Measuring photo-transferred thermoluminescence from feldspars in post-IR IRSL procedures using a new user defined command for the Risø TL/OSL reader

Riedesel, Svenja; Duller, G. A. T.

Published in:
Ancient TL

Publication date:
2022

Citation for published version (APA):
Riedesel, S., & Duller, G. A. T. (2022). Measuring photo-transferred thermoluminescence from feldspars in post-IR IRSL procedures using a new user defined command for the Risø TL/OSL reader. *Ancient TL*, *40*(1), 1-11. http://ancienttl.org/ATL_40-1_2022/ATL_40-1_Riedesel_p1-11.pdf

Ancient TL

Measuring photo-transferred thermoluminescence from feldspars in post-IR IRSL procedures using a new user defined command for the Risø TL/OSL reader

Svenja Riedesel[1,2*] and Geoff A.T. Duller[2]

[1] Institute of Geography, University of Cologne, Cologne, Germany
[2] Department of Geography and Earth Sciences, Aberystwyth University, United Kingdom
*Corresponding Author: riedeselsvenja@gmail.com

Received: February 17, 2022; in final form: May 13, 2022

Abstract

Stimulating a preheated feldspar sample with infrared light causes photo-transfer of charge within the crystal. The resulting photo-transferred luminescence signal is visible in thermoluminescence (TL) and infrared stimulated luminescence (IRSL) measurements, where it results in an increase in the measured luminescence signal intensity. This is particularly relevant when using a post-IR IRSL measurement protocol for dating feldspars, which consists of two consecutive IRSL measurements, one at low temperature (usually at 50 °C) and a second IRSL measurement at elevated temperature. In this case, the lower temperature IRSL measurement causes photo-transfer of charge, which then influences the subsequent higher temperature post-IR IRSL signal. This paper presents a new user defined command to be used with the Sequence Editor software on a Risø TL/OSL reader. This command can be used to assess the amount of photo-transfer that is caused by IR stimulation, and how large a signal this may contribute to a subsequent elevated temperature IRSL measurement. The command can also be useful for identifying the length of time an aliquot should be held at the measurement temperature prior to switching on the IR LEDs in order for any photo-transferred TL to have been reduced to a negligible level. To show the applicability of this new user defined command to feldspar post-IR IRSL measurements, this paper presents results from two feldspar samples measured using the new user defined command as part of three different preheat and post-IR IRSL temperature combinations.

Keywords: User defined command, photo-transferred TL, feldspar luminescence, post-IR IRSL

1. Introduction

Photo-transferred thermoluminescence (PTTL) or re-excitation (e.g., Bailiff, 1976; Schlesinger, 1965) describes luminescence that occurs as a result of optical excitation after irradiation and preheating and which is visible in TL curves measured after the optical excitation. It has been associated with redistribution of charge (Kaylor et al., 1995) and was observed in different minerals, including quartz (e.g., Kaylor et al., 1995; Schlesinger, 1965; Wintle & Murray, 1997), fluorapatite (e.g., Bailiff, 1976), zircon (e.g., Bailiff, 1976; Kristianpoller et al., 2006) and feldspar (e.g., Duller, 1995; Murray et al., 2009; Robertson et al., 1993; Tsukamoto et al., 2012). PTTL can easily be identified when comparing TL curves recorded after a preheat and TL curves recorded after a preheat and a subsequent optical stimulation step (cf. Fig. 1).

For feldspars it has been observed that stimulation of a sample with infrared (IR) light can result in an increase in TL signal intensity due to photo-transfer of charge (cf. Fig. 1). If one subtracts a TL curve measured after a preheat and an IR stimulation step from a TL curve measured after only a preheat (curve A – curve B), a net negative TL signal will be observed in the temperature range of the former preheat (cf. dashed line in Fig. 1; e.g. Duller 1995; Murray et al. 2009; Robertson et al. 1993; Tsukamoto et al. 2012). Besides affecting TL, PTTL may influence the intensity of the IR stimulated luminescence (IRSL) signal (e.g., Qin et al., 2015; Wang & Wintle, 2013; Wang et al., 2014), which is particularly important in light of the routine use of the post-IR IRSL

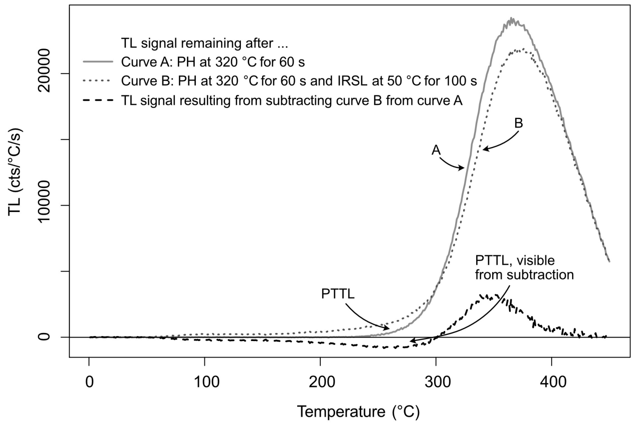

Figure 1: Feldspar TL signal recorded after a preheat of 320 °C for 60 s (solid line). From the dotted line (B) one can see the slight increase in TL signal intensity in the temperature range from ~ 100 °C to 290 °C. This is due to photo-transfer from IR stimulation. The dashed line results from subtracting the dotted line (B) from the solid line (A). Using this approach, the PTTL signal is visible from subtraction.

signal when dating feldspars. Usually, when applying a post-IR IRSL protocol, the signal used for dating is an IRSL signal recorded after a preheat and after one or multiple lower temperature IR stimulation step(s) (cf. Buylaert et al., 2012; Li & Li, 2011; Thiel et al., 2011; Thomsen et al., 2008). The lower temperature IRSL measurements will likely introduce a PTTL signal for the next higher temperature IRSL measurement, where it would then be released during the heating to this higher temperature IRSL measurement, potentially affecting the signal intensity and stability (e.g., Qin et al., 2015; Wang & Wintle, 2013; Wang et al., 2014). PTTL is clearly visible in TL measurements (cf. Fig. 1) and a first impression of PTTL in IRSL can be gained by using blank channels prior to turning on the stimulation light sources. However, the current set of commands for the Risø TL/OSL reader available through the Sequence Editor does not enable recording the PTTL signal to its full extent during IRSL measurements, hindering routine study of this PTTL signal, or any assessment of the likely impact it could have upon the subsequent post-IR IRSL measurement.

To gain further information on the PTTL signal and on its impact on the post-IR IRSL signal, it would be useful to monitor and minimise the impact of PTTL when dating feldspars. Here we present a new user defined command for the Risø TL/OSL reader that makes it possible to record and monitor the PTTL signal during routine IRSL measurements.

2. Previous studies exploring the effect of PTTL on post- IR IRSL signals

Whilst PTTL in feldspars has been known for a long time (e.g., Duller, 1995; Robertson et al., 1993), its presence in and effect on the post-IR IRSL signal has been of interest in more recent years. To highlight the importance of this photo-transferred signal for IRSL measurements, we here briefly review studies focussing on the PTTL signal arising in the post-IR IRSL measurement procedure.

