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

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Abstract

The cross-bleaching behaviour of automated Risø TL/OSL (DA-12, DA-15, DA-20) luminescence readers is investigated. By design, up to 24 or 48 aliquots can be stored on a carousel in a single measurement chamber. Due to this construction, irradiation or illumination on one sample may affect the adjacent position resulting in systematic errors.

Previously reported for blue LEDs, such cross-talk (cross-bleaching/illumination) has never been quantified explicitly for the infrared (IR) LEDs, although they are intensively used in IRSL measurements of e.g. feldspar and polymineral samples. In IRSL measurements of feldspar or polymineral samples it is important to keep the time constant between the (midpoint of the) irradiation and the subsequent read out to avoid the malign effects of anomalous fading in laboratory constructed dose response curves. This may be achieved by running all measurements for equivalent dose estimation on a single sample before moving to a subsequent sample (e.g. by using the “run 1 at a time” option in the Risø sequence editor). However, if the measurement sequence is not designed carefully, then using this option may result in a significant depletion of the natural signal on subsequent samples. Here we investigate the size of this reduction due to cross-bleaching from the IR diodes and quantify the cross-bleaching for 10 different Risø TL/OSL readers produced between 1994 and 2011. We find that cross-bleaching from the IR diodes is worse than from the blue diodes. Using the “run 1 at the time” option can result in significant dose underestimation (1) if the sequence is not split into different sets, or (2) if samples are not placed on every 2nd position. In addition, a newly designed flange for the optical unit of the TL/OSL reader is presented which appears to reduce cross-bleaching significantly.

Keywords: Cross-bleaching, IRSL, polymineral, feldspar, instrumentation, improved luminescence-reader design

Introduction

Dating of sediments using optically stimulated luminescence (OSL) was introduced in 1985 by Huntley et al. (1985) for quartz and using infrared stimulated luminescence (IRSL) for K-feldspar by Hütt et al. (1988). The introduction of the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) enabled a wide range of dating applications in a short time span. Due to the development of automated luminescence dating systems (e.g. Bøtter-Jensen, et al. 2003, Bøtter-Jensen 1997) OSL and IRSL dating results have been produced more and more rapidly.

The commonly used Risø TL/OSL readers accommodate up to 48 individual samples, which are located in the measurement chamber on a sample carousel (Fig. 1). The distance between the centres of neighbouring (adjacent) samples is 17 mm. For older readers with only 24 sample positions this distance is 32 mm. In the standard Risø TL/OSL reader, illumination is achieved using seven clusters of LEDs mounted concentrically in a ring-shaped holder (stimulation head). Each cluster contains seven LEDs and each individual diode is focused at the sample.

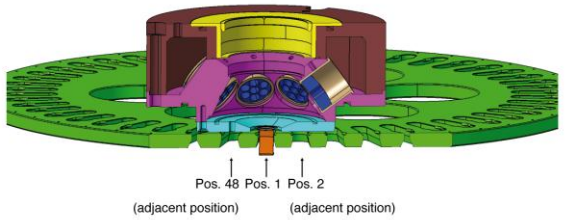

Figure 1: *Technical drawing of the stimulation head and the sample carousel of a Risø TL/OSL reader (DA-15, DA-20).*

The stimulation head normally contains three IR (870 Δ 40 nm) and four blue (470 Δ 30 nm) LED clusters. For illumination the sample is lifted up from the sample carousel into the stimulation head through a circular opening (ø 15.5 mm) in the bottom flange of the stimulation head (see Fig. 1 and Fig. 5). A consequence of the close proximity of the adjacent sample positions and the illumination geometry is that adjacent samples are also optically stimulated during illumination. This is known as cross-bleaching (cross-illumination, optical cross-talk) and has previously been determined to be 0.006% (Bøtter-Jensen et al. 2000) or 0.014% (Bray et al. 2002) for the blue LEDs. Although these cross-bleaching values appear small they may significantly reduce the luminescence signal of samples on adjacent (i.e. subsequent) positions if long illumination times are employed or if all illuminations in the measurement sequence are carried out on a single position before measurements of the adjacent sample (a long total/cumulative stimulation time). Bray et al. (2002) showed that if a sample is illuminated for a total of 1400 s by blue LEDs then the first signal measured from the adjacent sample position may be reduced by ~18% in a standard SAR run. In OSL dating procedures the first signal read out in a SAR sequence is the natural signal of a sample, and hence cross-bleaching will lead to age-underestimation. However, this is generally not an issue for quartz measurements because there is no demand that the time between irradiation and readout is kept constant, and so the illumination time of any one sample is typically ~40 s before the adjacent sample is measured and thus the effect of cross-bleaching is assumed to be negligible.

In contrast, the stimulation time on one position for feldspar measurements is markedly longer (e.g. up to 240 s Kadereit et al., 2010) and many feldspars have been shown to suffer from anomalous fading (e.g. Wintle, 1973; Spooner, 1994). Thus it is important to keep the time elapsed between irradiation and readout fixed. This is easily accomplished using the "run 1 at a time" option[1] in the Risø sequence editor but if care is not taken in the design of the measurement sequence cross-bleaching may be a significant problem.

