

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.

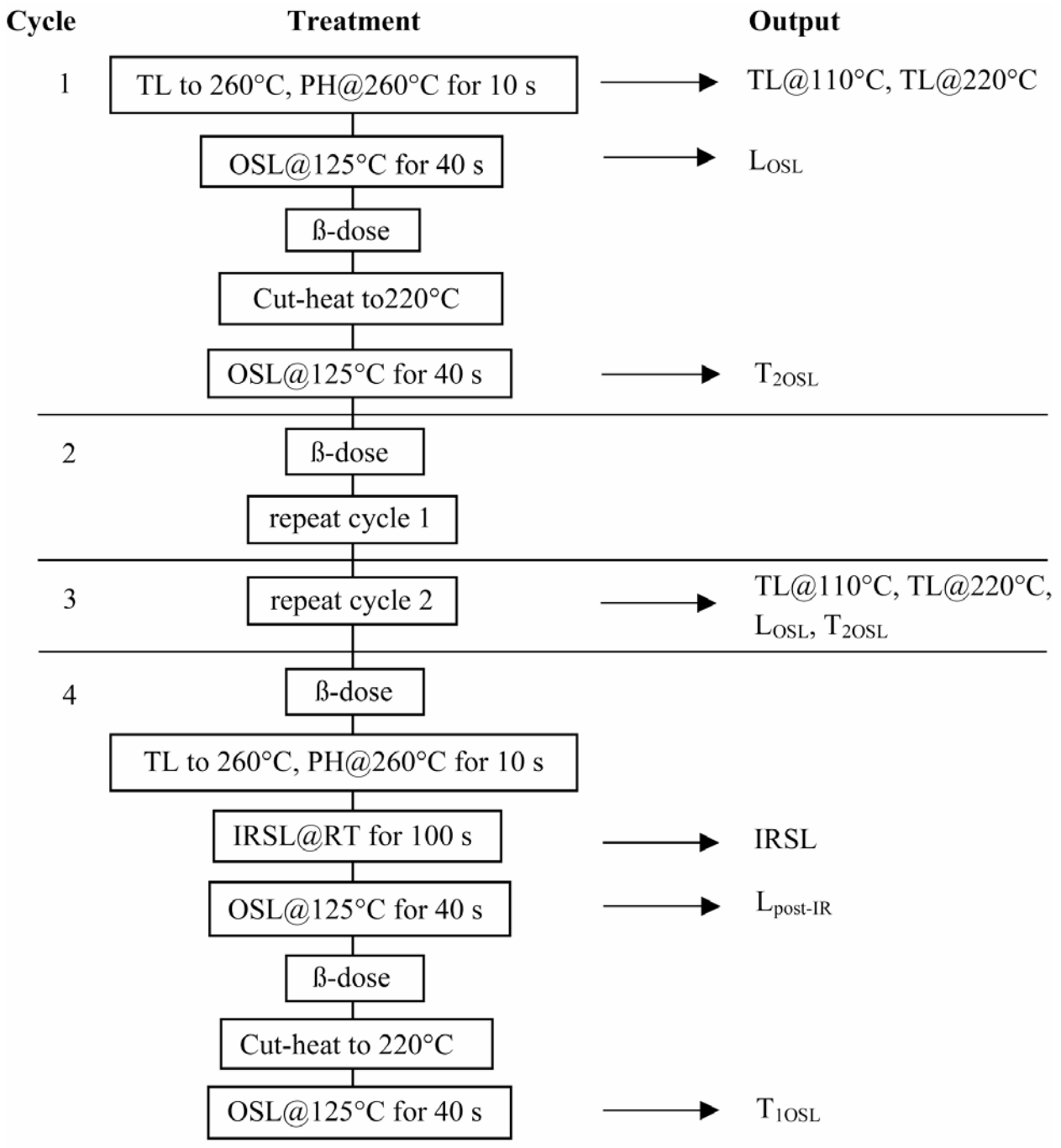


Figure 1: Measurement sequence designed to detect feldspar contaminants. For TL@110°C, TL@220°C, L_{OSL} and T_{2OSL} the output of cycle 3 was used, and for IRSL, $L_{post-IR}$ and T_{1OSL} the output of cycle 4 was used. Equal laboratory regenerative doses of ~10 Gy were administered in each cycle.

performed using either blue LEDs emitting at 470±30 nm (delivering ~30 mW cm⁻²) or IR LEDs emitting at 875±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.

