

- Supplementary Data -

Quantification of cross-bleaching during infrared (IR) light stimulation

Ancient TL

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1 Quantification of the cross-bleaching

1.1 Quantification using a multi-exponential function

An R [R Development Core Team, 2012] script was written to analyse the data extracted from the Risø BIN-file. First, all individual L_x curves were plotted alongside with the direct count signal on the measurement position and the corresponding T_x curves. This allows a rough estimation of the cross-bleaching by comparing the individual curves and potential changes of the signal during the measurement (see Sec. 2 this supplement). Secondly, the normalized IRSL (blue OSL) curve after bleach and dose cycles was fitted using a multi-exponential function given in equation

$$I(t) = \sum_{i=1}^n I_0_i \exp(-\lambda_i t) \quad (1)$$

where I_0 is the initial signal intensity, λ is the decay constant, t the illumination time. For the fitting, n (maximal number of components) was allowed to vary between 1 and 3 and was set to the value where the best fit of the sum curve was obtained. From the fitted curve, the halftime ($T_{0.5_{signal}}$), referring to the time it takes to halve the signal of the sample, was calculated by iteration. The same equation was applied for the fitting of the corresponding normalized L_x/T_x values of the adjacent position against the illumination time on the measurement position and also a halftime of the sum curve ($T_{0.5_{L_x/T_x}}$) was calculated. For calculating the L_x/T_x ratio the first 0.5 s of the shine-down curves were taken. The relative cross-bleaching in percentage is calculated by:

$$CB_{rel.} = \frac{T_{0.5_{signal}}}{T_{0.5_{L_x/T_x}}} \cdot 100 \quad (2)$$

1.2 Quantification using an inverse power law function

For analysis instead of the multi-exponential function for the fitting a modified inverse power law function (e.g. [Bailiff and Poolton, 1991, Baril, 2002]) was used:

