pIRIR analysis B19-LU 6

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Analysed file: 3-Permafrost-1mm-63-100-Fsp-pIR290-sample_B19-LU-6-24al.binx

Date of analysis: 01 Jan 2023

This report describes the data analysis work flow for the pIRIR measurement of the sample *B19-LU 6*, measured by Steffi Hesse and Dr. Tobias Lauer. The data analysis was performed by Dirk Mittelstraß on behalf of Dr. Margret Fuchs from HZDR Innovation GmbH and paid for by the HZDR Innovation GmbH.

The data was processed using the open-source statistical progamming language R and multiple open-source function libraries (called 'packages'), especially the R package Luminescence by Kreutzer et al. (2012, 2022). This PDF was formatted and created with Quarto.

1 Data structure

1.1 Sample set

First we prove if all aliquots are of the same sample and the same run by looking at the Risö Reader record parameters.

Table 1: Structure of the BIN file content

index	SAMPLE	DATE	RUN	SET	POSITION	records
1	Sample1	141022	2	1	1	48
2	Sample3	141022	2	1	3	48
3	Sample5	141022	2	1	5	48
4	Sample7	141022	2	1	7	48
5	Sample9	141022	2	1	9	48
6	Sample11	141022	2	1	11	48
7	Sample13	141022	2	1	13	48
8	Sample15	141022	2	1	15	48
9	Sample17	141022	2	1	17	48
10	Sample19	141022	2	1	19	48
11	Sample21	141022	2	1	21	48
12	Sample23	141022	2	1	23	48
13	Sample25	141022	2	1	25	48
14	Sample27	141022	2	1	27	48
15	Sample29	141022	2	1	29	48
16	Sample31	141022	2	1	31	48
17	Sample33	141022	2	1	33	48
18	Sample35	141022	2	1	35	48
19	Sample37	141022	2	1	37	48
20	Sample39	151022	2	1	39	48
21	Sample41	151022	2	1	41	48
22	Sample43	151022	2	1	43	48
23	Sample45	151022	2	1	45	48
24	Sample47	151022	2	1	47	48

24 aliquots were processed with the same sequence and were of the same measurement run.

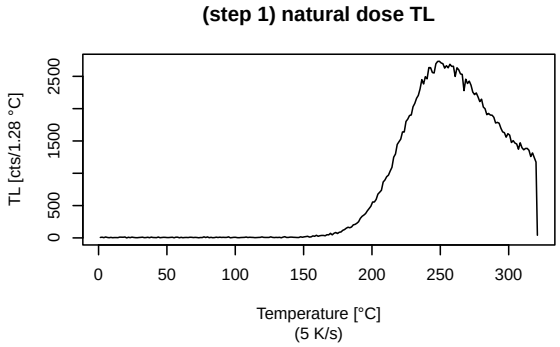
1.2 Measurement sequence

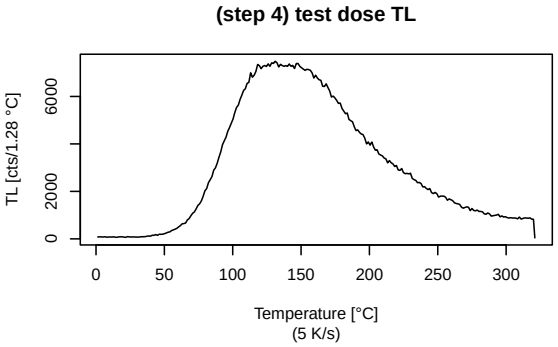
Table 2: Measurement sequence structure for aliquot 1

