Ancient TL

TL dating of fluvial quartz sands: a comparison of ages obtained at 325°C and 375°C

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Generally the validity of the ages determined for aeolian quartz sands using the 375°C TL peak has been accepted as providing an accurate measure of the time since the last deposition of the sediment (Gardner *et al.*, 1987, Nanson *et al.*, 1992). The reason for this acceptance is that it is felt that these sediments receive sufficient solar exposure prior to deposition to reduce the stored TL to a minimum level. Over the past number of years however there has been a growing concern that water borne sediments may not receive such an exposure and that these sediments may retain a remnant of their previously acquired TL signal (Spooner *et al.*, 1988). The retention of such a remnant would lead to an overestimation of the age of the sediment.

As a result of this concern there is an increasing move towards the use of the more easily removed TL peak occurring at 325°C in the age determination of water borne sediments. This trend however is not without its drawbacks as one must ensure that the level of light exposure during and following the sample collection is kept to an absolute minimum. This requirement is a far from desirable laboratory working condition.

Because of the large number of fluvial sedimentary studies undertaken by the laboratory and the concern regarding the validity of the results of these studies a comparison between the TL ages determined at 325°C and at 375°C was carried out. As the Wollongong practice, where possible, is to analyse most samples at a temperature of 375°C this comparison entailed re-analysis at 325°C. Also reported here are the results of tests conducted upon the laboratory lighting and its effect upon the TL stored within the sample quartz grains. It should be noted that wherever possible it has

long been the practice of the laboratory to perform corroborative dating exercises using independent dating methods (Nanson *et al.*, 1991, bin Hassan *et al.*, 1993).

The Wollongong laboratory in the main utilises the total bleach combined regenerative/additive method of TL analysis as described by Readhead (1984). Where necessary the polymineral fine grain (1-8 μm) assemblage, as described by Aitken (1985), is applied. Laboratory bleaching is achieved using a Philips 300MLU sunlamp, irradiations are performed by means of a strontium-90 plaque source and TL outputs are detected by an EMI 9635Q PM fitted with a Corning 7-59 filter. A heating rate of 5°C s^{-1} is used in the case of coarse grain work and 20°C s^{-1} for the fine grain experiments. All measurements are made under a high purity nitrogen atmosphere and each output is normalised using a second glow procedure. This is found preferable to weight normalisation as, unlike weight normalisation, it compensates for any slight difference in sample planchette colour or staining which has been found to have a marked effect upon TL output. There appears to be no systematic increase in TL sensitivity with increasing first radiation dose. Each sample aliquot is deposited upon an aluminium planchette using a volume technique which is described by Bell (1978). Sample aliquots are spread in a monolayer and adhered using a silicone spray found to be both stable at high temperatures and non-TL emitting.

The laboratory is illuminated by six single 36 watt fluorescent tubes. The outer fittings of these are covered with Cinemoid No.1 yellow plastic filter sheeting which has a sharp cut-off at 500 nm (Jensen

and Barbetti 1979). It is found preferable to cover the outer surface of the fitting rather than the tube as this prevents rapid deterioration of the filter material due to heating at the ends of the fluorescent tubes.

Table 1.

Comparison of TL ages determined at 325°C and 375°C

Coarse grain quartz: 90-125 μm

Sample Ref.	Temp. Plateau	TL Age (ka)	
		325°C (°C)	375°C
A	300-500	106±21	86.8±7.5
B	300-500	49.5±5.6	46.8±3.0
C	275-500	33.2±6.8	34.4±4.0
D	275-500	83.8±7.1	83.5±7.1
E	275-500	99.4±8.6	95.3±6.3
F	300-500	47.0±3.8	45.9±3.8
G	300-450	19.7±3.9	25.5±3.5
H	325-450	26.5±2.8	29.8±2.9
I	325-500	21.2±2.4	24.7±2.8
J	325-500	23.7±4.6	29.4±4.6
K	300-500	34.2±3.8	33.6±2.5
L	300-500	47.7±4.5	47.8±6.1
M	300-500	41.0±7.2	43.2±3.7
N	300-500	65.6±3.9	63.7±4.1
O	300-500	16.8±1.7	15.2±2.0
P	325-500	9.8±1.0	13.6±1.6
Q	300-500	19.1±1.9	18.2±1.5
R	300-450	19.8±2.1	17.9±2.5
S	300-500	3.8±0.3	4.1±0.3
T	300-500	7.0±0.9	7.1±0.6
U	300-500	1.9±0.2	1.6±0.2
V	300-500	122±7	115±7

Note:

The data shown have been taken from four widely separated study areas. Samples A-F are from Page *et al.* (1991), the remaining samples are from unpublished work.

Effect of laboratory lighting upon TL samples

In total three separate tests have been conducted over a period of seven years. These tests involved a total of 34 sample aliquots as previously described. Each sample batch was irradiated to a level of about 36 Gy and divided into two subsets. One of these was placed at laboratory bench level under the laboratory lights for a period of 24 hours while the other was stored under dark conditions. Following this procedure the TL output of the two subsets was measured and the mean TL output compared at 25°C intervals between 200 and

500°C. At 375°C there was a mean TL loss of 4.1% for the three tests conducted and at 325°C there was a mean loss of 14.5% over the 24 hour period. It should be pointed out that at all times samples are stored under dark conditions so minimising exposure to the laboratory lights. The majority of exposure takes place during the preparation stage and most of this is under liquid chemicals and in a less well lit fume hood. The Wollongong procedure does not make use of a prolonged HF etch period (Readhead 1984).