In 2013 Wang & Wintle associated parts of a measured post-IR IRSL signal of a perthitic feldspar sample with photo-transfer of charge during IR stimulation. These authors suggest that this charge is released thermally during the post-IR IRSL stimulation step at elevated temperature, resulting in a contribution to the overall post-IR IRSL signal. These authors identify contributions of this photo-transferred signal to the overall measured post-IR IRSL signal of up to 20 %. Wang & Wintle (2013) suggest that this photo-transferred TL signal should be removed prior to the measurement of the post-IR IRSL signal, because it is expected that the photo-transferred signal shows fading rates similar to the IRSL signal measured as a first step in the post-IR IRSL measurement procedure. Subsequently these authors suggest to either (i) insert a cut heat, at a temperature slightly below the preheat temperature, between the two IR measurements, or (ii) to hold the sample at the post-IR IRSL measurement temperature for a longer duration prior to switching on the LEDs. However, Wang & Wintle (2013) did not explore this second suggestion.

Wang et al. (2014) further investigated the effect of the photo-transferred signal on the post-IR IRSL signal. These authors suggest that the cut heat proposed by Wang & Wintle (2013) could affect the post-IR IRSL measurement negatively by resulting in re-trapping of some of the thermally excited electrons, which will then contribute to the post-IR IRSL signal. Subsequently, these authors advise against the use of a cut heat as a tool used to remove a PTTL signal. Qin et al. (2015) showed that the presence of a PTTL signal in a post-IR IRSL signal can lead to an underestimation of a given dose in dose recovery experiments and that the presence of a PTTL signal can lead to an underestimation of the thermal stability of the post-IR IRSL signals. Qin et al. (2015) show that increased preheat and post-IR IRSL stimulation temperatures (up to 400 °C) improve dose recovery ratios, compared to a post-IR $IRSL_{290}$ protocol with a preheat at 320 °C.

The application of a cut heat between the two IRSL measurements in a post-IR IRSL protocol results in other issues, as pointed out by Wang et al. (2014). Also, the suggestion by Qin et al. (2015) of using very high preheat and post-IR IRSL stimulation temperatures might influence the feldspar crystal, since exsolution features might be affected by heating the material to temperatures around 400 °C (e.g., Brown & Parsons, 1984; Lin & Yund, 1972; Parsons et al., 2015). Thus, holding the sample at the IRSL measurement temperature for a longer duration (Wang & Wintle, 2013) might be the most gentle and effective solution to remove the PTTL signal and its contribution to a post-IR IRSL signal. If this approach would be attempted, it would be important to evaluate how long such an isothermal holding step prior to switching on the IR LEDs should be. Therefore, a procedure for the routine assessment of the presence and influence of PTTL on post-IR IRSL measurements is needed.

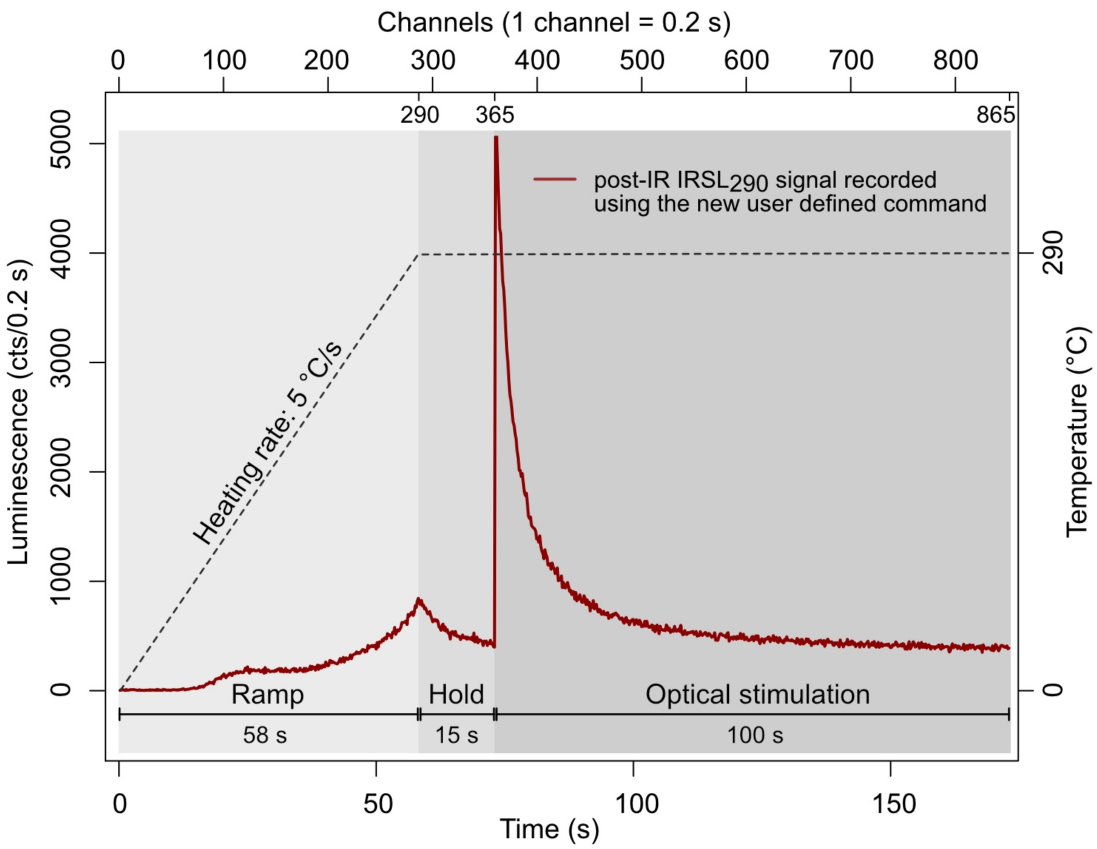

Figure 2: An example of a PTTL signal and IRSL decay curve recorded using the new user defined command. The figure shows the luminescence emitted during ramping of the sample to the measurement temperature (here: 290 °C), then during the 15 s pause prior to the IRSL measurement, and then during the actual IRSL measurement (here: 100 s of IR stimulation).

3. A new user defined command to record and monitor PTTL in feldspar IRSL measurements

3.1. Purpose and performance of the new user defined command

In the following we describe a newly developed user defined command for recording and monitoring PTTL in IRSL measurements using the Risø TL/OSL system. This new user defined command can be used instead of a routine OSL or IRSL measurement, where it will not only record the actual OSL/IRSL measurement, but also any luminescence emitted during heating of the sample to the measurement temperature and during potential isothermal pauses at the measurement temperature prior to optical stimulation. This will enable an estimation to be made of the contribution of this PTTL signal to the elevated temperature IRSL signal.

