pIRIR analysis B19-LU 5

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Analysed file: 1-Permafrost-1mm-63-100-Fsp-pIR290-sample_B19-LU-5-24al.binx

This report describes the data analysis work flow for the pIRIR measurement of the sample *B19-LU 5*, measured by 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	290822	2	1	1	48
2	Sample3	290822	2	1	3	48
3	Sample5	290822	2	1	5	48
4	Sample7	290822	2	1	7	48
5	Sample9	290822	2	1	9	48
6	Sample11	290822	2	1	11	48
7	Sample13	290822	2	1	13	48
8	Sample15	290822	2	1	15	48
9	Sample17	290822	2	1	17	48
10	Sample19	290822	2	1	19	48
11	Sample21	290822	2	1	21	48
12	Sample23	290822	2	1	23	48
13	Sample25	290822	2	1	25	48
14	Sample27	290822	2	1	27	48
15	Sample29	290822	2	1	29	48
16	Sample31	290822	2	1	31	48
17	Sample33	290822	2	1	33	48
18	Sample35	290822	2	1	35	48
19	Sample37	290822	2	1	37	48
20	Sample39	290822	2	1	39	48
21	Sample41	300822	2	1	41	48
22	Sample43	300822	2	1	43	48
23	Sample45	300822	2	1	45	48
24	Sample47	300822	2	1	47	48

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

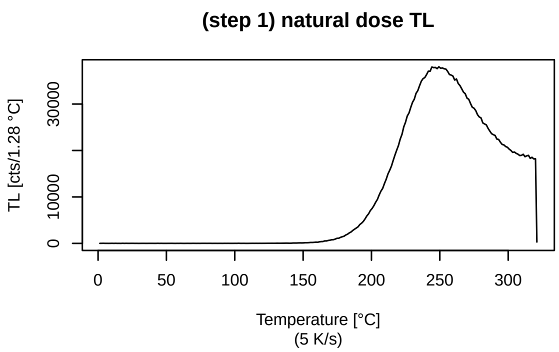
1.2 Measurement sequence

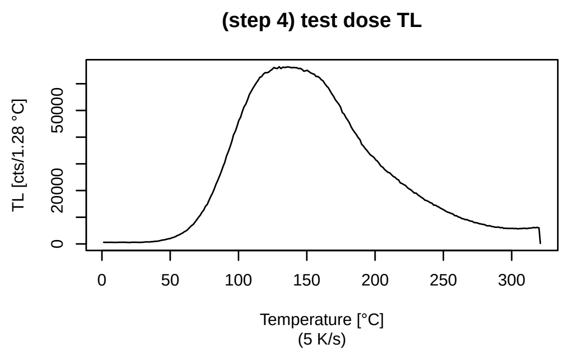
Table 2: Measurement sequence structure for aliquot 1

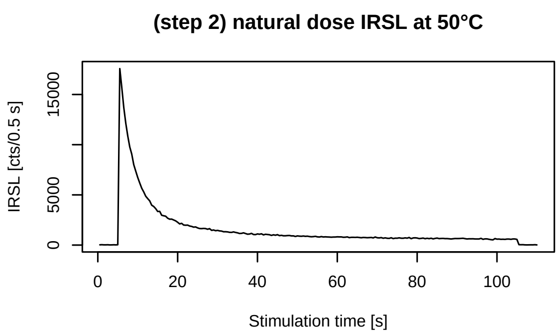
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
1	TL	321	0	250	1.285
2	IRSL	50	0	220	0.500
3	IRSL	290	0	420	0.500
4	TL	0	1000	250	1.285
5	IRSL	50	1000	220	0.500
6	IRSL	290	1000	420	0.500
7	IRSL	325	1000	420	0.238
8	TL	321	0	250	1.285
9	IRSL	50	0	220	0.500
10	IRSL	290	0	420	0.500
11	TL	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	2000	250	1.285
16	IRSL	50	2000	220	0.500
17	IRSL	290	2000	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	4000	250	1.285
23	IRSL	50	4000	220	0.500
24	IRSL	290	4000	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	6000	250	1.285
30	IRSL	50	6000	220	0.500
31	IRSL	290	6000	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	8000	250	1.285
37	IRSL	50	8000	220	0.500

