pIRIR analysis B19-LU 13

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Analysed file: 3-Permafrost-1mm-63-90-Fsp_LU-13_24al.binx

Date of analysis: 2023-07-23

This report describes the data analysis work flow for the pIRIR measurement of the sample *B19-LU 13* , 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 programming 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	LU-7	260423	2	1	1	55
2	LU-7	260423	2	1	3	55
3	LU-7	260423	2	1	5	55
4	LU-8	260423	2	1	7	55
5	LU-8	260423	2	1	9	55
6	LU-8	260423	2	1	11	55
7	LU-9	260423	2	1	13	55
8	LU-9	260423	2	1	15	55
9	LU-9	260423	2	1	17	55
10	LU-10	260423	2	1	19	55
11	LU-10	260423	2	1	21	55
12	LU-10	260423	2	1	23	55
13	LU-12	260423	2	1	25	55
14	LU-12	260423	2	1	27	55
15	LU-12	260423	2	1	29	55
16	LU-13	260423	2	1	31	55
17	LU-13	260423	2	1	33	55
18	LU-13	260423	2	1	35	55
19	LU-14	260423	2	1	37	55
20	LU-14	260423	2	1	39	55
21	LU-14	270423	2	1	41	55
22	Sample43	270423	2	1	43	55
23	Sample45	270423	2	1	45	55
24	Sample47	270423	2	1	47	55

According to the sample names, this was a mixed sample run. However, the relatively narrow dose distribution we will observe later points towards a single-sample measurement campaign.

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

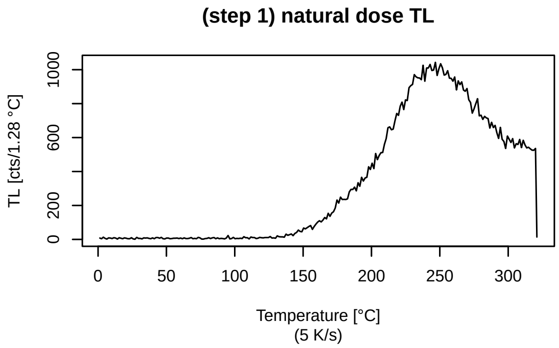
1.2 Measurement sequence

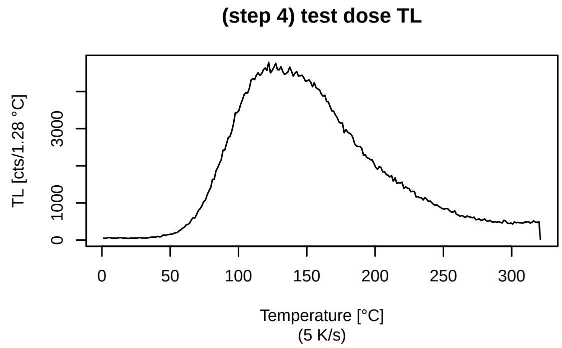
Table 2: Measurement sequence structure for aliquot 1

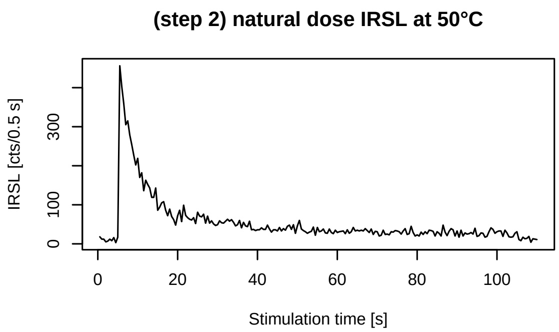
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
1	TL	321	0	250	1.285
2	IRSL	50	0	220	0.500
3	IRSL	290	0	420	0.500
4	TL	0	1000	250	1.285
5	IRSL	50	1000	220	0.500
6	IRSL	290	1000	420	0.500
7	IRSL	325	1000	420	0.238
8	TL	321	0	250	1.285
9	IRSL	50	0	220	0.500
10	IRSL	290	0	420	0.500
11	TL	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	500	250	1.285
16	IRSL	50	500	220	0.500
17	IRSL	290	500	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	1000	250	1.285
23	IRSL	50	1000	220	0.500
24	IRSL	290	1000	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	2000	250	1.285
30	IRSL	50	2000	220	0.500
31	IRSL	290	2000	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	3000	250	1.285
37	IRSL	50	3000	220	0.500