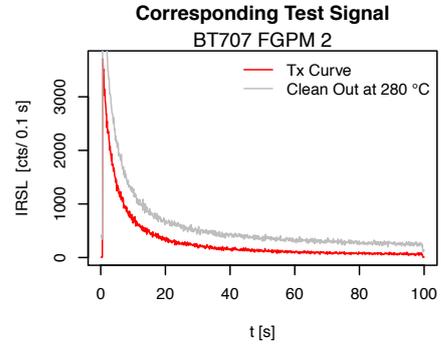
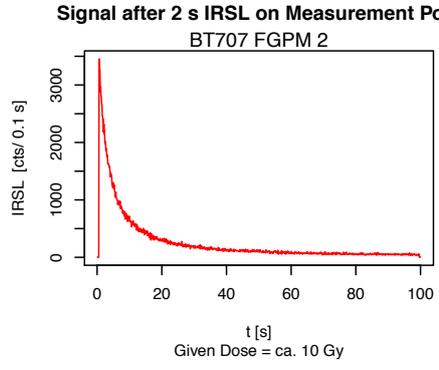
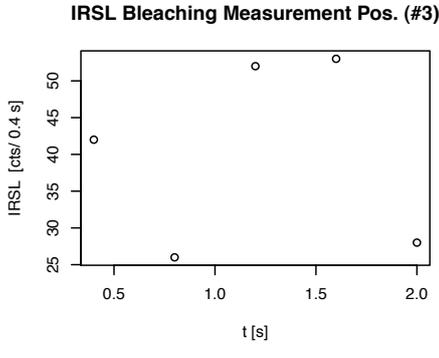
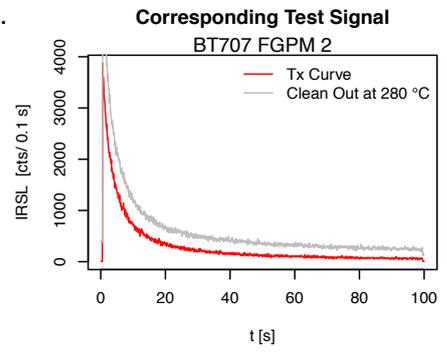
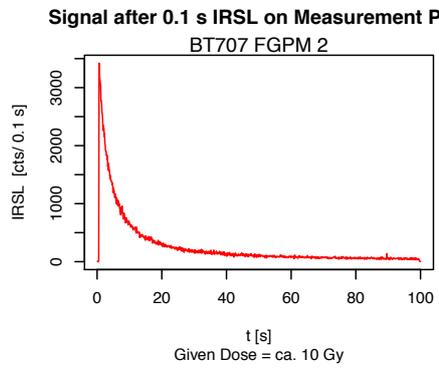
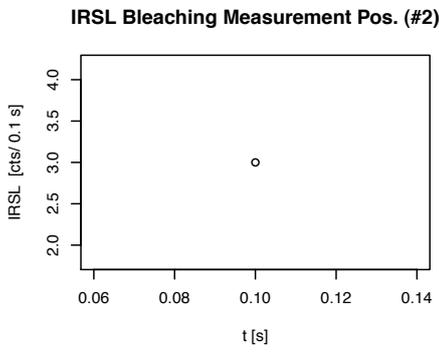
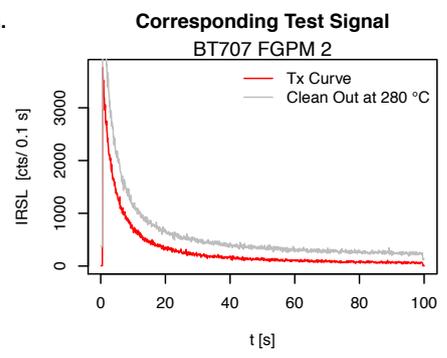
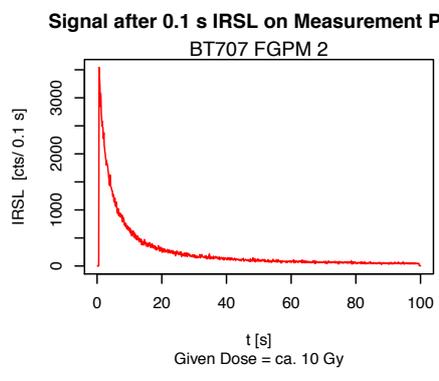
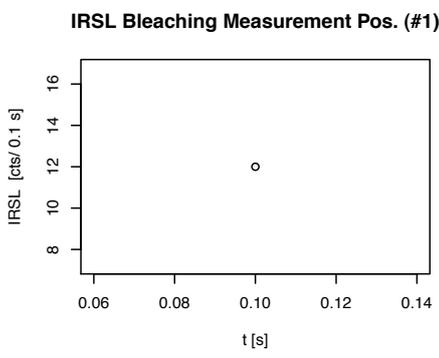
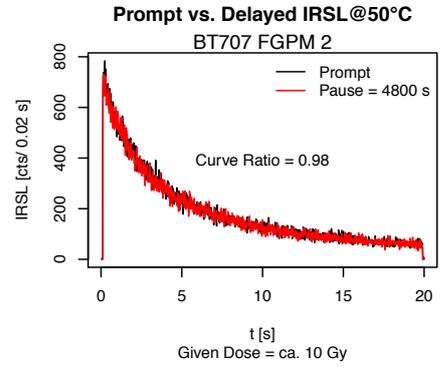
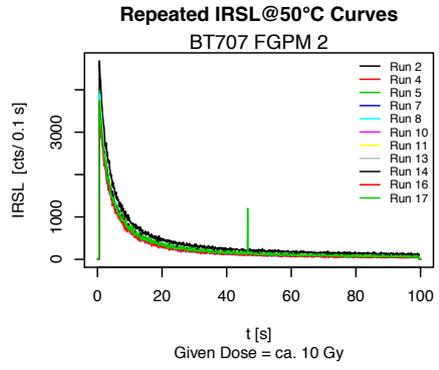
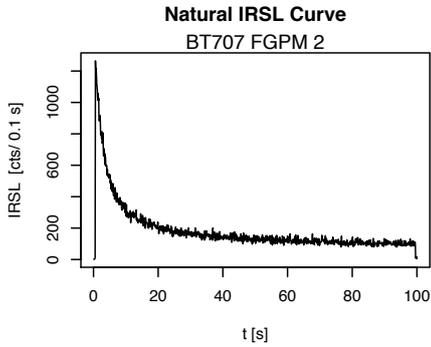
$$I(t)_{Signal Curve} = \frac{1}{(1 + P_2 \cdot t)^{P_3}} \quad (3)$$