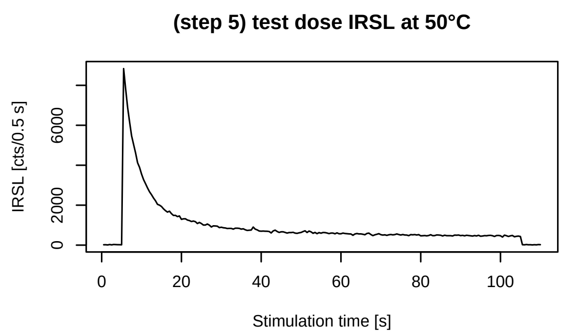
#	Type	max. Temp. [°C]	Irr. time [s]	Channels	Channel width [s]
38	IRSL	290	8000	420	0.500
39	TL	0	1000	250	1.285
40	IRSL	50	1000	220	0.500
41	IRSL	290	1000	420	0.500
42	IRSL	325	1000	420	0.238
43	TL	0	2000	250	1.285
44	IRSL	50	2000	220	0.500
45	IRSL	290	2000	420	0.500
46	TL	0	1000	250	1.285
47	IRSL	50	1000	220	0.500
48	IRSL	290	1000	420	0.500

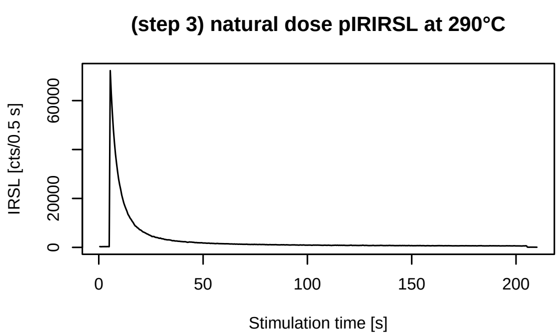
1.3 Measurement plots

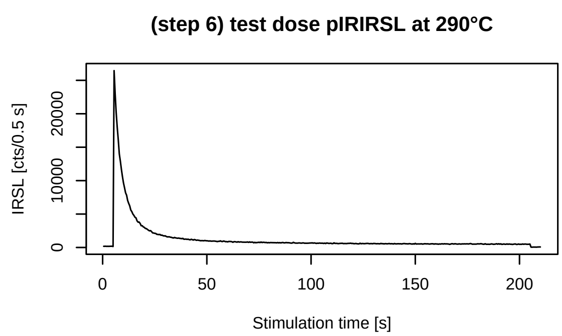


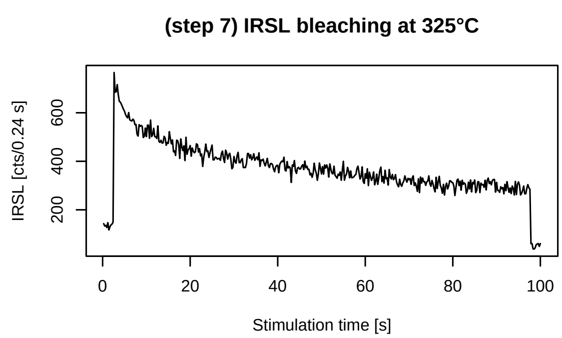












2 Data preparation

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: 1.02 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: 2.26 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: 0.33 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 IRSL2 records reduced from 210 s to 200 s
(time needed: 4.61 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 0.0768 ± 0.001 Gy/s measured at 2022/07/01. We calculate the dose rate at the day of measurement by using the function `calc_SourceDoseRate()` of the `Luminescence` package and write the results into the data set.

