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Testing heated flint palaeodose protocols using dose recovery procedures

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Abstract

Thermoluminescence (TL) dating of materials from archaeological contexts has been shown to be an accurate method when comparisons are made with other chronometric dating methods; however, little has been published on the verification of the measurement protocols used to determine the equivalent dose (palaeodose). Instead of testing TL dating protocols for heated flint using archaeological material with unknown thermal and radiation history, dose recovery tests for three samples of different geological origin are presented. These samples exhibit TL emission in the UV, blue and orange–red wavelengths. In addition to the two multiple aliquot protocols (standard additive-regeneration and normalization) generally used to determine the palaeodose, the single-aliquot-regenerative-dose (SAR) TL and OSL procedure, a 'short' SAR–TL and isothermal luminescence (IT) decay procedures are applied using detection windows limited to these emissions.

Accurate dose recovery is obtained for the standard and normalization protocols in the commonly employed detection window (UV-blue), the 'short' SAR in the orange–red window and some IT measurements. While the standard techniques give the most accurate and precise results, detection of the TL and IT orange–red emission in connection with a 'short' SAR protocol also gave accurate and precise results. Such procedures are especially suitable for samples too small for standard multiple aliquot techniques, which require large samples.

1. Introduction

The accuracy of dating archaeological sites with thermoluminescence (TL) methods has been shown for only a very limited number of samples when comparisons are made with other chronometric dating methods. Good agreement of dating results was obtained with independent methods such as radiocarbon (e.g. Abeyratne et al., 1997), K/Ar (e.g. Pillans et al., 1996), or with similar TL methods on different materials from the same site (e.g. Valladas and Valladas, 1987). Generally, such comparisons are satisfactory and give confidence in the methods, but only a few attempts to verify the technique (e.g. Tribolo et al., 2003) have been published, especially to check the measurement protocols used to determine the palaeodose (equivalent dose).

As a first step to test luminescence dating protocols for flints, geological samples rather than archaeological material are used here. Thus, no assumptions about the temperature and radiation history of the samples under investigation have to be made, which is in contrast to samples for dating purposes from archaeological contexts, for which irradiation and thermal histories are unknown a priori. The influence on the determination of the palaeodose of different radiation and thermal histories can be investigated in the laboratory by varying these parameters. The importance of such knowledge has been shown, for example, by Duttine et al. (2005) who found the palaeodose to be dependent on the original temperature reached by the archaeological samples. Recovering a known dose applied within a luminescence reader employs optimized experimental conditions. This restricts the evaluation to only the recovery protocol. Parameters such as bleaching after sampling (e.g. Tribolo et al., 2003), sample preparation (crushing, etc.) and dose rate effects do not have to be taken into consideration. Protocols thus can be rejected solely based on their failure in accurate dose recovery.

In contrast, experimental setups in actual dating applications are far from being this ideal, and a number of experimental parameters might influence the final results to a non-quantifiable degree. Therefore, a subsequent step by step analysis is necessary to experimentally determine the effects of a non-optimized procedure, eventually allowing the selection of the most suitable protocol(s) for dating application. By eliminating

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protocols which already fail to recover known doses under optimal conditions, the number of subsequent experiments in the search for the most accurate and precise way to date archaeologically heated flint can be reduced. The present study is aimed at protocols which need less material and are less labour intensive. Therefore, it focuses on single-aliquot-regeneration (SAR) procedures.

2. Sample material and general experimental setup

Three samples of geological origin were selected, for which an analysis of some luminescence properties, such as the emission spectra, are already available (Richter et al., 1999). Sample NORD-1 is a Cretaceous flint from the Baltic Sea, samples JU-2 and PLA-1 are Jurassic hornstones from southern Germany, where the former is from nodules and the latter from bands of flint (Richter et al., 1999). These samples represent three different modes of the genesis of what is commonly referred to as flint, chert, hornstone, etc. (see e.g. Church, 1994 for discussion).

Measurements were performed with a Risø DA-15 luminescence reader, equipped with a ^{90}Sr/^{90}Y-source and an EMI 9236QA bialkaline photomultiplier (PMT). Detection windows were restricted by using filters for UV (U-340), UV-blue (BG25+HA30), blue (GG420+B390), orange–red (600-FS40), only a heat absorption filter (HA30), or by the PMT response, which is always used in addition to the built-in quartz window (SICO SQ1) (Fig. 1).

