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Towards a SAR-ITL protocol for the equivalent dose estimate of burnt quartzites

C. Tribolo* and N. Mercier

Institut de Recherche sur les Archéomatériaux, UMR 5060 CNRS - Université de Bordeaux, Centre de Recherche en Physique Appliquée à l'Archéologie (CRP2A), Maison de l'archéologie, 33607 Pessac cedex, France

*corresponding author: ctribolo@u-bordeaux3.fr (C. Tribolo)

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Abstract

Up to now, the preferred protocol for determining the Equivalent Dose (D_e) of burnt lithics has been a multiple aliquot additive and regenerative dose (MAAD) approach based on the measurement of the thermoluminescence (TL) signal. The purpose of this study was to test a single aliquot regenerative dose (SAR) protocol measuring the isothermal thermoluminescence (ITL) signal. It is shown that this protocol is efficient for quartzites where the TL glow curve is dominated by the 280 and 375°C peaks. However, it fails with quartzites for which the relative contributions of TL peaks change with regenerative doses.

Introduction

Up to now, the determination of the equivalent dose (D_e) for burnt rocks (either flints or quartzites) has been done using the thermoluminescence signal (TL) at 370-380°C (for a heating rate of ~5°C.s^{-1}) and by applying mainly multiple-aliquot additive and regenerative dose (MAAD) protocols. Meanwhile, it is well known that multiple aliquot approaches present several drawbacks (in comparison with single aliquot approaches, e.g. Murray and Wintle, 2000), such as: 1) the large amount of material needed for obtaining one D_e estimate, 2) the lack of systematic controls for accuracy such as dose recovery tests (Richter and Temming, 2006), 3) the difficulties in correctly extrapolating the TL growth curve to a zero signal, more particularly when the D_e is close to saturation, and 4) the low reproducibility of the measured TL signals sometimes seen for a given dose.

Attempts at using TL single aliquot protocols for the D_e determination of flints have been performed through detecting their UV-blue emission (Valladas, personal comm.), but were not successful due to the impossibility of satisfactorily correcting for the sensitivity changes occurring in most samples during the first heating in the laboratory. Richter and Temming (2006) and Richter and Krebtschek (2006) were quite satisfied however with a TL short SAR procedure (single aliquot regenerative dose protocol, without any test dose measurements) for flints in detecting their orange emission as they observed that, in most cases, the sensitivity changes were low enough to be neglected. These authors also tested a protocol using isothermal measurement of the TL (ITL) in various wavelength ranges (UV-blue, orange–red and full spectrum) either with standard SAR or short SAR procedures. In a minority of cases only the SAR-ITL dose recovery ratios were close to unity at one sigma. Finally, these authors concluded that the standard MAAD-TL protocol in which the UV-blue emission is selected (Valladas, 1992) remains the best choice for flint dating.

Beyond these attempts focusing on burnt flints, studies using SAR-ITL protocols have been performed on sedimentary quartz grains with signal detection in the UV range and on volcanic materials with ITL detected at red wavelengths (Wintle 2010 and references therein). The main purpose of these studies was to extend the quartz dating range, as it was noticed that the ITL signal saturates at higher doses than the optically stimulated luminescence (OSL) signal (Murray and Wintle, 2000). Experiments on the origin, bleachability, thermal stability and dose response of the ITL signal have been performed at different temperatures (e.g. Jain et al., 2005, 2007 a and b), and various SAR-ITL protocols have been tested by different authors (Table 1). Choi et al. (2006) and Jain et al. (2005) (also see Gibling et al., 2005) obtained satisfying results with ITL measurements at 310 or 320°C, but Huot et al. (2006) and Buylaert et al. (2006) both noted that their SAR-ITL protocol (also at 310°C) is inefficient for their samples: a sensitivity change occurring during or just after the measurement of the natural signal is not properly corrected for by the following test dose.

Table1: *Summary of the different SAR-ITL protocols that have been proposed. In all cases but Barham et al. (2011), the same heat treatment is done for the natural and regenerative doses and for the test doses.*

