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Effect of single-grain versus multi-grain aliquots in determining age for K-feldspars from southwestern British Columbia

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Introduction

Using simulated data, Arnold and Roberts (2009) have argued that multi-grain aliquots, because of averaging effects among grains, cannot correctly distinguish different dose components in mixed-age samples. They argued that averaging effects may have less effect for determining the correct depositional age of partially bleached sediments, using the minimum age model (MAM), provided that partial bleaching is the main cause of dispersion. This paper presents empirical evidence to show that MAM on multi-grain aliquots may produce grossly over-estimated ages even from small aliquots in the range of 2-3 grains each. The samples are K-feldspar extracts from fluvial sediments collected from the Chilliwack Valley, southwestern British Columbia.

The Chilliwack Valley is a large mountainous watershed in the North Cascade Mountains. The valley was under 2 km of ice during the last glacial maximum, but during a late glacial phase of alpine glaciation, a series of retreats and advances of Fraser ice into Chilliwack Valley led to a moraine blocking the valley mouth and to the development of a sequence of intermittent glacial lakes and an outwash plain in the mid-valley (Saunders et al., 1987). With further ice retreat, valley base-level was lowered considerably (100 m or more) as the Chilliwack River began to cut down through (or incise) these glacial sediments, a process that extended well into the Holocene (Tunnicliffe et al., submitted). To strengthen the chronology of these events, nine luminescence samples were collected from a series of mid-valley terraces deposited during the incision process. Dates for eight of these samples are evaluated here.

Procedures

The samples form two sets, UW1326-29, which were collected in summer 2005, and UW1863-66, which were collected in spring 2008. All were retrieved from between 35 and 60 cm below the modern surface. Because quartz in these samples had little sensitivity using UV emission, K-feldspars were prepared. For UW1326-29, both 125-150μm and 180-212μm fractions were prepared, while for UW1863-66 only the larger fraction was prepared. This report focuses on the differences in results between the two size fractions of UW1326-29, mentioning the results of the other samples only for comparison.

Luminescence was measured using the single-grain attachment to a Risø TL/OSL DA-20 reader, using a 150mW 830nm IR laser for excitation. Because the single-grain disks have 300μm diameter holes, the 180-212μm grains generally provide single-grain resolution, but 2-3 grains of the 125-150μm grains will fit in each hole, providing effectively small multi-grain aliquots.

K-feldspars were isolated by sieving, treatment with HCl and H_2O_2, and density separation using a lithium metatungstate solution of 2.58 specific gravity. No HF was applied. For measuring luminescence, the laser was passed through a RG780 filter and set at variable power from 30-90%. A 7.5 mm blue filter pack (Scott 7-59 and BG-39) allowed emission in the 350-450 nm range. The variable power did not appear to have a detectable effect. Equivalent dose (D_e) values for two samples were 21.5±2.1 and 5.8±1.0 Gy for 30% power and 18.1±1.6 and 5.8±0.9 Gy for 70% power, respectively. The values were based on 20-60 grains for each sample, using the central age model. No significant differences in over-dispersion or ages corrected for anomalous fading were detected either.

D_e was determined using a single aliquot regenerative dose (SAR) method as adapted for feldspars (Auclair et al. 2003), with stimulation at 50°C and using a preheat of 250°C for 1 minute (after either

Table 1: *Luminescence dating data for samples arranged in stratigraphic order of terraces from oldest to youngest*

Sample	Terrace	U (ppm)	Th (ppm)	K (%)	Total dose rate (Gy/ka)*	Grain size (µm)	N	Age (ka) Central age model	Over-dispersion (%)	Age (ka) Minimum age model
UW1326	T1-outwash surface	1.38±0.11	3.77±0.76	1.40±0.01	2.64±0.20	180-212	36	10.9±0.7	1.3	11.5±2.2
					2.52±0.16	**125-150**	**245**	**87.1±4.2**	**53.3**	**36.1±2.8**
UW1863	T2-upper	1.03±0.08	1.63±0.49	1.35±0.05	2.27±0.05	180-212	108	15.1±2.2	132.6	2.9±0.8
UW1866	T2-upper	1.58±0.11	1.90±0.54	1.58±0.08	2.44±0.05	180-212	197	28.0±2.8	119.0	5.0±0.9
UW1327	T3-mid	1.39±0.10	1.85±0.53	1.21±0.05	2.36±0.20	180-212	86	23.8±3.4	119.1	3.5±0.7
					2.24±0.15	**125-150**	**328**	**64.1±2.7**	**65.6**	**20.0±1.3**
UW1865	T3-mid	1.85±0.17	9.77±1.17	1.73±0.08	2.34±0.06	180-212	188	24.9±2.2	106.3	4.4±0.6
UW1328	T3a-mid-eroded edge	1.76±0.12	2.90±0.68	1.20±0.04	2.69±0.20	180-212	85	30.4±4.4	117.7	4.3±0.8
					2.57±0.16	**125-150**	**250**	**46.6±3.1**	**87.0**	**10.5±1.0**
UW1864	T3a-mid-eroded edge	0.96±0.07	1.56±0.44	1.63±0.11	2.78±0.06	180-212	117	15.6±1.8	109.5	3.0±0.5
UW1329	T4-lower	1.70±0.12	3.02±0.68	1.21±0.02	1.95±0.07	180-212	100	11.5±0.6	109.6	1.1±0.2
					1.84±0.07	**125-150**	**38**	**31.6±5.4**	**91.3**	**7.0±1.8**

* For the alpha contribution, a b-value of 1.9 ± 1.0 was used to correct for lower efficiency. The dose rate is slightly higher for larger grains because of grain size effect on attenuations and internal K contribution.

regeneration or test dose). Anomalous fading rates were measured using the procedures of Auclair et al. (2003) for single aliquots. Ages were corrected following Huntley and Lamothe (2001). Storage times after irradiation of up to 3-5 days were employed to determine fading.

Dose rates were measured in the laboratory by alpha counting, beta counting and flame photometry and in situ by placing $CaSO_4$:Dy dosimeters. The dosimeters measured a somewhat higher external dose rate than that calculated from the laboratory measurement of the samples themselves, making a difference of about 12% in the total dose rate. The rate from the dosimeters, as a more direct measure, was used in the age calculation. The internal beta dose rate from K-feldspar grains was evaluated by determining the K-content on 39 grains (180-212μm) of samples UW1327-8 (also used for D_e measurement) using X-ray energy dispersive analysis attached to a scanning electron microscope. The K content averaged 8.2 ± 2.9%, compared to 14% for pure orthoclase. The dose rates in Table 1 were calculated using the former value. A moisture content of 10 ± 5% was used, which was about 2-5% higher than the measured values from summer-collected samples.

Results
Table 1 gives the samples, their provenance and a summary of results. The discussion here focuses on the age distributions. Aside from measurement of internal K on a few grains, no attempt (nor the ability to do so) was made to measure dose rate at a single-grain scale, so a bulk dose rate for each sample was used in the age calculations.

A well-noted advantage of single-grain dating is the opportunity to remove from analysis grains with unsuitable characteristics as judged by failure to meet a set of criteria. Grains were eliminated from this analysis if they (1) had poor signals (as judged from net natural signals less than three times above the standard deviation of the background), (2) had recycle ratios outside of the range 0.8-1.2, (3) yielded natural signals that did not intersect the regeneration growth curves, (4) had a signal larger than 10% of the natural signal after a zero dose, and (5) produced a D_e within 1-sigma of zero.

A total of 2951 single-grains of 180-212μm were measured. Of these, 1030 passed all the criteria, an acceptance rate of 35%. Of those rejected, 66% were because of poor signal. Of the other criteria mentioned, the largest number of rejections was due to the natural signal not intersecting the growth curve (17%). Most of these were probably due to the regeneration curves not being carried out far enough

to capture the natural signal, although some showed the regeneration curve saturating below the natural level, a phenomenon well documented in quartz, but not published to our knowledge for feldspar. Figure 1 shows some examples. Early measurements used about 300 Gy as the highest regeneration dose, but later measurements (on the UW1326-1329 180-212μm grains) reduced this to about 185 (for UW1327-8) or even 25 Gy (for UW1326, UW1329) to save machine time when it was realized the samples were much younger. This had the effect of increasing the number of rejections due to lack of natural intersection. The full distributions, even for those with a maximum regeneration dose of 300 Gy, are clipped at the high end. This should not affect minimum age determinations as long as the highest regeneration dose is well above that required for the minimum ages. To measure the effect, the distributions for single-grain samples where the highest regeneration dose was 300 Gy were artificially clipped for maximum D_e of 185 and 25 Gy (Table 2). No significant difference in minimum age is observed. A 25 Gy dose translates into an uncorrected age of about 10 ka. Almost half the rejections by this criterion came from one sample, UW1326, the sample from the highest terrace (and presumably the oldest).

A total of 1787 single grain holes containing 125-150 μm grains (small aliquots) were measured for 4 samples, and 946 were accepted, a rate of 53%. Of the rejections 30% were because of the natural signal not intersecting the growth curve, again the largest number (about half) from UW1326. After fading tests, there were further rejections for both single grains and small aliquots because high fading rates produced infinite corrected ages. The number of measurable ages totalled 917 for the single grains and 861 for the multi-grain aliquots.

A dose recovery test was done on 400 180-212μm grains from four samples, using an administered dose of 200s (~24 Gy) of beta irradiation. A total of 201 grains produced an acceptable signal. The weighted average, using the central age model, was 205 ± 3.4 s, but with an over-dispersion of 18.5%. These results suggest the procedures are generally appropriate, but the grains show wide variation in behavior. The 18.5% over-dispersion indicates that scatter of at least that magnitude can be expected in the natural samples even if all grains are the same age.

Fading rates were also highly variable. A weighted average of all g-values from the 180-212 μm grains yielded 3.9 ± 0.3% (normalized to two days), which is in the 3-5% range reported for southern British Columbia and northern Washington by Huntley and
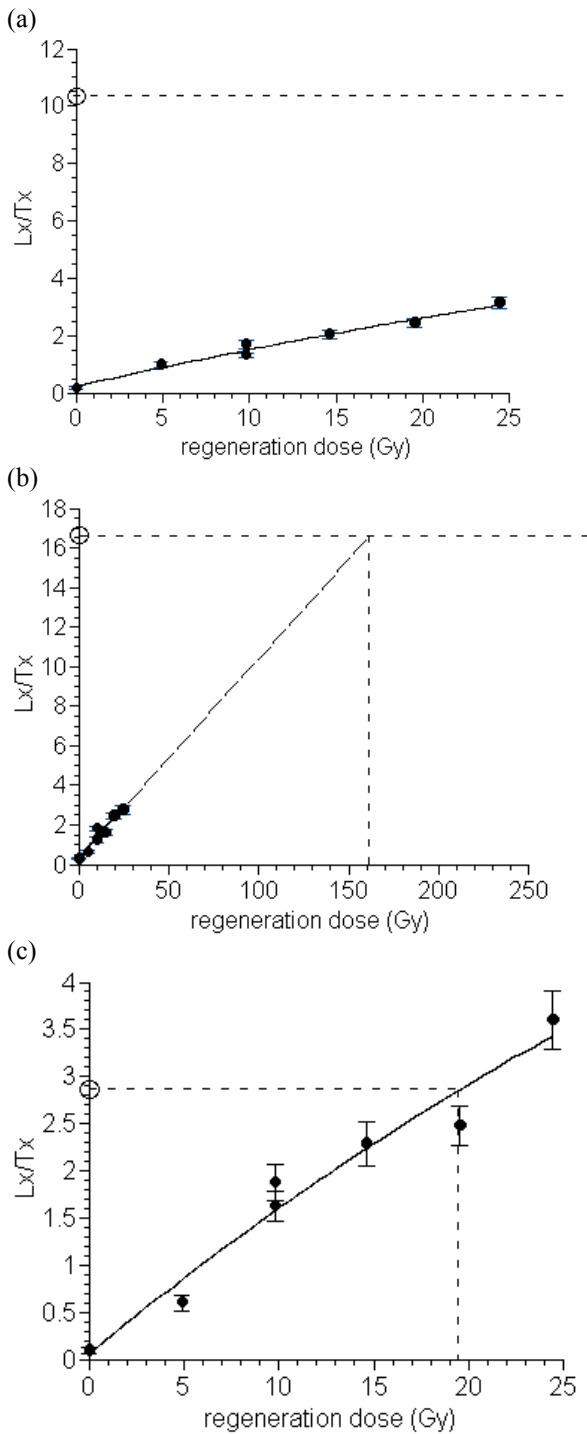
(a)

(b)

(c)

Figure 1: *Growth curves for three grains from UW1326, 180-212μm grains. a) grain where the regeneration curve saturates below the natural level, b) grain where an extrapolated saturating exponential curve is able to reach the natural level, c) grain where the natural level is within the limits of the regeneration growth curve.*

Sample UW	Age (ka) from MAM for maximum regeneration doses					
	300		185		25	
	N	Gy	N	Gy	N	Gy
1863	122	2.9±0.8	109	2.9±0.8	72	3.2±0.8
1864	131	3.0±0.5	124	3.0±0.5	58	2.9±0.6
1865	214	4.4±0.6	198	4.3±0.6	74	3.3±0.6
1866	226	5.0±0.9	193	4.6±0.9	84	5.0±1.4

Table 2 : *Minimum age values for different maximum regeneration doses. In each case, N, is the number of grains used in the MAM.*

Lamothe (2001). Leaving off negative values (17% of them), over-dispersion of the g-values was calculated at 43%.

