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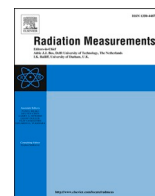
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On the use of new vs. heated sample carriers for luminescence measurements

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ABSTRACT

To ensure precise luminescence measurements, it is crucial to carry out measurements on clean sample carriers. Moreover, it is essential to ensure that the light emitted by the studied sample is reproducibly reflected towards the detector, particularly when the sample exhibits dim luminescence. This study reports observations made by three laboratories working in the field of retrospective dosimetry. The study discusses the results obtained using new sample carriers (made of stainless steel), instead of sample carriers previously heated to a temperature of 450 °C. The article describes the cleaning protocols routinely used by each laboratory and the treatment with distilled water that effectively removed spurious signals. When measuring TL signals of resistors using new sample carriers, a signal reduction of 10% was observed in the second measurement compared to the initial TL. For display glass samples from a mobile phone, a signal reduction between 15% and 32% was observed. However, there was negligible change in reproducibility when using the previously heated sample carriers. The reflectance of the sample carrier surface was indirectly measured using breakthrough signal measurements using violet or blue LEDs. A signal reduction of 12–14 % was observed when heating was applied for 20 new, unheated sample carriers using violet LEDs. The impact on dose estimation is discussed, and some recommendations are provided to guarantee precise luminescence measurements.

1. Introduction

Luminescence techniques, such as thermoluminescence (TL) and optically stimulated luminescence (OSL), are utilized in various dosimetry applications including dating or retrospective dosimetry. In recent decades, they have been widely applied to investigate the dosimetric properties of materials from personal items carried by the general population (ICRU, 2019). These materials could indeed be used as potential emergency dosimeters for a rapid and accurate retrospective assessment of the dose absorbed by victims in the event of a radiological accident. For example, measurement protocols were developed using OSL of resistors (Bassinet et al., 2014), TL of resistors (Ademola and Woda, 2017) and TL of display glass (Discher and Woda, 2013) from mobile phones. To conduct luminescence measurements, the prepared sample is put on a sample carrier specific to the luminescence reader. If necessary (such as for resistors), a thin layer of silicon oil is sprayed on the sample carrier surface to adhere the sample. To examine the dosimetric properties of a material, such as dose response or signal stability,

several “irradiation - luminescence measurement” cycles are generally performed on a single sample aliquot. In addition, the procedures developed to estimate a dose from a sample aliquot also include the repetition of the measurement cycle. The steps are as follows: First, measure the luminescence signal related to the unknown dose (i.e. “accident” dose) using optimized measurement parameters. Then, administer a known calibration dose to calibrate the radiation sensitivity of the sample aliquot and measure the resulting luminescence signal using the same measurement parameters. The dose can be estimated by analysing the intensity of the luminescence signals. Correction factors may be applied, particularly, to take into account for fading or energy dependence.

Among the prerequisites necessary for properly investigating the dosimetric properties of materials, it is crucial to eliminate any spurious signals in the luminescence integration range that could interfere with the luminescence signal of the material under study. This includes signals that could be attributed to the sample carrier. A protocol for cleaning carriers should be robust enough to remove any contamination

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caused by residual materials deposited from previous measurements, such as traces of residual samples and silicone oil (Vandenberghue et al., 2008). It is also important to clean new sample carriers, as they may have been contaminated during the manufacturing process (Schmidt et al., 2011; Simkins et al., 2013; Kazakis et al., 2015). Schmidt et al. (2011) proposed that a spurious signal may be caused by luminescent chemical compounds of the carrier material itself.

Additionally, it is important to ensure that the light emitted by the sample is reproducibly reflected towards the detector, including the reflectance at the surface of the sample and its carrier from one measurement cycle to the next. Otherwise, when studying the reproducibility of the luminescence signal for example, repeatedly giving the same dose and measuring the luminescence signal after each irradiation, changes in reflectivity could lead to incorrect highlighting of sensitivity changes.

The present study reports on the results obtained from using new stainless steel sample carriers (new cups) compared to sample carriers previously heated up to 450 °C (heated cups). Observations made by three laboratories involved in the field of retrospective dosimetry are reported. The effectiveness of the cleaning protocols used by these teams on unused sample carriers is evaluated. The study also discusses the impact of the difference in reflectance between new and heated sample carriers for dose estimation.

2. Materials and methods

2.1. Luminescence readers

At the *Salzburg laboratory*, two automated readers Lexsyg Research and Lexsyg Smart made by Freiberg Instruments (Richter et al., 2013, 2015) were used for luminescence measurements. The Lexsyg readers are equipped with a built-in Sr-90/Y-90 beta source for laboratory irradiations and calibrated for glass against air kerma using a Cs-137 gamma source. The Lexsyg Research and Lexsyg Smart sources delivered a dose rate of ≈ 0.046 Gy/s and ≈ 0.098 Gy/s, respectively. The Lexsyg readers are equipped with an optical stimulation module with three stimulation wavelengths, but for this study only two wavelengths were used: violet LEDs (405 ± 3 nm) on the Research reader and blue LEDs (458 ± 5 nm) on both readers. Both Lexsyg readers have a bi-alkaline cathode photomultiplier tube from Hamamatsu (H7360-02) used for detection.

At the *Institut de Radioprotection et de Sûreté Nucléaire* (IRSN), luminescence measurements were performed with an automated Lexsyg Smart reader, equipped with a Hamamatsu H7360-02 bi-alkaline photomultiplier tube. In this study, violet LEDs (405 ± 3 nm) and blue LEDs (458 ± 5 nm) were utilized. The reader is equipped with a built-in Sr-90/Y-90 beta source calibrated in air kerma from TL measurements of glasses (LCD) irradiated at the IRSN's linear accelerator (Elekta Synergy® Platform) with a 4 MV photon beam. The dose rate of the beta source was ≈ 48 mGy/s. The sample carriers (cups) of all Lexsyg readers are made of stainless steel (X15CrNiSi25-21). The material is heat resistant up to 1150 °C and the inner diameter of the sample carrier is 7.95 ± 0.10 mm (Mauz et al., 2021).