To enable recording and monitoring of the PTTL signal in OSL/IRSL measurements the Mini-Sys command file for user defined instructions (usermsll.cmd) originally installed with the software needs to be modified (Table 1). The new command results in the same treatment to the aliquot as occurs using the OSL command, but instead of only recording the luminescence emitted during the optical stimulation, this user defined command records data (i) during the ramping of the aliquot temperature from room temperature to the measurement temperature, (ii) during the period of time selected for the 'Pause' when the aliquot is held at the measurement temperature prior to starting the optical stimulation, and (iii) during the optical stimulation itself. Data from all three phases of the operation are concatenated, producing a single data record in the BINX file. The data can be viewed using the Analyst software (Duller, 2015), but because they combine together three different phases they are hard to interpret. To present the data more clearly they were exported from Analyst and then further processed using **R** (R Core Team, 2019). An example of the data produced by this command is shown in Figure 2. Figure 2 shows a post-IR IRSL$_{290}$ signal (red line) recorded using the new user defined command. The shaded areas indicate the three parts of the PTTL and IRSL measurement: (i) ramping to the measurement temperature (here 290 °C, see dashed line), (ii) the isothermal holding measurement for 15 s (also referred to as 'Pause') and (iii) the actual optical stimulation for 100 s. The second x-axis on top of the graph shows the channel numbers used, when recording this measurement with a resolution of

0.2 s per channel. We would also advise the user to use the same channel width for all three parts of the measurement using the new command to make data analysis simpler. The post-IR $IRSL_{290}$ signal shows a PTTL signal during the ramp and the isothermal holding measurement. Whilst the PTTL signal increases during the ramp, a decrease is observed during the 15 s pause prior to switching on the LEDs. However, one can see that the PTTL signal has not decreased to the background level after a 15 s pause, thus one can assume a contribution of the PTTL signal to the actual IRSL signal in this example.

3.2. Implementing and using the user defined command

To be able to use this user defined command, the Mini-Sys command file for user defined instructions (usermsll.cmd) needs to be changed. Details about user defined commands can be found in Armitage & Duller (2004). Information about the low-level commands that are used in the command file are listed in a handbook provided by Risø via their website (https://www.fysik.dtu.dk/english/research/radphys/research/radiation-instruments/tl_osl_reader/manuals; June 23, 2022). Once the latest Risø software package is installed on the user's hard drive, a PDF ("UserDef.pdf") containing information on how to edit and write user defined commands can be found in the "Manuals" folder of the Risø software package. Before modifying the usermsll.cmd file we strongly recommend to back-up the existing file and create a copy for editing. For the purpose of this paper we modified user defined command 4 (UserDef4), which already existed in the usermsll.cmd file. The text which needs to be inserted as a user defined command into the usermsll.cmd file is given in Table 1. The first part of Table 1 defines which command line in the Sequence Editor software describes which part of the command (see Table 1 and Fig. 3, and compare commands with $N). The second part contains the instructions for the Mini-Sys, so that the command can be performed. Once the user defined command is implemented, it can be tested. Figure 3 shows the Sequence Editor command window, where all individual commands ($N) can be edited depending on the settings required for the measurement. Please be aware that it is not possible to modify the title for each command. In the example displayed in Figure 3 an IRSL measurement at 290 °C was performed for 100 s. The corresponding IRSL decay curve and recorded PTTL is shown in Figure 2. Prior to the optical stimulation using IR LEDs, the sample was held at the measurement temperature for 15 s. In this example, command $5 defines the measurement temperature (*here* 290 °C) and command $7 the light source (*here* IR LEDs). Depending on the integration time for each channel, all commands referring to the data points recorded have to be adjusted for the respective duration of the individual parts of the measurement. As an example: the duration of the IR stimulation in this example is 100 s (see $3 in Fig. 3) and we would like the data to be collected with a channel width of 0.2 s, thus the number of data points during optical stimulation ($11 in Table 1 and Fig. 3) should be set to 500. Similar calculations and adjustments should be made for the ramp and the isothermal holding step (see corresponding command numbers in Table 1 and Fig. 3). All channels needed to record the ramp ($1), the isothermal hold ($19) and the actual stimulation step ($11) have to be added and the sum needs to be inserted in the field corresponding to $20 (in this case: 290 + 500 + 75 = 865).

New user defined function in usermsll.cmd script

```
; Record luminescence during heating and pause prior to OSL
; $0 Position
; $1 Data points during heating ramp
; $2 Not used
; $3 Length of optical stimulation
; $4 Heating rate during ramp
; $5 Heating temperature
; $6 Length of pause at temperature before optical stimulation
; $7 Light source
; $8 Optical power
; $9 Not used
; $10 Not used
; $11 Data points during optical stimulation
; $12-18 Not used
; $19 Data points during pause before optical stimulation
; $20 = $1 + $11 + $19

10=PS $0
20=#RS
25=#INITGRAPH $20
30=#TF
40=#RS
50=#WLT
60=LU
70=#RS
75=IR SET $8
80=TL $5 $4 $1 $5
90=#DATA
100=OS N $6 $19
110=#DATB
120=#CONCAT
130=OS $7 $3 $11
140=#DATB
150=#CONCAT
160=#SAVE
170=#ENDGRAPH
180=#WRITE
190=ST 0
200=LD
210=#RS
```

Table 1: User defined command as written for the Mini-Sys command file for user defined instructions (usermsll.cmd), which allows recording and monitoring of the PTTL signal in IRSL measurements. Since it is not possible to rename the individual commands in Sequence Editor or the usermsll.cmd file, the titles for each command are listed in this table for clarity.

