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Removal of the feldspar-derived luminescence component from polymineral fine silt samples for optical dating applications: evaluation of chemical treatment protocols and quality control procedures

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Abstract

In optical dating of fine silt samples, the use of the quartz fraction only is often preferred. This approach requires the elimination of contaminating luminescence signals from feldspars. We compare here the effectiveness of different chemical treatments using diluted and concentrated hydrofluoric (HF) acid, and describe different tests to monitor the extent of feldspar contamination. The results suggest that the use of concentrated acid for a short time is the most suitable option. Satisfactory results were achieved with diluted HF for several tens of minutes only if the impurity component was small. Four “feldspar contamination tests” were examined in this study. Three of these were based on infrared (IR)-stimulation response and one was based on thermoluminescence (TL) signals. All tests show similar trends of decreasing feldspar contamination with increasing etching time or increasing acid strength, but seem to provide different detection limits. None of the tests proved to be an unambiguous stand-alone test. Consequently, a combination of IR- and TL-based tests is recommended.

Introduction

The use of quartz in optical dating circumvents some disadvantages associated with feldspars. These include (1) a relatively low precision in the equivalent dose (D_e) determined using an additive-dose dating protocol, (2) the unresolved issue of sensitivity change correction using a single-aliquot regenerative-dose protocol, and (3) the need to investigate anomalous fading. To access the advantages of dating quartz from polymineral fine silt samples, the feldspar component has to be removed. Complete feldspar removal is crucial, since this impurity may contribute to the optically stimulated luminescence (OSL) signal and, thus, may lead to D_e -underestimation. Duller and Bøtter-Jensen (1993) showed that the infrared stimulated luminescence (IRSL, 880 nm), OSL (514.5 nm) and

thermoluminescence (TL) from coarse grain potassium-rich feldspars relate to different trap depth distributions (detection window was 340–460 nm). OSL and IRSL seem to share the same source traps, but some of these traps can generate only OSL (Duller and Bøtter-Jensen, 1993). Duller (2003) observed a large, slowly decaying OSL signal from single grain feldspars after exposure to IR.

Theoretically, some feldspars may not contribute to the OSL signal. But as long as the mineral composition of the impurity is not known, its contribution to the D_e -estimate cannot be assumed to be negligible and, thus, complete feldspar removal is necessary.

To isolate the quartz component from a polymineral fine silt sample, two additional steps need to be introduced in the dating procedure: first, the removal of the feldspar component from the sample without affecting the quartz component, and second, the development of a measurement protocol to monitor the completeness of the removal. Two different approaches are in use: (a) chemical removal using hydrofluoric (HF) acid or fluorosilicic (H_2FSi_6) acid, and (b) optical removal using IR-bleaching before measuring the OSL.

This present paper focuses on the chemical treatment and discusses possible quality control by luminescence means.

Chemical treatment using HF or H_2FSi_6 to remove feldspars from polymineral fine silt samples has been applied previously (e.g. Berger et al., 1980; Rees-Jones, 1995; Prasat, 2000; Roberts and Wintle, 2001; Stokes et al., 2003a, b). These authors used diluted HF (5% or 10%) for 80–120 min, or H_2FSi_6 (35%) for 30 h and longer.

A rigorous test is essential to evaluate the effectiveness of the different chemical treatments. Low-cost and relatively simple tests based on mineralogical-chemical analysis (e.g. x-ray diffractometry) are usually not sensitive enough for optical dating purposes and, moreover, lead to

substantial loss of sample material. Thus, the only alternative is a luminescence-based test. Several tests are in use based on luminescence properties characteristic of quartz and of feldspars: Henshilwood et al. (2002) used the ratio of the sensitivity-corrected [post-IR]-OSL ($L_{x\text{post-IR}}/T_{x\text{OSL}}$) to the sensitivity-corrected OSL ($L_{x\text{OSL}}/T_{x\text{OSL}}$). Duller (2003) further investigated this test ("OSL IR depletion ratio") which is based on the assumption that the UV recombination centres in feldspars responsible for IRSL and OSL share the same source traps, as indicated also by Jain and Singhvi (2001). Li et al. (2002) proposed the ratio of the IRSL signal to the 110°C TL peak. The TL signal is obtained during preheating of the test dose given as part of the irradiation cycle in a single-aliquot regenerative-dose (SAR) protocol. Olley et al. (2004) compared decay curves of OSL and [post-IR] OSL and rejected quartz grains with OSL decay curves that did not reach background within 1 s of laser stimulation. Besides these relatively quantitative approaches, a qualitative test observing the shape and intensity of an IRSL decay curve is often used to assess feldspar presence in a sample. Hence, different chemical treatments are in use and different views exist on which OSL properties are the most appropriate to reveal feldspar contamination.

Ideally, one would want to check for impurities by analysing exclusively the OSL decay curves used for D_e -estimation (Olley et al., 2004; Duller, 2003). But such analysis requires detailed fitting procedures (e.g. Bailey et al., 1997; Bulur, 2000) and often needs additional support from measurements such as linearly ramped OSL or IR at elevated temperature (Duller, 2003). An alternative to such investigations could be to observe luminescence properties unique to each of the minerals and compare the corresponding data collected during routine dating protocols.

Here we report our attempts at isolating pure quartz extracts from polymineral fine grain samples based on different etching procedures, and assess the existing tests to verify successful isolation. We also propose a further test based on the ratio of the TL@220°C to the TL@110°C.

Physical background of feldspar contamination tests

In most studies, a signal above background in response to IR stimulation is attributed to feldspars. At room temperature (RT), the fast component of quartz OSL is not stimulated by IR (Aitken, 1998), whereas a large variety of feldspars respond to IR excitation by emitting UV wavelengths. Indeed, some K-rich feldspars (e.g. orthoclase and microcline), most Na-rich feldspars (e.g. albite) and some Ca-rich feldspars (e.g. oligoclase) emit IRSL at 280, 330 and

380 nm (Krbetschek and Rieser, 1995; Krbetschek et al., 1996). However, other K-rich feldspars (e.g. sanidine and some microclines) and plagioclases (e.g. anorthoclase and labradorite) do not emit UV under IR excitation (Krbetschek and Rieser, 1995; Krbetschek et al., 1996). Consequently, an IR-sensitive electron trap giving rise to an IRSL signal in the UV wavelength range is a characteristic property of many, but not all, feldspars, as long as excitation is performed at RT. (At elevated temperature, the signal may also come from the quartz fast component; Singarayer and Bailey, 2004.) Thus, RT-IRSL does not necessarily monitor the presence of all feldspar contaminants in a quartz sample.