The dose rate is 0.0765 ± 0.001 Gy/s. 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

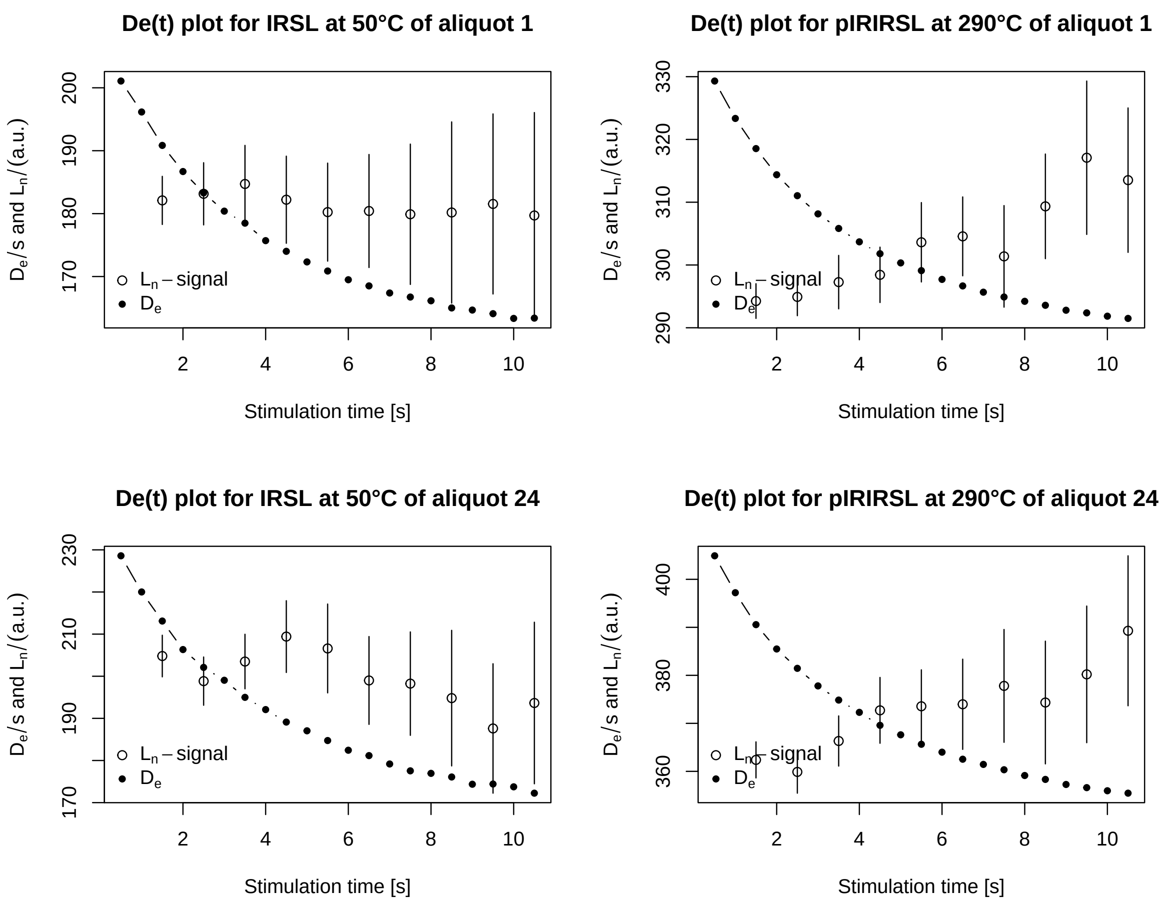
Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
1	TL	321	0.0	321.25	1.285	1	preheat
2	IRSL	50	0.0	100.00	0.500	1	Lx
3	IRSL2	290	0.0	200.00	0.500	1	Lx
4	TL	0	76.5	321.25	1.285	1	preheat
5	IRSL	50	76.5	100.00	0.500	1	Tx
6	IRSL2	290	76.5	200.00	0.500	1	Tx
7	IRSL3	325	76.5	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	200.00	0.500	2	Lx
11	TL	0	76.5	321.25	1.285	2	preheat
12	IRSL	50	76.5	100.00	0.500	2	Tx
13	IRSL2	290	76.5	200.00	0.500	2	Tx
14	IRSL3	325	76.5	99.96	0.238	2	bleaching
15	TL	0	153.0	321.25	1.285	3	preheat
16	IRSL	50	153.0	100.00	0.500	3	Lx
17	IRSL2	290	153.0	200.00	0.500	3	Lx
18	TL	0	76.5	321.25	1.285	3	preheat
19	IRSL	50	76.5	100.00	0.500	3	Tx
20	IRSL2	290	76.5	200.00	0.500	3	Tx
21	IRSL3	325	76.5	99.96	0.238	3	bleaching

