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D_e determination for young samples using the standardised OSL response of coarse-grain quartz

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Abstract

It has recently been shown that it is possible to construct standardised curves of the sensitivity corrected growth in optically stimulated luminescence (OSL) with exposure to ionising radiation, and that they may be used in the dating of quartz and polymineral samples. Standardised growth curves are particularly advantageous where measurement time is limited, as once they have been defined, only the natural signal and the response to a subsequent test dose are required in order to determine the equivalent dose of a sub-sample. The present study is concerned with the application of the standardised growth curve approach to OSL dating of Holocene age samples. Systematic changes in the shape of the standardised growth curve of coarse-grain quartz are identified as the size of the test dose is varied, because of non-proportionality between the test dose and the luminescence test response. The effect is characterised by fitting the change in gradient of the standardised growth curve as test dose is varied. An equation is defined to describe standardised growth as a function of regenerative dose and test dose.

Regenerative dose responses of other samples in this study are treated as unknowns and recovered through different growth curves to compare precision and accuracy of various methods of D_e determination. The standardised growth curve is found to yield similar precision to conventional fits of single aliquot regenerative data, but slightly poorer accuracy. The standardised growth curve approach was refined by incorporating the measurement of one regenerative response for each aliquot as well as its natural signal. Measurements of this additional data point for aliquots of 22 samples were used to adjust the standardised growth equation, improving its accuracy. The incorporation of this additional data point also indicated a systematic uncertainty of 2.4% in the estimates of D_e.

Keywords: Standardised growth curve; Quartz; Optically stimulated luminescence; Late Holocene; Old Scatness; Sumburgh Hotel Gardens; Shetland

1. Introduction

This paper explores methodology for the measurement of equivalent dose (D_e) distributions for young (late Holocene) quartz samples, which have D_e values below saturation. It focuses on the application of a standardised growth curve approach, where a single growth curve is defined independently from one or more samples, before application to other samples, which then require only minimal measurements to determine a value for D_e. This approach is of particular value where large numbers of samples and/or aliquots are to be examined from a particular site or region.

Previous single aliquot approaches to the measurement of optically stimulated luminescence (OSL) from large numbers of quartz aliquots have suggested the use of a single

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Table 1
Standardised growth parameters from Eq. (1) for quartz

	D_T (Gy)	I_0	I_{Max}	D_0	I_{Max}/D_0
Roberts and Duller (2004)	3.1,4.1	0.03 ± 0.13	51.7 ± 0.6	55.1 ± 1.3	0.9383 ± 0.0003
Aber/42/J9	6	0.14 ± 0.07	47.8 ± 0.6	46.9 ± 1.1	1.0187 ± 0.0003
	5	0.20 ± 0.08	45.4 ± 0.6	46.3 ± 1.3	0.9813 ± 0.0004
	4	0.16 ± 0.08	49.3 ± 0.7	50.5 ± 1.3	0.9768 ± 0.0004
	3	0.14 ± 0.07	42.8 ± 0.6	44.2 ± 1.2	0.9669 ± 0.0003
	2	0.07 ± 0.03	36.9 ± 0.2	38.0 ± 0.4	0.9707 ± 0.0001
	1	0.12 ± 0.09	36.0 ± 0.5	38.6 ± 1.0	0.9326 ± 0.0004

Roberts and Duller (2004) measured 27 aliquots from six samples. Groups of fouraliquots from sample Aber/42/J9 were measured using a range of test doses (Fig. 1(c)).

measured regenerative point (e.g. Olley et al., 1999), rather than the generation of a full growth curve. However, this results in some systematic error in D_e when the value is either below or particularly above the regenerative dose used, and greater scatter is also expected because of the reduction in the number of regenerative points used. The fitting of exponential curves to a range of sensitivity corrected regenerative data from each individual aliquot (e.g. the single aliquot regenerative (SAR), dose approach of Murray and Wintle, 2000) might therefore offer optimum results in terms of precision and accuracy, whilst requiring many more measurements to be made. Using a standardised growth curve approach reduces the total number of measurements made, but some systematic deviation from a sample's growth characteristic might be expected at all dose levels, since the curve is not fitted to observed data. The question is: how significant will that deviation be?

The standardised luminescence signal is defined by Roberts and Duller (2004) as the luminescence signal following natural or laboratory irradiation, L_X, divided by the luminescence response to a subsequent test dose, T_X, multiplied by the magnitude of that test dose, D_T. Roberts and Duller (2004) found that the growth in standardised luminescence with increased regenerative laboratory dose was well fitted by the saturating exponential equation:

$$I = I_0 + I_{Max}(1 - e^{-D/D_0}). \quad (1)$$

In Eq. (1), I = standardised luminescence ($D_T \cdot L_X/T_X$), I_0 = standardised luminescence at zero dose, I_{Max} = standardised luminescence at saturation, D = natural or regenerative dose, and D_0 = the characteristic dose at which $I = I_{Max}(1 - e^{-1})$. Roberts and Duller (2004) found that growth in the standardised OSL signal from coarse-grained quartz from a variety of locations worldwide was well described up to 125 Gy, when the values of I_0, I_{Max} and D_0 given in Table 1 were used. This implied that growth in the standardised OSL signal from these quartzes was independent of the origin of the quartz. Nevertheless, where large numbers of samples were to be dated from a given site or region, Roberts and Duller (2004) advocated that a standardised growth curve should be defined for that site/region using a selection of samples, before being tested and applied to other samples in that study. The accuracy of D_e values derived in this way depends on the standardised growth curve being consistent between different samples, sub-samples, and within a range of measurement conditions.

The standardised growth parameters of Roberts and Duller (2004) (Table 1) were derived from SAR dose measurements (Murray and Wintle, 2000) made using "preheats" of 160 °C for 10 s prior to L_X measurements, and "cutheats" of 160 °C with immediate cooling prior to T_X measurements. Test doses for these measurements were 3.1 and 4.1 Gy, but the parameters obtained were used to predict D_e for 22 other samples, where test doses between 0.7 and 4 Gy were used. However, reduction in trapping probability as D_T is increased is expected to reduce the magnitude of T_X/D_T , and hence increase the standardised luminescence at a given value of regenerative dose (Roberts and Duller, 2004). Some inaccuracy should thus be present in estimates of D_e made using a standardised growth curve, if it was defined using measurements made with a different test dose.