Reference		Murray and Wintle (2000)	Jain et al. 2005; Gibling et al. 2005	Choi et al. 2006	Huot et al. 2006	Buylaert et al. 2006	Richter and Temming 2006	Vanderberghe et al. 2009	Pagonis et al. 2011	Barham et al. 2011
Material		Fluvial quartz	Fluvial or aeolian quartz from Indo-Gangetic plain	Various sedimentary quartz	Various sedimentary quartz	Chinese loess	Flint	Various sedimentary quartz	Simulation	Quartz from river bench in Zambia
Wavelength		UV	UV	UV	UV	UV	Various	UV		
Heating Rate			2°C/s	5°C/s	5°C/s	5°C/s	5°C/s	2°C/s	5°C/s	
	Preheat	340°C cut, OSL at 330°C for 5s	No	310°C for 10 s	No	No	350°C cut	300°C for 10s	300°C for 10s	280°C for 10s
Protocol	ITL	330°C for 1000s	320°C for 500s	310°C for 500s	310°C for 100s or 300s	310°C for 300s	340°C for 500s	270°C for 600s	310°C for 600s	310°C for 250s except for the natural: 310°C for 3s + 110 min bleaching in solar simulator
	Cleaning	no	no	no	no	no	no	OSL 280°C for 40s	OSL 280°C for 40s	no
Notes			Works fine for most; for some samples sensitivity change during 1st measurement is suspected		Sensitivity change during 1st measurement	Sensitivity change during 1st measurement. SARA preferred	Accurate at 2 sigma only		Sensitivity change during 1st measurement	

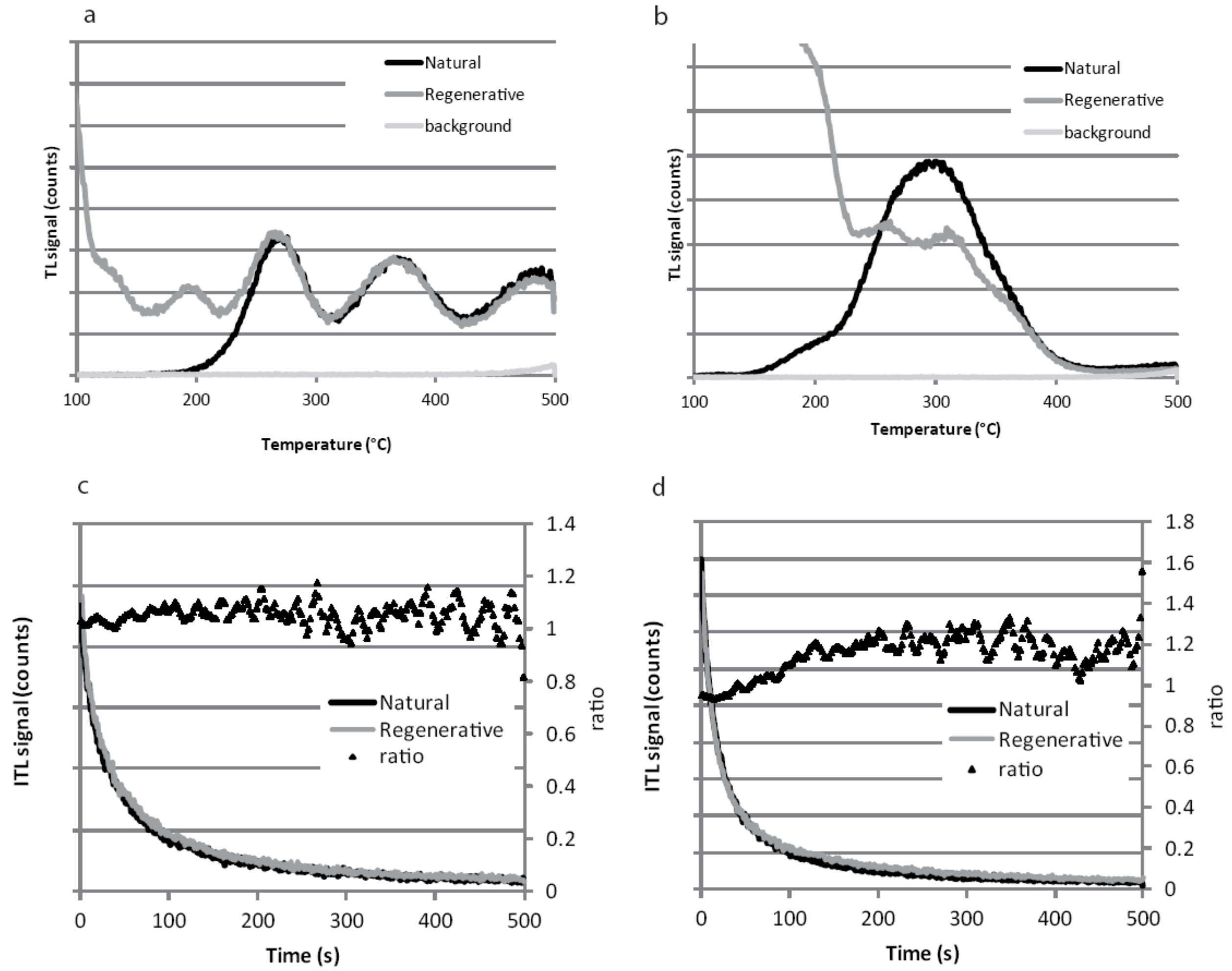


Figure 1: *Natural, regenerative and background TL glow curves obtained when the temperature is increased at 5°C/s. a) DRS178, b) DRS196. Natural and regenerative ITL decay curves at 300°C after a 10s preheat at 300°C. The ratio between the two curves is also plotted; c) DRS178, d) DRS196.*