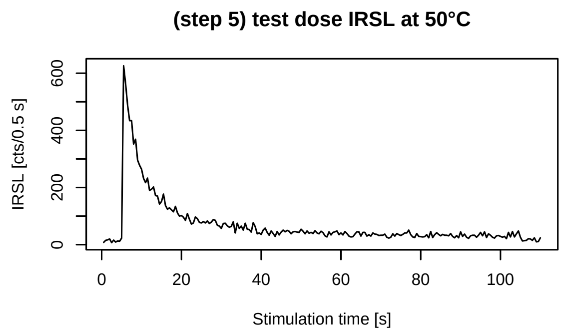
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
38	IRSL	290	3000	420	0.500
39	TL	0	1000	250	1.285
40	IRSL	50	1000	220	0.500
41	IRSL	290	1000	420	0.500
42	IRSL	325	1000	420	0.238
43	TL	0	5000	250	1.285
44	IRSL	50	5000	220	0.500
45	IRSL	290	5000	420	0.500
46	TL	0	1000	250	1.285
47	IRSL	50	1000	220	0.500
48	IRSL	290	1000	420	0.500
49	IRSL	325	1000	420	0.238
50	TL	0	500	250	1.285
51	IRSL	50	500	220	0.500
52	IRSL	290	500	420	0.500
53	TL	0	1000	250	1.285
54	IRSL	50	1000	220	0.500
55	IRSL	290	1000	420	0.500