Once a standardised growth curve has been adopted, only measurement of the natural signal, L_N, and the response to a single subsequent test dose, T_N, are required for each aliquot in order to determine its equivalent dose, D_e. As such, the standardised growth curve approach is particularly effective in reducing measurement time where D_e distributions are to be examined (e.g. Olley et al., 1999; Spencer et al., 2003), since large numbers of aliquots are typically measured per sample. However, small aliquots (typically < 50 grains) are often used to minimise averaging effects in such studies, and sensitivity corrected growth curve shape has been shown to vary between individual grains and small aliquots of quartz (e.g. Duller et al., 2000; Vandenberghe et al., 2003). The application of a single standardised growth curve might therefore produce inaccuracies in estimates of D_e for small aliquots, adversely affecting the D_e distribution observed for the sample as a whole. However, the major differences in growth curve shape arise from variability between aliquots in the dose at which growth in the OSL signal saturates (I_{Max}, Eq. (1)) (Duller et al., 2000), so differences

should be less evident at low doses. Small aliquots containing low doses yield low signal levels and consequently large statistical uncertainties, so any inaccuracy in the standardised growth curve might be outweighed by improvements in the precision of D_e determinations because the standardised growth curve is more precisely defined.

The aims of the present study were first to test for changes in standardised luminescence with test dose and to correct for any observed changes, and second to test the applicability of the standardised growth curve approach for the determination of small aliquot D_e distributions from young samples.

2. Samples and methods

As part of a wider study looking at the OSL dating of agricultural deposits, large numbers of samples were taken from agricultural infield deposits associated with the Old Scatness Broch (samples: Aber/34, /41), and nearby Sumburgh Hotel Gardens (samples: Aber/42) archaeological sites (Burbidge, 2003). The deposits were inhomogeneous in nature, and included material expected to date to the late Holocene (Burbidge et al., 2001; Rhodes et al., 2003).

The 180–212 μm quartz fraction was separated by standard HCl and H_2O_2 treatment, followed by dry sieving, density separation using sodium polytungstate solution, HF etching (40% for 45 min), and resieving. Luminescence measurements were conducted using a Risø TL/OSL-DA-10 reader, with Hoya U340 detection filters (2 × 2.5 mm, 50% transmission between 290 and 370 nm). Optical stimulation was from blue LEDs (470 nm), delivering 2.3 mW/cm^2 to the sample material, deposited as a monolayer on aluminium disks. The reader contained a 40 mCi ^{90}Sr/^{90}Y beta source that delivered approximately 1 Gy/min to the samples during the period in which the measurements were conducted.

OSL measurements were made at 125 °C, using a preheat of 170 °C for 10 s and a cutheat of 160 °C. Test doses applied to the Old Scatness and Sumburgh Hotel Gardens samples lay in the range 1–6 Gy. D_T was generally ∼ 70% of the expected D_e, but a minimum value of 1 Gy was applied. This was to ensure that precision was not limited by low T_X signal levels, but at the same time to avoid compromising the accuracy of the D_e values (Burbidge, 2003, Chapter 5).

3. An equation to account for non-linearity in both test and regenerative dose response

In order to test for changes in standardised luminescence with D_T, regenerative growth curves were measured for aliquots from a single sample, Aber/42/J9 ($D_e \approx 7.2$ Gy), using the range of test doses applied to all the archaeological samples in this study (1–6 Gy). OSL decay and regenerative growth up to 64 Gy are plotted in Fig. 1(a and b). The growth curves obtained for each test dose are much more similar

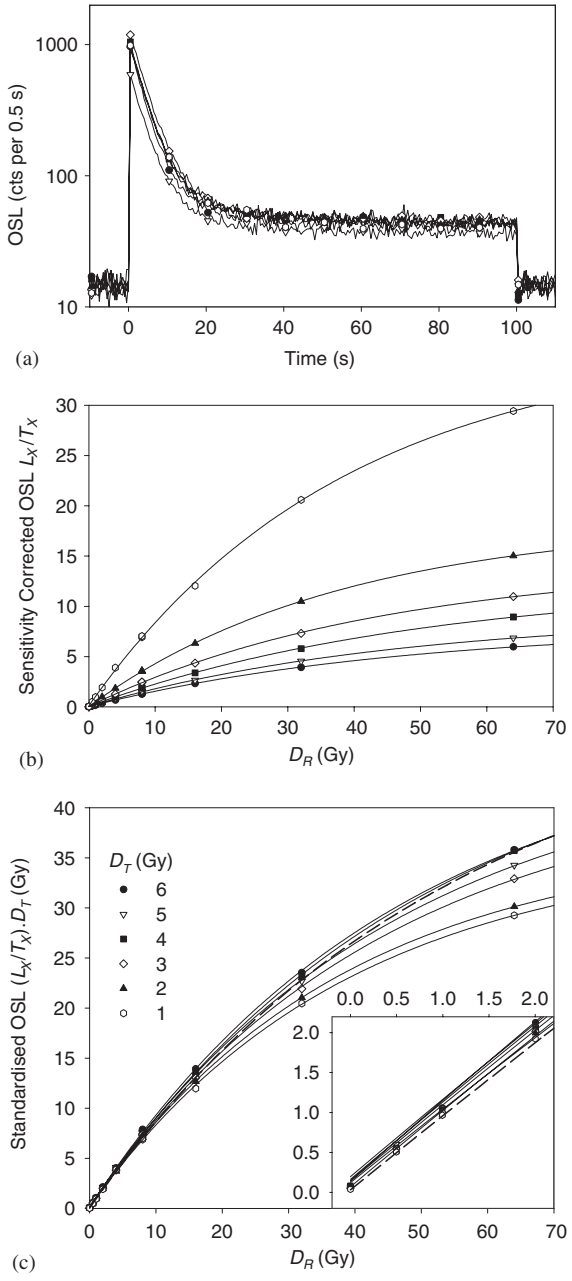


Fig. 1. (a) OSL signal decay following a regenerative dose of 8 Gy (note log OSL axis), (b) growth in sensitivity corrected OSL with regenerative dose (D_R), and (c) growth in standardised OSL. Each data point is the weighted mean of measurements on four aliquots of sample Aber/42/J9. Each group of four aliquots was measured using a different test dose, being 1–6 Gy in 1 Gy increments. Curves in (b) and (c) take the form of Eq. (1). Also shown in (c), as a dashed line, is the standardised growth curve for quartz of Roberts and Duller (2004). Details in Table 1.

to each other when standardised, or corrected for absolute sensitivity (i.e. $D_T \cdot L_X/T_X$, Fig. 1(c)), than when only corrected for relative sensitivity (i.e. L_X/T_X, Fig. 1(b)). However, the shape of the standardised growth curves does vary systematically with test dose (Fig. 1(c)).