Table 1 gives the ages determined by the central age model as well as the over-dispersion. Except for the single-grains of UW1326, the over-dispersion is high, over 100% for all other single-grain samples. The central age is higher and over-dispersion lower for the small aliquots, on a sample to sample comparison. The lack of over-dispersion for single-grains of UW1326 can be explained by the use of a maximum regeneration dose of 25 Gy. The older grains for this sample were simply not accepted because the natural signals were too large. A total of 43% of grains from this sample were rejected for this reason. The accepted grains form an effective single-age sample. It is possible the age for this sample is underestimated because the regeneration doses were not carried out far enough, but the determined age is consistent with independent evidence. We also reanalyzed this sample accepting all the grains where the natural signal was larger than the highest regeneration dose, using extrapolation, either linear or saturating exponential, of unknown reliability. This resulted in 183 acceptable values (after the fading correction). No longer consistent with a single age, the minimum age for this sample was 15.0 ± 1.51, somewhat older but within error terms of the single grain age determination from the clipped data set.

The most likely cause of high over-dispersion for the other samples is partial bleaching. The central age model produces ages that are Pleistocene or very early Holocene, which is not in accordance with the geological evidence that the terrace sedimentation is post-glacial. Radiocarbon evidence strongly points to deposition on the uppermost terrace ending some time after 11,400 ^{14}C yrs. B.P. (Saunders et al., 1987). Post-depositional mixing is not likely, because it is not clear where the older grains would be coming

from. The older ages more likely represent grains whose signal was not fully reset at the time of deposition. Partial bleaching is not uncommon for high energy streams. Another possible cause of over-dispersion is differential dose rate caused by variation in K content within individual grains. For the 33 grains for which both K content and age were determined, there is no significant correlation between K and age ($R^2 = 0.01$). Reducing the internal dose rate by a third (which would be appropriate for lower K grains) only reduces the total dose rate by 10%, which cannot account for the differences by a factor of 5 to 10 between the central age and minimum age for these samples.

If partial bleaching is the cause of over-dispersion, then the minimum age model is appropriate. We used the model of Galbraith et al. (1999), with three unknown variables, and adding 18.5% error in quadrature to the error on each age (the 18.5% value coming from dose recovery). Minimum ages are given in Table 1, with 125-150 μm results in bold. The minimum ages for the 180-212 μm grains are in accordance with the post-glacial expectations from the geology, and agree with some independent dating evidence, including radiocarbon analyses from the outwash surface of Terrace 1. They also roughly agree with the chronological order of the terraces, although errors are high. The ages of the 180-212 μm fraction from UW1327-1328 also agree with counterparts UW1864-1865 from the same terraces, even though the latter were measured with higher regeneration doses. The minimum ages of the 125-150 μm small aliquots, on the other hand, are much older for every sample, two of them Pleistocene in age.

Figure 2 shows radial graphs of the age distributions for both grain sizes for UW1327. The two shaded areas represent the 2σ range for the minimum age and central age for the 180-212 μm fraction. The third line represents a possible higher component. Both graphs show the older components, but the younger ages are mostly absent with single-aliquots using smaller grain sizes. This can be readily explained by averaging effects, particularly considering the high acceptance rate for these samples and the high number of partially bleached grains. The smaller signals associated with the younger grains are simply swamped by the larger signals from the more plentiful older, partially bleached grains. Perhaps if enough measurements were made, at considerable cost in machine time, a large enough population of younger ages could be measured so that they would show up statistically as a minimum age. The larger the aliquot the less likely this would be possible (Fig 4. in Olley et al. 1999).
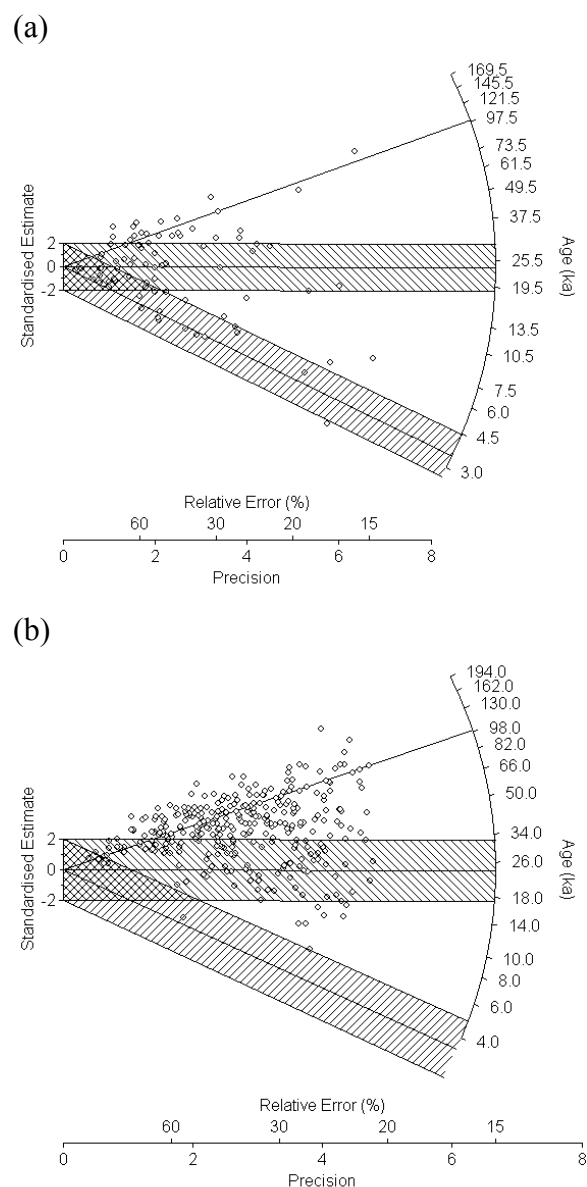
Figure 2. *Radial graphs showing central age and minimum age for UW1327 using (a) 180-212μm single grains and (b) 125-150μm multi-grain aliquots. The reference lines in both graphs are the minimum age and central age results from the 180-212μm fraction plus a possible older component around 100ka. The shaded areas around the minimum age and central age reference lines represent 2σ range.*

The data presented here suggest that for partially bleached sediments, particularly involving highly sensitive feldspars, accurate minimum age determinations will require single-grain analysis.

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Reviewer
M. Lamothe

Reviewers Comment:
Feldspar single grains are back! This is a nice demonstration that unless the most simple sedimentary object is used, that is a single grain, luminescence dating will not provide an accurate evaluation of burial time for partially-bleached sediments. More research in feldspar single grain laser technology is timely, to increase precision, a malign collateral "damage" of anomalous fading.

Optical dating of young feldspars: the zeroing question

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Abstract

We present evidence that when using 1.4 eV excitation for optical dating of young K-feldspars, one must recognize that the state of the mineral after a long light exposure depends on the spectrum of that light. Hence, a modern analogue is necessary for the determination of the equivalent dose. In particular, it is required for determining the spectrum of light exposure to be used for the thermal transfer correction.

Introduction

The ages of young sedimentary deposits (< 1000 years) are frequently of interest to geoscientists. Ages can be used to assess rates of sediment accretion and deposit stability, for example, and optical dating has become increasing popular for obtaining them. A review focused on young sediments has recently been published by Madsen and Murray (2009). They clearly show that current optical dating techniques hold significant promise for dating a variety of Holocene sedimentary deposits, in some cases even as young as ten years. However, in the case of feldspars they did not discuss what we term the "zeroing question". The objective of this note is to emphasize the need to consider this question when dating young feldspars, because getting the right answer to it is necessary if one is to get correct ages for young sediments.

Madsen and Murray (2009) identified the main problems associated with the dating of young sediments as: (1) insufficient luminescence to determine equivalent doses, (2) thermal transfer during the heating that takes place prior to luminescence measurement, (3) insufficient sunlight exposure just prior to burial, and (4) changes in the dose rate with time. Their review deals mainly with quartz from sediments, but includes work on feldspars from sediments when there was relevant research to discuss. Although they suggest the necessity of having a zero age analogue when dating "older" samples, they do not discuss any relationship between the spectrum of the bleaching at the time of deposition and the spectrum of the bleach used for
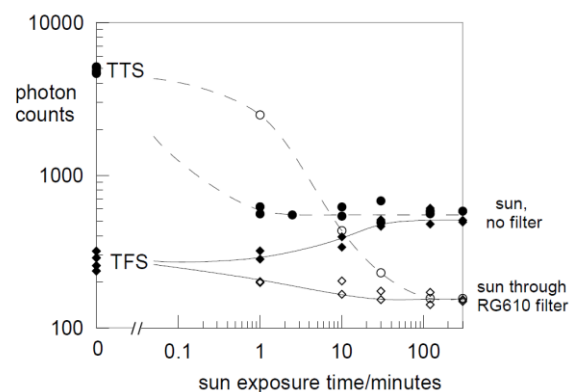
Figure 1: *Reproduction of data presented in Figure 11 of Huntley and Clague (1996), showing the effect of a prior light exposure on the luminescence intensity due to 1.4 eV excitation. Aliquots of a 300-year-old tsunami sand (TTS) and of a zero-age source sand (TFS) were exposed to unfiltered (solid symbols) and filtered (open symbols) sunlight. No preheat was used.*

thermal transfer measurements.

This issue is illustrated in the Appendix of Huntley and Clague (1996); their key result is reproduced here as Figure 1. It is seen that, compared to the luminescence from aliquots not exposed to light, the 1.4-eV excited luminescence of K-feldspars extracted from the modern sediment (TFS) was significantly larger (up to 2x more) if the grains had been previously exposed to several hours of sunlight, whereas it was smaller (about ½) if there was a prior exposure to infrared light. Related effects were observed in a 300-year-old sample of the same sediment (TTS). Note that the endpoints for the filtered sunlight exposure are the same for both samples. The same is essentially true for the unfiltered sunlight exposure but these endpoints are significantly higher.

It was quite clear that the natural bleach for TFS could not have been unfiltered sunlight, but that some of the high photon energy component had been

removed. Knowing this was essential to obtaining ages consistent with independent information. Huntley and Clague (1996) suggested that the effect might be attributed to the higher energy photons of sunlight causing transfer of electrons into the principal traps (those being sampled by the 1.4 eV excitation photons), either from other traps or from the valence band. They further noted that such a transfer was observed by Bailiff and Poolton (1991) for feldspars using photons of about 4 eV energy. This work illustrates that the spectrum of sunlight exposure at deposition, and consequently the spectrum of the bleach used for the thermal transfer measurement, matter.

Long Point Experiment

Work done to date a young sample of known age from Long Point, Ontario, Canada, further illustrates this issue. Long Point is a sandy barrier spit located on the north shore of Lake Erie. A severe storm in December, 1985 opened many overwashes along the spit, including two closely related ones near the distal end. These washovers were activated again in mid-December, 1987. Rebuilding of the foredunes at the sites of these two washovers was monitored by Davidson-Arnott and Fisher (1992; their study site WSS3). Their data provided the framework for the samples described here.

Sample C was collected on 15 August 1995 from the lower part of a young dune that formed in the throat of the eastern 1985/1987 overwash. Because the dune rebuilding at the site of this overwash was monitored by Davidson-Arnott and Fisher (1992), it was known that the dune had been stable for 8-10 years prior to sampling. Thus, an optical age of 9 ± 1 years was expected. Sample D was collected from the upper 2 mm of beach sand lakeward of the foredunes as presumed source material for the dunes. For it, the time elapsed since a significant sunlight exposure, and hence optical age, was expected to be < 1 year.