Index	Record Type	Temperature (°C)	Dose (Gy)	Meas. time (sec)	Ch. width (sec)	Meas. cycle	Step
22	TL	0	306.0	321.25	1.285	4	preheat
23	IRSL	50	306.0	100.00	0.500	4	Lx
24	IRSL2	290	306.0	200.00	0.500	4	Lx
25	TL	0	76.5	321.25	1.285	4	preheat
26	IRSL	50	76.5	100.00	0.500	4	Tx
27	IRSL2	290	76.5	200.00	0.500	4	Tx
28	IRSL3	325	76.5	99.96	0.238	4	bleaching
29	TL	0	459.0	321.25	1.285	5	preheat
30	IRSL	50	459.0	100.00	0.500	5	Lx
31	IRSL2	290	459.0	200.00	0.500	5	Lx
32	TL	0	76.5	321.25	1.285	5	preheat
33	IRSL	50	76.5	100.00	0.500	5	Tx
34	IRSL2	290	76.5	200.00	0.500	5	Tx
35	IRSL3	325	76.5	99.96	0.238	5	bleaching
36	TL	0	612.0	321.25	1.285	6	preheat
37	IRSL	50	612.0	100.00	0.500	6	Lx
38	IRSL2	290	612.0	200.00	0.500	6	Lx
39	TL	0	76.5	321.25	1.285	6	preheat
40	IRSL	50	76.5	100.00	0.500	6	Tx
41	IRSL2	290	76.5	200.00	0.500	6	Tx
42	IRSL3	325	76.5	99.96	0.238	6	bleaching
43	TL	0	153.0	321.25	1.285	7	preheat
44	IRSL	50	153.0	100.00	0.500	7	Lx
45	IRSL2	290	153.0	200.00	0.500	7	Lx
46	TL	0	76.5	321.25	1.285	7	preheat
47	IRSL	50	76.5	100.00	0.500	7	Tx
48	IRSL2	290	76.5	200.00	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:



Similar as for sample BU19-LU 3, the IRSL at 50°C measured equivalent doses are much lower than the pIR-IRSL measured, as already observed in preliminary tests in February 2022. This 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, as in the plot examples can be seen, the IRSL signal at 50°C lead to about stable De values for all aliquots. Which leads to the conclusion, that the signals are probably not affected by effects other than fading, which can not be detected by De(t) plots.

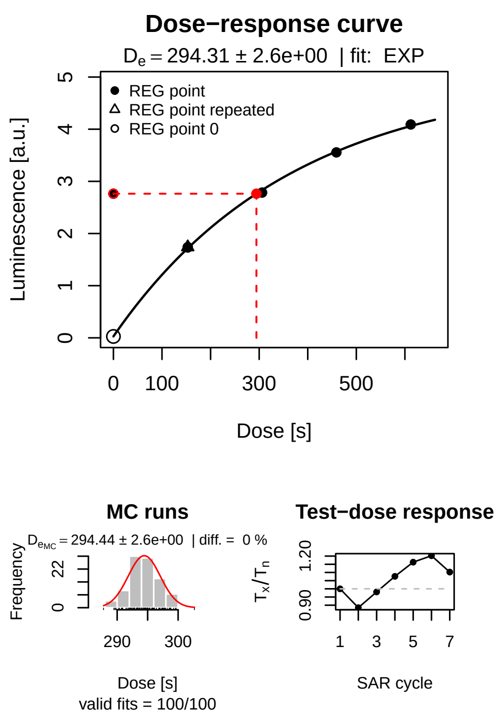
The pIR-IRSL De(t) plots show an De increase of around 10 % during the beginning of the stimulation for almost all aliquots, like it can be seen in the plots above. This points towards a potential systematic error in the results, which however will not be further investigated with this data set.

