Appendix A: Additional information on the luminescence dating

Luminescence protocols and settings

Sampling in the field

The pits were dug with a shovel, and excess water was removed with plastic buckets. The location and elevation of the pits were recorded using Real Time Kinetic GPS. The soil was described the same way as the other boreholes. The luminescence samples were taken using PVC tubes with a 5cm diameter and a length of approximately 20 cm. A piece of styrofoam was placed on 1 side of the tube. The tube was hammered horizontally in the soil with the styrofoam part first, using rubber hammers, such that the styrofoam was pushed to the other side. The depths of the luminescence samples were recorded using a measurement tape. The luminescence samples were dug out, after which the ends were taped, to prevent exposure to light.

Lab analysis

Quartz Single Aliquot Regenerative (Murray & Wintle, 2003) was used for OSL-samples NCL2223043 - NCL2223048 and for samples NCL2223045-NLC2223047 an additional single grain pIRIR175 feldspar measurement (Reimann et al., 2012) was used. The latter three samples originate from a mixed horizon and therefore the variation of the equivalent doses of individual grains might result in an overestimation of the age. In addition, the variation could provide insight into the formation of this horizon. The samples were analysed at Netherlands Centre for Luminescence dating (NCL) in Wageningen. Light exposure to the grains was minimised using subdued amber light. Each sample was split in two, one part was used to measure the dose rate, and the other part was used to measure the equivalent dose.

Dose rate determination

The outer 3 cm of sediment was removed from the tubes and used to determine the dose rate. This part of the sample contains grains which are exposed to light during the sampling. This material was also used to determine the water content and the organic matter content. This was done by placing them in the oven overnight at 105 $\circ$C and 550 $\circ$C respectively. A scale was used to record the mass loss. Afterwards, the samples were mixed with wax and shaped into pucks. The pucks were sent to RIKILT (Wageningen), where the dose rate was derived from the activity of 40-K and parts of the uranium and thorium decay chains. The following factors were also included in the dose rate calculations: an internal alpha dose of 0.010 ± 0.005 Gy/ka after Vandenberghe et al.(2008), cosmogenic contributions after Prescott & Hutton (1994), grain-size attenuation after Mejdahl (1979) and attenuation by water content and organic material after Aitken (1998).

Equivalent dose determination

The equivalent dose was determined with collected material not exposed to light. This material was sieved to obtain the material with grain sizes 212 µm till 250 µm. Next, the calcareous material was removed using 10% HCl for 40 minutes, organic material was removed using H_2O_2 for over 4 days. The magnetic material was removed, using a Frantz LB1. The non-magnetic material was separated based on density (ρ = 2.58 g/cm3). This resulted in a feldspar (lighter) and a quartz fraction. The Quartz grains were treated with HF to etch the quartz grains (10 minutes with 10% HF and 40 minutes with 40% HF). The quartz grains were afterwards treated with HCl to remove any remaining HF.

Multigrain quartz OSL

The quartz grains were analysed using the SAR procedure with a blue (470nm) LED at 125 ◦C (Murray & Wintle, 2003). For each sample, the equivalent doses of 48 aliquots with a 2mm diameter were measured.

Quartz SAR sequence

Step	Action
1	Natural dose or beta dose
2	10s preheat to 200°C
3	20s blue stimulation at 125°C
4	30 s Beta test dose
5	Cutheat to 200°C
6	20s blue stimulation at 125°C
7	40s blue bleach at 210°C
8	Repeat step 1-7 for beta dose for 50 s, 100 s, 200 s, 400 s, 0 s, 50 s, 50 s

In addition, a thermal transfer test was applied to the grains in line with (Truelsen & Wallinga, 2003), this was used to select suitable preheat temperatures. The samples were heated in steps of 20 ◦C, ranging from 160 to 300 ◦C, before the luminescence signal was measured. Finally, a dose recovery test was performed. During a dose recovery test, the grains are bleached and a known dose is given. Afterwards, the luminescence signal is measured and validates whether the measured dose corresponds with the given dose. A Risoe TL/OSL reader equipped with U-340 detection filters and a Strontium-90 beta source was used for all the quartz analyses.

