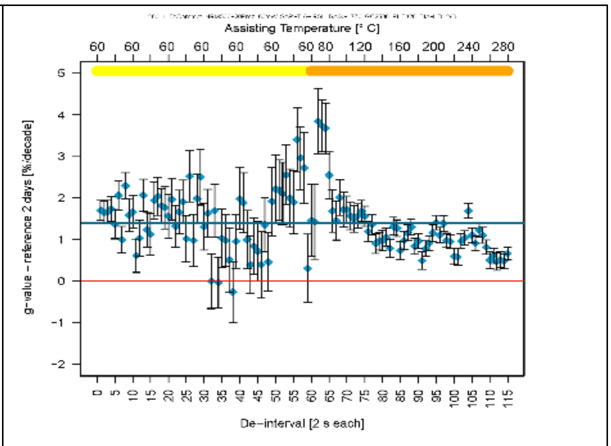
Figure S5.3.1. MS_206_1–MS_206_12. g-value determination on five aliquots of HDS-1776 previously used for De determination (cf. **MS_205a–MS_205e)** applying the same PET-IRSL SAR protocol as in **MS_205**: DRC aiming at 2500 s (100 %), NRM 20 % of DRC-AIM, PHT 320 °C, PET$_{max}$ and HBL 280 °C. Customary measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. LAB$_{FAD}$ 1750 s. Five prompt readouts with ca 1143 s delay after half of the beta irradiation time of 1750 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C).

Measurements 206_1 – MS_206_12, each Aliquot 1–5

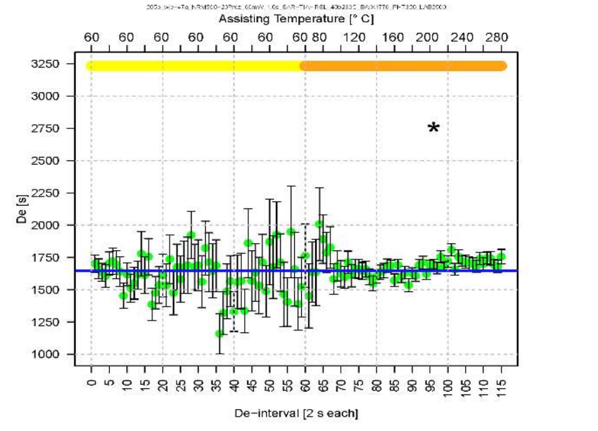
S5.3.1. Contrasting g-value data curves and De-value data curves of 5 aliquots of BAIX-1776 (cf. MS_205a–205b) – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – GC 2500 s – NRM 20 % of DRC-AIM – LAB 1750 s – PHT 320 °C – PET_{max} and HBL 280 °C



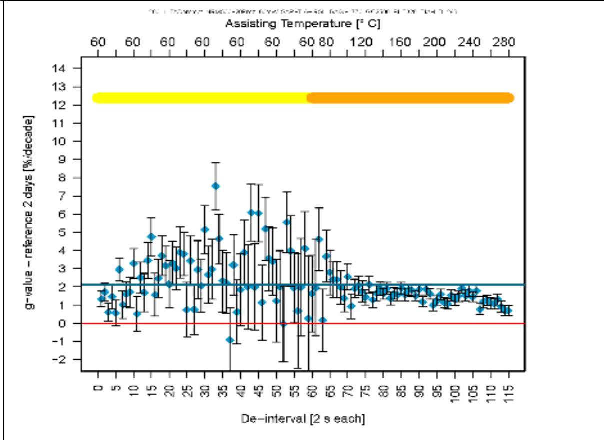
(a-1) Aliquot 1 – MS_205a



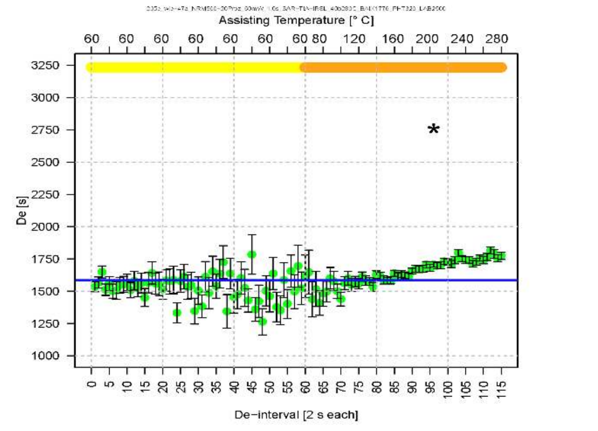
(a-2) Aliquot 1 – from MS_205a



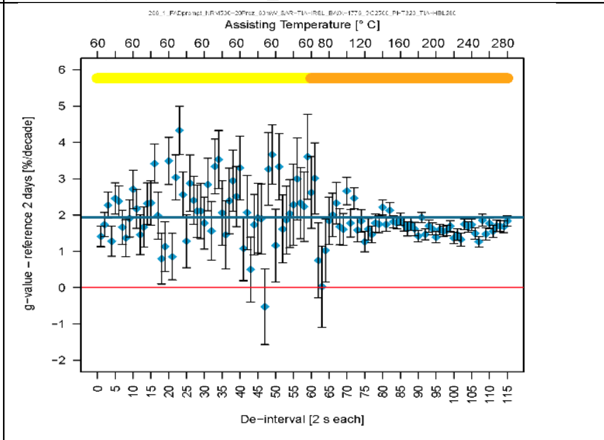
(b-1) Aliquot 2 – MS_205b



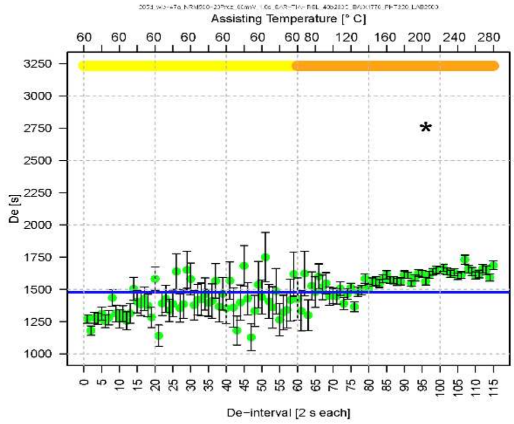
(b-2) Aliquot 2 – from MS_205b



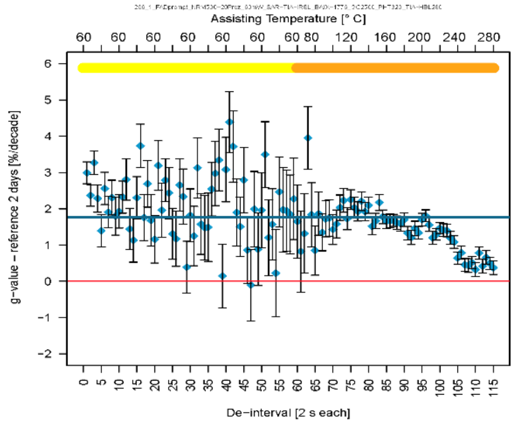
(c-1) Aliquot 3 – MS_205c



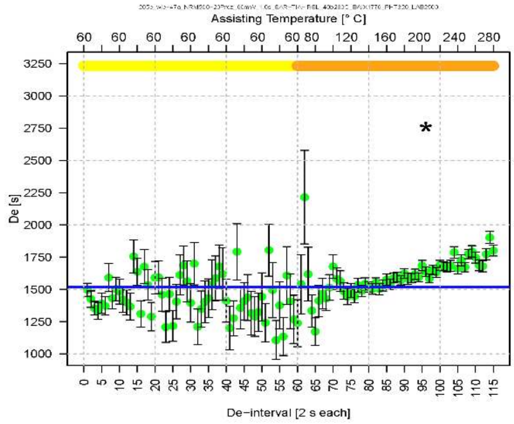
(c-2) Aliquot 3 – from MS_205c



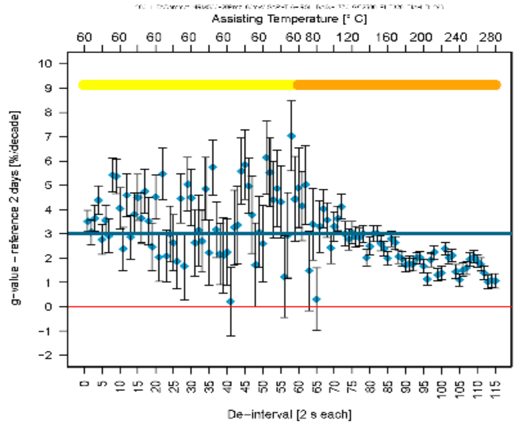
(d-1) Aliquot 4 – MS_205d



(d-2) Aliquot 4 – from MS_205d



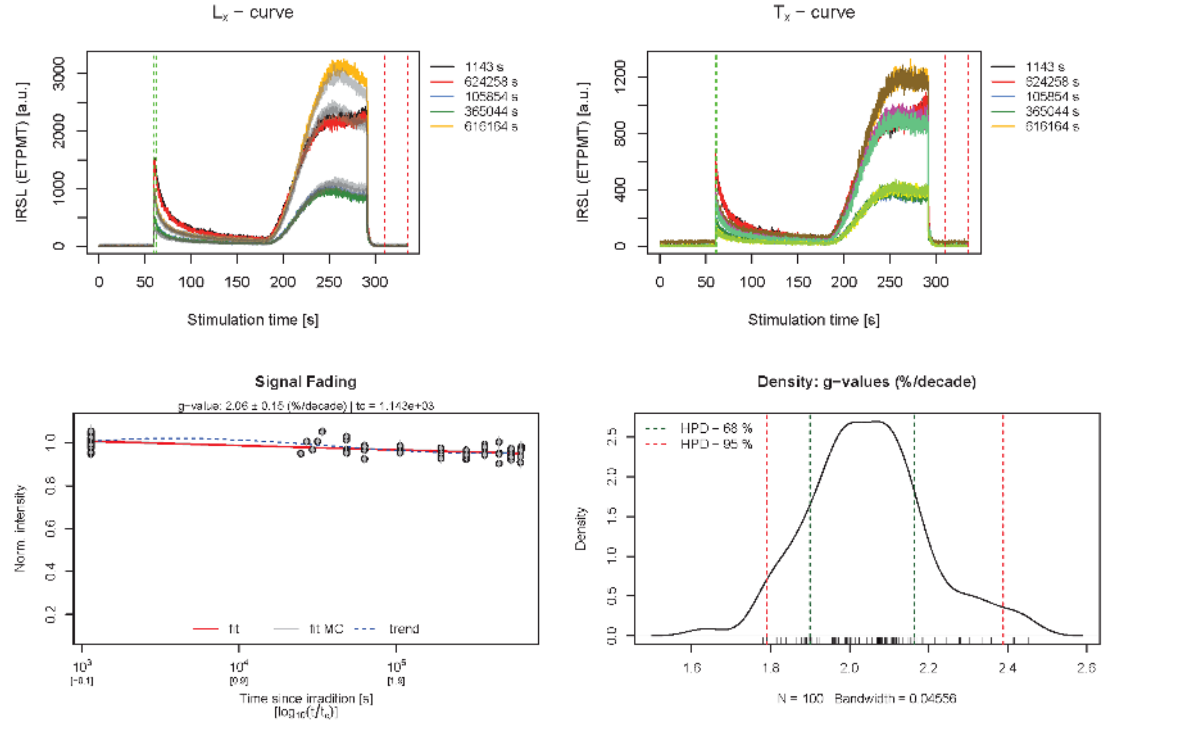
(e-1) Aliquot 5 – MS_205e



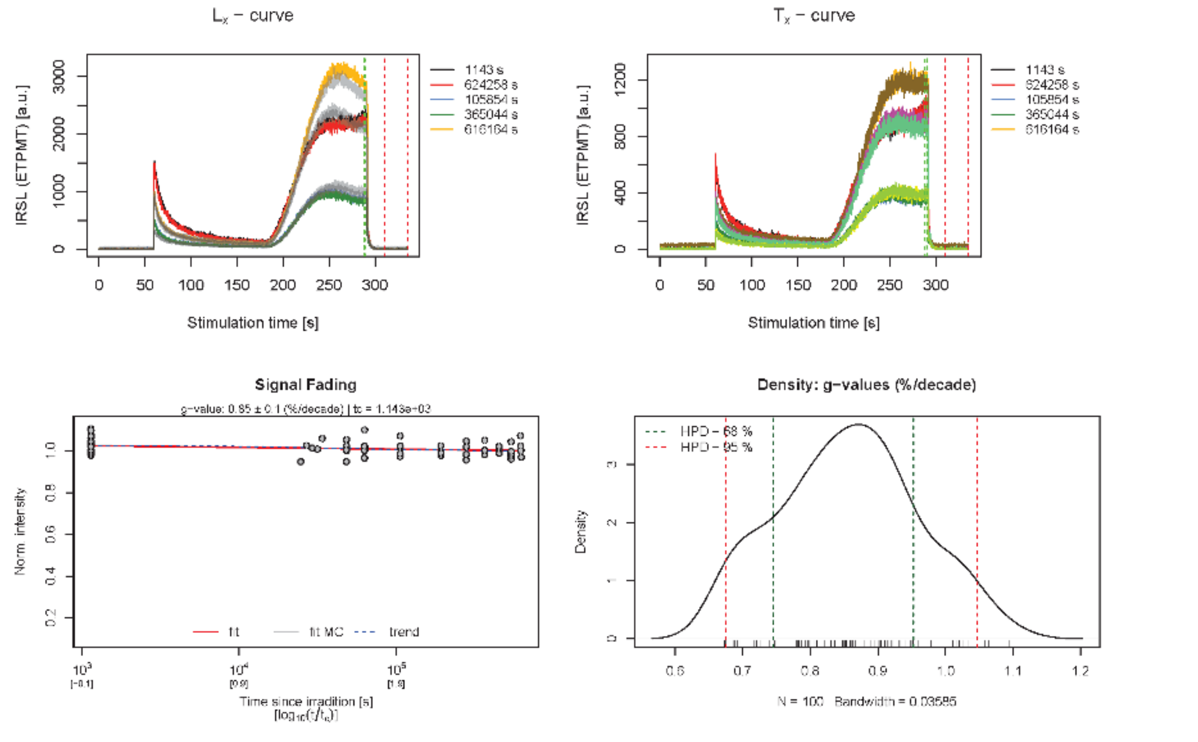
(e-2) Aliquot 5 – from MS_205e

Figure S5.3.2. MS_206_1–MS_206_12. De-value determination (left) and g-value determination (right) on five aliquots of HDS-1776 previously used for De determination (cf. **MS_205a–MS_205e)** applying the same PET-IRSL SAR protocol as in **MS_205**: DRC aiming at 2500 s (100 %), NRM 20 % of DRC-AIM, PHT 320 °C, PET_{max} and HBL 280 °C. Customary measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. LAB_{FAD} 1750 s**.** Five prompt readouts with ca 1143 s delay after half of the beta irradiation time of 1750 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). The graphs in the right column are identical with those from **Fig. S5.3.1a–e.**