A procedure similar to that used by Ollerhead et al. (1994) was used in July 1997 to determine equivalent doses for both samples using the 250-355 μm K-feldspar fraction. The excitation source was an array of 1.4 eV light-emitting diodes fitted with a Schott RG-630 filter. Luminescence was measured using an EMI 9635Q photomultiplier tube fitted with a Schott BG-39 and a Kopp 5-56 filter. This combination was designed to exclude measurement of unwanted quartz and plagioclase that were expected to be present along with the K-feldspar grains (e.g., Huntley and Baril, 1997). The measurement system was most sensitive to the 3.1 eV (violet) emission from K-feldspar; quartz does not respond to the 1.4 eV (infrared) excitation used and plagioclase emits predominantly at 2.2 eV (yellow-green).
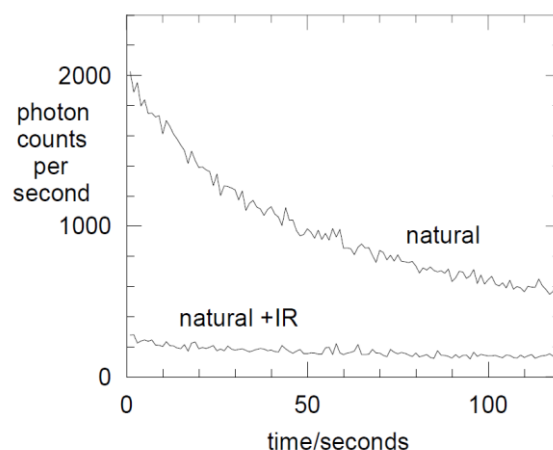
Figure 2: *Shine-down curves for a natural and an IR-bleached aliquot for Long Point sample D. These aliquots were not given any dose or preheat. Excitation was 1.4 eV photons.*

The luminescence resulting from a 5 second exposure to 1.4 eV excitation was measured for each aliquot prior to any treatment. The ratio of measured luminescence to the mean value for a set was used to normalize data from treated samples in order to account for inter-aliquot variability. Equivalent doses were obtained using the multiple-aliquot additive dose method with a thermal transfer correction (Ollerhead et al., 1994; Wolfe et al., 2001). To start with, the thermal transfer correction was done using an infrared bleach obtained from a quartz-halogen lamp with a Schott RG-715 filter to absorb visible and ultraviolet (UV) light. Except for a few control aliquots, all treated aliquots were preheated at 120°C for 21 hours and then stored at ambient temperature for at least a month before measurement.

The results obtained indicated both promise and problems. Of immediate concern was that the luminescence intensity vs time plot, the shine-down curve, for untreated control aliquots from sample D showed a decrease, which was troubling because sample D was thoroughly bleached by sunlight prior to collection. Figure 2 shows representative luminescence intensity vs time curves for a natural aliquot and for a similar aliquot first given a laboratory infrared bleach. These aliquots were not given any dose or preheat. This showed that determining an optical age for this sample using the "usual" method would not produce the expected age of zero, and it did not. The equivalent doses obtained for samples D and C are provided in Table 1 and lead to apparent ages of 70-90 years. It is clear from these results that the equivalent doses determined using the infrared bleach were incorrect.

Sample	D_{eq} (Gy)	Optical age (a)	Known age (a)
IR bleach			
D	0.23 ± 0.02		< 1
C	0.28 ± 0.02		9 ± 1
(D_{eq} for C) - (D_{eq} for D)	0.05 ± 0.03	19 ± 12	9 ± 1
Sunlight bleach			
D	0.06 ± 0.04		<1
C	0.05 ± 0.03	19 ± 12	9 ± 1

Table 1: *Equivalent doses, optical ages corrected for anomalous fading, and expected ages for samples C and D for both IR and real sunlight bleaching to determine the thermal transfer correction are presented. The IR bleach was 3 hours in the laboratory, the real sunlight bleach was 1 minute. The optical ages were corrected for anomalous fading using the procedure described by Huntley and Lamothe (2001) using a measured fading rate of 6.7 ± 0.3 %/decade starting 2 days after irradiation; the delay between irradiation and measurement was 220 days for the IR bleach and 65 days for the sunlight bleach. The corrections increased the ages by about 10%. The dose rate for sample C was 3.0 ± 0.1 Gy·ka^{-1}.*

If one treats sample D as a modern analogue for C and subtracts the equivalent dose, then an age of 19 ± 12 years results, consistent with the known age of 9 ± 1 years (Table 1).

Discussion

The fact that our surface sand sample did not yield an optical age consistent with zero when using an IR bleach for the thermal transfer correction could be a result of two factors: (i) the sample was not well bleached prior to collection, (ii) the technique for determining the equivalent dose was inappropriate, or both.

Insufficient sunlight exposure was unlikely for the following reasons: (a) sample D was known to have been exposed to bright sunlight for > 6 hours prior to collection, (b) the equivalent dose vs time curve did not show an increase in the later part of the intensity decay, and (c) for each sample the aliquots were divided into two sets, those with normalization values

below the median and those with values above the median; the two sets gave consistent results. With insufficient sunlight exposure, grains with less exposure would have been expected to have dominated the high normalization values and have led to larger equivalent doses.

Thus, we attributed the failure to obtain an optical age of zero for this sample to use of an inappropriate method. As noted previously, Huntley and Clague (1996) found the use of a sunlight bleach to be inappropriate for their modern source material as the luminescence caused by 1.4 eV excitation was larger if there was a prior exposure to sunlight. Their tests showed that aliquots shielded from the short-wavelength component of natural sunlight reached a lower luminescence level than aliquots exposed to unfiltered sunlight. The shielding mechanism is not known in that case, but possibilities include coatings on grains, scattering of light by particulate matter in water, and/or absorption of light by dissolved organic compounds in water (Bricaud et al., 1981).

Consequently, Huntley and Clague (1996) used an IR bleach in place of a sunlight bleach for their thermal transfer correction. Since this bleach has been used on many other samples with no adverse effects found, it was used for the Long Point samples too. However, as Figure 2 clearly shows, the difference in luminescence between a sample exposed to natural sunlight and one exposed to an IR bleach was substantial for these samples. The explanation would appear to lie in the observation of Huntley and Clague (1996) that sunlight exposure, no matter how long, leaves electrons in traps sampled by 1.4 eV excitation. This, therefore, was the justification for subtracting the equivalent dose for sample D from the equivalent dose for sample C to obtain an age for sample C using the infrared bleach for the thermal transfer correction.

In order to further our understanding, an experiment similar to the bleaching experiment described by Huntley and Clague (1996) was performed on sample D. The delay between bleaching and measurement was 4-5 days. The results are presented in Figure 3 where it is seen that the same effects were found. The infrared bleach empties the principal traps (N + IR data), whereas sunlight exposure refills them (N + IR + sun data). For natural aliquots, sunlight exposure has little or no effect (N + sun data). The decrease in photons emitted with long exposures to sunlight appears to be real (Figure 3). It may be that the UV component of the sunlight bleach empties hard-to-bleach or "deep" traps and that the electrons are being re-trapped in the principal traps; if so, these deep traps would require sunlight exposures of much longer than 100 minutes to empty them.
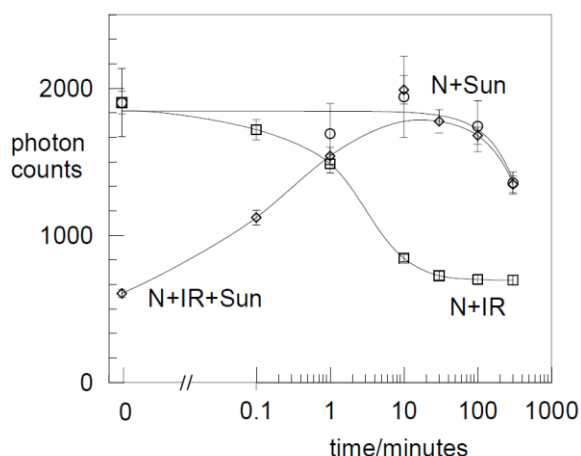
Figure 3: *Luminescence response to 1.4 eV excitation of K-feldspar aliquots from Long Point sample D, showing that a prior infrared bleach reduces the response whereas a sunlight bleach brings it back to the original level. IR stands for sunlight through an RG-610 filter as in Figure 1. N+IR+Sun means that aliquots were given a 300 minute IR exposure and then exposed to sunlight for the length of time given on the abscissa.*

The message is apparently clear. For this sample at least, a real sunlight bleach to evaluate the thermal transfer correction must be used. Thus, the equivalent dose measurements were repeated doing just that. The results are shown in Table 1, where it is seen that the two samples gave equivalent doses similar to each other. Nevertheless, sample D (the modern sample) gave an equivalent dose consistent with zero. Sample C yielded an age of 19 ± 12 years, consistent with the known age of 9 ± 1 years. Improvements in technology would likely allow these uncertainties to be reduced if this work were repeated today.

The results of these experiments informed the work of Wolfe et al. (2002) which was done shortly afterwards. They studied this matter by comparing equivalent doses obtained using an infrared bleach with those obtained using a sunlight bleach for the thermal transfer correction, for surface and near-modern dune samples. For three surface samples dated by Wolfe et al. (2002), the infrared bleach yielded equivalent doses on the order of + 0.1 Gy, while a sunlight bleach gave equivalent doses ranging from 0 to - 0.08 Gy. These figures give some idea of the uncertainty in equivalent dose arising from the uncertainty as to the appropriate bleach to use. For each of the three near-modern samples, subtraction of the equivalent dose for the surface sample gave results that were the same for both bleaches, yielding ages of 1 ± 7, 8 ± 8 and 38 ± 7 years.

It therefore appears to us that when dating young feldspar sediments, one requires a modern analogue

or source material for each sample and experiment to determine the appropriate bleach to use for the thermal transfer correction. Alternatively, a subtraction of equivalent doses method can be used (as shown in Table 1). Subtraction of ages is not appropriate.

The age resolution in these examples is about 10 years; with more intense excitation and better light collection (i.e., better technology), it should be possible to reduce this to about 1 year for the samples we studied. This level of resolution would be comparable to the best results that can be obtained for quartz from sediments (Madsen and Murray, 2009).

Conclusion

This work illustrates that in addition to the problems associated with the dating of "young" sediments identified by Madsen and Murray (2009), in the case of K-feldspars the "zeroing question" must also be considered. For a sample well bleached at about the time of deposition, its state depends on the spectrum of the bleach. A modern analogue is needed to determine this state, and consequently to determine the type of laboratory bleach to use if correct optical ages are to be obtained. One method of achieving this would be to choose a laboratory bleach that has no effect on the 1.4 eV-excited luminescence of the modern analogue.

It is possible that the bleaching spectrum just prior to burial depends on such things as the time of year, time of day, type of cloud cover, etc. In which case, since what matters is the exposure during the last few minutes before burial, it may not be possible to identify a true modern analogue. This could lead to a fundamental uncertainty in the optical age, maybe as much as a few decades.

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Reviewer
S. Tsukamoto

Reviewers Comment:
This article nicely shows that the light spectrum at the time of bleaching affects the residual (thermal transfer) of the IRSL signal, and the spectrum seems to be different from sample to sample. I think this paper also gives an insight in understanding the meaning of residual in the post-IR IRSL dating, which is now widely used.

On the luminescence signals of empty sample carriers

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Abstract

Luminescence dating is a leading technique for a large spectrum of Quaternary dating applications. Since the development of automated reader systems, handling great amounts of samples has become possible. A large quantity of data is produced in a short time and a detailed check of every single curve is often impractical. Therefore, it is important to be confident in excluding any kind of unwanted signal contributions, such as those from sample carriers. For commonly used types of steel and aluminium (Al) carriers from three laboratories, luminescence characteristics of spurious and radiation-induced signals are presented. TL and OSL emissions of discs show natural (Al) and regenerated thermally stable signals in the UV, UV-blue and red detection range. These signals have characteristic saturation doses of several hundred Gy. Furthermore, we demonstrate light insensitive signal components and phototransferred thermoluminescence (PTTL). Due to high scatter between discs, the proportion of unwanted disc signal contribution to the entire signal is difficult to predict, without direct measurement. The sources of these signals are possibly chemical compounds acting as luminophores or oxide layers (Al_2O_3 layers in case of Al discs).

Introduction

Since the beginning of thermoluminescence (TL) and later optically stimulated luminescence (OSL) dating much effort has been focused on avoiding unwanted signal contributions to those used for dating purposes (Aitken 1985, Aitken 1998). These contributions can originate from the sample itself or the measurement setup, e.g. the atmosphere during TL measurements or phosphorescence from the filters. The choice of the sample carrier is therefore of paramount importance for measuring a clean luminescence signal from the sample only.