Comparison of TL ages determined at 325°C and 375°C

This comparison involved the re-analysis of 22 fluvial samples which had previously been analysed at 375°C. The initial 16 samples were selected at random by a non-TL expert and the remainder by the author in order to fill out age gaps in the data. In each case comparison between the amplitude of the natural sample TL and that of a regenerated TL glow curve. selected to give a ratio of approximately unity. provided a plateau region generally extending between 300 and 500°C. The result of this comparison is shown at Table 1 and in graphic form at Figure 1.

Discussion

As indicated at Figure 1 there is an excellent overall agreement between the two sets of TL ages determined with a least mean squares regression coefficient of 0.99 and a slope of 0.90. If the three higher age values are ignored the slope becomes 0.96. As tempting as it may be, with the limited data available. it is not suggested that the relationship between the two data sets is other than linear beyond 100 ka.

The ages determined at 375°C for samples G. H, I, J and P exceeded those computed at the lower temperature TL peak. This was particularly noticeable in the case of samples G. J and P. Samples H. I. J and P displayed temperature plateaux which did not extend below 325°C although this is probably closer to 315°C allowing for a heating rate of 5°C s⁻¹ and that the 110°C reference peak occurs at about 100°C. Examination of the glow curves for all of these samples suggests that this age difference might well be due to a mineralogy problem rather than one of
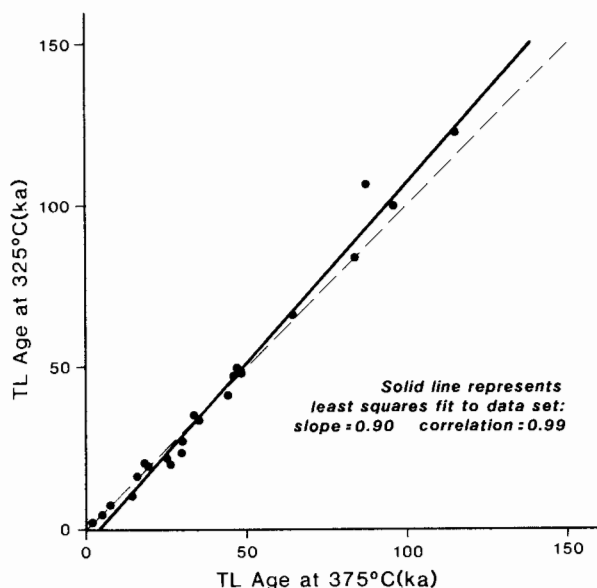
Figure 1.

Comparison of TL ages at 325 °C and 375 °C. The solid line represents the least squares fit to the data set, slope 0.90, correlation 0.99.
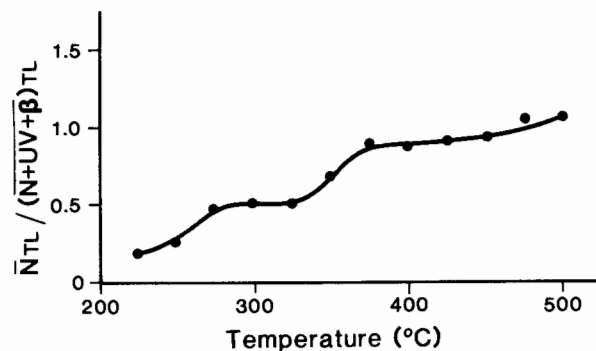

Figure 2.

Variation of natural TL/regenerated TL with temperature. Showing a two step temperature plateau relationship thought to be caused by partial re-exposure due to bioturbation. TL ages computed at 325°C and 400°C are 5.1± 0.8 ka and 9.1± 0.9 ka respectively. The associated ratio errors lie within the width of the plotted points. Site, Western Arnhem Land Plateau, N.T. (unpublished).

incomplete removal of the previously acquired TL although this possibility cannot be totally excluded. Each of the regenerated and second TL glow curves exhibited by these samples showed evidence of the presence of strong TL interference at around 200°C. This effect was not consistent between sample aliquots and not at all evident from the natural TL glow curves suggesting a relatively short lifetime. This interference has a much greater effect upon the lower 325°C regenerated TL glow curves as compared to the 375°C TL outputs. Thus the 325°C TL ages are thought to represent underestimates as the natural TL signal represents a lesser proportion of the enhanced TL peak. This effect could be due to the presence of inclusions within the quartz grains as reported by Fragoulis and Readhead (1991). The possibility of the problem being caused by interference from other TL sensitive minerals is further supported in that each of the samples in question originated from the same site.

Unlike the TL glow curves of Spooner et al. (1988) the majority of samples from this study exhibit dominant TL peaks in the 375°C region and this is found to be more often than not the normal situation. The exceptions to this were samples C and E which displayed dominant 325°C TL maxima. The criterion for the selection of a particular analysis temperature at the Wollongong laboratory is one of judging each sample upon its merits and the rate of change of the TL signal with temperature is a consideration in this choice. Where possible a point at which a slow change is taking place is preferred to one of rapid change. The existence of an extended natural/regenerated TL versus temperature plateau region is of prime importance.