This effect has also been observed by Pagonis et al. (2011) for simulated SAR-ITL experiments. To get round this problem, three kinds of solutions have been proposed: 1) allowing for this initial sensitivity change with a Single Aliquot Regenerative and Additive dose (SARA) protocol (Buylaert et al., 2006), 2) minimizing the sensitivity changes by working with lower temperatures (Vandenberghe et al., 2009), or 3) by replacing the long heat of the natural by a short heat (3 s only instead of 250 s for the test and regenerative doses) followed by an optical bleaching in a solar simulator (Barham et al., 2011). It is not clear whether the successes or failures obtained within the cited works are due to differences in the protocols or to samples that may or may not stand the SAR-ITL protocols. Meanwhile, as the main problem that was identified for these bleached sedimentary quartz seemed to be linked with the first heating of the sample, it is possible that it would be less stringent for quartz that were already submitted to a high temperature heat (at least 350°C) in the past. The purpose of this paper is to present SAR-ITL tests performed on such burnt quartzites.

Samples and Measurements

The quartzite samples come from Diepkloof Rock Shelter (South Africa), a thick Middle Stone Age deposit (Parkington et al., in prep; Porraz et al., in prep; Texier et al., 2010; Tribolo et al., 2009, in prep). The samples can be described as a cluster of quartz grains, the granulometric distribution of these grains varies from sample to sample. The core of each quartzite was sawed and crushed so that a quartz powder with an artificial grain size of 100-160µm was obtained.

Measurements were performed with a TL/OSL DA15 Risø reader, equipped with an EMI Q9235 photomultiplier tube, preceded by a combination of Schott BG39 and Corning 7-59 filters for detection in the UV-blue wavelength range (about 330-450 nm). A $^{90}Sr/^{90}Y$ beta source was used for irradiations and the heating rate was 5°C.s^{-1} for all experiments.

Based on the observation of the natural and regenerative TL glow curves, two groups of samples can be distinguished: in the first group, the natural TL signal presents two peaks at 280 and 375°C (the 325°C peak being likely dominated by this last one), and the shape of the regenerative glow curve is similar to the natural one above 280°C (e.g. DRS178 on Figure 1a). For the second group, the 325°C peak contributes significantly to a broad signal dominating the first glow curve but its relative contribution (in comparison with the 280 and 375°C peaks) is different in the regenerative curve. This allows us to distinguish these three peaks in this last curve but as a consequence, the natural and regenerative glow curves are not homothetic (e.g. DRS196 on Figure 1b). It is therefore unreasonable to combine the growth curves built from the additive and regenerative TL glow curves in order to determine the D_e of these samples. Therefore, when MAAD protocols are applied, the samples from this group are usually discarded (which represents about 45% of burnt stones from Diepkloof). Figure 1c and d display natural and regenerative ITL glow curves recorded at 300°C for samples DRS178 (first group) and 196 (second group) respectively, following a 10s preheat at 300°C. In both cases, the regenerative and natural decay curves seem very similar, though calculation of the ITL signal ratio reveals that the curves are not perfectly homothetic for DRS196, with a 10% increase during the first 100 s. Nonetheless, the question behind this is whether using an ITL protocol instead of a TL one could avoid the need to discard group 2 samples from the dating process.

The SAR-ITL protocol used in our experiments is presented in Figure 2. Each cycle is composed of the following steps: after irradiation, a 10s preheat at temperature T is applied and the ITL measurement is performed at the same temperature for 500 s. A 52 Gy test dose is given and the same preheat and ITL measurements are performed once again. A 500°C TL measurement has been included at the end of each cycle to avoid signal build-up (data not shown). This cycle is repeated for regeneration doses of D, 2D, 4D, 8D, 0, D and 8D, where D is close to the expected D_e. Unless otherwise specified, the first 20 s and the last 50 s of the ITL signals were considered as signal and background, respectively.