A minimum of three discs were used for each dose point or experiment presented here, but the majority of the measurements is based on 6–10 discs. For analysis, the TL data was integrated over ±30 °C of the peak temperature after immediate background subtraction. For blue optical stimulated luminescence (OSL) and isothermal luminescence (IT) data the first 0.8 and 5 s and the last 3 and 5 s background subtraction were used, respectively.

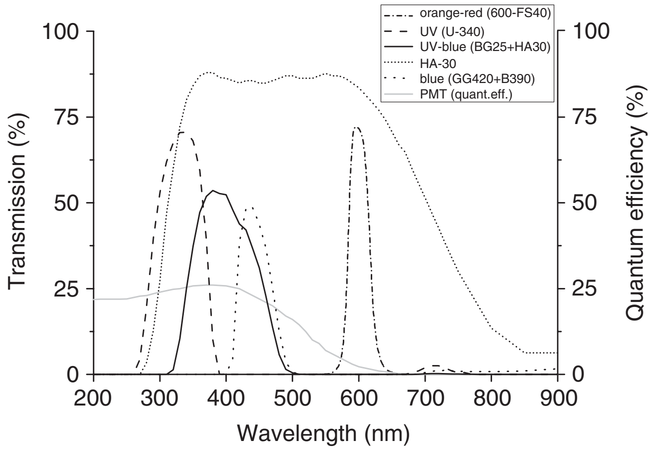

Fig. 1. Transmission of filters used to restrict the luminescence detection to UV (U-340), UV-blue (BG25+HA30), blue (GG420+B390), orange–red (600-FS40), broad (HA30), or restricted only by the efficiency of the PMT (EMI 9236QA), given as quantum efficiency. No correction was made for the built-in quartz window (SICO SQ1).

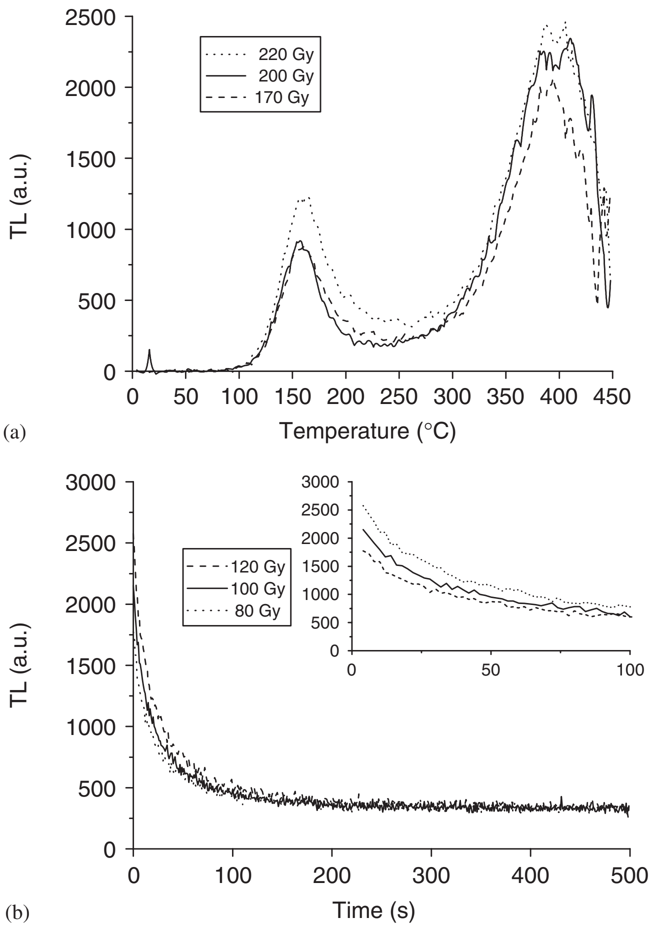

Fig. 2. Examples of orange–red luminescence from sample JU-2. Data is for one aliquot of (a) TL ramped at 10 °C s^{-1} up to 450 °C for 170, 200, 220 Gy and another aliquot of (b) IT for 80, 100, 120 Gy held at 340 °C for 500 s.