Most feldspars show TL at around 110°C (in the UV detection window); this TL signal is not in the form of a distinct peak, but instead represents the rising limb of higher temperature TL peaks. In contrast, quartz shows a distinct 110°C TL peak. Thus, a distinct 110°C TL peak detected in the UV wavelength range is seen as characteristic of quartz.

At 280 and 330 nm, some K-rich feldspars (e.g. microcline) and most Na-rich and Ca-rich feldspars (e.g. albite, oligoclase and andesine) have TL peaks at ~180°C, ~220°C and ~250°C (5°C s⁻¹ heating rate) resulting from several overlapping TL emissions (Krbetschek et al., 1997). However, not all feldspars emit TL above background between 180°C and 250°C (Krbetschek et al., 1997).

Occasionally, quartz can show TL peaks at 180°C and 220°C (3.1°C s⁻¹ heating rate; Franklin et al., 1995). According to our observation, these peaks are not very common. Almost all of our quartz samples have a TL background signal at ~220°C (based on a standard SAR protocol with delay times between beta-irradiation and preheating of 30–80 min and a heating rate of 5°C s⁻¹).

To summarise, most, but not all, feldspars have an IR-sensitive electron trap that gives rise to an IRSL signal in the UV, which quartz does not show at RT. Most, but not all, feldspars show one to two TL peaks between 180°C and 250°C, while most, but not all, quartz samples do not show TL peaks in this temperature region. In addition, the 110°C TL peak is a characteristic property of quartz.

Materials and experiments

The samples used here originate from the German North Sea coast (LV 01, LV 05 and LV 08), the English east coast (LV 17) and the South German loess area (loess sample). Samples were treated in the laboratory following conventional procedures for fine silt sample preparation (Mauz et al., 2002). All measurements were performed using an automated Risø TL/OSL reader equipped with an EMI 9635QA photomultiplier. Stimulation of samples was

Sample	Etch procedure (% HF, min)	Post-IR OSL/OSL	IR/TL	TL/TL	IRSL
LV 01	0	4.833±0.002	1.73±0.09	2.2929±0.0001	340207±1669
	2%, 80 min	0.314±0.002	0.56±0.03	2.2350±0.0001	259466±1385
	4%, 80 min	0.8252±0.0004	0.152±0.008	0.67096±0.00004	69230±690
	10%, 80 min	0.96642±0.00005	0.138±0.007	1.1441±0.0001	34947±477
	20%, 5 min	0.95765±0.00005	0.094±0.005	0.45133±0.00003	49426±128
	20%, 15 min	1.0097±0.0009	0.0080±0.0005	0.09906±0.00006	860±34
	20%, 40 min	0.970±0.001	0.0086±0.0005	0.0723±0.0009	1056±34
	48%, 5 min	0.982±0.002	0.023±0.001	0.1372±0.0001	1314±47
LV 05	0	2.781±0.001	0.223±0.015	1.27523±0.00003	404709±1699
	2%, 90 min	0.429±0.002	0.163±0.011	0.7490±0.0001	35576±436
	4%, 120 min	0.635±0.006	0.166±0.012	0.69620±0.00001	31071±435
	5%, 80 min	0.8338±0.0001	0.083±0.004	0.2805±0.0003	471675±2142
	10%, 80 min	0.9682±0.0001	0.0141±0.0008	0.16945±0.00001	8267±174
	20%, 5 min	0.891±0.002	0.072±0.004	0.36156±0.00007	8966±219
	20%, 15 min	0.99300±0.00004	0.0350±0.002	0.08130±0.00002	6357±169
	48%, 5 min	0.9941±0.0001	0.0011±0.00009	0.03990±0.00005	146±6
LV 08	0	4.690±0.001	0.954±0.051	2.7524±0.0002	406042±1713
	5%, 80 min	0.8869±0.0001	0.75±0.04	2.3254±0.0001	217572±1256
	10%, 80 min	0.9919±0.0002	0.030±0.002	0.18130±0.00002	5156±127
	20%, 5 min	0.990±0.002	0.028±0.002	0.1903±0.0001	1760±58
	20%, 15 min	1.0075±0.0001	0.068±0.004	0.36868±0.00004	17360±313
	48%, 5 min	0.9960±0.0002	0.0039±0.0003	0.1142±0.0001	188±8
LV 17	2%, 80 min	0.466±0.008	0.27±0.01	1.6083±0.0003	32975±494
	10%, 80 min	0.942±0.004	0.092±0.006	0.7101±0.0004	2936±90
Loess	0	7.276±0.005	0.523±0.029	2.1077±0.0005	40707±437
	5%, 80 min	1.1610±0.0004	0.290±0.016	1.7344±0.0003	39084±445
	10%, 80 min	0.8994±0.0004	0.025±0.001	0.1240±0.0006	1922±44
	20%, 15 min	0.9921±0.0002	0.114±0.007	0.4826±0.0002	4595±128
	20%, 40 min	0.996±0.002	0.0105±0.0007	0.1653±0.0009	502±21
	48 %, 5 min	1.045±0.003	0.017±0.001	0.0563±0.0001	1784±51

Table 1: Data used to compare the feldspar contamination tests and to test etching procedures. Data were obtained using the measurement protocol depicted in Fig. 1. Post-IR OSL/OSL: $\frac{L_{\text{post-IR}}}{T_{\text{OSL}}}$ where $\frac{L_{\text{OSL}}}{T_{\text{OSL}}}$ is the OSL normalised by the test dose OSL and $\frac{L_{\text{post-IR}}}{T_{\text{OSL}}}$ is the [post-IR] OSL normalised by the test dose OSL after an equal laboratory regenerative dose had been administered; IR/TL: $\frac{\text{IRSL}}{TL@110^\circ\text{C}}$ where IRSL is the signal integral between 0 s and 10 s illumination time, after subtraction of the background count rate calculated from the 60–100 s interval, and TL@110 °C is the integral 90–140 °C; TL/TL: $\frac{TL@220^\circ\text{C}}{TL@110^\circ\text{C}}$ where TL@220 °C is the integral 200–250 °C and TL@110 °C is the integral 90–140 °C; IRSL: signal integral between 0 s and 10 s illumination time, after subtraction of the background count rate calculated from the 60–100 s interval. Note that data are derived from one aliquot only and that statistically outlying data may occur.