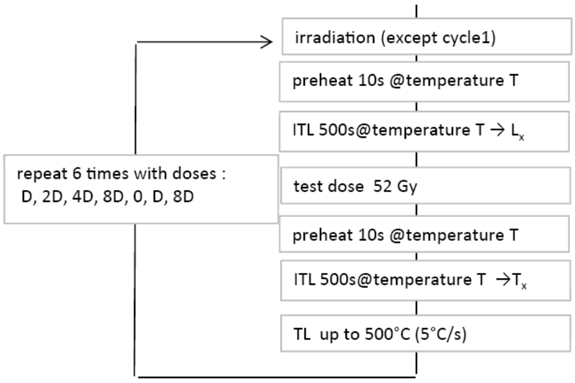
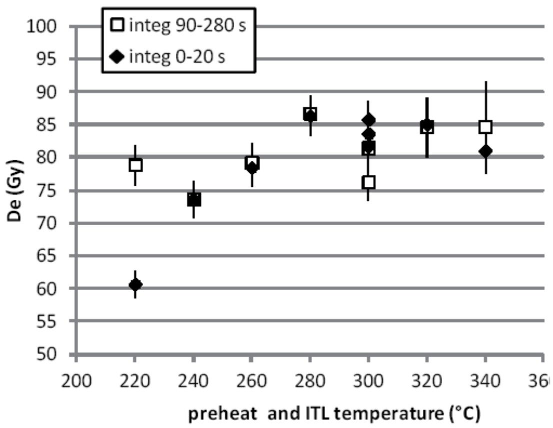


Figure 2: *SAR-ITL protocol.*

(a)



(b)

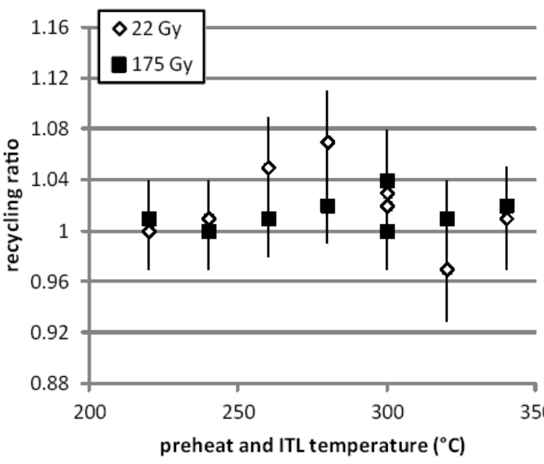


Figure 3: *a) SAR-ITL D_e values as a function of preheat and ITL temperature for DRS178. Signal is integrated either over the first 20s (black diamonds) or over the 90-280s interval (the last 50 s are used for background in both cases). b) recycling ratios for the SAR-ITL experiment as a function of the preheat and ITL temperature (integration: first 20s).*

Study of samples from group 1

SAR -ITL

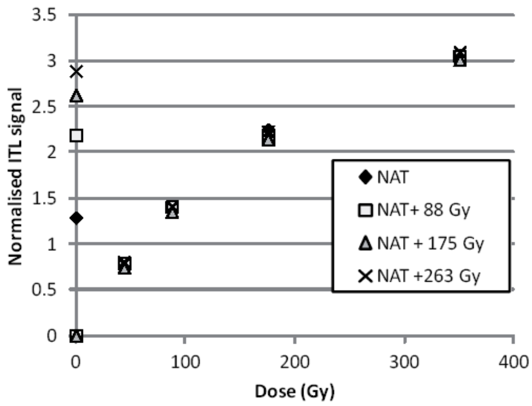
Figure3a shows the D_e as a function of the temperature T, which was varied between 220 and 340°C by steps of 20°C, for sample DRS178[1]. Figure 3b displays the corresponding recycling ratios for the lowest and highest regenerative doses. These ratios are all consistent with unity at one or two sigma whatever the temperature, showing that the sensitivity correction is efficient at least from the second cycle. Meanwhile, the calculated D_e values increase from 240 to 280°C (Figure 3a) and then remain constant up to 340°C (mean D_e on 280-340°C: 84±3Gy), whatever the integration interval: the D_e obtained when integrating a later part of the signal (e.g. 90-280s) is consistent at one sigma with the D_e estimates obtained for the first 20s, except at 220°C. Experiments that were carried out in order to correlate the ITL and TL signals suggest that at 220°C, the ITL signal can be entirely associated with the 280°C TL peak (data not shown), while above this temperature, the ITL signal is dominated by the 325° and 375°C TL peaks. As the 280°C TL peak is known to be unstable at long timescales (>10ka; Spooner and Questiaux, 2000), it is therefore not surprising that the apparent D_e decreases when its contribution to the ITL signal increases.