Measurements 206_1 – MS_206_12, each Aliquot 1–5



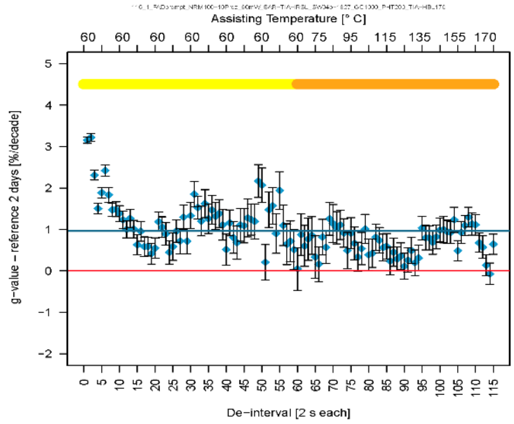
(a) g-value determination for De integral 1



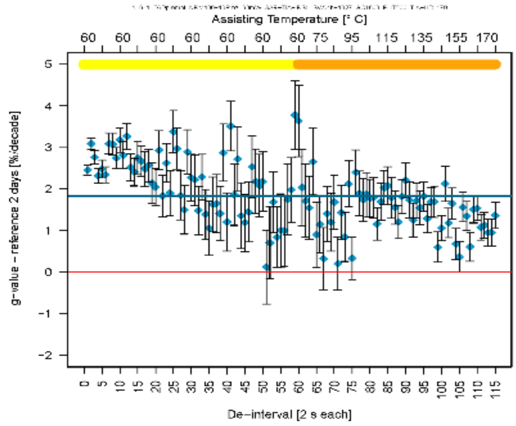
(b) g-value determination for De integral 115

Figure S5.3.3. MS_206_1–MS_206_12. g-value determination of all 5 aliquots of HDS-1776 together. Graphical output of the function "analyse_FadingMeasurement" (version 0.1.23) (Kreutzer & Burow 2025) for the first and last of 115 De integrals.

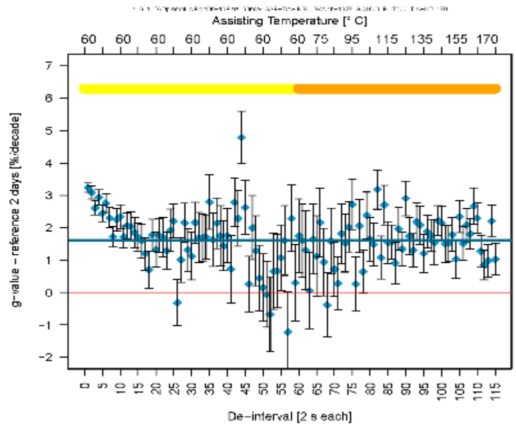
S5.4. g-value determination on 5 aliquots of SW04b-1827 (cf. MS_144b–114d, 114h, 114t) – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – LAB 400 s – PHT 200 °C – PET$_{max}$ and HBL 170 °C



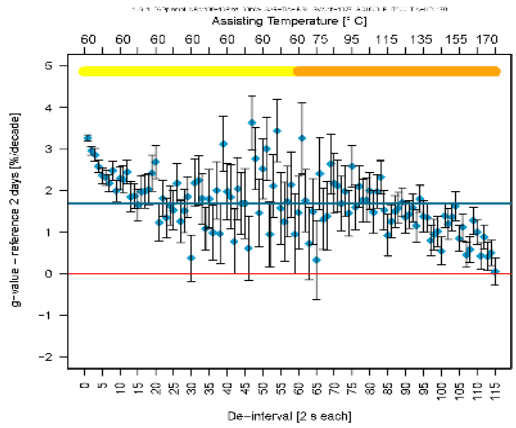
(a) Aliquot 1 – from MS_114b



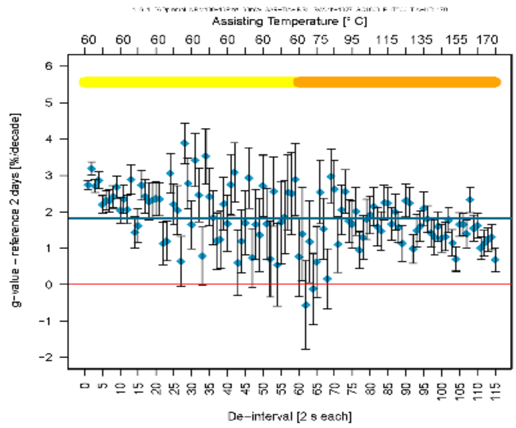
(b) Aliquot 2 – from MS_114c



(c) Aliquot 3 – from MS_114d



(d) Aliquot 4 – from MS_114h

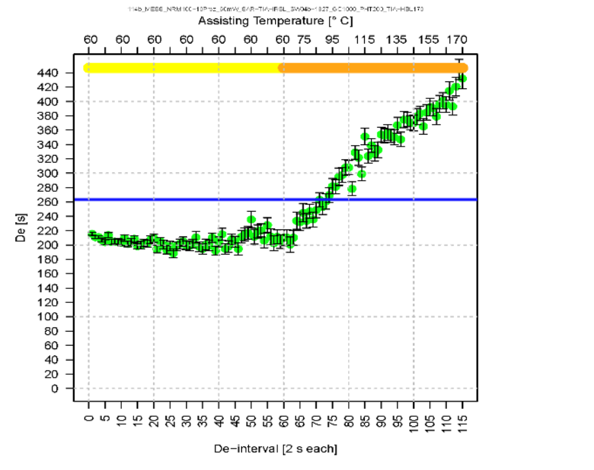


(e) Aliquot 5 – from MS_114t

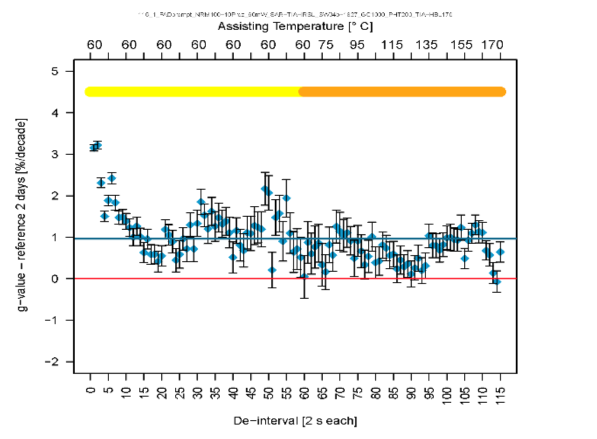
Figure S5.4.1. MS_116_1–MS_116_12. g-value determination on five aliquots of HDS-1827 previously used for De determination (cf. **MS_114b, 144c, 114d, 114h, 114t**) applying the same PET-IRSL SAR protocol as in **MS_116**: DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET$_{max}$ and HBL 170 °C. Customary measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. LAB$_{FAD}$ 400 s. Five prompt readouts with ca 440 s delay after half of the beta irradiation time of 400 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C).

Measurements 116_1 – MS_116_12, Aliquot 1–5

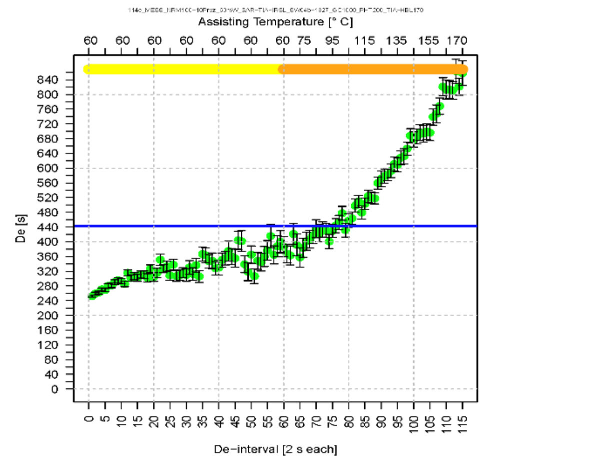
S5.4.1. Contrasting g-value data curves and De-value data curves of 5 aliquots of SW04b-1827 (cf. MS_144b–114d, 114h, 114t) – PET "classic" – IR 60 mW/cm² – GRSL hotbleach every SAR half-cycle – DRC 1000 s – NRM 10 % of DRC-AIM – LAB 400 s – PHT 200 °C – PET_{max} and HBL 170 °C