The standardised growth curves at higher test doses appear similar to, but more curved than, that determined by Roberts and Duller (2004) (dotted line in Fig. 1(c)). The values of I_{Max} and D_0 for Aber/42/J9 are all lower than those of Roberts and Duller (2004) (Table 1). This may be because the maximum regenerative dose of 64 Gy was approximately half that used by Roberts and Duller (2004), allowing greater curvature in the less constrained fits.

In Eq. (1), I_{Max}/D_0 is the gradient of the curve at $D = 0$, and $-1/D_0$ controls the curvature (i.e. the change in gradient as D is increased). For Aber/42/J9, the curvature changes by $\sim 20\%$ across the 1–6 Gy range of D_T, but the individual values are scattered (Table 1). The gradient at D=0 and the value of the intercept, I_0, show some indication of dependence on D_T, but this is not likely to be significant considering the expected errors on the data (Table 1).

It is also apparent that Eq. (1) did not fit the standardised growth data closely at low doses (Fig. 1(c), inset). The effect persisted when weighted fits ($w = 1/\text{variance}$) were made. These ill fits at low doses are important because the present study aims to apply a standardised growth curve to young samples. The regenerative points at 32 and 64 Gy were thus removed, which markedly increased the errors on the parameter estimates using Eq. (1). For the limited dose range (0–16 Gy), closer fitting and more precise parameter estimation was obtained using a quadratic equation (Eq. (2)) to describe the standardised luminescence growth curve

$$I = aD^2 + bD + c. \quad (2)$$

Unlike the saturating exponential fit (Eq. (1)), the parameters (a, b, c) of the quadratic equation (Eq. (2)) do not relate directly to basic physical models for the process resulting in the growth of standardised luminescence (I) with dose (D). However, the individual parameters of a quadratic equation do relate directly, and individually, to the form of the curve. Where $D = 0$, $I = c$, the gradient of the quadratic curve at $D = 0$ is b, and change in gradient with D is $2a$.

Growth in standardised luminescence ($L_X/T_X D_T$) as a function of regenerative dose (D_R) was closely fitted by quadratic equations in the regenerative dose range 0–16 Gy, for each of the six test doses (D_T) employed (Fig. 2). Of the parameters a, b, and c, derived from these fits, a (change in gradient with D_R) and c (I at D=0) did not vary significantly with D_T (Fig. 3 (a and c)). However, there was a relatively strong, consistent linear relationship between b (gradient at $D = 0$) and D_T (Fig. 3(b)). Weighted mean values for a and c, and the linear equation fitted to b versus D_T are given by

$$a = -0.0130 \pm 0.0005, \quad (3)$$

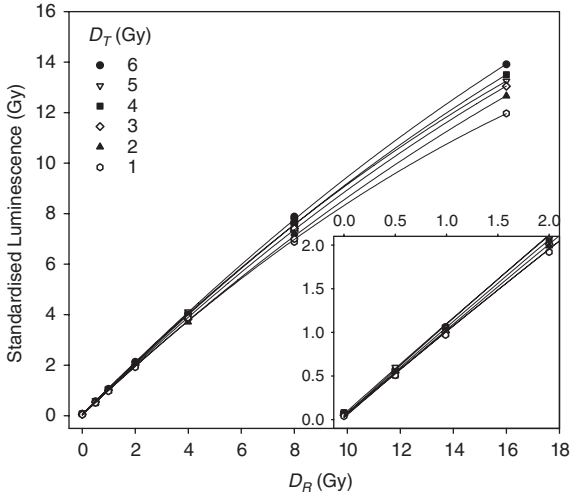


Fig. 2. Standardised growth curves for test doses between 1 and 6 Gy. Quadratic fits (Eq. (2)) were used in place of saturating exponential ones (Eq. (1)). Each point is the mean of measurements on four aliquots of sample Aber/42/J9. The dose range 0–2 Gy is expanded inset.

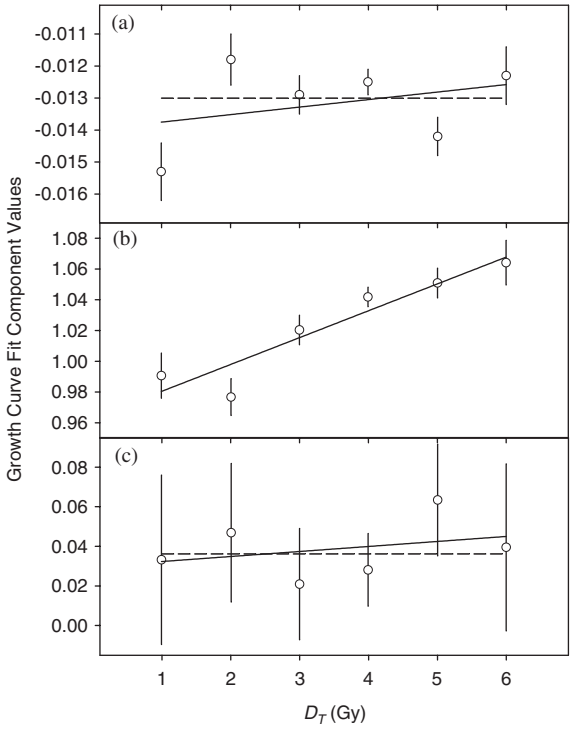


Fig. 3. Parameter values for quadratic fits to the standardised growth curves measured for sample Aber/42/J9 using test doses between 1 and 6 Gy (Fig. 2). Parameter a is plotted in a, b in b, and c in c (Eq. (2)). Linear fits to change in the parameters with D_T are plotted as solid lines, they have R^2 values of 0.11, 0.89, 0.10 for a, b, and c, respectively. The weighted means of a and c are indicated by dashed lines.