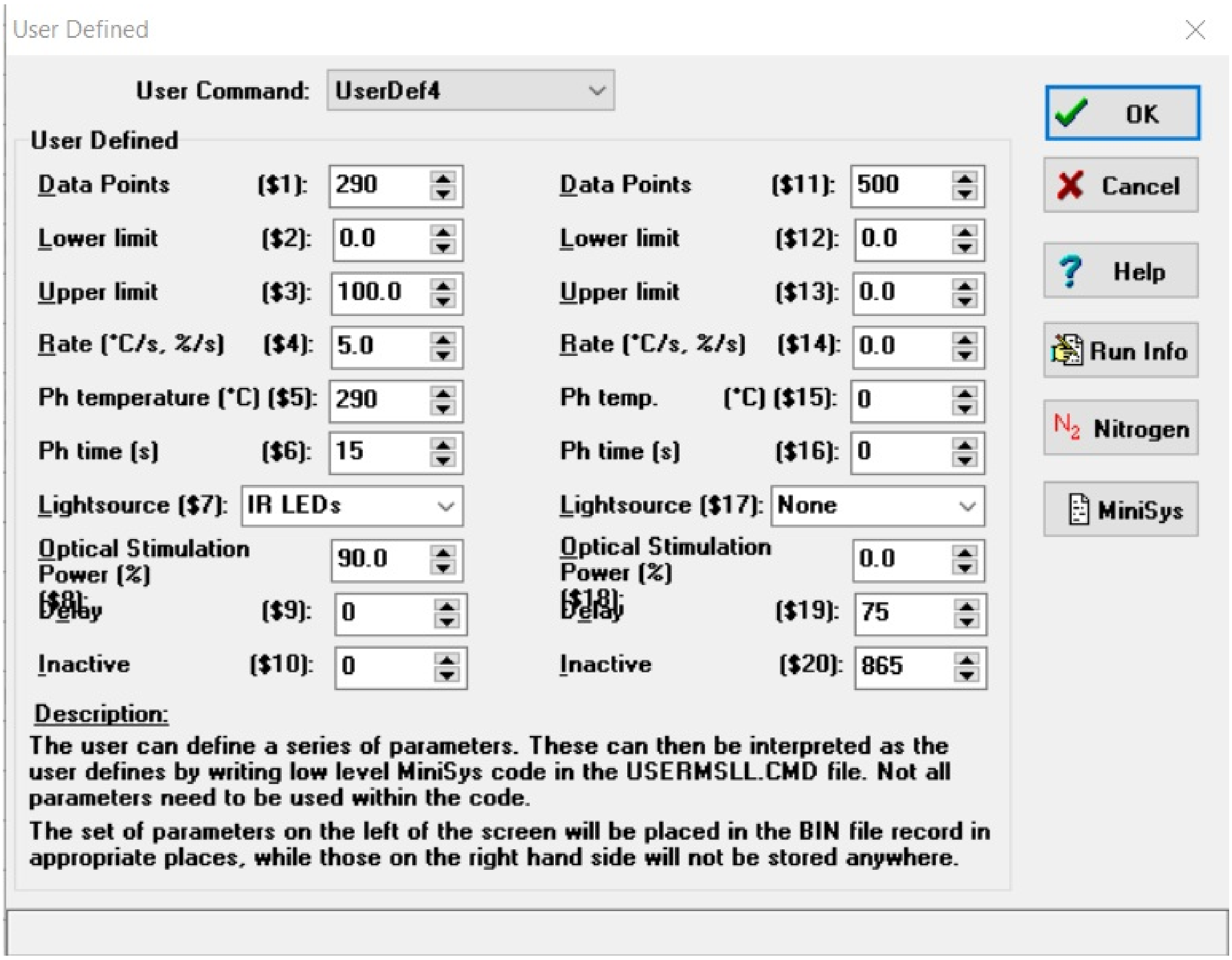

Figure 3: Screen shot of the Sequence Editor command window, as prepared for a 100 s IRSL measurement at 290 °C (parameter $5), with a 15 s isothermal measurement step ('pause') prior to switching on IR LEDs (parameter $6). The time included in each channel was defined as 0.2 s for the ramp, the isothermal measurement and the actual IRSL measurement. Each parameter in this window has to be edited to fit desired measurement settings, such as stimulation temperature, duration, channel width and light source.

After ensuring appropriate settings in the Sequence Editor command window, the user defined command can be used instead of the routine IRSL measurement.

4. Application of the new user defined command to explore PTTL

To show the potential use of the new user defined command, the amount of PTTL in TL and IRSL measurements in two feldspar samples (one sedimentary K-rich feldspar sample and one Na-rich feldspar sample extracted from bedrock) was examined using three different preheat and stimulation temperature combinations.

4.1. Samples

The two feldspar samples were selected because of their different chemical composition and because they show very different laboratory irradiated TL signals (inset Fig. 4a and c) and different PTTL signal intensities (Fig. 4b and d). Both samples are feldspar-rich grain mixtures, such as those commonly used during feldspar luminescence dating. Sample WHB-7 is a sediment extract from the Channelled Scablands, Washington State, USA. The sediment sample WHB-7 was treated using hydrochloric acid (HCl; 10 %) and hydrogen peroxide (H_2O_2; 10 %) to remove carbonates and organic matter. Subsequently the sample was dry sieved to 180-212 µm and density separated to $2.53 < \rho < 2.58$ g cm^{-3} using sodium polytungstate. Thermoluminescence emissions and anomalous fading rates of sample WHB-7 have been explored previously by Riedesel et al. (2021). KTB-383-C originates from a bedrock core obtained from the KTB borehole in southern Germany. The grain extract of this sample was obtained by crushing the rock; subsequently the material was sieved to isolate the fraction of 180-250 µm. This fraction was used to isolate the alkali feldspar grains using sodium polytungstate at a density of 2.58 g cm^{-3}. The density separated sample material of KTB-383-C was etched in 10 % HF for 40 min and subsequently washed in 10 % HCl for 20 min to remove any fluorides (see supplementary material of Guralnik et al. (2015) for details regarding the sample preparation procedure of KTB-383-C). Further information on the luminescence of sample KTB-383-C can be found in Guralnik et al. (2015) and Riedesel et al. (2019).

The chemical compositions of the samples were determined using X-ray fluorescence (XRF) and the mineral phases present in the grain mixtures were determined using X-ray diffraction (XRD). Details regarding these two methods and the respective sample preparation can be found in Riedesel et al. (2021). Sample WHB-7 consists of microcline (8 %), albite (54 %), quartz (15 %) and muscovite (23 %) and its feldspar chemistry has been determined as 64 % K-feldspar, 32 % Na-feldspar and 5 % Ca-feldspar, when assuming 100 % feldspar (cf. Table 2). Sample KTB-383-C contains 22 % albite, 31 % quartz, 45 % muscovite and 2 % other mineral phases. Its chemical composition is determined as 8 % K-feldspar, 80 % Na-feldspar and 13 % Ca-feldspar, when assuming 100 % feldspar (cf. Table 2).

4.2. Instrumentation and the new user defined command

For luminescence measurements, sample grains were mounted as 2 mm aliquots on stainless steel discs (~0.095 g in mass) using silicone oil. The luminescence measurements were performed at the Aberystwyth Luminescence Research Laboratory using a Risø DA20 TL/OSL reader equipped with a $^{90}Sr/^{90}Y$ beta source delivering ~0.08 Gy s^{-1} at the sample position. For stimulation, IR LEDs with an emission at 870 nm (FWHM 40 nm) delivering ~145 mW cm^{-2} (Risø, D. T.