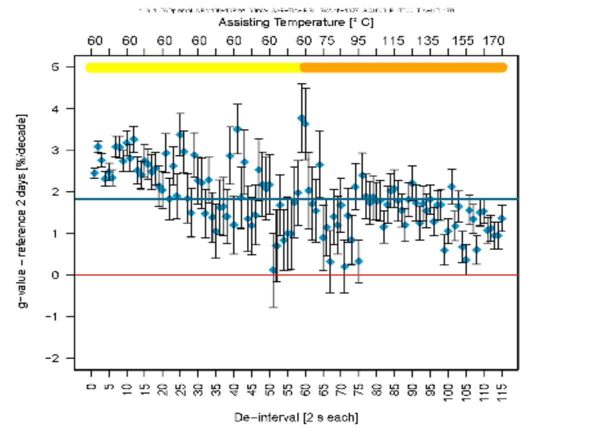
(a-1) Aliquot 1 – MS_114b



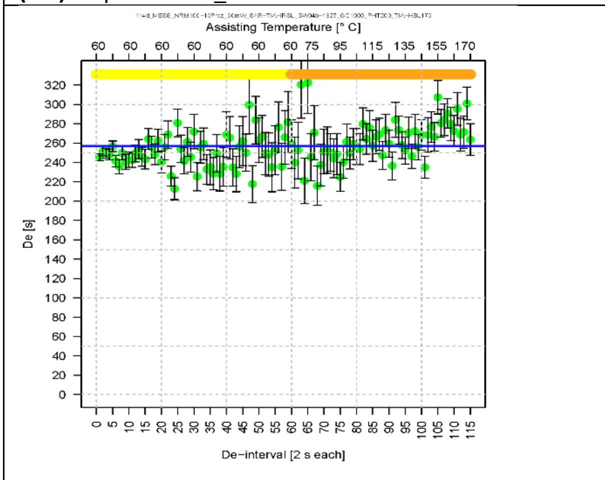
(a-2) Aliquot 1 – from MS_114b



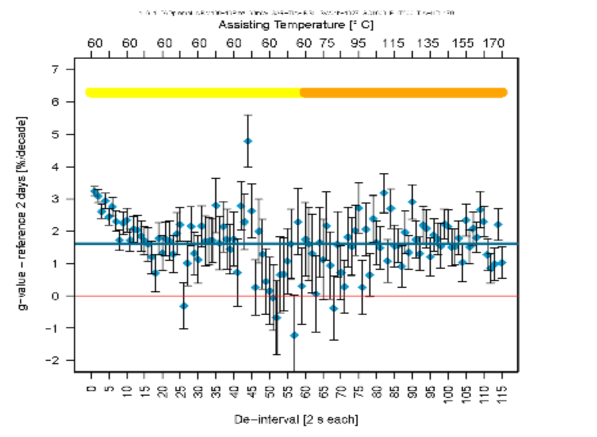
(b-1) Aliquot 2 – MS_114c



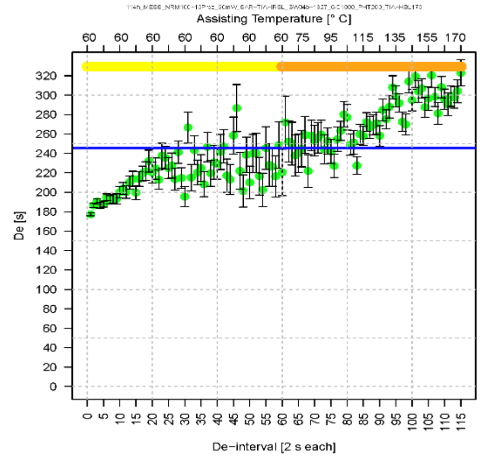
(b-2) Aliquot 2 – from MS_114c



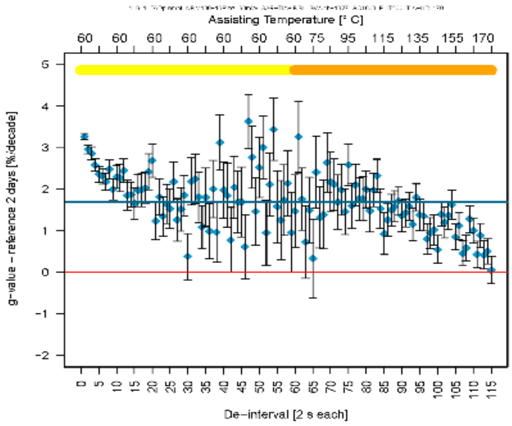
(c-1) Aliquot 3 – MS_114d



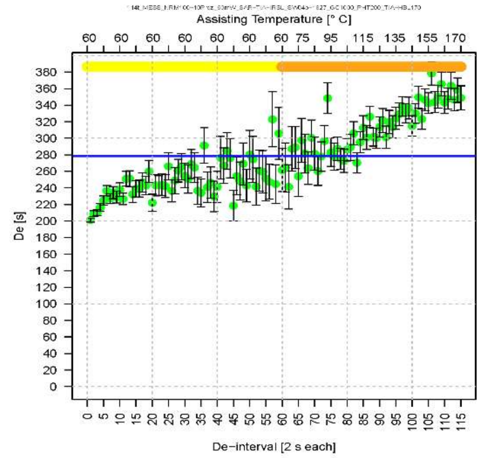
(c-2) Aliquot 3 – from MS_114d



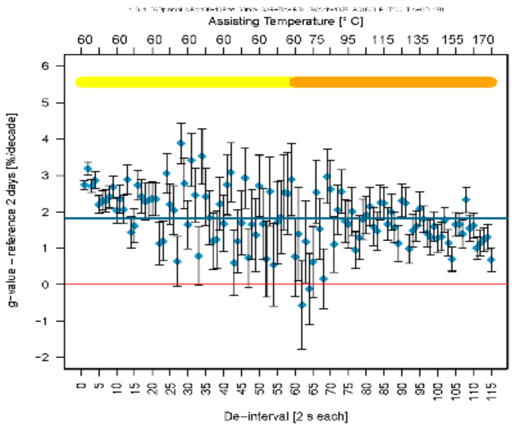
(d-1) Aliquot 4 – MS_114h



(d-2) Aliquot 4 – from MS_114h



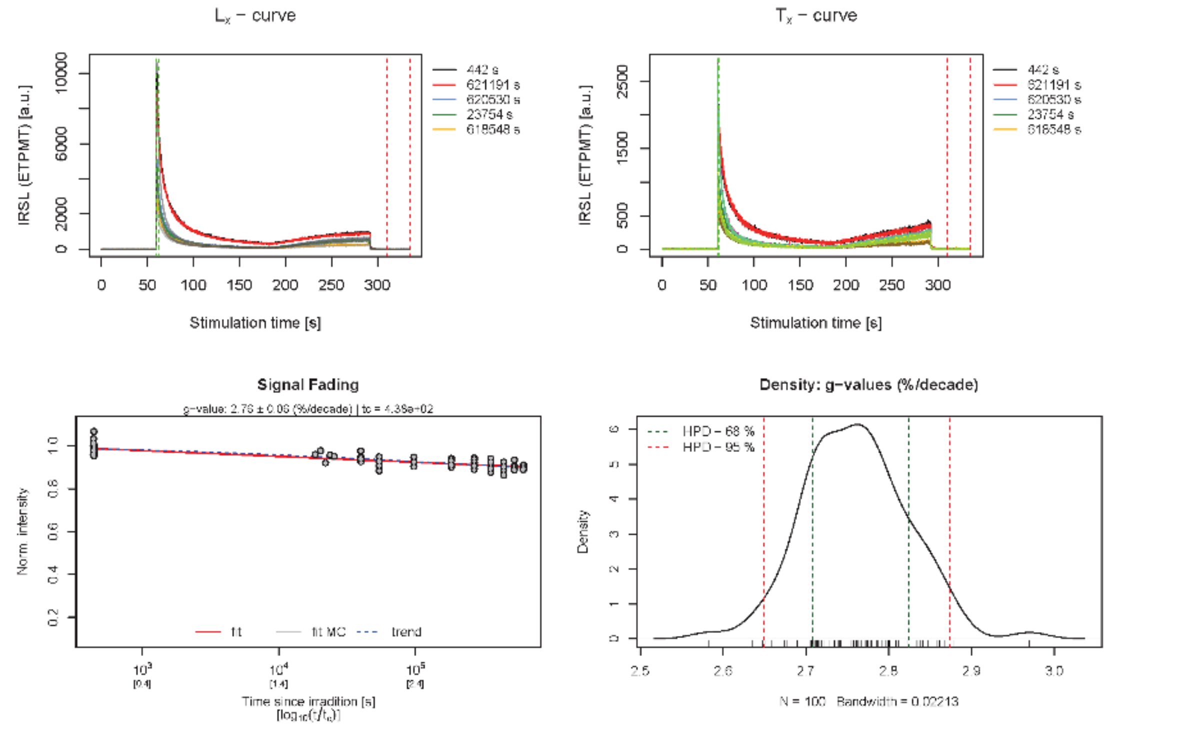
(e-1) Aliquot 5 – MS_115t



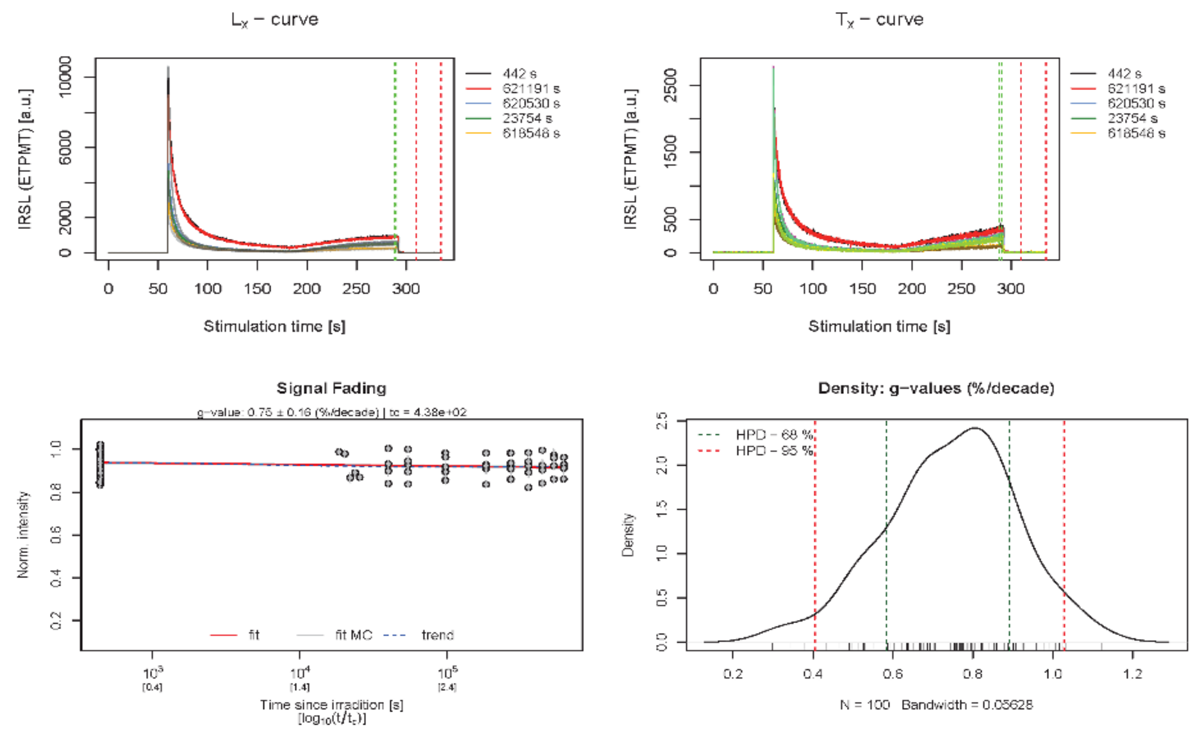
(e-2) Aliquot 5 – from MS_114t

Figure S5.4.2. MS_116_1–MS_116_12. De-value determination (left) and g-value determination (right) on five aliquots of HDS-1827 previously used for De determination (cf. **MS_114b, 144c, 114d, 114h, 114t**) applying the same PET-IRSL SAR protocol as in **MS_116**: DRC aiming at 1000 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. Customary measurement parameters: IR 60 mW/cm², GRSL hotbleach in each SAR half-cycle. LAB_{FAD} 400 s. Five prompt readouts with ca 440 s delay after half of the beta irradiation time of 400 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). The graphs in the right column are identical with those from **Fig. S5.4.1a–e.**

Measurements 116_1 – MS_116_12, Aliquot 1–5



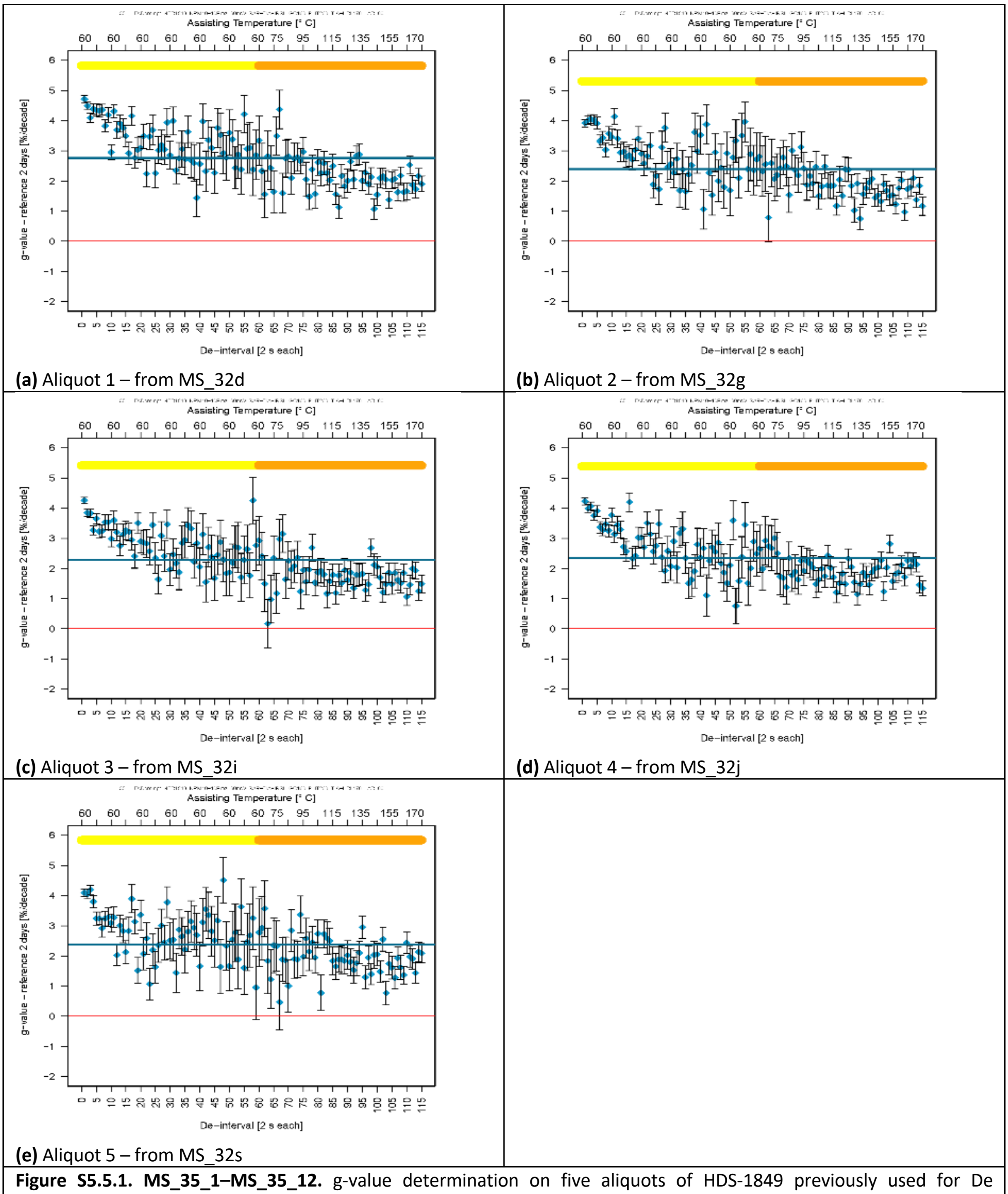
(a) g-value determination for De integral 1



(b) g-value determination for De integral 115

Figure S5.4.3. MS_116_1–MS_116_12. g-value determination of all 5 aliquots of HDS-1827 together. Graphical output of the function “analyse_FadingMeasurement” (version 0.1.23) (Kreutzer & Burow 2025) for the first and last of 115 De integrals.

S5.5. g-value determination on 5 aliquots of NEB-1849 (cf. MS_32d, 32g, 32i, 32j, 32s) – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % of DRC-AIM – LAB 100 s – PHT 200 °C – PET_{max} and HBL 170 °C



(a) Aliquot 1 – from MS_32d

(b) Aliquot 2 – from MS_32g

(c) Aliquot 3 – from MS_32i

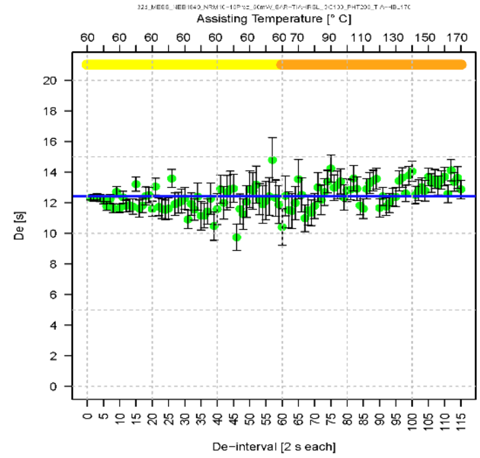
(d) Aliquot 4 – from MS_32j

(e) Aliquot 5 – from MS_32s

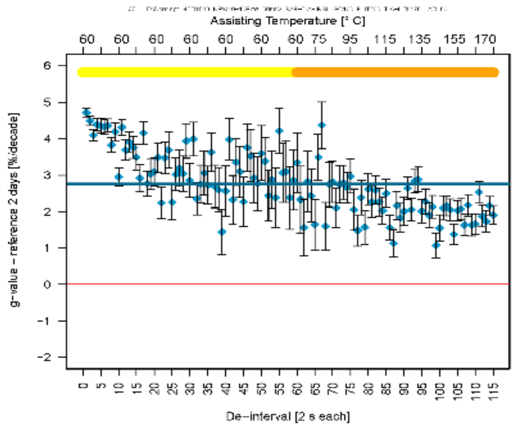
Figure S5.5.1. MS_35_1–MS_35_12. g-value determination on five aliquots of HDS-1849 previously used for De determination (cf. **MS_32d, 32g, 32i, 32j, 32s**) applying the same PET-IRSL SAR protocol as in **MS_32**: DRC aiming at 100 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. Customary measurement parameters: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. LAB_{FAD} 100 s. Five prompt readouts with ca 288 s delay after half of the beta irradiation time of 100 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C).