At the *Korea Atomic Energy Research Institute* (KAERI), a Risø-TL/OSL-DA-20 reader (Lapp et al., 2015) with a detection and stimulation head (DASH) upgrade unit was available. The optical stimulation was carried out by built-in 470 nm blue LEDs. A blue-sensitive PMT (Electron Tube PDM9107Q-AP-TTL-03) was used to detect luminescence signals. Samples were irradiated using a built-in beta source (Sr-90/Y-90) with a dose rate of ≈ 6.6 mGy/s in terms of Cs-137 equivalent air kerma for display glasses and resistors. The Risø reader uses stainless steel sample carriers with a diameter of 11.7 mm.

Table 1 lists the various filter combinations for the Lexsyg and Risø readers for different measurement protocols.

Table 1

Overview of the protocols, measurement parameters and detection filter combinations used for each institute.

Protocol	Used measurement parameters	Detection filters	Institute
TL with blue filter combination	TL measurement up to 450 °C with a heating rate of 2 °C/s	Wideband blue filter pack: Schott BG39 + BG25 + KG3	IRSN
		Wideband blue filter pack: Schott BG39 + BG25 + KG3 Blue filter pack: Schott BG3 and BG39	Salzburg KAERI
TL with UV filter combination	TL measurement up to 450 °C with a heating rate of 2 °C/s	UV filter combination: Hoya U340 and Delta-BP 365/50 EX	IRSN
		UV filter combination: Hoya U-340 and Delta-BP 365/50 EX	Salzburg
“Pre-bleached TL” protocol for glass	TL measurement up to 450 °C with a 500 s blue LEDs pre-bleaching	UV filter: Hoya U-340 and Delta-BP 365/50 EX	IRSN
		UV filter: Hoya U-340 and Delta-BP 365/50 EX	Salzburg
“OSL with preheat” protocol for resistors	OSL measurement at 100 °C for 30 s after a preheat of 10 s at 120 °C	UV filter: Hoya U-340	KAERI
		UV filter: Hoya U-340	KAERI
“TL” protocol for resistors	TL measurement up to 400 °C with a heating rate of 2 °C/s	UV filter: Hoya U-340	KAERI
Protocol to estimate the reflectance of the carrier's surface	VSL measurement for 100 s at room temperature	UV filter: Hoya U340 and Semrock HC 340/26	IRSN
		UV filter: Hoya U340 and AHP-BrightLine HC340/26	Salzburg
		Blue filter pack: Schott BG3 and BG39 and a neutral density filter OD6	KAERI

2.2. Protocols developed for retrospective dosimetry used in the present study

The main measurement parameters of protocols developed for retrospective dosimetry and applied in the present study are described below. For the “OSL with preheat” protocol for resistors (Bassin et al., 2014), OSL measurement is performed at 100 °C for 30 s after a preheat of 10 s at 120 °C. Early background signals from 6 to 12 s are subtracted from initial OSL signals from 0 to 6 s. For the “TL” protocol for resistors (Ademola and Woda, 2017), electronic components are heated up to 400 °C with a heating rate of 2 °C/s. To suppress the black body radiation effect at high temperature regions, the second TL reading is subtracted from the first one. The TL signal integration window is set to 100–200 °C. For the “pre-bleached TL” protocol for glass (Discher and Woda, 2013), TL measurements are performed after a 500 s optical blue stimulation (pre-bleaching). A heating rate of 2 °C/s is used to reach the readout temperature ($T_{\max} = 450$ °C) with a hold time of 10 s. A second TL reading is performed for thermal background subtraction. The recorded TL signals are integrated between 100 and 250 °C for analysis. All TL measurements are performed in a N₂ atmosphere.

2.3. Samples used to test the measurement reproducibility and their preparation

At Salzburg and at IRSN, glass samples extracted from display of mobile phones (size approx. $5 \times 5 \text{ mm}^2$) were used in this study. They belonged to category A, according to the glow curve characterization found in Discher and Woda (2013). A TL measurement to a maximum temperature of $450 \text{ }^\circ\text{C}$ with a hold time of 120 s was carried out to eliminate any native signals before using the glass. The “pre-bleached TL” protocol for glass (Discher and Woda, 2013) was used to test the reproducibility of the measurement.

At KAERI, resistors with a size of $1.00 \times 0.50 \times 0.35 \text{ mm}^3$ (0402 package type; SMD 1005, 510 Ohm, Samsung Electro Mechanics Corporation) were used from a roll of resistors. 10 resistors were mounted per sample carrier. The typical composition of a resistor is known to be 97% Al_2O_3 and 3% SiO_2 (Inrig et al., 2008). The resistors were heated to $450 \text{ }^\circ\text{C}$ to remove any native signals prior to measurements and to avoid any sensitivity changes due to heat treatments. The “OSL with preheat” protocol (Bassin et al., 2014) and the “TL” protocol (Ademola and Woda, 2017) for resistors were then used to test the reproducibility of the measurements. The studied glass samples were extracted from the AMOLED display of a mobile phone (Galaxy S3 model). The TL glow curve of category A was confirmed in the immediately preceding measurement and the glass samples were annealed to remove any native signals. The “pre-bleached TL” protocol for glass (Discher and Woda, 2013) was then used to test the measurement reproducibility.

2.4. Cleaning procedures of sample carriers

Sample carriers of the Lexsyg readers are routinely cleaned at the Salzburg laboratory by using butanone and methanol in an ultrasonic bath for 10 min. Similarly, at IRSN, the sample carriers of the Lexsyg Smart reader are routinely cleaned using acetone in an ultrasonic bath for 10 min (twice) and distilled water for 10 min. At KAERI, sample carriers of the Risø reader are routinely cleaned using ethanol in an ultrasonic bath for 20 min.

To ensure that no spurious signal remains in the sample carriers, the efficiency of the cleaning procedures routinely used by the three laboratories was tested against three protocols developed for retrospective dosimetry (see section 2.2). In each laboratory, for each retrospective dosimetry protocol, new empty sample carriers were cleaned. Their luminescence signal was then recorded after beta irradiation ($\sim 5 \text{ Gy}$) using the measurement parameters specific to the protocol investigated.