In this work, we report cross-bleaching values measured for the IR LEDs on 10 different Risø TL/OSL readers produced between 1994 and 2011. The cross-bleaching values obtained for the IR LEDs are compared with values for blue LEDs from the literature and with values determined by our own measurements. We further investigate the effect which cross-bleaching may have on equivalent dose determination if this issue is not kept in mind when designing the measurement sequence. Finally, we show that the new stimulation head flange, developed by Risø, reduces the cross-bleaching significantly.

Experimental design

Instrumentation

Cross-bleaching was investigated for Risø TL/OSL (DA-12, DA-15, DA-20) readers. All readers are equipped with bialkali photomultiplier tubes (EMI 9235Q) and ^{90}Sr/^{90}Y β-sources. The luminescence in the UV/blue band (polymineral and potassium rich feldspar, infrared stimulation) was measured with a 3 mm Chroma D410/30x interference filter (410 Δ 30 nm) or a blue filter pack (390 Δ 60 nm, 4 mm Corning 7–59 in combination with 2 mm Schott BG 39). For the UV band (quartz, blue stimulation) a 7.5 mm Hoya U340 glass filter (290–370 nm) was used.

IRSL measurements were done at 50°C for 100s after a preheat of 250°C for 60 s. In between the various SAR cycles (L_x and T_x) a hotbleach at 280°C for 100 s was used. Measurements of the quartz OSL signal using blue stimulation were carried out at 125°C for 100 s after a preheat of 260°C for 10 s. Both IR and blue stimulated signals reported here have been summed using the initial 0.5 s of the decay curves after subtraction of the last 10 s of the shine-down curve. For the signal curves (direct stimulation, Fig. 2b) only the first 20 s are recorded. Table 2 shows relevant data for the various readers investigated in this work.

Samples

All measurements were carried out on sedimentary samples. To avoid a dependency of the results on a single sample, different natural coarse grain (potassium feldspar, quartz) and fine grain (polymineral, quartz) samples were used. However, the cross-bleaching results seem not to be markedly affected by the chosen samples. A list of all investigated samples with their references is given in the supplementary information (Table S1).

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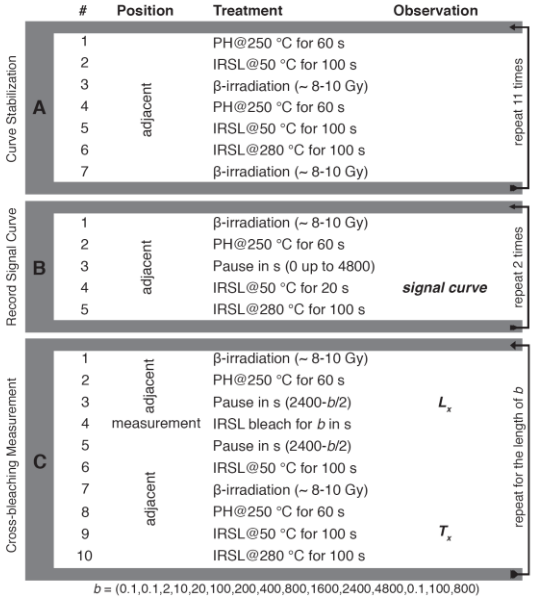
[1] All operations in a given set are carried out on a single sample before any operation is carried out on the next sample.



	#	Position	Treatment	Observation	
Curve Stabilization **A**	1	adjacent	PH@250 °C for 60 s		repeat 11 times
	2		IRSL@50 °C for 100 s		
	3		β-irradiation (~ 8-10 Gy)		
	4		PH@250 °C for 60 s		
	5		IRSL@50 °C for 100 s		
	6		IRSL@280 °C for 100 s		
	7		β-irradiation (~ 8-10 Gy)		
Record Signal Curve **B**	1	adjacent	β-irradiation (~ 8-10 Gy)		repeat 2 times
	2		PH@250 °C for 60 s		
	3		Pause in s (0 up to 4800)		
	4		IRSL@50 °C for 20 s	***signal curve***	
	5		IRSL@280 °C for 100 s		
Cross-bleaching Measurement **C**	1	adjacent	β-irradiation (~ 8-10 Gy)		repeat for the length of *b*
	2		PH@250 °C for 60 s		
	3		Pause in s (2400-*b*/2)	L_x	
	4	measurement	IRSL bleach for *b* in s		
	5	adjacent	Pause in s (2400-*b*/2)		
	6		IRSL@50 °C for 100 s		
	7		β-irradiation (~ 8-10 Gy)		
	8		PH@250 °C for 60 s		
	9		IRSL@50 °C for 100 s	T_x	
	10		IRSL@280 °C for 100 s		

b = (0.1,0.1,2,10,20,100,200,400,800,1600,2400,4800,0.1,100,800)