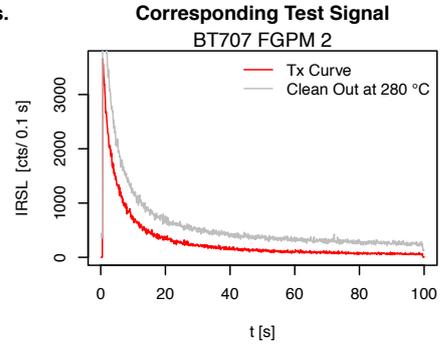
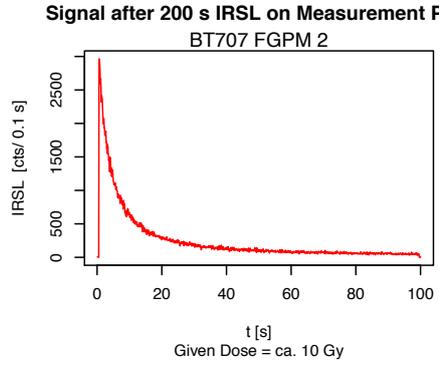
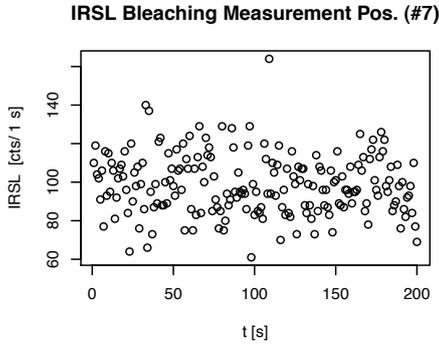
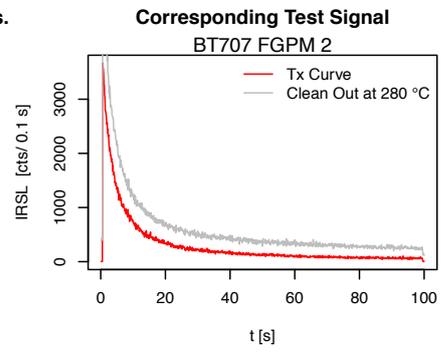
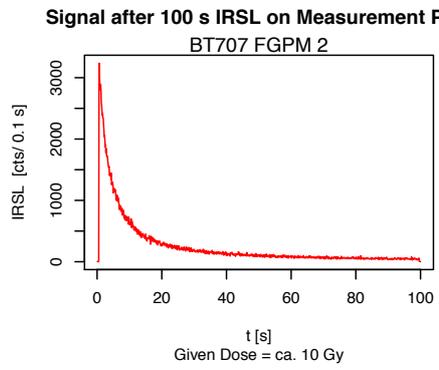
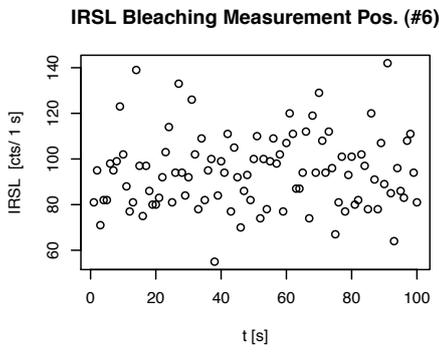
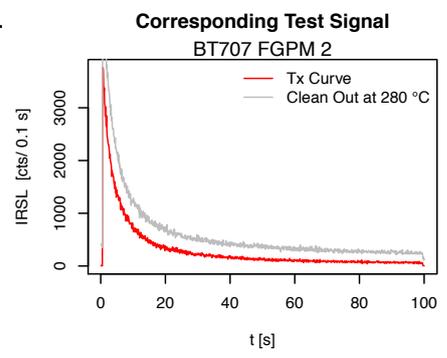
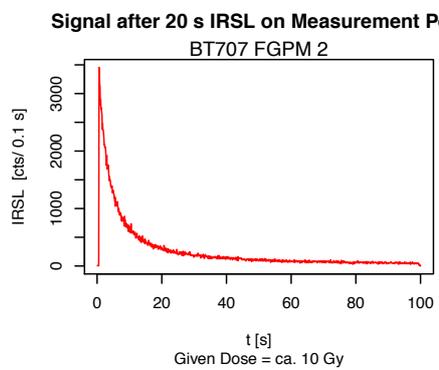
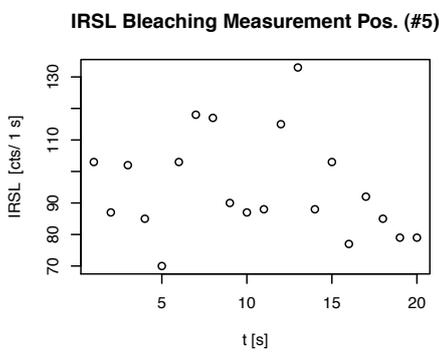
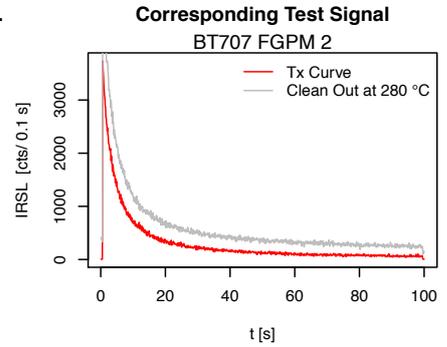
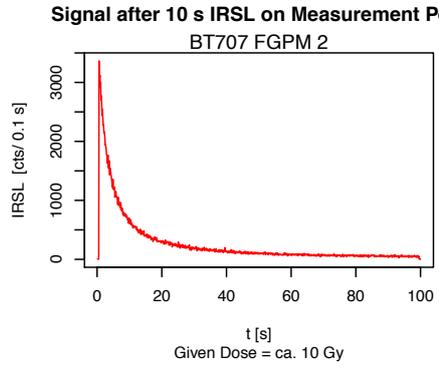
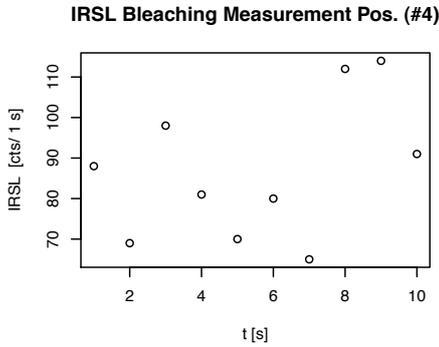
$$I(t)_{L_x/T_x} = \frac{1}{(1 + P_1 \cdot P_2 \cdot t)^{P_3}} \quad (4)$$