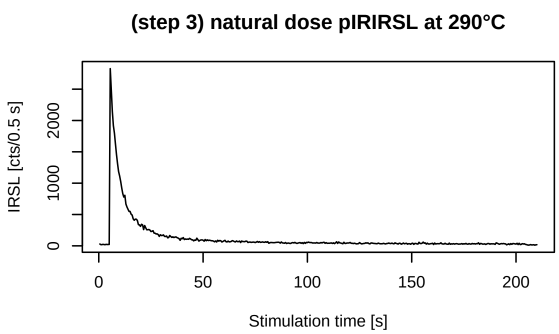
1.3 Measurement plots

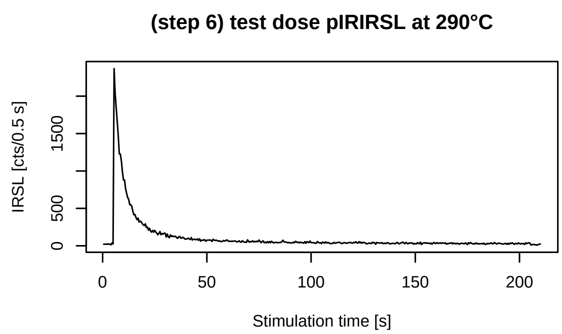


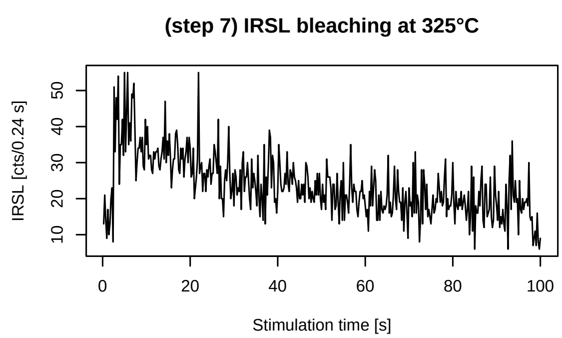












2 Data preparation

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       384        IRSL
3               420, 0.5       384       IRSL2
2 420, 0.238095238095238       168       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: 1.54 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 384 IRSL records reduced from 110 s to 100 s
(time needed: 3.71 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: 0.58 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 384 IRSL2 records reduced from 210 s to 205 s
(time needed: 6.38 s)
```

2.2 Dose rate calculation

Dr. Tobias Lauer provided a calibrated laboratory dose rate value of 0.1053 ± 0.001 Gy/s measured in October 2022. We recalculate the dose rate to the date of the measurement and set as common uncertainty value 5 %.

The recalculated dose rate is 0.104 Gy/s with a dose rate error of 0.0052 Gy/s.

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

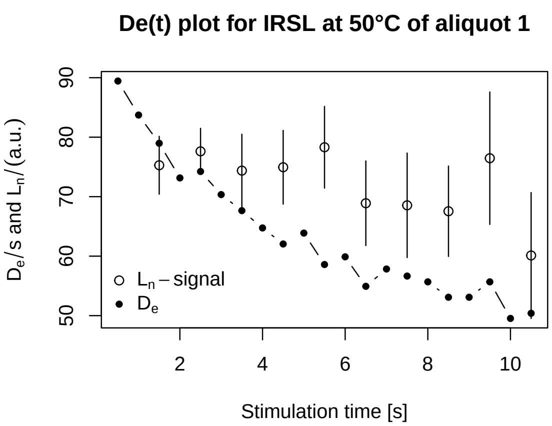
Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
1	TL	321	0.0	321.25	1.285	1	preheat
2	IRSL	50	0.0	100.00	0.500	1	Lx
3	IRSL2	290	0.0	205.00	0.500	1	Lx
4	TL	0	104.0	321.25	1.285	1	preheat
5	IRSL	50	104.0	100.00	0.500	1	Tx
6	IRSL2	290	104.0	205.00	0.500	1	Tx
7	IRSL3	325	104.0	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	104.0	321.25	1.285	2	preheat