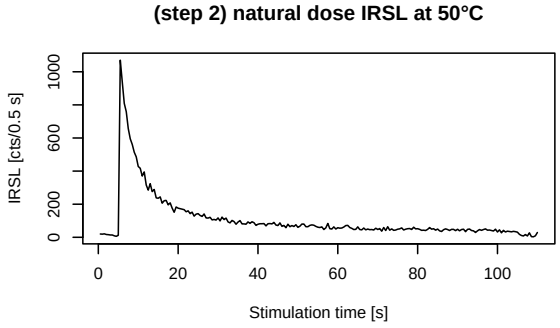
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
1	TL	321	0	250	1.285
2	IRSL	50	0	220	0.500
3	IRSL	290	0	420	0.500
4	TL	0	1000	250	1.285
5	IRSL	50	1000	220	0.500
6	IRSL	290	1000	420	0.500
7	IRSL	325	1000	420	0.238
8	TL	321	0	250	1.285
9	IRSL	50	0	220	0.500
10	IRSL	290	0	420	0.500
11	TL	0	1000	250	1.285
12	IRSL	50	1000	220	0.500
13	IRSL	290	1000	420	0.500
14	IRSL	325	1000	420	0.238
15	TL	0	1000	250	1.285
16	IRSL	50	1000	220	0.500
17	IRSL	290	1000	420	0.500
18	TL	0	1000	250	1.285
19	IRSL	50	1000	220	0.500
20	IRSL	290	1000	420	0.500
21	IRSL	325	1000	420	0.238
22	TL	0	2000	250	1.285
23	IRSL	50	2000	220	0.500
24	IRSL	290	2000	420	0.500
25	TL	0	1000	250	1.285
26	IRSL	50	1000	220	0.500
27	IRSL	290	1000	420	0.500
28	IRSL	325	1000	420	0.238
29	TL	0	4000	250	1.285
30	IRSL	50	4000	220	0.500
31	IRSL	290	4000	420	0.500
32	TL	0	1000	250	1.285
33	IRSL	50	1000	220	0.500
34	IRSL	290	1000	420	0.500
35	IRSL	325	1000	420	0.238
36	TL	0	6000	250	1.285
37	IRSL	50	6000	220	0.500

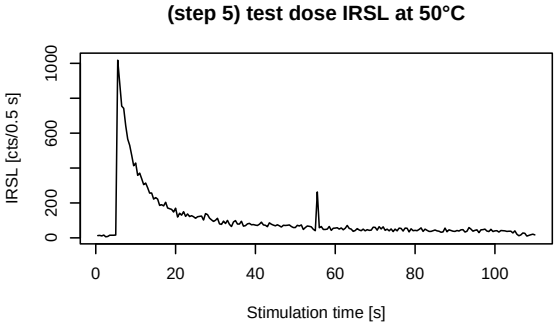
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
38	IRSL	290	6000	420	0.500
39	TL	0	1000	250	1.285
40	IRSL	50	1000	220	0.500
41	IRSL	290	1000	420	0.500
42	IRSL	325	1000	420	0.238
43	TL	0	1000	250	1.285
44	IRSL	50	1000	220	0.500
45	IRSL	290	1000	420	0.500
46	TL	0	1000	250	1.285
47	IRSL	50	1000	220	0.500
48	IRSL	290	1000	420	0.500

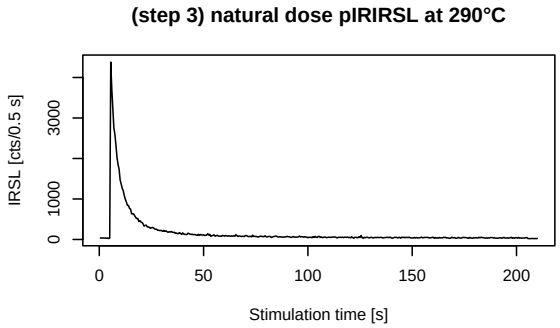
1.3 Measurement plots

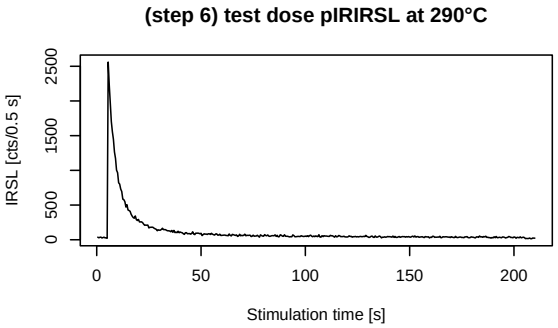


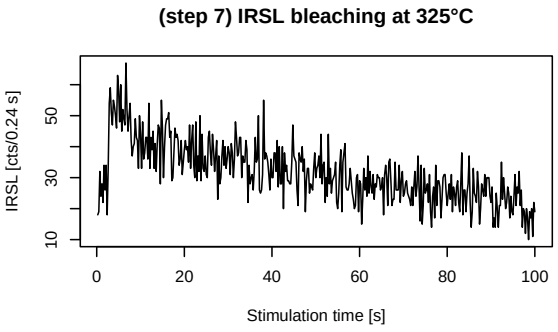












2 Data preparation

2.1 Data cleaning

The sequence structure and the record plots reveal two issues which need to be solved before the data can be further analysed with the function `analyse_SAR.CWOSL()` of the `Luminescence` package:

- The data contains three different types of IRSL steps
- At the beginning and the end of the measurement records, the stimulation light was turned off.