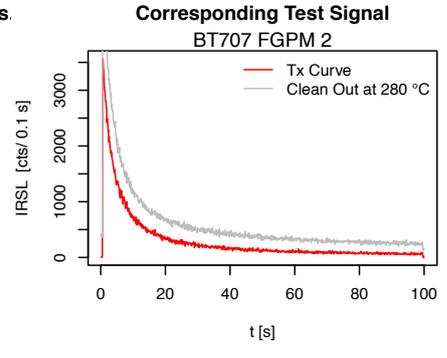
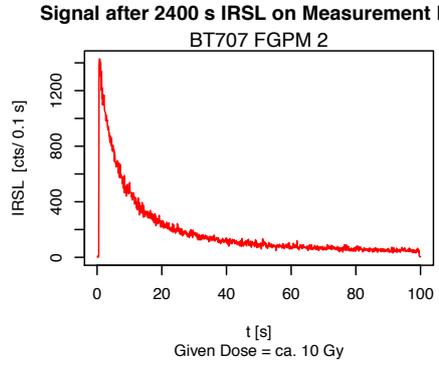
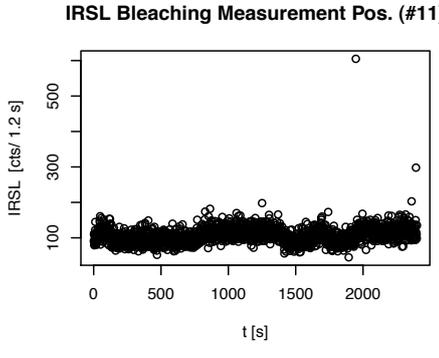
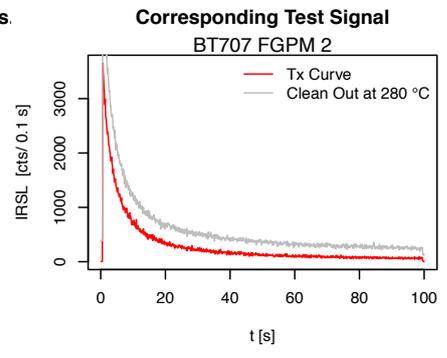
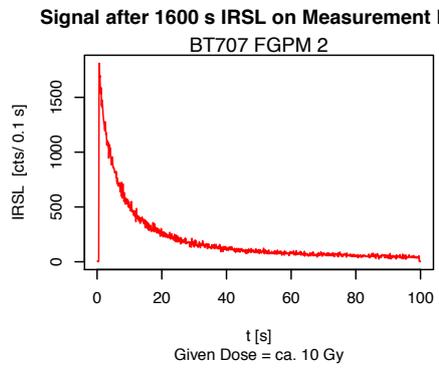
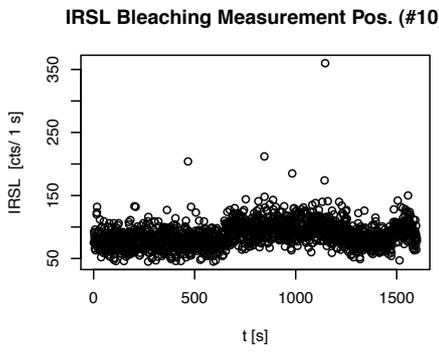
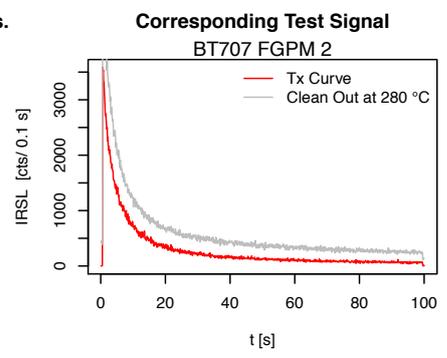
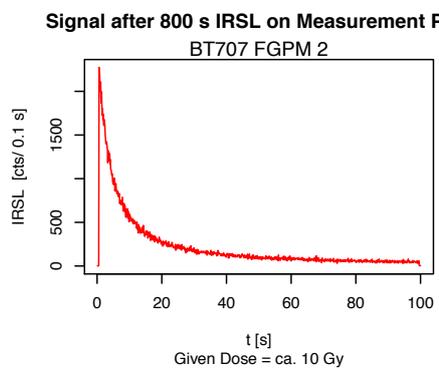
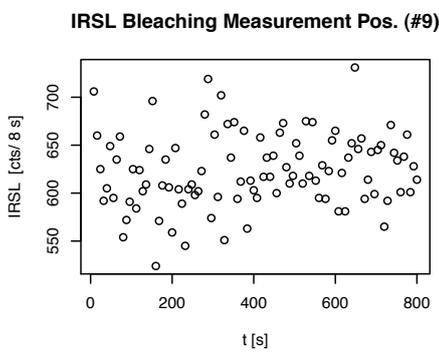
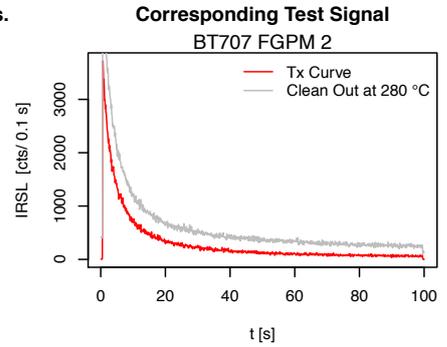
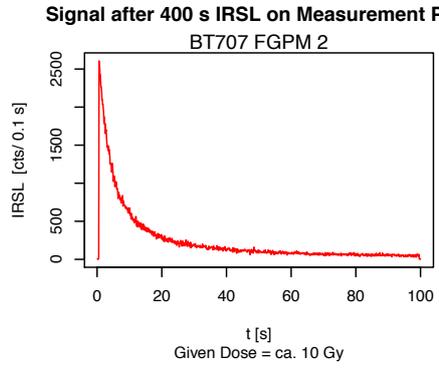
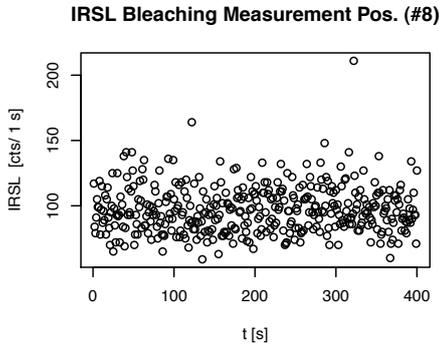
where P_1 , P_2 and P_3 are fitting parameters. The loss in L_x/T_x as percentage of loss caused by direct stimulation is then expressed by the parameter P_1 .