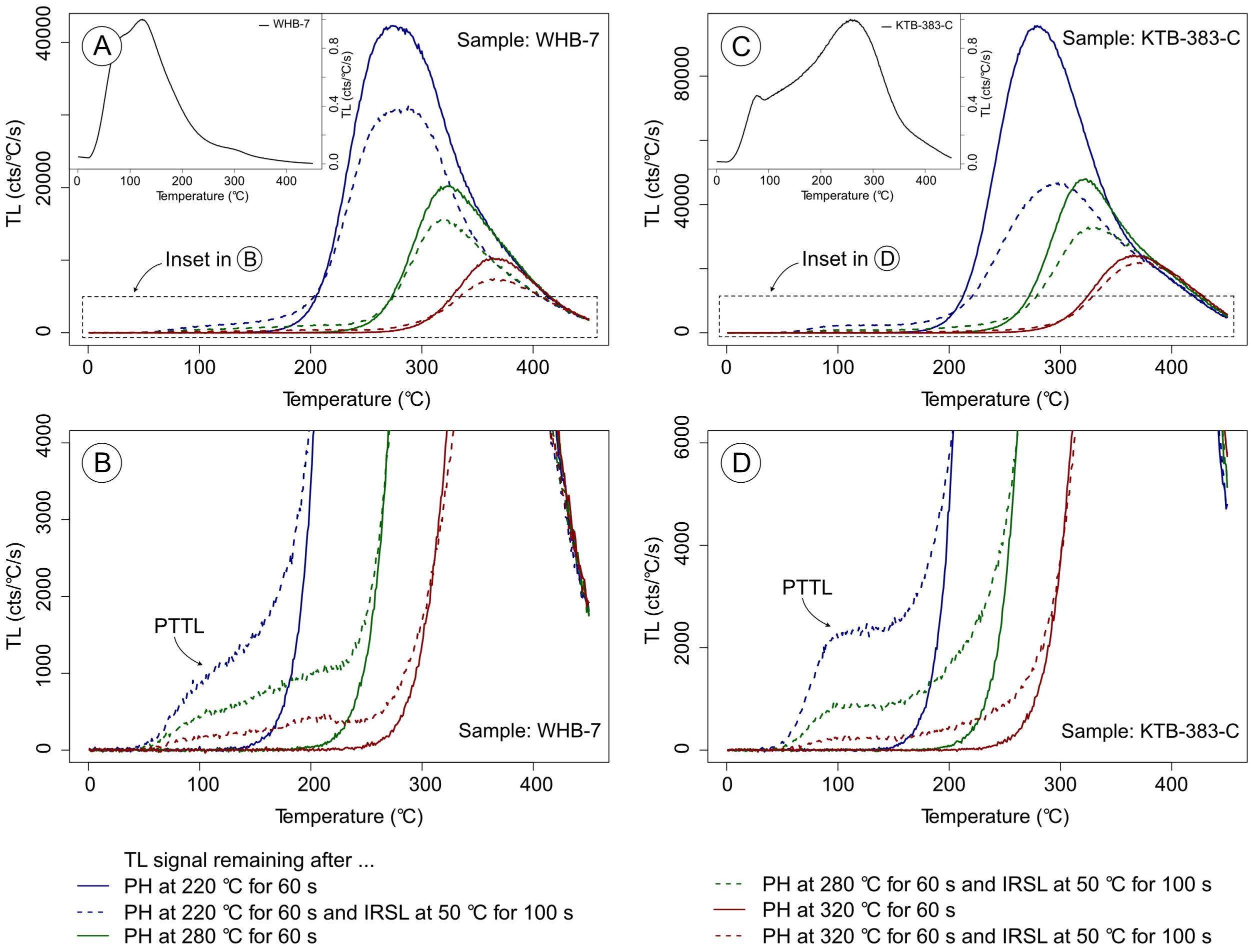

Figure 4: The insets of Figs (A) and (C) show the TL curve recorded immediately after irradiation. (A) TL signal remaining after preheating the laboratory irradiated material to 220 °C (blue line), 280 °C (green line) and 320 °C (red line) and after preheating and IRSL50 (dashed lines of respective colours) of sample WHB-7. Besides the decrease in signal intensity in TL at higher temperatures, an increase in TL intensity is visible in the temperature range below preheat temperature for TL curves recorded after preheating and stimulating with IR photons at 50 °C. This signal increase is associated with photo-transfer and a close-up of this temperature range is shown in (B). (C) and (D) show the same as (A) and (B), but for sample KTB-383-C.

Sample ID	Sampling location	Chemical compositions of feldspars (in %, assuming 100 % feldspar)			Mineral phases present (%)					References
		K-FS	Na-FS	Ca-FS	Microcline	Albite	Quartz	Muscovite	Other	
WHB-7	White Bluffs, Channelled Scablands, Washington State, USA	63.9	31.5	4.6	8	54	15	23	NA	Riedesel et al. (2021)
KTB-383-C	KTB-borehole, Germany	7.9	79.1	13	NA	22	31	45	2	Guralnik et al. (2015), Riedesel et al. (2019)

Table 2: Details of the samples used in this paper. Their chemical composition was determined using XRF and the phases present in the grain mixtures were determined using XRD. References are given to studies in which the luminescence properties of the samples have previously been explored.

Table 3

(a) Measurement protocol used to visualise the impact of phototransfer on feldspar TL (Curve A in Fig. 1).

Step	Treatment
1	Irradiation (48 Gy)
2	Preheat for 60 s at 220, 280 or 320 °C, 5 °C s-1
3	Pause for 100 s
4	Pause for 100 s
5	TL to 450 °C, 1 °C s-1

(b) Measurement protocol used to visualise the impact of phototransfer on feldspar TL (Curve B in Fig. 1).

Step	Treatment
1	Irradiation (48 Gy)
2	Preheat for 60 s at 220, 280 or 320 °C, 5 °C s-1
3	IRSL for 100 s at 50 °C
4	Pause for 100 s
5	TL to 450 °C, 1 °C s-1

(c) Measurement protocol used to visualise the impact of phototransfer from $IRSL_{50}$ to post-IR IRSL using the new user defined command to record luminescence during ramping, isothermal holding and IR stimulation steps, as part of the IRSL measurement. A TL signal was recorded after the post-IR IRSL measurement to check for any remaining photo-transferred signal.

Step	Treatment
1	Irradiation (48 Gy)
2	Preheat for 60 s at 220, 280 or 320 °C, 5 °C s-1
3	IRSL for 100 s at 50 °C – measured using the new user defined command
4	IRSL for 100 s at 190, 250 or 290 °C – measured using the new user defined command
5	TL to 450 °C, 1 °C s-1

U. Nutech, 2015) were operated at 90 % stimulation power. The TL and IRSL signals were recorded through a combination of Schott BG 39 (2 mm thick, 45 mm in diameter) and Corning 7-59 (4 mm thick, 45 mm in diameter) optical filters and were detected using an EMI 9235QA photomultiplier tube.

The amount of PTTL in TL and IRSL measurements was examined using three different protocols with three different preheat and stimulation temperature combinations (Table 3a, 3b and 3c). To keep the time differences between irradiation and TL measurements the same for all three protocols, pauses were inserted in Table 3a (steps 3 and 4) and Table 3b (step 4) instead of IRSL commands. TL measurements were recorded up to 450 °C using a heating rate of 1 °C s^{-1}. Measured TL curves were background corrected by subtracting a second TL measurement from the first measurement. This was done automatically, by enabling the command “background subtraction” in the TL command in the Sequence Editor software. TL measurements after an irradiation step, but without any preheat and IRSL stimulation steps were performed before and after the steps outlined in Tables 3a, 3b and 3c to monitor potential sensitivity changes. We observed that once the sample material had been heated to 450 °C all subsequent measurements only display very little change in intensity, and TL measurements could be conducted with good reproducibility.

Preheating of the sample material prior to IRSL measurements was conducted using the TL command available in the Sequence Editor software. The preheat temperatures of 220 °C, 280 °C or 320 °C were reached using a heating rate of 5 °C s^{-1} and the sample was then kept at the preheat temperature for 60 s. All IRSL measurements were performed using the new user defined command. IRSL measurements were conducted for 100 s at 50 °C (Table 3b and Table 3c). The temperature for the post-IR IRSL stimulation was either 190 °C, 250 °C or 290 °C, depending on the preheat temperature, and the duration of this stimulation was 100 s (Table 3c). The IRSL measurement temperature was reached by heating at a rate of 5 °C s^{-1} and IR LEDs were switched on after the sample had been kept at the IRSL measurement temperature for 15 s. To visualise the decay of the PTTL signal during the actual IRSL measurements using the user defined