Single grain feldspar

A Risoe TL/OSL reader equipped with an 830 nm IR laser, a LOT/ORIEL D410 interference filter and a strontium beta source was used for all the feldspar analyses. The grains are expected to be less than 1000 years old as they are part of a plaggen layer. Therefore, the equivalent dose of the feldspars was determined using the single grain pIRIR protocol at 175 ◦C (Reimann et al., 2012). Three 100-grain discs were analysed for samples NCL-2223046 and NCL-2223047. For sample NCL-2223045 the number of grains with a luminescence signal was lower, in total six 100-grain discs were analysed. All three samples originate from the same mixed A-horizon.

Feldspar single grain pIRIR175 sequence

Step	Action
1	Natural dose (or Beta dose)
2	50 s preheat to 200 °C
3	2 s single-grain IR laser stimulation at 50 °C
4	2 s single-grain IR laser stimulation at 175 °C
5	300 s IR stimulation at 175 °C
6	50 s Beta test dose
7	50 s cutheat to 200 °C
8	2 s single-grain IR laser stimulation at 50 °C
9	2 s single-grain IR laser stimulation at 175 °C
10	300 s IR stimulation at 200 °C
11	Repeat step 1-10 for beta dose for 100 s, 200 s, 400 s, 800 s, 0 s, 100 s

A dose recovery test was applied to samples NCL-2223045 and NCL-2223047. The outcome of those dose recovery tests is considered representative of NCL-2223046 as the sample is located in between the other two samples. A small portion of the grains was bleached on an aluminium tray for a minimum of 48 hours using a solar simulator (Hönle SOL2). The pIRIR signal is not completely removed by bleaching, this remnant is measured. The residual dose was determined on two 100-grain disks for each sample. The bleached grains are given a known laboratory dose, and subsequently, the resulting luminescence signal is measured. The laboratory dose and the measured signal are then compared. The stability of the feldspar pIRIR signal was determined using a fading test. pIRIR were measured on three aliquots per sample, with a delay time of 4000 s. Here, pIRIR responses are measured on two aliquots per sample, with different delay times in between irradiation dose and post-IR IRSL measurement. The results were fitted to obtain the g-value, which is a measure of the decay of the signal.

Soil description of pit N100 sampled for luminescence dating

Depth (cm)	horizon	texture	grain size um	sorting	Colour	remarks
0-20	Ap	slight loamy sand	90-125	poor	10YR3/1	contains brick particles, charcoal, and no Fe
20-45	Apg	slight loamy sand	90-125	poor	10YR3/1	slightly compacted, contains brick particles, charcoal, and some Fe
45-65	Apg2	slight loamy sand	63-90	moderate	10YR3/2	contains brick particles, charcoal, and much Fe
65-70	Aтg	slight loamy sand	63-90	poor	10YR2/1	contains light sand stains, brick particles, charcoal, and much Fe
70-100	Abg	moderately loamy sand	63-90	moderate	10YR2/1	contains brick particles, Fe

						concretions and less charcoal than Aapg2
> 100	C	slight loamy sand	250-355	poor	10YR6/3	contains plant remains (fluvial reworked sand)

Figure A.1: A picture of pit N100, the pit is 100 cm deep.

Soil description of pit N101 sampled for luminescence dating

Depth (cm)	horizon	texture	grain size um	sorting	Colour	remarks
0-35	Apg1	slight loamy sand	90-125	moderat e	10YR3/1	contains some brick particles, some charcoal, and some Fe