2 Cross-bleaching sequence output

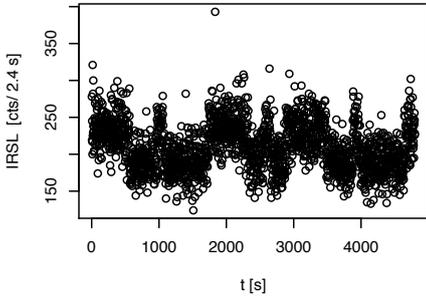
The following four pages show the output of the cross-bleaching sequences for the reader with the highest amount of cross-bleaching (ID 189) using a polymineral fine grain sample.



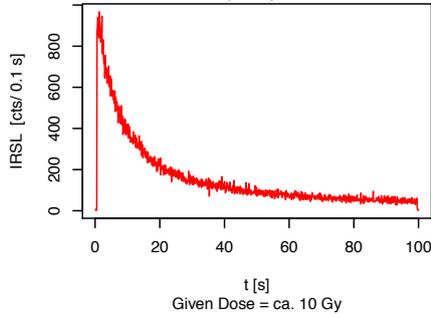




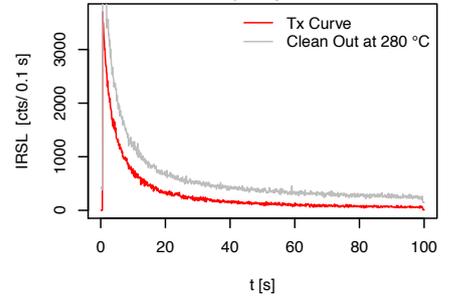
IRSL Bleaching Measurement Pos. (#12)



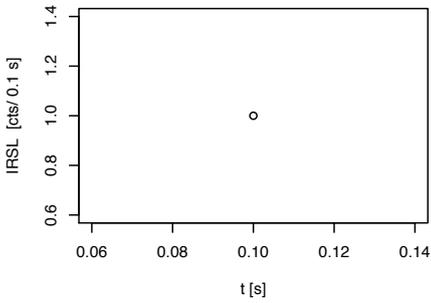
Signal after 4800 s IRSL on Measurement Pos. BT707 FGPM 2



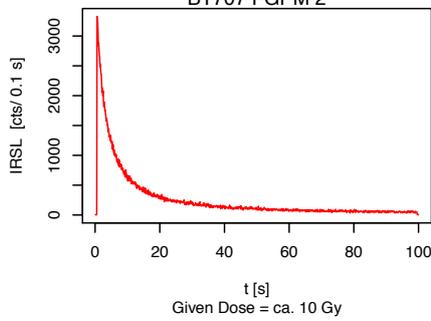
Corresponding Test Signal BT707 FGPM 2



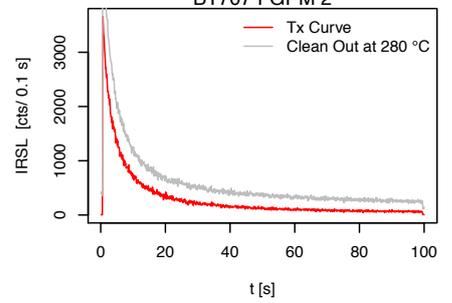
IRSL Bleaching Measurement Pos. (#13)



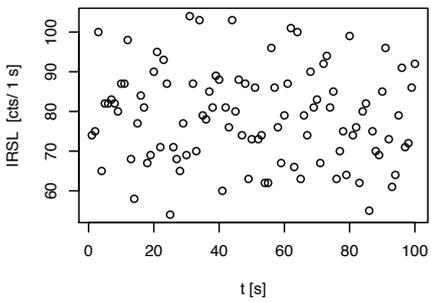
Signal after 0.1 s IRSL on Measurement Pos. BT707 FGPM 2