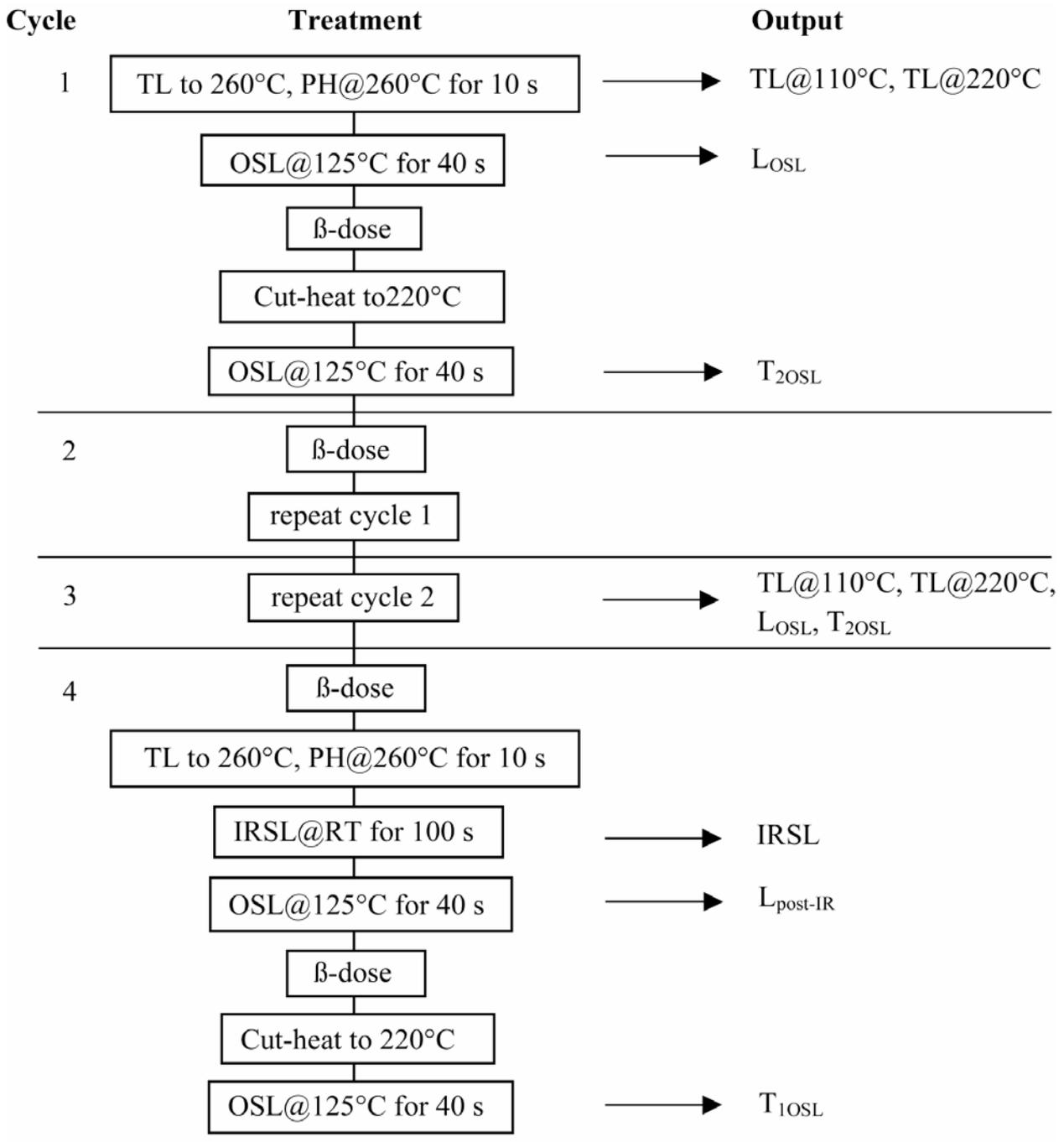


Figure 1: Measurement sequence designed to detect feldspar contaminants. For TL@110°C, TL@220°C, L_{OSL} and T_{2OSL} the output of cycle 3 was used, and for IRSL, $L_{post-IR}$ and T_{1OSL} the output of cycle 4 was used. Equal laboratory regenerative doses of ~10 Gy were administered in each cycle.

performed using either blue LEDs emitting at $470\Delta 30$ nm (delivering ~ 30 mW cm⁻²) or IR LEDs emitting at $875\Delta 40$ nm (delivering ~ 110 mW cm⁻²). The OSL, IRSL and TL emissions were detected through an optical filter (Hoya U340, 7.5 mm) transmitting 260 to 390 nm wavelengths.

The samples were etched using HF diluted to 2%, 4%, 5%, 10% and 20%, and concentrated HF (48%), for various durations (Table 1). During etching, samples were continuously shaken by applying an orbital movement to the beakers. Subsequently, samples were first washed in 10% HCl for approximately 1 hour and then washed in distilled water and 4 M NaOH. The presence of remnant feldspar was detected using the measurement sequence shown in Fig. 1: (1) OSL for 40 s at 125°C, (2) IRSL for 100 s at RT, (3) [post-IR] OSL for 40 s at 125°C, and (4) TL from RT to 260°C using a heating rate of 5°C s⁻¹ and constant nitrogen flow into the measurement chamber. The TL signals recorded during preheating were integrated between 90°C and 140°C (denoted here as TL@110°C) and between 200°C and 250°C (denoted here as TL@220°C) and an average low temperature background (RT to 60°C) was subtracted (assuming negligible temperature-related dark counts in this temperature region). The IRSL signal was integrated between 0 s and 10 s stimulation time, and the average signal from the integral 60–100 s was subtracted as background; the net signal is denoted here as IRSL). All measurements were performed on one aliquot of each sample after having administered a laboratory regenerative dose of ~ 10 Gy.

Three “feldspar contamination tests” were chosen to monitor possible feldspar remains after chemical treatment. The first test followed Henshilwood et al. (2002) and Duller (2003) using the OSL IR depletion ratio $\frac{L_{post-IR}}{T_{1OSL}} \frac{L_{OSL}}{T_{2OSL}}$ (denoted here as post-IR OSL/OSL). This test assumes that an IRSL signal at RT is emitted by feldspars. Thus, the ratio is at unity if no feldspar component is present and it is < 1 in the case of contamination. The second test followed Li et al. (2002) by computing the ratio $\frac{IRSL}{TL@110^\circ C}$ (denoted

here as IR/TL). This test compares two properties, which are supposed to be characteristic of feldspars (IRSL) and quartz (TL@110°C) respectively. The ratio approaches zero if no feldspar component is present. It is expected that these ratios do not reach absolute values of 1 (post-IR OSL/OSL) and zero (IR/TL), as sedimentary quartz can show a weak IR-stimulated emission associated with the Al impurity in the quartz crystal lattice (Godfrey-Smith and Cada, 1996; Jaek et al., 1999).