The first TL measurements with relevance to recent dating applications were carried out with samples mounted on glass plates (Daniels et al. 1953). Several other materials were used as sample holders in the following decades, such as aluminium (Al), platinum, nickel, steel (e.g. Bøtter-Jensen 1997), or silver (e.g. Yawata and Hashimoto 2007). Only sparse information on the applicability of those substances is given in the literature (Berger 1982, Aitken 1998). The authors report on 'parasite' luminescence signals generated by Al holders. Today, most laboratories use stainless steel or Al carriers for sample placement in the commonly used Risø readers, usually termed as discs (flat plate) or cups (with depression). It should be mentioned, however, that one of the first materials used for TL dosimetric purposes was Al oxide (Al_2O_3) (Osvay and Biró 1980).

However, to our best knowledge, no systematic study has been published in the contemporary luminescence dating literature to prove the absence of parasite luminescence for commonly used sample holders. Standard measurement protocols do not check for unwanted signal contributions. Thus, the registered net luminescence (background subtracted) is routinely attributed in its entirety to the sample.

Since we found hints on spurious (i.e. non-radiation-induced) and dose dependent luminescence signals of a variety of different disc materials during experimental measurements, we conducted a study to investigate the features of these potentially problematic signals and their expected influence on dose determination. Therefore, we measured various sample carriers (Al and steel) from the luminescence laboratories in Oxford, Cologne and Bayreuth using different stimulation methods (TL, OSL, IRSL) and detection windows (UV: 340 Δ 80 nm, UV-blue: 420 Δ 30 nm, red: 625 Δ 25 nm and 630 Δ 30 nm). Furthermore, we examined the dose response characteristics and the bleachability of the signals as

well as the phototransferred thermoluminescence (PTTL, e.g. Furetta 2010, Kalchgruber 2002) of empty discs. An exemplary equivalent dose determination (SAR protocol, after Murray and Wintle 2000) of a well-studied sediment sample acts as reference point for assessing signal interference. Finally, an alternative method for cleaning the discs is presented and its effectiveness is tested.

Materials and methods
Sample carriers
To investigate the luminescence behaviour of sample carriers, three kinds of empty discs or cups commonly employed in laboratories were tested:
1) Stainless steel discs used in Oxford and Cologne (Cr-Ni steel V4A, diameter 9.8 mm, thickness 0.5 mm)
2) Al discs used in Oxford, Cologne and Bayreuth (unknown Al composition, diameter 9.8 mm or 9.6 mm, thickness 0.45 mm)
3) Al cups, manufactured in Bayreuth (Goodfellow Al foil, purity 99.0 %, diameter 12.0 mm, thickness 0.1 mm)

From each kind of discs or cups, at least five exemplars were measured. We chose all discs randomly from the 'clean disc box' (for the applied cleaning procedure, see below) and measured them without further chemical and physical treatments. Regarding Al discs we tested both used and new discs. Al cups from Bayreuth were new, because cleaning without damaging is difficult due to their low thickness.

Disc cleaning procedures
To test the effects of disc cleaning procedures on spurious and dose-dependent signals two methods were applied on used discs in Cologne.
1) Steel and Al discs were cleaned in an ultra sonic bath in addition to mechanical rubbing (sponge) and washing with rinsing agent to remove remaining silicon oil. Afterwards, the discs were flushed with distilled water and purged in acetone.
2) A mechanically more severe procedure was applied to remove the oxidized layers from steel and Al discs. Several tens of discs were placed in a bottle of chalk suspension and kept on the shaking table for polishing for 24 h, or 72 h if they were scrubbed with scouring agent in advance. Washing with distilled water afterwards prevents measuring spurious signals from carbonates. The loss in mass is negligible.

Measurement setup
The luminescence measurements were carried out on different Risø DA-12, DA-15 and DA-20 readers in Cologne, Oxford and Bayreuth, equipped with a standard bialkali photomultiplier tube (EMI 9235QB). In addition, for the red detection a cooled trialkaline photomultiplier tube (EMI 9658B) described in Fattahi and Stokes (2005) was used in Oxford. The luminescence was measured placing the following filters consecutively between the disc and the photomultiplier:
1) Hoya U340 (7.5 mm, 340 Δ 80 nm),
2) Combination of Schott GG400 (3.0 mm), Corning 7-59 (2.0 mm), Schott BG39 (1.0 mm) and HA3 (4.0 mm): UV-blue detection centred at 420 nm,
3) Chroma D410/30x interference filter for UV-blue detection (410 Δ 30 nm) for IRSL,
4) Chroma D630/60 M (630 Δ 30 nm, Cologne) or Omega D625/DF50 (625 Δ 25 nm, Oxford) interference filters for red detection, respectively.

The heating rates were set to values of 2 or 5 K s^{-1} and the measurement chamber was flushed with N_2 for two minutes before each measurement exceeding 160°C, except where indicated otherwise. Maximum temperatures for UV TL (UVTL) and UV-blue TL (BTL) measurements were 500°C and for red TL (RTL) 450°C. The background for each measurement was recorded immediately afterwards. For OSL measurements of the UV emissions, blue LEDs (470 Δ 30 nm) and for IRSL measurements, infrared LEDs (870 Δ 40 nm) were used.

The discs received radiation doses from ^{90}Sr/^{90}Y β-sources delivering around 5 Gy min^{-1} (DA-12 and DA-15) or 7 Gy min^{-1} (DA-20). These dose rates are usually calculated for mineral grains mounted on the discs and not for the discs themselves. Therefore, the dose rates are approximations.

Dose response measurements
For dose response measurements, blank discs were irradiated with incremental doses up to ca. 1 or 2 kGy and subsequently TL and OSL were measured. A standardized luminescence efficiency value, with units cts (K Gy)$^{-1}$ for TL and cts (s Gy)$^{-1}$ for OSL, respectively, allows comparing measurements with differing parameters, e.g. heating rate or measurement channels (suggested by M. Krbetschek, pers. comm.). The TL signal was integrated over the thermally stable range of 300-400°C for UVTL and BTL. To avoid the influence of increasingly noisy net signals above 350°C, the integration interval for RTL was lowered to 250-350°C.
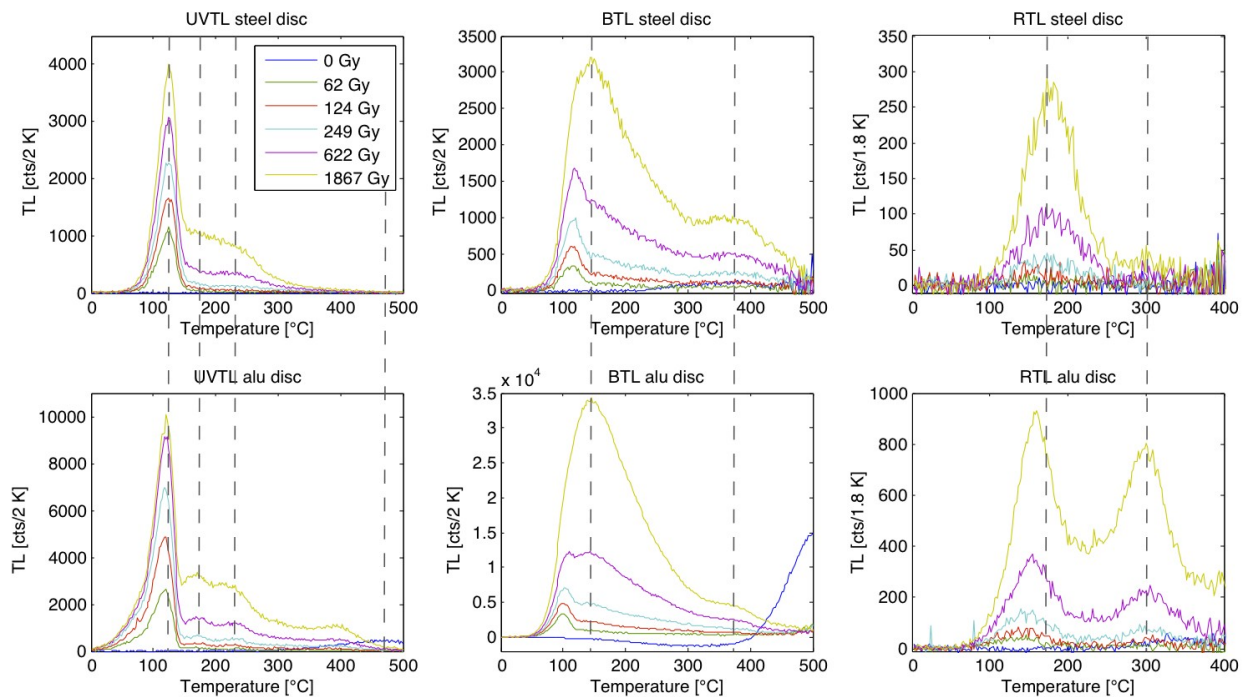
Figure 1: *Natural and regenerated TL signals from single steel and Al discs for different detection wavelengths. All measurements were performed in Cologne and carried out at 5 K s⁻¹. For further details, see text.*

The OSL/IRSL decay curves were measured at 125°C read temperature after a preheat of 260°C (OSL) for 10 s and read temperatures of 50°C after a preheat of 250°C for 60 s or 270°C for 10 s (IRSL).

Experimental details and results
Thermoluminescence signal
 - Steel discs
Steel discs from the Cologne and Oxford laboratories were heated to 500°C (UVTL and BTL) or 450°C (RTL) and TL was measured first without irradiation, then after incremental β-doses. Between the dose steps, a test dose was given to record potential sensitivity changes.

No signal above background could be detected for non-irradiated steel discs except for a small 380°C peak in the blue range (Fig. 1).

Significant TL signals resulted from exposure to ionizing radiation. UVTL and BTL showed a strong 110°C peak, whereas for BTL this peak slightly shifts towards higher temperatures or is superposed by another peak on its high temperature shoulder for high doses (> 1 kGy). At higher temperatures, we observed at least two other peaks which form a broad continuously decreasing shoulder between 150 and 300°C in the UV range. Little UVTL signal is

detected above 300°C. A similar shoulder occurs in the blue window, followed by a distinct peak centred at 380°C in the high temperature region. In the red detection window, steel discs are far less sensitive to irradiation. We only observed signals above background noise for doses > 250 Gy (peak at 180°C).

 -Al carriers
We applied the same measurement routine to Al discs from the Oxford and Cologne laboratories. In contrast to steel discs, Al discs yielded a notable TL signal without artificial irradiation. The peak positions for this spurious signal are 480°C (UV) and ≥ 500°C (UV-blue) with count rates extending from a few hundred cts K⁻¹ in the UV and red (no peak observable) up to several thousand cts K⁻¹ in the blue detection window. For BTL, however, difficulties with the background subtraction led to a negative net signal and the 'natural' peak may also suffer from this problem. The natural UVTL and BTL signal is not depleted completely by measurements up to 500°C. Hence, remaining trapped charge appears to cause a spurious signal also in subsequent measurements (data not shown).

Following β-irradiation, the 110°C UVTL peak of Al discs appears at slightly lower temperatures in
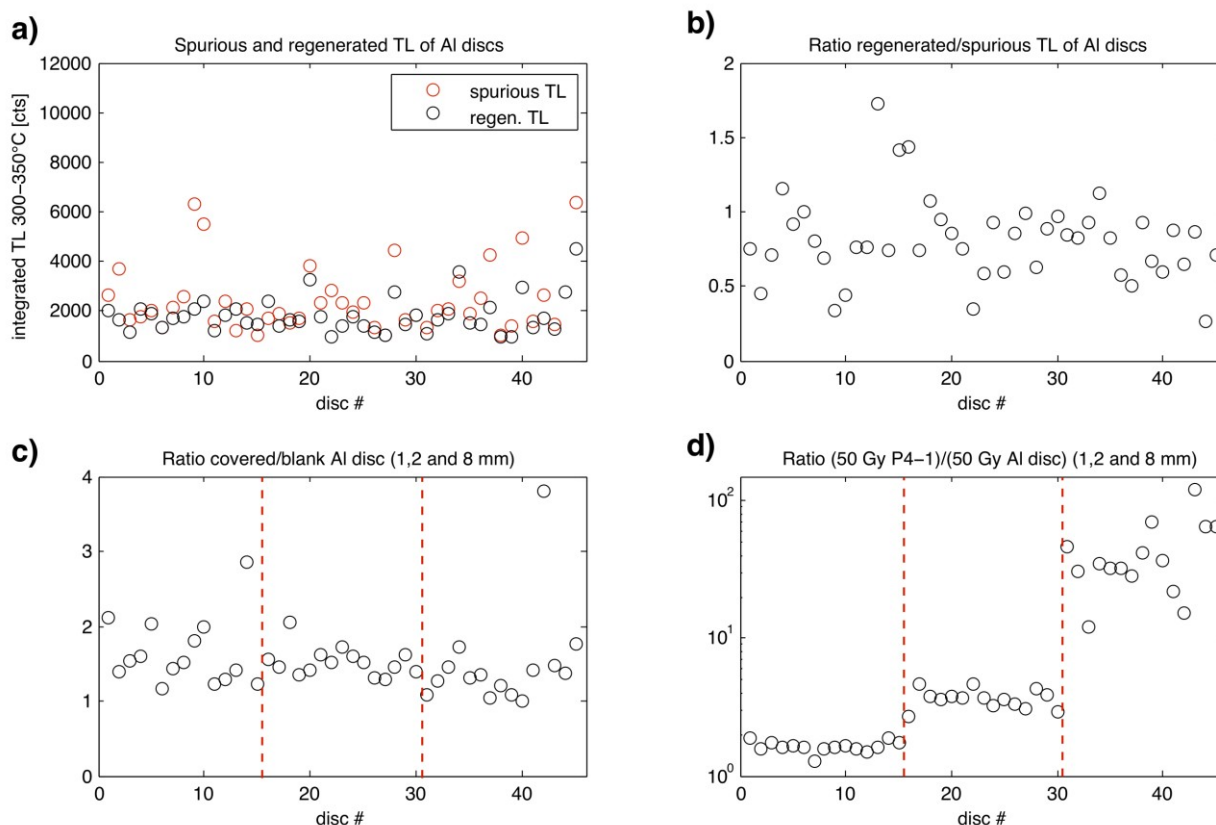
Figure 2: *Results of the experiment to measure the influence of grain coverage on unwanted signal contributions of used Al discs (UV detection range). Experimental conditions and detailed descriptions are given in the text.*