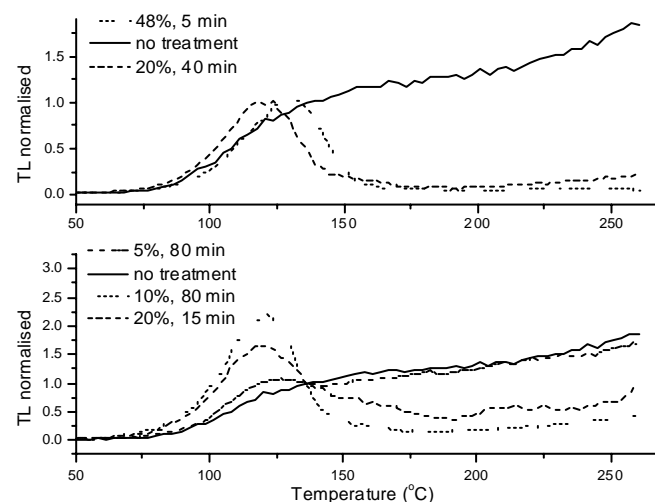


Figure 2: TL curves to 260°C (heating rate 5°C s⁻¹) for the loess sample. TL curves are normalised by the 110°C peak (but not shifted). Each curve represents a different HF treatment. With increasing acid strength and, consequently, with increasing removal of the feldspar component, the TL signal at 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.

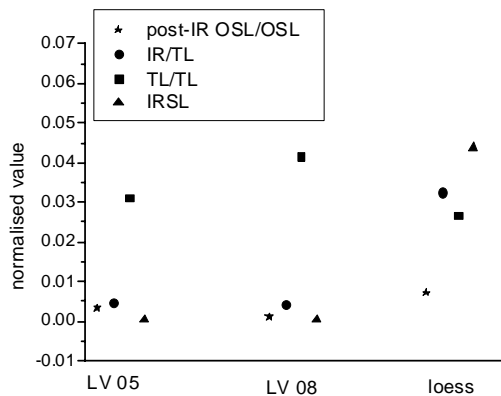


Figure 3: Results from feldspar contamination tests after sample treatment in 48% HF for 5 minutes. The post-IR OSL/OSL values are calculated from the differences between the measured values after etching and the 'feldspar-free' value of 1, divided (normalised) by the differences between the 'no treatment'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

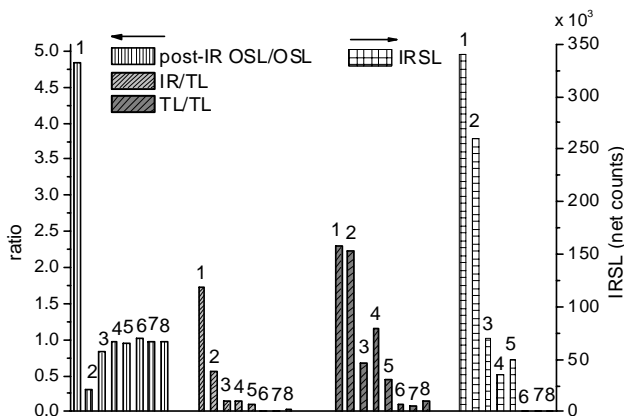


Figure 4: The effect of various etching procedures on the decrease in feldspar content for sample LV 01. 1: no treatment, 2: 2%, 80 min, 3: 4%, 80 min, 4: 10%, 80 min, 5: 20%, 5 min, 6: 20%, 15 min, 7: 20%, 40 min, 8: 48%, 5 min (for data see Table 1).

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.

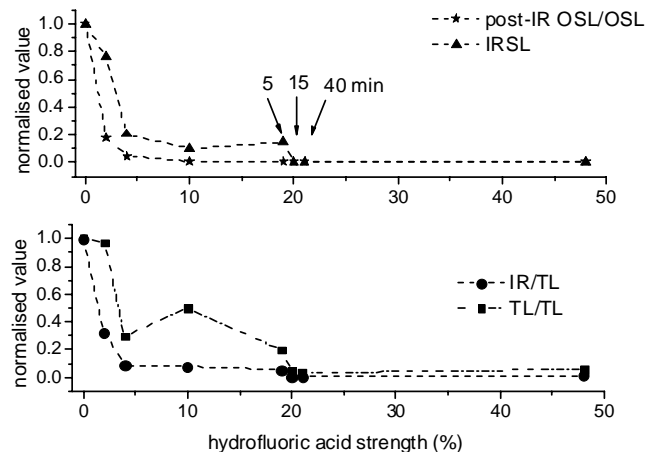


Figure 5: Comparison of the feldspar contamination tests for sample LV 01. The plots display the normalised values (calculated as described in the Fig. 4 caption) versus HF strength. The time difference of treatment with 20% HF (5, 15 and 40 min) is depicted by three data points centred on 20% HF strength. 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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