12	IRSL	50	104.0	100.00	0.500	2	Tx
13	IRSL2	290	104.0	205.00	0.500	2	Tx
14	IRSL3	325	104.0	99.96	0.238	2	bleaching
15	TL	0	52.0	321.25	1.285	3	preheat
16	IRSL	50	52.0	100.00	0.500	3	Lx
17	IRSL2	290	52.0	205.00	0.500	3	Lx
18	TL	0	104.0	321.25	1.285	3	preheat
19	IRSL	50	104.0	100.00	0.500	3	Tx
20	IRSL2	290	104.0	205.00	0.500	3	Tx
21	IRSL3	325	104.0	99.96	0.238	3	bleaching
22	TL	0	104.0	321.25	1.285	4	preheat
23	IRSL	50	104.0	100.00	0.500	4	Lx
24	IRSL2	290	104.0	205.00	0.500	4	Lx

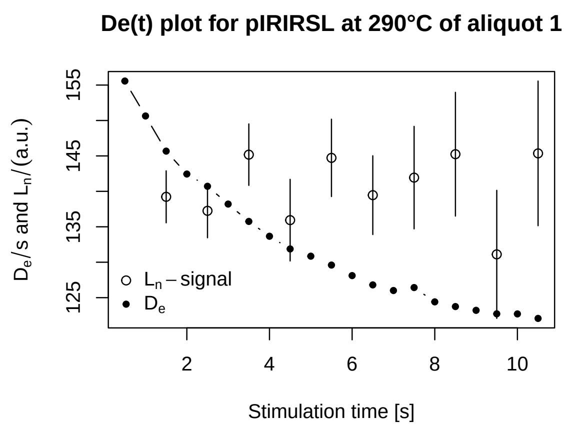
Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
25	TL	0	104.0	321.25	1.285	4	preheat
26	IRSL	50	104.0	100.00	0.500	4	Tx
27	IRSL2	290	104.0	205.00	0.500	4	Tx
28	IRSL3	325	104.0	99.96	0.238	4	bleaching
29	TL	0	207.9	321.25	1.285	5	preheat
30	IRSL	50	207.9	100.00	0.500	5	Lx
31	IRSL2	290	207.9	205.00	0.500	5	Lx
32	TL	0	104.0	321.25	1.285	5	preheat
33	IRSL	50	104.0	100.00	0.500	5	Tx
34	IRSL2	290	104.0	205.00	0.500	5	Tx
35	IRSL3	325	104.0	99.96	0.238	5	bleaching
36	TL	0	311.9	321.25	1.285	6	preheat
37	IRSL	50	311.9	100.00	0.500	6	Lx
38	IRSL2	290	311.9	205.00	0.500	6	Lx
39	TL	0	104.0	321.25	1.285	6	preheat
40	IRSL	50	104.0	100.00	0.500	6	Tx
41	IRSL2	290	104.0	205.00	0.500	6	Tx
42	IRSL3	325	104.0	99.96	0.238	6	bleaching
43	TL	0	519.8	321.25	1.285	7	preheat
44	IRSL	50	519.8	100.00	0.500	7	Lx
45	IRSL2	290	519.8	205.00	0.500	7	Lx
46	TL	0	104.0	321.25	1.285	7	preheat
47	IRSL	50	104.0	100.00	0.500	7	Tx
48	IRSL2	290	104.0	205.00	0.500	7	Tx
49	IRSL3	325	104.0	99.96	0.238	7	bleaching