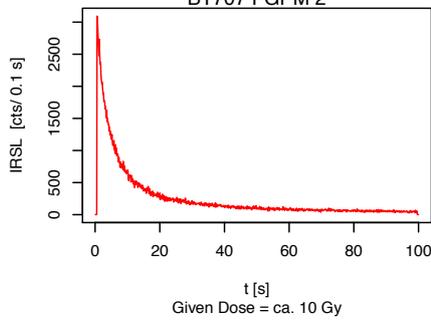
Corresponding Test Signal BT707 FGPM 2



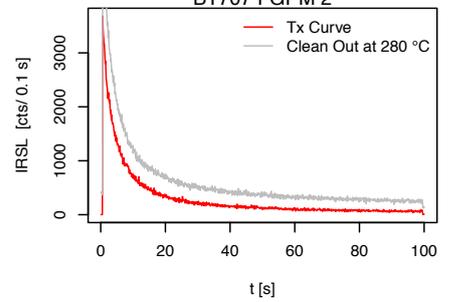
IRSL Bleaching Measurement Pos. (#14)



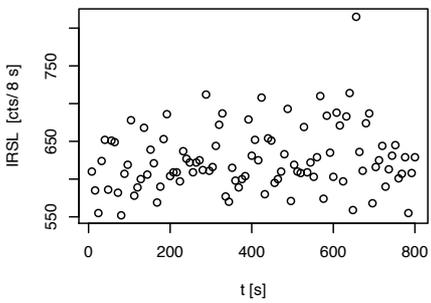
Signal after 100 s IRSL on Measurement Pos. BT707 FGPM 2



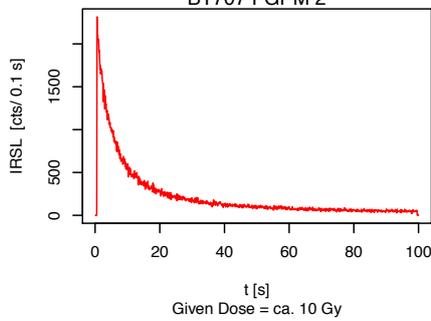
Corresponding Test Signal BT707 FGPM 2



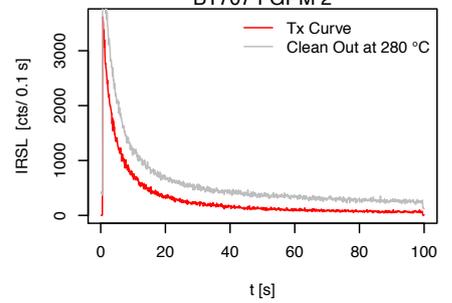
IRSL Bleaching Measurement Pos. (#15)



Signal after 800 s IRSL on Measurement Pos. BT707 FGPM 2



Corresponding Test Signal BT707 FGPM 2



3 Additional figures and tables

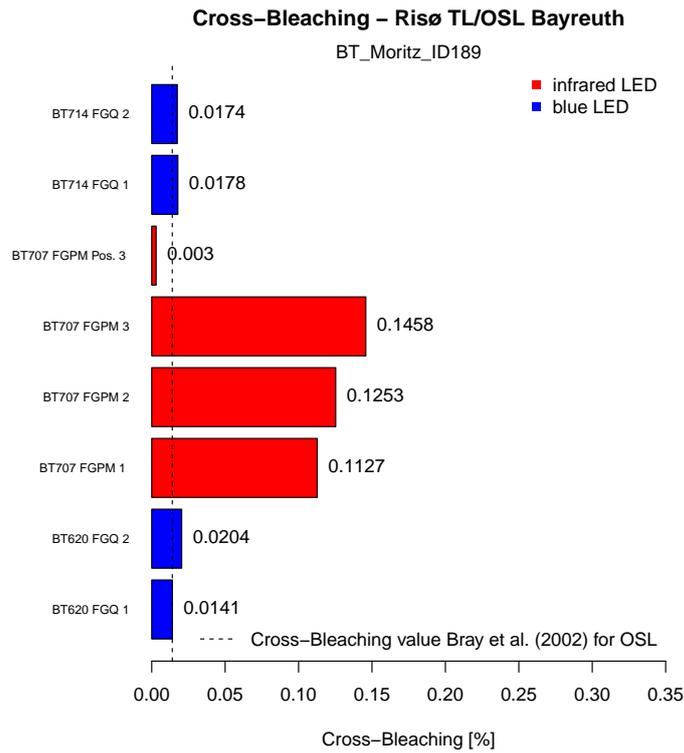


Figure 1: Cross-bleaching results for all measurements carried out on reader ID 189. Three samples were used for the experiments one polymineral fine grain sample (BT707, IR stimulation) and two fine grain quartz samples (BT620 and BT714, blue stimulation). The obtained cross-bleaching values can differ markedly even for one sample. Note: BT707 FGPM Pos. 3 is a measurement on the 2nd adjacent position (position 3 on the sample carousel).