comparison to steel due to the higher thermal conductivity of Al and thus reduced thermal lag between heater plate and upper disc surface. We observed three other UVTL peaks at 180, 220 and 400°C for β-doses > ca. 250 Gy (Fig. 1). Similar to the BTL emission of steel, Al discs yield a 110°C peak that is surpassed in growth by a peak at 140°C for doses > 600 Gy. A further small BTL peak of Al is centred at 380°C. The RTL emission of Al discs is dominated by two maxima at 150 and 300°C. The low temperature peak seems to correspond to the RTL peak observed for steel discs, whereas steel discs only show a very weak 300°C peak.

The positions of the TL peaks in the various detection windows for steel and Al discs are roughly the same for all measured discs. However, the intensities of the luminescence emissions and the relation of peak heights vary considerably between individual discs.

We also detected low spurious TL signals in the UV range for Al cups as a slowly growing shoulder from 200 up to 450°C. Following β-irradiation up to ca. 1 kGy we found UVTL peaks at 110°C and at around 400°C for doses > 500 Gy (see supplementary data at

www.aber.ac.uk/ancient-tl). BTL glow curves of several Al cups exhibit a very weak peak at 110°C after the highest regeneration dose (> 1 kGy). In general, used and cleaned Al and steel discs produce much higher spurious and regenerated signal intensities in the UV and blue detection range than new Al cups. RTL emissions of Al cups were not investigated.

Fig. 2 presents the results of an experiment to measure the influence of grain coverage on unwanted signal contributions of used Al discs. All signals were recorded in the UV detection window. Fig. 2a shows the integrated TL (300-350°C) of the spurious signal and the regenerated signal after 50 Gy β-irradiation for 45 discs. The ratio of these signals for each disc is plotted in Fig. 2b. The annealed discs were then irradiated with 50 Gy and immediately covered with annealed quartz grains (BT781, loessic sample from Nussloch, Germany, unit P4-1, 100-200 μm, heated at 500°C for 1 h) using masks of different diameter (discs 1-15: 1 mm; discs 16-30: 2 mm; discs 31-45: 8 mm). Fig. 2c shows the ratio of the regenerated TL signal of grain-covered discs and uncovered discs

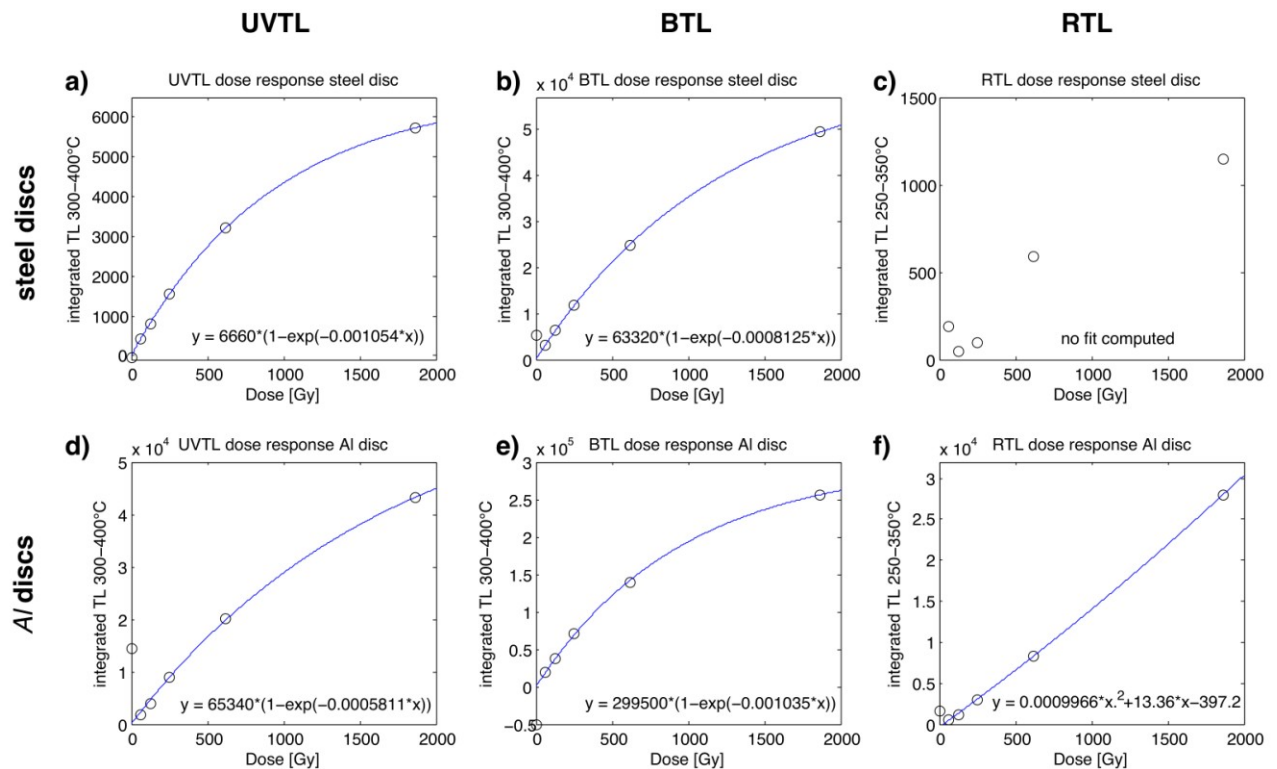**Figure 3:** *TL dose response curves of steel and Al discs for different emissions. The investigated discs are the same as shown in Fig. 1. Integration limits are as indicated on the ordinate. For further details, see text.*

(measured after cleaning the discs individually afterwards). These measurements indicate that the effect of light shielding through grain coverage is negligible. The fact that the ratio is higher than 1 is probably a result of the cleaning procedure. Fig. 2d shows the ratio of the 50 Gy irradiated quartz sample (mounted on Al discs using the same order and mask diameters as in Fig. 2c) and the regenerated signal after the same dose of the same discs without quartz layer.

-Dose response

Growth curves of UVTL and BTL emissions of steel discs can be fitted to single saturating exponentials with characteristic saturation doses (D_0) of ca. 950 Gy and 1.23 kGy, respectively (Fig. 3). For low β-doses (< 200 Gy), the dose response can be approximated with a linear fit. In this range, the dose response amounts to about 3 cts (K Gy)$^{-1}$ for UVTL and 24 cts (K Gy)$^{-1}$ for BTL. For higher doses, those values decrease due to saturation. The RTL signals in the thermally stable region are too low to estimate their dose response. The UVTL and BTL growth curves of Al discs show similar behaviour with approximately linear growth up to doses of 500 Gy (UVTL) and 300 Gy (BTL) and characteristic saturation doses of 1.72 kGy and 970 Gy,

respectively. In contrast, the RTL Al emission reveals supralinear increase for doses < 2 kGy. Up to onset of saturation, the dose response of Al discs can be expressed as ca. 20 cts (K Gy)$^{-1}$ for UVTL, ca. 180 cts (K Gy)$^{-1}$ for BTL and ca. 4-6 cts (K Gy)$^{-1}$ for RTL emissions.

Test dose monitoring shows little sensitization for steel discs and Al discs and cups. The effect is most distinct for the 110°C TL peak in the UV and blue detection window and the BTL high temperature peaks of Al discs, but not quantifiable for other high temperature peaks.

-Growth rate of the spurious signal

As spurious and dose-dependent signals of Al discs may derive from oxide layers, the signal response was investigated for different durations of oxygen exposure. Fresh chalk-polished Al discs (Cologne, procedure see above) were stored in an acetone bath in a closed bottle to prevent contact with oxygen. Batches of 5 discs were successively removed at defined times. The remaining time to the UVTL measurements is then the oxygen exposure time. Those were set to 10 minutes (approximated 0 days), 2, 5, 20 and 41 days. The TL signal was integrated over the range 250-450°C.

(a)

(a)

(b)

(b)
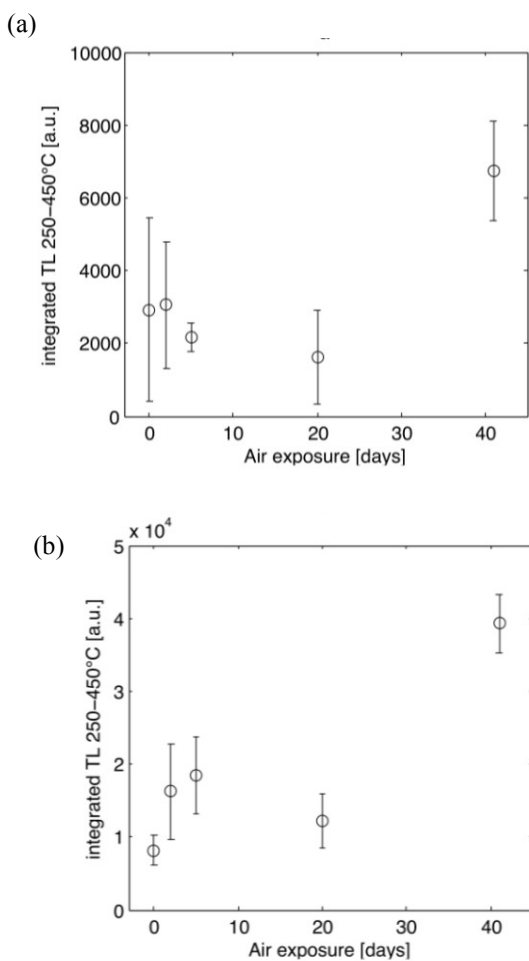
Figure 4: *Growth of (a) spurious and (b) 250 Gy regenerated UVTL signals of Al discs with air (oxygen) exposure duration. The error bars represent the standard deviation of 5 discs each.*

Figure 5: *Natural (0 Gy) and regenerated OSL signals of single (a) steel and (b) Al discs after preheat (260 °C for 10 s). The inset in (a) shows the residual UVTL measured after β-irradiation of 1867 Gy and 40 s blue optical stimulation (470 Δ 30 nm). For further details, see text.*

We observed no growth, within measurement uncertainty, up to oxygen exposure durations of 20 days due to high scatter of the spurious TL signal between discs (Fig. 4a). For longer contact with air, the discs showed a substantially increased signal. In order to check whether there is a correlation between spurious and regenerated signal, all discs received a β-dose of 250 Gy after initial spurious signal readout (Fig. 4b). We calculated the ratio of both signals in the same temperature interval. The ratio values (regenerated signal/spurious signal) denote high disc-to-disc scatter and suggest that both signals are not closely correlated (data not shown). However, Fig. 4 indicates that longer oxygen exposure durations generate both stronger spurious and radiation-induced TL signals.