The third test comprises the ratio $\frac{TL@220^\circ C}{TL@110^\circ C}$

(denoted here as TL/TL). The rationale for this ratio is based on our observation that a feldspar-free quartz sample does not show a TL signal above background in the range 180–250°C, as shown in Fig. 2. Similar to the IR/TL ratio, the TL/TL ratio also approaches, but might not reach, the absolute value of zero if no feldspar component is present. The three ratios were compared with the IRSL signals. Uncertainties were quantified based on counting statistics of luminescence signals and background signals, and applying error propagation.

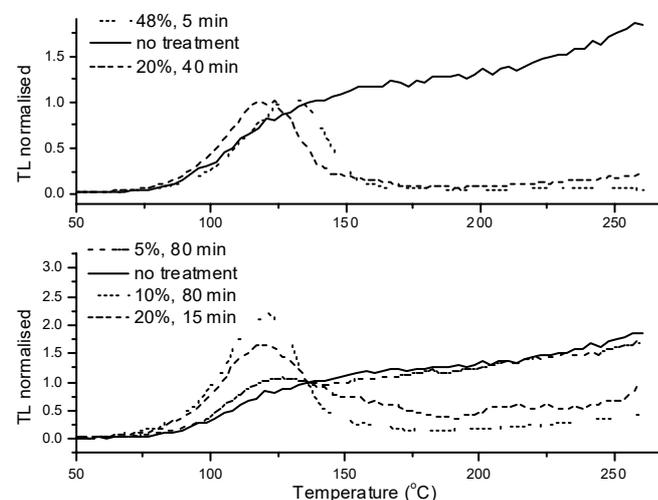


Figure 2: TL curves to 260°C (heating rate 5°C s⁻¹) for the loess sample. TL curves are normalised by the 110°C peak (but not shifted). Each curve represents a different HF treatment. With increasing acid strength and, consequently, with increasing removal of the feldspar component, the TL signal at 150–260°C decreases and is at background when (we infer) no feldspar component is present.

Results and discussion

All data used in this study to compare the feldspar contamination tests and to test the efficacy of the etching procedures are listed in Table 1 and depicted in Figs. 3–5.

For each etching procedure, the results were assessed to determine if all three ratio-based test results were in agreement. When there was disagreement, the results were then examined in terms of the different luminescence properties on which the tests were based.

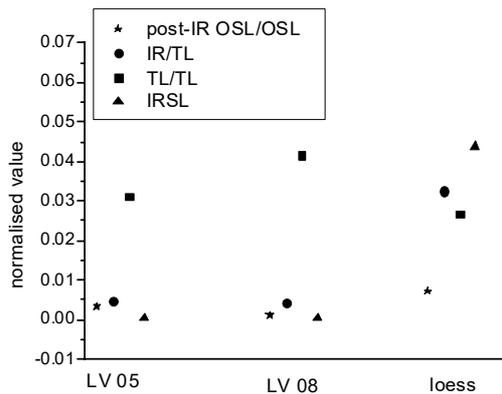


Figure 3: Results from feldspar contamination tests after sample treatment in 48% HF for 5 minutes. The post-IR OSL/OSL values are calculated from the differences between the measured values after etching and the 'feldspar-free' value of 1, divided (normalised) by the differences between the 'no treatment' values and the feldspar-free value of 1. The IR/TL, TL/TL and IRSL values are calculated from the measured values after etching, divided by their respective 'no treatment' values. For data see Table 1.

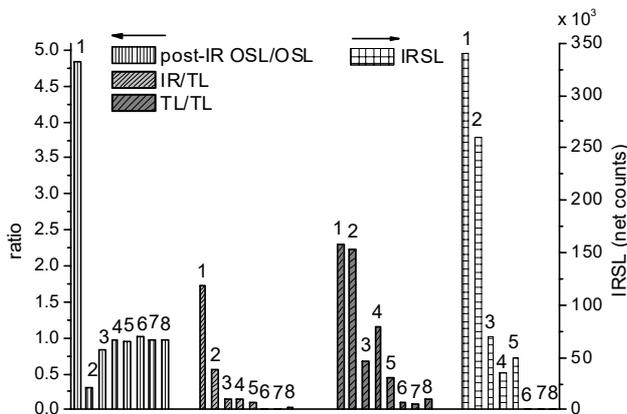


Figure 4: The effect of various etching procedures on the decrease in feldspar content for sample LV 01. 1: no treatment, 2: 2%, 80 min, 3: 4%, 80 min, 4: 10%, 80 min, 5: 20%, 5 min, 6: 20%, 15 min, 7: 20%, 40 min, 8: 48%, 5 min (for data see Table 1).

There was satisfactory agreement between tests regarding complete removal of feldspar when the samples were treated in concentrated HF for 5 minutes (Fig. 3). Treatment in diluted HF (2%, 4%, 5%, 10%) for 80, 90 or 120 minutes resulted in the incomplete removal (e.g. sample LV 01, Fig. 4), as confirmed by all three tests. An exception here was LV 17: treatment in a weak (10%) acid for several

tens of minutes was sufficient to remove most of the impurity component. The tests also agreed that there was an insignificant difference between treatment in 10% HF for 80 min and 20% HF for 5 min (sample LV 08, Table 1), both being reasonably effective at removing the feldspar contaminants.

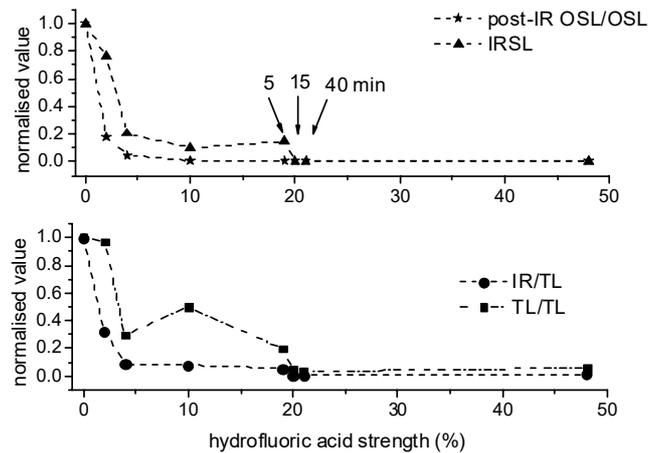


Figure 5: Comparison of the feldspar contamination tests for sample LV 01. The plots display the normalised values (calculated as described in the Fig. 4 caption) versus HF strength. The time difference of treatment with 20% HF (5, 15 and 40 min) is depicted by three data points centred on 20% HF strength. IRSL and TL/TL show a relatively high sensitivity to feldspar contamination whereas IR/TL and [post-IR] OSL/OSL seem to be less sensitive.