Examples of orange–red TL and IT measurements are given in Figs. 2a and b with a heating rate of 10 °C s^{-1} and a temperature of 340 °C, respectively.

3. Sensitivity change experiment

Any procedure in luminescence dating of heated flint also employs the measurement of signals after the sample material has been heated in the laboratory. Such a heating can cause a change in sensitivity to ionizing radiation, which is strongly dependent on the temperature and length of heating time (Bowman and Seeley, 1978; Göksu et al., 1989). This is especially true for SAR TL procedures, where the same material is used for regeneration after natural luminescence measurement of up to 450/500 °C. Regeneration material in non-SAR approaches is heated/zeroed at much lower temperatures and smaller sensitivity changes are thus expected. It is therefore necessary to understand the influence of these parameters on the sensitivity of the material under investigation.

Crushed geological material (90–160 μm) was treated with 10% HCl and mounted on discs. Discs were heated in 20 °C temperature steps from 380% to 560 °C and held at each temperature for 20 s. Temperatures in open fires reach up to 650 °C, with peak temperatures reaching 900 °C (e.g. Richter, 1995).

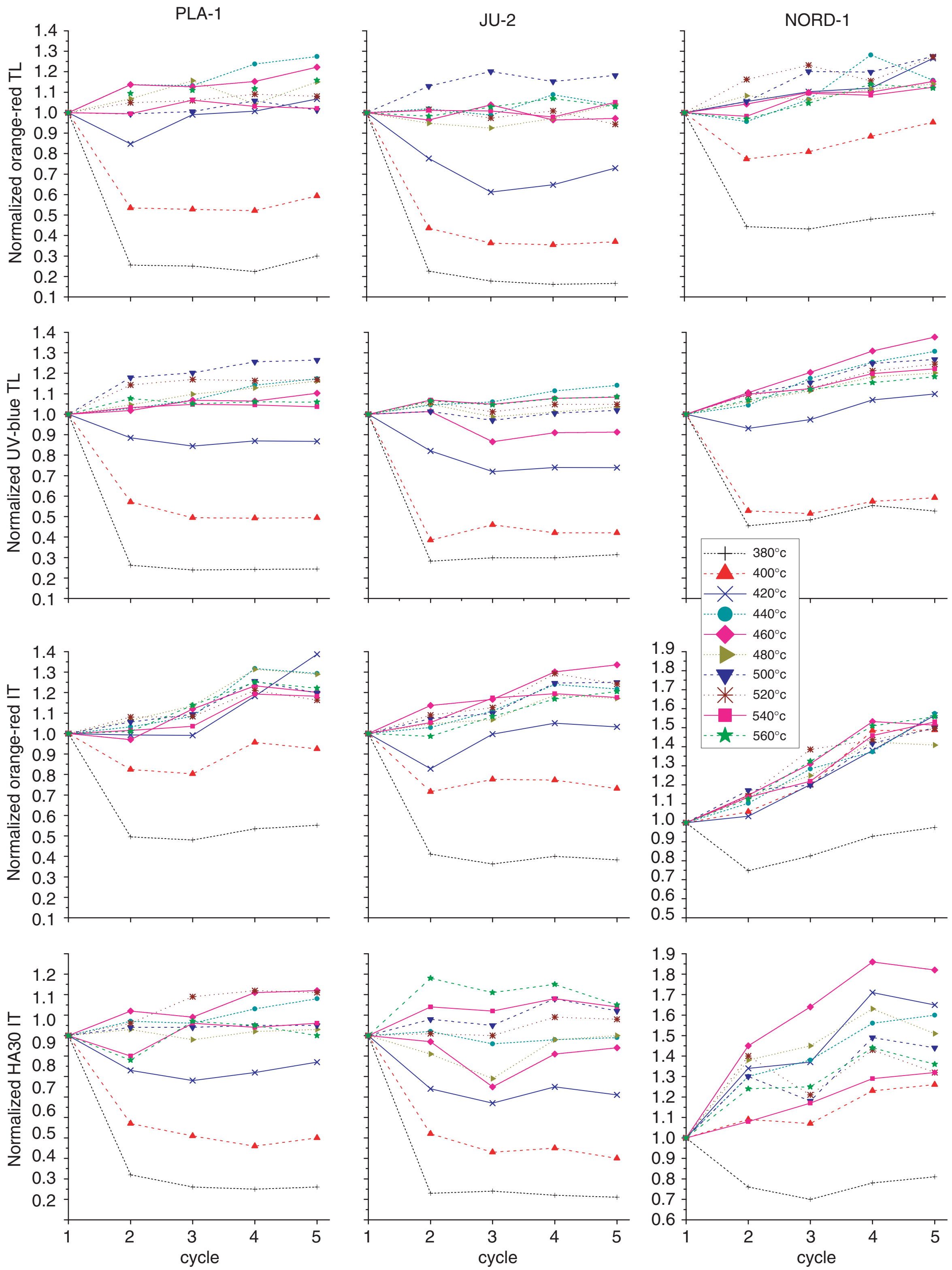

Fig. 3. Results of sensitivity change experiment with samples PLA-1, JU-2 and NORD-1 for orange–red TL (top row), UV-blue TL (second row), orange–red IT (third row) and broad (HA30) IT (bottom row) detection. Initial temperatures to simulate a prehistoric heating were 380–560 °C for 20 s in steps of 20 °C. The luminescence was then measured (TL up to 450 °C, IT at 340 °C after a 350 °C cut heat) for 5 cycles of 20 Gy β-irradiation, which was normalized to the first measurement.

Calcination of rock material has been reported to occur at temperatures as low as 540 °C (Holleman and Wiberg, 1952) and has been observed in open fires for flint as well (Richter, pers. observation). Calcined flints disintegrate easily and are not used for dating purposes, because of the structural changes caused by the strong heating. On the other hand, very long heating times are required to achieve such temperatures within rocks (Valladas, 1981). It is therefore unlikely that many archaeological flints used for dating were exposed to the α–β quartz phase transition temperature of 573 °C. A major sensitivity change is expected to occur at this temperature, which would probably mask any other pattern. The maximum temperature for this experiment was therefore set below the phase transition. In this experiment, it was assumed that an insignificant number of grains cracked because of the heating and no carbonates were exposed which could have given rise to spurious TL (Valladas, 1978), for which no indications were found in