Measurements 35_1 – MS_35_12, Aliquot 1–5

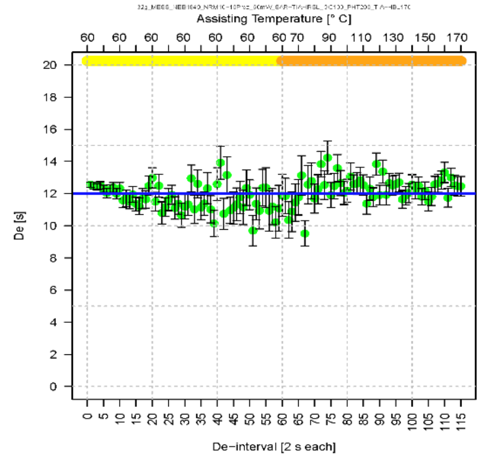
S5.5.1. Contrasting g-value data curves and De-value data curves of 5 aliquots of NEB-1849 (cf. MS_32d, 32g, 32i, 32j, 32s) – IR 60 mW/cm² – BLSL hotbleach every SAR half-cycle – DRC 100 s – NRM 10 % of DRC-AIM – LAB 100 s – PHT 200 °C – PET_{max} and HBL 170 °C



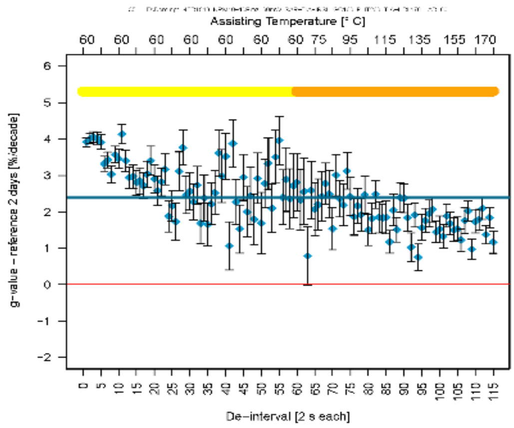
(a-1) Aliquot 1 – MS_32d



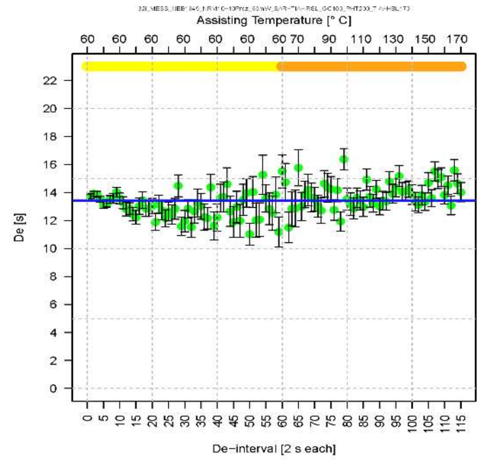
(a-2) Aliquot 1 – from MS_32d



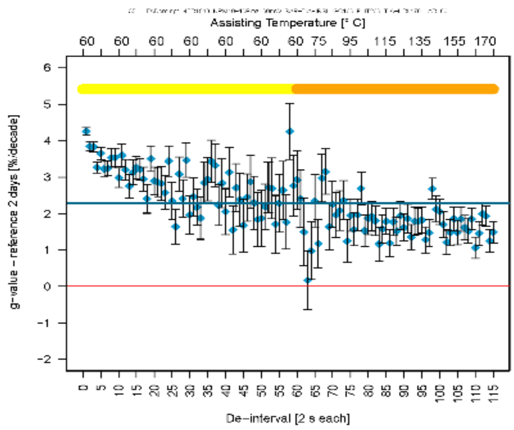
(b-1) Aliquot 2 – MS_32g



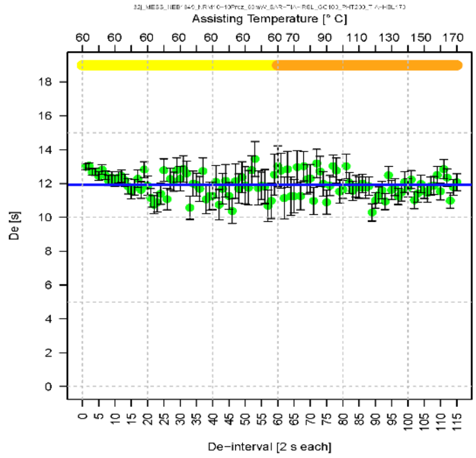
(b-2) Aliquot 2 – from MS_32g



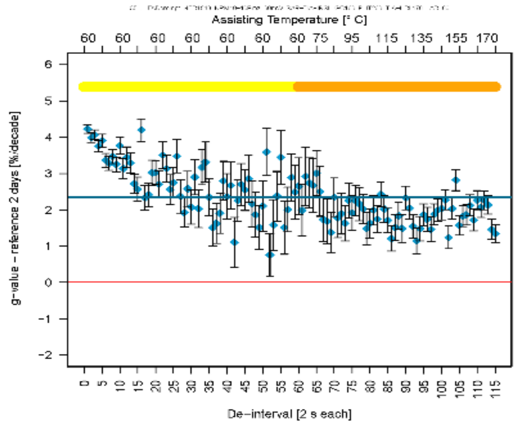
(c-1) Aliquot 3 – MS_32i



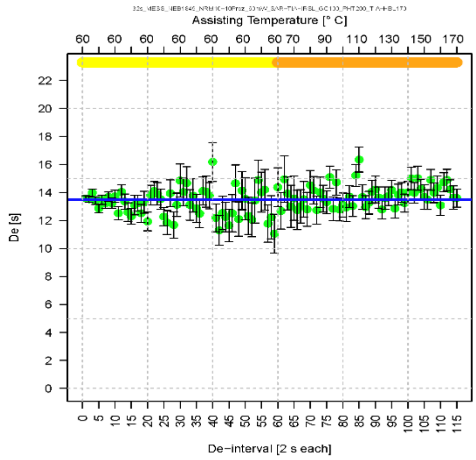
(c-2) Aliquot 3 – from MS_32i



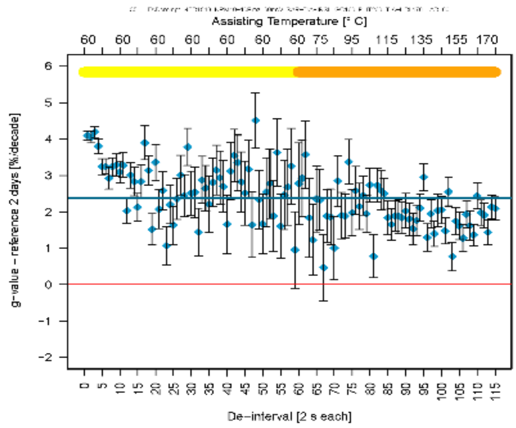
(d-1) Aliquot 4 – MS_32j



(d-2) Aliquot 4 – from MS_32j



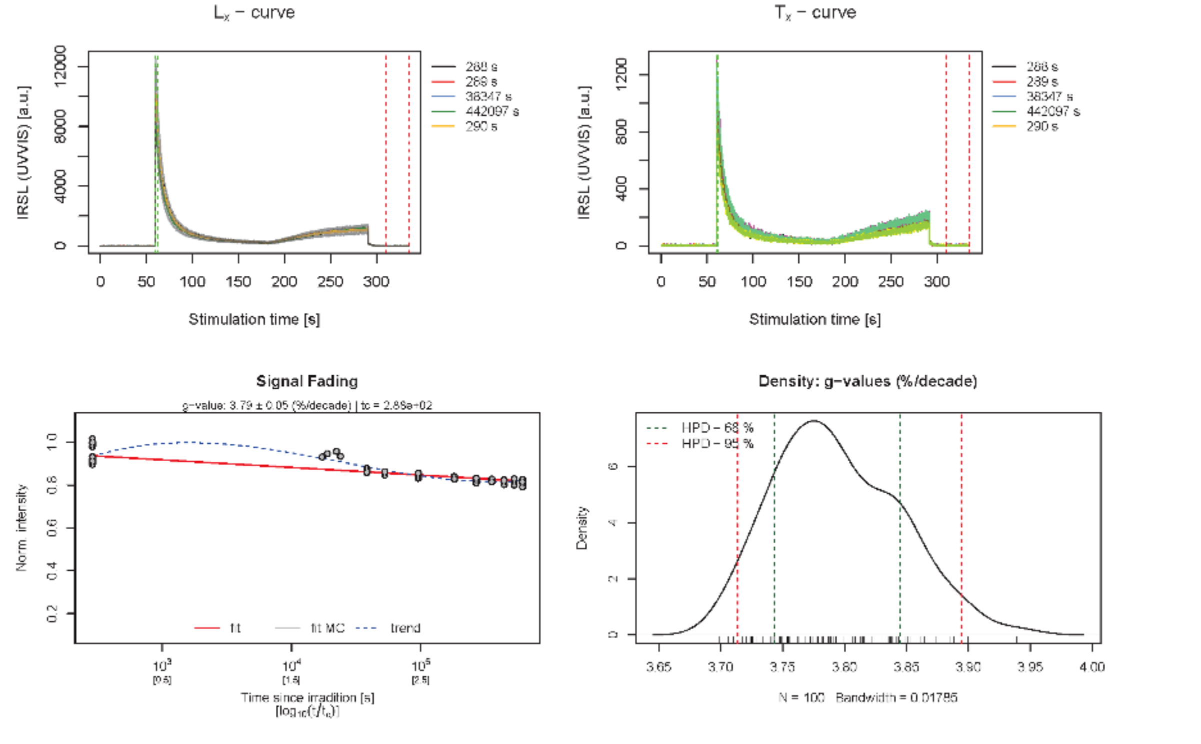
(e-1) Aliquot 5 – MS_32s



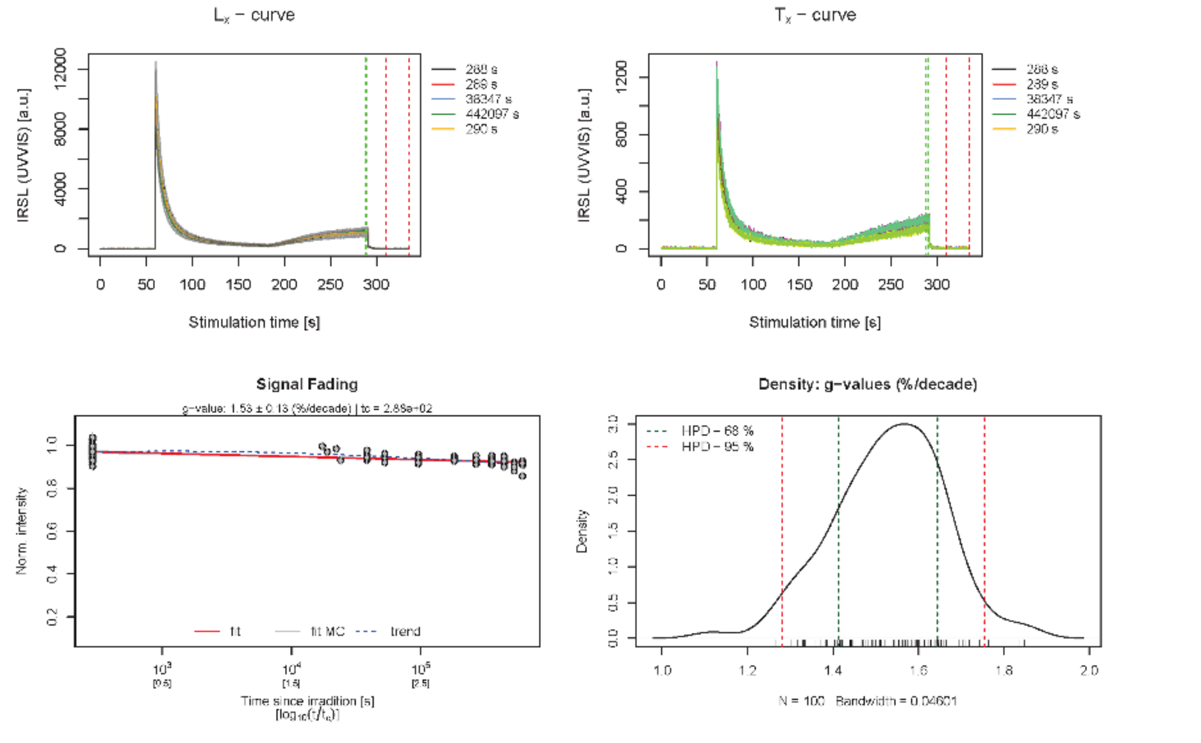
(e-2) Aliquot 5 – from MS_32s

Figure S5.5.2. MS_35_1–MS_35_12. g-value determination on five aliquots of HDS-1849 previously used for De determination (cf. **MS_32d, 32g, 32i, 32j, 32s**) applying the same PET-IRSL SAR protocol as in **MS_32**: DRC aiming at 100 s (100 %), NRM 10 % of DRC-AIM, PHT 200 °C, PET_{max} and HBL 170 °C. Customary measurement parameters: IR 60 mW/cm², BLSL hotbleach in each SAR half-cycle. LAB_{FAD} 100 s. Five prompt readouts with ca 288 s delay after half of the beta irradiation time of 100 s; ten readouts after pauses of ca 4 h to 168 h (7 days); three prompt readouts. Blue line: Mean of all g-values from De-signal integral 1–115 (2 s each). Thin red line: Potential level of zero fading. Yellow bar: Low-temperature PET-IRSL range (constant at 60 °C). Orange bar: High-temperature PET-IRSL range (60–170 °C). The graphs in the right column are identical with those from **Fig. S5.5.1a–e.**

Measurements 35_1 – MS_35_12, Aliquot 1–5



(a) g-value determination for De integral 1



(b) g-value determination for De integral 115

Figure S5.5.3. MS_35_1–MS_35_12. g-value determination of all 5 aliquots of HDS-1849 together. Graphical output of the function "analyse_FadingMeasurement" (version 0.1.23) (Kreutzer & Burow 2025) for the first and last of 115 De integrals.