Optically and infrared stimulated signals -OSL

All steel discs were preheated to 260°C for 10 s at a rate of 5 K s^{-1} to simulate conditions equal to routine OSL measurements. We observed negligible OSL signals without artificial irradiation, but count rates up to 20 cts (s Gy)$^{-1}$ for β-doses < 100 Gy (Fig. 5). Pronounced scatter of signal intensity and growth for single discs is typical. Al discs show OSL signals of about 2 cts (s Gy)$^{-1}$ after a 260°C preheat for 10 s up to the highest regeneration dose. Al cups produce OSL signal above background in the range of 10 cts (s Gy)$^{-1}$ in the first one or two measurement channels after irradiation, indicating a rapidly decaying signal component (supplementary data). As for steel discs, initial OSL signal intensities of Al cups differ considerably from cup to cup. Due to low signal-to-
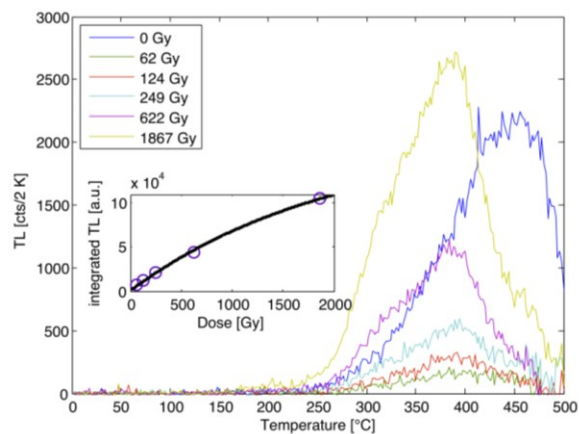
Figure 6: *Dose response of residual UVTL after blue optical stimulation (470 Δ 30 nm) for 40 s for one Al disc. The inset displays the signal growth for integrated TL (340-440°C) as a function of the regeneration dose, fitted to a single saturating exponential (y = a*(1-exp(-b*x)), where a = 1.74E+05, b = 4.95E-04 and x is dose in Gy).*

noise ratios of the test dose signals we cannot provide any information on potential sensitivity changes for OSL of steel and Al sample carriers.

-IRSL

We also checked the IRSL signals from discs during common IRSL (feldspar) measurement conditions. After a 270°C preheat for 10 s or 250°C for 60 s IRSL was measured at 50°C. Steel discs as well as Al cups completely lack IRSL signals (natural and after irradiation). Al discs reveal a dose dependent, slowly decaying signal with count rates in the range of 2-5 cts (s Gy)$^{-1}$, but no natural IRSL signal (supplementary data).

-Residual TL

After OSL signal depletion and subsequent TL measurement we detected a non-bleachable or slowly bleachable signal component at high temperatures for both steel and Al discs. An example of the residual UVTL of a steel disc after 1867 Gy β-irradiation is shown in the inset of Fig. 5a. In general, the residual UVTL signal of Al discs shows higher count rates than that for steel discs (Fig. 6). Furthermore, the peak positions of natural and regenerated residual glow curves of Al discs differ significantly. As the empty discs are handled under room light conditions (and therefore light sensitive components should be removed), it is probable that the 0 Gy peak in Fig. 1 (lower left) is identical to the 0 Gy peak shown in Fig. 6. The dose response of Al residual UVTL is approximately linear up to β-doses of 500 Gy, but shows exponential saturation for higher doses ($D_0 \approx 2$ kGy, Fig. 6).

-Photo-transferred thermoluminescence (PTTL)

For the measurement of the PTTL the discs were first irradiated with 250 Gy, twice annealed to 500°C, and subsequently two cycles of OSL and UVTL were measured without further irradiation. The OSL signals (max. about 1500 cts s^{-1}) of the Al discs show an initial increase before decaying exponentially as expected from a typical OSL curve. This is contrary to the OSL signals observed after lower preheat temperatures. Optical stimulation induced a weak PTTL signal (ca. 60 cts K^{-1}) with a peak at 190°C (Fig. 7c). The dose response of the PTTL signal was not investigated. Our results indicate that steel discs are free from PTTL signals.

Influence of cleaning procedures

Since the BTL emissions of both steel and Al discs showed the strongest luminescence, we used them as an indicator of signal reduction attributed to the chalk cleaning procedure. The polishing of steel discs with chalk reduced the spurious and dose-dependent BTL signals by about 40-50 % but did not suppress them completely (data not shown). Though natural signals are lacking, we still observed a prominent 110°C peak of about 10 cts (K Gy)$^{-1}$.

Al discs showed a natural BTL signal with a peak at 450°C (ca. 100-200 cts K^{-1}) and a regenerated emission of 1-2 cts (K Gy)$^{-1}$ after air storage of several days. Accordingly, the disturbing signal can be effectively reduced but not fully eliminated.

Influence of the disc signals on D_e determination

In order to test the effect of unwanted signal contributions of sample discs during an equivalent dose determination, a standard SAR protocol was carried out (Murray and Wintle 2000) using a coarse grain quartz sample (BT781) whose reliable luminescence characteristics are known (Zöller et al. 1988, Tissoux et al. 2010). Half of 10 steel and half of 10 Al discs were annealed (500°C for 30 s), the other half remained untreated prior to grain deposition (aliquots of 2 mm diameter). A preheat temperature of 240°C, a cutheat of 220°C and a read temperature of 125°C were chosen and a hot bleach (OSL at 280°C for 40 s) was applied at the end of each SAR cycle. Excluding one outlier (annealed steel disc no. 4, supplementary data Fig. 3a), the mean D_e values determined with natural discs are ca. 20 % (steel, n = 4) and 35 % (Al, n = 5) higher than those measured with annealed discs (n = 5 each). This indicates that the natural disc signal contributes verifiably to the initially measured geological signal of the sample. In addition, the measurement uncertainty of each single D_e value increases substantially if the discs are not annealed before the measurement. This effect is more distinct for steel
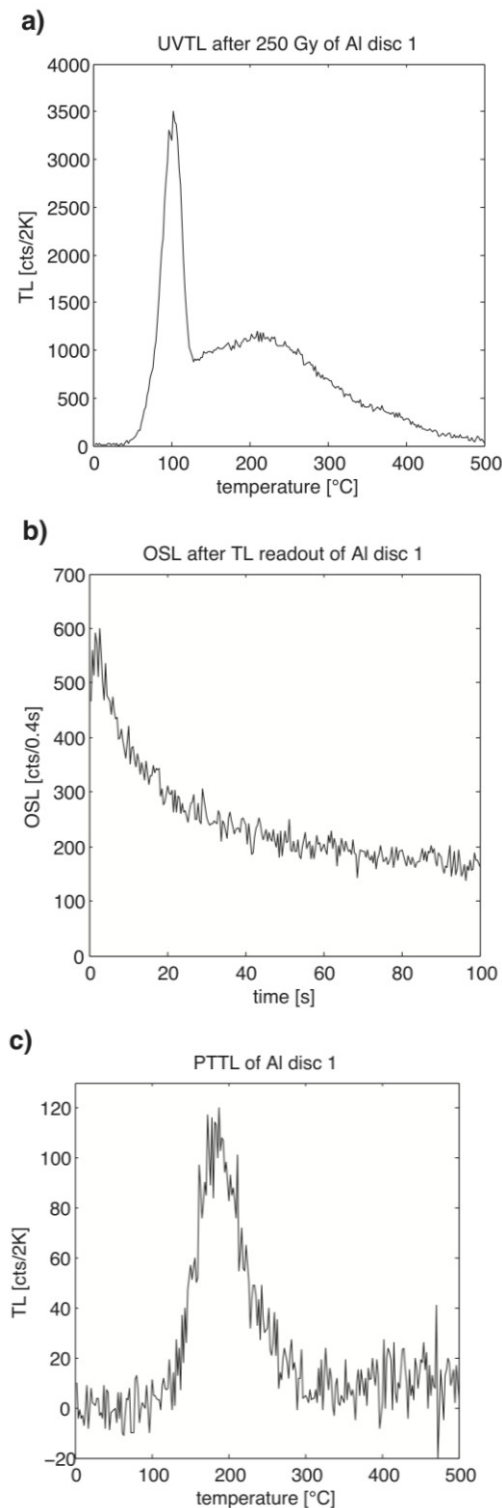
Figure 7: *PTTL measurement in the UV of an Al disc after a β-dose of 250 Gy was given: a) background subtracted TL measurement up to 500 °C after irradiation, b) subsequently recorded blue stimulated OSL signal, c) photo-transferred TL signal after optical signal depletion.*

than for Al discs, i.e., the mean uncertainty increases by about 90 % and 72 %, respectively, if the discs remain untreated prior to the measurement. Differences in the recycling ratio for annealed and non-annealed discs are not significant. However, statistical validity is limited by the small number of measured aliquots and further measurements are needed (supplementary data).

Discussion
The striking difference of TL peak temperatures for spurious and radiation-induced signals from Al discs (Fig. 1) suggests that different kinds of traps are involved in charge storage, but that potentially the same suit of recombination pathways are used owing to the common emission band (although detailed spectral measurements would be needed in order to confirm this). We observed several UVTL and BTL peaks between 100-400°C that cannot be distinguished without curve fitting. The RTL glow curve on the other hand consists of only two visually distinguishable peaks, suggesting involvement of fewer types of charge traps. In contrast to Cr-Ni steel, the charge occupation sites connected to the spurious and the dose-dependent signal of Al both include non-bleachable (insensitive to visible light) residual TL, suggesting that full signal erasure may only be possible by heat treatment.

Due to the high affinity of Al to O, aluminium oxide layers build up within a few minutes when the (hypothetically) clean disc is exposed to oxygen. These layers reach thicknesses of up to 30 nm on pure Al at high temperatures (> 300°C) (Ostermann 2007). Diffusion of O is then not possible anymore and the layer protects against continuing corrosion (Bargel and Schulze 2008). Possibly, intrinsic defects of Al_2O_3 are responsible for part of the luminescence signal of the Al discs.

Similar to silicon dioxide (SiO_2), Al_2O_3 can also facilitate vacancies and interstitials (due to impurities) in its molecular structure, such as oxygen vacancies which in quartz act as hole traps (Kelly and Laubitz 1967, Preusser et al. 2009). Oxygen vacancies are described in Al_2O_3 as well, but acting as traps for electrons instead of holes (Hakanen et al. 1997). The remarkable luminescence sensitivity of Al_2O_3 is commonly used for dosimetric applications, where the dosimeters are additionally doped with e.g. C, Fe or recently with Tb and Tm (Osvay and Biró 1980, Aypar 1986, Akselrod et al. 1993, Barros et al. 2010). Luminescence properties are then, however, strongly influenced by the elements used for doping.

Stainless steel, as used for the discs, contains small amounts of carbon and silicon which might together

form luminophores (carbides) (pers. comm. M. Krbetschek). It cannot be excluded that those contribute to the luminescence signal of the investigated steel discs. Furthermore, we cannot rule out that a small layer of Cr and Fe oxides (a few atom layers thick, arising from the admixture of Cr to the alloy) on Cr-Ni steel, causes part of the dose-dependent luminescence signal (Blasek and Weihert 1979, Bargel and Schulze 2008).

Although we observed minor dissimilarities between glow curves of steel and Al discs, the main peaks seem to correspond for both materials and all measured emissions (Fig. 1). This fact suggests that there is one common source for all disc materials.

Previous studies dealing with spurious luminescence from silicone oil revealed ambiguous findings. In combination with Al discs, considerable spurious TL signals were found, but not with steel discs (Murray 1981). Vandenberghe et al. (2008) reported that silicone oil (Willy Rüsch GmbH) potentially contributes to disturbing OSL signals. In both investigations, sample carrier materials are not described in detail. In contrast, Fuchs (2001) found silicone oil (from the same manufacturer) on Al cups to be free from spurious and radiation-induced OSL signals. The fact that Vandenberghe et al. (2008) observed disturbing signals of used and cleaned steel cups indicates that adherent silicone residues are a potential source. This would imply that conventional cleaning techniques (as described above) are not capable of removing silicone oil completely. As silicone is made up of silicon and oxygen atoms (among others), there might be chemical reactions during heat treatment or storage resulting in some kind of luminophore. Spurious signal levels of new sample holders possibly depend on the cleaning procedure prior to first use (if cleaned at all) because in some workshops lubricant oils (containing forms of silicone) might be used for disc or cup manufacturing. Beside the technique mentioned above, there are also alternative methods to remove silicone relics and to clean the discs, such as using propanol or butanone (Vandenberghe et al. 2008) or short acid treatments (e.g. 1-2 % aqua regia or diluted phosphoric acid for a few minutes).

In summary, there are two main sources potentially causing the observed disc signals: luminescent chemical compounds of the carrier material itself, or some kind of contamination resulting from grain deposition (silicone oil) that resists the cleaning procedures. Similar glow curves for both investigated materials favour contaminations as main source. However, further investigation is needed for conclusive determination of the origin of the observed signals.