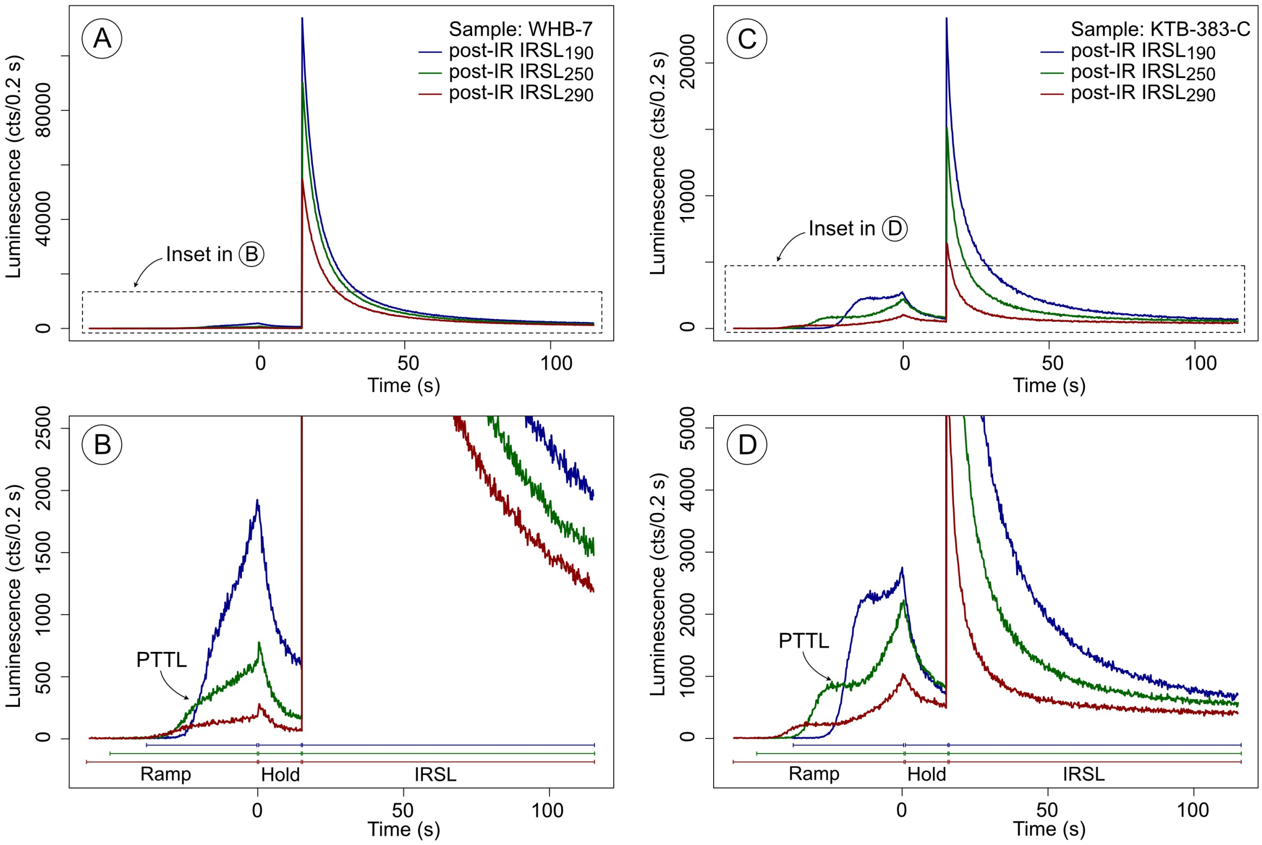

Figure 5: Post-IR IRSL signals recorded of sample WHB-7 (A and B) and of sample KTB-383-C (C and D) using the user defined command (Table 2). Figures (A) and (C) show the intensity of the photo-transferred signal, which is emitted during the ramping up to the IRSL measurement temperature and during isothermal holding of the sample at the measurement temperature prior to switching on the IR LEDs. The close-up figures (B) and (D) show the shape of the photo-transferred signal.

command, the post-IR IRSL measurements were performed using IR stimulation (Fig. 5 and Fig. 6) and without enabling the stimulation light source (Fig. 6, dashed lines) by selecting 'None' for the Lightsource ($7 in Fig. 3).

4.3. Results: Monitoring PTTL in TL measurements

Figure 4 shows the effect of $IRSL_{50}$ stimulation on the low temperature part of the TL curve of preheated samples when comparing the TL curves measured with (dashed lines in Fig. 4, Table 3b) and without $IRSL_{50}$ (solid lines in Fig. 4, Table 3a). The TL curves recorded immediately after a preheat (solid lines, Fig. 4) exhibit no TL signal up to ~30 °C below the preheat temperature, where the TL signal starts to grow. TL curves recorded after an $IRSL_{50}$ measurement (dashed lines Fig. 4, cf. protocol in Table 3b) show an increase in TL signal intensity from about 50 °C until ~20 °C below the preheat temperature: this is photo-transfer of charge, induced by IR stimulation.

The shape of the PTTL signal differs slightly for the two samples investigated, with the PTTL signal of KTB-383-C measured in the low temperature protocol showing a clear plateau region, but the TL curves after $IRSL_{50}$ stimulation show a similar trend in both samples: the PTTL signal is larger when lower temperature preheats were used compared to measurements after higher temperature preheats (Fig. 4B and D).

4.4. Results: Monitoring PTTL as part of IRSL measurements using the new user defined command

In section 4.3 we have shown that both samples show PTTL in TL curves. Especially important for feldspar luminescence dating is the potential influence of PTTL on post-IR IRSL signals, and the influence of this is explored in the following.

Figure 5 illustrates the amount of PTTL in post-IR IRSL signals of the two samples studied here. In Figure 5 the different coloured IRSL curves correspond to the different protocols used, with the blue curve representing the post-IR $IRSL_{190}$ signal, green represents the post-IR $IRSL_{250}$ signal and red the post-IR $IRSL_{290}$ signal. Due to the different temperatures used for these measurements, the time needed to ramp up to the measurement temperature differs, which results in the off-set of the start of the ramp in the three curves (Fig. 5). The different parts of the measurement using the new user defined command are indicated below the x-axis in