2.5. Measurement protocol used for reflectance test

To indirectly quantify the reflectance of the sample carrier surface, the breakthrough signal caused by the blue or violet LED light and the used filter combination was recorded for a duration of 100 s. The LEDs were switched on only for the first 50 s, and the subsequent 50 s were used to monitor the background level of the readout system. For analysis, the average of the first 50 s and its standard deviation were calculated.

3. Results and discussions

3.1. Efficiency of the cleaning procedures routinely used by the three laboratories

The results obtained by each laboratory using its own cleaning protocol on new and empty sample carriers and the parameters optimized for retrospective dosimetry protocols showed that the spurious signals were negligible over the integration range because the spurious signals were indistinguishable from the dark count level of the system. Thus, the efficiency of the cleaning procedures was confirmed for the use of these protocols by the laboratories.

However, it is important to emphasize that the results are highly dependent on the filters and the sensitivity of the readout system used. To illustrate this, TL measurements of uncleaned and unused sample carriers were recorded after beta irradiation (approx. 5 Gy) up to $450 \text{ }^\circ\text{C}$ for the different readers (Risø, Lexsyg Research and both Lexsyg Smart readers) using a UV or a blue filter combination (see Fig. 1). Fig. 1a shows that TL signals appear in the higher temperature ranges ($T > 200 \text{ }^\circ\text{C}$) for the sample carriers of a Lexsyg Smart reader when using the blue filter combination. These signals which are suspected to be caused by impurities from the manufacturing process completely disappeared in a second cycle of beta irradiation and TL readout. No spurious TL signals were detected for TL measurements of uncleaned and unused sample carriers using the UV filter combination and the same luminescence reader (Fig. 1b).

Spurious TL signals were also observed when using the blue detection filter combination but a different Lexsyg Smart reader system (Fig. 1c). However, using the blue filter combination on the Lexsyg Research reader the signals were detected at significantly lower intensities (Fig. 1d) which can be explained by the greater distance between the sample and the PMT. Spurious TL signals ($T > 150 \text{ }^\circ\text{C}$) were also observed for the sample carriers of the Risø reader when using the blue filter combination, but with a different shape (Fig. 1e).

The reduction of spurious TL signals solely by a cleaning treatment with distilled water for 10 min was tested. The results are shown in Fig. 2. Comparing the recorded signals in Figs. 1a and 2a (Lexsyg Smart reader) and in Figs. 1e and 2b (Risø reader) respectively, the distilled water cleaning method is very efficient in eliminating the TL spurious signals.

3.2. Study of new vs. heated Lexsyg sample carriers

20 new sample carriers were used for this experiment at Salzburg laboratory. Again, in order to exclude any contamination of the previously cleaned sample carriers, all 20 sample carriers were irradiated ($\sim 10.4 \text{ Gy}$) and a measurement with violet LEDs was performed to check for any luminescence signals. All sample carriers passed this pre-test, and it was proven that all sample carriers were clean.

The next step was to anneal 10 glass aliquots on sample carriers (at pos. 1–10) by applying a TL measurement, so consequently these 10 sample carriers were exposed to heat up to $450 \text{ }^\circ\text{C}$ for the first time. Then, a simple measurement repetition test of the annealed glass samples was performed using the “pre-bleached TL” protocol (Discher and Woda, 2013) to test the reproducibility and monitor any sensitivity changes of the radiation-induced signals. Sequential irradiation and measurement cycles ($N = 5$) were performed with a dose of $\sim 1 \text{ Gy}$. The TL glow curves of a glass aliquot are shown in Fig. 3a. After 5 cycles, the change in sensitivity was $< 3\%$. Similar observations were made for the other nine glass aliquots. The same measurement sequence was repeated with sample carriers (at pos. 11–20) that had not been previously exposed to heat, and the glass aliquots from the previous measurements were recycled. The TL glow curves of one glass aliquot are shown in Fig. 3b. It was the same glass aliquot as shown in Fig. 3a. Contrary to the results obtained with the heated sample carrier, a significant decrease in the TL intensity was observed after the second cycle compared to the first cycle for the glass aliquot on an unheated sample carrier (see Fig. 3b). Similar observations were confirmed for the other nine glass aliquots on the unheated sample carriers.

To quantify the signal reduction effect of an unheated sample carrier on the luminescence intensity compared to a heated sample carrier, the integrated TL signals ($100\text{--}250 \text{ }^\circ\text{C}$) for each cycle were normalized to those of the first measurement (cycle #1). The ratios are depicted in Fig. 4.

Small variations (between 1 and 5%) of the signals were observed for all aliquots using the heated sample carriers on pos. 1–10. The results are consistent with the findings of a reproducibility test by Discher and Woda (2013). For the unheated sample carriers on pos. 11–20, a