We use the function `RLum.OSL_correction()` of the package `OSLdecomposition` (Mittelstraß et al. 2022) to separate IRSL records and to remove the zero-stimulation parts of the records. The correction procedure log is shown below.

```
CORRECTION STEP 1 ----- Check records for consistency in the detection settings -----
Frequency table of different sets of detection settings (Channels, Channel width):
                settings frequency record_type
1               220, 0.5       336        IRSL
3               420, 0.5       336       IRSL2
2 420, 0.238095238095238       144       IRSL3
RLum.Data.Curve@RecordType changed to IRSL2 or IRSL3 in sequence: 1, 2, 3, 4, 5, 6, 7, 8, 9,
Further data manipulations are performed just on IRSL records
(time needed: 0.65 s)

CORRECTION STEP 2 ----- Remove not stimulated measurement parts -----
Measurement parts with stimulation light turned off detected and removed:
 5 s at the beginning and 5 s at the end.
-> Length of 336 IRSL records reduced from 110 s to 100 s
(time needed: 1.7 s)
```

We perform the code again but only for IRSL2 records to clean also 290°C IRSL records.

```
CORRECTION STEP 1 ----- Check records for consistency in the detection settings -----
All IRSL2 records have the same detection settings
(time needed: 0.28 s)

CORRECTION STEP 2 ----- Remove not stimulated measurement parts -----
Measurement parts with stimulation light turned off detected and removed:
 5 s at the beginning and 0 s at the end.
-> Length of 336 IRSL2 records reduced from 210 s to 205 s
(time needed: 3.12 s)
```

2.2 Dose rate calculation

The beta-source dose rate is not given in the data set. However, Dr. Tobias Lauer provided a calibrated dose rate value of 0.1053 ± 0.001 Gy/s measured in the same wwek as these measurements were performed. Thus it is not necessary to recalculate the dose rate.

We apply the dose rate on the IRR_TIME property of the records to get the applied doses in each step. The dose rate error will be added to the equivalent dose error in a later step.

2.3 Corrected data set

The above data preparation steps accumulate to a more reasonable sequence structure, where we also translate the record length into measurement time, add measurement cycle and step names and write proper column names:

Table 3: Measurement sequence structure for aliquot 1

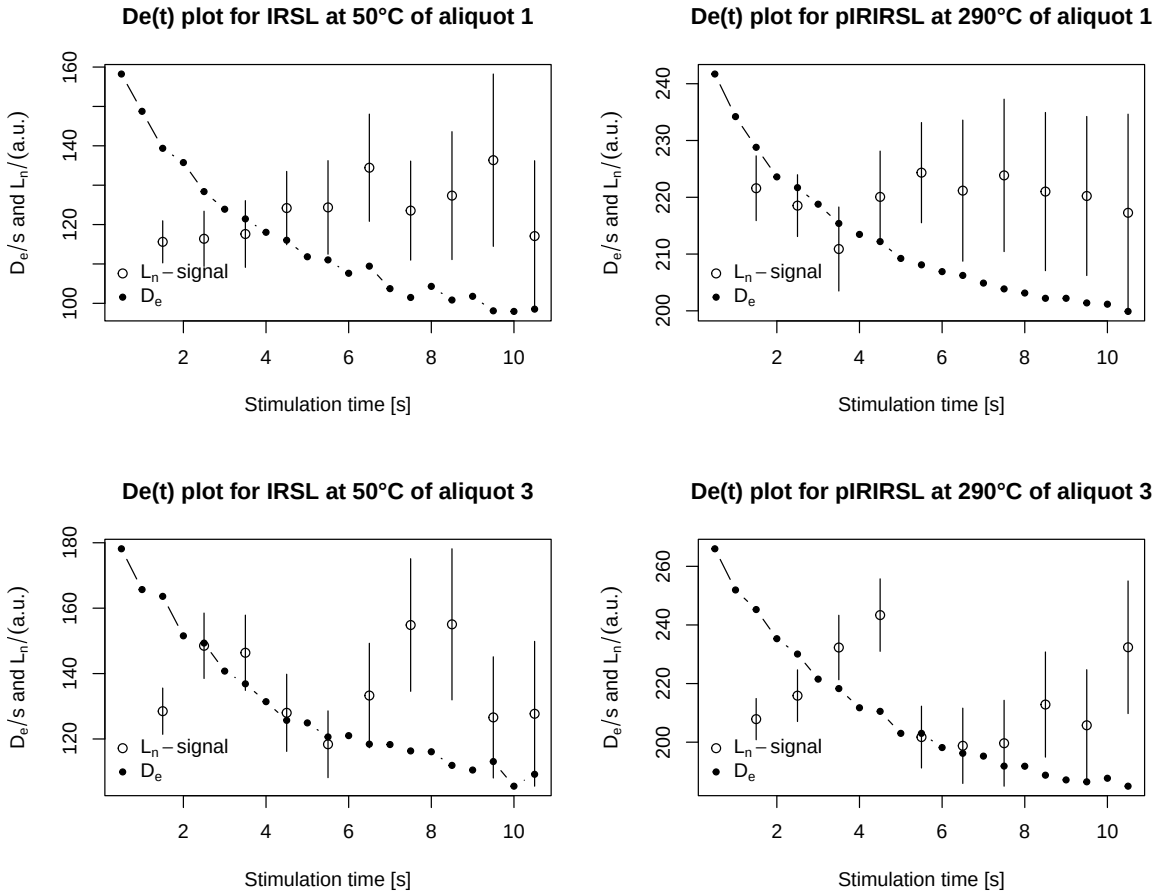
Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
1	TL	321	0.0	321.25	1.285	1	preheat
2	IRSL	50	0.0	100.00	0.500	1	Lx
3	IRSL2	290	0.0	205.00	0.500	1	Lx
4	TL	0	105.3	321.25	1.285	1	preheat
5	IRSL	50	105.3	100.00	0.500	1	Tx
6	IRSL2	290	105.3	205.00	0.500	1	Tx
7	IRSL3	325	105.3	99.96	0.238	1	bleaching
8	TL	321	0.0	321.25	1.285	2	preheat
9	IRSL	50	0.0	100.00	0.500	2	Lx
10	IRSL2	290	0.0	205.00	0.500	2	Lx
11	TL	0	105.3	321.25	1.285	2	preheat
12	IRSL	50	105.3	100.00	0.500	2	Tx
13	IRSL2	290	105.3	205.00	0.500	2	Tx
14	IRSL3	325	105.3	99.96	0.238	2	bleaching
15	TL	0	105.3	321.25	1.285	3	preheat
16	IRSL	50	105.3	100.00	0.500	3	Lx
17	IRSL2	290	105.3	205.00	0.500	3	Lx
18	TL	0	105.3	321.25	1.285	3	preheat
19	IRSL	50	105.3	100.00	0.500	3	Tx
20	IRSL2	290	105.3	205.00	0.500	3	Tx
21	IRSL3	325	105.3	99.96	0.238	3	bleaching
22	TL	0	210.6	321.25	1.285	4	preheat
23	IRSL	50	210.6	100.00	0.500	4	Lx

Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
24	IRSL2	290	210.6	205.00	0.500	4	Lx
25	TL	0	105.3	321.25	1.285	4	preheat
26	IRSL	50	105.3	100.00	0.500	4	Tx
27	IRSL2	290	105.3	205.00	0.500	4	Tx
28	IRSL3	325	105.3	99.96	0.238	4	bleaching
29	TL	0	421.2	321.25	1.285	5	preheat
30	IRSL	50	421.2	100.00	0.500	5	Lx
31	IRSL2	290	421.2	205.00	0.500	5	Lx
32	TL	0	105.3	321.25	1.285	5	preheat