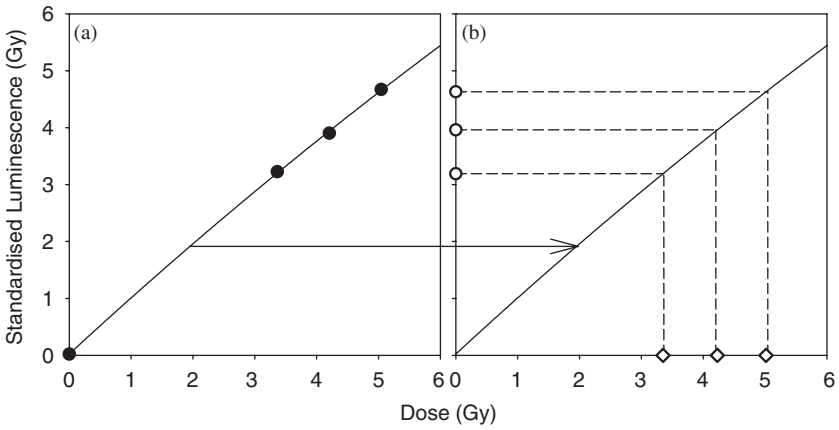


Fig. 4. Derivation of "D_{eR}" values from recycled regenerative points, for one 8 mm diameter aliquot from sample Aber/34/2108a. Measured using SAR, with regenerative doses: 3.2, 4.1, 5.0, 3.2, 4.1, 5.0 Gy. (a) Curve is fitted to the first three regenerative dose responses (●). (b) Curve is used to derive D_e values (◊) for the second three regenerative responses (○), treated as unknowns. The second set of regenerative doses were made equal to the first set used for fitting, so that recycling ratios could be checked in each case.

$$b = 0.018(\pm 0.003)D_T + 0.96(\pm 0.01), \quad (4)$$

$$c = 0.036 \pm 0.006. \quad (5)$$

By substituting Eqs. (3)–(5) into Eq. (2), I can be calculated directly for a given combination of test dose and regenerative dose

$$I = -0.013D^2 + (0.018D_T + 0.96)D + 0.036. \quad (6)$$

D_e can then be predicted by substituting the standardised luminescence signal of the natural sample into Eq. (6) and solving for D. This equation has been derived from measurements on a single sample (Aber/42/J9). Is it then applicable to other samples, and does it provide more accurate and/or more precise estimates of D_e than other approximations of luminescence growth characteristics?

4. Testing the equation for standardised growth on large aliquots and comparison with other methods of D_e determination

The extent to which Eq. (6), derived from measurements of sample Aber/42/J9, is applicable to other samples was tested using sample Aber/34/2108a. An experiment was devised that would allow comparison of four different methods for determination of D_e using the data from a single set of measurements. The first method is to construct a conventional SAR growth curve (Murray and Wintle, 2000) for each aliquot. The second is to take the ratio of the sensitivity corrected signal from the natural to that of a single regenerative point. This has been used previously to speed up the determination of D_e distributions for young samples (e.g. Olley et al., 1999) and if the growth curve is linear or the D_e of the sample is close to the regenerative dose used, it is expected to produce accurate results. The third method uses the sensitivity corrected value of the natural signal and the standardised growth curve equation of Roberts and Duller (2004), whilst the fourth uses the standardised growth curve in Eq. (6) of this paper.

The experiment was undertaken on 24 large (8 mm diameter) aliquots, to minimise random uncertainties, so that any systematic effects could more clearly be observed. The SAR procedure was used with test doses of 2.5 Gy: the natural D_e of the sample was ~ 4.8 Gy. However, the natural D_e is not known independently, and was expected to show significant scatter. Therefore, three regeneration doses were measured twice for each aliquot: 3.2, 4.1, 5, 3.2, 4.1, 5 Gy. The second set of three provided values of L_X/T_X which could be treated as though they were measurements of unknown doses, and a value of "equivalent dose" calculated for each one (Fig. 4). This value is here termed "D_{eR}". By making two sets of regenerative measurements one can avoid circularity, since the first set of data are used to construct the SAR growth curve for each aliquot. This type of approach is similar to the use of recycled points in the assessment of reproducibility by Duller et al. (2000), and the replacement plots of Bailey et al. (2003).

Calculation of D_{eR} values is of limited utility for assessing the accuracy of natural D_e determinations, since they use only data taken from within the regenerative sequence of measurements. However, in the present case the use of repeat regenerative datapoints has the distinct advantage that the dose received by an aliquot is very precisely known, and the reproducibility of the aliquot's response to each dose can be directly assessed by calculating a "recycling ratio" (Murray and Wintle, 2000). Thus, the relative accuracy and precision of determinations made using different growth curve types can be assessed, after accounting for instrument reproducibility and in the absence of natural variability.

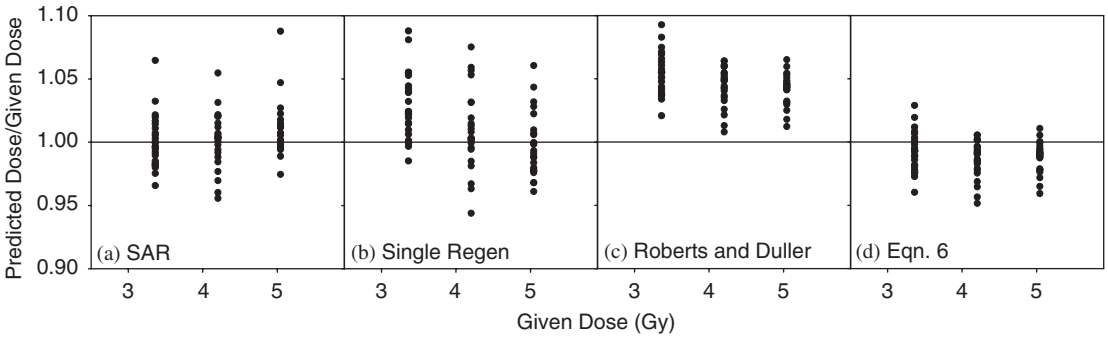


Fig. 5. Predicted dose (D_{eR}) as a fraction of the given dose for each of 24 large aliquots (8 mm diameter) from sample Aber/34/2108a. (a) D_{eR} calculated using SAR. For each aliquot the regenerative response has been fitted using a weighted exponential curve. (b) D_{eR} calculated using the response of each aliquot to a regenerative dose of 4.2 Gy. (c) D_{eR} calculated using the standardised growth curve of Roberts and Duller (2004). (d) D_{eR} calculated using Eq. (6).