Smith et al. (1985) recognised that, in certain circumstances, it is possible for a sediment to exhibit a two-step equivalent dose plateau. Partial exposure of the sediment during transport is thought to be sufficient

to release those electrons trapped at lower energy levels but insufficient to release those stored at deeper levels. As is shown by Prescott (1985) a single plateau is not necessarily an indication of complete resetting of previously acquired TL over the entire temperature range. Figure 2, taken from work currently under way in this laboratory, shows a two-step plateau which is attributed to partial re-exposure of the sediment by ants (*Aphaenogaster* sp.) which are reported to be extremely active in the area (Bowman, pers. comm.). Further work is under way to verify this.

Conclusion

The objective of this exercise was to verify that, given sufficient care, it is possible to accurately determine the depositional age of water borne sediments by means of the 375°C quartz TL peak. The results of this study would seem to achieve this purpose and indicate that the level of sample exposure to the laboratory lighting is insufficient to have a detrimental effect on either the 325°C or the 375°C TL energy levels. Of paramount importance in the analysis phase of the sample is the existence of a lengthy temperature plateau region and that each sample should be judged upon its own merits rather than to a preconceived formulation.

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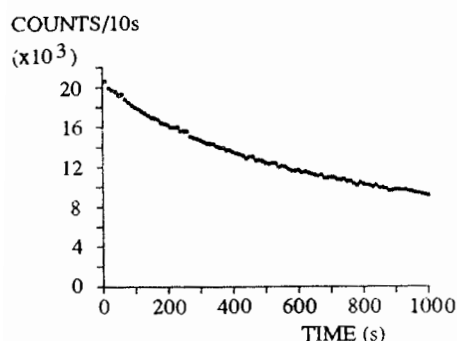
Figure 1.
An example of the time dependence of luminescence stimulated by the green LED system from a sample of microcline feldspar previously bleached in a SOL-2 solar simulator, dosed to 250 Gy and preheated at 220 °C for 10 minutes.
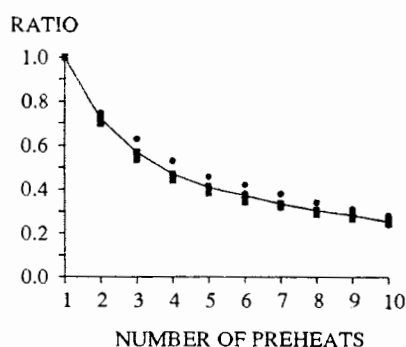

Figure 2.
The decay in luminescence signal due to successive preheating and measurement sequences. Preheating was at 220 °C for 10 minutes and measurement for 10 s exposure to the green LEDs. Five samples of microcline feldspar were used, dosed to 125 Gy. The line joins the average of the 5 measurements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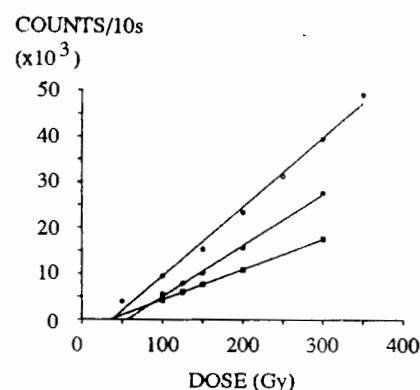

Figure 3.
Three single aliquot measurements, corrected by the method of Duller (1991), of the dose dependence of green stimulated luminescence from microcline feldspar previously bleached in a SOL-2 solar simulator. Preheating was at 220 °C for 10 minutes and measurement for 10 s exposure to the green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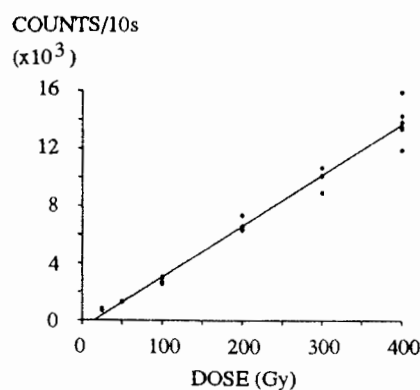

Figure 4.
Conventional multiple aliquot determination of the dose dependence of green stimulated luminescence from microcline feldspar with preheating at 220 °C for 10 minutes and measurement for 10 s exposure to the green light. 23 aliquots were used. The least squares linear fit to the data points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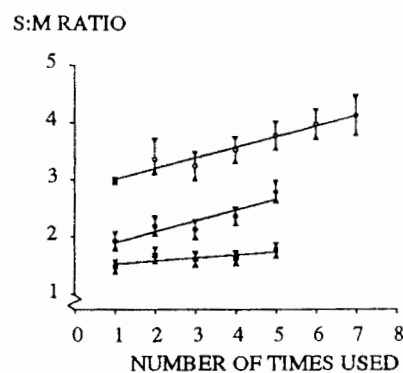

Figure 5.
Ratio of single to multiple aliquot counts plotted against the number of dose, preheat and green light for single aliquot measurements, from data in Figs. 3 & 4. Ratios >1 reflect differing quantities of deposited material between sets of aliquots. Percentage change in sensitivity per re-use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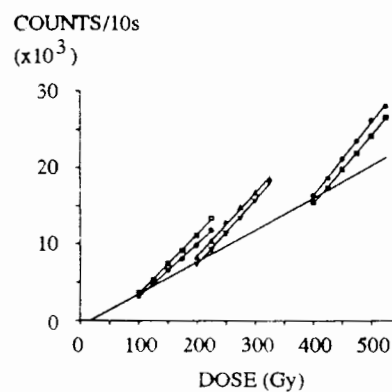