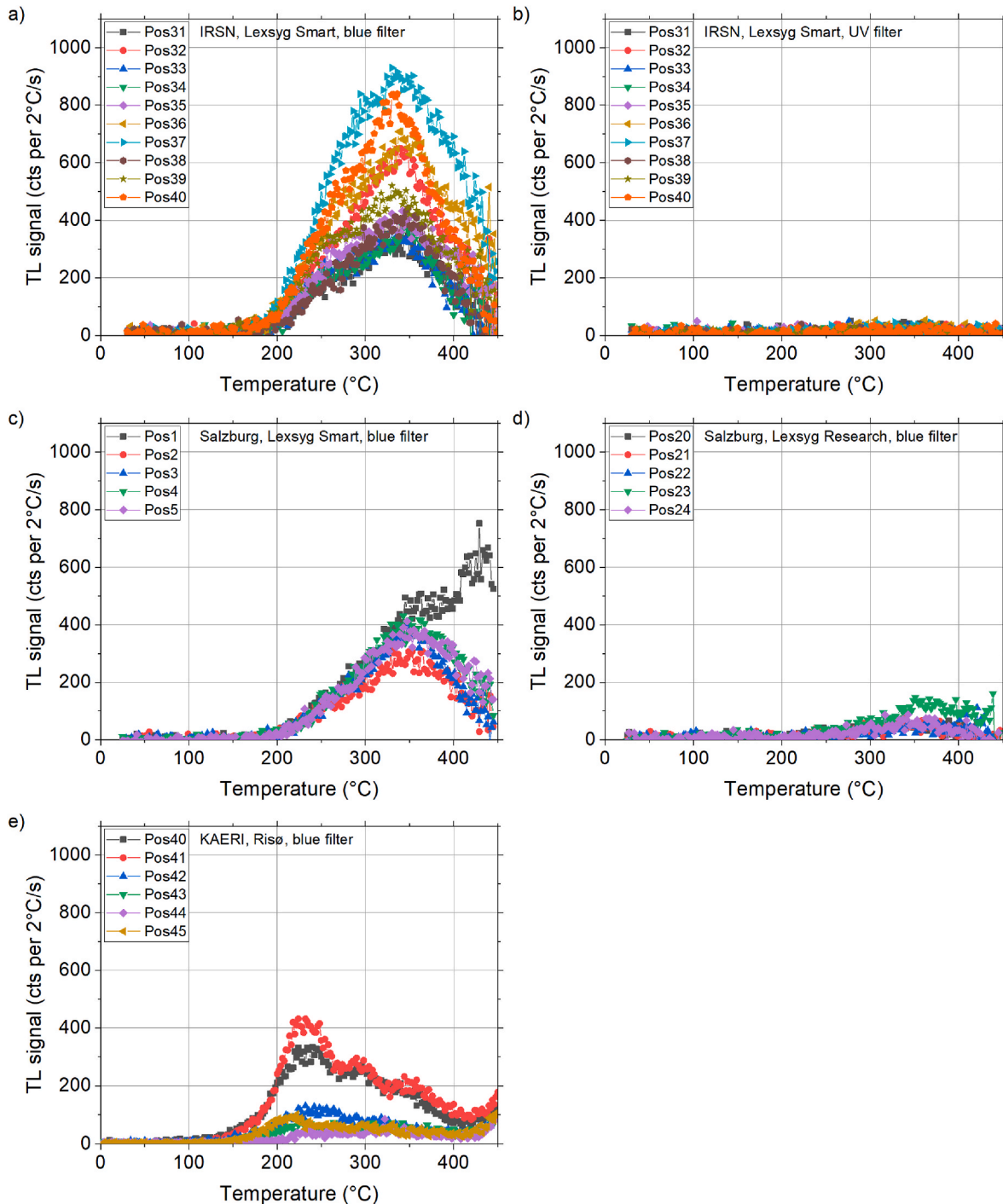


Fig. 1. TL signals of empty, uncleaned and unheated sample carriers recorded using different filter combinations and readers: a) IRSN Leksy Smart using blue filter combination, b) IRSN Leksy Smart using UV filter combination, c) Salzburg Leksy Smart using blue filter combination, d) Salzburg Leksy Research using blue filter combination, e) KAERI Risø using blue filter combination.

significant reduction of the integrated TL signals was observed when comparing cycle #1 to cycle #2. The reduction ranges from 15 to 32%. After the first heat exposure during the TL measurement, the reproducibility of measurement cycles #2–5 is in the expected range of (1–5%) and re-irradiation and readout do not significantly change the signal sensitivity.

The blue stimulated luminescence (BSL) signals measured during the pre-bleaching process, which are recorded before the TL reading, were also analyzed to quantify the effect of using unheated sample carriers on the luminescence intensity. Similar to the TL measurements, the ratios of

the integrated BSL signals (0–1 s) to the first measurement (cycle #1) were calculated and normalized to the first cycle. In Fig. 5 the ratios are shown.

For heated sample carriers on pos. 1–10, a reduction of the integrated BSL signals in the range of 5–9% was observed when comparing cycle #1 to cycle #2. For unheated sample carriers on pos. 11–20, a significant reduction of the BSL signals was observed, ranging between 32 and 52%. After the first measurement cycle, the sensitivity trends to still decrease. It is expected that the degree of reflectivity of the sample carrier surface is affected by the first heat exposure (such as the first TL measurement).

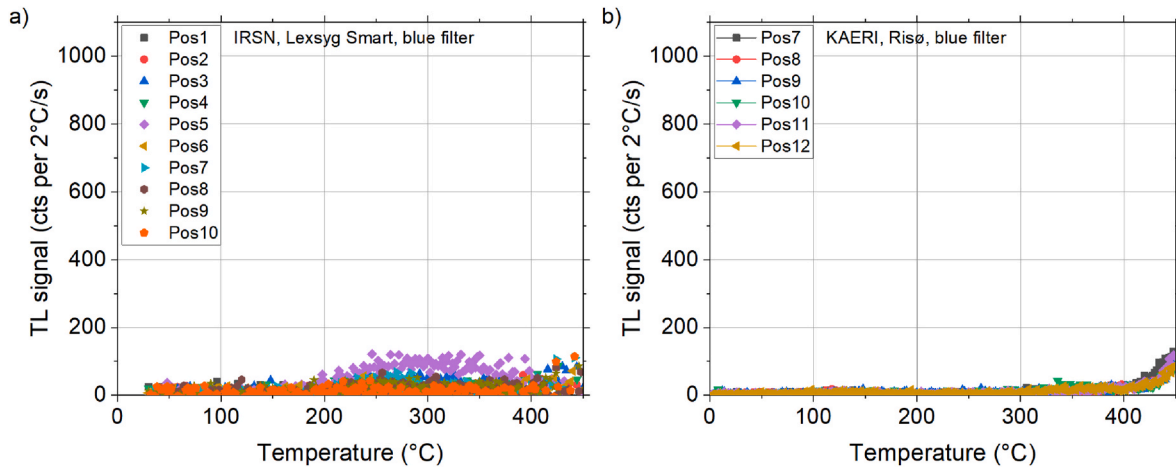


Fig. 2. TL signals of empty and unheated sample carriers recorded after cleaning with distilled water on a Lexsyg Smart (a) and Risø reader (b).