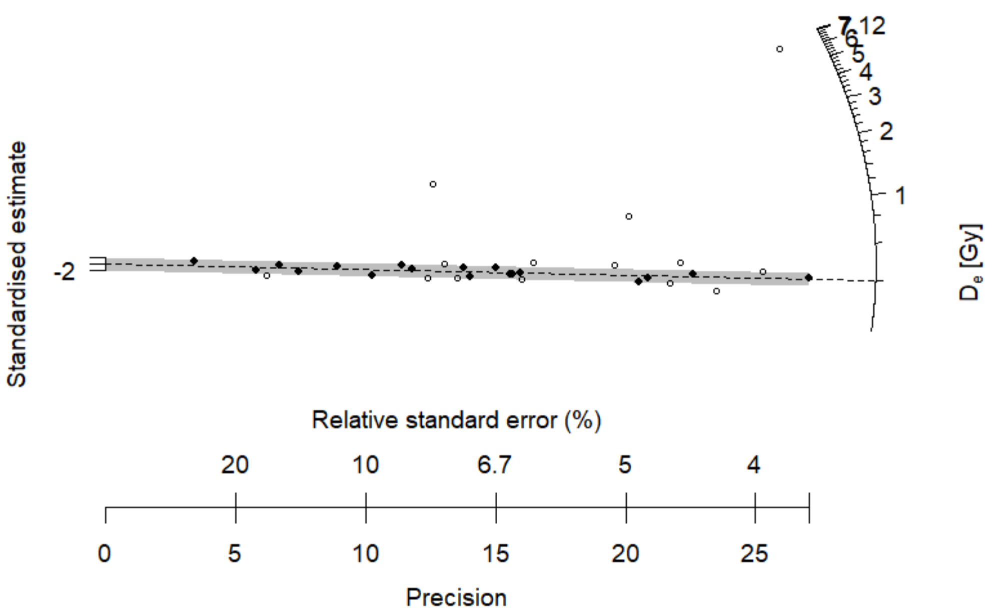
35-49	Apg2	slight loamy sand	90-125	poor	10YR3/1	contains some brick particles, much charcoal, and some Fe
49-85	Aтg	slight loamy sand	63-90	poor	10YR3/2	contains some brick particles, light sand inclusions, some charcoal, and some Fe
> 85	Cg	Strongly loamy sand	63-90		10YR7/1	Brabant Loam

Figure A1.2: A picture of pit N101, the pit is 85 cm deep.