Table 1: *Cross-bleaching protocol.*

Cross-bleaching protocol

To estimate the cross-bleaching value on an adjacent sample position a blank sample holder was placed on position 1 (measurement position) and a sample on position 2 (adjacent position). Sample holders were either cups or discs (ø 9.7 mm; stainless steel or aluminium). Initially the sample was repeatedly dosed, preheated and read out to stabilize the aliquot in terms of sensitivity change. The full sequence used to estimate the cross-bleaching is given in Table 1. The basic structure of the sequence is that the sample is dosed and preheated but before read out, the blank sample holder (measurement position) is illuminated for a time t_x varying between 0 and $t_{\max}$ (for most estimates $t_{\max}$ is 4,800 s but in some cases it was increased to 15,000 s). Subsequent to each illumination the sample on the adjacent position is read out (L_x) followed by a test dose (T_x) measurement. In between the various SAR cycles (L_x/T_x) the sample was bleached at 280°C for 100 s. Three recycling points were inserted to ensure that any sensitivity change was corrected adequately. To eliminate the effects of anomalous fading the time elapsed between irradiation and read out (L_x) was kept constant by inserting a pause of duration x = $t_{\max} - t_x$. In practice this pause was split in two parts of equal duration, one inserted before and one after the illumination of the empty measurement position. The sequences were run using the "run 1 at a time" option.

In order to derive a value for cross-bleaching, the observed reduction of the luminescence signal of the sample on the adjacent position is compared to that measured by direct stimulation of the same sample (Table 1 part B, Fig. 2b).

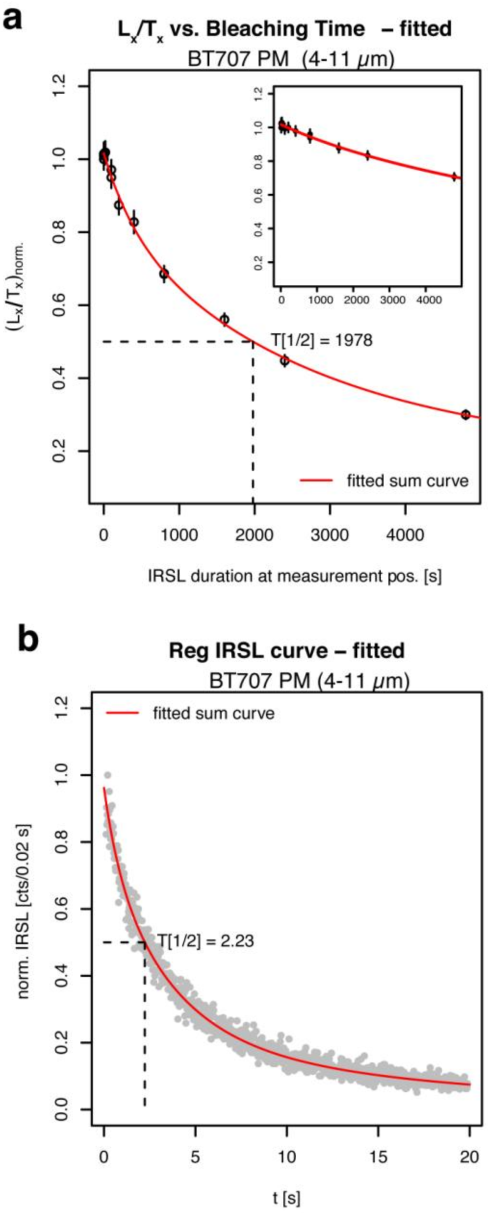


Figure 2: *Normalized L_x/T_x values shown as a function of IR illumination time for the reader with the highest relative cross-bleaching value. (a) Fitted L_x/T_x on the adjacent position (y-axis), each gained with the cross-bleaching protocol (Part B, protocol Table 1) after a respective illumination time on the measurement position (x-axis). L_x/T_x values normalized to the first value and then fitted with a multi-exponential function (n_{max} = 3). The halftime (T[1/2]) is derived iteratively. The obtained halftime from (a) is then compared with the halftime from (b) which is signal curve fitted to the IRSL decay curve data of the sample illuminated for 20 s (part B protocol, Table 1). The inset shows the fitted L_x/T_x values obtained for a reader with lower cross-bleaching behaviour.*

All L_x/T_x ratios were plotted as a function of illumination time and fitted (1) using either a multi-exponential function, where the number of components varied between 1 and 3 (reader ID 45 to ID 262) or (2) using an inverse power law function (reader ID 326). In the first approach the halftime of the fitted curve of the L_x/T_x ratios is compared to the halftime determined from the decay curve observed from direct stimulation (signal curve). In the second approach, the parameters in the inverse power law function fitted to the decay curve from direct stimulation are used to determine the equivalent loss resulting from cross-bleaching. In practice, these two approaches do not result in significantly different cross-bleaching values. Nevertheless, it should be noted that using a multi-exponential function for fitting is not related to any physical model. Details on the quantification procedures are given in the supplementary information (Section 1 supplement).

Measuring the effect of cross-bleaching on dose estimation

Cross-bleaching values are primarily a technical characteristic of a reader. They do not give any information on the degree of the depletion of a luminescence signal or on the corresponding reduction of the equivalent dose (D_e) of a dating sample. In the literature (Bray et al. 2002) it has already been demonstrated that cross-bleaching from the blue LEDs can reduce the estimated equivalent dose significantly if the entire dose-estimation sequence is carried out on a single aliquot before the next aliquot is measured. Here, we report the results from a similar experiment using the IR LEDs.