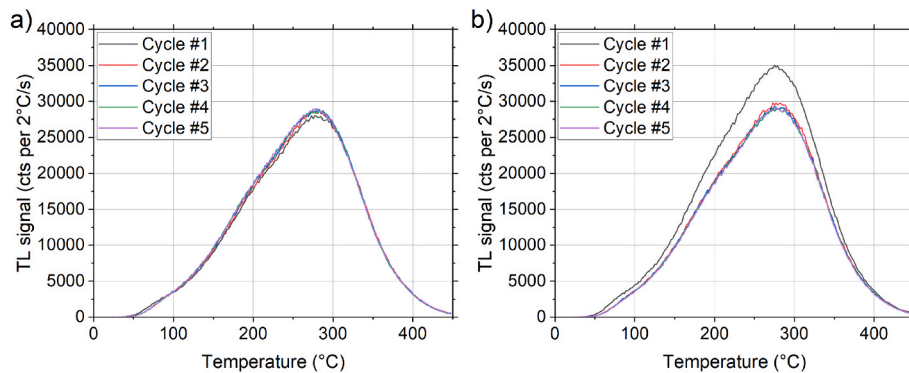


Fig. 3. Pre-bleached TL glow curves of the same glass aliquot placed on a previously heated sample carrier (a) and on an unheated sample carrier (b).

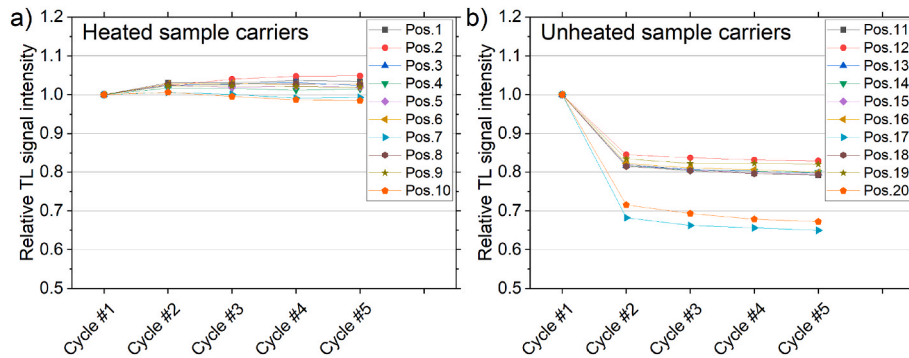


Fig. 4. Ratios of the integrated TL signals (100–250 °C) for the 10 glass aliquots placed on the (a) previously heated (Pos. 1–10) and (b) unheated sample carriers (Pos. 11–20). The data are normalized to the first cycle.

Obviously, the heating history of the sample carrier plays a more dominant role for OSL measurements of glasses due to the fact that this material is transparent and the optical properties (reflectance) of the sample carrier surface are considerable importance.

The same type of experiments was conducted at IRSN on the Lexsyg Smart using a different batch of new sample carriers and glass aliquots, and similar results were observed (see Fig. A1 in the Appendix).

3.3. Study of previously heated vs. unheated Risø sample carriers

For this study, 30 new, unheated sample carriers were used. Half of them were annealed using the Risø reader at 450 °C for 120 s to mimic a

used sample carrier. The other half represents the unused sample carrier group. Reproducibility according to the measurement protocols and materials (“OSL with preheat” protocol for resistors, “TL” protocol for resistors and “pre-bleached TL” protocol for glass, see section 2.2) and the initial state of a sample carrier was obtained by applying a test dose (approx. 1 Gy) and repeating the irradiation-reading cycle 10 times as shown in Fig. 6. Signals were normalized to the first measurement. To show a statistical trend, each reproducibility curve represents the averaged signal from 5 sample carriers, and their standard deviation is presented as error bars.

In Fig. 6a, no significant changes in sensitivity were observed for the “OSL with preheat” protocol for resistors. Although the protocol

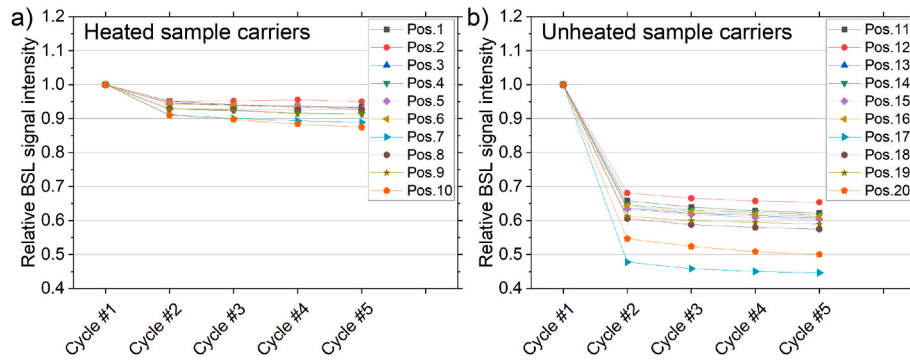


Fig. 5. Ratios of the integrated BSL signals (integral 0–1 s) obtained from the pre-bleaching step of the protocol for the glass aliquots placed on the (a) previously heated on pos. 1–10 and (b) unheated sample carriers on pos. 11–20. The data are normalized to the first cycle.