Figure 6.
Pairs of single aliquot measurements starting from three different initial doses, with the initial measurements providing the response independently of the single aliquot corrections. The corrected single aliquot responses rise above the response indicated by the initial measurements.
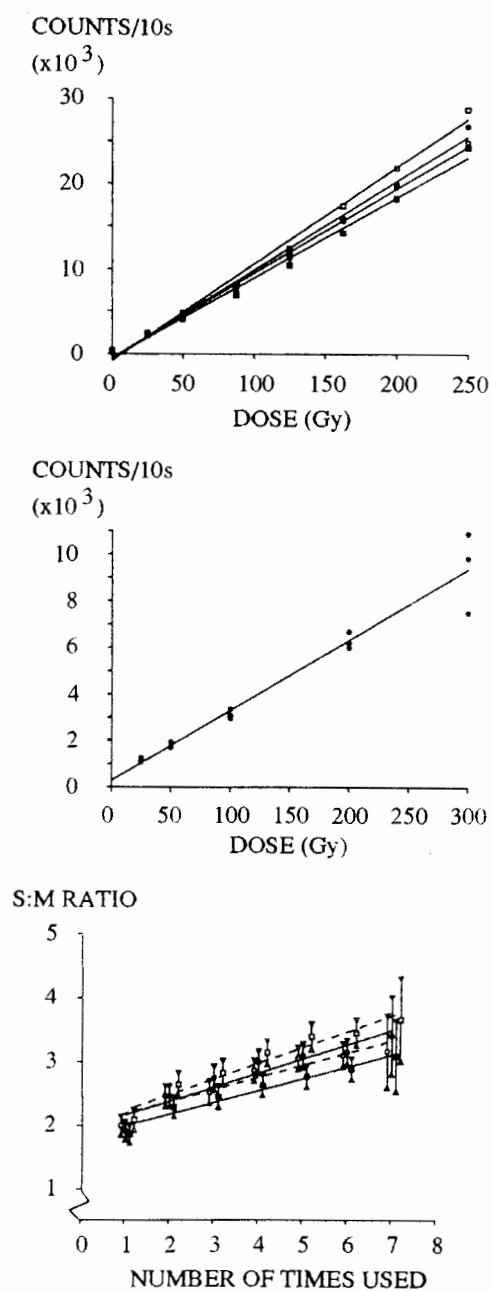
Figure 7.

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.
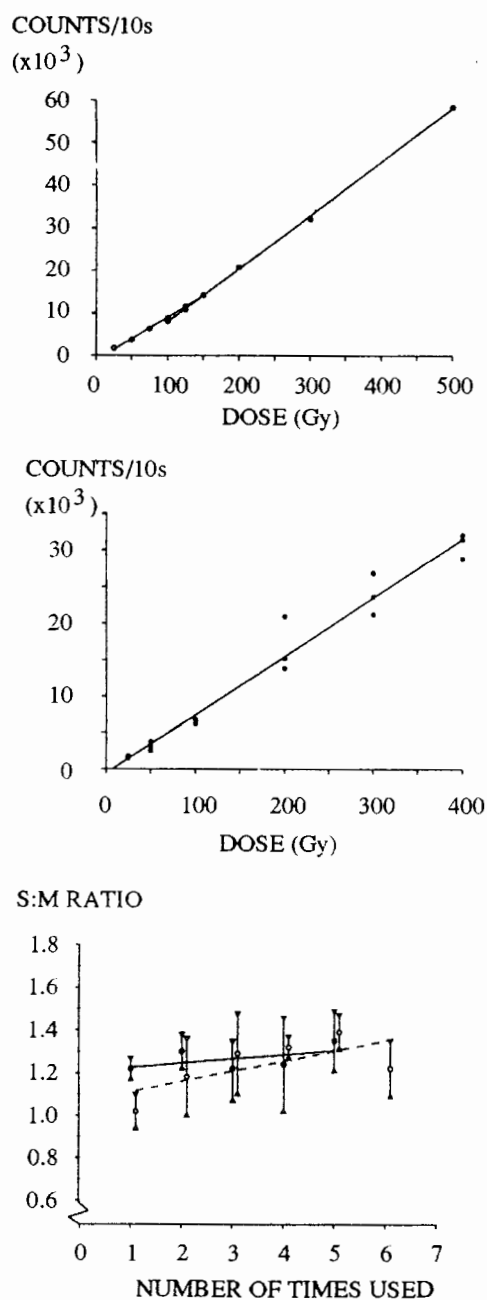
Figure 8.

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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Tests of luminescence dating making use of paleomagnetic reversals

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Introduction

Since the beginning of sediment dating the necessity of testing techniques on samples of known age has been recognized. The purpose of this note is to summarize the results obtained from samples independently dated making use of palaeomagnetic field reversals for which our tests have been successful.

Samples are rare for which a sufficient light exposure has occurred at the time of deposition and for which ages over 60,000 years are well established, due to the shortage of suitable dating techniques beyond the range of radiocarbon dating. One can make some use of the known time scale of climatic variations; however the best clear time marker is the reversal of the direction of the Earth's magnetic field, and hence the magnetization of sediments, that occurred at 0.78 Ma. This marker is referred to as the Brunhes-Matuyama (or B/M) boundary as it is the dividing point between the period of present normal polarity (Brunhes epoch) and the last major period of reversed polarity (Matuyama epoch) of the geomagnetic field. The reversed Matuyama period occurred from 2.60 to 0.78 Ma, except for 3 periods of normal polarity, the most recent of which was from 1.05 to 0.98 Ma which is called the Jaramillo event (the dates quoted here are from Cande and Kent (1992).