OSL quartz dating radial plots

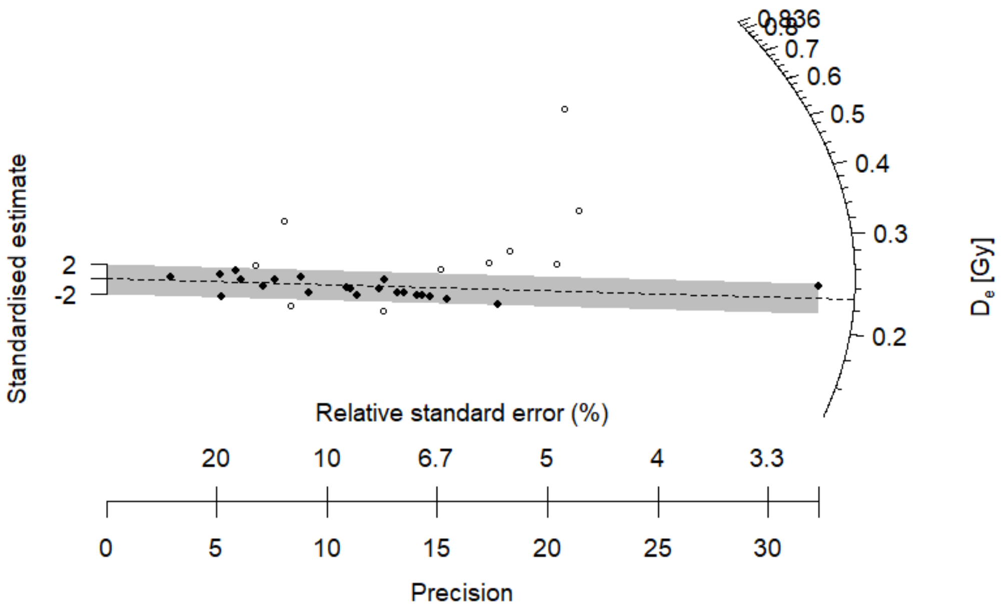
NCL-2223043

BsMAM = 0.48 ± 0.06 Gy



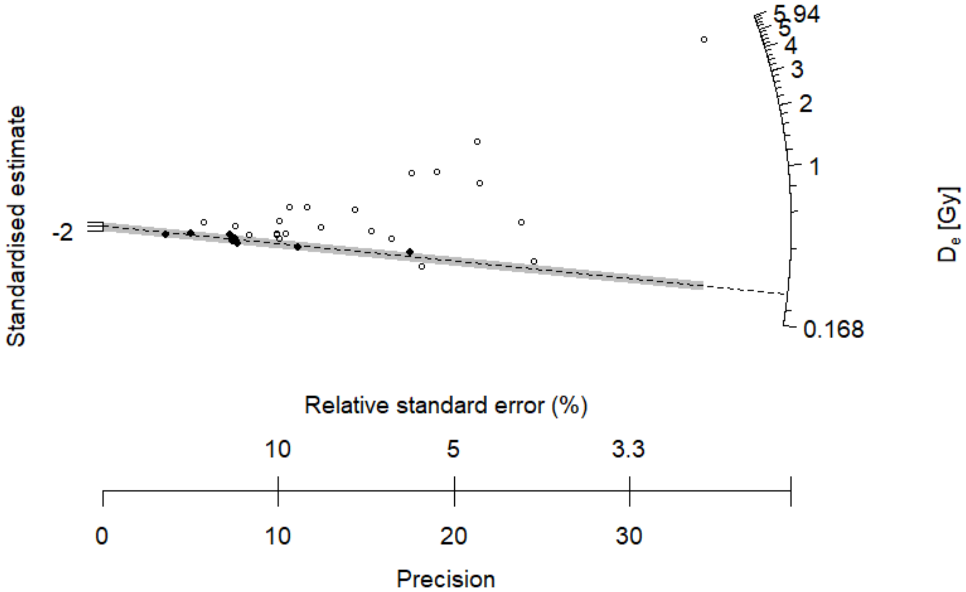
NCL-2223044

BsMAM = 0.23 ± 0.02 Gy