Two feldspar samples (US-C, KG-1) were heated to 500°C for two hours to completely eliminate the IRSL signal before giving to five portions of each sample gamma (^{60}Co) doses of 4.45 Gy, 8.9 Gy, 13.35 Gy, 26.7 Gy and 40.5 Gy, respectively.

12 aliquots of each gamma-dosed portion were subsequently measured using a routine SAR IRSL protocol with six regeneration doses and IR stimulation at 50°C for 300 s. The sequence was written in a single set in the Risø sequence editor and the “run 1 at a time” option was chosen. This means that the first aliquot was stimulated for a total of 4,200 s (14 × 300 s), before the natural signal of the subsequent aliquot was read out. For each portion irradiated with a given gamma-dose, aliquots were placed at positions 1–4, 10–13 and 20–23. Thus, there are three measurement positions (1, 10, 20) for which optical cross-bleaching is assumed to be insignificant, and nine measurement positions for which cross-bleaching is likely to affect the dose estimate. Sample US-C was measured on a single reader (ID 262, gamma-dose portions as stated above) to investigate a potential dose-dependency, whereas sample KG-1 was measured on three different readers (ID 262, 133 and 45) to investigate the variability of cross-bleaching characteristics between individual readers.

Results

Cross-bleaching values

In Fig. 2a the IRSL L_x/T_x measurements are shown as a function of the illumination time for reader ID 189. For this particular reader, 4,800 s of illumination results in a depletion of the signal of the adjacent aliquot of ~70%. For the second nearest position an illumination of 4,800 s results in signal depletion of ~5%. Both data sets have been fitted using a linear combination of exponentially decaying functions. In Fig. 2b a decay curve obtained under direct stimulation is shown. In summary, for this particular reader, cross-bleaching values of 0.128 ± 0.017% (n=4) and 0.003% (n=1) were derived for the nearest (adjacent) and second nearest (adjacent of the adjacent) position, respectively. These cross-bleaching values are significantly higher than what has been reported for the blue LEDs in the literature before and what has been observed for the other readers investigated in this study. The inset shows the fitted L_x/T_{x}-values obtained for a reader with lower cross-bleaching behaviour (ID 150). All derived cross-bleaching values are summarized in Table 2. Errors of the cross-bleaching values were only given, if at least two repetitive measurements were carried out (see Table 2). Error bars show the standard deviation.

Our experiments show that the cross-bleaching value for the IR LEDs on the adjacent position range from <0.0001% (ID 60) to 0.1279 ± 0.0167% (ID 189). In comparison, the cross-bleaching values derived for the blue LEDs range from 0.0019% (ID 150) to 0.0176% (ID 189). Thus, in all cases the cross-bleaching resulting from the IR LEDs is approximately one order of magnitude larger than that from the blue LEDs (Table 2, Fig. 3).

The lowest cross-bleaching values were observed for the oldest readers (ID 45 and ID 60), which both accommodate sample carousels with only 24 sample positions (as opposed to 48 for all readers produced after 1996). For these sample carousels, the distance between the centres of adjacent sample positions are 32 mm (compared to 17 mm on a carousel accommodating up to 48 samples

The derived cross-bleaching values for a given type of stimulation source (IR- or blue LEDs) vary markedly also between readers and they even scatter for a single sample on the same reader (e.g. ~13% difference between repetitive determinations of the IR cross-bleaching value for reader ID 189, Fig. S1). We further observed a dependency of the IR cross-bleaching values on the chosen signal integral

Risø ID	Year	Type	Head	Sample Carousel	Blue Power [mW cm^{-2}]	IR Power [mW cm^{-2}]	Sample Carrier	Sample Code	Stimulation	Cross-Bleaching			
										Nearest [%]	Nearest Error [%]	2nd Nearest [%]	2nd Nearest Error [%]
45	1994	DA-12	std	24			steel discs	ME S2	IR	0.0065	NA	NV	NV
60	1996	DA-12	SG	24	53	140	steel discs	ME S2	IR	<0.0001	NA	NV	NV
98	2000	DA-15	SG	48	89	147	steel discs	ME S2	IR	0.0167	NA	NV	NV
133	2002	DA-15	SG	48		140	steel discs	ME S2	IR	0.0138	NA	NV	NV
150	2003	DA-15	std	48	40	120	Al discs	BT707, BT711	IR	0.0239	0.0042	NV	NV
							Al discs	BT620	Blue	0.0019	NA	<0.0001	NA
189	2005	DA-15	std	48	36	123	Al discs	BT707	IR	0.1279	0.0167	0.003	NA
							Al discs	BT714, BT620	Blue	0.0176	0.0003	NA	NA
240	2008	DA-20	std	48	45	131	Al discs	HDS-493m, HBII	IR	0.0387	NA	NV	NV
245	2008	DA-20	std	48	47	137	Al discs	HDS-499m, HBII	IR	0.0398	NA	NV	NV
							Al cups	BolivienQ 1409 LM1	Blue	0.0028	0.0004	NV	NV
262	2008	DA-20	std	48	80	128	steel discs	ME S2	IR	0.0296	0.0071	NV	NV
326	2011	DA-20	std	48	78	157	steel discs	914807	Blue	0.0020	NA	0.00016	NA
							steel cups	970425	IR	0.0160	NA	0.0004	NA