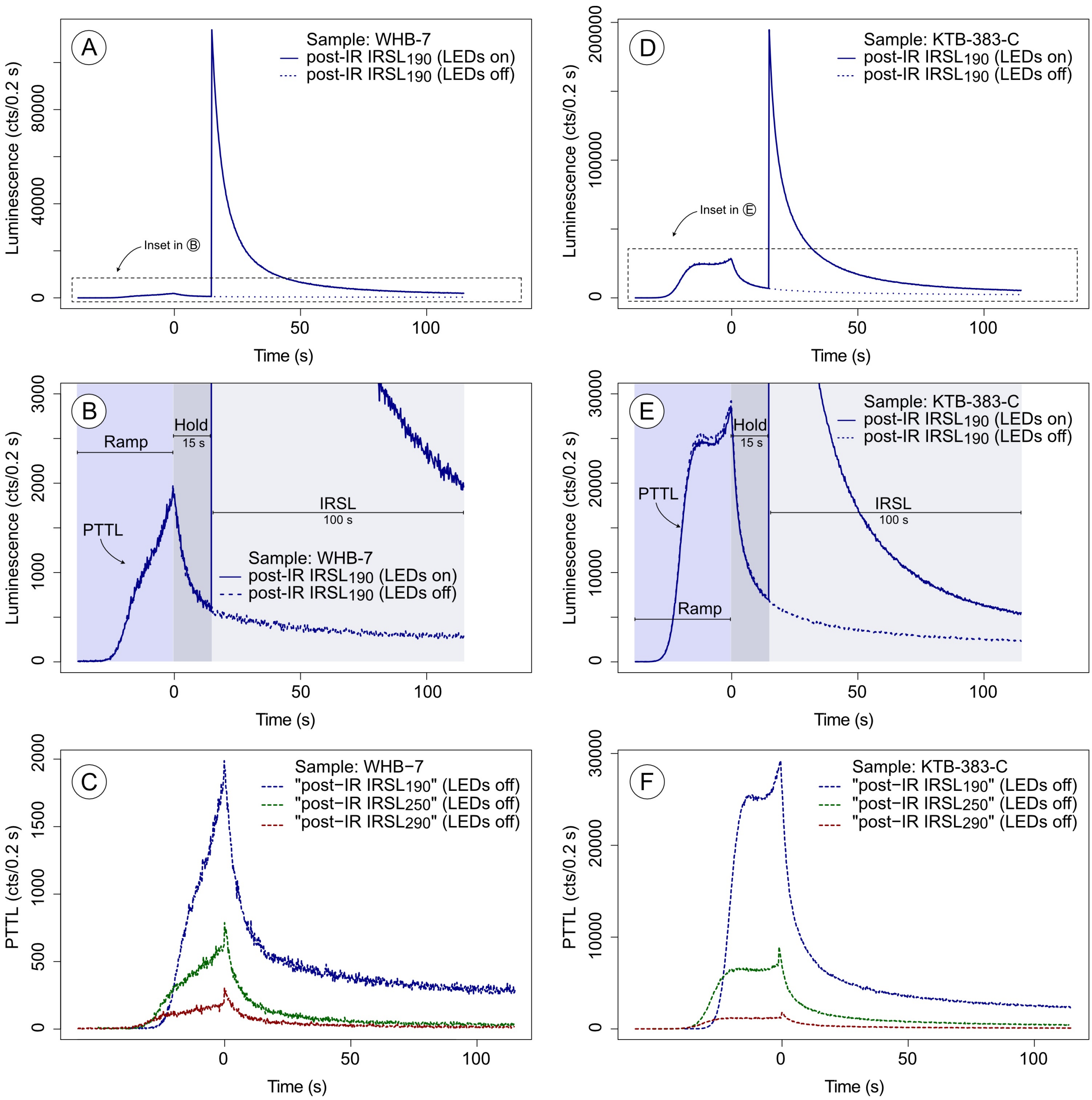

Figure 6: (A) Post-IR $IRSL_{190}$ signal of sample WHB-7 recorded using the protocol shown in Table 3c. The solid line shows the signal recorded with IR LEDs switched on. The dotted line represents the same measurement, but without IR LED stimulation. (B) Close-up of the luminescence signals shown in (A). Subfigures (D) and (E) show results from the same measurements made on sample KTB-383-C. Subfigures (C) and (F) show a comparison of the PTTL "post-IR IRSL" signal recorded for the three different protocols. For these measurements the "post-IR IRSL" signal was recorded using the user defined command, but without optical stimulation.

Figures 5B and 5D.

As already seen for TL, the post-IR IRSL signal recorded using the new user defined command shows a PTTL signal for both samples and for all preheat and post-IR IRSL stimulation temperature combinations. In the recorded post-IR IRSL measurements using the user defined command, the PTTL signal increases during the ramp up to the IRSL measurement temperature. At the end of this ramp the PTTL signal reaches its maximum intensity. During the isothermal holding step, prior to switching on the IR LEDs, the PTTL signal decreases rapidly (Fig. 6). The fastest decay of the PTTL signal is observed in the initial few seconds of the pause, afterwards signal decays more slowly. The intensity and decay behaviour of the PTTL signal is particularly visi-

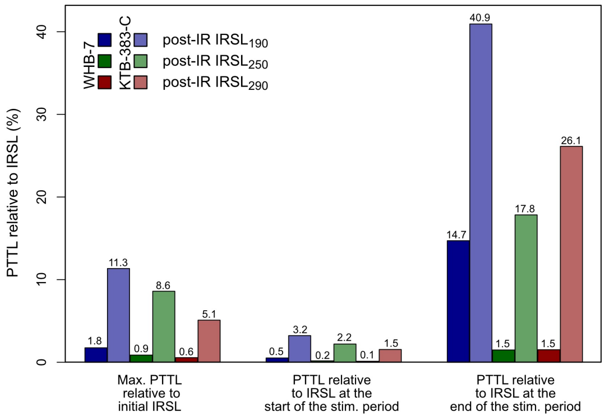

Figure 7: PTTL signal in relation to the measured IRSL signal for WHB-7 and KTB-383-C for all three protocols tested. The respective signals are measured with an integration interval of 0.2 s and the comparison here is based on one channel each. Left: Maximal PTTL signal relative to the maximum IRSL signal intensity. Middle: PTTL signal relative to the IRSL signal, both measured at the time of the start of the stimulation period. Right: Relative comparison of the PTTL and IRSL signal in the last channel of the 100 s stimulation period. The data displayed in the graph corresponds to the data presented in Figure 5

ble in Figure 6, where the PTTL signal is recorded without IR stimulation (dashed curve in Fig. 6) and compared to the corresponding signal with optical stimulation (solid curve in Fig. 6). Figures 6A, B and D, E show the post-IR $IRSL_{190}$ protocol with a 60 s preheat at 220 °C, because this low temperature post-IR IRSL signal shows the largest PTTL signal. For sample WHB-7 the PTTL decreases significantly during the first 5 s of the isothermal holding step, afterwards the decrease stabilises, but remains at a high signal count level, which is even at ~41 % of the post-IR $IRSL_{190}$ signal for KTB-383-C in the later part of the post-IR $IRSL_{190}$ signal (cf. Fig. 6E). Comparing only the PTTL signals for the three different protocols using the new user defined command, it becomes apparent that the PTTL signal of the post-IR $IRSL_{190}$ signal is significantly higher than the PTTL signals of the two higher temperature post-IR IRSL signals (cf. Fig. 6C and F). Whilst for the post-IR $IRSL_{250}$ and post-IR $IRSL_{290}$ a stable background level is reached after 15 to 30 s, dependent on the signal and sample investigated, the PTTL signal of the post-IR $IRSL_{190}$ signal stabilises, but remains at a high level, even after 100 s (cf. Fig. 6C and F; Fig. 7).

Like in TL (Fig. 4), PTTL is largest for the lowest temperature measurement (post-IR $IRSL_{190}$, 220 preheat) and decreases with increasing preheat temperature (Fig. 5). The amount of PTTL present in post-IR IRSL seems to be sample dependent: Whilst the highest PTTL signal intensity is about 11 % of the initial post-IR $IRSL_{190}$ signal for KTB-383-C, it is only 2 % of the initial post-IR $IRSL_{190}$ signal of sample WHB-7 (Fig. 7). However, most important is the finding that the PTTL signal intensity can be reduced in both samples during an isothermal measurement step prior to switching on the IR LEDs. Comparing the IRSL signal intensity and the PTTL intensity at the start of the stimulation period after the isothermal holding step of 15 s reveals that the relative PTTL signal has decreased to ~0.2 % in WHB-7 and to ~2.2 % in KTB-383-C (Fig. 7) – highlighting the importance of the isothermal holding step prior to turning on the stimulation light source. This suggests that the selection of appropriate isothermal holding durations should be based on the properties of each sample and the protocol used. Our data also shows that dependent on the sample and protocol used, the PTTL signal can remain at a high level even after 100 s (cf. Fig. 7).