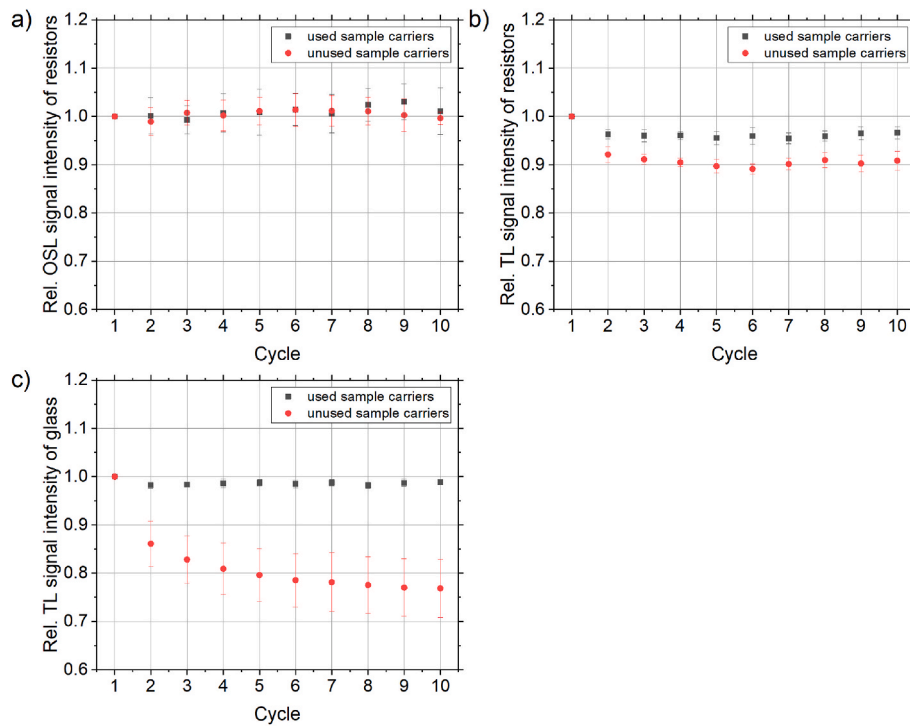


Fig. 6. Comparison of reproducibility between used and unused sample carriers for the “OSL with preheat” protocol for resistors (a), the “TL” protocol for resistors (b) and “pre-bleached TL” protocol for glass (c).

includes the preheating at 120 °C for 10 s, the heat treatment is considered insufficient to cause a significant change in sensitivity. The higher error bars compared to the “TL” protocol for resistors in Fig. 6b are due to the sample-to-sample variation caused by the lower intensity of OSL than TL.

The “TL” protocol for resistors causes a difference in sensitivity between used and unused sample carriers in Fig. 6b. In the case of the unused sample carriers, a decrease of up to 10% from the initial signal is confirmed, and there is no further change between the other measurements. The used sample carriers also show a change in sensitivity at the second measurement, but it is less than 5%. This slight decrease may indicate that the heat treatment to mimic a used sample was insufficient. Fig. 6c shows the similar result to section 3.2 for glass samples. For the used sample carrier, the highest signal decrease of about 15% is found between the first and second measurement, and the signal decrease is maintained up to 8 times, showing a maximum average decrease of 23% compared to the first measurement. The discrepancy in the duration of the signal decrease can be attributed to differences in the equipment (i.e. the readers and sample carriers, respectively). In addition, the

sensitivity changes of the unused sample carriers showed about 8% standard deviation, confirming that the sample variation was large.

The results show that the use of new sample carriers in dose assessment using TL of fortuitous materials, such as resistors and glasses, can introduce an overestimation in dose, and the effect is more pronounced for glass samples. The reason why the signal reduction of glasses is greater than that of resistors is speculated to be the effect of back reflection due to the difference in transparency.

3.4. Indirect measurement of the reflectivity of sample carriers

Violet stimulated luminescence (VSL) measurements from luminescence readers can be used to qualitatively investigate the effect of initial heat exposure on an unheated sample carrier and the reduction of its reflectance. A breakthrough signal is detected with the available filter combination of the reader when the violet LEDs are switched on.

At Salzburg laboratory, the 20 sample carriers used in section 3.2 were measured with the Lexsyg Research reader using VSL before any heat treatment and after several cycles of TL measurements during the

measurement campaign of the reproducibility tests. Fig. 7 shows the signals detected by the PMT for the same sample carrier as an example.

For further analysis, the mean of the breakthrough signal (0–50 s) was calculated. The reduction of the signal (R_{signal}) was simply calculated by: $R_{\text{signal}} = 100\% - (\text{Sig}_{\text{heated cup}}/\text{Sig}_{\text{unheated cup}})$.

For the 20 sample carriers the R_{signal} value ranges between 12 and 14 % ($13 \pm 1\%$ in average).

A series of breakthrough signal measurements were performed on a new, unheated sample to measure the effect of the heat exposure on the sample carrier. Between each measurement, the sample carrier was heated to 450 °C and held for 60 s. The results of 10 cycles are shown in Fig. 8 for the signals normalized to the initial measurement. The largest difference in relative signal is shown between the first and second measurements. Afterwards a continuous decrease is observed until the 10th measurement, showing a plateau of an initial signal reduction of 15%. The result supports the hypothesis that the reflected breakthrough signals are reduced by a change in the sample carrier surface after a heat treatment.

The same type of experiments was conducted at IRSN on the Lexsyg Smart reader and at KAERI on the Risø reader using new sample carriers. Similar results were observed for the Lexsyg sample carriers using the VSL measurement, but the Risø sample carriers using the BSL measurement showed a continuous decrease, and no plateau was observed after 11 measurement cycles (see Fig. A2 in the Appendix).

4. Conclusions and recommendations

This study discovers that knowing the heating history of the sample carriers is important in luminescence studies. The authors made the following general recommendations:

- New sample carriers should always be cleaned to remove any contamination from the manufacturing process using a standard cleaning method and should be routinely tested by each laboratory. A final cleaning step with distilled water is recommended.
- Separation of the heated and unheated sample carriers in the laboratory (sample carriers for TL vs. sample carriers for OSL measurements) is recommended.
- With respect to TL, a heat exposure up to 450 °C of the new, unused sample carriers is necessary to avoid overestimations of TL signal. The use of an oven or a simple TL measurement of empty sample carriers is recommended to reduce this effect.
- With regard to OSL, unheated sample carriers are recommended when measuring weak OSL signals and when no heating is applied. It is important to note that reduced reflectivity due to heat treatment at 450 °C can cause a significant signal reduction of about 50%, which should be considered for weakly luminescent materials or samples with low sensitivity due to low dose. However, a heated sample carrier should be used for OSL measurements that involve heating processes, to avoid sensitivity changes.