S6. $pIR_{1st}IR_{2nd}$ and MET-SAR measurements, PET-IRSL measurements on empty sample carriers, dose-rate data, and IRSL spectra

S6.1. De-range test with MET protocol after Fu & Li (2013) on sample HDS-1827

Table *De range test with MET protocol after Fu & Li (2013) on sample HDS-1827 from drilling core SW04b. 1000 s (ca. 75.2 Gy) dose-response curve. NRM 20 % (200 s)*

	HDS 1827				
Aliquot/Position	1 / 1	2 / 3	3 / 5	4 / 7	5 / 9
	De	De	De	De	De
IR 50	623.78 ± 17.44	327.41 ± 12.30	197.65 ± 10.17	364.59 ± 11.65	531.76 ± 14.31
IR 80	858.61 ± 93.36	NAN NAN	280.82 ± 51.25	464.65 ± 80.01	514.75 ± 79.27
IR 110	674.33 ± 72.72	430.23 ± 269.12	307.21 ± 61.18	378.41 ± 102.19	423.45 ± 117.83
IR 140	1056.53 ± 130.03	NAN NAN	311.03 ± 63.98	707.54 ± 185.40	296.27 ± 179.45
IR 170	989.20 ± 124.54	638.94 ± 310.97	427.61 ± 81.68	420.23 ± 102.79	292.73 ± 111.04
	up and down	no plateau	no plateau	up and down	decrease

IR 50				IR 80			IR 110
De	Err_De	Recu	Rejection	DE	Recu	Rejection	DE
623.78	± 17.44	1.59 ± 0.13		858.61 ± 93.36	1.10 ± 1.06	RecRat, MPE	674.33 ±72
327.41	± 12.30	1.37 ± 0.17		NAN NAN	11.65 ± 9.25	MTDE, MPE	430.23 ±26
197.65	± 10.17	5.73 ± 0.66		280.82 ± 51.25	6.28 ± 5.46	MTDE, MPE	307.21 ±61
364.59	± 11.65	0.90 ± 0.16		464.65 ± 80.01	1.73 ± 4.95	MTDE, MPE	378.41 ±10
531.76	± 14.31	1.07 ± 0.10		514.75 ± 79.27	2.24 ± 2.41	MTDE, MPE	423.45 ±11

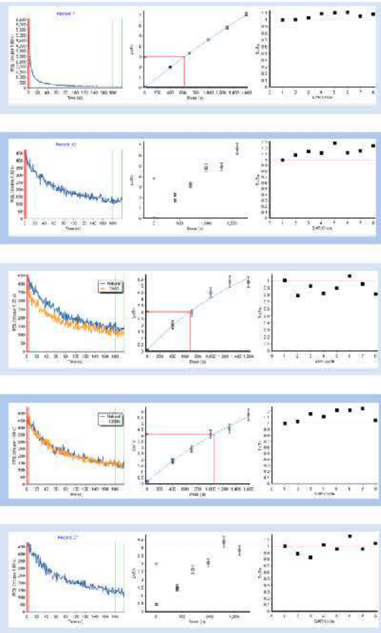
IR50
NRM 20 %

IR80
NRM 20 %

IR110
NRM 20 %

IR140
NRM 20 %

IR170
NRM 20 %



Figures left: Aliquot 1 (position 1), NRM 20 % (200 s) - no fit, if rejectio

De range test, MET after (Fu & Li, 2013)

DRC-AIM: 1000 s (ca. 73.1 Gy)

DRC: 400/700/1000/1300/1600/0/400 s

NRM: 200 s (20 %)

DRC = dose response curve
LAB = laboratory dose
NRM = nomalisation dose (test dose)

Sample HDS-1827

drilling core SW04b, 3 - 4 m b.g.l.

Luminescence reader

Risø TL/OSL Reader Model DA-20 ("Athenaeum")

dose rate [Gy/s]: 0.07306723 0.003704483
reference date: 07. Jul 25

Comments

The natural signal starts at the beginning of the IR50 shine down with ca
Many measurements would not meet the 10 % acceptance/rejection cri
When accepting all measurements ups and downs (aliquots 1, 4) as well
That aliquots do not show a plateau may be expected for a fluvial depos

Figure S6.1. De-range test with MET protocol after Fu & Li (2013) on sample HDS-1827 from drilling core SW04b. 1000 s (ca. 75.2 Gy) dose-response curve (DRC). NRM 20 % (200 s)

S6.2. Tests of pIR$_{1st}$IR$_{2nd}$ for HDS-1776 on Risø model DA20 (No. 240, "Athenaeum") – Comparing pIR$_{60}$IR$_{225}$ after preheat 280 °C with pIR$_{60}$IR$_{280}$ after preheat 320 °C and "one-run-at-a-time"

HDS-1776 had been dated with a pIR$_{60}$IR$_{225}$ protocol using a preheat of 280 °C for 60 s (Pfaffner et al., 2025; 1st measurement; **Fig. S6.2.1**). In this study, we used a preheat of 320 °C for 60 s for the PET-IRSL method and repeated the pIR$_{60}$IR$_{225}$ SAR measurement on the same luminescence reader type Risø model DA20 (No. 240, "Athenaeum") as utilized by Pfaffner et al. (2025), this time applying a preheat of 320 °C for 60 s (2nd measurement; **Fig. S6.2.2**). In addition, we tested preheat 320 °C for 60 s on the Risø reader in a "run one at a time" mode (**Fig. S6.2.1**), which compares to the way of measurement on the *lexsygresearch* readers. The results are compiled in **Fig. S6.2.1–S6.2.3** showing no significant difference between the three De-value data sets. We conclude that the higher preheat temperature does not have a significant effect on the resulting De values. However, a higher preheat temperature allows for producing a longer, still meaningful PET-IRSL De-value data curve up to 280 °C PET$_{max}$, whereas PET-IRSL readout would be meaningful only up to ca 260 °C at maximum for preheat 280 °C. As in this study we want to monitor PET-IRSL readout to the greatest possible extent, we used a maximally high but still reasonable PHT of 320 °C, at least for the oldest sample HDS-1776. For the younger samples HDS-1827 and HDS-1849, however, such high preheat temperature proved not adequate (cf. main text, **section 4.1–4.3**).

turntable position [#]	aliquot [#]	determined dose [s irradiation time] [0 - 1 s]		determined dose [s irradiation time] [0 - 4 s]		Diff long vs. short De integral [s irradiation time] [0 - 4 s] - [0 - 1 s]
1	1	2951.53	96.78	2945.67	95.18	-5.86
3	2	2702.90	118.91	2662.48	96.40	-40.42
5	3	2626.76	87.63	2646.23	83.01	19.47
7	4	2487.33	84.63	2461.29	78.25	-26.04
9	5	2639.41	103.44	2630.97	91.86	-8.44
11	6	2627.29	78.73	2631.26	78.40	3.97
13	7	3402.51	147.31	3535.99	126.85	133.48
15	8	2278.23	79.37	2281.39	71.79	3.16
17	9	2725.76	87.73	2834.26	84.40	108.50
19	10	2600.89	75.60	2673.12	75.85	72.23
21	11	2344.51	110.50	2409.90	116.23	65.39
23	12	2707.56	87.42	2744.31	83.76	36.75
25	13	2509.07	76.71	2558.31	75.33	49.24
27	14	2986.67	84.29	2954.98	80.61	-31.69
29	15	2805.22	91.81	2852.35	92.90	47.13
31	16	n.a.		n.a.		
33	17	2696.17	77.56	2697.15	76.96	0.98
35	18	3155.04	100.51	3144.17	99.98	-10.87
37	19	3227.81	115.12	3197.45	102.99	-30.36
39	20	3011.29	85.41	2981.53	84.64	-29.76
41	21	2008.77	80.95	2057.09	68.46	48.32
43	22	2509.08	88.51	2520.23	75.74	11.15
45	23	3532.65	133.90	3547.10	126.94	14.45
47	24	2527.97	74.58	2523.19	72.26	-4.78
	central dose:	2720.50	71.20	2738.90	71.20	
						426.00 but not

Figure S6.2.1. $pIR_{60}IR_{225}$ after preheat 280 °C on HDS-1776. 1st measurement (from Pfaffner et al., 2025).

	turntable position [#]	aliquot [#]	determined dose [s irradiation time] [0 - 1 s]		determined dose [s irradiation time] [0 - 4 s]		Diff long vs. short De integral [s irradiation time] [0 - 4 s] - [0 - 1 s]
	1	1	4175.06	174.36	4122.12	168.82	-52.94
	3	2	3530.47	161.04	3349.41	142.22	-181.06
	5	3	2610.15	101.63	2681.52	99.13	71.37
RecRat	7	4	3161,7	191.87	3142,13	170.98	
RecRat	9	5	3211,34	182.35	3226,35	176.39	
RecRat	11	6	3089,5	194.90	3138,73	174.96	
	13	7	2858.96	94.08	2878.17	91.84	19.21
RecRat	15	8	3145,11	148.64	3131.62	131.82	
RecRat	17	9	2910,33	133.73	2993.44	125.84	
	19	10	2412.60	130.59	2348.45	108.34	-64.15
	21	11	2609.64	155.41	2639.43	148.55	29.79
	23	12	3218.17	177.58	3251.23	170.25	33.06
	25	13	2797.50	106.91	2794.03	96.72	-3.47
	27	14	3435.27	154.99	3395.66	143.12	-39.61
	29	15	2792.04	124.90	2824.76	115.39	32.72
	31	16	3139.79	155.28	3155.81	131.51	
RecRat	33	17	2787,66	129.60	2754.99	110.53	
	35	18	3044.08	103.91	3030.06	100.19	-14.02
	37	19	2330.92	83.93	2383.47	82.73	52.55
	39	20	2889.08	114.28	2861.39	98.29	-27.69
	41	21	2213.42	135.72	2235.69	99.84	22.27
	43	22	2815.57	133.43	2784.23	120.61	-31.34
	45	23	3002.95	120.44	2972.22	109.33	-30.73
	47	24	2582.72	104.27	2634.50	95.92	51.78
		central dose:	2881.90	102.20	2888.50	84.20	
							-132.26 but n

Figure S6.2.2. pIR$_{60}$IR$_{280}$ after preheat 320 °C on HDS-1776. 2nd measurement.

turntable position [#]	aliquot [#]	determined dose [s irradiation time] [0 - 1 s]		determined dose [s irradiation time] [0 - 4 s]		Diff long vs. short De integral [s irradiation time] [0 - 4 s] - [0 - 1 s]	
							N = 24 /
1	1	4170.75	179.38	4179.22	172.94	8.47	
3	2	3545.08	143.87	3414.10	125.52	-130.98	
5	3	2673.53	98.32	2771.79	90.12	98.26	
7	4	2810.03	101.96	2839.18	94.52	29.15	
9	5	2791.15	104.83	2805.28	104.00	14.13	
11	6	2701.09	103.11	2770.77	96.24	69.68	
13	7	2859.31	98.20	2862.72	95.26	3.41	
15	8	3202.37	119.22	3201.19	112.68	-1.18	
17	9	2991.96	113.65	3065.30	108.77	73.34	
19	10	2232.52	97.69	2202.44	85.13	-30.08	
21	11	2460.09	89.42	2529.33	87.20	69.24	
23	12	3044.06	116.60	3075.32	114.39	31.26	
25	13	2750.81	112.22	2765.89	100.99	15.08	
27	14	3358.37	133.97	3295.77	120.63	-62.60	
29	15	2796.88	97.01	2808.34	94.95	11.46	
31	16	3202.65	112.91	3190.02	107.39	-12.63	
33	17	2801.24	95.83	2755.20	90.54	-46.04	
35	18	3023.91	101.86	3034.33	98.33	10.42	
37	19	2248.28	87.12	2292.61	85.28	44.33	
39	20	2802.21	109.80	2796.40	96.56	-5.81	Start
41	21	1960.26	143.21	2006.06	99.05	45.80	Dose ra
43	22	2582.27	90.44	2605.68	86.99	23.41	dose ra
45	23	3034.08	102.98	2995.93	95.83	-38.15	De [s]
47	24	2566.15	89.13	2628.12	89.09	61.97	age [ka
	central dose:	2831.10	35.50	2841.30	83.20		
						281.94	but not systematicall

Figure S6.2.3. $pIR_{60}IR_{280}$ after preheat 320 °C on HDS-1776. 3rd measurement ("one-run-at-a-time" mode.

S6.3. $pIR_{50}IR_{170}$ after preheat 200 °C on HDS-1849. Dose-recovery tests, zero-dose test and De-measurements

Table **Dose recovery tests, zero-dose test and De-measurements with $pIR_{50}IR_{170}$ protocol on sample HDS-1849 from**

Measurement-No.	Measurement-Type	Expected De [s]	IR50 De [s]	IR50 Err_De [s]	IR170 De [s]	IR170 Err_De [s]
MS_47a	DRT-NRM10	15^	16.15	0.36	15.17	0.41
MS_47b	DRT-NRM20	15^	16.53	0.30	15.05	0.38
MS_47c	DRT-NRM30	15^	16.32	0.28	15.76	0.28
MS_47d	DRT-NRM40	15^	16.75	0.25	16.14	0.30
MS_47e	DRT-NRM50	15^	16.61	0.21	16.22	0.21
MS_49a	DRT-Zero-Dose - NRM10	0#	0.50	0.05	2.93	0.11
MS_48a	MESS - NRM10	ca 13 - 15*	13.22	0.32	14.16	0.40
MS_48b	MESS - NRM10	ca 13 - 15*	11.57	0.23	12.93	0.27
MS_48c	MESS - NRM10	ca 13 - 15*	11.81	0.30	12.48	0.39

^ Administered dose (LAB)
# Aliquot bleached, no dose applied
* Expectation based on PET-IRSL SAR measurements

Comments

- The dose recovery tests recover the administered dose of 15 s reasonably well.
- The zero-dose test shows a larger residual dose for IR170 than PET-IRSL SAR.
- The De measurements corroborate the tentative PET-IRSL SAR De determinations.

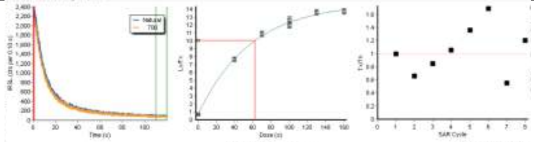
$pIR_{50}IR_{170}$-protocol largely following Buckland et al. (2019) except for:

- PHT-TL at 200 °C for 60 s (instead of 10 s)
- IRSL-HBL at 280 °C for 100 s (instead of 290 °C for 50 s)
- REGs for DRC-construction 40/70/100/130/160/0/100 s

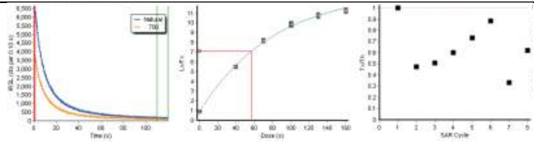
Figure S6.3. $pIR_{50}IR_{170}$ after preheat 200 °C on HDS-1849. Dose-recovery tests, zero-dose test and De-measurements.