Inconsistencies between the test results within individual samples appear after prolonged treatment in weak acid, or after short treatment in strong acid, when the feldspar impurity has decreased from an initial high level. This is evident for several samples: (1) LV 05 (10%, 80 min) where the IR/TL ratio indicates a pure quartz sample but the other test results do not; (2) LV 08 (20%, 5 min) where the post-IR OSL/OSL and IR/TL ratios indicate pure quartz, but the TL/TL ratio and IRSL signal do not; (3) loess (5%, 80 min) where the post-IR OSL/OSL ratio indicates a pure quartz sample but the other test results do not; (4) LV 01 (10%, 80 min; 20%, 5 min), where the post-IR OSL/OSL ratios and IRSL signals show little difference between the two treatments but the IR/TL and TL/TL ratios indicate a reduction of feldspar contamination after 20% HF for 5 min; and (5) LV 05 (10%, 80 min; 20%, 5 min) where the IRSL signals indicate little difference between the two treatments but all three ratios show that 10% HF for 80 min was more effective.

The IRSL test responds to the IR-stimulated component in the sample and, thus, detects some K-rich feldspars, most Na-rich feldspars and some Ca-

rich feldspars. The post-IR OSL/OSL test responds to the presence of these contaminants relative to the intensity of the quartz OSL fast component. And the IR/TL test responds to these contaminants relative to the size of the quartz 110°C TL peak. Consequently, these three tests are based on the IR-sensitive electron trap giving rise to an IRSL signal in the UV wavelength range and are, therefore, expected to give similar results. Table 1 shows that this is the case for most data. Deviations can be explained in terms of statistical variations, as one aliquot only was measured for each test. Contradictory results were commonly obtained between post-IR OSL/OSL, IR/TL and IRSL on the one hand and TL/TL on the other, indicating that the TL/TL test reflects additional complexities of the feldspar UV emission. Probably, the TL/TL test responds also to some K-rich feldspars (e.g. sanidine and some microclines) and some plagioclases (e.g. anorthoclase and labradorite), which are not detected by the IR-based tests. Quartz can show TL@220°C, however, so a TL/TL ratio significantly above zero cannot be unambiguously attributed to a feldspar impurity only. Thus, the relatively high sensitivity of the TL/TL test, as shown in Fig. 5, is not an unambiguous indicator of quartz purity.

For the untreated samples, the post-IR OSL/OSL test shows ratios above unity, which dropped below unity when the feldspar component decreased. This result could be related to IR-induced charge transfer during SAR measurement, but further investigations are needed to fully understand this phenomenon.

Relatively small IR/TL ratios from untreated samples (e.g. loess and LV 05) reveal that wrong conclusions about quartz purity can be drawn, as these tests are based on TL signal intensity but not on TL peak shape. As shown in Fig. 2, chemical treatment in weak acid does not significantly enhance the definition of the quartz 110°C TL peak. This prominent and discrete peak starts to appear only after treatment in 10% HF and is fully developed after treatment in 20% HF for several tens of minutes. Both the IR/TL and the TL/TL tests may, therefore, need an additional qualitative check regarding TL signal shape.

Conclusions

The experiments with various etching procedures suggest that the use of a strong acid for a relatively short time is preferable to a weak acid for a long time. The treatment using relatively weak acid for several tens of minutes was successful if the impurity content was small (e.g. LV 17).

Three "feldspar contamination tests" were investigated in this study using data accrued during

standard dating protocols. All test results show the same trend with increasing etching time or increasing acid strength, but the different tests had different detection limits. None of the tests proved to be a stand-alone test. The post-IR OSL/OSL, IR/TL and IRSL tests are not truly independent of each other, as each is based on the IR-sensitive electron trap giving rise to an IRSL signal in the UV wavelength range; thus, they detect virtually the same contaminants. The TL/TL test seems to respond to some K-rich feldspars and some plagioclases that are not detected by IR-based tests. We conclude, therefore, that IR-based and TL-based tests complement each other and should be applied together. Furthermore, for both the IR/TL and the TL/TL tests, which utilise the quartz 110°C TL peak, we recommend that the shape of the peak be observed, in addition to determining the numerical ratios.

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Reviewer

R.G. Roberts

A user defined command for pulsed-irradiation on Risø TL-OSL readers

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Abstract

It has recently been proposed that large laboratory radiation doses to quartz should be administered in small pulses separated by a cut-heat, rather than the conventional method of administering doses in a single pulse. This paper explains the structure of the software system used to control the Risø TL/OSL reader and presents code for a user defined command which allows pulsed-irradiation to be performed conveniently. This approach could also be used to undertake any complex sequence of operations in a flexible and straightforward manner.

Keywords

Quartz, optical dating, dose-rate, pulsed-irradiation, age, accuracy.

Introduction to the use of pulsed-irradiation

The single-aliquot regenerative-dose (SAR, Murray and Wintle, 2000) method has been used to determine the equivalent dose (D_e) to quartz from a wide range of environments. The SAR method generally yields ages which are in good agreement with independent age control (e.g. Hilgers et al. 2001, Murray and Clemmensen, 2001, Murray and Olley, 2002, Murray and Funder, 2003, Stokes et al. 2003). However, a recent study using modelled data (Bailey, 2004) indicates that the SAR method may overestimate the equivalent dose (D_e), when the D_e is greater than ~40Gy. In addition, the SAR procedure appears to produce incorrect results for individual aliquots at high equivalent doses, where the natural luminescence intensity is sometimes greater than the saturation intensity observed due to laboratory irradiation (e.g. Armitage et al. 2000, Figure 5, Yoshida et al. 2000, Class 3 grains).

According to Bailey (2004) this effect is caused by the relatively high dose rates used during laboratory irradiation, leading to the trapping of a significant population of holes at a thermally unstable, non-radiative recombination centre (R_1 -centre).