5. Conclusions

This paper shows that implementing a user defined command in the Mini-Sys can help to assess the intensity and influence of a photo-transferred luminescence signal on a post-IR IRSL measurement. The user defined command can be used instead of a standard IRSL measurement and will then enable the recording of the ramp up to the IRSL measurement temperature, an isothermal holding step at the IRSL measurement temperature and the IRSL measurement using IR LEDs. To allow the recording of the PTTL signal only, the command can be used without stimulation light source. The measurements with and without stimulation can then be compared, particularly to assess an appropriate isothermal holding step prior to optical stimulation.

Acknowledgement

SR's PhD research was funded through an AberDoc PhD scholarship awarded by Aberystwyth University. The authors would like to thank Dr. Anthony M.T. Bell (Sheffield Hallam University, United Kingdom) for performing the XRF and XRD measurements. Benny Guralnik (DTU, Denmark) is thanked for the provision of KTB-383-C. Sample WHB-7 was collected during a field campaign in 2018 which was financially supported by an RGS-IBG postgraduate research grant awarded to SR. We would like to thank Kristina Thomsen for her constructive feedback and review.

References

Armitage, S. J. and Duller, G. A. T. *A user defined command for pulsed-irradiation on Risø TL-OSL readers*. Ancient TL, 22: 9–12, 2004.

Bailiff, I. K. *Use of phototransfer for the anomalous fading of thermoluminescence*. Nature, 264: 531–533, 1976.

Brown, W. L. and Parsons, I. *Exsolution and coarsening mechanisms and kinetics in an ordered cryptoperthite series*. Contributions to Mineralogy and Petrology, 86: 3–18, 1984.

Buylaert, J.-P., Jain, M., Murray, A. S., Thomsen, K. J., Thiel, C., and Sohbati, R. *A robust feldspar luminescence dating method for Middle and Late Pleistocene sediments*. Boreas, 41: 435–451, 2012.

Duller, G. A. T. *Infrared bleaching of the thermoluminescence of four feldspars*. Journal of Physics D: Applied Physics, 28: 1244–1528, 1995.

Duller, G. A. T. *The Analyst software package for luminescence data: overview and recent improvements*. Ancient TL, 33: 35–41, 2015.

Guralnik, B., Jain, M., Herman, F., Ankjærgaard, C., Murray, A. S., Valla, P. G., Preusser, F., King, G. E., Chen, R., Lowick, S. E., Kook, M., and Rhodes, E. J. *OSL-thermochronometry of feldspar from the KTB borehole, Germany*. Earth and Planetary Science Letters, 423: 232–243, 2015.

Kaylor, R. M., Feathers, J., Hornyak, W. F., and Franklin, A. D. *Optically stimulated luminescence in Kalahari quartz: Bleaching of the 325 °C peak as the source of the luminescence*. Journal of Luminescence, 65: 1–6, 1995.

Kristianpoller, N., Weiss, D., and Chen, R. *Effects of photostimulation in natural zircon*. Radiation Measurements, 41: 961–966, 2006.

Li, B. and Li, S.-H. *Thermal stability of infrared stimulated luminescence of sedimentary K-feldspar*. Radiation Measurements, 46: 29–36, 2011.

Lin, T.-H. and Yund, R. A. *Potassium and Sodium Self-Diffusion in Alkali Feldspar*. Contributions to Mineralogy and Petrology, 34: 177–184, 1972.

Murray, A. S., Buylaert, J. P., Thomsen, K. J., and Jain, M. *The effect of preheating on the IRSL signal from feldspar*. Radiation Measurements, 44: 554–559, 2009.

Parsons, I., Fitz Gerald, J. D., and Lee, M. R. *Routine characterization and interpretation of complex alkali feldspar intergrowths*. American Mineralogist, 100: 1277–1303, 2015.

Qin, J., Chen, J., and Salisbury, J. B. *Photon transferred TL signals from potassium feldspars and their effects on post-IR IRSL measurements*. Journal of Luminescence, 160: 1–8, 2015.

R Core Team. *R: A Language and Environment for Statistical Computing*. Vienna, Austria, 2019. URL https://r-project.org.

Riedesel, S., King, G. E., Prasad, A. K., Kumar, R., Finch, A. A., and Jain, M. *Optical determination of the width of the band-tail states, and the excited and ground state energies of the principal dosimetric trap in feldspar*. Radiation Measurements, 125: 40–51, 2019.

Riedesel, S., Bell, A. M. T., Duller, G. A. T., Finch, A. A., Jain, M., King, G. E., Pearce, N. J., and Roberts, H. M. *Exploring sources of variation in thermoluminescence emissions and anomalous fading in alkali feldspar*. Radiation Measurements, 141: 106541, 2021.

Risø, D. T. U. Nutech. *Guide to The Risø TL/OSL Reader*. User manual. DTU Nutech. Denmark, 2015.

Robertson, G. B., Prescott, J. R., and Hutton, J. T. *Bleaching of the thermoluminescence of feldspars by selected wavelengths present in sunlight*. Nuclear Tracks and Radiation Measurements, 21: 245–251, 1993.

Schlesinger, M. *Optical studies of electron and hole trapping levels in quartz*. Journal of Physics and Chemistry of Solids, 26: 1761–1766, 1965.

Thiel, C., Buylaert, J.-P., Murray, A. S., Terhorst, B., Hofer, I., Tsukamoto, S., and Frechen, M. *Luminescence dating of the Stratzing loess profile (Austria) - Testing the potential of an elevated temperature post-IR IRSL protocol*. Quaternary International, 234: 23–31, 2011.

Thomsen, K. J., Murray, A. S., Jain, M., and Bøtter-Jensen, L. *Laboratory fading rates of various luminescence signals from feldspar-rich sediment extracts*. Radiation Measurements, 43: 1474–1486, 2008.

Tsukamoto, S., Jain, M., Murray, A. S., Thiel, C., Schmidt, E., Wacha, L., Dohrmann, R., and Frechen, M. *A comparative study of the luminescence characteristics of polymineral fine grains and coarse-grained K- and Na-rich feldspars*. Radiation Measurements, 47: 903–908, 2012.

Wang, X. L. and Wintle, A. G. *Investigating the contribution of recuperated TL to post-IR IRSL signals in perthitic feldspar*. Radiation Measurements, 49: 82–87, 2013.

Wang, X. L., Wintle, A. G., and Adamiec, G. *Post-IR IRSL production in perthitic feldspar*. Radiation Measurements, 64: 1–8, 2014.

Wintle, A. G. and Murray, A. S. *The relationship between quartz thermoluminescence, photo-transferred thermoluminescence, and optically stimulated luminescence*. Radiation Measurements, 27: 611–624, 1997.

Reviewer

Kristina Thomsen