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A test of the single aliquot method of equivalent dose determination for feldspar stimulated by green light

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Introduction

Duller (1991) has described an attractive single aliquot additive dose method of equivalent dose determination applicable to feldspar stimulated by infra red. His account addressed various of the potential pitfalls in a single aliquot approach and illustrated the success of the method by comparing the equivalent dose determined by the single aliquot method with that determined by other methods for three different samples. For two of the three samples all equivalent dose values agreed within the quoted standard deviations (about ±8%) while for the other sample the various methods showed a greater spread. The question addressed here is whether the same method is successful in the case of feldspar stimulation by green light. The test undertaken was to look closely at whether the single aliquot method reproduces the same form of relationship between luminescence and dose as a conventional multiple aliquot method.

The measurements

A green light emitting diode system with peak emission at 565 nm was used for stimulation (Galloway, 1992; 1993) while a combination of HA3, BG39, UG11, 7-59 and 7-60 filters with a peak transmission at 355 nm preceded the 9635QA photomultiplier which counted the luminescence photons. Figure 1 shows an example of the time dependence of luminescence stimulated from a feldspar sample using the system.

Fine grain samples of a Norwegian microcline feldspar were prepared by sedimentation and bleached in a Honle SOL-2 solar simulator for 24 hours. Five aliquots were given a beta dose of 125 Gy and used to determine the decay in luminescence due to successive preheating at 220°C for 10 minutes and reading for 10 s exposure to the green light, Figure 2. This decay curve was used as recommended by Duller (1991) to correct each single aliquot set of additive dose measurements to produce the corrected dose response curves in Figure 3. Least squares linear fits to the data points are drawn, although inspection of the lower dose points shows that the curves are initially supralinear, as indicated by the linear fits having a negative intercept on the counts axis. A similar response is observed by conventional multiple aliquot determination, Figure 4. If the response takes precisely the same form in both methods of measurement, the ratio of the single aliquot counts/s to the multiple aliquot counts/s for each beta dose should be a constant independent of dose. Figure 5 shows for each of the single aliquot measurements this ratio plotted against the number of dose, preheat and green light exposure sequences involved in the single aliquot measurement. In each case an increase in sensitivity is indicated, detailed in Table 1 and averaging about 6% per re-use of the aliquot. The error bars in Figure 5 arise from the spread in the multiple aliquot measurements (Fig. 4). A similar increase in sensitivity is shown in all cases and so may well reflect a genuine effect.

The increase in sensitivity due to successive re-use is strikingly illustrated in Figure 6, in which pairs of corrected single aliquot measurements built up from first beta doses of 100, 200 and 400 Gy are compared with the response based only on these first dose measurements, which of course requires no correction. The data in Figure 6 indicate a sensitivity increase of about 8% per re-use of the aliquot (Table 1) although in this simple illustration the 'multiple aliquot' response is interpolated and extrapolated from only 6 aliquots.

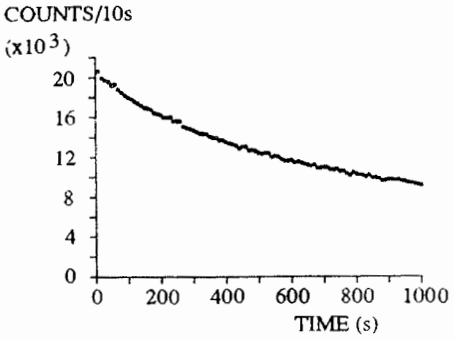

Figure 1.
An example of the time dependence of luminescence stimulated by the green LED system from a sample of microcline feldspar previously bleached in a SOL-2 solar simulator, dosed to 250 Gy and preheated at 220°C for 10 minutes.

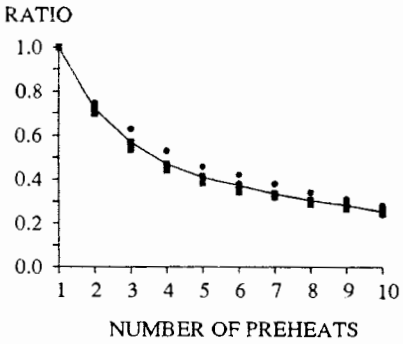

Figure 2.
The decay in luminescence signal due to successive preheating and measurement sequences. Preheating was at 220°C for 10 minutes and measurement for 10 s exposure to the green LEDs. Five samples of microcline feldspar were used, dosed to 125 Gy. The line joins the average of the 5 measurements at each successive preheat.

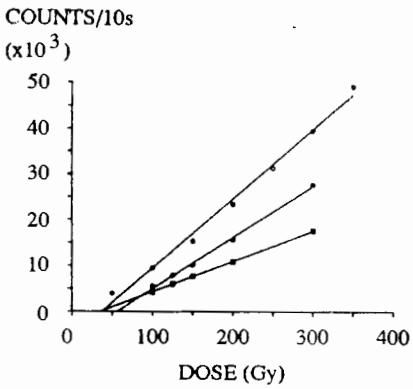

Figure 3.
Three single aliquot measurements, corrected by the method of Duller (1991), of the dose dependence of green stimulated luminescence from microcline feldspar previously bleached in a SOL-2 solar simulator. Preheating was at 220°C for 10 minutes and measurement for 10 s exposure to the green LEDs. The lines are least square linear fits to the data points.