CRediT authorship contribution statement

Michael Discher: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Céline Bassinet:** Writing – review & editing, Writing – original draft, Validation, Investigation, Formal analysis, Conceptualization. **Hyoungtaek Kim:** Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

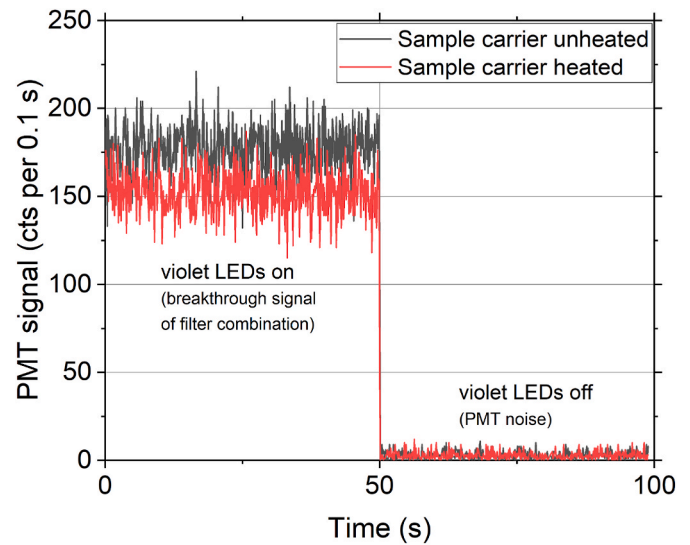


Fig. 7. Breakthrough signal of the filter combination by the violet LEDs. The PMT recorded 100 s in total, however, the LEDs were only switched on for the first 50 s. The same sample carrier was compared.

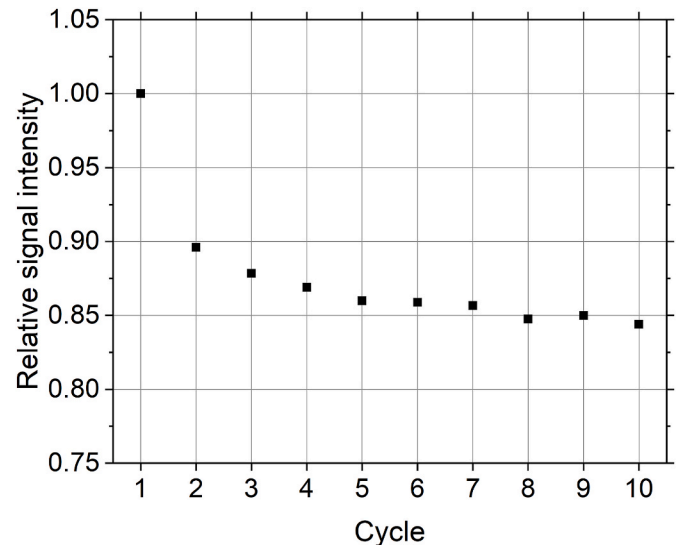


Fig. 8. Series of breakthrough signal measurements. Between each measurement the sample carrier was heated to 450 °C and held for 60 s in order to measure the impact of the heat exposure. Data are normalized to the first cycle.

Data availability

Data will be made available on request.

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Appendix

The same type of experiments as presented in section 3.2 were performed at IRSN on the Lexsyg Smart reader using a different batch of new sample carriers and glass aliquots. Similar results were observed and are shown in Fig. A1.