33	IRSL	50	105.3	100.00	0.500	5	Tx
34	IRSL2	290	105.3	205.00	0.500	5	Tx
35	IRSL3	325	105.3	99.96	0.238	5	bleaching
36	TL	0	631.8	321.25	1.285	6	preheat
37	IRSL	50	631.8	100.00	0.500	6	Lx
38	IRSL2	290	631.8	205.00	0.500	6	Lx
39	TL	0	105.3	321.25	1.285	6	preheat
40	IRSL	50	105.3	100.00	0.500	6	Tx
41	IRSL2	290	105.3	205.00	0.500	6	Tx
42	IRSL3	325	105.3	99.96	0.238	6	bleaching
43	TL	0	105.3	321.25	1.285	7	preheat
44	IRSL	50	105.3	100.00	0.500	7	Lx
45	IRSL2	290	105.3	205.00	0.500	7	Lx
46	TL	0	105.3	321.25	1.285	7	preheat
47	IRSL	50	105.3	100.00	0.500	7	Tx
48	IRSL2	290	105.3	205.00	0.500	7	Tx

3 Dose calculation

3.1 De(t) plots

De(t) plotting helps to identify potential age over- or underestimation due to partial signal resetting, unstable signal components or other signal related issues (Bailey et al. 2003). Thus, we evaluated the De(t) plots for the first 10 seconds of stimulation for all measurements using the `plot_DetPlot()` function of the `Luminescence` package. Below are shown the plots for aliquot 1 and aliquot 3 as examples:



Like for other B19-LU samples the IRSL at 50°C measured equivalent doses are lower than the pIR-IRSL measured, which is a indication of thermal fading of the luminescence signal in the samples and thus also a indication of De underestimation in the IRSL at 50°C signals (Thomsen et al. 2008).