Consequently, during room temperature laboratory irradiation, the R_1 -centre competes for charge in the conduction band, reducing the charge available for trapping at the OSL traps. The R_1 -centre does not compete for charge in nature due to its low thermal stability. Consequently, the laboratory regenerated OSL signal intensity is lower per unit dose than for natural rate irradiation and hence the D_e calculated is erroneously large. By administering laboratory doses either at raised temperature, or in short pulses separated by thermal treatments (pulsed-irradiation), the malign effects of the R_1 -centre can be reduced. Bailey et al. (in press) present empirical data which supports the prediction by Bailey (2004) that SAR and pulsed-irradiation produce different growth curves, with the latter yielding lower equivalent doses. However no known age samples were measured in this study, precluding firm conclusions regarding the accuracy of either method.

Although a raised temperature irradiation facility is available for the Risø TL-OSL reader (Bøtter-Jensen et al. 2003), raised temperature irradiation cannot be performed in many laboratories. In addition, temperature dependent changes in the trapping cross-section of the OSL traps appear to invalidate this approach (Wallinga et al. 2002). This paper presents code for a user defined command for pulsed-irradiation, which can be performed on all Risø readers which use a MiniSys. This pulsed-irradiation command performs irradiation in a series of pulses (e.g. 10 Gy). Following each pulse, the sample is heated to a definable temperature (during which the resulting thermoluminescence is measured) and immediately allowed to cool to room temperature. The heating step releases holes trapped at the thermally unstable R_1 -centre. Consequently, the R_1 -centre competes less strongly for charge in the conduction band, approximating the situation found in nature and preventing an overestimate of the equivalent dose. Administering a series of radiation

pulses, with intervening heating stages, is possible using conventional software, but is unwieldy since each irradiation would require many instructions. By utilising a new facility in the Risø reader, namely user defined commands, the process can be made much simpler.

How the Risø reader is controlled

Current versions of the Risø TL/OSL reader consist of three functional units. The first is the reader itself, consisting of the measurement chamber, the heater plate, irradiator, other facilities and associated control electronics. This hardware is controlled by the second unit, a dedicated PC based controller called a MiniSys (Markey et al. 1997). The MiniSys directly interfaces with the hardware and continuously monitors it to check for hardware faults. The software at the heart of the MiniSys is a command interpreter. The MiniSys language consists of two character commands, each of which instruct the MiniSys to perform a single operation, e.g. "LU" instructs the MiniSys to raise the lift. Parameters can be added to these commands where appropriate, e.g. "ST 160 5" instructs the MiniSys to heat the hotplate to 160°C at a rate of 5°C per second. A full list of these commands are supplied with each Risø reader and is available from the second author. The third functional unit is a host computer running a programme that can issue sequences of MiniSys codes, and that can collect any data that is generated. The Sequence Editor programme supplied with the reader serves this function. When a command is entered in the Sequence Editor, it is stored in a .SEQ file. When a sequence is executed, the Sequence Editor translates this file into commands which are implemented by the MiniSys. A standard (or "high level") command such as "Pre-Heat" requires several low-level commands to be issued to the MiniSys (e.g. lift up, heat to temperature, pause at temperature, lift down). The Sequence Editor translates high-level commands into low-level MiniSys commands using the TLMSLL.CMD file. For each high-level command (e.g Preheat or Irradiation) this file lists the low-level commands required to perform each high-level command. An example of this is given in figure 1 which shows the section of the TLMSLL.CMD file that the Sequence Editor uses to convert the high-level 'Pre-Heat' command into MiniSys commands.

The pulsed-irradiation user defined command

In late 2000, the capability to define non-standard high-level commands was added to the Risø Sequence Editor (Duller, 2000). This allows the operator to use the low-level MiniSys language without having to write control code to issue commands and collect data. Any of these MiniSys commands can be linked together, both to control the

TL/OSL reader and to collect data, and these user defined commands can then be intermixed with standard commands in a Sequence.

```
[PREHEAT]
; $1 Temp
; $2 Heat Rate
; $3 Time
;
10=PS $0
20=#RS
30=#TF
40=#WLT
50=LU
60=#RS
70=ST $1 $2
80=#RS
90=PA $3
100=#RS
110=LD
120=#RS
130=ST 0
```

Figure 1: *The section of the TLMSLL.CMD file that the Sequence Editor uses to convert the high-level command 'Preheat' into low-level MiniSys codes. To undertake a Preheat operation the Sequence Editor reads each of the numbered lines (10 to 130). A number of parameters, prefixed by the symbol '\$' can be passed by the Sequence Editor. These correspond to parameters entered by the user in the Sequence Editor. In the case of the Preheat command \$1 is the preheat temperature, \$2 is the heating rate at which to raise the temperature and \$3 is the period of time that that temperature is to be held. For all commands, the position on the carousel of the sample that is to be analysed is passed as the parameter \$0. Once any parameters have been replaced by the correct numerical values, the text to the right of the equals sign is sent to the MiniSys. Thus the first operation is to send a 'PS 12' command (assuming that the current sample that is to be analysed is sample number 12). This will move the carousel so that position 12 is over the hotplate. In line 20, #RS (read status) is a metacommand that will pause the Sequence Editor until the current operation (in this case moving the carousel) is complete. #TF will check for a thermal failure and #WLT will pause until the hotplate temperature is lower than the threshold specified in the Sequence Editor (default 60°C).*

For the current example, the code for the pulsed-irradiation command is given in Figure 2. These user defined commands should be written in a separate command file called USERMSLL.CMD, to prevent accidental alteration of the standard high-level commands in TLMSLL.CMD. To be able to use this pulsed-irradiation command, the code must be added

to the USERMSLL.CMD file using a text editor. The pulsed-irradiation command is then ready to be used from within the Sequence Editor.

```
[UserDef0]
; Pulsed-irradiation with cut heats in gaps
; $1 No of datapoints during cut-heat
; $3 Number of cycles required
; $4 Heating Rate (°C/s)
; $5 Maximum cut-heat temperature (°C)
; $6 Irradiation time (s)
;
10=#LOOP 1 $3
20=BP $0
30=#RS
40=BI $6
50=#RS
60=PS $0
70=#RS
80=#INITGRAPH $1
90=#TF
100=#WLT
110=LU
120=#RS
130=TL $5 $4 $1 0
140=#DATA
150=#SAVE
160=#ENDGRAPH
170=LD
180=#RS
190=#ENDLOOP
```

Figure 2: Text for the USERMSLL.CMD file to define the pulsed-irradiation command. The #LOOP (line 10) and #ENDLOOP (line 190) commands allow the sequence of commands in between (lines 20 to 180) to be repeated a number of times. The loop counter will count from 1 to \$3.