When making use of the Brunhes-Matuyama, or any other reversal, one has to establish its identity and take care that one does not confuse it with a reversal associated with a short polarity excursion which may only be found locally. Thus a single observation of reversed polarity is quite inadequate for identification;

a suite of observations, such as can be made in deep ocean sediments, or in continuous loess deposits, provides the required confidence. Alternatively, as in the present paper, reliable dates on associated material may help with establishment of the identity of the reversal.

The samples on which we are reporting come from the stranded dune sequence of south-east South Australia and from a peat deposit. The dune sequence has been described in detail elsewhere (Huntley *et al.* 1993a and references therein). Here we shall simply note that the dunes can be matched to high sea-stands and that all the dunes are normally magnetized except for the East Naracoorte dune which is reversed (Idnurm and Cook, 1980). This places the B/M reversal between the West Naracoorte and East Naracoorte dunes. Detailed matching to the $\delta^{18}O$ curve leads to an age of 0.69 or 0.78 Ma for West Naracoorte, and of 0.95 Ma for East Naracoorte (these differ from our earlier figures as the time scale prior to 0.62 Ma has since been revised; Shackleton *et al.* 1990, Spell and McDougall, 1992).

The Salmon Springs peat is part of a non-glacial sequence of (from bottom to top) volcanic ash, silt and peat located in northwestern Washington, U.S.A. The lower part of the silt layer has reversed magnetic polarity whereas the upper part has normal polarity, indicating that a palaeomagnetic reversal occurred during deposition of the silt (Easterbrook *et al.*, 1981). A fission-track age of 0.84 ± 0.22 Ma has been obtained on zircon from the ash. Fission-track ages on the same (Lake Tapps) tephra at other localities are 0.87 ± 0.30 Ma (zircon) and 1.06 ± 0.11 Ma (glass)

Table

Sample	laboratory identification	reference	D_{eq} Gy	dose rate Gy ka^{-1}	Age ka
thermoluminescence dating, quartz					
East Naracoorte dune	SESA-63	a	455 ± 44	0.63 ± 0.02	720 ± 70
West Naracoorte dune	SESA-74	b	327 ± 30	0.41 ± 0.03	800 ± 100
optical dating, 1.4 eV, inclusions in quartz					
West Naracoorte dune	SESA-74	c	280 ± 50	0.41 ± 0.03	680 ± 130
optical dating, 1.4 eV, 4-11 μm polymineral grains					
Salmon Springs peat	SSP2-6	d	250 ± 30	0.37 ± 0.05	660 ± 120

a = Huntley *et al*. 1993a
b = Huntley *et al*. 1994
c = Huntley *et al*. 1993b (for method)
d = Lian *et al*. in preparation. Most of the details can be found in Hu (1994).
    These are: additive dose, 7 day 140°C preheat, detection with EMI 9635 with BG-39 filter, no anomalous fading test.

(Westgate *et al*. 1987). The peat must therefore have been deposited shortly after the start of the Brunhes epoch at 0.78 Ma or shortly after the start of the Jaramillo event at 1.05 Ma, the latter seeming the more likely. A TL age of $> 0.48 \pm 0.06$ Ma for this peat was reported earlier by Huntley *et al*. (1983); the lower limit was given because of the observation of anomalous fading.

Results
The table summarizes our results. A variety of methods was used and these are indicated in the table in which specific references are given.

The TL date for West Naracoorte is in accord with the known age while that for East Naracoorte may be considered to be a little low for the age of 0.95 Ma. As we have noted elsewhere the dosimetry for both samples is not as secure as for others in the sequence, due to inhomogeneities, and this may be the cause of any inaccuracies. Huntley *et al*. (1993a) reported on a different West Naracoorte sample which did not exhibit a satisfactory TL behaviour, and we have since then found several samples of this age or older for which the N+dose and regeneration data do not meet our criterion for acceptance. We thus regard the TL ages for the West and East Naracoorte dunes to be at

the limit of what can be done with this method on this quartz.

The optical age for West Naracoorte, obtained using 1.4 eV excitation, is encouraging because it indicates that this method can be used on samples as old as this, and that the thermal lifetime of the traps appears not to be a limiting feature. Using the same method and regeneration data we have obtained an age of 1.2 ± 0.1 Ma (sample NL1, $D_{eq} = 900 \pm 40$ Gy, $\dot{D} = 0.75 \pm 0.04$ Gy ka^{-1}) on a quite different sample, of unknown age but classified as older than 1 Ma on geological grounds.

The optical age of the Salmon Springs peat was obtained using 1.4 eV excitation of 4-11 μm polymineral grains separated from the peat. Additive dose measurements similar to those described in Huntley *et al*. (1993b) were used. Details will be published elsewhere. The optical age is concordant if the true age is 780 ka, but is decidedly too young if the true age is 1.05 Ma.

In contrast with the above, an earlier attempt (by DJH) to test fine-grain TL dating on samples of loess from Tadzhikistan, which straddled the Brunhes-Matuyama reversal, gave apparent ages that were significantly too low.