this experiment. For each temperature, one disc was used for each protocol of TL and IT detection in different wavelength windows (Fig. 3). Five cycles of giving a 20 Gy β-dose with measurement up to 450 °C for the TL, and at 340 °C for 500 s after a 350 °C cut heat when IT measurements were performed. The resulting data were normalized to the first measurement, which could be regarded as being equivalent to an 'archaeological' signal.These data were used to assess the sensitivity changes caused by the luminescence measurements in relation to the sensitivity obtained by the initial heating. In contrast to prehistorically heated material, with a dose that is unknown a priori, it is possible in this experiment to observe the sensitivity change caused by the measurement of the first 20 Gy dose signal, which is dependent on the original temperature history of the sample. So far, no observations have been made on dose dependency of flint TL, but future sensitivity change experiments need to take this into account (oral communication, Mercier, 2005).

Based on previous results (e.g. Bowman and Seeley, 1978) there appears to be a relationship between increased sensitivity and rising temperature. Therefore, one would expect to observe the sensitivity to increase when the measurement temperature is higher then the original heating temperature. It follows, that the lower measurement temperature required for IT measurements would probably allow the dating of samples for which the zeroing was sufficient, but lower than the temperature needed for a TL measurement (up to 450 °C), and thus the change in sensitivity would be smaller for IT compared to TL.

It has to be noted, that any sensitivity change for samples which were zeroed at low temperatures certainly will be masked by the incomplete resetting of the geological signal.

4. Results of sensitivity change experiments

It should be emphasized that the background for subtraction was determined immediately after the luminescence measurement. This is necessary especially for any orange–red signal from flint to be detected, because the observation is dominated by the incandescence light (Richter and Krbetschek, 2006). For comparison, the same procedure was employed for the other measurements as well. Therefore, the actual number of times the sample has been heated is twice the number of cycles shown. The currently employed protocols for measuring TL from heated flint employ only one heating cycle after the original prehistoric heating (equivalent to cycle 2 in Fig. 3), which is in contrast to SAR protocols, in which the sample is heated several times. The interest of a sensitivity study is thus in the sensitivity change observed for the second cycle, the third cycle in case of the 'short' SAR, and more cycles for standard SAR procedures.