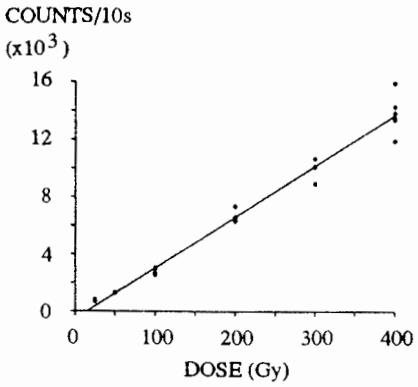

Figure 4.
Conventional multiple aliquot determination of the dose dependence of green stimulated luminescence from microcline feldspar with preheating at 220°C for 10 minutes and measurement for 10 s exposure to the green light. 23 aliquots were used. The least squares linear fit to the data points is shown.

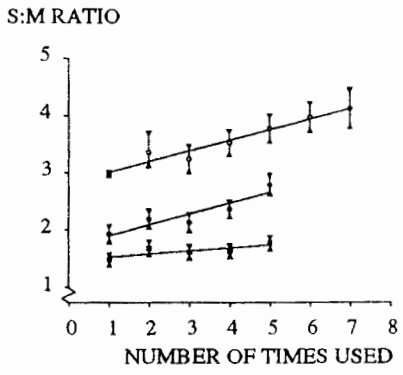

Figure 5.
Ratio of single to multiple aliquot counts plotted against the number of dose, preheat and green light for single aliquot measurements, from data in Figs. 3 & 4. Ratios >1 reflect differing quantities of deposited material between sets of aliquots. Percentage change in sensitivity per re-use of the aliquot from these plots is listed in Table 1.

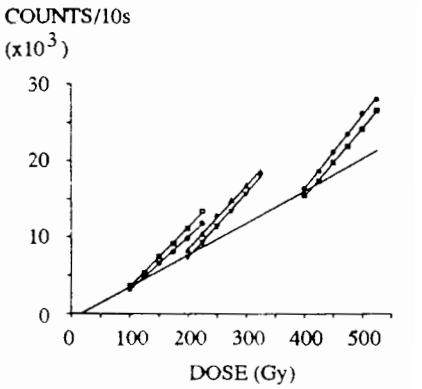

Figure 6.
Pairs of single aliquot measurements starting from three different initial doses, with the initial measurements providing the response independently of the single aliquot corrections. The corrected single aliquot responses rise above the response indicated by the initial measurements.

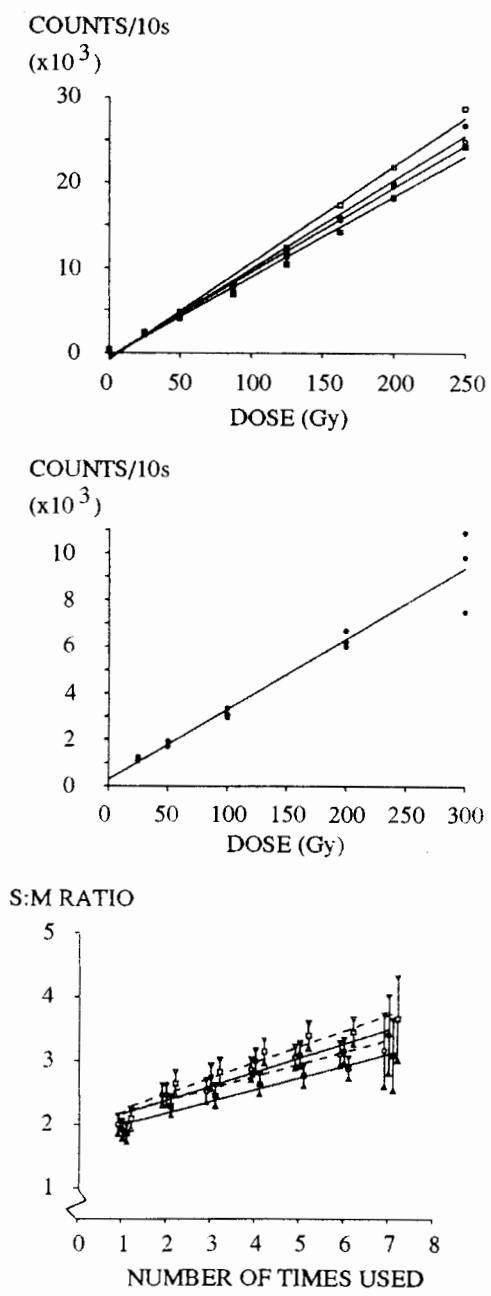

Figure 7.
Microcline feldspar bleached by heating to 500°C; (a;top) corrected response for one aliquot over the dose range 25 - 150 Gy and another over the range 100 - 500 Gy, (b; middle) conventional 18 aliquot determination of the response, (c; lower) dependence of the ratio of single to multiple aliquot response at the same beta dose on the number of times the aliquot has been used.