However, the significant increase in the De values when shifting the signal integration window towards higher which can be observed for at least 50 % of the aliquots of B19-LU 3, 4 and 5, are less frequent in B19-LU 6.

3.2 Dose-response curve fitting optimization

To calculate the De values relevant for dating, we discard the IRSL at 50°C signals and use just the pIR IRSL measurements. The De values are calculated using the `analyse_SAR.CWOSL()` function of the `Luminescence` package.

As the dose calculation of sample **B19 LU-1** revealed a dependence of the De results and the rejection test results from the growth-curve parameters, see details in the B19 LU-1 report), the same parameters were also tested for B19 LU-6. The tables below compare the number of rejected aliquots as well as the Central Age Model (CAM) results in dependence to the chosen parameters:

Table 4: Analysis results setting `fit.method = EXP` (default model).

Signal integration window	Aliquots that passed RC (of 24)	CAM paleodose [Gy]	Overdispersion [%]
1.5 sec	24	240.48 +- 4.26	8.10
3.5 sec	24	241.50 +- 4.02	7.79

Table 5: Analysis results setting `fit.method = GOK` (model by Guralnik et al. (2015)).

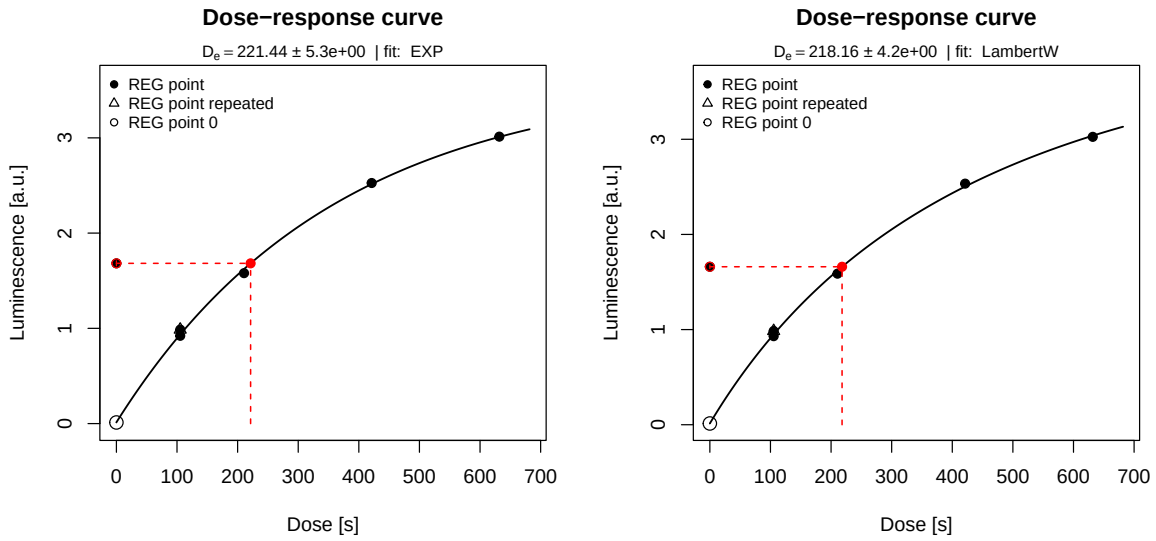
Signal integration window	Aliquots that passed RC (of 24)	CAM paleodose [Gy]	Overdispersion [%]
1.5 sec	24	244.30 +- 4.17	7.67
3.5 sec	24	245.74 +- 3.93	7.37

Table 6: Analysis results setting `fit.method = LambertW` (model by Pagonis et al. (2020)).

Signal integration window	Aliquots that passed RC (of 24)	CAM paleodose [Gy]	Overdispersion [%]
1.5 sec	24	244.87 +- 4.11	7.39
3.5 sec	**24**	**246.12 +- 3.90**	**7.25**

Changing the growth curve parameters has no significant impact on the result. Thus, the same parameters as used for B19 LU-1 were used for this data set (**bold** table row) to ensure

parameter uniformity. Please be aware that the results displayed in the tables above are not exactly reproducible as the used calculation procedures deploy Monte Carlo methods.