The results of the sensitivity change study (Fig. 3) show a general tendency of sensitivity to increase for temperatures above 440 °C, but lack a strict relationship between temperature and increased sensitivity. It appears that the orange–red TL (upper row in Fig. 3) exhibits the least sensitivity changes, but the differences for the UV-blue TL (second row in Fig. 3) are minor, especially for the second and third cycles. Sample NORD-1 always exhibits the strongest sensitivity changes, whereas the sensitivity changes for PLA-1 and JU-2 only increase significantly after the third cycle for most of the temperatures above 440 °C.

The expectation of smaller sensitivity changes for IT measurements is not supported by this data (third and bottom rows in Fig. 3), and the changes actually appear to be more severe compared to those for TL measurements in the rows above. For samples PLA-1 and JU-2 sensitivity changes are small for several repeated TL measurements and only minor for samples heated around 500 °C. The data for measurements in the blue detection window (GG420 + B-390 in Fig. 1) are similar to those for the UV-blue and are not shown.

5. Recovery dose (D_R) protocols and rejection criteria

To test measurement protocols, dose recovery tests were performed with luminescence detection limited to several different detection windows (Fig. 1). The recovery doses (D_R) were chosen with reference to the UV-blue TL dose-response curve (not shown), i.e. in the linear region (30 Gy) and at the onset of saturation (200 Gy), or in between (100 Gy).

The samples were crushed (90–160 μm) in a steel mortar and zeroed in the laboratory by holding at 500 °C for 30 min. Since heating could break up some grains and expose carbonates which could give rise to spurious TL (Valladas, 1978), the samples were treated with 10% HCl after heating. All TL measurements were recorded in a N_2-atmosphere using $5\,°C\,s^{-1}$ ($10\,°C\,s^{-1}$ for orange–red TL) heating rate to 450 °C with immediate measurement of the background.

Standard protocols in heated flint TL dating use multiple aliquots additive and regeneration growth curves, where the latter are used to correct for the supralinear behaviour of the material (Aitken, 1985). The extrapolation protocol, after Aitken (1985), was used, as well as the 'normalization' procedure (after Valladas, 1992; Mercier et al., 1992) for the recovery dose of 200 Gy.

SAR protocols either follow Murray and Wintle (2000a,b) or a 'short' SAR TL approach (Richter and Krbetschek, 2006), which does not use a test dose and needs only two closely set regeneration doses. These two points are set to produce signals which narrowly bracket the natural luminescence. A straight line between two points very close to each other is a sufficiently good representation of the dose-response curve, even with a strong curvature when close to saturation, and thus allows the determination of the dose. Such a procedure can be used only if no, or very limited, sensitivity changes have occurred. These two different SAR procedures were employed as well for 500 s isothermal (IT) measurements (Murray and Wintle, 2000a,b) in N_2 at 340 °C after a 350 °C cut heat. Additionally, the recovery of a 30 Gy dose was attempted with blue-OSL using a SAR protocol (Murray and Wintle, 2000b) and UV-detection (U-340 filter).

Unlike in a dating application, by only using one kind of radiation (β) for the recovery dose (D_R) no corrections are necessary for differences in the relative efficiencies of the different types of radiation. Such differences have been found to be responsible for different results in palaeodose determination for archaeological samples, when using different wavelength detection, e.g. UV-blue or orange–red (Richter and Krbetschek, 2006).

The following rejection criteria were applied in the analysis: For multiple aliquot procedures, measurements not within two standard deviations (2σ) of the average for each dose point were rejected.

For all SAR protocols, the failure to recycle a dose point within 10% was used as a rejection criterion. Additionally, measurements where the recovery dose signal ('natural signal') was not bracketed by the two regeneration points in the 'short' SAR protocol were rejected.