3.2 De calculation

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. We select the first three channels (1.5 sec) as signal integral and the last 100 channels (50 sec) as background signal. These settings are arbitrarily chosen but in accordance to the De(t) plot.

Below, the resulting signal-dose growth curve for the first aliquot is shown.

```
[plot_GrowthCurve()] Fit: EXP (interpolation) | De = 294.31 | D01 = 370.23
```



The results of all aliquots in the table below include the dose rate error, which increases the De error values a bit.

Table 4: Equivalent doses for pIRIR290 measurements

#	De [Gy]	De error [Gy]	D01 [Gy]	D01 error [Gy]	Rejection criteria
1	294.31	4.34	370.23	4.37	OK
2	274.55	5.37	493.33	15.87	OK
3	258.05	4.33	320.84	4.64	OK
4	256.18	4.19	428.24	9.41	OK
5	352.88	5.02	558.68	7.73	OK
6	271.87	4.73	330.17	5.61	OK
7	269.22	4.22	402.94	6.40	OK
8	270.86	4.20	395.00	6.52	OK
9	265.69	5.06	398.71	8.17	OK
10	379.58	5.84	438.84	6.04	OK
11	333.46	5.90	404.46	8.91	OK
12	279.54	4.78	398.36	7.30	OK
13	337.74	5.17	408.46	5.99	OK
14	266.31	4.09	381.69	5.45	OK
15	309.13	5.24	382.20	6.42	OK
16	277.71	5.23	373.00	6.57	OK
17	301.30	4.80	424.23	6.33	OK
18	247.18	3.90	387.09	5.71	OK
19	307.04	4.81	397.38	5.74	OK
20	265.41	4.64	359.98	5.83	OK
21	243.59	4.36	347.35	5.46	OK
22	304.57	5.02	399.63	7.52	OK
23	300.83	4.66	405.32	5.61	OK
24	362.27	5.76	406.61	5.05	OK