While observing that the D_e is not dependent on the temperature at least between 280 and 340°C is satisfying, it does not ensure that the obtained D_e is correct. A dose recovery test was thus performed in order to check for the efficiency of the protocol: the signal was zeroed by heat (450°C for 1h30min)[2], a known dose close to the expected D_e was given and the protocol was applied for a temperature of 320°C. One aliquot of DRS178 was tested and yielded a good recovery (estimated to expected dose) ratio of 1.01±0.03.

SARA -ITL

Huot at al. (2006) and Buylaert et al. (2006) noticed that the sensitivity change occurring during the first heat (ITL measurement) was not properly corrected for by the use of a test dose, but this correction was working fine for the following cycles. It was therefore feared that the dose recovery test performed

(a)



(b)

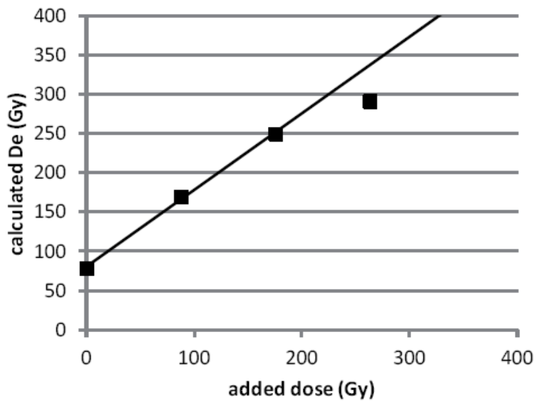


Figure 4: *a) SAR-ITL growth curves built for testing a SARA protocol with sample DRS178 (ITL and preheat temperature: 300°C). The L_x/T_x signals for the natural and natural+added doses are projected on the interpolated growth curve. b) The estimated equivalent doses are then plotted as a function of the added doses.*

in the previous section was somewhat biased since the resetting was done by heating the sample. As suggested by Buylaert at al. (2006), a SARA protocol would help answer this question since it allows the very first sensitivity change to be taken into account (assuming that all aliquots are affected by the same sensitivity change). It was then attempted on DRS178. Four aliquots were used, on which the SAR-ITL protocol at 300°C was applied, except that doses of 88, 175 and 263 Gy were added to the natural dose for three out of the four aliquots (Figure 4a). For each aliquot, the estimated dose was then plotted as a function of the added dose (Figure 4b). For the SARA protocol to be successful, the growth curve has to remain linear. In this condition, the intercept on the Y axis of the D_e versus added dose straight line corresponds, when it is divided by the slope of this line, to the corrected D_e (Mejdahl and Bøtter-Jensen, 1994).

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[1] Note that we used here a cut edge of the stone, which was submitted to beta and alpha radiations from the surrounding sediment. The D_e of this part is therefore different from the D_e obtained for the core, presented in Tribolo et al., (in prep).

[2] 450°C is the temperature indicated by the thermocouple of the oven. The actual temperature within the sample, measured with a thermocouple in contact with the quartz powder, is somewhat different: rapid increase from 20 to 200°C in <5min, 340°C reached by 30min, 390°C reached by 1h00 and 400°C by 1h30.

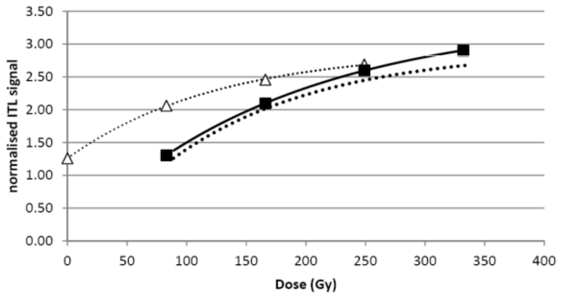

Figure 5: *Additive (white triangles) and regenerative (black squares) growth curves obtained with multiple aliquots for sample DRS178. The bold dotted line represents the additive growth curve after slide.*

On Figure 4a, it can be seen that the growth curve is linear in the dose interval lower than 200 Gy. Consequently, only the three added doses of 0, 88 and 175 Gy were considered for the D_e calculation (Figure 4b). The computation of the slope indicated that its value is close to unity (0.97±0.02) and the corrected SARA D_e was found to be 86±2 Gy, consistent with the D_e estimated with the SAR-ITL protocol (84±3 Gy), suggesting that for this sample there was no sensitivity change during the natural measurement cycle.