50	TL	0	52.0	321.25	1.285	NA	NA
51	IRSL	50	52.0	100.00	0.500	NA	NA
52	IRSL2	290	52.0	205.00	0.500	NA	NA
53	TL	0	104.0	321.25	1.285	NA	NA
54	IRSL	50	104.0	100.00	0.500	NA	NA
55	IRSL2	290	104.0	205.00	0.500	NA	NA

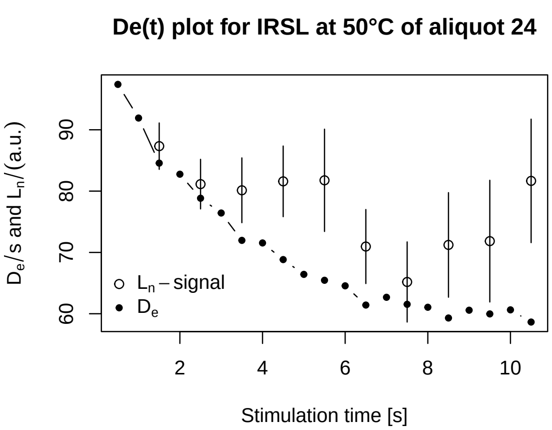
3 Dose calculation

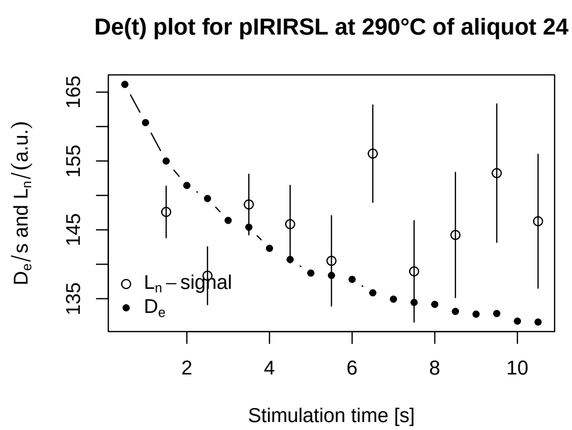
3.1 De(t) plots

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 of two aliquots as examples:









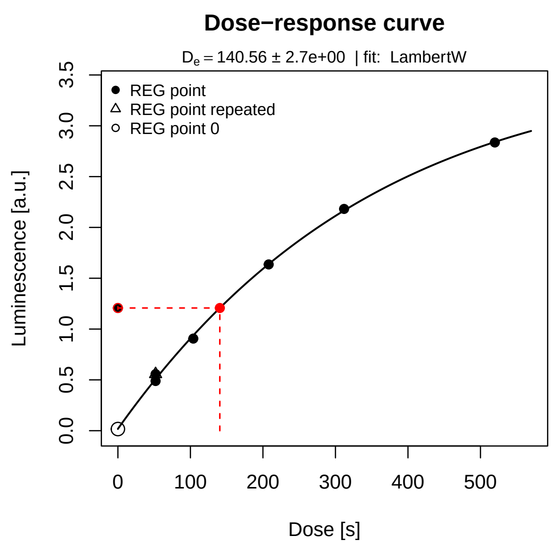
Like for other B19-LU samples the IRSL at 50°C measured equivalent doses are lower than the pIR-IRSL measured, which is a indication of thermal fading of the luminescence signal in the samples and thus also a indication of De underestimation in the IRSL at 50°C signals (Thomsen et al. 2008).

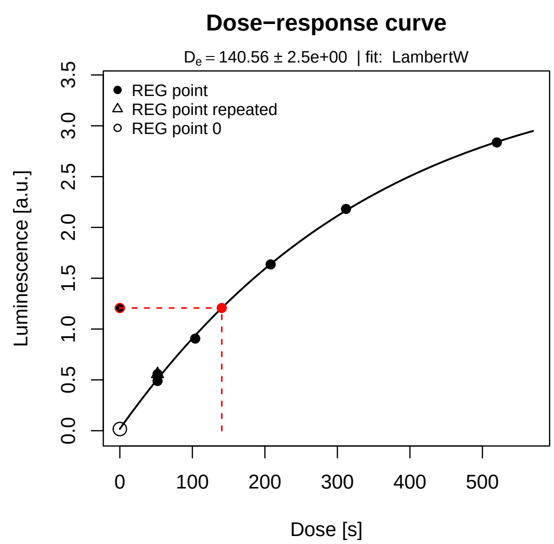
However, the increase in the De values when shifting the signal integration window towards higher values which can be observed for other B19 samples can also be observed for some of the aliquots of this sample.

3.2 Dose-response curve fitting

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 value scattering and the likelihood of aliquot rejections from the growth-curve parameters (see details in the B19 LU-1 report). The optimum parameters for **B19 LU-1** and **B19 LU-6** are also used for this sample. Thus, we use as fitting method `fit.method = LambertW` (model by Pagonis et al. (2020)) and integrate over the first seven channels (3.5 seconds). As background integral, we choose the last 100 channels (150 to 200 sec).