Notes
Year - production year. Some of the readers have been upgraded since production.
Stimulation head – ‘std’ is the standard stimulation head, ‘SG’ is a special stimulation head used on readers with a single grain attachment
Sample carousel - the number of available sample positions
Stimulation - stimulation light source (blue and IR). The IR stimulations were detected in the blue range, whereas the blue stimulated signals were detected in the UV range
NA - Not Available (not calculated in cases were the measurement was repeated only once)
NV - No Value (not measured)

Table 2: *Reader characteristics and cross-bleaching results.*

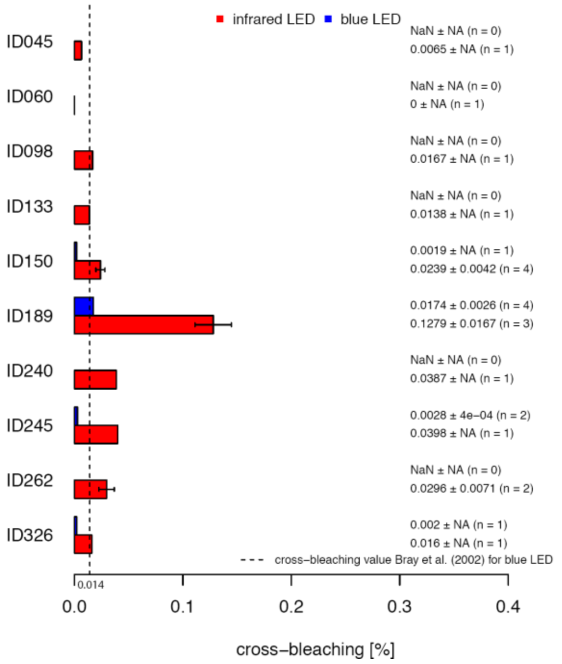


Figure 3: *Cross-bleaching on the adjacent position for IR- and blue LEDs of the investigated readers. If only one measurement was carried out, no error is given. The dashed line denotes the cross-bleaching value for blue LEDs (0.014%) published by Bray et al. (2002).*

(Fig. S2) which decrease with longer signal integrals. However, for all experiments the chosen signal integral was kept constant (see Sec. 2.3).

Except for a single outlier (ID 189) the DA-15 readers (produced between 1997 and 2005) yield mean optical cross-talk values for the blue LEDs of 0.017 ± 0.003% (n=3) which is similar within errors to the cross-bleaching value of 0.014% reported by Bray et al. (2002) for the blue LEDs. The highest cross-bleaching values for the IR LEDs were measured for the newest DA-20 series (produced since 2006) with a mean value of 0.028 ± 0.002% (n=3) for discs. Thus, it would appear that the cross-talk of the IR LEDs have increased by a factor of two. In Table 2 the power density at the sample position at 100% LED power is given for each reader and it is apparent that there is no correlation between the power of the IR LEDs and the measured relative optical cross-talk. This observation seems to be confirmed by an additional experiment on reader ID 189 using a polymineral fine grain sample (BT714). For this sample, no correlation was found between IR stimulation power densities of 31 mW cm^{-2} (25% LED power) and 62 mW cm^{-2} (50% LED power), respectively, and the observed cross-bleaching values (data not shown). No dependency of the IR cross-bleaching intensity on the type of stimulation units (i.e. standard or single-grain) was observed.

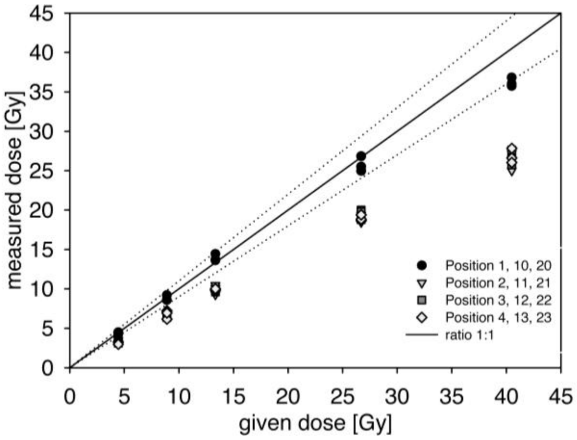


Figure 4: *Measured dose plotted as a function of given dose. All results derived by using the 'run 1 at a time' option for the measurement positions 1–4, 10–13 and 20–23 on reader ID 262 (model DA-20). The aliquots on the positions 1, 10 and 20, which are not affected by cross-bleaching, are displayed as black circles; all other aliquots (influenced by cross-bleaching) are given as grey rectangles and triangles. Also shown is the 1:1 line (black) and the ± 10% lines (dotted lines).*

Effect of cross-bleaching on dose estimation

Figure 4 shows that the measured equivalent doses for sample US-C measured on reader ID 262 reproduce the given dose within ± 10% for the three aliquots on the positions 1, 10 and 20. The tendency of D_e underestimation for the larger doses may be interpreted in terms of fading as the time span between irradiation and measurement was a couple of weeks. In contrast, the measured D_e values of all nine aliquots without an empty position to the left are systematically lower than the given dose. The relative underestimation of ~36 ± 3.2% is not dependent on the given dose. The reason for this is that the stimulation time, and thus the time when cross-bleaching occurs, is constant. Furthermore, it can be deduced that cross-irradiation does not appear to be significant. If it was, we would expect to observe dose-dependent differences.