MAAD-ITL

As an alternative to the SARA protocol, a multi aliquot (MAAD) approach was also attempted. Four aliquots of DRS178 were used for building an additive dose growth curve (added doses: 0, 83, 146 and 249 Gy), while a regenerative growth curve was built with four other aliquots (signal reset with a 500°C cut heat reached at 5°C.s^{-1}, regenerative doses: 83, 166, 249 and 332 Gy) (Figure 5). Since only one signal per dose was available, each one was attributed a ±5% uncertainty (this value being likely higher than the reproducibility of our machine for this type of measurements). For calculating the D_e, both slide and extrapolation methods were used with either a quadratic or a saturating exponential fit. The D_e estimates obtained with the extrapolation methods have large uncertainties (77±22 Gy for the quadratic fit, 73±19 Gy for the exponential fit) and are therefore not informative. When slide methods are used, the D_e estimates are 87±8 Gy whatever the fitting model and are consistent with the D_e estimated with SAR-ITL or SARA-ITL protocols, indicating that all the ITL procedures (based on single or multiple aliquots) lead to equivalent results.

Application to other samples from group 1

SAR, SARA and MAAD-ITL protocols give consistent results for DRS178, suggesting the sensitivity corrections in SAR are effective and thus,

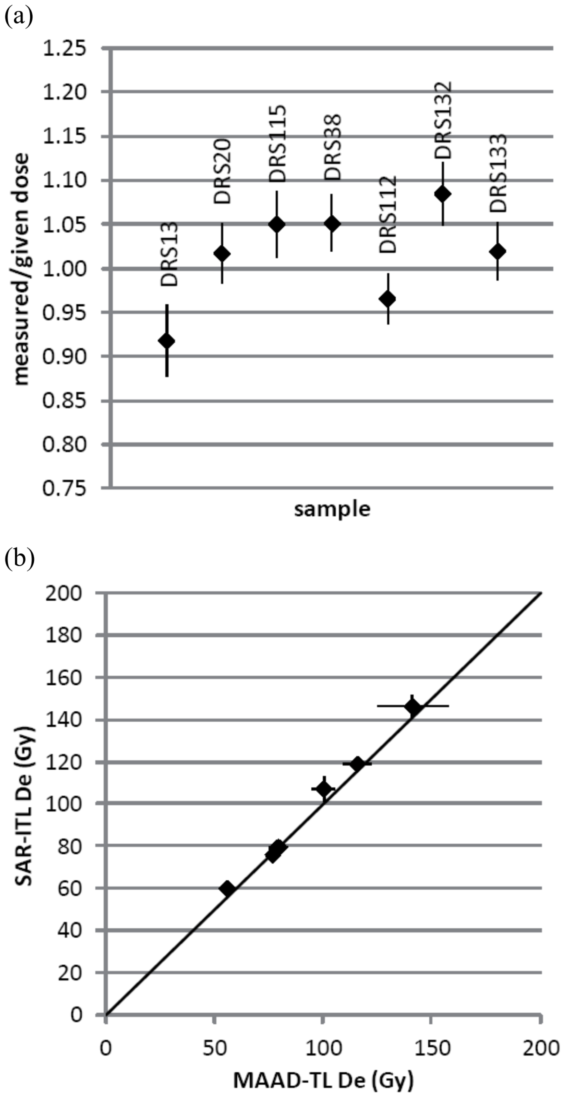

Figure 6: *Application of the SAR-ITL protocol to seven samples from group 1. a) dose recovery ratios for one aliquot per sample (mean: 1.02±0.05). b) Comparison of the SAR-ITL and MAAD-TL D_e values for these samples (slope: 1.05±0.04).*

that the D_e is accurate. In order to extend this observation, the SAR-ITL protocol was also applied to 7 samples from group 1 for which the D_e, within a 50-150 Gy interval, had been previously measured with a MAAD-TL protocol (Tribolo et al., in prep). Figure 6a displays the dose recovery ratios obtained for one aliquot of each sample with this protocol (preheat and ITL temperature at 320°C). They are all within 10% of unity. The SAR-ITL was then applied to these samples for temperatures between 280 and 340°C. As for DRS178, the D_e is both independent of the temperature for all samples (except DRS38, 112, 132 for which this interval is restricted to 300-340°C only) and of the integration zone. In all cases, the