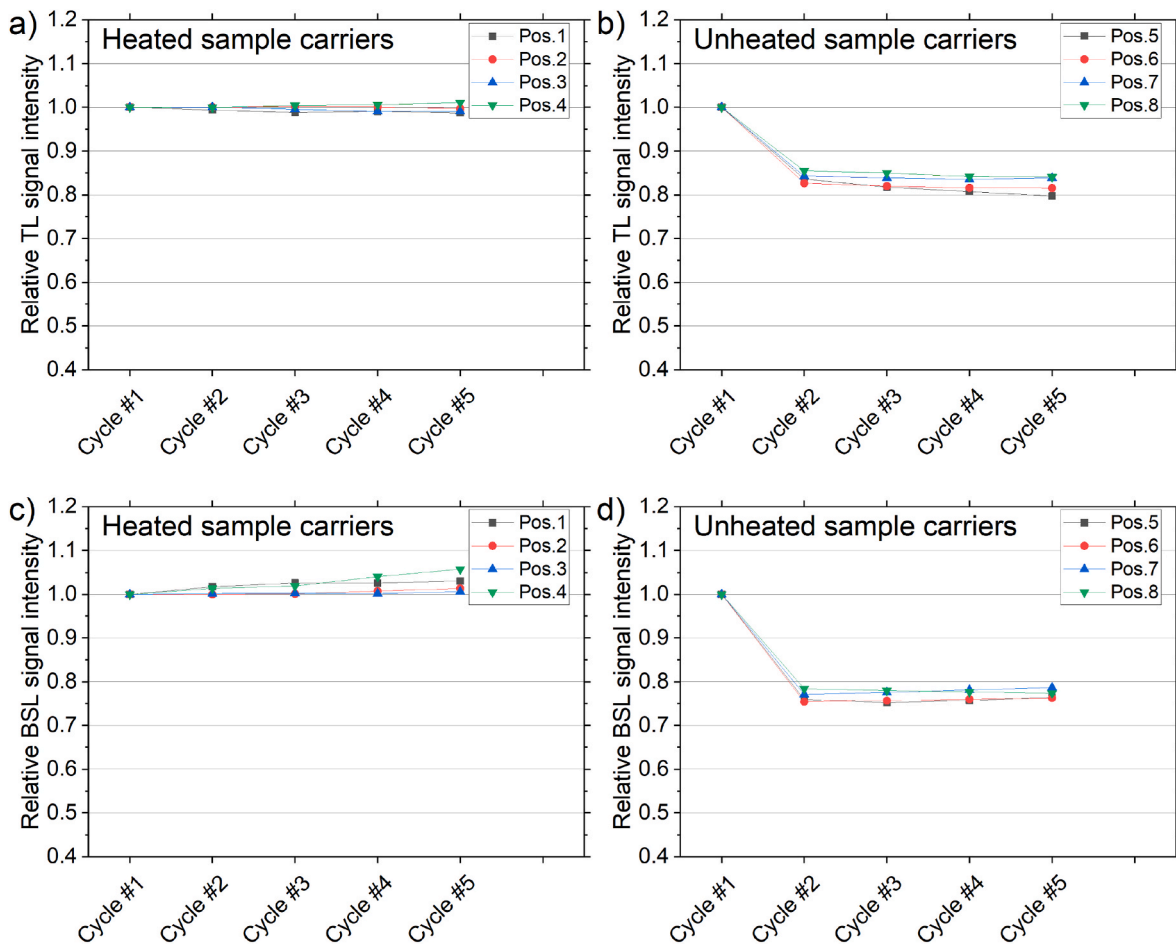


Fig. A1. Ratios of the integrated TL signals (100–250 °C) for the 10 glass aliquots placed on the (a) previously heated sample carrier on pos. 1–4 and (b) unheated sample carriers on pos. 5–8. Ratios of the integrated BSL signals (integral 0–1 s) obtained from the pre-bleaching step of the protocol for the glass aliquots placed on the (c) previously heated (on pos. 1–4) and (d) unheated (on pos. 5–8) sample carriers. Data are normalized to the first cycle.

Same kinds of experiments as presented in section 3.4 were conducted at IRSN on the Lexsyg Smart reader and KAERI on the Risø reader. Data are presented in Fig. A2.

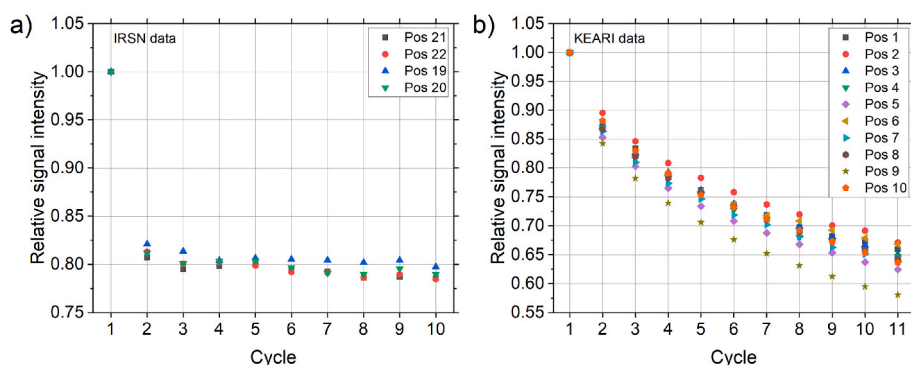


Fig. A2. Series of breakthrough signal measurements measured at IRSN (a) and at KAERI (b). Between each measurement the sample carrier was heated to 450 °C and held for 60 s in order to measure the impact of the heat exposure. VSL was measured at IRSN and BSL was measured at KAERI. Data are normalized to the first cycle.

References

- Ademola, J.A., Woda, C., 2017. Thermoluminescence of electronic components from mobile phones for determination of accident doses. *Radiat. Meas.* 104, 13–21.
- Bassinat, C., Woda, C., Bortolin, E., Della Monaca, S., Fattibene, P., Quattrini, M.C., Bulanek, B., Ekendahl, D., Burbidge, C.I., Cauwels, V., Kouroukla, E., Geber-Bergstrand, T., Mrozik, A., Marczewska, B., Bilski, P., Sholom, S., McKeever, S.W.S., Smith, R.W., Veronese, I., Galli, A., Panzeri, L., Martini, M., 2014. Retrospective radiation dosimetry using OSL of electronic components: results of an inter-laboratory comparison. *Radiat. Meas.* 71, 475–479.
- Discher, M., Woda, C., 2013. Thermoluminescence of glass display from mobile phones for retrospective and accident dosimetry. *Radiat. Meas.* 53–54, 12–21.
- ICRU, 2019. Methods for Initial-phase Assessment of Individual Doses Following Acute Exposures to Ionizing Radiation. ICRU Report 94. International Commission on Radiation Units and Measurements, Bethesda, MD.
- Inrig, E.L., Godfrey-Smith, D.I., Khanna, S., 2008. Optically stimulated luminescence of electronic components for forensic, retrospective, and accident dosimetry. *Radiat. Meas.* 43 (2–6), 726–730.
- Kazakis, N.A., Kitis, G., Tsirliganis, N.C., 2015. A cleaning method to minimize contaminant luminescence signal of empty sample carriers using off-the-shelf chemical agents. *Appl. Radiat. Isot.* 95, 226–232.
- Lapp, T., Kook, M., Murray, A.S., Thomsen, K.J., Buylaert, J.-P., Jain, M., 2015. A new luminescence detection and stimulation head for the Risø TL/OSL reader. *Radiat. Meas.* 81, 178–184.
- Mauz, B., Martin, L., Discher, M., Tribolo, C., Kreutzer, S., Bahl, C., Lang, A., Mercier, N., 2021. Technical note: on the reliability of laboratory beta-source calibration for luminescence dating. *Geochronology* 3, 371–381.
- Richter, D., Richter, A., Dornich, K., 2013. Lexsyg – a new system for luminescence research. *Geochronometria* 40 (4), 220–228, 2013.
- Richter, D., Richter, A., Dornich, K., 2015. Lexsyg smart – a luminescence detection system for dosimetry, material research and dating application. *Geochronometria* 42, 202–209.
- Schmidt, C., Kreutzer, S., Fattahi, M., Bailey, R., Zander, A., Zöller, L., 2011. On the luminescence signals of empty sample carriers. *Ancient TL* 29 (2), 65–74.
- Simkins, L.M., DeWitt, R., Simms, A., 2013. Methods to reduce sample carrier contamination for luminescence measurements. *Ancient TL* 31 (1), 19–28.
- Vandenbergh, D.A.G., Jain, M., Murray, A.S., 2008. A note on spurious luminescence from silicone oil. *Ancient TL* 26 (2), 29–32.