Although, these effects have not been explicitly investigated in this study, in addition two samples of cross-irradiation measurements using a quartz sample on reader ID 240 are provided in the supplementary information (Figs. S4 and S5). For this reader, the cross-irradiation was found to be ~0.01% on the adjacent position and ~0.001% on the 2nd adjacent position.

Stimulation	**Mineral**	**Sample Carrier**	**Flange**	**t_{max} [s]**	**Nearest [%]**	**2^{nd} nearest [%]**
Blue	Q	steel disc	std	15000	0.00200	0.00016
Blue	Q	steel cups	std	15000	0.00100	0.00003
Blue	Q	steel cups	new	15000	0.00005	
Blue	KF	steel cups	new	10000	0.00010	
IR	KF	steel cups	std	4800	0.01600	0.00040
IR	KF	steel cups	new	10000	0.00100	
IR	KF	steel disc	new	10000	0.00300	

Table 3: *Cross-bleaching results on reader ID 326 using the standard and the new bottom flange.*

However, it should be noted that this value is dependent on the built in irradiation source and therefore may vary from reader to reader (compare e.g. Thomsen et al., 2006; Bray et al., 2003; Bøtter-Jensen et al., 2000; Markey et al. 1997).

The second sample, KG-E, was measured on three different readers (ID 262, 133 and 45). For reader ID 262 a D_e underestimation of 37 ± 3.3% supports the reduction determined for sample US-C above. From the typical decay curves of both samples, it can be deduced that this reduction is equivalent to the depletion caused by ~1.7 s of direct stimulation. Dividing these 1.7 s by the total illumination time (4,200 s) of a complete SAR-protocol, the IR cross-bleaching value is estimated to be ~ 0.04%.

For reader ID 133 the underestimation of 11–18% corresponds to ~0.58 s of direct stimulation giving an estimated cross-bleaching value of ~ 0.014%.

Thus the obtained cross-bleaching values for reader ID 262 and ID 133 are of a similar order of magnitude as the values obtained by the measurements described above.

For reader ID 45 cross-bleaching causes a D_e underestimation of 9–15% giving an estimated cross-bleaching value of 0.011%.[2] A smaller cross-bleaching value was expected for this reader as it has a 24 position sample carousel, i.e. adjacent sample positions are further apart.

Our measurements indicate that sequences should not be written in a single set (writing measurements steps within one single row in Sequence Editor), if the “run 1 at a time option” is chosen or long illumination times on a measurement position are applied. They also show that cross-bleaching may vary significantly from reader to reader.

Instrument modification to reduce cross-bleaching

To reduce the effect of cross-bleaching a new flange has been designed by Risø which instead of a circular opening has an opening shaped as the lift itself (see Fig. 5). A series of experiments were undertaken using reader ID 326 (produced in 2011) to assess the cross-bleaching using this new flange. The results are presented in Table 3 and can be summarised as follows: (1) It appears that by using stainless steel cups instead of stainless steel discs the cross-bleaching value is reduced by a factor of ~ 2.5. (2) The cross-bleaching value for the second nearest position is ~4% of the value from the adjacent (nearest) position. (3) These measurements confirm the previous observation that the cross-bleaching resulting from the IR LEDs is an order of magnitude larger than that from the blue LEDs. Finally, the results indicate that the new flange reduces cross-bleaching by a factor of ~20 to a value of 0.001% for the IR LEDs. The corresponding value for the blue LEDs is 0.00005%. The new flange is now the standard flange for all readers produced from the middle of 2012.

Discussions

Our results show that cross-bleaching varies significantly between the investigated readers. The differences are only partly correlated with the type series. For the DA-12 series, for which the distance between the centres of adjacent samples is 32 mm on the carousels with 24 positions one would a priori expect cross-bleaching to be negligible. This was only confirmed for one reader (ID 60). For the second investigated DA-12 reader (ID 45), however, we measured an optical cross-talk of 0.0065% indicating that cross-bleaching can be relevant also for the readers of the DA-12 series. This agrees with Bøtter-Jensen et al. (2000) who report on an improved lift mechanism for the at that time newly introduced DA-15 series, which reduced cross-bleaching as compared to the older DA-12 readers. Therefore, an old lift mechanism (differences in the uplift height) may be responsible for the observed cross-bleaching value of reader ID 45 that was not confirmed for reader ID 60. This reader, however, has been subjected to technical modifications (e.g. single grain attachment) over the years. The potential for anomalous fading (e.g. Wintle 1979; Visocekas

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[2] Note that this value might not be comparable to the value derived by the cross-bleaching protocol in Table 1 because the stimulation unit was modified after the measurements described here.