In conclusion we note that three different methods, TL dating of quartz, optical dating of inclusions in quartz, and possibly optical dating of fine grains, all appear to be capable of yielding correct ages up to 800,000 years at some sites and with appropriate procedures. Berger *et al.* (1992, 1994) have previously shown this to be true for TL dating of fine grains.

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PR Martin Aitken
These results take us well beyond the half-million year mark that seemed ambitious only a few years ago; they will be encouraging news for all in the luminescence community, notwithstanding the qualification ".... at some sites and with appropriate procedures." The concordant optical age obtained for inclusions in quartz from West Naracoorte is of particular interest since, along with the referenced results of Berger, it indicates that there are other minerals besides quartz with a clean bill of health, intrinsically, as far as fading is concerned.

Use of UV-regenerated IRSL for normalization

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Introduction

In a study of IRSL signals from compressed loess pellets, Li and Wintle (1994) found that there was a considerable difference in the natural IRSL output from pellets of a similar age. It was also found that pellets from a palaeosol gave a lower natural signal than those from loess above it. It is therefore necessary to normalize the signal to allow for individual pellet sensitivity. Different normalization procedures for sample discs have been introduced in luminescence dating techniques (Aitken, 1985; Rhodes, 1990). These involve measurement of the luminescence signals after alpha, beta, or gamma irradiation, but such procedures are not suitable for pellets and a new normalization method has to be introduced.

Exposure of feldspar grains to short wavelength radiation (<300 nm) shows that the IRSL signal is increased for a sample previously bleached by sunlight (Bailiff and Poolton, 1991; Li, 1992). Here we discuss the use of the ultraviolet (UV) regenerated signal for normalization of the IRSL signal from pellets.

Experimental

The equipment used in IRSL measurement was a modified Daybreak manual TL reader (Duller et al., 1992; Li and Wintle, 1992). IR stimulation was provided by a diode array. An optical filter of 2 mm thick Schott BG39 was placed in front of the EMI 9635QB photomultiplier tube used to detect the luminescence emitted from the sample. The IRSL signals were measured with 1 second IR exposure.

The UV radiation source was a mineral lamp (UVP Inc., U.S.A.). The output wavelength peaked at 254 nm. The distance between the UV lamp and the pellets was 10 cm. To minimize the UV power differences, samples were placed within a relatively small area (5x5 cm) beneath the lamp. Under these conditions, the UV-regenerated IRSL signal increased to a maximum level after 1 h UV exposure, and this time was chosen for subsequent experiments.

Pellets were made by compressing with a modified car jack. Each pellet was cylindrical, 10 mm in diameter and weighted about 0.5 g. The height of each pellet was around 6 mm. The pellets were from mixtures of loess with either quartz or carbonate. After the natural IRSL signals had been measured, the pellets were then exposed to sunlight for 6 h to completely remove the natural signal, checked by measuring the IRSL after bleaching. After exposure to UV for 1 h the samples were stored for 16 h and the UV regenerated signal was then measured at room temperature.