Using the pulsed-irradiation command

Like standard high-level commands, a user defined command is selected in the Sequence Editor (selecting “User Defined”). The dialogue box shown in Figure 3 will then appear.

Up to eight user defined commands can be stored in USERMSLL.CMD, and the appropriate command is selected from the User Command drop-down menu at the top of the dialogue box. The most important point to note is that the titles for each box are only meant as suggestions for its function. The actual meaning of each parameter is specified by the user defined command in USERMSLL.CMD, with the parameter number in brackets beside each box (\$1, \$2, \$3 etc) being the critical link. For example, in the pulsed-irradiation command presented in this paper, \$6 (Ph

time in Figure 3) is actually used to specify the beta irradiation time required in each pulse (see Table 1).

Figure 3: The user defined command as used for the pulsed irradiation example described in the text. This command will pass parameters \$1, \$3, \$4, \$5 and \$6 to the section of the USERMSLL.CMD file. By comparing with Figure 2 it can be seen that this set of parameters will give 8 doses, each with a duration of 120 seconds. After each pulsed irradiation the sample will be heated to 240 °C at 5 °C per second, recording the TL in 250 channels.

The pulsed-irradiation command only uses five of the available parameters. These are listed towards the top of the code in Figure 2, and constraints on their use are given in Table 1. Values for each of these parameters are entered in the same manner as for a standard high-level command. Sequence Editor will ignore all other parameters, irrespective of the values they contain. The sequence is executed in the normal manner.

Summary

The user defined command described here provides flexibility in the control of the Risø reader, beyond what is possible using the standard Sequence Editor, but without the complication of writing software to interact with the MiniSys directly. The example given here is of a command to undertake pulsed-irradiation. Such pulsing would be possible with the conventional commands within the Sequence Editor, but would require so many individual commands that only very simple irradiations could be fitted into a single sequence. More complex user defined commands can be defined involving any of the capabilities of the Risø system.

Parameter	Function	Constraints
\$1	Datapoints recorded during the cut-heat.	Must be greater than zero
\$3	The number of irradiation and heating cycles required.	Must be greater than 1, e.g. 50Gy using 10Gy pulses requires 5 cycles.
\$4	Heating rate during cut-heat (°C/s).	None, Bailey et al. (in press) used 5°C/s.
\$5	Maximum temperature reached during cut heat (°C).	None, Bailey et al. (in press) recommended ~240°C.
\$6	Beta irradiation time per pulse (s).	None, Bailey et al. (in press) recommended 10Gy.

Table 1 : Parameters and constraints for the pulsed-irradiation user defined command.

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Reviewer

Andrew Murray

Thesis Abstracts

Author: Kristina Jørkov Thomsen
Thesis Title: Optically stimulated luminescence techniques in retrospective dosimetry using single grains of quartz extracted from unheated materials
Grade: PhD
Date: February 2004
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This work investigates the possibility of applying optically stimulated luminescence (OSL) in retrospective dose determinations using unheated materials. It focuses on identifying materials suitable for use in assessment of doses absorbed as a consequence of radiation accidents (i.e. accident dosimetry). Special attention has been paid to quartz extracted from unheated building materials such as concrete and mortar. The single-aliquot regeneration-dose (SAR) protocol has been used to determine absorbed doses in small aliquots as well as single grains of quartz. It is shown that OSL measurements of single grains of quartz extracted from poorly-bleached building materials can provide useful information on radiation accident doses, even when the luminescence sensitivity is low. Sources of variance in well-bleached single grain dose distributions have been investigated in detail and it is concluded that the observed variability in the data is consistent with the sum (in quadrature) of a component, which depends on the number of photons detected from each grain, and a fixed component independent of light level. Dose depth profiles through laboratory irradiated concrete bricks have successfully been measured and minimum detection limits of less than 100 mGy are derived. Measurements of thermal transfer in single grains of poorly-bleached quartz show that thermal transfer is variable on a grain-to-grain basis and that it can be a source of variance in single-grain dose distributions. Furthermore, the potential of using common household and workplace chemicals, such as table salt, washing powder and water softener, in retrospective dosimetry has been investigated. It is concluded that such materials should be considered

as retrospective dosimeters in the event of a radiation accident.

This thesis is available online at <http://www.risoe.dk/rispubl/NUK/ris-phd-1.htm>

Author: Jeong-Heon Choi
Thesis Title: Luminescence ages of Quaternary marine sediments on the Eastern coast of Korea and their geomorphic implications
Grade: PhD
Date: August 2004
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Several sets of marine terrace are exposed along the southeastern coast of the Korean peninsula. The formation ages of these terraces have attracted considerable attention because they provide essential information on local crustal stability. Over the last few years, considerable effort has been put into the determination of these ages using optically stimulated luminescence (OSL) dating of the marine sediments from which the terraces were built. However, previous efforts to establish a chronology using OSL methods have produced controversial results, particularly because of stratigraphic inconsistency and poor reproducibility. In this work, the application of OSL dating based on the single-aliquot regenerative-dose (SAR) protocol for quartz is investigated. The dependence of equivalent dose on the preheat and cut-heat temperatures (thermal treatment of the regeneration and test-doses, respectively) are examined. Linearly modulated luminescence signals from chemically cleaned quartz samples are used to identify the presence of a thermally unstable component with a large optical cross-section (component A'), which in part affects the ability to correct for sensitivity changes during measurements, and thus the reliability of the equivalent dose estimates. In some samples, a higher heat treatment after the test-dose is shown to improve the ability to measure a dose given in the laboratory before any heat treatment (dose recovery test). This higher temperature treatment effectively removes component A', and hence improves sensitivity correction. Despite of the removal of component A',

the samples from one site (Oryu Terrace) still exhibit various undesirable OSL characteristics, which result in stratigraphically inconsistent OSL ages. To resolve this stratigraphic inconsistency, these characteristics are investigated, and luminescence component separation used.