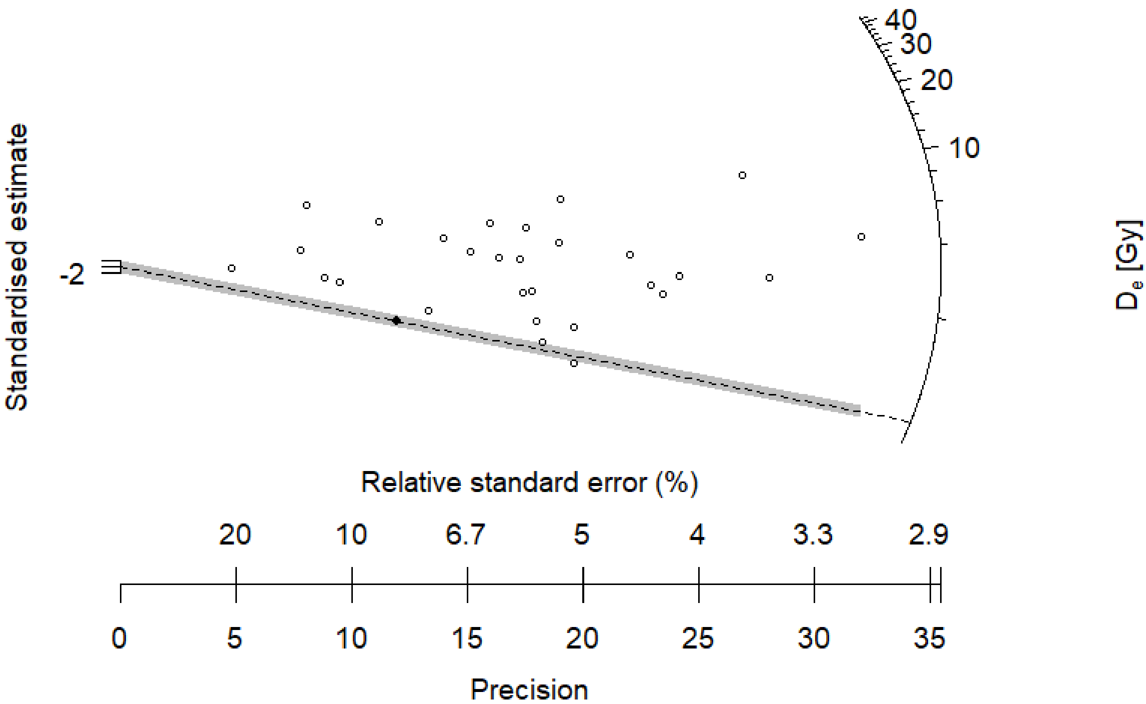
NCL-2223045

BsMAM = 0.24 ± 0.03 Gy



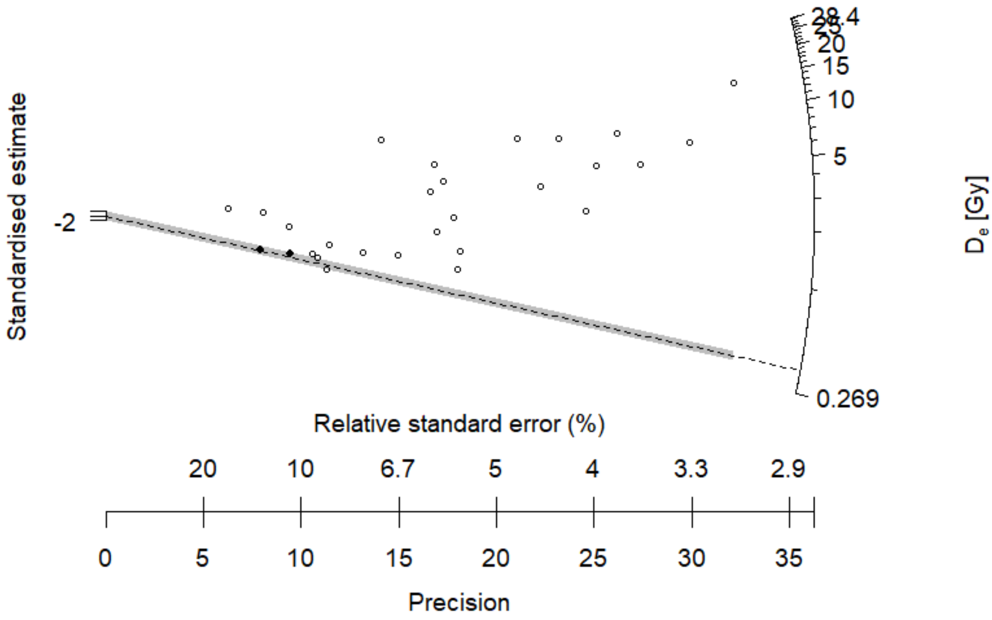
NCL-2223046

BsMAM = 0.73 ± 0.11 Gy



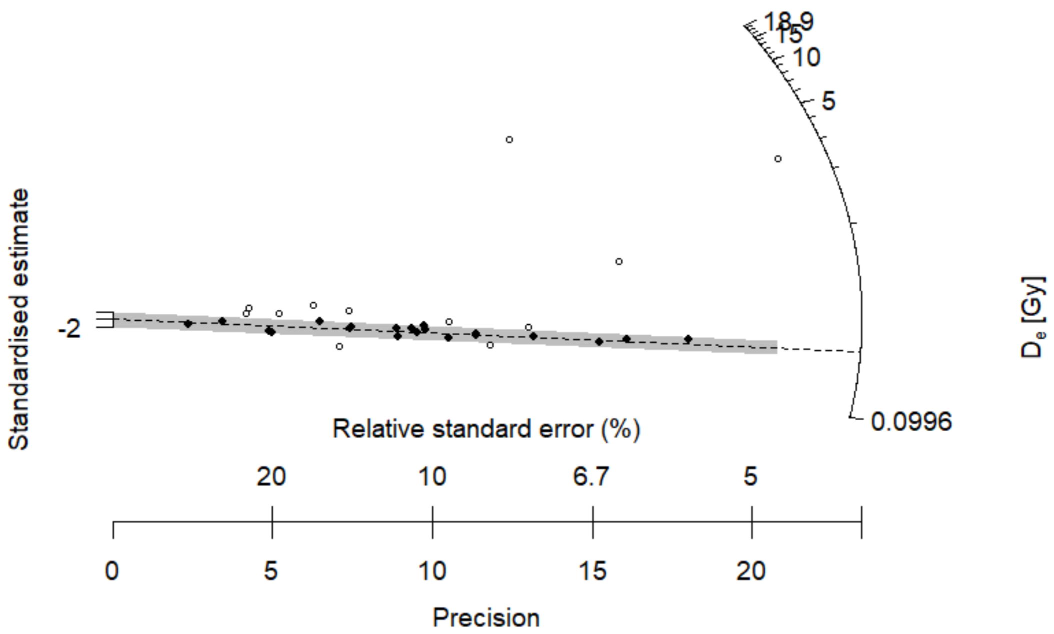