3.3 Rejection criteria

All measurements passed the rejection criteria

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

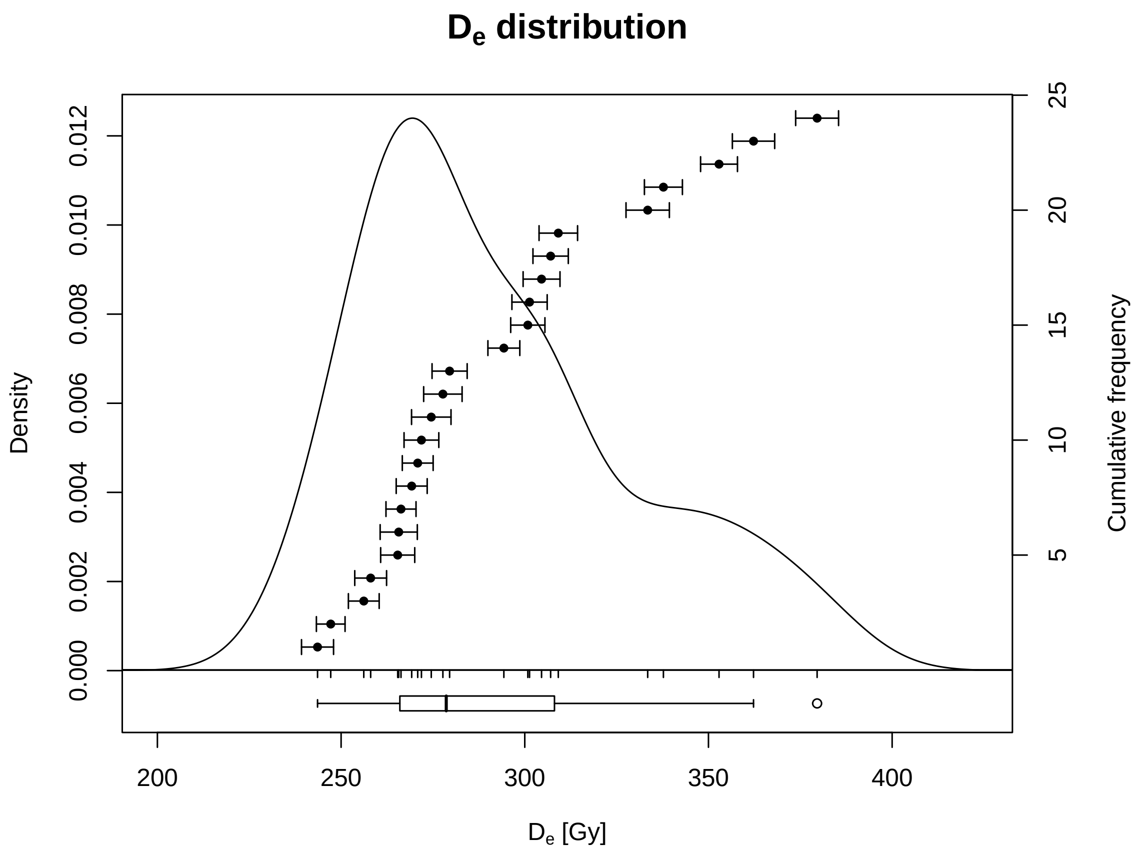
#	Criterium	Threshold
A	Recycling ratio (R5/R1)	0.100
B	Recuperation rate 1	0.100
C	Testdose error	0.100
D	Palaeodose error	0.100
E	De > max. dose point	612.023

#	A	B	C	D	E
1	1.004	0.010	0.004	0.007	294.313
2	1.025	0.010	0.009	0.015	274.555
3	1.031	0.014	0.005	0.011	258.052
4	1.016	0.011	0.006	0.010	256.176
5	1.008	0.007	0.003	0.006	352.882
6	1.001	0.012	0.006	0.012	271.874
7	1.021	0.011	0.005	0.009	269.217
8	1.020	0.007	0.004	0.008	270.856
9	1.035	0.013	0.007	0.014	265.690
10	1.034	0.007	0.004	0.008	379.581
11	0.981	0.012	0.006	0.012	333.462
12	1.011	0.008	0.005	0.011	279.539
13	1.032	0.012	0.003	0.008	337.740
14	1.026	0.009	0.005	0.008	266.307
15	1.016	0.013	0.005	0.011	309.134
16	0.987	0.013	0.007	0.014	277.712
17	1.044	0.010	0.004	0.009	301.302
18	1.037	0.012	0.005	0.009	247.179
19	1.045	0.011	0.005	0.009	307.041
20	1.020	0.009	0.006	0.012	265.408
21	1.018	0.008	0.006	0.012	243.592
22	1.024	0.008	0.006	0.010	304.571
23	1.033	0.010	0.004	0.008	300.825
24	1.002	0.008	0.004	0.009	362.273

