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# Optical dating of young feldspars: the zeroing question

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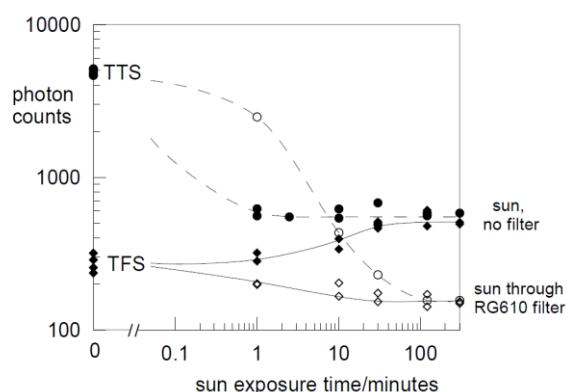
## Abstract

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 increasingly 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

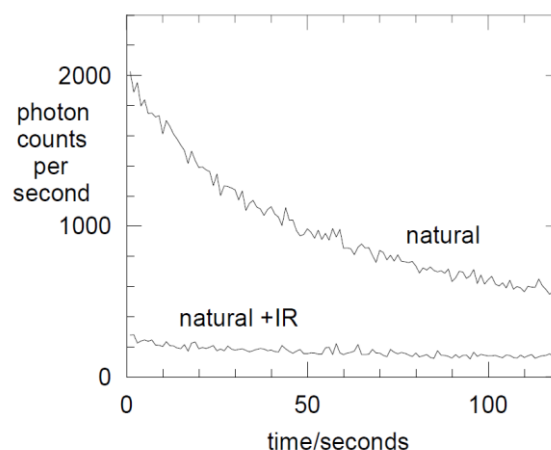
removed. Knowing this was essential to obtaining ages consistent with independent information. Huntley and Clague (1996) suggested that the effect might be attributed to the higher energy photons of sunlight causing transfer of electrons into the principal traps (those being sampled by the 1.4 eV excitation 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  $9 \pm 1$  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  $\mu\text{m}$  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 predominately 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 <sub>eq</sub> (Gy)	Optical age (a)	Known age (a)
<i>IR bleach</i>			
D	0.23 ± 0.02		< 1
C	0.28 ± 0.02		9 ± 1
(D <sub>eq</sub> for C) - (D <sub>eq</sub> for D)	0.05 ± 0.03	19 ± 12	9 ± 1
<i>Sunlight bleach</i>			
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  $6.7 \pm 0.3$  %/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  $3.0 \pm 0.1$  Gy·ka<sup>-1</sup>.

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

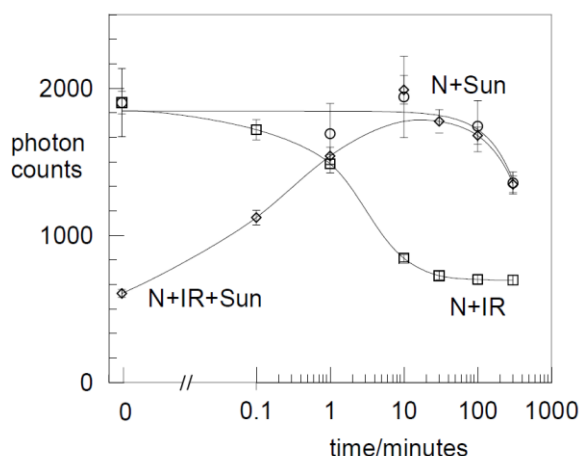
Insufficient sunlight exposure was unlikely for the following reasons: (a) sample D was known to have been exposed to bright sunlight for > 6 hours prior to collection, (b) the equivalent dose vs time curve did not show an increase in the later part of the intensity decay, and (c) for each sample the 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  $19 \pm 12$  years, consistent with the known age of  $9 \pm 1$  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  $1 \pm 7$ ,  $8 \pm 8$  and  $38 \pm 7$  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.

### Acknowledgements

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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.