With the exception of the normalization procedure and the 'short' SAR, the analysis was performed with the ANALYST software package. It was not necessary to consider errors associated with source calibrations, because the same source was always used for recovering the dose. Errors given for the 'short' SAR are the σ of the individual measurements only. Error estimates for the normalization procedure are based on the error propagation of the individual dose point σ values and following Brumby (1992) for linear extrapolation.

6. Results of dose recovery experiments

6.1. OSL

The data for the SAR recovery of a 30 Gy dose with OSL are not presented. The preheat test revealed a large scatter for recuperation, recycling ratios and recovered doses for all three samples under investigation. No obvious trends were observed and a preheat temperature of 270 °C was chosen. The regeneration growth curves were found to be already at the onset of saturation at approximately 100 Gy. The majority of measurements had to be rejected due to recycling ratios exceeding 15%. Therefore all results for sample JU-2 had to be rejected and the D_R was overestimated by 15–20% for the few remaining discs (about 2 out of 6 measured) for the other two samples. The results are statistically in agreement with the dose to be recovered, but the associated errors exceeded 25%. No acceptable results at all were obtained if the 110 °C TL-peak of the test dose was used for sensitivity correction instead of the OSL response. This study thus supports the view of Poolton et al. (1995), that OSL dating of flint is probably not possible.

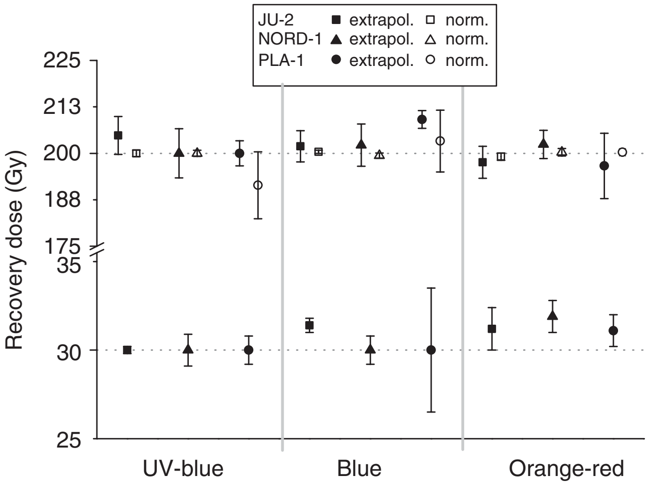

Fig. 4. Results (±1σ) for 30 and 200 Gy recovery doses using standard protocols of extrapolation (extrapol.) and normalization (norm.) for TL detection in the UV-blue, blue and orange–red wavelength bands (see Fig. 1).

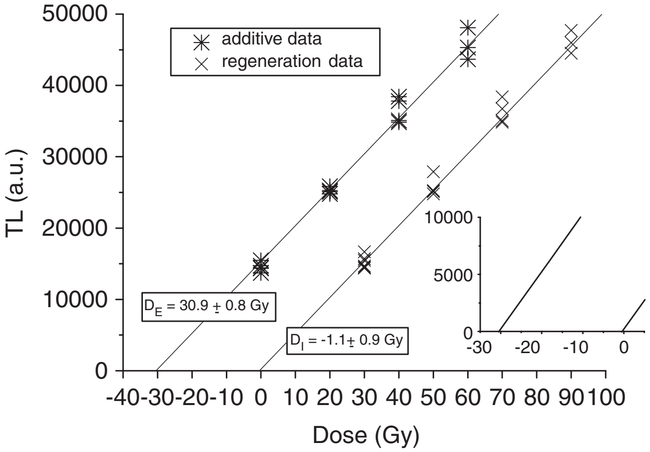

Fig. 5. Examples of additive and regeneration growth curves of orange–red TL (PLA-1). The insert shows the x-intercepts of the linear fits for the measured dose and supralinearity correction. Note the 'negative' value for the latter.

6.2. Standard extrapolation and normalization procedures for TL and IT

Recovery results for 30 and 200 Gy employing the standard extrapolation protocols for the UV-blue TL-detection window were excellent and gave values in agreement well within 1σ (Fig. 4). Results obtained for the same experiments but with the blue detection window gave significantly larger error estimates. The precision of extrapolation methods for the orange–red TL was even worse, but had a better accuracy than the blue detection window (Fig. 4), for which two experiments failed to recover the dose within 1σ. It should be noted that for the orange–red TL, the extrapolations for supralinearity correction intercepted the y-axis at negative dose values for all samples (Fig. 5), and thus were not incorporated in Fig. 4.