The D_{eR} values calculated for each aliquot are shown in Fig. 5 as a fraction of the given doses. As expected, use of a complete SAR growth curve appeared to predict the given doses most consistently (Fig. 5(a)), while use of the ratio to the 4.2 Gy regeneration dose (Fig. 5(b)) gave results closest to unity for the 4.2 Gy dose, tended to overestimate at the lower dose and underestimate at the higher dose. There was some systematic deviation apparent when the standardised growth curves were used (Fig. 5(c and d)). However, use of the standardised curves appeared to result in the least scatter of all the approaches.

Using the weighted mean of the calculated D_{eR} for all 24 large aliquots illustrates systematic trends with dose more clearly than the individual data (Fig. 6 (a)). The SAR method yielded predicted doses within 1.0% of the given doses. D_{eR} calculated relative to the regenerative point at 4.2 Gy predicted the given doses within 2.7%, but there was a slight trend for doses lower than 4.2 Gy to be overestimated, and for those above it to be underestimated. Use of the standardised growth equation of Roberts and Duller (2004) overestimated the given dose by between 4.2% and 5.4%. Use of Eq. (6) underestimated the given dose by between 0.8% and 1.3%, with no apparent trend with dose. The weighted mean recycling ratios at each dose point were all within 1% of unity. The small deviations of the predictions made using exponential fitting of the SAR data might thus be explained by uncertainty in the dose response being recovered, rather than deviation in the fits.

To assess scatter about the weighted mean D_{eR} values, the "external error" on the weighted mean was used (α_e)

$$\alpha_e^2 = \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2 / se_i^2}{(n-1)\sum_{i=1}^{n} 1/se_i^2}, \quad (7)$$

where x_i are the individual dose estimates, $\bar{x}$ is the weighted mean, se_i are the individual standard error estimates on the dose estimates x_i, and n is the total number of measurements. α_e combines information on both the individual error estimates, and the deviation from the weighted mean: after Thomsen et al. (2003), although it should be noted that in their equation α_e actually represents expected variance.

When a complete SAR growth curve was used, expected deviation in the weighted mean D_{eR} values was between 0.11% and 0.23% of the given doses (Fig. 6(b)). Using a single regenerative point it was higher, between 0.51% and 0.60%. Use of the standardised growth curves resulted in values higher than for the SAR method, but lower than for the single regenerative point. Both standardised curves yielded expected deviations of between 0.25% and 0.32% of given dose, which reduced as the given dose increased.

These results indicate that exponential fitting of the SAR regenerative responses from individual large aliquots would provide the most precise and accurate assessment of growth for each aliquot, and hence D_e. However, they also indicate that these levels of precision and accuracy could be closely approximated using a standardised growth curve.

5. Testing the equation for standardised growth on small aliquots

Both statistical and behavioural variability in growth curve shape are expected to increase as aliquot size is reduced, but for samples where there is the possibility of a mixture of grains with different apparent doses (such as at Old Scatness), small aliquots are required to accurately measure D_e distributions. Therefore, aliquots in which the sample only covered a 1.75 mm diameter circle in the centre of the disc were prepared for samples Aber/34/2108a and Aber/41/K23. The aliquots were loaded very lightly with material, such that an average of 22 grains was counted on a subset of six aliquots taken from Aber/41/K23. It was therefore expected that very few grains would contribute the majority of the signal on each aliquot (Duller et al., 2000),

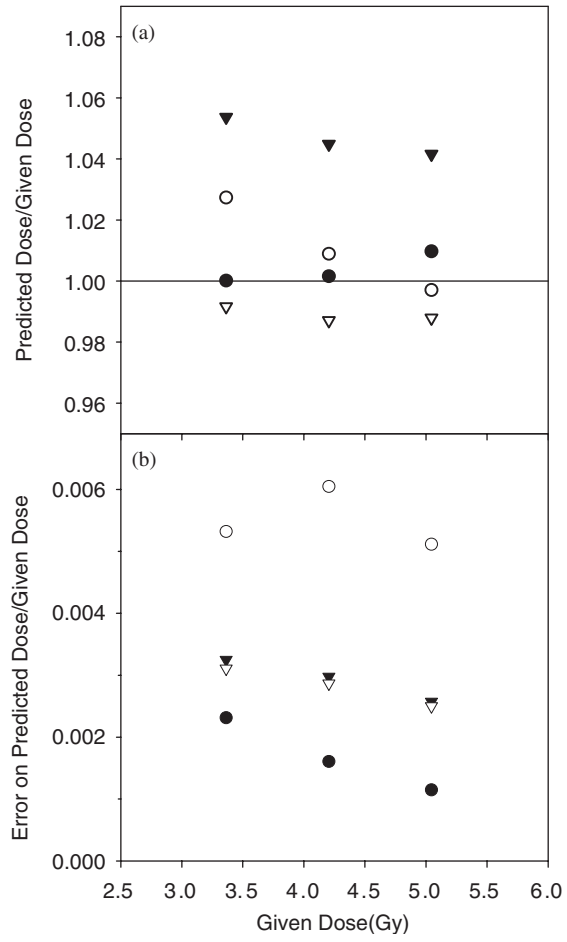


Fig. 6. (a) Weighted mean calculated dose (D_{eR}) as a fraction of the given dose for 24 large aliquots (8 mm diameter) from sample Aber/34/2108a. D_{eR} was calculated using weighted exponential fits to the regenerative responses of each aliquot (●), using the response of each aliquot to a regenerative dose of 4.2 Gy (◯), using the standardised growth curve of Roberts and Duller (2004) (▼), and using Eq. (6) (▽). (b) Expected deviation of the predicted doses (D_{eR}) as a fraction of the given dose, calculated as the "external error on the weighted mean" (Eq. (7)).

so that any real differences in the growth curve shapes of these grains would still be evident.