S6.4. p$IR_{60}IR_{280}$ – NEB-1849 – LR04 (COLOUR)

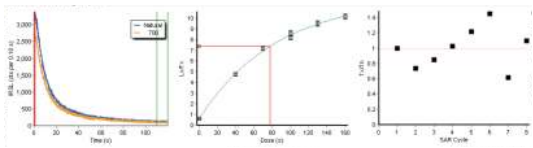
S6.4.1. NRM-DRT p$IR_{60}IR_{280}$ – LAB 100



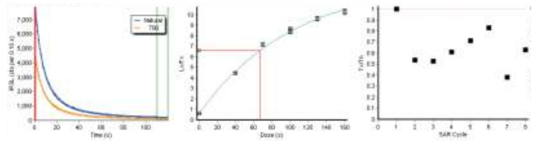
(a) IR_{60} – NRM 2.5 % – De = 62.21 ± 3.93 s, Recu = 6.8 ± 0.4 %



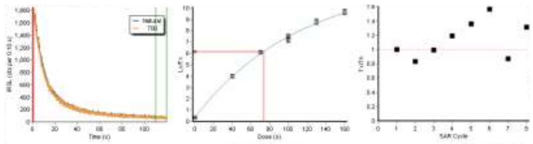
(b) IR_{280} – NRM 2.5 % – De = 57.13 ± 2.45 s, Recu = 12.3 ± 0.5 %



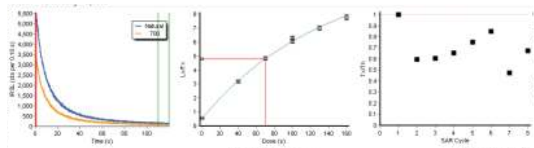
(c) IR_{60} – NRM 5 % – De = 77.57 ± 3.88 s, Recu = 8.2 ± 0.4 %



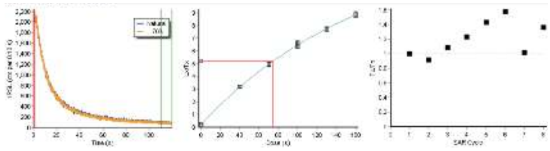
(d) IR_{280} – NRM 5 % – De = 66.74 ± 2.70 s, Recu = 9.1 ± 0.4 %



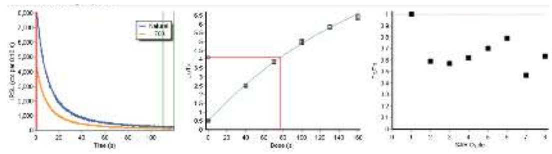
(e) IR_{60} – NRM 7.5 % – De = 73.42 ± 3.38 s, Recu = 5.7 ± 0.3 %



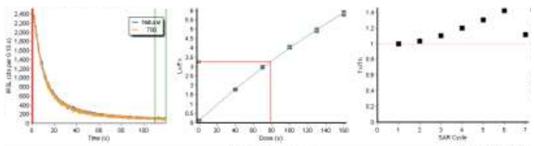
(f) IR_{280} – NRM 7.5 % – De = 70.06 ± 2.58 s, Recu = 11.2 ± 0.4 %



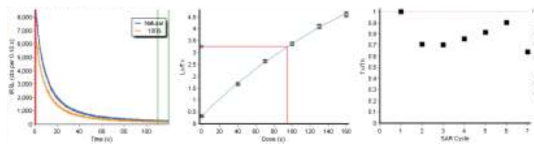
(g) IR_{60} – NRM 10 % – De = 74.12 ± 2.73 s, Recu = 4.3 ± 0.2 %



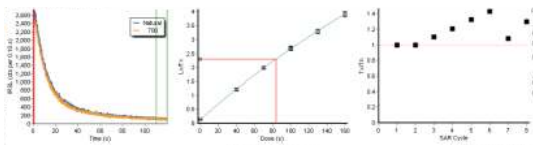
(h) IR_{280} – NRM 10 % – De = 77.38 ± 2.67 s, Recu = 12.6 ± 0.5 %



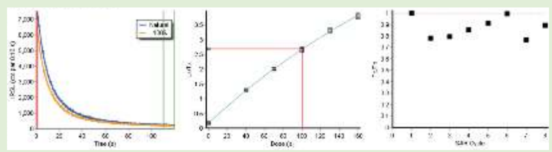
(i) IR_{60} – NRM 20 % – De = 79.36 ± 2.34 s, Recu = 3.5 ± 0.2 %



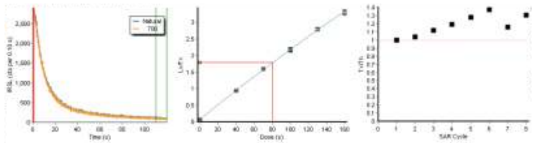
(j) IR_{280} – NRM 20 % – De = 94.88 ± 2.97 s, Recu = 10.1 ± 0.4 %



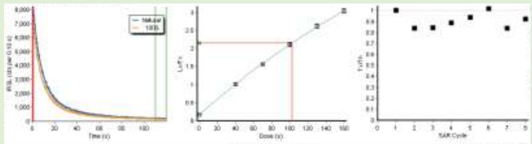
(k) IR_{60} – NRM 30 % – De = 83.68 ± 2.41 s, Recu = 5.7 ± 0.2 %



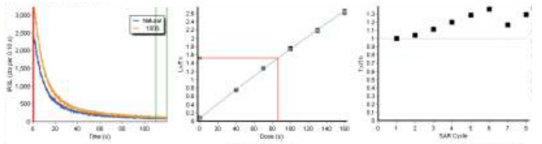
(l) IR_{280} – NRM 30 % – De = 100.78 ± 2.89 s, Recu = 6.6 ± 0.2 %



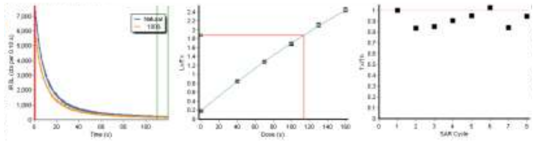
(m) IR_{60} – NRM 40 % – De = 80.44 ± 2.09 s, Recu = 4.0 ± 0.2 %



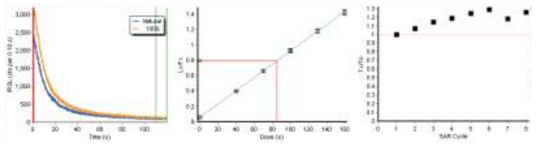
(n) IR_{280} – NRM 40 % – De = 102.19 ± 2.84 s, Recu = 7.6 ± 0.3 %



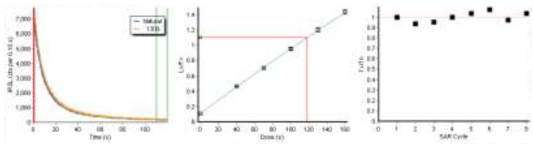
(o) IR_{60} – NRM 50 % – De = 86.57 ± 2.29 s, Recu = 5.0 ± 0.2 %



(p) IR_{280} – NRM 50 % – De = 113.93 ± 3.29 s, Recu = 9.8 ± 0.3 %



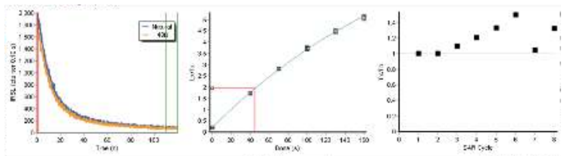
(q) IR_{60} – NRM 100 % – De = 85.09 ± 2.14 s, Recu = 7.3 ± 0.3 %



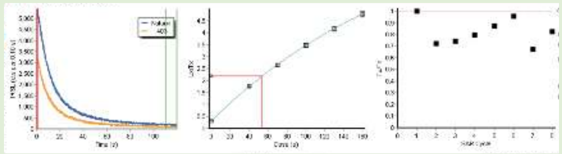
(r) IR_{280} – NRM 100 % – De = 118.17 ± 3.04 s, Recu = 9.6 ± 0.3 %

Figure S6.4.1. NRM-DRT test on sample HDS-1849 (Nebraska) with LAB 100 s

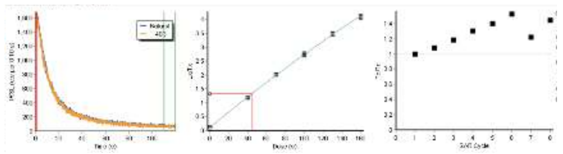
S6.4.2. NRM-DRT pIR$_{60}$IR$_{280}$ – LAB 50



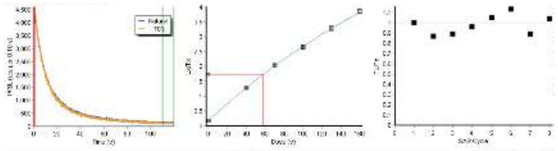
(a) IR$_{60}$ – NRM 20 % – De = 44.94 ± 1.44 s, Recu = 10.4 ± 0.4 %



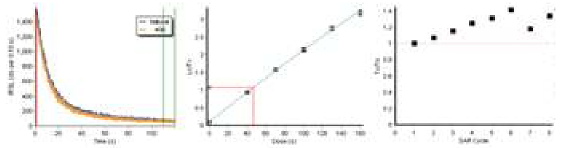
(b) IR$_{280}$ – NRM 20 % – De = 53.61 ± 1.62 s, Recu = 14.1 ± 0.5 %



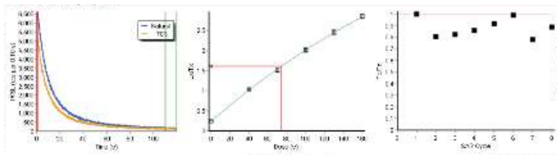
(c) IR$_{60}$ – NRM 30 % – De = 44.82 ± 1.30 s, Recu = 8.5 ± 0.4 %



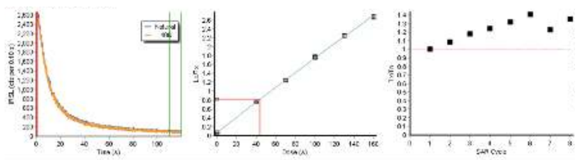
(d) IR$_{280}$ – NRM 30 % – De = 58.31 ± 1.67 s, Recu = 10.3 ± 0.4 %



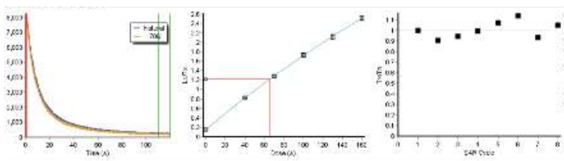
(e) IR$_{60}$ – NRM 40 % – De = 46.89 ± 1.40 s, Recu = 8.8 ± 0.4 %



(f) IR$_{280}$ – NRM 40 % – De = 74.05 ± 2.18 s, Recu = 14.6 ± 0.5 %



(g) IR$_{60}$ – NRM 50 % – De = 43.97 ± 1.20 s, Recu = 8.5 ± 0.3 %

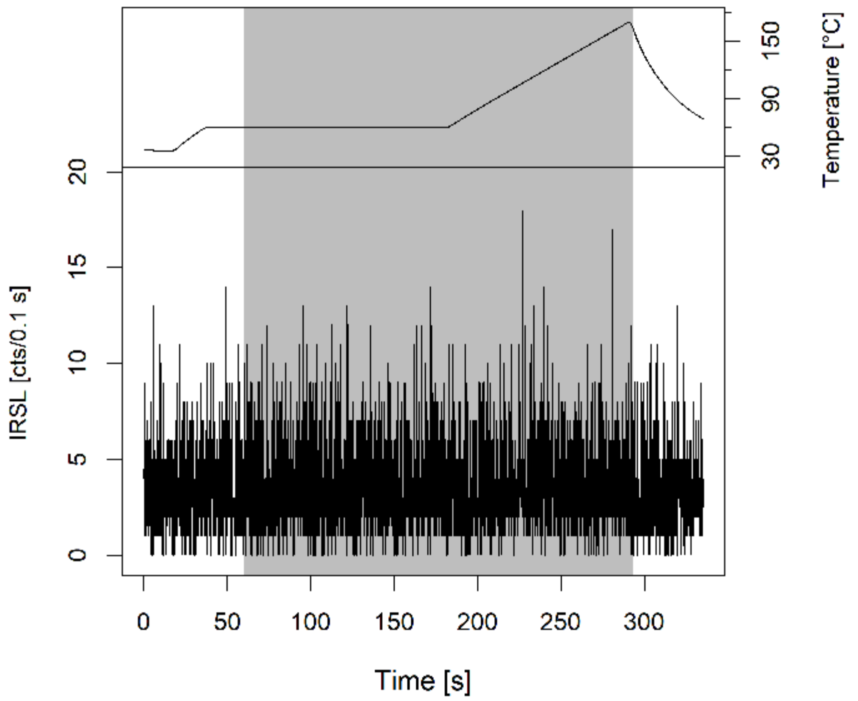


(h) IR$_{280}$ – NRM 50 % – De = 65.79 ± 1.77 s, Recu = 12.1 ± 0.4 %

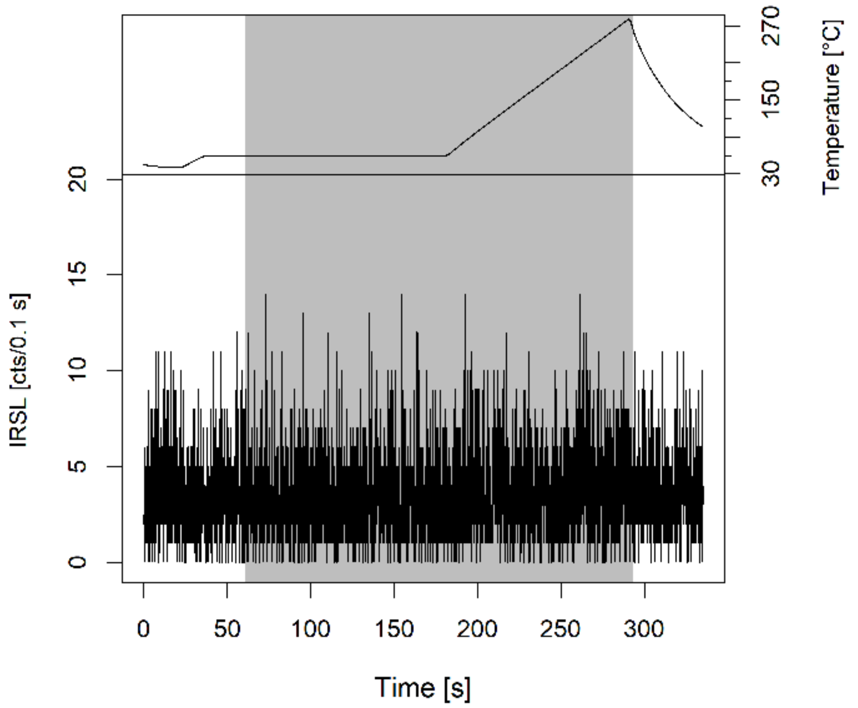
Figure 6.4.2. NRM-DRT test on sample HDS-1849 (Nebraska) with LAB 50 s

S6.5. Empty aliquots subjected to PET-IRSL measurement

No increased background signal was observed on empty aliquots during the actual PET-IRSL readout 60–170 °C or 60–280 °C (cf. grey shaded areas in **Fig. S6.5a and S6.5b**, respectively.



(a)



(b)

Figure S6.5. Empty aliquots subjected to PET-IRSL measurement. **(a)** PET-IRSL readout up to PET_{max} 170 °C. **(b)** PET-IRSL readout up to PET_{max} 280 °C.