UV normalization

The UV normalization was tested by normalizing pellets which contained known proportions of IR sensitive and insensitive materials. Since no IRSL signal has been found in calcite and most quartz (Spooner and Questiaux, 1989; Short and Huntley, 1992), but a signal is found in most feldspar minerals, the IRSL signal will decrease when IRSL insensitive material, such as quartz, is added to a sample with a fixed concentration of feldspar. A 70 ka old Chinese loess was chosen as a homogenous sample containing about 10% feldspar as the IR sensitive material. Finely ground quartz and carbonate from a lake sediment were chosen as the two diluting materials. No IRSL signals were detected from either after a 100 Gy dose. For each material two sets of pellets were made from well-homogenized mixtures with the loess content varying
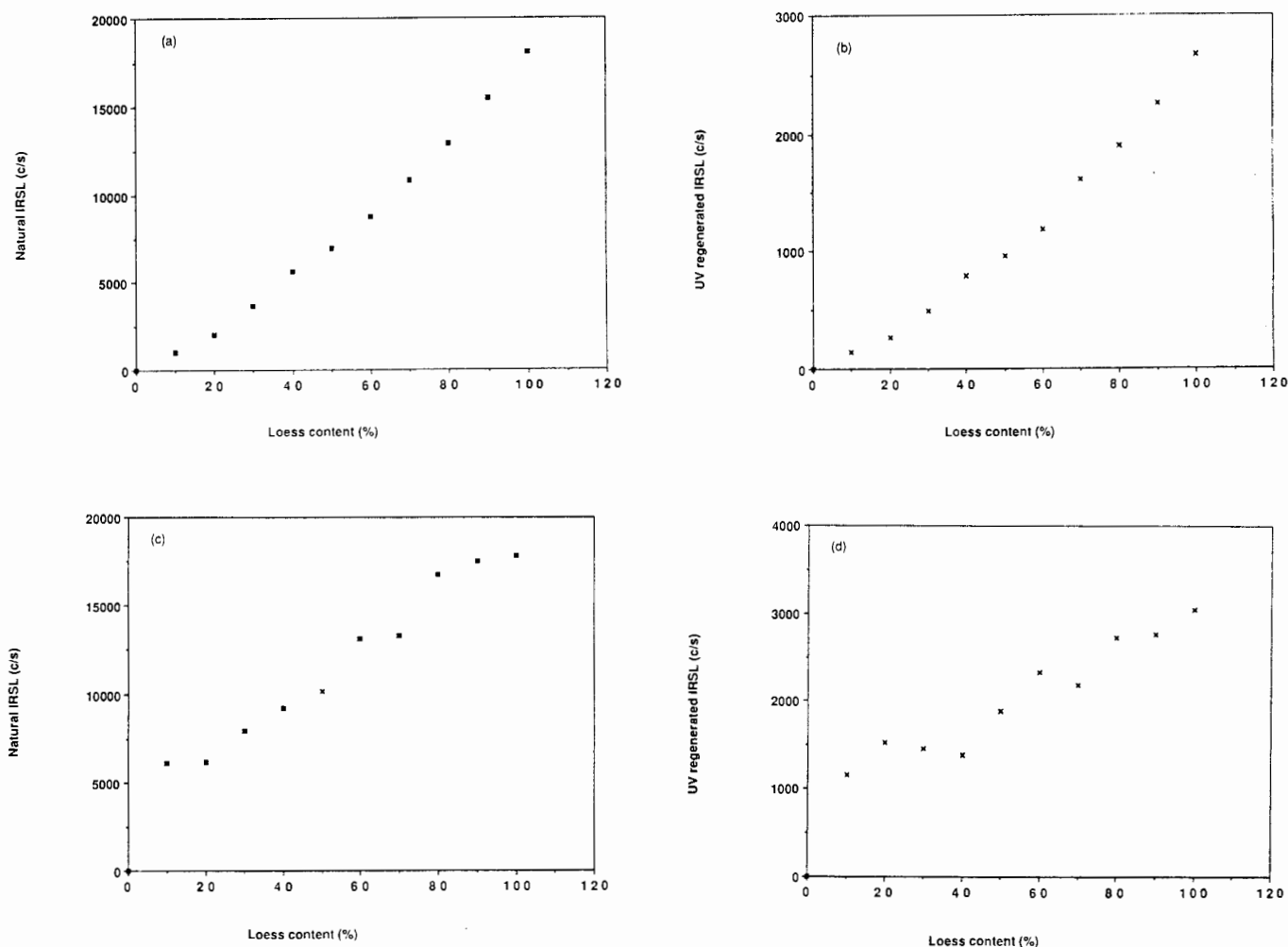
Figure 1.
IRSL signal of pellets of loess mixed with carbonate or quartz. (a) natural signal of loess mixed with carbonate, (b) UV-regenerated signal after sunlight bleaching, the pellets were the same as (a), (c) natural signal of loess mixed with quartz, (d) UV-regenerated signal after sunlight bleaching , the pellets were the same as (c).

Figure 2.
Schematic diagram for regeneration of the IRSL signal from pellets. (a) loess mixed with carbonate, (b) loess mixed with quartz. Feldspars give rise to the IRSL signal; carbonate can be treated as a mineral which absorbs the IRSL; quartz passes the IRSL signal. D is penetration depth of IR. d is equivalent optical depth for stimulated luminescence.
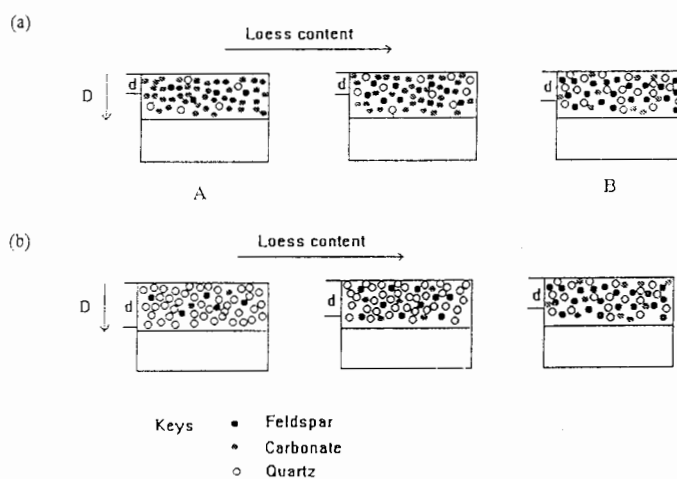
Table 1. Ratio of normalized IRSL signal from pellets relative to that from 100% loess.

| % loess | Ratio | |
	Mixture of loess & carbonate	Mixture of loess & quartz*
10	1.07	0.90
20	1.13	0.69
30	1.11	0.93
40	1.06	1.13
50	1.08	0.92
60	1.09	0.96
70	0.99	1.04
80	1.01	1.05
90	1.01	1.08
100	1.00	1.00

N.B. Pellets containing 0, 10 and 20% of loess in the mixture of loess and quartz were not as robust as other pellets and grains were lost during the experiment.

from 0 to 100% in 10% steps. The pellets were sufficiently robust to be handled, except those three pellets which had the highest quartz content.

For both sets of pellets, the natural signals increased monotonically with loess content, though not linearly (Figs 1a & 1c). The UV-regenerated signals also increased with loess content giving a similar pattern as for the natural signals (Figs 1b & 1d). A clear difference can be seen between the response when diluting with quartz and with carbonate. The UV-regenerated signal was used to correct for the response of different loess contents and the results for duplicate pellets are given in Table 1. Values close to unity were obtained suggesting that normalization of the natural IRSL signal can be achieved using this approach.