Twenty-four aliquots of Aber/34/2108a were measured in the same way as the 8 mm diameter aliquots of the same sample described above (Section 4). Response to the 2.5 Gy test dose, following measurement of the natural signal, was between 4 and 170 cps for the small aliquots (signal integrated over the first 4 s, background subtracted), compared to between 1530 and 3100 cps from the large aliquots. The results were also analysed in the same way as the large aliquot data, except that saturating exponential curves could not be fitted to the scattered SAR data obtained from many of the small aliquots. Weighted linear fitting ($w = 1/\mathrm{se}_i^2$) was used instead: the weighting reduced the effects of often poorly defined outlying data points.

The D_{eR} values obtained for the individual small aliquots (Fig. 7) illustrate that scatter was an order of magnitude higher than for the large aliquots, such that any systematic trends were no longer apparent. However, some of the most outlying D_{eR} values obtained using both linear fitting and a single regenerative point (Fig. 7(a, b)), were not present when standardised growth curves were used (Fig. 7(c, d)). Apparently, even the weighted fitting was not sufficient to remove the effects of variability in the regenerative responses of some aliquots. These aliquots could have been removed from the analysis, on the basis of low signal levels, for example, but they were retained for the purposes of examining the scatter in D_e obtained using the different approaches to defining the growth curve. This indicated that the standardised growth curve approach may allow more data to be retained for the analysis of young, dim samples than is possible when taking a more conventional SAR approach. However, even with the scattered (but poorly known) points included, expected deviations of the weighted mean D_{eR} values were similar for each of the D_e determination methods (1.7–2.3%, Fig. 8(b)). Within this small range, the standardised growth curve approaches often yielded slightly lower values at a given dose level.

For sample Aber/41/K23, 48 small aliquots were measured, using a wider range of regeneration doses (0.5, 1, 2, 4, 5, 8, and 16 Gy, Fig. 9). Unlike the previous experiments, this sequence of regeneration doses was only applied once, and so to avoid circularity, no growth curve was fitted to calculate D_{eR} as would be required using a conventional SAR approach. Instead, comparisons were made against a single regenerative dose point, against the standardised growth curve equation of Roberts and Duller (2004) and with Eq. (6), to determine D_{eR}. Use of the single regenerative point at 5 Gy resulted in overestimation at low doses, and underestimation at high doses, but values were within errors close to 5 Gy, and within ~ 5% of the given doses between 2 and 8 Gy. The equation of Roberts and Duller (2004) yielded results within 5% of all the given doses, and was better as dose increased to around 16 Gy. However, use of Eq. (6) consistently underestimated given dose by ~ 5% (Fig. 9(a)). Levels of scatter in the predicted doses were ~ 1.5% for each growth curve type at given doses above 2 Gy, and increased at lower doses (Fig. 9(b)). Use of the single regenerative point yielded slightly lower levels of scatter than the two standardised growth approaches, but at this level the difference may depend on the measure used (in this case the, "external error on the weighted mean" (α_e, Eq. (7)).

Based on large aliquots to indicate systematic deviations (Fig. 6(a)), and small aliquots to indicate levels of scatter (Figs. 8(b) and 9), the use of Eq. (6) would result in systematic deviations below levels of expected scatter. Furthermore, these would themselves be of a similar or lower magnitude to those derived from weighted fits to regenerative data (it should be noted that for the small aliquot results the

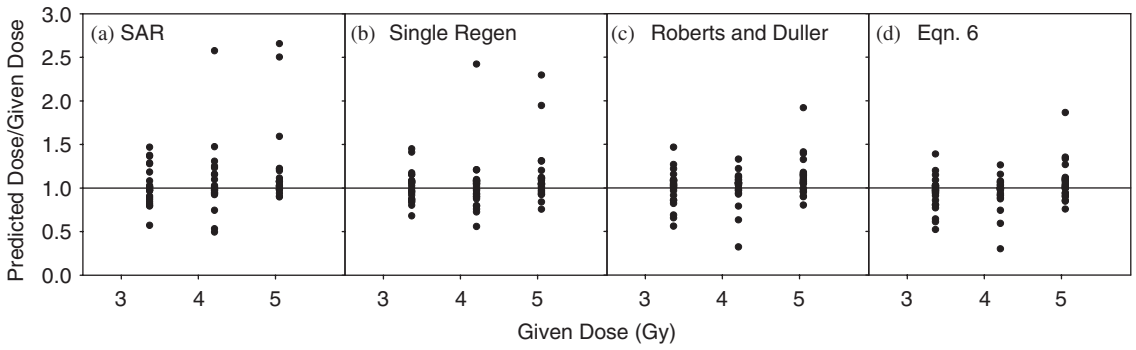
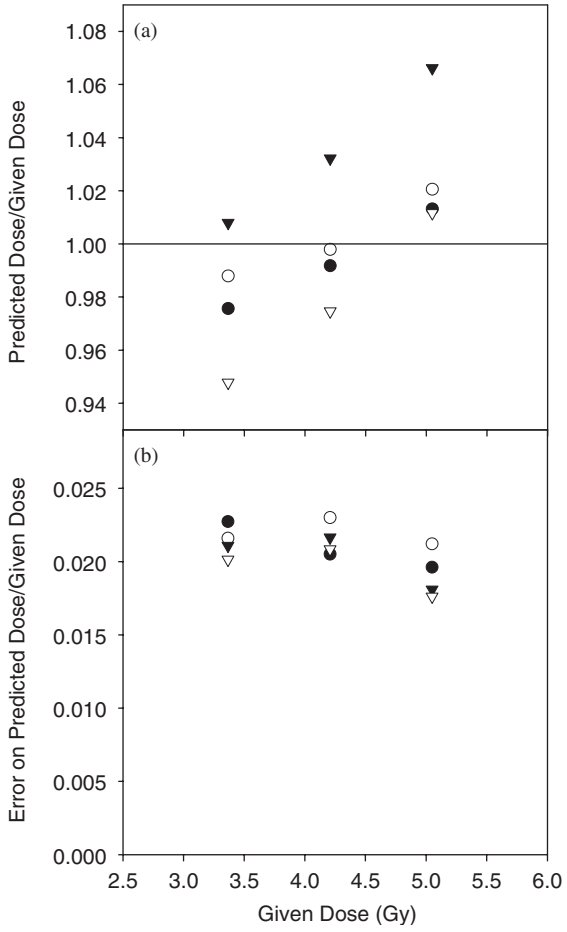