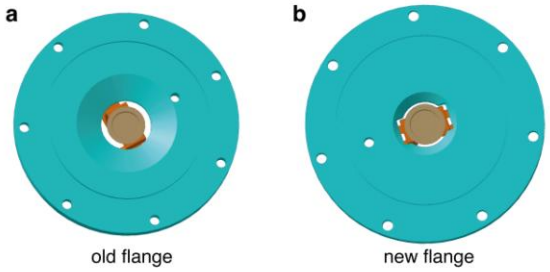

Figure 5: *Old (a) and new (b) bottom flange of the Risø reader. The opening has been minimized to reduce optical cross-talk.*

1985) to cause any signal loss was minimised by the sequence design and should therefore not be responsible for the signal loss observed on the DA-12 reader (ID 45).

Furthermore, it should be noted that our results for these readers are based on single measurements (one per reader). Due to technical modifications on reader ID 45 after the experiment the measurement could not be repeated.

For the DA-15 and DA-20 readers (excluding reader ID 189) we obtained a mean IR cross-bleaching value of 0.026% with a standard deviation of 0.011% consistently in the same order of magnitude as all investigated readers except for reader ID 189. Our findings indicate that the obtained IR cross-bleaching value of 0.1279 ± 0.0167% for this particular reader is not normally expected. As described in the introduction (see Fig. 1) the sample is lifted into the stimulation head through a circular opening in the bottom flange of the stimulation head. The differences are likely to arise from differences in how high the sample is lifted into the stimulation head, from the distance of the sample carousel with respect to the lid and/or from the thickness of the bottom flange. However, the actual reason for the observed behaviour remains unknown so far.

Surprisingly, the cross-bleaching value also varied for an identical sample on one reader and to a greater extent between assumed similar readers for one sample. Using an identical sample on one reader the measurement conditions were kept constant. Therefore it is likely that the differences result from the method applied for the calculation of the cross-bleaching value. The halftime of the obtained L_x/T_x-values on the adjacent position is compared with the halftime of a directly measured decay curve. Both halftimes are mathematically derived from previously fitted functions. Therefore, any (stochastic) variation in the fitted curve shapes yield different halftimes and cross-bleaching values.

Furthermore, our results show that the optical cross-talk for the IR LEDs is significantly higher than that for the blue LEDs. The reasons for this observation could not be further investigated in this study and may reflect differences in the stimulation geometry and/or the optical characteristics of the LEDs.

Of more practical relevance might be the implications of the deduced, somehow artificial, and apparently highly reader-specific, cross-bleaching values. Our results suggest (e.g. Table 2) that the optical cross-talk of the IR LEDs on the Risø TL/OSL readers can be significant in routine dating applications, if care is not taken when designing the measurement sequences. In this respect, the most important question is: How much (cumulative/total) stimulation time on the measurement position is acceptable for a given threshold value of signal reduction on the adjacent position? To account for this question two normalized typical natural decay curves (BT711, fine grain, polymineral and quartz) measured at 90% LED power (IR and blue LED) were plotted against the cumulative stimulation time on the measurement position for given cross-bleaching values of 0.02% (IR LED) and 0.002% (blue LED). The results are shown in Fig. S3. Based on a fitted inverse power law (IR LED, polymineral sample, Fig. S3a) and a single exponential function (blue LED, quartz, Fig. S3b) the cumulative stimulation time on the measurement position for a given value of signal reduction on the adjacent position (1%, 10%, 25%, 50%) were calculated. For the fitting the first five seconds of the decay curve of the polymineral sample and the first two seconds of that of the quartz sample were used respectively. For example: For a signal reduction of 25% of the polymineral sample on the adjacent position, ~5168 s of IR stimulation (90% LED power) on the measurement position are needed (polymineral, Fig. S3a). Our considerations allow a calculation of all possible combinations of cumulative stimulations times on the measurement position and cross-bleaching values for a presumed threshold value of the signal reduction (isoline).

Figure 6 shows the 1% to 5% signal reduction isolines of a typical polymineral (IR stimulation, Fig. 6a) and quartz (blue stimulation, Fig. 6b) fine grain sample (BT711) for a realistic range of cross-bleaching values. Considering the overall precision of the cross-bleaching measurements the allowed value of stimulation time on the measurement position for a given value of signal reduction can be read off with sufficient accuracy from the figure. Thus, an assumed cross-bleaching value for the IR LEDs of 0.03% allows a stimulation time of ~100 s on the measurement position if a signal reduction of 1% on the adjacent position is accepted. Using the newly designed bottom flange, or every 2nd position on the sample carousel, in case the new flange has not yet