The assumption of the proportionality of the two glow curves for using the normalization method was validated (Mercier et al., 1992), and the results for all three investigated detection windows were both accurate and precise (Fig. 4).

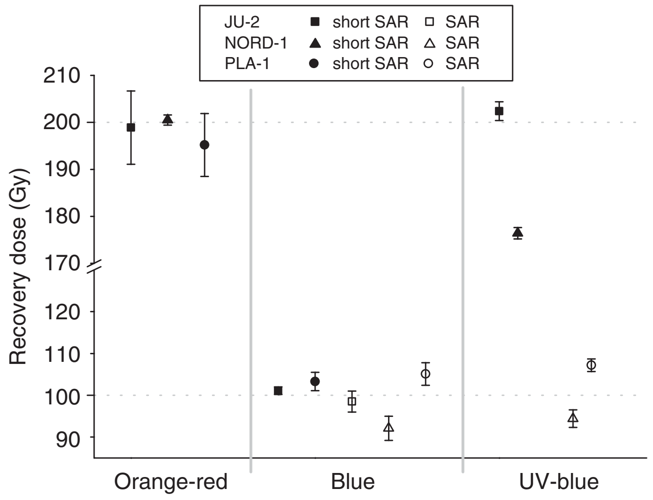

Fig. 6. Results ($\pm 1\sigma$) of dose recovery tests with TL SAR protocols for recovery doses of 100 and 200 Gy, using short SAR and standard SAR.

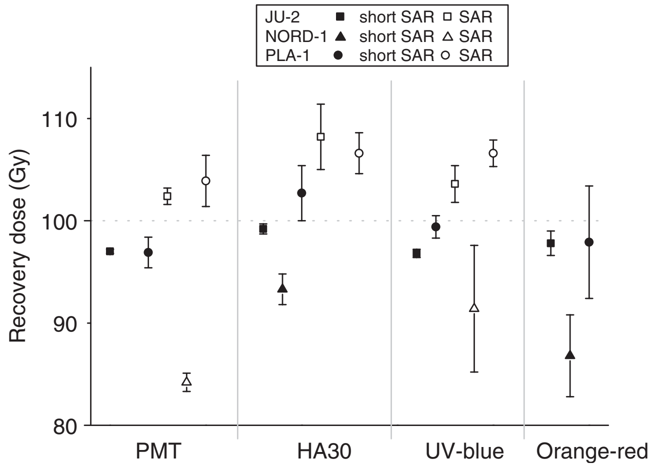

Fig. 7. Results ($\pm 1\sigma$) of recovery tests for a dose of 100 Gy with isothermal decay (IT) SAR protocols for a variety of wavelength detection windows.

6.3. Single-aliquots-regenerative dose (SAR) procedures for TL and IT

A number of SAR experiments failed to give results (Fig. 6) due to the aforementioned rejection criteria. Notably, the 'short' SAR procedure failed for TL detection in the UV-blue window. Only results with at least three successful aliquots are presented, equivalent to a 'success rate' of 50% or better in most cases.

Dose recovery for 200 Gy was good for the orange–red signal (Fig. 6), which is consistent with results obtained on archaeological samples (Richter and Krbetschek, 2006). Using a 30 Gy recovery dose (data not shown) the results were better than within 4% of the dose to be recovered, and have associated errors of less than 4%. Recovery of a 100 Gy dose using the blue detection window was fair, but less satisfactory than for the UV-blue TL (Fig. 6). Over estimation, as well as under estimation, was observed for this wavelength for 100 and 200 Gy recovery doses. Results for the orange–red TL were statistically identical (within 2σ) to the dose to be recovered, which was true for the blue detection window as well, but not for the UV-blue.