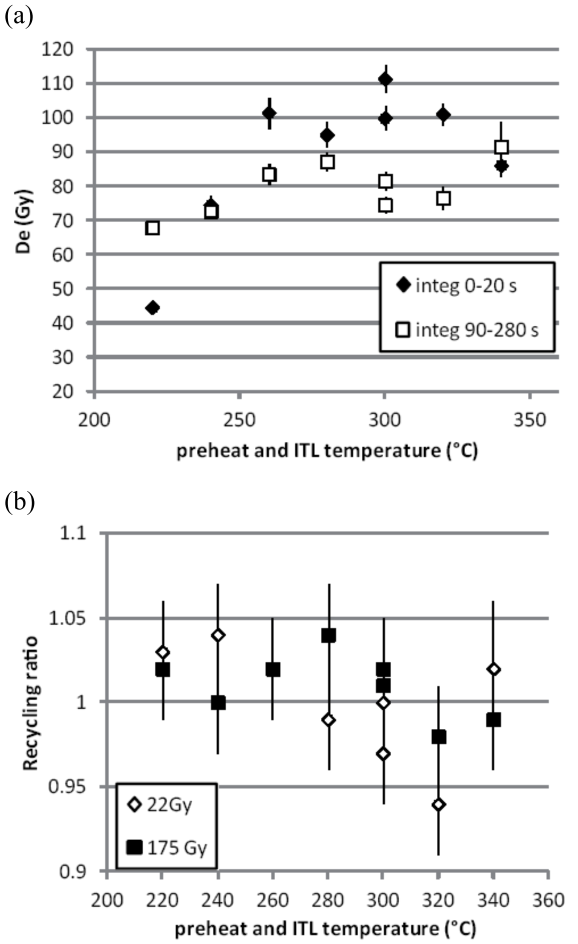

Figure 7: *a) SAR-ITL D_e as a function of the preheat and ITL temperature for sample DRS196. Signal is integrated either over the first 20s (black diamonds) or over the 90-280s interval (the last 50 s are used for background in both cases). b) recycling ratios for the SAR-ITL experiment as a function of the preheat and ITL temperature (integration: first 20s).*

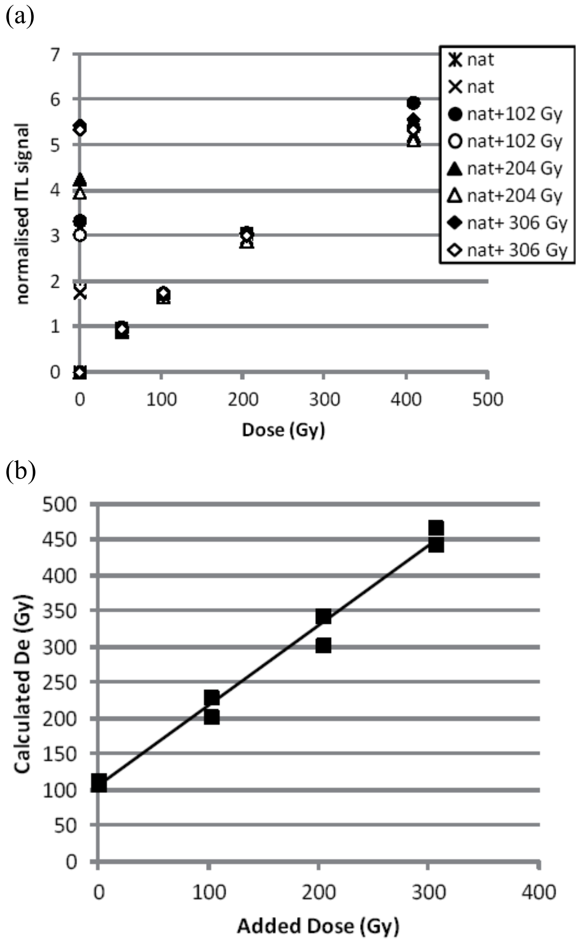

Figure 8: *a) SAR-ITL growth curves built for testing a SARA protocol with sample DRS196 (ITL and preheat temperature: 300°C). The L_x/T_x signals for the natural and natural+added doses are projected onto the interpolated growth curve. b) The estimated equivalent doses are then plotted as a function of the added doses.*

recycling ratios are within 10% of unity (data not shown). On Figure 6b, the SAR-ITL D_e estimates are plotted as a function of the MAAD-TL D_e values. The agreement is very satisfying, all points being consistent at one sigma with the 1:1 line.