3.3 De calculation result table

Table 7: Equivalent doses for pIRIR290 measurements

#	De [Gy]	De error [Gy]	D01 [Gy]	D01 error [Gy]	Rejection criteria
1	218.16	4.72	NA	NA	OK
2	243.66	6.13	NA	NA	OK
3	225.72	7.92	NA	NA	OK
4	287.60	12.27	NA	NA	OK
5	211.27	4.10	NA	NA	OK
6	268.94	7.84	NA	NA	OK
7	247.31	7.05	NA	NA	OK
8	257.46	8.61	NA	NA	OK
9	243.85	5.19	NA	NA	OK
10	268.34	7.15	NA	NA	OK
11	222.19	4.77	NA	NA	OK
12	251.82	5.45	NA	NA	OK
13	248.43	8.31	NA	NA	OK
14	258.56	4.27	NA	NA	OK
15	252.35	5.82	NA	NA	OK
16	223.90	6.91	NA	NA	OK

#	De [Gy]	De error [Gy]	D01 [Gy]	D01 error [Gy]	Rejection criteria
17	267.54	8.26	NA	NA	OK
18	251.75	8.32	NA	NA	OK
19	248.89	8.56	NA	NA	OK
20	217.38	5.99	NA	NA	OK
21	260.50	7.23	NA	NA	OK
22	268.36	5.05	NA	NA	OK
23	248.82	7.93	NA	NA	OK
24	238.39	8.17	NA	NA	OK

24 of all aliquots passed the rejection criteria. The results of all aliquots in the table above include the dose rate errors.

3.4 Rejection criteria

Table 8: Rejection criteria thresholds (left) and results (right)

#	Criterium	Threshold
A	Recycling ratio (R5/R1)	0.1
B	Recuperation rate 1	0.1
C	Testdose error	0.1
D	Palaeodose error	0.1
E	De > max. dose point	631.8

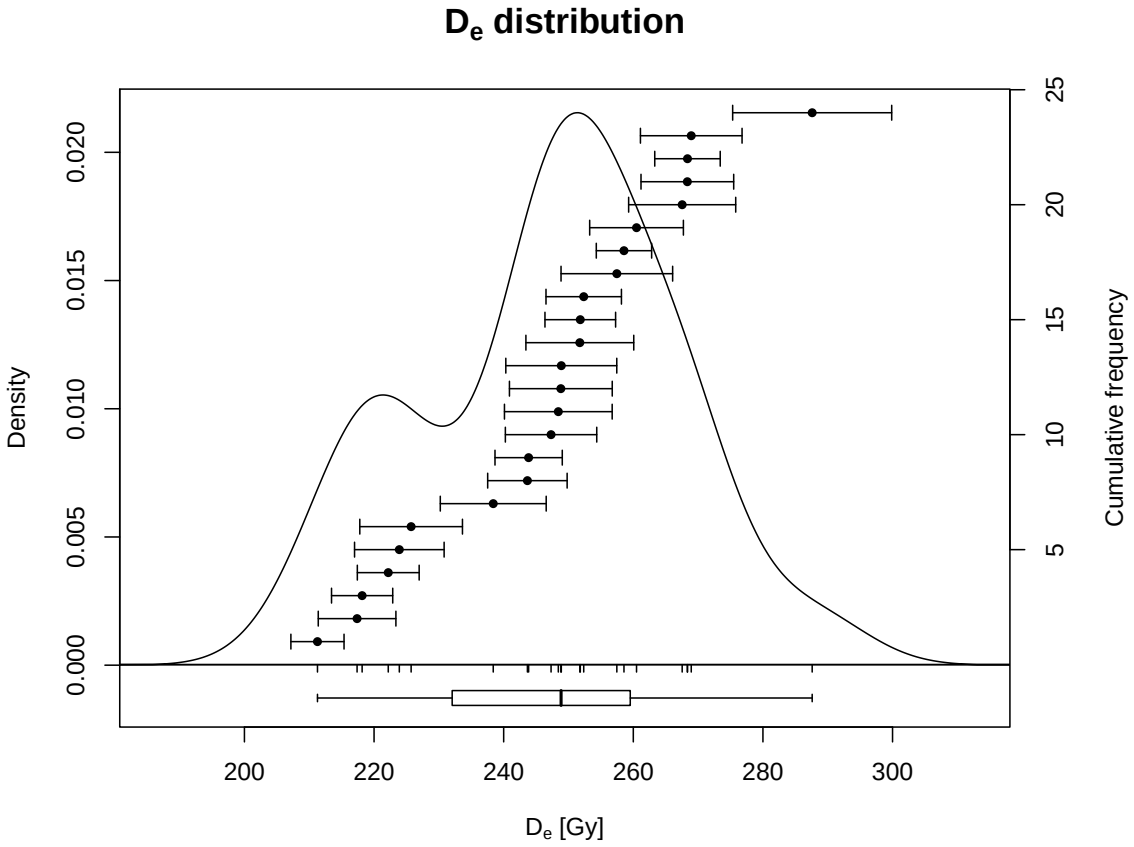
#	A	B	C	D	E
1	1.058	0.008	0.009	0.019	218.161
2	1.004	0.010	0.010	0.023	243.660
3	1.012	0.006	0.012	0.034	225.722
4	1.019	0.009	0.018	0.042	287.597
5	1.028	0.007	0.007	0.017	211.271
6	1.026	0.012	0.012	0.028	268.944
7	1.023	0.011	0.011	0.027	247.310
8	1.048	0.023	0.012	0.032	257.464
9	1.026	0.005	0.010	0.019	243.847
10	1.052	0.010	0.009	0.025	268.337
11	1.073	0.009	0.008	0.019	222.194
12	0.990	0.012	0.007	0.019	251.820
13	1.024	0.005	0.012	0.032	248.427
14	1.040	0.007	0.005	0.014	258.560
15	1.046	0.009	0.011	0.021	252.350
16	1.015	0.007	0.013	0.029	223.900
17	1.058	0.009	0.011	0.029	267.541
18	1.024	0.007	0.013	0.032	251.755
19	0.998	0.013	0.013	0.033	248.887
20	1.024	0.008	0.009	0.026	217.384
21	1.042	0.011	0.011	0.026	260.501
22	1.008	0.009	0.008	0.016	268.360
23	1.028	0.007	0.014	0.030	248.818
24	1.010	0.016	0.014	0.033	238.386