Results thus obtained from 2nd terraces, which is located at the elevation of 7~18m (a.m.s.l), are reproducible at each sampling location, and give ages grouping broadly into 50~70 ka (Oryu ~ Kwansong Terraces, Nasa ~ Bihag Terraces) and 110~120 ka (Weseong Terrace), but laterally discontinuous on a scale of tens of km. The OSL results for the younger group are supported by radiocarbon ages from overlying terrestrial deposits. The OSL age of one site from 3rd terrace (Suryeom Terrace; ~43m a.m.s.l) has recently been dated to about ~100 ka. From these observations, it is suspected that the Weseong Terrace (110~120 ka; ~ 17m) belongs to 3rd Terrace (rather than 2nd Terrace), and that differential crustal uplift or vertical dislocation between Oryu-Kwansong block and Weseong-Bihag block has led to its misidentification as 2nd Terrace. This argument is examined using OSL ages of aeolian sand dunes locate in the Pohang area.

Author: Zenobia Jacobs
Thesis Title: Development of luminescence techniques for dating Middle Stone Age sites in South Africa
Grade: PhD
Date: May 2004
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Middle Stone Age (MSA) archaeological sites in South Africa can contribute to the debates on the origins of modern humans and modern human behaviour. Participation in these debates requires accurate and precise chronologies. Most of the MSA dates to beyond the range of radiocarbon dating and optically stimulated luminescence (OSL) dating is an appropriate alternative method.

This study is primarily concerned with testing and developing luminescence measurement techniques appropriate for dating complex sedimentary deposits at archaeological sites. Instrument behavioural tests are performed to test the reproducibility of the Risø single grain system and the appropriateness of the comparison between results obtained from single aliquots and single grains, when using different stimulation wavelengths. Single aliquots of quartz are measured to assess the appropriateness of the

conventional and modified single aliquot regenerative-dose (SAR) measurement procedures. Single grains of quartz are measured to investigate the grain-to-grain variability in quartz OSL behaviour and how this will impact on derivation of equivalent dose (De) using the conventional or modified procedures.

Application of single grain measurements is necessary because of the likelihood of depositional and post-depositional processes which may result in the under- or overestimation of the true burial age. Single grain De distributions are discussed in terms of how instrumental, quartz behavioural and depositional and post-depositional processes can influence the shape of the distributions. Rejection criteria are proposed to eliminate both quartz grains for which the measurement procedure is inappropriate and feldspar grains.

Dose rate evaluation methods are discussed and results obtained using a number of different laboratory-based and field-based methods are compared.

OSL ages from Blombos Cave and Sibudu provided an age range between ~50 ka and 140 ka for the pre- and post-Howiesons Poort cultures. These ages are used to discuss the chronology of the MSA in South Africa and the evidence for early modern humans and modern human behaviour at Blombos Cave.

Author: Gunter Erfurt
Thesis Title: Radioluminescence spectroscopy and dosimetry on feldspars and synthetic luminophors for geochronometry applications
Grade: PhD
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This PhD thesis reports on new results of the characteristics of photo- and radioluminescence emissions at 865 nm (1.43 eV) and 910 nm (1.36 eV) emitted by potassium-rich feldspars (KAlSi₃O₈). For the physical interpretation of its behaviour, the IR luminescence in potassium-rich feldspars is compared to a well investigated IR luminescence occurring in lead-doped potassium chloride (KCl:Pb) and to a study on the IR luminescence of lead-rich KAlSi₃O₈ (amazonite). The emissions in both materials, potassium-rich feldspar and potassium chloride, are most likely due to electron transitions in

Pb⁺ from the 72P1/2 and 72S1/2 excited states to the 62S1/2 ground state of Pb⁺. Pb⁺ originates from interaction processes of Pb²⁺ cations, substituted for K⁺ cations in the tetrahedral feldspar framework or the cubic lattice of KCl, with ionising radiation (Pb²⁺ + β , γ , ... \rightarrow Pb⁺). Another result of the experiments reported is the fact, that this radiatively induced conversion is reversed by thermal treatment of the minerals (Pb⁺ + T \rightarrow Pb²⁺). Also, the Pb⁺ centres can be “optically bleached”, which is also interpreted as reversed conversion Pb⁺ + $h\nu$ \rightarrow Pb²⁺. Implications for the theory of the infrared optically stimulated luminescence (IR-OSL) are discussed.

Following upon these results, a fully automated radioluminescence (RL) measurement instrument for dating and dosimetry was designed and built. The instrument is based on a commercial Daybreak 1100 automated TL reader system, widely used in thermoluminescence (TL) dating. It was re-designed and highly modified to adapt it to the physical and methodological needs of the IR-RF dating technique and other RL dosimetry applications. This new system holds up to 10 samples, has an integrated bleaching and irradiation unit, and measures the radio-fluorescence (RF) (excitation using 10 ¹³⁷Cs sources, each 5 MBq activity). All technical requirements for the measurement of optically excited luminescence were implemented in order to investigate the defect structure of luminescent materials. Because of the broad wavelength range and the high sensitivity of the photomultiplier detector used, the system is suitable for a great many luminescent materials, natural and synthetic.

Furthermore, a calibration method and the dosimetric concepts based on the Bragg-Gray cavity theory is described in detail. This calibration method uses the blue RF emission of Al₂O₃:C at 415 nm (3.0 eV) for the β source dose rate estimation and shows much lower calibration errors than yielded by the application of calibration procedures using natural dosimeter materials such as feldspar and quartz. Finally, examples of IR-RF dating results on Quaternary sediments together with independent age control are presented.

The conclusions of this PhD thesis have important implications for the physical precision of luminescence dating methods in principal and especially for the determination of the deposition time of Quaternary sediments using the infrared radiofluorescence (IR-RF) of potassium-rich feldspar grains.

This thesis is available online at <https://fridolin.tu-freiberg.de/archiv/html/PhysikErfurtGunter946007.html>

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25 – 29 July 2005

University of Cologne, Germany

The University of Cologne invites you to the 11th *International Conference on Luminescence and Electron Spin Resonance Dating*. LED 2005 will bring together different experts in the field of trapped charge dating from all around the world. The topics will range from fundamental studies of the physical basics of trapped charge dating, through advances in equipment technology and analytical procedures, to applications in dating Quaternary deposits and archaeological material. A few invited lectures will provide an overview on the main topics. A half-day workshop preceding the conference is planned for Sunday, July 24th. This workshop will focus on basic problems involved with the application of trapped charge dating and will be relevant for non-specialists and students.

Persons interested in attending the conference are kindly asked to contact the organisers (U. Radtke, Department of Geography, University of Cologne: e-mail: LED2005@uni-koeln.de; FAX ++49 221 470 5124). The deadline for registration is 1st February 2005. We recommend the conference WEB-page at <http://www.uni-koeln.de/LED2005> for regular updates and links.

U. Radtke