Fig. 7. Predicted dose (D_{eR}) as a fraction of the given dose for each of 24 small aliquots (1.75 mm diameter) from sample Aber/34/2108a. (a) D_{eR} calculated using weighted exponential fits to the regenerative responses of each aliquot. (b) D_{eR} calculated using the response of each aliquot to a regenerative dose of 4.2 Gy. (c) D_{eR} calculated using the standardised growth curve of Roberts and Duller (2004). (d) D_{eR} calculated using Eq. (6).



use of non-weighted fits would result in very highly scattered results). However, despite having been measured on small aliquots, the consistent ~ 5% different between observed and predicted dose for sample K23 (Fig. 9(a)) indicated that Eq. (6) might be improved. Eq. (6) was derived from measurements on 24 aliquots of one sample only, so there was clearly potential to improve it by comparison with results from large numbers of aliquots from large numbers of other samples.

6. Refinement and application of the equation for standardised growth using a short SAR measurement protocol

Twenty-two samples from the Old Scatness Broch and Sumburgh Hotel Gardens sites were measured, using a single regenerative dose point close to the expected D_e value of each sample. Measurement in this way took longer than the minimum of L_N and T_N required for D_e determination using a standardised growth curve, but it allowed a value of D_{eR} predicted using Eq. (6) to be compared with the value of the regeneration dose given to each sample, thus providing a check on the appropriateness of the standardised growth curve employed. It should be noted that the 5 Gy regenerative data from sample Aber/41/K23 (above) were included in this analysis, as well as 8 Gy regenerative data from a

Fig. 8. (a) Weighted mean predicted dose (D_{eR}) as a fraction of the given dose for 24 small aliquots (1.75 mm diameter) from sample Aber/34/2108a. D_{eR} was calculated using weighted linear fits to the regenerative responses of each aliquot (●), using the responses of each aliquot to a regenerative dose of 4.2 Gy (◯), using the standardised growth curve of Roberts and Duller (2004) (▼), and using Eq. (6) (▽). (b) Expected deviation of the predicted doses (D_{eR}) as a fraction of the given dose, calculated as the "external error on the weighted mean" (Eq. (7)).

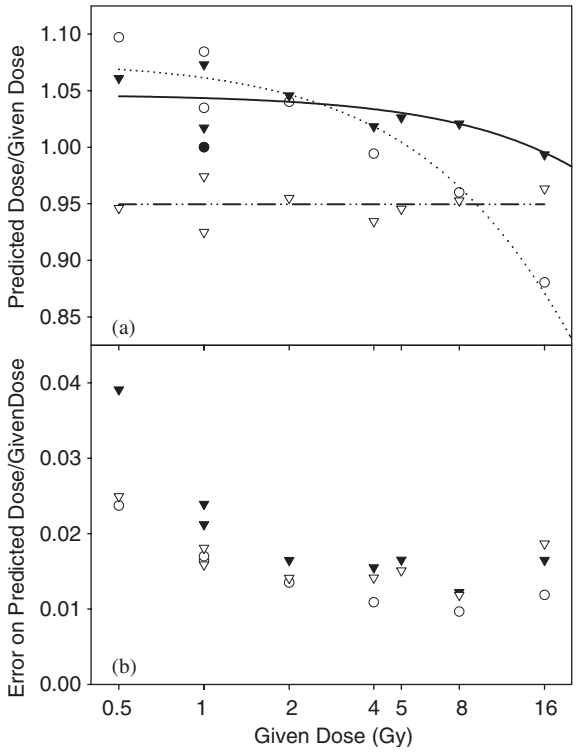


Fig. 9. (a) Weighted mean predicted dose (D_{eR}) as a fraction of the given dose for 48 small aliquots (1.75 mm diameter) from sample Aber/41/K23. D_{eR} was calculated using the response of each aliquot to a regenerative dose of 5 Gy (◯), using the standardised growth curve of Roberts and Duller (2004) (▼), and using Eq. (6) (▽). Note logged given dose axis. (b) Expected deviation of the predicted doses (D_{eR}) as a fraction of the given dose, calculated as the "external error on the weighted mean" (Eq. (7)).

new set of 48 small aliquots of sample Aber/42/J9 (the sample used to derive Eq. (6)). The other 20 samples were taken from different layers down each of two of the sections sampled at Old Scatness.

As with the larger datasets for samples Aber/34/2108a and Aber/41/K23 (above), D_{eR} values predicted using Eq. (6) commonly underestimated the given dose, albeit only by a few percent (Fig. 10(a)). This was also the case for the new aliquots of Aber/42/J9. An alternative way of viewing this