Table 1: Samples used for the cross-bleaching tests

sample id	mineral	grain size [μm]	laboratory	reference
BT620	quartz	4–11	Bayreuth	[Kreutzer et al., 2012]
BT707	polymineral	4–11	Bayreuth	[Meszner et al., 2012]
BT711	polymineral	4–11	Bayreuth	[Meszner et al., 2012]
BT714	polymineral	4–11	Bayreuth	[Meszner et al., 2012]
HDS-493, HBII 78 - 85 cm	polymineral	4–11	Heidelberg	[Kadereit, 2002]
HDS-499, HBII 380 - 387 cm	polymineral	4–11	Heidelberg	[Kadereit, 2002]
Bolivien	quartz	125–212	Heidelberg	(unpublished)
US-C	K-feldspar	100–200	Cologne	[Hülle, 2011]
KG-1	K-feldspar	100–200	Cologne	[Hülle, 2011]
ME-S2	K-feldspar	100–200	Cologne	[Hülle, 2011]
914807	quartz	180–250	NU Risø	(calibration quartz)
970425	K-feldspar	180–250	NU Risø	-

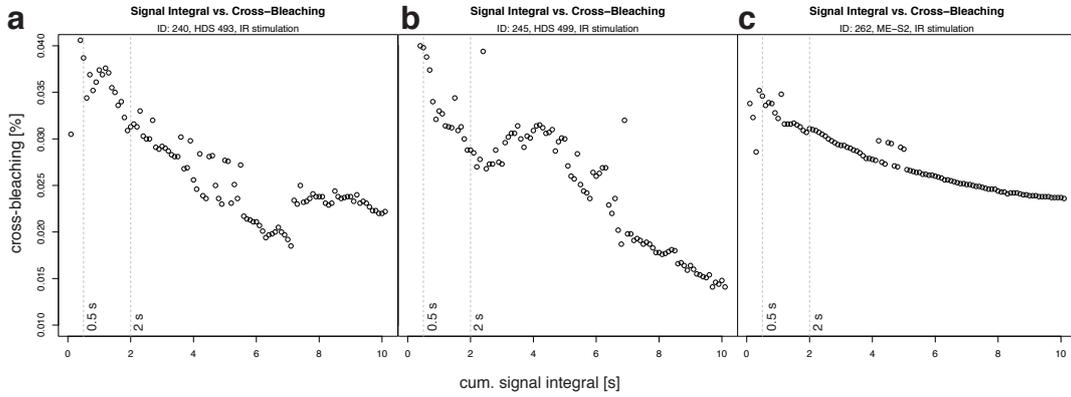


Figure 2: Signal integral vs. cross-bleaching results for IR-LEDs on three different readers (a: ID 240, b: ID 245, c: ID 262) using different samples (a: HDS-493, b: HDS-499, c: ME-S2). The results show that the chosen signal integral seems to be sample dependent and may markedly influence the obtained cross-bleaching value. The dashed lines mark the values for the 0.5 s and the 2 s signal integral.

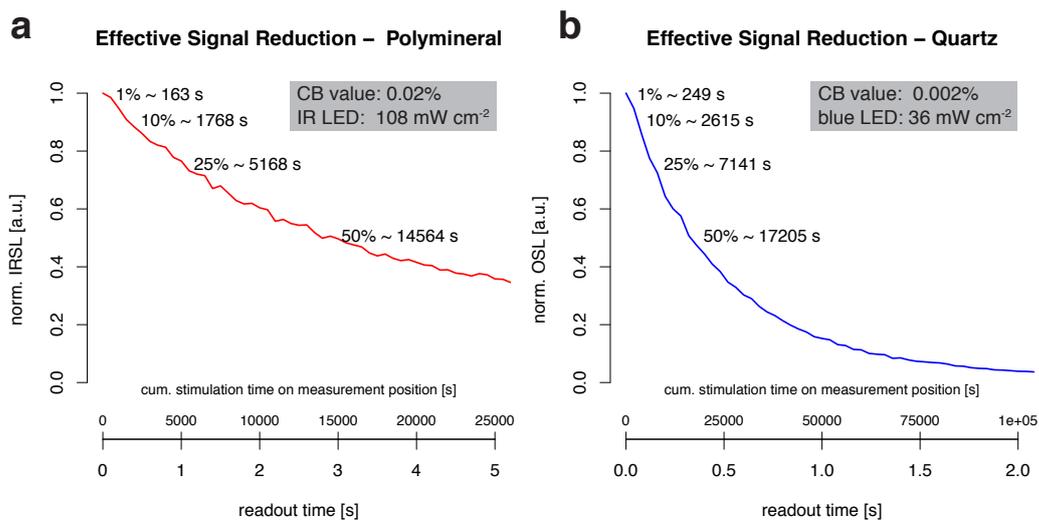


Figure 3: Effective signal reduction for a given cross-bleaching value as function of the cumulative stimulation on the measurement position (upper x-axis). The first seconds of a normalized shine-down curves of polymineral fine grain sample (BT711, a) and fine grain quartz sample (BT711, b) are shown. An artificial cross-bleaching value of 0.02 % was assumed for IR stimulation (a) and 0.002 % for blue stimulation (b). Note that the plotted stimulation times on the measurement position in the plot depend on the stimulation power density. That means for a higher photon flux less stimulation time on the measurement position for the same amount of signal reduction is needed, independent of the (relative) cross-bleaching value.