S6.6. Assessment of the effective dose rates for HDS-1827 and HDS-1849

Lab.-No.	De_bet	Err_De_bet	OD.Proz	Err_OD.Proz	DL_kosm	Err_DL_kosm	U	Err_U	Th	Err_Th	K	Err_K	Feuchte_mess	Feuchte_mod	Err_Feu
HDS-1849 - 12.5 %	1.326	0.049	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 12.0 % - Norflaot Potash	1.326	0.049	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 10.4 % - G40	1.326	0.049	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 4.1 % - F20	1.326	0.049	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 12.5 %	1.432	0.053	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 12.0 % - Norflaot Potash	1.432	0.053	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 10.4 % - G40	1.432	0.053	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 4.1 % - F20	1.432	0.053	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 12.5 %	1.538	0.056	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 12.0 % - Norflaot Potash	1.538	0.056	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 10.4 % - G40	1.538	0.056	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1849 - 4.1 % - F20	1.538	0.056	NA	NA	0.1	0.05	0.658706449	0.107798476	3.53463101	0.361396907	1.918499756	0.061394789	1.01	1.01	
HDS-1827 - 12.5 %	NA	NA	NA	NA	0.1	0.05	0.9771	0.0923	4.780975415	0.314530279	1.3712	0.0585	1.5	1.5	
HDS-1827 - 12.0 % - Norflaot Potash	NA	NA	NA	NA	0.1	0.05	0.9771	0.0923	4.780975415	0.314530279	1.3712	0.0585	1.5	1.5	
HDS-1827 - 10.4 % - G40	NA	NA	NA	NA	0.1	0.05	0.9771	0.0923	4.780975415	0.314530279	1.3712	0.0585	1.5	1.5	
HDS-1827 - 4.1 % - F20	NA	NA	NA	NA	0.1	0.05	0.9771	0.0923	4.780975415	0.314530279	1.3712	0.0585	1.5	1.5	
	Line 2-5:	Fig. 18c, 12.5 s													
	Line 7-10:	Fig. 18b, 13.5 s													
	Line 12-15:	Fig. 18b, 14.5 s													

Figure S6.6. Assessment of the effective dose rates for HDS-1827 and HDS-1849 based on the range of potassium contents of the three feldspar specimens Norfloat potash, G40 and F20.

S6.7. Results of the IRSL spectral measurements on the sediment samples and feldspar specimens

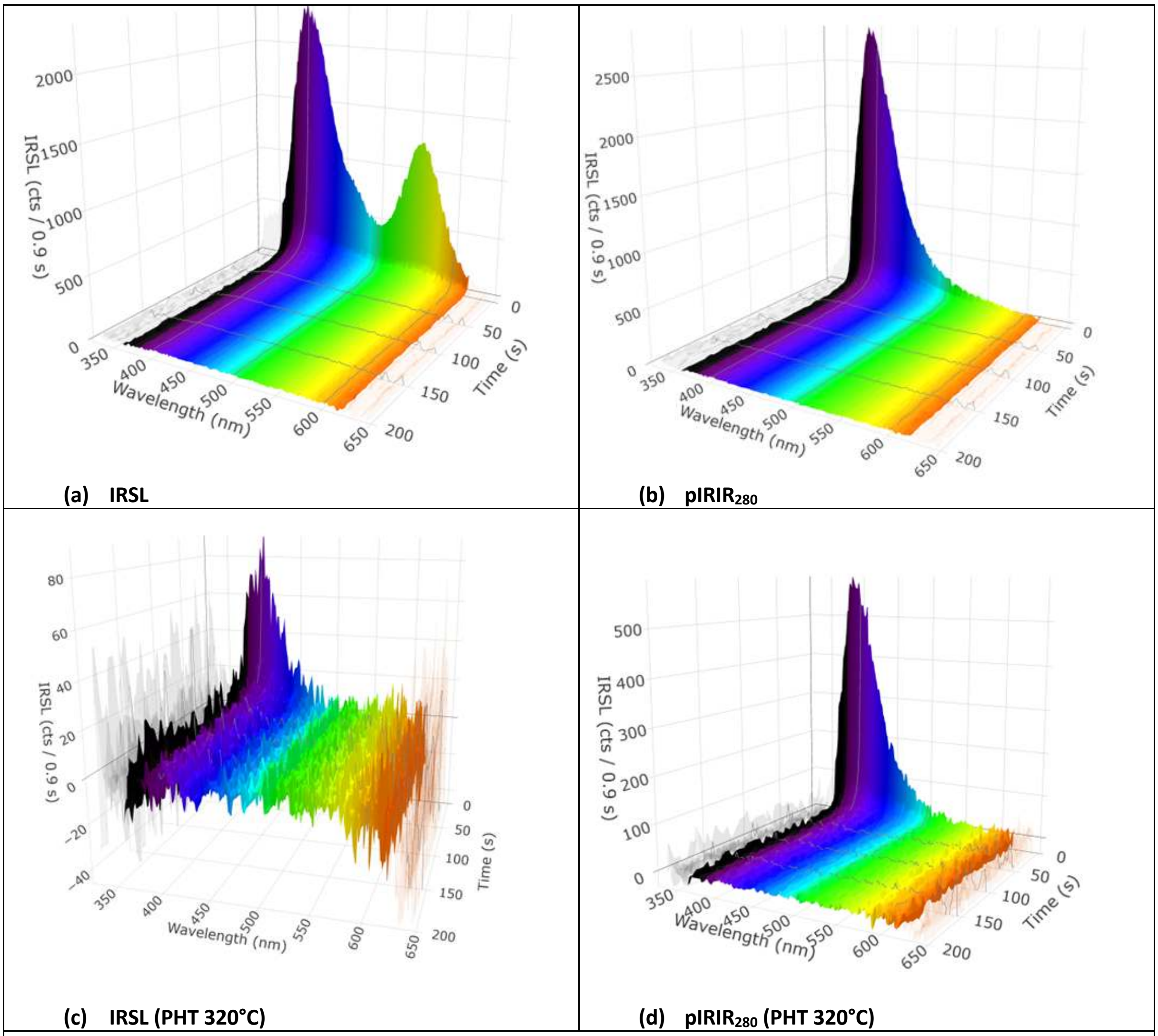
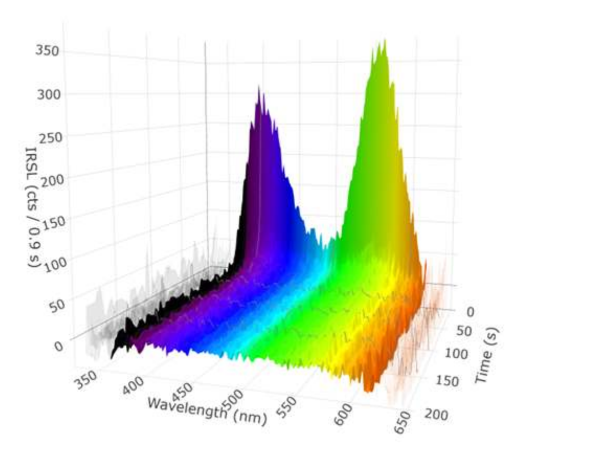
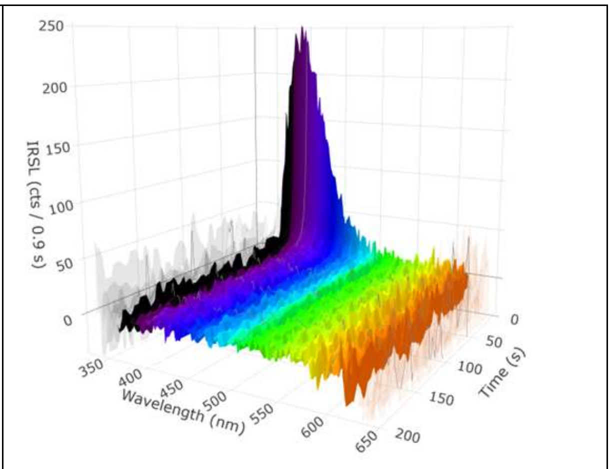


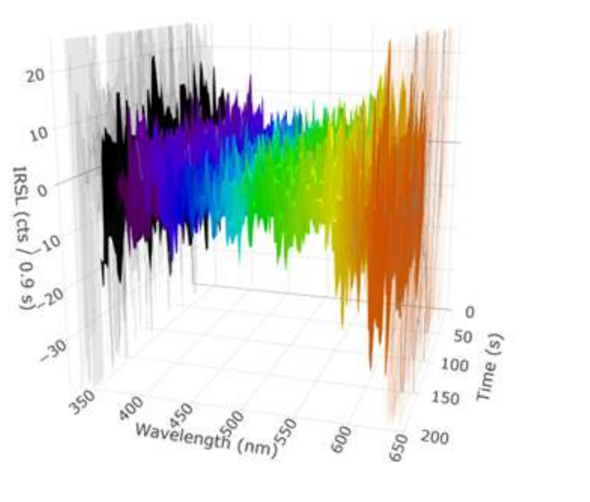
Figure S6.7.1. Spectra obtained from pIRIR measurements on sample HDS-1776 (loess-palaeosol section Baix, France) (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step.



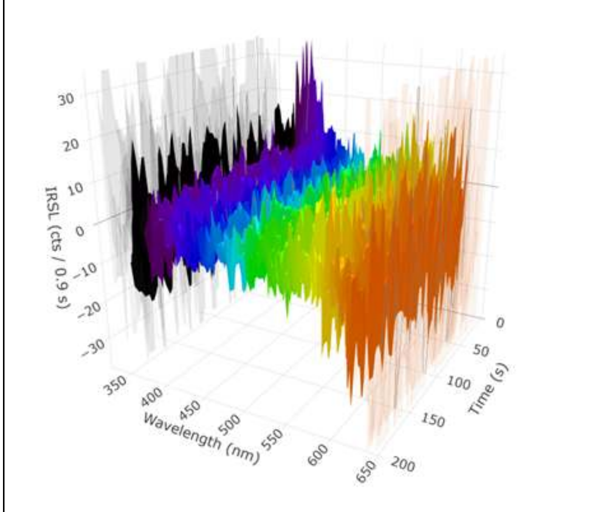
(a) IRSL



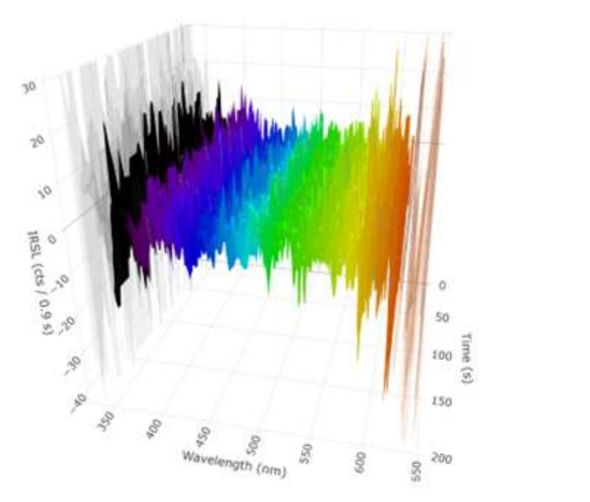
(b) pIRIR$_{280}$



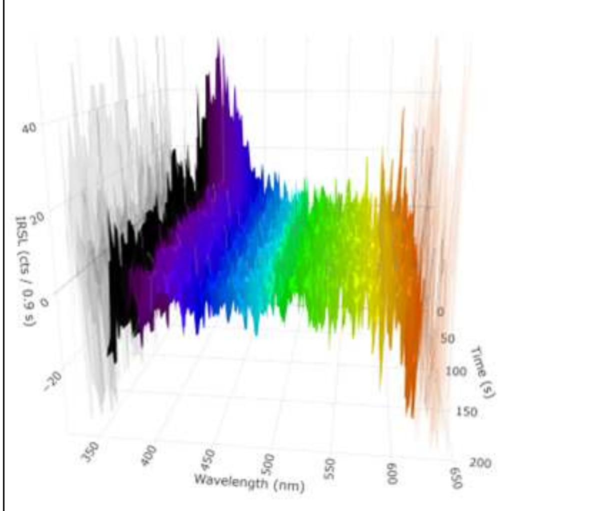
(c) IRSL (PHT 320°C)



(d) pIRIR$_{280}$ (PHT 320°C)



(e) IRSL (PHT 320°C; 800 Gy)



(f) pIRIR$_{280}$ (PHT 320°C; 800 Gy)

Figure S6.7.2. Spectra obtained from pIRIR measurements on sample HDS-1827 (Bergstraßenneckar, Germany) (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step. (e,f) Given the low signal intensity obtained for this sample, the measurement with a preheat was repeated with a higher given dose of ca. 800 Gy prior to the preheat step.