For TL measurements it is clearly advisable to use Al cups of the type described above or steel discs, despite lower thermal conductivity and higher costs for steel discs. Al discs show lower unwanted signals with regard to OSL measurements. During our investigations we applied relatively high β-doses. The majority of luminescence samples require less irradiation for D_e determination, so that the effect of the disc signal is less pronounced. However, when measuring very dim samples or for basic studies, one should always be aware of the 'disc problem' and its influence on weak luminescence signals.

The contribution of the disc signal to the entire recorded signal depends only weakly on light shielding by the grain-covered area of the disc for coarse grains (Fig. 2), but shielding is likely to be more effective for fine grain layers. Correction for disc influence requires careful investigation of each individual disc, especially in the case of Al discs. Consequently, alternative and chemically more inert disc materials (nickel, silver, rhodium, gold etc.) should be investigated in detail.

Conclusions
Spurious and dose-dependent TL and OSL emissions from commonly used sample carriers were investigated in this study. We observed:
1) Al discs showed significant TL and OSL signals in the UV, UV-blue and red detection range.
2) Steel discs showed no significant spurious but dose-induced signals in the UV and UV-blue windows.
3) For Al cups (from Bayreuth) we found no spurious signals for UVTL and generally low sensitivity to irradiation.
4) The provided cleaning procedures for discs (Al and steel) can reduce spurious and dose-dependent signals but cannot eliminate them completely.
5) For most bright samples, the influence of disc signals is expected to be negligible but further investigation for the case of single grain discs is needed.

These phenomena seem to be widespread among laboratories as they were observed in at least three luminescence dating facilities and with various kinds of sample holders. However, this paper is not a comprehensive luminescence study of specific disc materials. The influence of spurious and dose-dependent signals on particular measurements is different for each disc and sample and difficult to assess because of highly differing characteristics

between discs. In short, the résumé of this paper is to alert the reader to the problem of unwanted luminescence contributions from the sample holder and to advise careful measurement of these signals when measuring relatively young and/or dim samples (including single grains).

Acknowledgements

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Reviewer

R. DeWitt

Thesis Abstracts

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Thesis Title: Numerical modelling of environmental dose rate and its application to trapped-charge dating
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Accurate estimation of environmental dose rate is essential for high-resolution trapped-charge dating. Beta and gamma emissions from simulated sediments containing radioactive uranium, thorium and potassium are modelled in contexts that are spatially heterogeneous. Dose rate was modelled using Monte Carlo radiation transport codes MCNP and PENELOPE. A number of key issues that affect dose rate evaluation are examined and updated corrections are calculated.

Granular structures used for geometrical input into the models were simulated using randomly packed ellipsoids. The pair correlation function and chord length distributions were derived. The effects of water content on dose rate were modelled and compared with cavity theory. Apart from activity dilution, the variation of grain size or water content was shown to be significant for gamma radiations due to the transition from charged particle equilibrium. The standard correction for beta dose rate due to grain size was found to be satisfactory although sensitivity to grain shape and material should be taken into account.

Dose rate modeling was applied to three dating studies of early human fossils: Skhul V, Israel skull; Hofmeyr, South Africa skull and the Forbes' Quarry, Gibraltar skull. The spatial modelling was implemented using computerised tomographic (CT) images and dose rate found to be modified significantly by the presence of the skull in the sediment. Time evolution of the dose rate was examined for the latter two skulls and dates of 36±3ka (Hofmeyr) and 55-95ka (Forbes' Quarry) were calculated.

This thesis is available as a PDF from: www.aber.ac.uk/ancient-tl

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The dispersal of anatomically modern humans (AMH, *Homo sapiens*) out of Africa is a pivotal event in the global expansion of our species. As the major dispersal routes cut through the vast Arabian Desert, favourable climatic and environmental conditions with sufficient supply of surface water must have been critical factors for AMH expansion into Arabia and beyond. The modern climate of Arabia is governed by subtropical high pressure with very scarce moisture, generally delivered by the northwesterly winds. A limited area in the far south experiences tropical summer precipitation under the influence of the Indian Summer Monsoon. From regional climate archives it is known, that the northern limit of monsoon rainfall was highly variable in the past, reaching latitudinal positions far north of its present summer position. How far north, however, remains unknown because of large gaps between clusters of existing climate archives. Relict lake deposits scattered among the dunes are the primary repository of past climate conditions in Arabia due to their potential to record environmental conditions during humid periods. Yet, until a few years ago, dating of Pleistocene relict lake deposits was limited to contaminated radiocarbon ages of the 70s, systematically underestimating the real ages. Hence, this PhD study focused on re-dating the relict lake deposits using optically stimulated luminescence (OSL). Owing to early signal saturation, the OSL dating limit was reached at ages of ~100 ka and required the testing of a novel measuring protocol to expand the dating range (TT-OSL). Application of the approved protocol on samples from south-western Saudi Arabia revealed humid phases with a savannah like environment at ~80 ka (MIS 5a), ~100 ka (MIS 5c) and ~125 ka (MIS 5e). A second study on fluvial and lacustrine deposits in the interior of Oman, suggested a short humid period sometime between 132 and 104 ka. A final study on relict lake deposits from northern Saudi Arabia indicated humid periods

centred at ~410, 320, 125 and 100 ka. A comparison with speleothem records of the southern Negev desert shows a very similar timing for humid periods. The agreement with the southern Negev, and disagreement with the central and northern Negev, makes a Mediterranean moisture source unlikely and suggests a tropical source, possibly from an enhanced African Monsoon. Favourable environmental conditions during all these periods allowed AMH to migrate across Arabia. Between ~75 and 10.5 ka arid conditions prevailed and turned Arabia into a natural barrier for human dispersal. Thus, expansion of AMH into Arabia and beyond must have taken place before 75 ka, possibly in multiple dispersals.

Author:	Tobias Lauer
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Fluvial aggradation and erosion is triggered by mechanisms like climate variations, tectonics, sea-level change and human impact. The Rhine system is one of the largest drainage systems in Europe and its sediments therefore provide important information about the palaeo-climate and tectonic evolution of Central Europe. To understand at what time for instance tectonic impulses or changes in climate, regulating sediment supply and sediment preservation, occurred, a reliable chronology for the fluvial sediments is mandatory.

In this thesis Luminescence and Infrared Radiofluorescence (IR-RF) dating were applied to fluvial deposited collected from the Heidelberg Basin (northern Upper Rhine Graben) and Lower Rhine Embayment, Germany. Optical dating of fluvial deposits is still challenging because in many cases the luminescence or IR-RF signal was not reset completely before burial. Further problems like feldspar impurities disturbing the quartz-OSL signal or a weak luminescence signal (bad signal to noise ratio) of quartz can occur. Potassium feldspar gives much more luminescence (higher signal intensity) but it is affected by anomalous fading which has to be corrected. Fading corrections are problematic especially for old sediments. One aim of this thesis was to better overcome these problems and to contribute to the methodological progress among optical dating with focus on fluvial deposits. To do so, different dating approaches were tested on fluvial samples for which age control is available. Furthermore it was intended to establish a better chronological framework for Holocene, Upper and Middle Pleistocene fluvial sediments from the Rhine system.

The sediments in the Heidelberg Basin are characterized by heterogeneous, gravel-rich layers (cold stage deposits) and intercalated fine-grained layers hosting organic material (so called Interlayer). The latter were deposited during warmer climate periods. It was intended to obtain a reliable chronology for both, the warm stage and cold stage deposits. The quartz OSL ages demonstrate that the upper fluvial units (sediments above the Upper Interlayer) were deposited during the Last Glacial period (Weichselian).

To frame the sedimentation age of the Upper Interlayer and sediments below, IR-RF was used. For the Upper Interlayer the IR-RF ages point to a sedimentation age of ~ 300 ka. This shows that there is a huge chronological gap between the Weichselian fluvial sediments and this interlayer. For the fluvial units below the Upper Interlayer it was possible to date up to ~ 640 ka (100 m core depth at the Viernheim drilling site). For the Middle Pleistocene differences in the intensity of subsidence of the Heidelberg Basin mainly regulated the fluvial aggradation. During times of increased subsidence, accumulation space was created and the sediments could be preserved. Hence, the IR-RF ages help now to better estimate the timing of subsidence of the Heidelberg Basin.

For the Lower Rhine the luminescence ages now yield a higher chronological resolution for the studied sections (mainly Lower Terrace) and help to better understand the past fluvial dynamics of the Rhine. It could for instance be shown that fluvial aggradation of many meters of sediments can happen within a very short time period. Samples which were taken with a vertical distance of > 5 meters from a section at Monheim-Hitdorf all yield equal OSL ages.

For some of the samples taken at the LRE independent age control was provided by Laacher See pumice (age ~ 12.9 ka). For these samples quartz OSL and feldspar measurements were conducted and the results were checked against the age control. For feldspar dating, the IRSL at 50°C was measured and after this, the post-IRSL signal was detected stimulated with red LED at 225°C (pIRIR signal).

Next to this a new protocol was applied which includes the detection of a feldspar signal stimulated with yellow LED at 260°C after depleting the IRSL (50°C) signal (pIR-YOSL). The latter protocol was developed within this thesis.

It turned out that quartz dating worked well for the fluvial samples under study. The ages are in agreement with the age control and also the feldspar dating results agree with the quartz ages.

Further samples were taken from a Roman harbour exposed at Cologne. For these samples Roman artefacts gave a very precise age control. This gave the opportunity to test different statistical approaches for these incompletely bleached sands. Furthermore, different protocols were applied to minimize the feldspar signal in contaminated quartz and it was shown that pulsed OSL but also an IRSL bleach at 225°C prior to the detection of the quartz (blue stimulated) signal have very good potential to obtain a purer quartz signal.

The results which are presented in this thesis show that luminescence and IR-RF dating are powerful tools to establish a reliable chronological framework for fluvial deposits. There are still challenges (e.g. which statistical approach one should apply if samples are incompletely bleached). Nevertheless, the here applied dating approaches yielded reliable ages as for example demonstrated by quartz and feldspar ages from the Monheim-Hitdorf site (Lower Rhine). It was also of high relevance to point out that IR-RF dating could successfully be applied to samples being older than 600 ka.

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Obituary

John Russell Prescott
(May 31st 1924 - September 1st 2011)

John Russell Prescott, affectionately known to his colleagues and students as "Prof", was born in Egypt on May 31st, 1924, in sight of the pyramids of Giza. However, John spent only the first three months of his life there as his family was about to move to Adelaide, where his father was to become the first Professor of Agricultural Chemistry at the Waite Agricultural Research Institute and later, in 1938, the Director of that Institute. While his father was Director, the family lived in the historic Urrbrae House, of which John continued to be a dedicated Friend until the time of his death. He attended Scotch College where he excelled both academically and on the sports field, and in 1942 entered the University of Adelaide to study Physics. He graduated in 1945 with the degree of BSc (Honours) and in the same year became engaged to Josephine Wylde. He moved to Melbourne to undertake his PhD studies, and he and Jo were married in 1947. In 1948 John was elected an Associate of the Institute of Physics, and in April 1949 was awarded his PhD for his dissertation on cosmic ray showers and bursts. This was one of the first two PhDs in Physics to be awarded by the University of Melbourne. During his time in Melbourne John also worked as one of the two foundation employees of the nascent Australian Atomic Energy Commission.

Apparently not satisfied with one doctorate degree John, now married to Jo and starting a family, then travelled to Oxford to take up a scholarship awarded by Christchurch College to study for the degree of DPhil.. During his time in England he took his wife and young son on a trip through Europe, and could not resist repeating Galileo's famous experiment at the Leaning Tower of Pisa. He danced with his toddler son "sur le pont d'Avignon" before returning to Oxford; he received the D.Phil. (Oxon) in 1953. His thesis was on the nuclear structures of some of the heavy elements, and John always regarded his work on the decay of Bi-207 to be his greatest scientific achievement. In 1953 the family returned to Australia where John rejoined the AAEC at the Physics Department of the University of Melbourne.