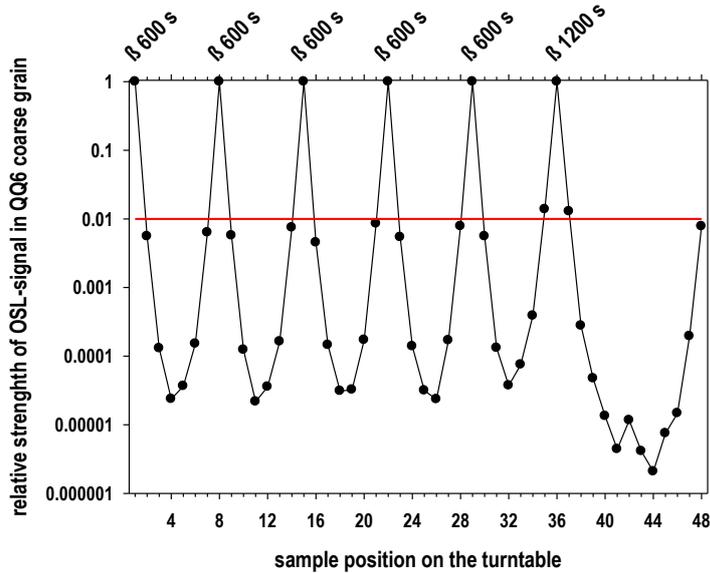


Figure 4: Cross-irradiation test on reader ID 240. Relative OSL-signal of quartz samples on irradiated positions (positions 1, 8, 15, 22 and 29 600 s β -irradiation resp. ca. 56 Gy and position 36 with 120 s resp. ca. 112 Gy) and on not directly irradiated positions. Whereas aliquots on most of the not irradiated sample positions do not show any significant OSL-signal, the aliquots on the sample positions next to the irradiated positions show a relative signal strength of 0.5 % to 1.4 % of the nearest directly irradiated position. The red line denotes a potential 1 % proportion of the relative OSL-signal of the nearest directly irradiated turntable position. Relative OSL-signal means that the OSL-signals of the 47 different aliquots on positions 2–48 were normalized against the aliquot on position 1 in order to correct for varying signal strengths. As pointed out by Regina DeWitt (personal communication) the relatively high values are most likely due to the circumstance that the chosen quartz (QQ6) is probably already in saturation at 56 Gy, and even more so at 112 Gy. Therefore a second measurement was carried out, using 60 s resp. ca. 9.3 Gy as the laboratory dose on six of the 48 turntable positions, which this time were evenly spread on the turntable.

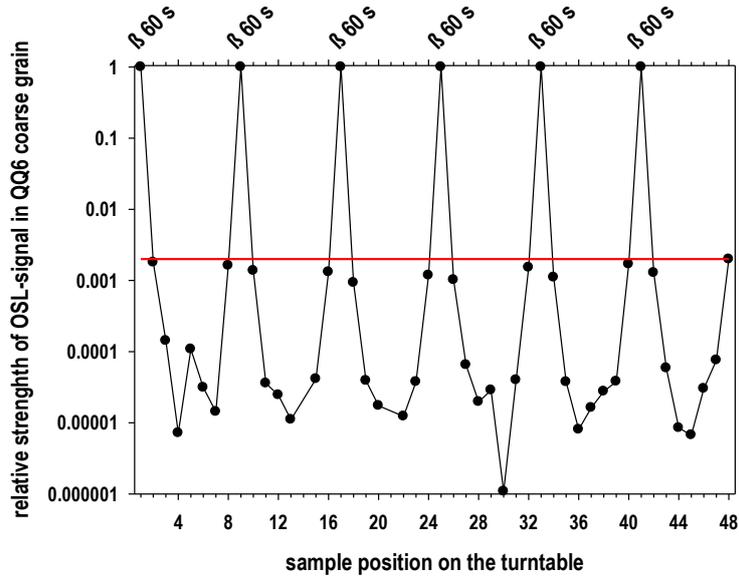


Figure 5: Cross-irradiation test on reader ID 240. Relative OSL-signal of quartz samples on irradiated positions (positions 1, 9, 17, 25, 33 and 41 with 60 s β -irradiation resp. ca. 9.3 Gy) and on not directly irradiated positions. Whereas aliquots on most of the not irradiated sample positions do not show any significant OSL-signal, the aliquots on the sample positions next to the irradiated positions show a relative signal strength of 0.09 % (position 18) to 0.20 % (position 48) of the nearest directly irradiated position. The red line denotes a potential 0.2 % proportion of the relative OSL-signal of the nearest directly irradiated turntable position. Relative OSL-signal means that the OSL-signals of the 47 different aliquots on positions 2–48 were normalized against the aliquot on position 1 in order to correct for varying signal strengths. As also seen in Fig. 4 cross talk is slightly asymmetric with the higher values ca. 0.2 % being on the left side of the irradiated position, the lower values around 0.1% being on the right side. The values meet the order of 0.17% for quartz coarse grain as published by [Bøtter-Jensen et al., 2000] but exceed the values of 0.04 % for α -Al₂O₃ and CaF₂:Mn as quoted by [Kalchgruber, 2002].

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