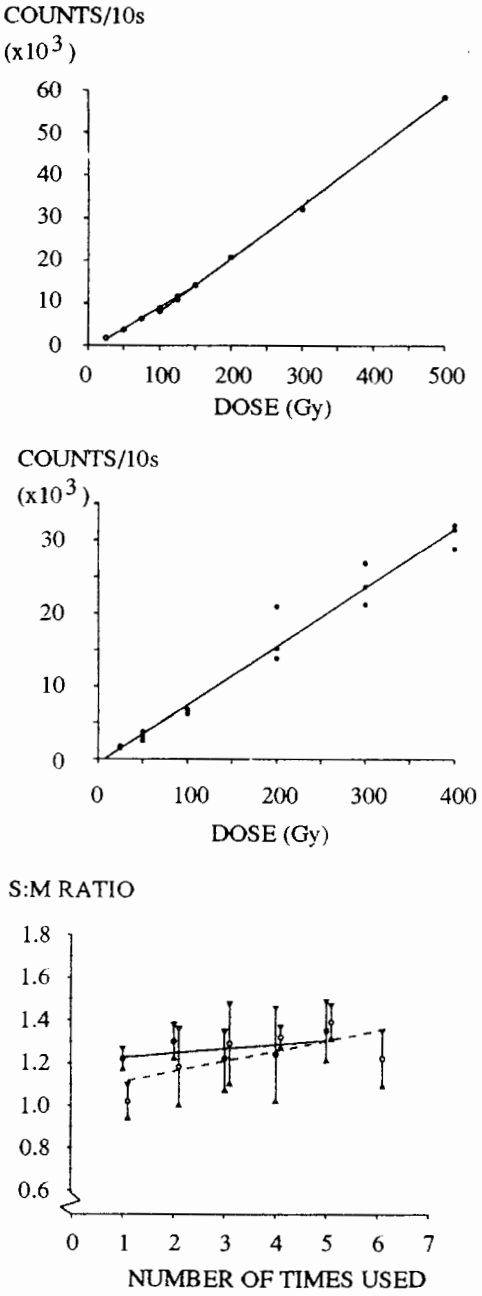

Figure 8.
Microcline feldspar bleached by daylight; (a; top) four corrected single aliquot measurements of the response, (b; middle) a conventional 15 aliquot determination of the response and (c; lower) dependence of the ratio of single to multiple aliquot response at the same beta dose on the number of times the aliquot has been used.

Some measurements were made on a set of aliquots which were bleached by heating to 500°C instead of exposure in the solar simulator. Three aliquots were used to determine the correction for re-use and corrected single aliquot determinations of the dose response over the range 25-150 Gy and 100-500 Gy are shown in Figure 7a. A conventional 18 aliquot determination of the response for comparison is shown in Figure 7b and the ratios of single to multiple aliquot response at equal beta doses are plotted in Figure 7c. The two sets of ratios in Figure 7c indicate an increase in sensitivity of about 3% per re-use of the aliquot (Table 1) but could be consistent with zero within the uncertainties indicated. This hint at a dependence of sensitivity change with re-use on the method of bleaching the sample material made it important to test samples bleached by daylight.

Samples of the same microcline feldspar were exposed to daylight for three weeks and used for similar tests with the results shown in Figure 8 and summarised in Table 1. The sensitivity change with re-use, about 10%, is comparable to the value for the material bleached in the SOL-2 solar simulator.

Finally a set of measurements were made on aliquots prepared from an orthoclase feldspar from a different source which had been bleached in daylight for three weeks, with results similar to Figure 8 as detailed in Table 1. Again a comparable, 7%, change in sensitivity per re-use was found.

Conclusions

All the corrected single aliquot measurements made on feldspar bleached in the SOL-2 or by daylight show an increase in sensitivity with re-use (Figs 5, 6 & 8c; Table 1). Although the measurement uncertainty is relatively large in each individual case, that all show an increase cannot be dismissed in this way. Thus it would seem that the attractive single aliquot method devised by Duller (1991) for equivalent dose determination by infra red stimulated luminescence from feldspar cannot be applied to green stimulation without the complication of making allowance for the change in sensitivity of the aliquot with re-use.

Table 1. Change in sensitivity per re-use of aliquots of feldspar.

Sample	microcline	microcline	microcline	microcline	orthoclase
Bleaching	SOL	SOL	500°C	daylight	daylight
Pre heat	all 220°C / 10 min				
Data in Fig.	5	6	7	8	*
% change	6	6	2	11	7
	10	6	4	10	7
	3	7		9	8
	5*	9		9	7
	4*	9			
	4*	9			
	7*				
Mean	6±2	8±2	3±2	10±2	7±2

* Not illustrated for economy of space.

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