NCL-2223047

BsMAM = 0.36 ± 0.06 Gy



NCL-2223048

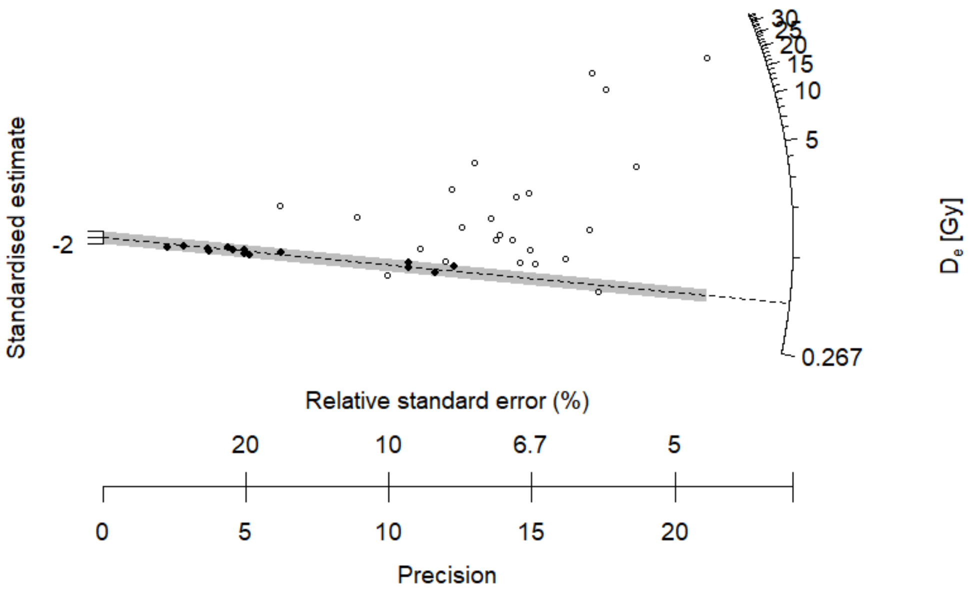
BsMAM = 0.22 ± 0.01 Gy



Feldspar single grain radial plots

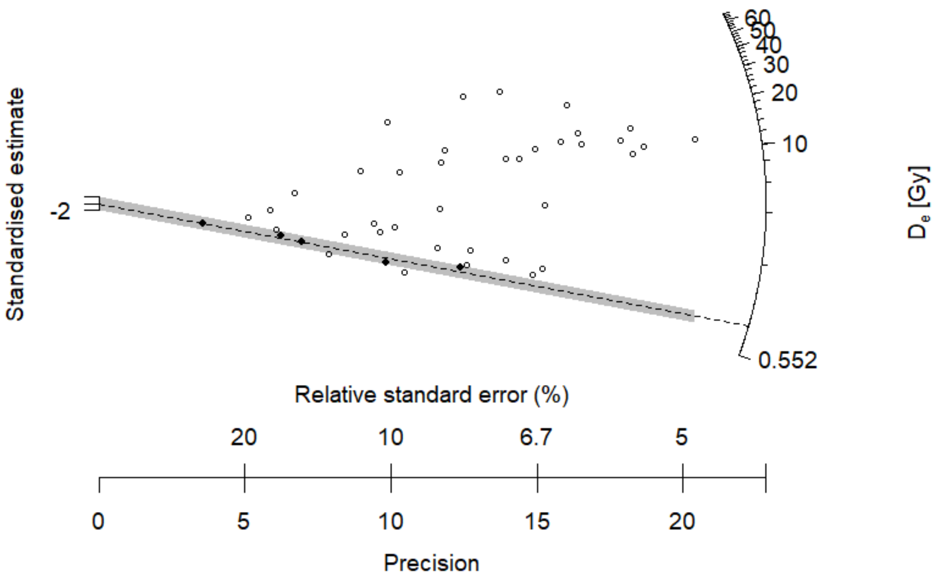