Study of samples from group 2

SAR-ITL

The SAR-ITL protocol that was successfully tested on DRS178 and other samples from group 1 was applied to DRS196. The apparent D_e values are plotted on Figure7a as a function of the preheat and ITL temperature. They increase from 220 to 260°C, then are consistent at one sigma up to 320°C (mean 102±4 Gy) and apparently decrease again at 340°C. The recycling ratios are consistent with unity whatever the temperature (Figure 7b). However, it can be noticed that, contrary to DRS178, the D_e values obtained with a later part of the ITL signal (e.g. 90-280s: mean 81±4 Gy) are generally lower than those obtained for the first 20s, despite the recycling ratios being consistent with unity in these cases as well. It is therefore likely that the sensitivity correction is not efficient for the entire signal, maybe because this signal is composed of several components (Figure 1b).

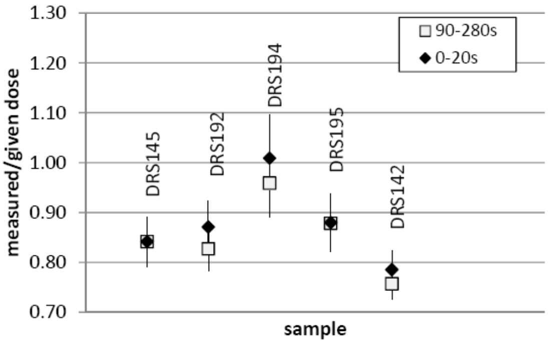

Figure 9: *Dose recovery ratios for samples of group 2 obtained with a modified SAR-ITL protocol (after Vandenberghe et al., 2009) (mean and standard deviation for the 0-20s interval: 0.88±0.07).*

SARA-ITL

The SARA-ITL protocol was thus applied to four aliquots of DRS196 (preheat and ITL temperatures fixed at 300°C). The growth curve is almost linear (Figure 8a), and so is the curve describing the D_e value as a function of the added dose (Figure 8b), suggesting that the sensitivity change is the same for each aliquot. The slope of this last curve is consistent with unity at three sigma only (1.12±0.05) confirming that the sensitivity correction of each aliquot is not efficient. When a later part of the signal is integrated, the slope is significantly lower than unity (e.g. 0.68±0.03 for 20-240 s), suggesting that the sensitivity change during the first measurement is even more important. The corrected D_e values for the 0-20s and 20-240s intervals are 96±10 and 132±10 Gy respectively, i.e. they are consistent with each other at 2 sigma only, indicating that for this sample the SARA protocol does not allow the recovery of the correct D_e.

Other protocols

The SAR-ITL protocol apparently failed because of uncorrected sensitivity changes during the first measurements. In order to minimize this effect, Vandenberghe et al. (2009) proposed applying a preheat at 310°C for 10s and performing the ITL measurement at 270°C only. A dose recovery test for this protocol was attempted on five samples of group 2 (DRS 142, 145, 192, 194, 195), except that the optical cleaning at 280°C between each cycle suggested by Vandenberghe et al. (Table 1) was replaced by a 500°C cut-heat. For the five samples, the recycling ratios were within 10% of unity (data not shown) and the D_e values were not dependent on the integration zone of the ITL signals. However for 4 out of 5 samples, the dose recovery ratio was not consistent with unity at two sigma, showing that this modified SAR protocol is inefficient for most of our samples (Figure 9).

Finally, as single aliquot protocols did not yield satisfactory results, a MAAD-ITL protocol was attempted with sample DRS142, in which a test dose and its induced ITL signal was used for normalization. These measurements showed that the test dose signal increases with the added dose, indicating a predose effect. This effect might partly explain the failure of the SAR protocols, though it does not seem to occur in all samples (e.g. it does not with DRS196).

Conclusions

It has been shown that the SAR-ITL protocol is generally accurate for samples of group 1 (i.e. with dominant peaks centered at 280°C and 375°C, showing no strong change of glow curve shape after a regenerative dose is given). In future works however, the efficiency of the protocol will be systematically tested for each new sample of this group, based - as for standard SAR-OSL protocols - on dose recovery tests, dose recycling tests, preheat and ITL temperatures plateau tests, and independence of the D_e on the signal integration zone.

It can be noticed that the preheat for 10 s before the ITL measurement was maintained in this work, while several authors have suggested that this might be useless, since the ITL itself contains a heating ramp (e.g. Jain et al., 2005). The effect of removal of the preheat will be tested in future works.

Up to now, no satisfying protocol has been found for group 2 samples (i.e. samples whose glow curve shape for regenerative doses is significantly different from the natural). Pagonis et al. (2011) recommended examining the size of the test dose. Ongoing preliminary tests also suggest that the use of a lower heating rate (e.g. $1°C.s^{-1}$ instead of $5°C.s^{-1}$) could be a way to explore, but further analyses are needed before strong conclusions can be drawn.

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Reviewer

S. Huot