Before long the adventure of an overseas post again beckoned and in 1956 the family moved to Canada where John was appointed Lecturer at the University of British Columbia in Vancouver. In 1960 he moved to the University of Calgary, Alberta, as Assistant Professor, where he remained for eleven years. John
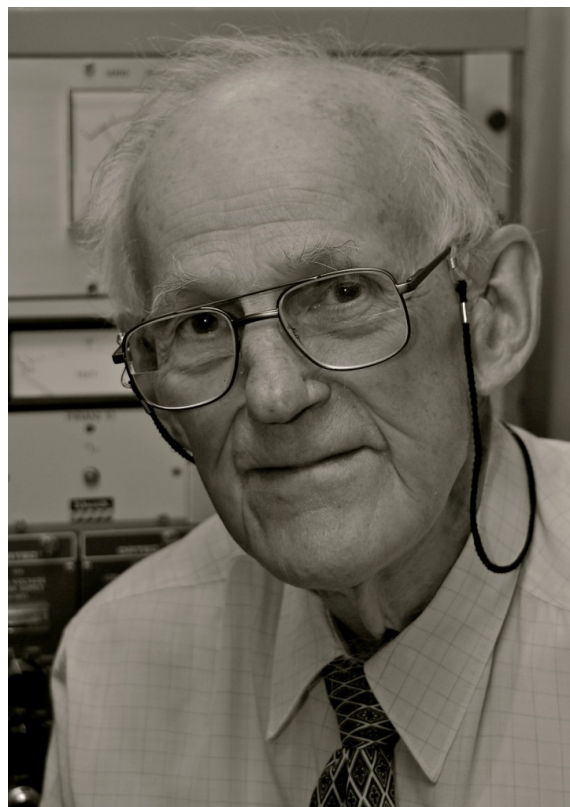
was promoted to Professor in 1968. It was in Alberta that he revived his interest in cosmic ray physics through studies at a high-altitude site at Sulphur Mountain in the Canadian Rockies, and started pioneering work on radio emission from cosmic ray showers at the Dominion Radio Astrophysical Observatory near Penticton. In addition to his research activities, and giving dynamic lectures to his students, John sang in choirs, played cricket, explored the Rockies, skated, skied, and culminated a hockey career of 40 years by qualifying as a grass hockey umpire and umpiring in the 1967 Pan Am games.

In 1971 John returned to Australia where he took up a position as Professor of Physics at the University of Adelaide, and in 1982 he was appointed Elder Professor of Physics, a distinguished title dating back to the Nobel prize-winning Sir William Bragg, who was appointed to the position in 1886.

John was passionate about teaching, and subscribed to the view that physics academic staff should be able to lecture on any undergraduate physics course. He made a special study of the teaching of elementary physics courses, particularly undergraduate laboratories in which he enjoyed performing entertaining experiments. His yellow Volvo with its sticker "Physics is Phun" will be fondly remembered. His main research interest continued to be in cosmic rays, and together with colleagues, he relocated the Penticton cosmic ray detectors to the University of

Adelaide's research station at Buckland Park. This was the beginning of the Adelaide Cosmic Ray Physics (later named High Energy Astrophysics) group which John led for the next 20 years, until his retirement.

He also played a major role in raising awareness of employment opportunities in physics, and published extensively on physics education. He surveyed all advertisements for physics employment in "The Australian" newspaper weekly for 25 years and regularly published the results and trends, for which he received the Australian Institute of Physics Outstanding Service award in 2003. In between all this activity John found time to build a harpsichord.

During the mid-1970s John and Jo participated as volunteers in archaeological digs at Roonka on the River Murray. This led John to ask the question of how could physics assist archaeology? He was advised that dating would help, and hence developed an interest in the relatively new technique of thermoluminescence dating then decided that he would start up a laboratory as a side-line to his core research interest in cosmic rays. He reasoned that it would make a good hobby in his retirement, which at that stage was still fifteen years off! Luminescence soon began to dominate his academic research, and John wrote over a hundred papers on the subject. His field activities extended to Lake Mungo, Lake Amadeus, the Flinders Ranges and many other localities in Australia, and as far afield as China and Thailand. Back in the laboratory he and his students developed and built a "Three Dimensional" Fourier Transform spectrometer for the study of the wavelength of luminescence from mineral grains – the first such instrument to be built and still the only one of its calibre in the world. At the same time his interest in cosmic rays did not diminish and he applied his knowledge of it to luminescence: his paper on the relationship of cosmic ray dose-rate to depth, density and latitude is one of the most cited articles in the luminescence literature.

In 1990 he officially retired and delivered his valedictory lecture at which, among other things, he demonstrated how to cook eggs in liquid nitrogen.

John and Jo became members of the Glenunga Croquet Club, and in characteristic fashion John gave the game his very best, soon winning his first of several competitions. They were also active and valued members of the Field Geology Club of South Australia. In 2007 John and Jo celebrated their 60th wedding anniversary.

In the academic world John continued to receive acclaim, including a special award from the University of Melbourne for being among its first PhD awards, and in 2002 the Royal Society of South Australia's Verco medal for an outstanding contribution to science, which had special meaning for John as his father had been awarded that same medal 64 years previously.

Retirement to John meant coming into the Physics Department five days a week and honing his expertise in luminescence dating and the physics of luminescence. He was also a regular contributor to the "Would you believe it?" column of the Adelaide daily newspaper, the "Advertiser". In 2004 a special conference and celebratory dinner were held at the University of Adelaide in honour of John's 80th birthday. His only concession to octagenarianism, however, was to reduce his attendance in the lab to just 3-4 days a week (Wednesday mornings being dedicated to croquet) and, following a knee operation, to cease riding his bicycle to work and to use the lift instead of the stairs to reach his office. His publication output did not decrease and he continued with pioneering research, particularly that involving use of his 3D spectrometer, right to the end. His last message to his colleagues at the luminescence lab, only three days before he went into hospital, was "I may not be in for the rest of this week. Please keep my laptop charged up".

John's service to the University of Adelaide has been outstanding, including periods as Dean of Science, Chairman of Physics and later of Physics and Mathematical Physics, and also Chairman of the Education Committee. He was a wonderful colleague and friend, held in the greatest esteem by the Cosmic Ray and Luminescence Groups which he had founded, by his colleagues in Physics, Geology and Geography, and by the worldwide communities in those disciplines. A gifted and caring teacher, mentor and supervisor, he always put his students and their welfare above all else, giving generously of his time, knowledge and expertise. Although he was a firm disciplinarian, his heart was of gold. Often a colleague would find on their desk an apple, a cluster of delicious peaches, capsicums or other products of his garden for, among his many other talents, he was a dedicated gardener and delighted in sharing the results. His kindness and generosity, sense of humour, love of jokes, deep love of science, breadth of knowledge and intellectual curiosity were, and will continue to be, an inspiration to all who knew him. He will be sorely missed. He is survived by his wife Jo, with whom he shared a true partnership for 70 years, and children James, Ann and Kate.

Frances Williams, Nigel Spooner, Bruce Dawson
Department of Physics,
University of Adelaide,
Australia

Obituary

Glenn Berger (1945-2011)

The luminescence dating community, as well as the broader field of Quaternary geochronology has lost one of its' pioneering scientists, with the sudden and untimely death of Glenn Berger on September 17, 2011. Glenn will be remembered for his role in the development and application of luminescence dating techniques to understand Quaternary environments and rates of surface processes.

His many collaborators and colleagues will miss Glenn's intellectual rigor, careful attention to detail, scientific insights, and dry humor. Our deep sense of loss is compounded by the realization that his contributions to Quaternary geochronology were cut off in their prime.

Glenn became interested in the development and application of luminescence dating to Quaternary deposits in the late 1970's, following an early career interest in the development and application of $^{40}Ar/^{39}Ar$ dating. Coning from a background in physics and geophysics, he was fortunate to acquire a thorough grounding in the physics and procedures of luminescence dating from one of the pioneers in the field - Dr David Huntley of the Department of Physics at Simon Fraser University. In 1986, Glenn went to Western Washington University to establish one of the first generation of luminescence dating laboratories in the USA. He moved to the Desert Research Institute in Reno Nevada, in 1994 to head the E.L. Cord Geochronology Laboratory, which he directed until his untimely passing.

Glenn's contributions to Quaternary geochronology were many and ranged from detailed experimental studies of the luminescence characteristics of sediments in varied environments to innovative applications to new and challenging environmental settings. Glenn constantly strived to understand which grains in a sediment would be amenable to luminescence dating. He developed meticulous sampling procedures to extract these grains and worked diligently to analyze them in the most appropriate manner. Glenn was drawn to applications of luminescence dating to challenging environments where conventional geochronological techniques proved difficult to apply or produced inconsistent results. He was particularly interested in the application of luminescence dating to water-lain sediments, especially in lacustrine and marine

environments, as illustrated by his recent work on Arctic and Antarctic marine sediments; and lacustrine sediments in the Dry Valleys of Antarctica.

Glenn Berger was always a perfectionist in his field and laboratory research and worked hard to make sure that the chronometric information he produced was as accurate and precise as could possibly be achieved. He will be missed by all who had the privilege to work with him.

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Conference Announcements

UK Luminescence and ESR meeting

12th-14th September 2012

The next UK luminescence and ESR dating research meeting will be held at Aberystwyth University from the 12-14th September 2012. The meeting is intended to provide a forum for the presentation and discussion of the latest research in luminescence and ESR dating and related work. Presentations covering basic physics, methodological issues and the application of these techniques are all welcome. The meeting will consist of both oral and poster presentations, and presentations by research students are particularly encouraged.

Further details will be posted on the Aberystwyth Luminescence Research Laboratory website (http://www.aber.ac.uk/en/iges/research-groups/quaternary/luminescence-research-laboratory) early in 2012.

Professor Geoff Duller
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Submission of articles to Ancient TL

Reviewing System

In order to ensure acceptable standards and minimize delay in publication, a modification of the conventional refereeing system has been devised for Ancient TL:

Articles can be sent directly by authors to a member of the Reviewers Panel chosen on the basis of the subject matter, but who is not in any of the authors' laboratories. **At the discretion of the Editor**, reviewers who are not listed in the Panel may be used.

The reviewing system aims to encourage direct dialogue between author and reviewer. The Editor should be kept advised of the progress of articles under review by sending him copies of all correspondence. He is available for advice where reviewing difficulties have arisen. Authors whose mother tongue is not English are required to have their manuscript revised for English *before* submitting it.

We ask reviewers to specify (where required) the minimum of revision that is consistent with achieving a clear explanation of the subject of the paper, the emphasis being on *rapid* publication; reviewers are encouraged to make a brief written comment for publication at the end of the paper. Where a contribution is judged not to meet an adequate standard without substantial modification, the author will be advised that the contribution is not suitable for publication. Articles that are not considered to be of sufficient interest may also be rejected.

Procedures

1. Articles should be submitted to an appropriate member of the Reviewing Panel or Editorial Board, chosen on the basis of the subject matter, but who is not in any of the authors' laboratories.
2. Articles should not normally exceed the equivalent of 5000 words inclusive of diagrams, tables and references. Greater space will be appropriate for certain topics; for these the Editor should first be consulted.
   Short notes and letters are also invited. These should not exceed two printed pages in Ancient TL, including diagrams, tables and references (equivalent to ~1400 words of text).
3. Diagrams and labels should be ready for direct reproduction and not normally exceed 12 cm wide by 10 cm high. Where possible, high quality electronic versions of figures should be submitted. Separate figure captions should be supplied. Inappropriately scaled drawings and labels will be returned for alteration.
4. Authors are asked to submit the paper, including diagrams, to the Reviewer and a duplicate copy to the Editor.
   The final version of the text must be submitted to the Editor electronically using a standard format (Microsoft Word for PC is currently used for producing Ancient TL). Electronic copies of Diagrams and Tables should also be submitted.
5. Upon receipt of an article, the Editor will send an acknowledgement to the author. If the Reviewer is unable to deal with the contribution within **4 weeks** he/she will inform the author and advise the Editor.

Requirements under various situations

When agreement concerning an article has been reached:
The Editor should receive a copy of the final version of the paper, both as hard copy and electronically. The Reviewer should send their final decision, including comments for publication if any, to the Editor.

If the article has not been rejected, but agreement on its final form cannot be reached or where there are protracted delays in the reviewing process:
The Editor may request an assessment from the Reviewer and responsibility passes to the Editor.

If the article is rejected:
The Editor and author receive notification from the Reviewer, with an indication of the reason for rejection.

Thesis abstracts are to be sent to the Editor and in principle do not need reviewing. However, authors are requested to make sure that the English is correct before submission. Thesis abstracts should not exceed 750 words, and figures and tables are not accepted.

Advertising. Formal information on equipment can be published in Ancient TL. It should not exceed one printed page. Current charges are displayed on the website (http://www.aber.ac.uk/ancient-tl)

Subscriptions to Ancient TL

Ancient TL is published 2 times a year and is sent Airmail to subscribers outside the United Kingdom. While every attempt will be made to keep to publication schedule, the Editorial Board may need to alter the number and frequency of issues, depending on the number of available articles which have been accepted by reviewers.

The subscription rate for 2012 is £15 for individual subscribers and £25 for Institutional subscription, plus any taxes where required. Payment must be in pounds sterling. Enquiries and orders must be sent to the Editor. Payment may be by cheques, made payable to 'Aberystwyth University', by credit/debit cards or by bank transfers. Further information on subscriptions is available on the Ancient TL web site (**http://www.aber.ac.uk/ancient-tl**)