

Supplement 1

Fading tests for MAA-dating

In the multiple aliquot additive (MAA) approach aliquots with a sample's natural dose (N) are administered an (additional) laboratory dose ($N+\beta$). Usually, each dose point, including the natural dose, is represented by several aliquots and the mean signal of a group of aliquots is used for constructing the dose-response curve. For MAA measurements of feldspar or feldspar-bearing polymineral fine grains it is common practice to store samples for several weeks in the dark in between laboratory irradiation and IRSL readout in order to eliminate electrons from traps with a short lifetime (e.g., Lang & Wagner 1996). To determine possible fading, a second set of aliquots of one dose point (e.g., the highest additive dose point, $N+\beta_{\max}$), is irradiated and preheated together with the aliquots used for dating. Further, an extra set of aliquots with the natural luminescence signal (N) is preheated together with the aliquots used for dating. The aliquots used for the fading test (henceforth: fading aliquots) are stored for a longer period than the aliquots used for dating (dating aliquots) and read out at a later point in time (e.g., ca. 3 months after laboratory irradiation, whereas dating aliquots are measured ca. 4 weeks after laboratory irradiation). In order to monitor and correct for any possible changes in the measurement configuration (e.g., aging of the photomultiplier tube (PMT) used for detecting the IRSL signal), the fading measurement is normalized to the dating measurement by comparing the signals of the two sets of aliquots with the natural signal: $[N+\beta_{\text{fad}}]_{\text{cor}} = [N+\beta_{\text{fad}}] * N/N_{\text{fad}}$. If $[N+\beta_{\text{fad}}]_{\text{cor}}$ is within error margins identical with $N+\beta$, the palaeodose is used for calculating an IRSL age, which is regarded to represent the sample's true age. Otherwise the IRSL age is regarded as the sample's minimum age, or the samples are subjected to a fading correction to produce fading-corrected ages (e.g. Mejdahl 1988, Balescu et al. 2019).

Heat control

In contrast to earlier studies when in the Heidelberg Luminescence Laboratory reader Athenaeum was relatively 'cool' as compared to the relatively 'hotter' reader No. 245 (cf. Kadereit & Kreutzer 2013), actual comparative measurements between these two readers as well as reader No. 83 (TL/OSL DA15, updated to DA20) show only minor variations in the position of the instable quartz thermoluminescence (UV) peak induced by laboratory irradiation which is supposed to occur at 110 °C. The observed peak positions range between ca. 90 °C for reader Athenaeum, 92 °C for No. 245 and 95 °C for No. 83 (heating rate 10 °C/s). This shows that the temperature control of the recent MiniSys software was significantly improved.

Importance of the temperature control during luminescence readout – feldspar *versus* quartz

The importance of the temperature control during the IRSL-readout of a fading test is demonstrated by a test illustrated in Fig. i. In this test we compared the behaviour of the IRSL-signal of the polymineral fine-grain separate of sample HDS-1712 from Lake Chapalla with the behaviour of the BLSL-signal of a quartz coarse grain separate of sample HDS-1339 from Cameroon (Sprafke et al. 2019). The IRSL-measurement occurred on reader Athenaeum, the BLSL measurement on reader DA15.

Sample HDS-1712 received repeatedly an LAB of 400 s and was afterwards read out with a usual SAR-cycle (NRM 400 s; preheat 60 s at 250 °C): While the IRSL-readout temperature of the SAR-normalisation dose sub-cycle was kept constant at 60 °C, the IRSL-readout temperature of the LAB sub-cycle was increased from 41 °C to 80 °C, in steps of 1 K. The results are presented in Fig. i for the integral 1 – 20 s, both as gross signals and as net signals after late-light subtraction. Like in the figures of the

main text, the results are presented as double normalized values (usual SAR-normalisation in a first step (L_x/T_x); normalisation to the first IRSL-readout at 41 °C). Over the span of $\Delta 40$ K the values increase by approximately 40 % (Fig. ia), suggesting that the IRSL signal increases in the range of 41 – 80 °C by approximately 1 % per additional K readout-temperature. Basically this test conforms earlier observations of IRSL-counts increasing with increasing readout-temperature (e.g. Habermann 2000).

An almost identical sequence was used to measure the quartz sample on reader DA15, in which only the IR-stimulation unit was exchanged for the blue LEDs and the detection occurred in the UV band (around 340 nm) rather than in the blue-violet band (around 410 nm). In contrast to the IRSL feldspar signal, the BLSL quartz signal does not increase with increasing readout temperature but drops about 10 % over the here investigated temperature range 41 – 80 °C (Fig. ib). The observed course of the data values could possibly be caused by thermal quenching, an effect reducing the efficiency of luminescent charge recombination with increasing temperature (e.g. Gurney and Mott 1938; Wintle 1975; Friedrich et al. 2018). Assuming that our hypothesis is correct that a decreasing readout temperature (in a luminescence reader with insufficient temperature control) during a fading test with increasing delay of the IRSL readout and with the delay pause being placed in between the preheat and the IRSL-readout (Auclair et al. 2003) may cause or at least assist the phenomenon of a fading-like downward trend of the IRSL signal, the same temperature effect would in quartz lead to a stabilized BLSL signal or a slightly increasing trend. This might possibly explain, why fading is generally observed for feldspar but not for quartz.

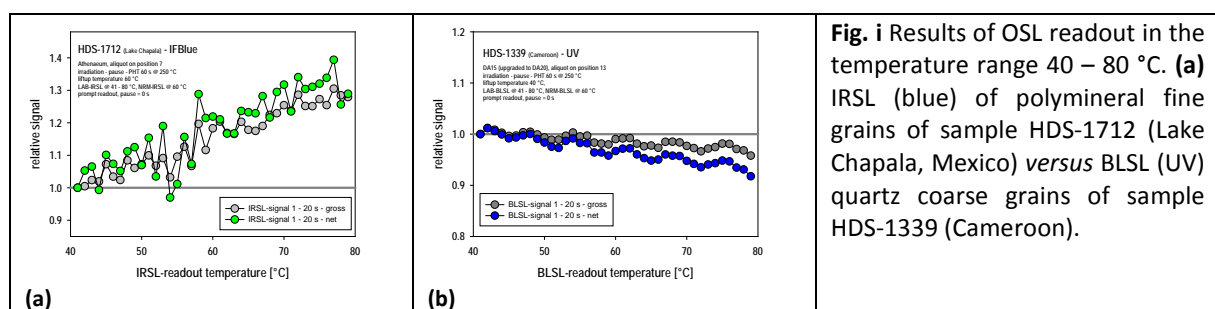


Fig. i Results of OSL readout in the temperature range 40 – 80 °C. **(a)** IRSL (blue) of polymineral fine grains of sample HDS-1712 (Lake Chapala, Mexico) versus BLSL (UV) quartz coarse grains of sample HDS-1339 (Cameroon).

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