3.3 De calculation result table

Table 4: Equivalent doses for pIRIR290 measurements

#	De [Gy]	De error [Gy]	D01 [Gy]	D01 error [Gy]	Rejection criteria
1	140.56	7.36	NA	NA	FAILED
2	149.89	7.98	NA	NA	OK

#	De [Gy]	De error [Gy]	D01 [Gy]	D01 error [Gy]	Rejection criteria
3	175.45	9.03	NA	NA	OK
4	163.95	8.65	NA	NA	FAILED
5	149.33	7.94	NA	NA	FAILED
6	131.77	6.86	NA	NA	FAILED
7	166.61	8.53	NA	NA	OK
8	147.83	8.04	NA	NA	OK
9	149.91	7.81	NA	NA	OK
10	156.61	8.29	NA	NA	OK
11	167.86	8.57	NA	NA	OK
12	166.71	9.27	NA	NA	FAILED
13	169.69	8.93	NA	NA	OK
14	153.01	8.05	NA	NA	OK
15	134.03	8.93	NA	NA	FAILED
16	138.67	7.46	NA	NA	OK
17	167.90	8.80	NA	NA	OK
18	159.99	8.60	NA	NA	OK
19	151.81	7.92	NA	NA	OK
20	167.05	8.64	NA	NA	OK
21	147.18	8.11	NA	NA	OK
22	143.33	7.61	NA	NA	OK
23	181.94	9.72	NA	NA	FAILED
24	146.46	7.84	NA	NA	OK

17 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 5: Rejection criteria thresholds (left) and results (right)

#	Criterium	Threshold
A	Recycling ratio (R6/R1)	0.100
B	Recuperation rate 1	0.100
C	Testdose error	0.100
D	Palaeodose error	0.100
E	De > max. dose point	519.831

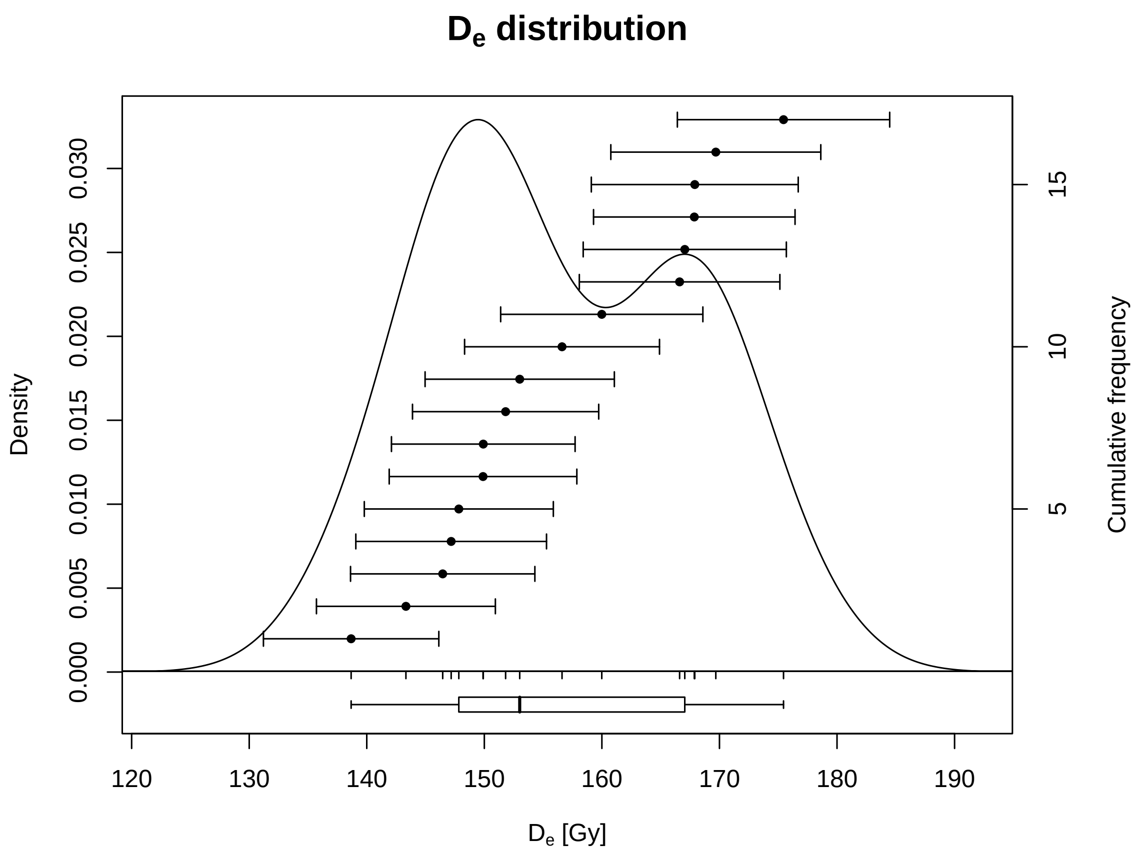
#	A	B	C	D	E
1	1.138	0.013	0.010	0.015	140.562
2	1.059	0.010	0.009	0.018	149.885
3	1.064	0.006	0.007	0.012	175.453
4	1.104	0.012	0.008	0.017	163.947
5	1.110	0.011	0.010	0.018	149.329
6	1.126	0.012	0.008	0.014	131.774
7	1.062	0.017	0.005	0.011	166.612
8	1.084	0.013	0.011	0.021	147.831
9	1.093	0.015	0.007	0.015	149.913
10	1.076	0.012	0.008	0.017	156.614