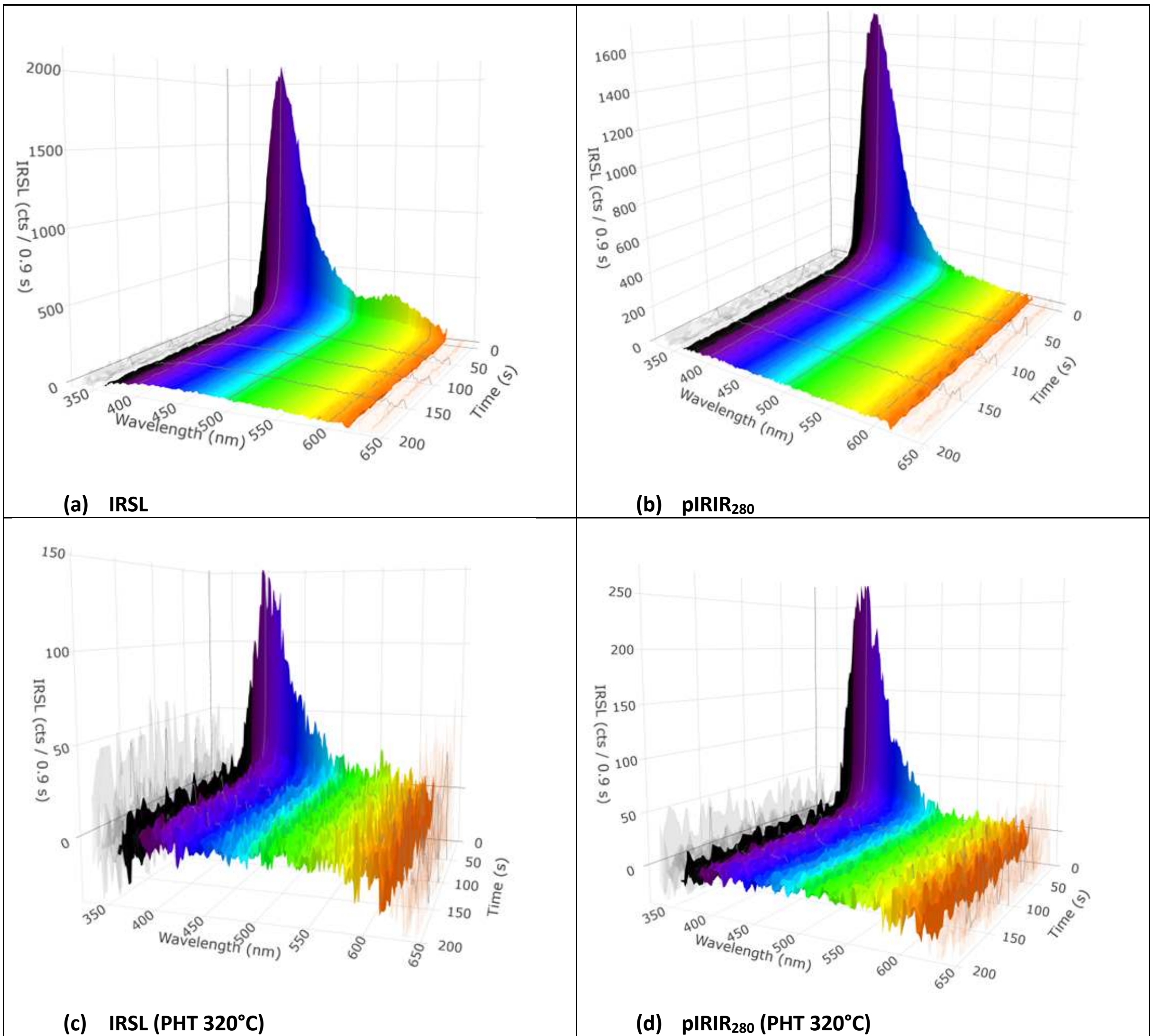
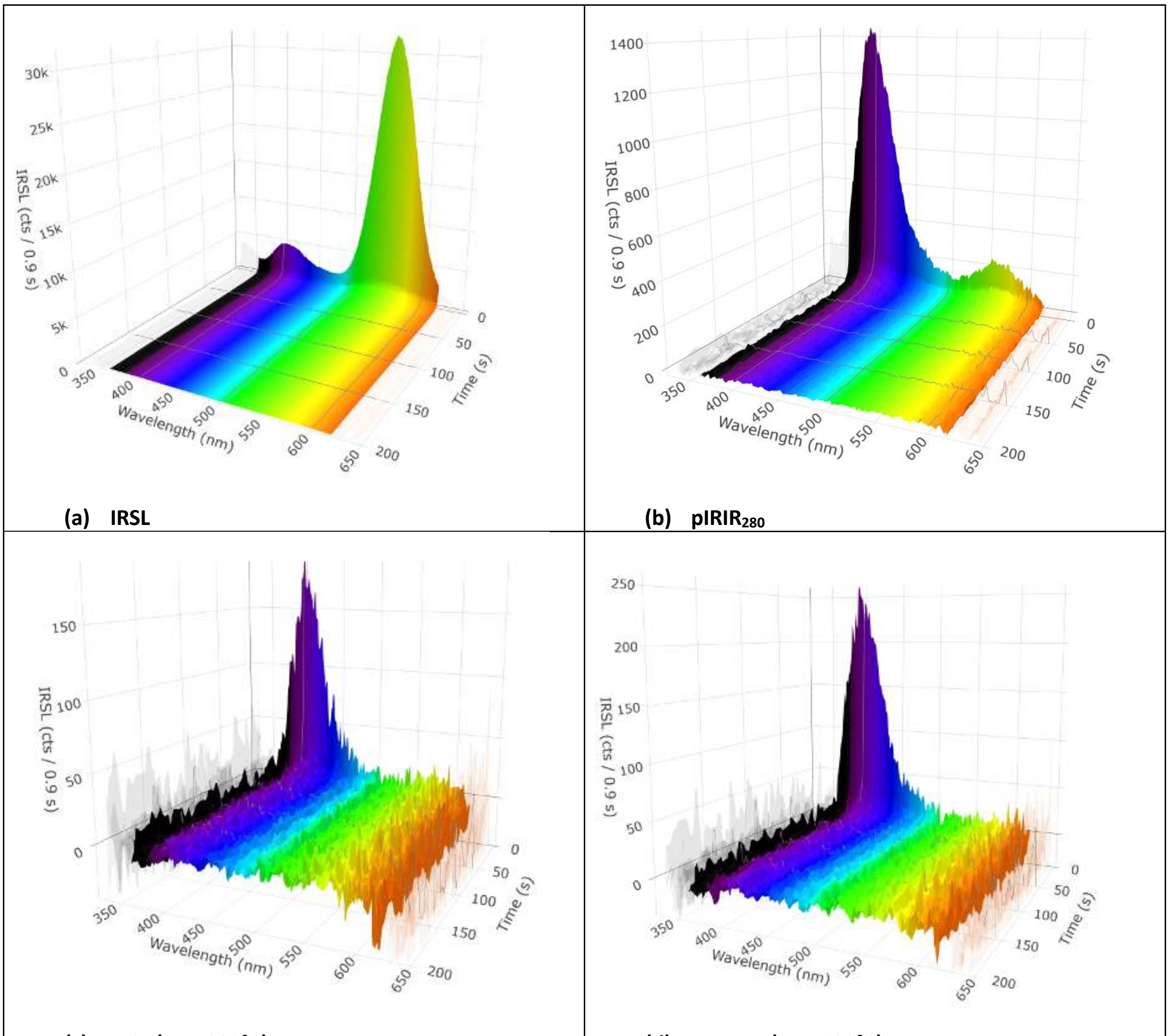


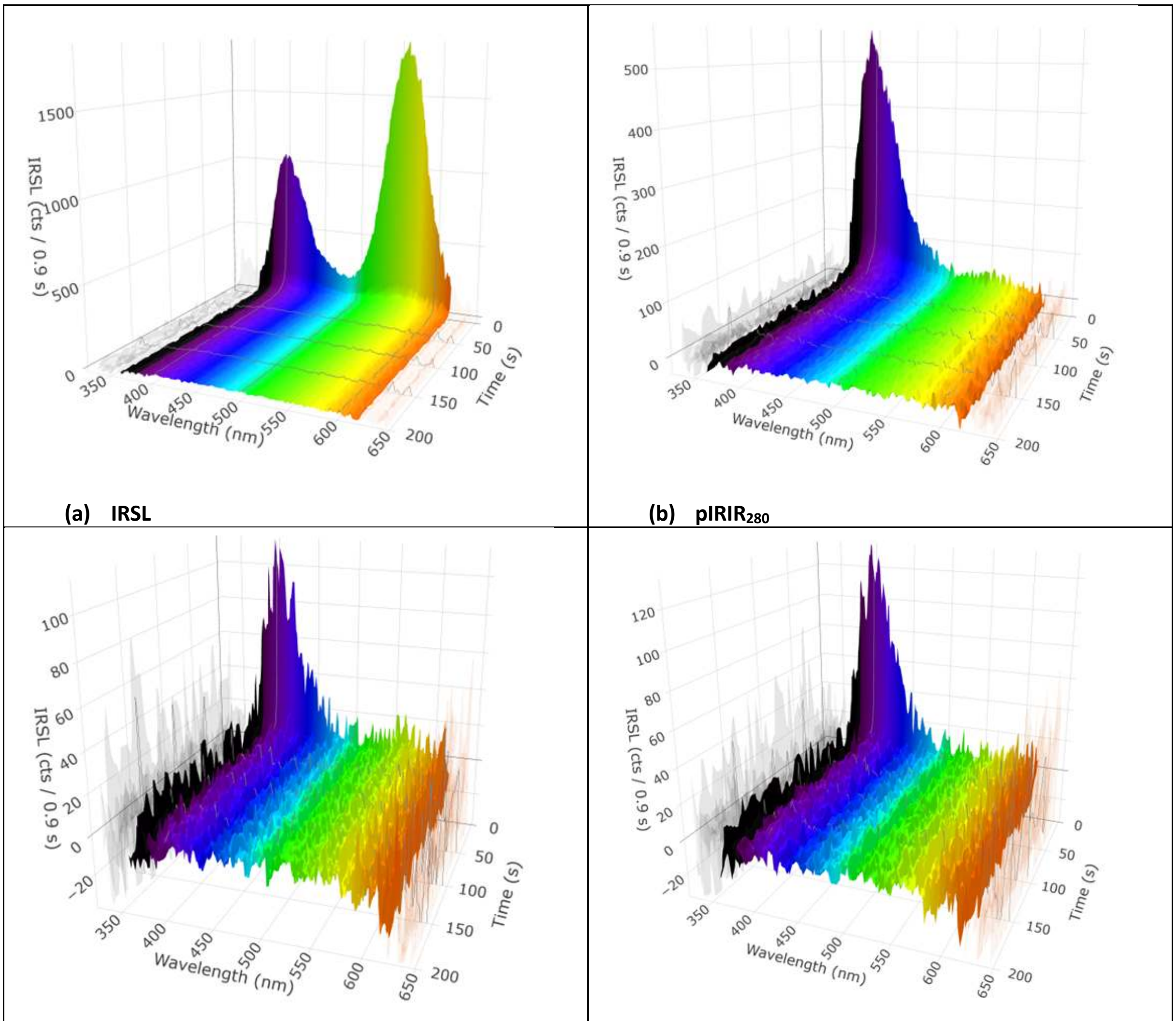
Figure S6.7.3. Spectra obtained from pIRIR measurements on sample HDS-1849 (Nebraska Sand Hills, USA) (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step.



(a) IRSL **(b) pIRIR$_{280}$**

(c) IRSL (PHT 320°C) **(d) pIRIR$_{280}$ (PHT 320°C)**

Figure S6.7.4. Spectra obtained from pIRIR measurements on sample Norfloat-Potash-Feldspar (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step.



(a) IRSL **(b) pIRIR$_{280}$**

(c) IRSL (PHT 320°C) **(d) pIRIR$_{280}$ (PHT 320°C)**

Figure S6.7.5. Spectra obtained from pIRIR measurements on sample G-40-Feldspar (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step.

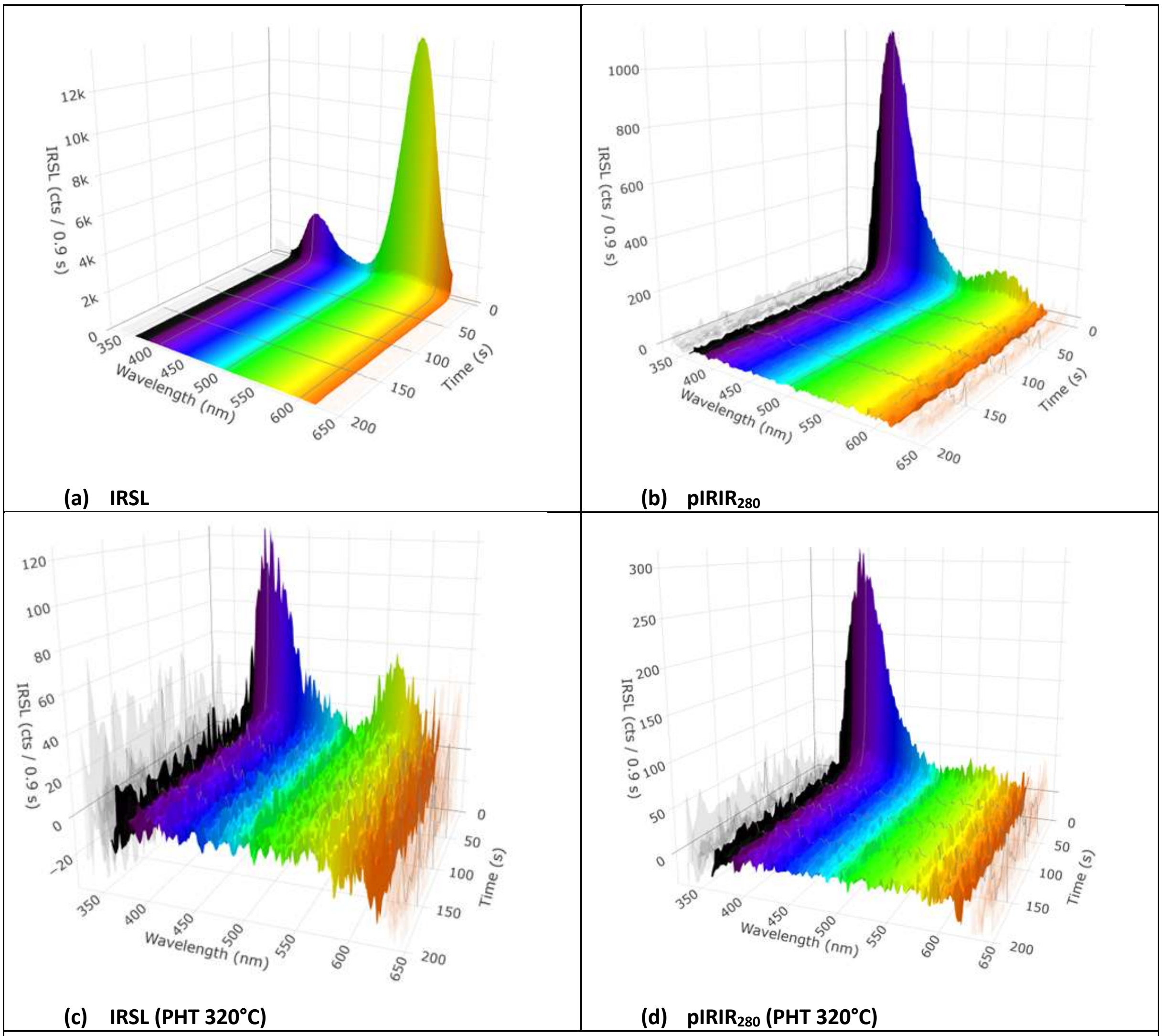


Figure S6.7.6. Spectra obtained from pIRIR measurements on sample F-20-Feldspar (a,b) without any preheat treatment or (c,d) with a preheat to 320°C. Spectra are shown for the first IRSL step and the pIRIR step of the same sequence. The same aliquot was used for all measurements, dosed with ca. 400 Gy prior to (a) the IRSL step or (c) the preheat step.