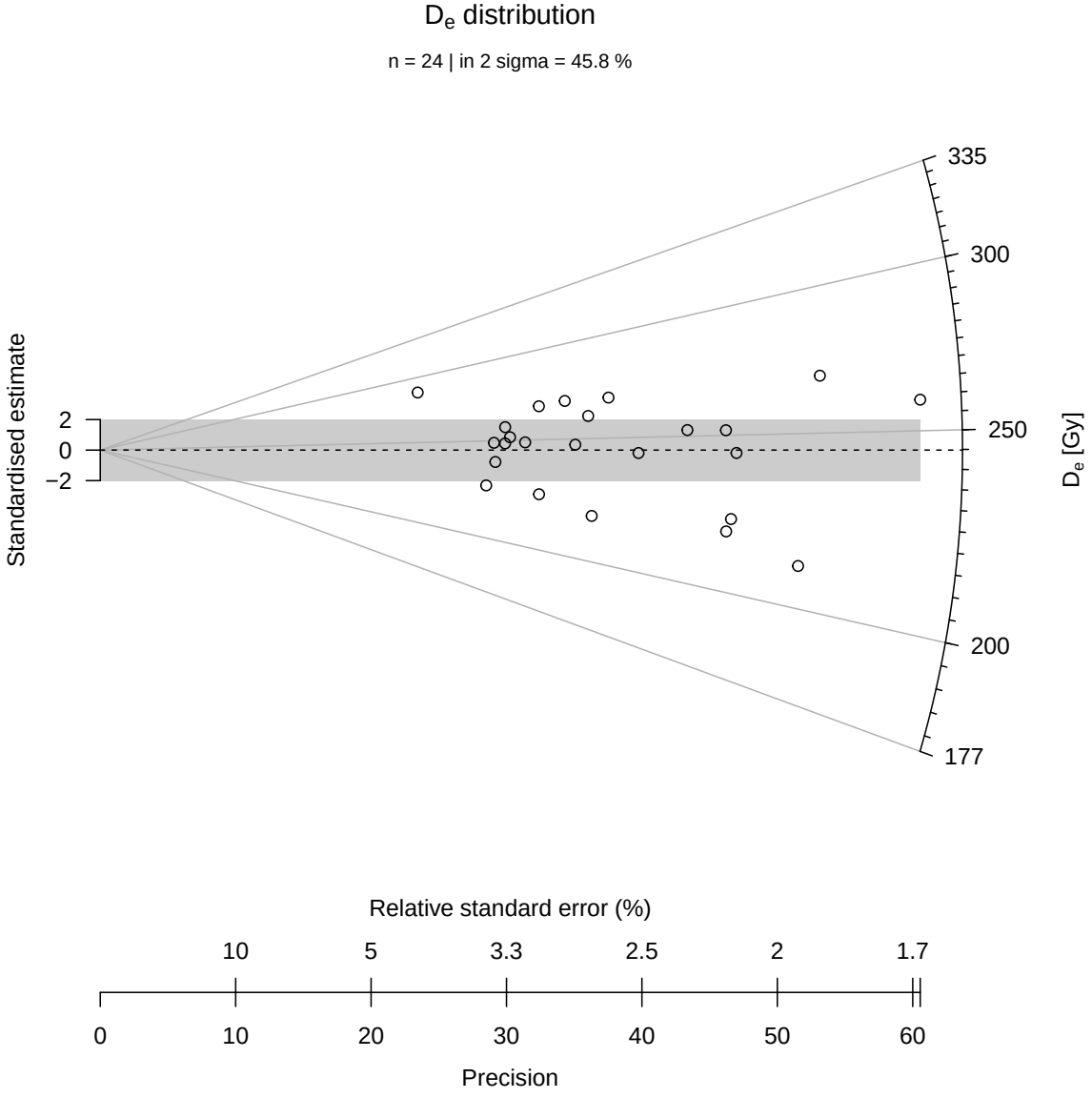
4 Dose distribution

Important: Those aliquots which did not passed the rejection criteria, where not included in any of the dose distribution calculations.

4.1 Distribution plots

The dose distribution is plotted below with the functions `plot_KDE()` and `plot_RadialPlot()` of the `Luminescence` package.




De distribution
n = 24 | in 2 sigma = 45.8 %
Standardised estimate
2
0
−2
335
300
250
200
177
De [Gy]
Relative standard error (%)
10
5
3.3
2.5
2
1.7
0
10
20
30
40
50
60
Precision

4.2 Central age model

Below is output of the function `calc_CentralDose()` of the `Luminescence` package shown, which calculates the central dose and the over-dispersion of the De distribution in accordance to the model given by Galbraith et al. (1999) .

```
 [calc_CentralDose]

---------- meta data ----------------
 n:                       24
 log:                     TRUE
---------- dose estimate ------------
 abs. central dose:       246.08
 abs. SE:                 3.91
 rel. SE [%]:             1.59
---------- overdispersion -----------
 abs. OD:                 17.83
 abs. SE:                 2.96
 OD [%]:                  7.25
 SE [%]:                  1.20
-------------------------------------
```

SE = standard error, OD = over-dispersion

5 References

Bailey, R.M., Singarayer, J.S., Ward, S., Stokes, S., 2003. Identification of partial resetting using De as a function of illumination time. Radiation Measurements 37, 511-518. doi:10.1016/S1350-4487(03)00063-5

Galbraith, R.F., Roberts, R.G., Laslett, G.M., Yoshida, H. & Olley, J.M., 1999. Optical dating of single grains of quartz from Jinmium rock shelter, northern Australia. Part I: experimental design and statistical models. Archaeometry 41, 339-364.

Guralnik, B., Li, B., Jain, M., Chen, R., Paris, R.B., Murray, A.S., Li, S.-H., Pagonis, P., Herman, F., 2015. Radiation-induced growth and isothermal decay of infrared-stimulated luminescence from feldspar. Radiation Measurements 81, 224-231.

Kreutzer S, Burow C, Dietze M, Fuchs M, Schmidt C, Fischer M, Friedrich J, Mercier N, Philippe A, Riedesel S, Autzen M, Mittelstrass D, Gray H, Galharret J (2022). *Luminescence: Comprehensive Luminescence Dating Data Analysis*. R package version 0.9.20, https://CRAN.R-project.org/package=Luminescence.

Kreutzer S, Schmidt C, Fuchs MC, Dietze M, Fischer M, Fuchs M (2012). "Introducing an R package for luminescence dating analysis." *Ancient TL*, *30*(1), 1-8.

Mittelstraß D, Kreutzer S, Schmidt C (2022). *OSLdecomposition: Signal Component Analysis for Optically Stimulated Luminescence*. R package version 1.0.0, https://CRAN.R-project.org/package=OSLdecomposition.

Pagonis, V., Kitis, G., Chen, R., 2020. A new analytical equation for the dose response of dosimetric materials, based on the Lambert W function. Journal of Luminescence 225, 117333.

Thomsen, K.J., Murray, A.S., Jain, M., Bøtter-Jensen, L., 2008. Laboratory fading rates of various luminescence signals from feldspar-rich sediment extracts. Radiation Measurements 43, 1474–1486.