NCL-2223045

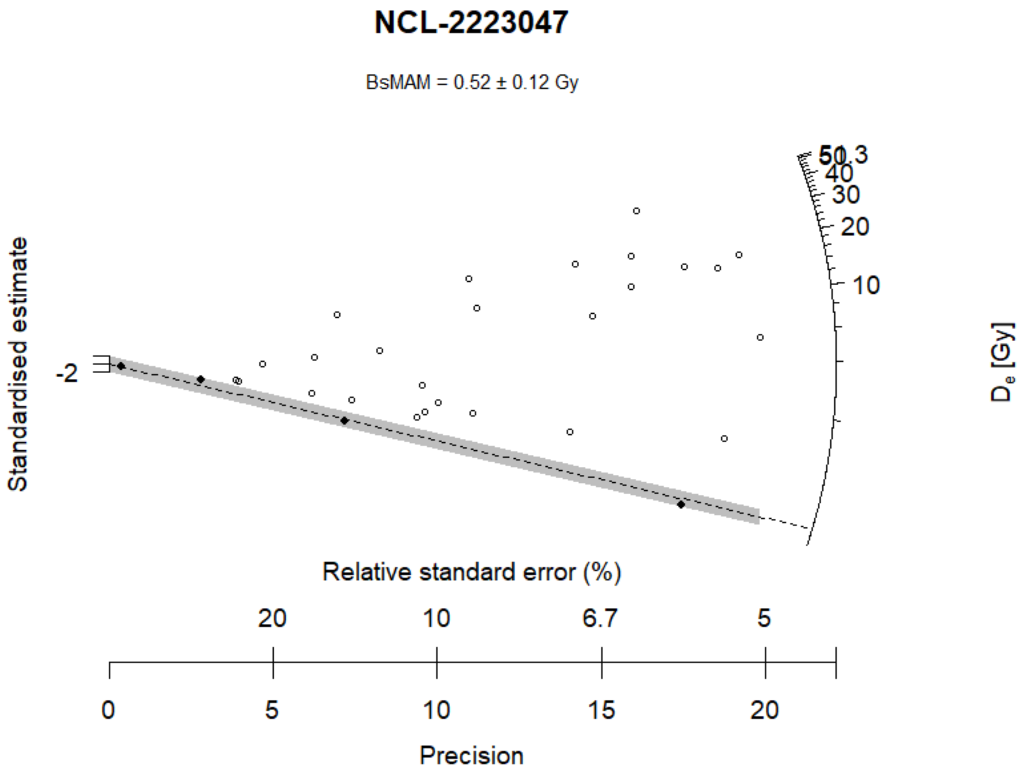
BsMAM = 0.54 ± 0.05 Gy



NCL-2223046

BsMAM = 0.85 ± 0.15 Gy





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