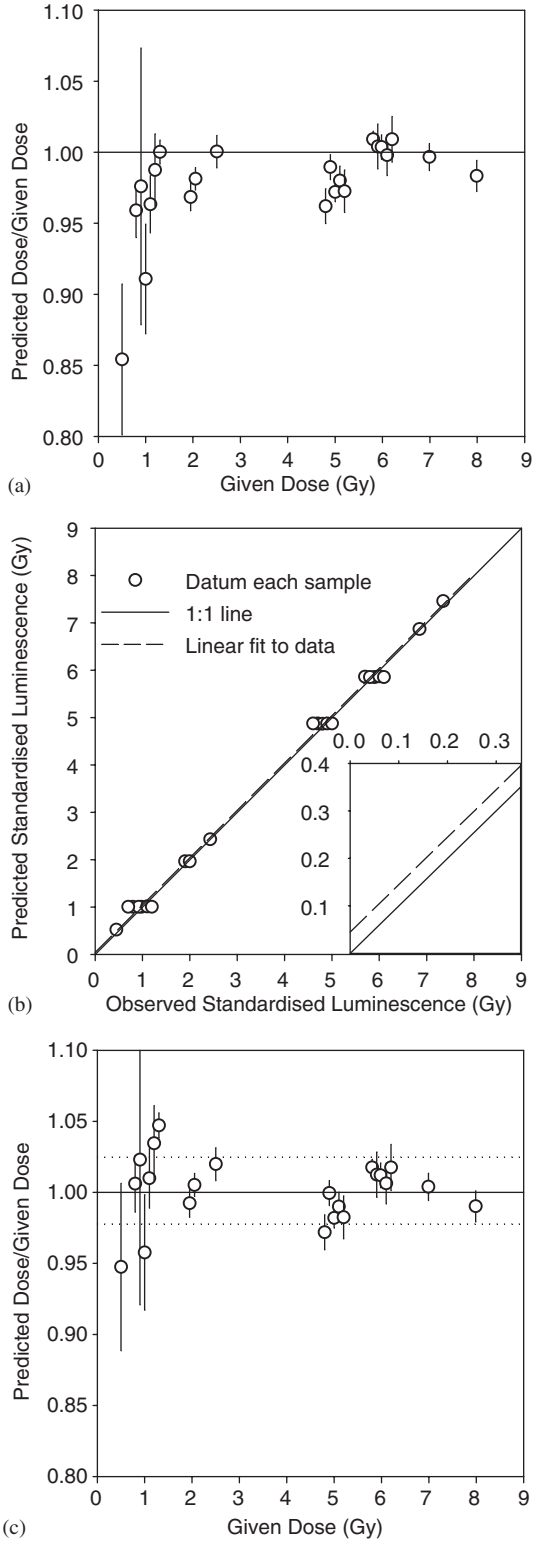


Fig. 10. (a) Weighted mean predicted dose (D_{eR} calculated using Eq. (6)) as a fraction of the given dose for between 24 and 48 aliquots from each of 22 different samples from Old Scatness Broch and Sumburgh Hotel Gardens. (b) Standardised luminescence predicted using Eq. (6), versus weighted mean observed standardised luminescence for each sample. Note that data in groups have been spread along the *x*-axis so that individual symbols are visible. (c) Weighted mean predicted dose as a fraction of the given dose for each sample (D_{eR} calculated using Eq. (10)).

is that Eq. (6) overestimates the standardised luminescence response to a given dose. This can be examined by using Eq. (6) to predict the standardised luminescence response ($L_X/T_X D_T$) that would be expected from a given dose, and comparing this with the observed standardised luminescence (Fig. 10(b)). The values appeared very similar (i.e. close to the 1:1 line), but a linear fit was made to better describe the relationship between them:

$$\text{Predicted} = 0.999(\pm 0.006)\text{Observed} + 0.04(\pm 0.02). \quad (8)$$

The gradient of the fit was within errors of one, indicating that there was no significant divergence of the predictions from the observed values in the range 0–8 Gy. However, there was a small, but not insignificant, intercept of 0.04 (±0.02) Gy (Fig. 10(b inset)). A value of 0.04 Gy would constitute a significant percentage of D_e values much below 1 Gy. Subtraction of this constant from Eq. (6) gives

$$c = -0.01 \pm 0.02, \quad (9)$$

$$I = -0.013D^2 + (0.018D_T + 0.96)D - 0.01. \quad (10)$$

The intercept c was thus made consistent with zero, which is reassuring since it means that at zero dose Eq. (10) predicts a luminescence signal well within errors of zero.

The weighted means of doses predicted using Eq. (10) were within 5% of the given doses for all the 22 samples (Fig. 10(c)). Subtraction of the offset from Eq. (6) meant that the average of the predictions over all the samples was equal to the average observed values (1:1 line, Fig. 10(c)). There was therefore no systematic deviation of the predictions over all the 22 samples, but scatter in the results from individual samples was ±2.4% in predictions of D_e made using Eq. (10).

7. Conclusions

The shape of the standardised OSL growth curve measured from quartz changes systematically with test dose, even between relatively low test doses in the range 1–6 Gy. Change in growth curve shape was accounted for by fitting the dependence of initial gradient on test dose. The use of quadratic fits, rather than saturating exponential fits, aided the close fitting of growth curve shape and precise determination of fit parameters and their variation with test dose. This is because the individual parameters of the quadratic equation directly and individually relate to curve shape.

This study was aimed at the dating of young (late Holocene) samples, so precise fitting at low doses was the most important consideration, and the unsuitability of the quadratic form for fitting at high doses was not a concern here. In a study focussing only on larger doses it should be possible to account for changes in saturating exponential fit parameters with test dose, since better precision in the parameter estimates will be obtained from fits to larger ranges of doses. However, the present study indicates that this will be to the detriment of accuracy for low doses, and in any case there is likely to be less pressure to vary the level of the test dose used in measurements on samples expected to contain large natural doses.

Use of the quadratic standardised growth curve on very small aliquots yielded levels of precision similar to those obtained from weighted fitting of regenerative data. Statistical variability in growth curve shape, not present when applying a standardised growth curve, therefore contributed similarly to expected error as did genuine differences in growth curve shape between aliquots (grains), which would not be present when fitting SAR data. This observation indicates that contrary to initial concerns (Roberts and Duller, 2004), it may be fruitful to pursue the application of standardised growth curves to single grain datasets, even where differences in growth curve shape between aliquots would be most severe.

Plots of individual recovered regenerative points versus their given doses indicated a reduction in outliers when using standardised growth curves. However, using the "external error" on the weighted mean (Eq. (7)) to assess spread reduced the effect of the poorly known outliers in any case. The quadratic standardised growth curve as initially defined (Eq. (6)), exhibited systematic deviations of up to 5%. These were consistent for a particular sample or dataset as the given dose was increased, but were averaged out by comparing results from 22 different samples (Eq. (10)). The comparison indicated inter-sample variability of ±2.4% in the accuracy of the standardised growth curve, and thus in the weighted mean growth curve shapes of each sample.

One potential approach to the use of a standardised growth curve that was pursued in the present work is to define the curve (e.g. Eq. (6)), but then measure a single regenerative response from each aliquot as well as the natural response. The single regenerative responses can be averaged to check that the standardised growth curve is accurate, and if it is not they may be used either to refine the standardised growth equation (e.g. Eq. (10)), or simply to calculate D_e proportional to the dose response of each aliquot. The present study has demonstrated that small but significant improvements in dose recovery can be obtained in this manner.

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