11	1.072	0.011	0.005	0.010	167.862
12	1.154	0.028	0.012	0.024	166.711
13	1.076	0.010	0.009	0.016	169.688
14	1.096	0.013	0.008	0.016	153.010
15	1.140	0.024	0.026	0.044	134.030
16	1.086	0.013	0.009	0.020	138.669
17	1.072	0.013	0.008	0.016	167.905
18	1.051	0.021	0.010	0.020	159.995
19	1.059	0.010	0.007	0.015	151.813
20	1.075	0.012	0.007	0.013	167.046
21	1.076	0.011	0.013	0.023	147.178
22	1.082	0.006	0.008	0.018	143.326
23	1.126	0.009	0.009	0.019	181.936
24	1.097	0.013	0.009	0.019	146.465

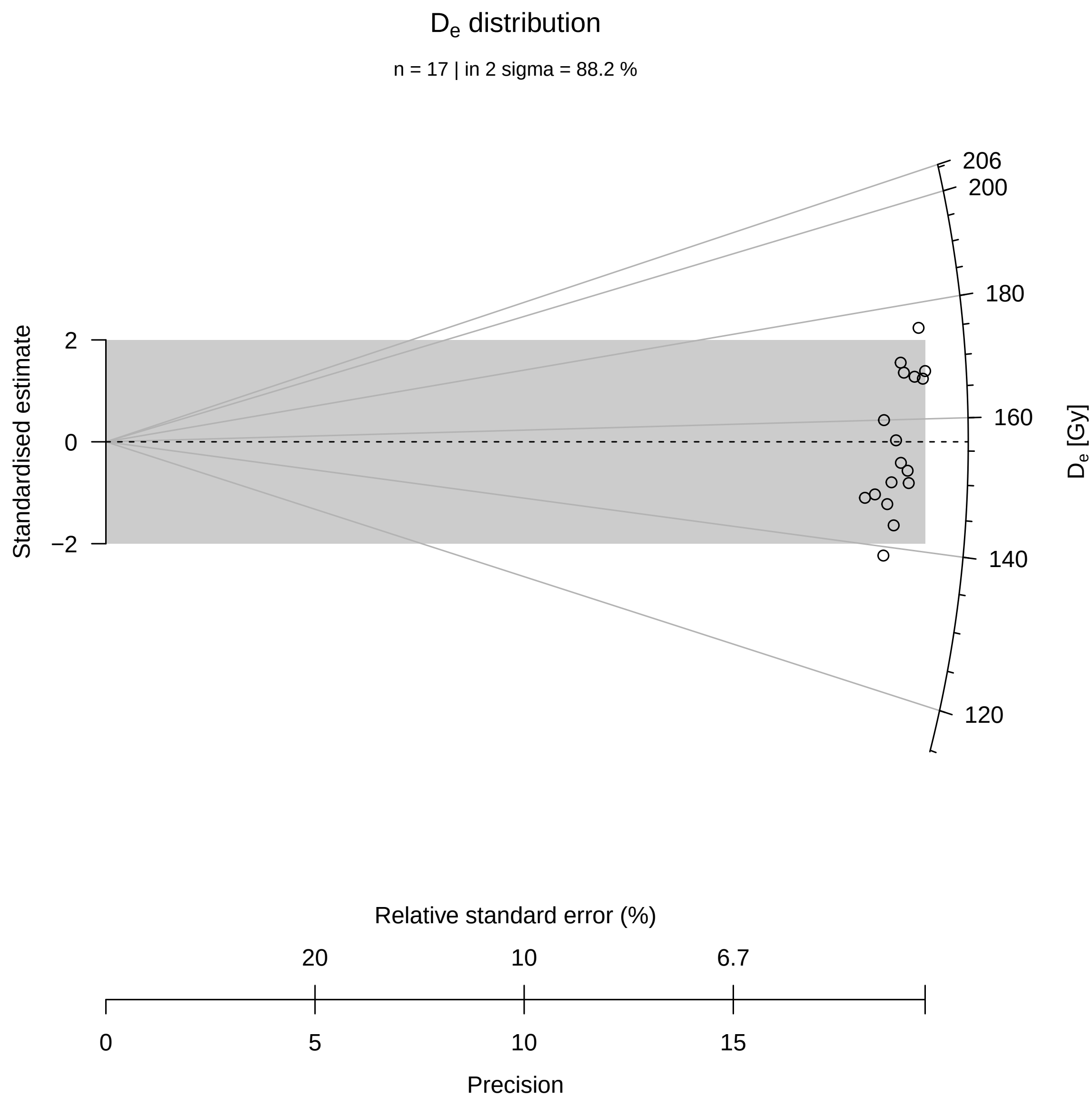
4 Dose distribution

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.





D_e distribution
n = 17 | in 2 sigma = 88.2 %
Standardised estimate
2
0
−2
206
200
180
160
140
120
D_e [Gy]
Relative standard error (%)
20
10
6.7
0
5
10
15
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:                        17
 log:                      TRUE
----------- dose estimate ------------
 abs. central dose:        156.26
 abs. SE:                  2.56
 rel. SE [%]:              1.64
----------- overdispersion -----------
 abs. OD:                  6.57
 abs. SE:                  2.90
 OD [%]:                   4.20
 SE [%]:                   1.86
-------------------------------------
```

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.

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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.

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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.