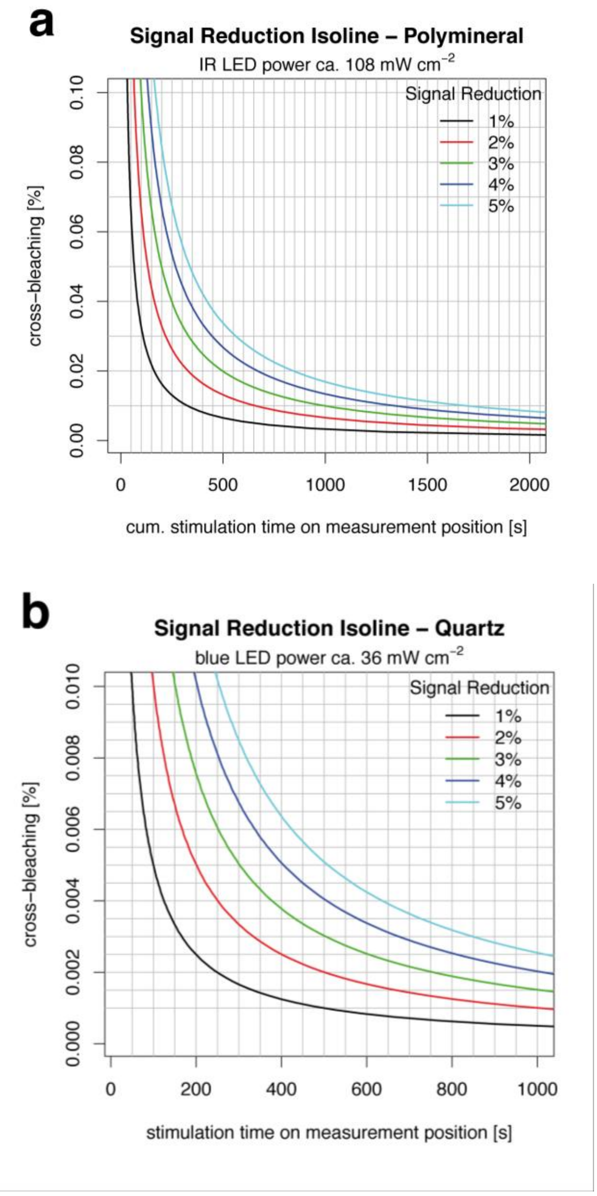

Figure 6: *Signal reduction on adjacent position vs. cross-bleaching values. Corresponding isolines for five hypothetically tolerated thresholds (1% to 5%) by cross-bleaching on the adjacent position. The lines represent all possible combinations of cumulative stimulation times (up to 2000s for IR stimulation) on the measurement position (x-axis) and cross-bleaching values of a reader (y-axis) for constant value of signal reduction. The signal-reduction isolines for a typical polymineral (a) and quartz (b) fine grain sample are shown using IR stimulation and blue stimulation respectively. Note that the shapes of the isolines depend slightly on the absolute power density of the stimulation LEDs. For further details see main text.*

been installed, a cross-bleaching value of <0.003% for the IR LEDs can be assumed. For the same amount of tolerated signal reduction, this allows stimulation times at least larger by a factor of ten. Thus cross-bleaching will become negligible for ordinary SAR-measurements.

In summary, for routine dating applications an appropriate measurement sequence design or the use of every 2nd position is considered as adequate to reduce any unwanted malign effect of cross-bleaching below the detection threshold.

Nevertheless, it is worth mentioning that the cross-bleaching values were measured using a blank sample carrier on the measurement position. Any sediment on the sample carriers may cause different (material dependent) scattering effects. Such effects have not been investigated in the presented study.

Conclusions

In this study the cross-bleaching (optical cross-talk) of the IR LEDs for the adjacent position has been investigated on various types of Risø TL/OSL readers. For the quantification three different methods were applied. The results were compared with findings from the literature and our own measurements of the cross-talk of the blue LEDs on the same reader. In addition, a newly designed flange to reduce the cross-bleaching was tested and the results were compared with the measurements run carried out using the standard flange. In summary:

1. We confirm that cross-bleaching exists for the IR LEDs on almost all investigated readers (~0.026% for DA-15 and DA-20 readers).
2. Our findings indicate that the cross-bleaching is significantly higher (ca. one order of magnitude) for the IR than for the blue LEDs.
3. The relative cross-bleaching seems to be independent of the stimulation power.
4. The cross-bleaching value varies with the used sample carrier and is observed to be higher for steel discs than for steel cups.
5. The newly designed bottom flange reduces the cross-bleaching by a factor of ~20 resulting in negligible signal reduction by cross-bleaching on the adjacent position.
6. The cross-bleaching effect can lead to significant age underestimations if care is not taken in the measurement-sequence design. It is therefore highly recommended to split the sequence in different sets when using the “run 1 at a time” option or to use only every 2nd measurement position on the sample carousel (for an assumed cross-bleaching value <0.0001% on the 2nd nearest position). A sequence template splitting up the steps in different sets is provided as supplementary data on the *Ancient TL* website.

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Supplementary Information for this article is available at www.aber.ac.uk/ancient-tl

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Reviewer

R. Bailey

Reviewer's comment

As the equipment for measuring OSL becomes increasingly sophisticated, reliable, and also easier to use, the user arguably becomes more removed from the process of measurement. It is easy to forget the details of what is happening inside the equipment and to ignore the potential for systematic effects. The careful analysis described in this paper aids both the users and the designers of automated equipment in our shared goal of reducing systematic effects where we find them and improving the reliability of our results. Other aspects of the dating process will no doubt benefit from similar investigation.