Similar behaviour was observed for the IT–SAR measurements for a 100 Gy dose (Fig. 7), where the majority of results agree within 2σ. The 'short' SAR approach produced the highest precision. It should be noted that the dose was almost always underestimated for sample NORD-1, and no results at all were obtained for an orange–red standard SAR procedure. No clear tendency could be observed, except that 'short' SAR seems to underestimate and standard SAR seems to overestimate doses.

7. Discussion

The sensitivity change experiments (Fig. 3) reveal a large dependency of sensitivity changes on the sample material, and to a lesser degree on the detection window. The effect of the incompleteness of the resetting of the geological TL is obvious for at least the first two heating steps (380 and 400 °C); this is responsible for the apparent strong sensitivity changes for lower initial temperatures. Thus, such strong sensitivity changes can serve as an additional indicator of the incompleteness of resetting in a prehistorically heated sample. Small sensitivity changes should thus be found only for well-zeroed samples, i.e. heated above about 450 °C.

The expected lower sensitivity change for IT protocols is not confirmed, because TL protocols exhibit smaller sensitivity changes. It appears that TL–SAR procedures should be feasible for samples heated at around 500 °C (equivalent temperature to these experimental conditions), because the sensitivity changes are small for UV-blue and orange–red. This is especially true for the first three cycles, thus favouring a 'short' SAR approach.

The protocols of choice for recovering doses for heated flint are standard protocols in the UV-blue detection waveband, with the most precise results obtained by the 'normalization' technique (Fig. 4). The suggestions by Richter et al. (1999) that the inclusion of some UV TL-component with these methods might be problematic and that detection windows should be better restricted to blue emission only is not supported by this study. Extrapolation methods also seem to work for the orange–red TL in general, but give large error estimates, which are mostly due to the low signal intensities. This TL emission needs to be investigated for its growth at low doses, because it might, in contrast to UV-blue, not exhibit supralinear behaviour, as indicated by the failure to obtain a meaningful result with regeneration curves for all three samples (Fig. 5). However, the 'negative' results for supralinearity correction could be due to an inappropriate background subtraction. Nevertheless, given the associated errors of these corrections, linear growth even at low doses seems to be more likely, because for all three samples extrapolation to zero is within 2σ of the least square fit of the data.

The large sensitivity changes observed for IT measurements indicate that time plays a role in sensitivity changes as well as geology (e.g. NORD-1). Overall, the data are not conclusive but indicate the potential application of SAR procedures, notably the 'short' SAR TL approach for well-heated flint samples.

The apparent failure of standard SAR TL protocols to correct for sensitivity changes, as indicated by the complete failure of successful dose recovery for UV-blue detection, favours the ‘short’ SAR approach coupled with orange–red detection.

For the finest grained sample (NORD-1) recovered doses were the least accurate, especially for SAR-procedures. Dependency of luminescence on mineralogical composition in flints and cherts has been reported (e.g. Akridge and Benoit, 2001). Sample NORD-1 is a very cryptocrystalline sample, so its signal is likely to be dominated by the ‘amorphous’ silica phase, which might be more sensitive to sensitivity changes than larger quartz crystals, which are found more frequently in the other two samples.

8. Conclusions

As outlined earlier, this study is a first step in an effort to determine procedures especially suitable for small samples and a reduction of the preparation as well as analysis time required. In future studies, the effects of sampling and sample preparation have to be tested for the protocols which gave results in agreement with the applied doses, notably SAR-TL and SAR-IT procedures for the orange–red emission. Because IT signals can be measured at temperatures where the heat radiation is still low, the use of a red sensitive PMT might lead to improvements.

Additional tests are required for the determination of differences in alpha sensitivities for the various wavelengths used, because this may be the reason for failure to recover comparable archaeological doses (e.g. Richter and Krbetschek, 2006). The various protocols have to be tested not only for more geological material, but also on archaeological samples with good age control (e.g. K/Ar) for their last heating. The use of the SARA procedure, instead of SAR as used in this study, might overcome some problems (oral communication, N. Mercier, 2005).

To conclude, standard protocols and TL detection in the UV-blue seem to be the best choice for dating heated archaeological flint whenever enough material is available. This work shows the potential for successful application of the detection of the orange–red TL with a ‘short’ SAR (see Richter and Krbetschek, 2006) for small samples not yielding enough material for standard protocols.

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