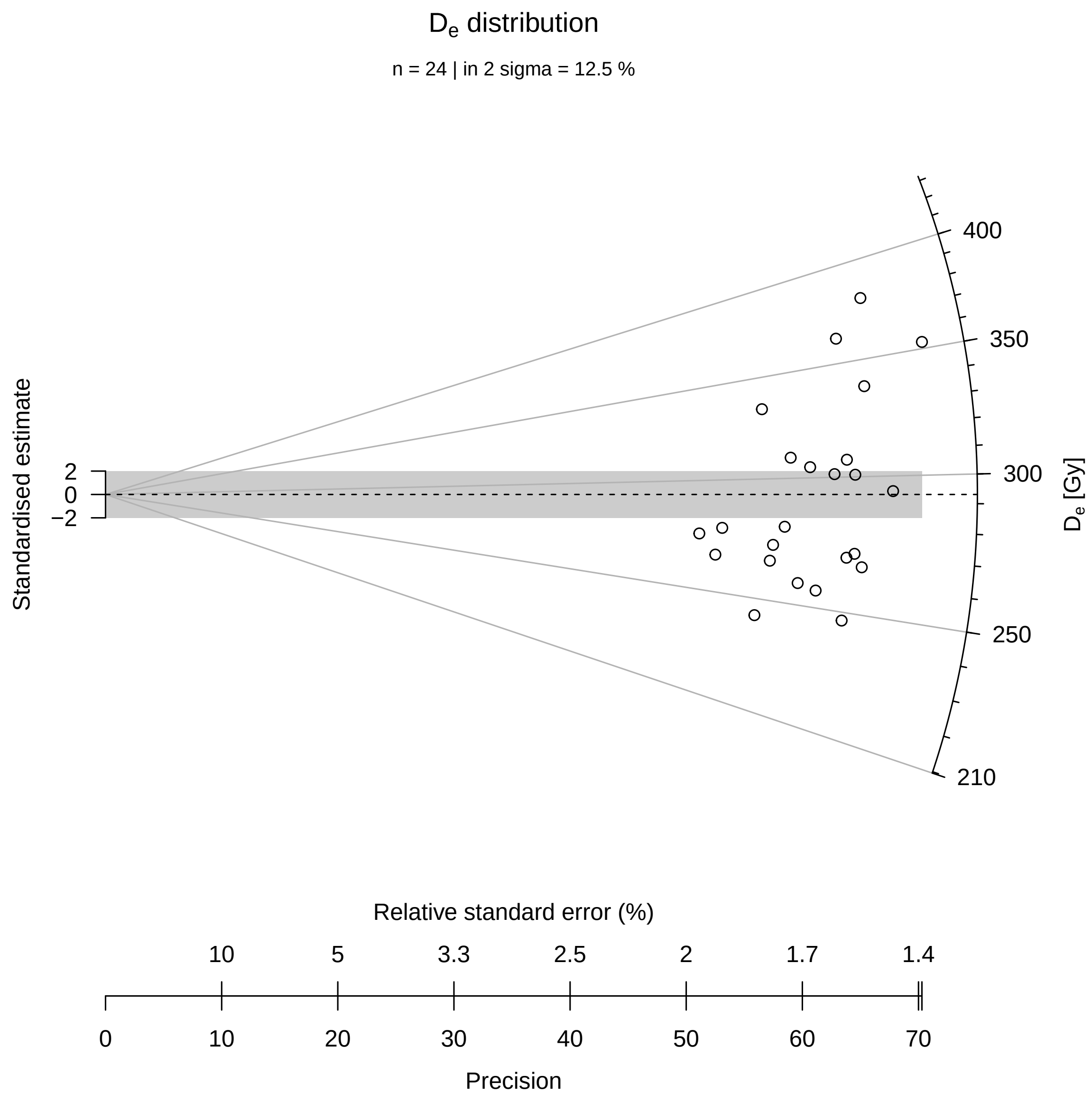
4 Dose distribution

4.1 Distribution plots

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




```
[1] "Attention, small standardised estimate scatter. Toggle off y.ticks?"
```



D_e distribution
n = 24 | in 2 sigma = 12.5 %
Standardised estimate
2
0
−2
400
350
300
250
210
D_e [Gy]
Relative standard error (%)
10
5
3.3
2.5
2
1.7
1.4
0
10
20
30
40
50
60
70
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:        290.79
 abs. SE:                  7.11
 rel. SE [%]:              2.45
----------- overdispersion -----------
 abs. OD:                  34.52
 abs. SE:                  5.08
 OD [%]:                   11.87
 SE [%]:                   1.75
-------------------------------------
```

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.

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.

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. https://doi.org/10.1016/j.radmeas.2008.06.002