Effective depth and IRSL

The different responses that occur with carbonate or quartz dilution (shown in Fig. 1) may be explained by invoking an effective depth involved in the production of the natural and UV-regenerated IRSL signals. This is shown schematically in Figure 2. For high carbonate concentrations (A) the IRSL signal from the feldspar grains at depth D (the maximum depth of IR penetration) would be absorbed by the carbonate before it could get through to the surface and be detected. The

effective depth d is relatively shallow in this case, with only a few feldspar grains near the surface giving rise to the signal. As the loess concentration was increased (B), the quartz concentration also increased since the loess contains a high percentage of quartz. If quartz is transparent to IRSL, then more IRSL can pass through the quartz grain and thus get through to the surface. The effective depth d is therefore greater. Thus increasing the loess content not only increases the amount of IR sensitive material (feldspars), but also increases the effective depth d of observation of IRSL. Hence, a monotonic but non-linear increase of the IRSL signal would be expected with the increase of loess content, as found in Figures 1a and 1b.

For dilution with quartz, the higher quartz concentration allows most of the IRSL to get through to the surface and results in a greater effective depth d. Although high quartz means there is less feldspar in the pellet, the increased sampling depth makes the IRSL signal decrease more slowly with decreasing loess content, as observed in Figs 1c and 1d. Similarly, in the UV regenerated IRSL signal, the effective depth can also be affected by UV exposure if the penetration depth of the UV is less for carbonate than for quartz. As can be seen in Table 1, a marginal difference in the ratio between pellets containing lowest loess contents is found between both mixtures. This indicates the relative deeper penetration depth of UV in quartz than in carbonate resulting in a relatively large UV regenerated IRSL signal for higher quartz content. However, this has an insignificant effect on the normalization results as close to unity results were obtained in both mixtures.

The UV normalization method has been used satisfactorily in Aberystwyth for several years for pellets and has the potential to be used for grain discs. As has been shown in this study, the UV-regenerated IRSL is related to the feldspar concentration in the sample. The changes in feldspar concentration resulted from weathering and diluting will be reflected in the UV-regenerated signal and therefore further sedimentary information can be provided by the signal (Li, 1992; Li and Wintle, 1994). It is possible that the implications and interpretation of the UV-regenerated signal are more important than just as a normalization method.

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An alpha irradiator for ESR dating

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Due to the large sample size required for ESR measurements (10-20 mg), ESR alpha irradiation experiments on thin fine grain layers have rarely been reported (e.g. Yokoyama *et al.* 1981, Grün 1985, Mudelsee 1990). Some studies have used an accelerator to determine alpha radiation effects on speleothems (Lyons & Brennan 1989, 1991) others have experimented with [210]Po-doping (DeCanniere *et al.* 1986).

In order to perform alpha irradiation on thin fine grain samples, we have constructed a large [241]Am-irradiator (see Fig. 1). The irradiation chamber is constructed from a 150 mm ID stainless steel tube which is cut in two halves. The top contains the alpha source and a valve to flush the chamber with argon when not in use in order to avoid oxidation of the [241]Am foils. The bottom half contains an adjustable plate for holding samples and the collimator. The maximum distance between the source and the samples is 100 mm, the positioning can be read from a steel ruler. A side valve is used to evacuate the chamber to about 10^{-3} Torr.

The alpha source (Fig. 2) has a diameter of 100 mm and is built by four [241]Am smoke detector foils (Amersham glued the foils into holder that we supplied). Since the foils have an inactive strip of 1mm at each side, the alpha emission is not homogeneous. Therefore the sample holder has four fixed sample positions for disks with a diameter of 31.5 mm. In order to guarantee a steep impact angle (>65°) of the alpha particles, we have constructed a 64 mm high brass collimator. The use of metal material avoids electrostatic charging which may occur in plastic. Figure 3 shows the sample holder and the collimator.

The energy of the alpha particles at the surface of the

foils is 4 MeV (according to Amersham). Each fixed sample position was calibrated against the alpha source of the Max Planck Institute in Heidelberg by TL using $CaSO_4$:Dy. The dose rates for position 1 to 4 in a distance of 65 mm using the collimator are 31.64±0.69, 27.73±1.05, 29.44±1.48 and 29.91±0.09 Gy h^{-1}, respectively. The errors result from repeated measurements.

We have carried out some alpha efficiency measurements. Fine grains were separated from ground samples with deposition in acetone as described by Huxtable (1979). About 20-22 mg were effectively deposited on the aluminium sample holders (diameter: 31.5 mm; thickness: 5 mm) which corresponds to an average thickness of about 9 to 10 μm. The range of 4 MeV alpha particles is about 15 μm in a matrix with a density of 2.95 g cm^{-3} (Rytz 1979). This energy is high enough for the alpha particles to pass completely through a 10 μm layer of constant thickness (with the above configuration).

Gamma irradiation was carried out with a calibrated ^{60}Co source at Louvain-la-Neuve. Alpha and gamma irradiations were performed on aragonitic mollusc shells, corals and on tooth enamel (hydroxyapatite). ESR measurements were performed with a Bruker ER 200 tt ESR spectrometer at room temperature.

Figure 4 shows the dose response curves for gamma and alpha irradiation on recent enamel (g=2.0018) and mollusc shells (over-modulated signal with 0.63 mTpp; see Katzenberger, 1989), respectively. The best fits using a single saturating exponential function are shown in Figure 4. These curves have not been forced through zero. The scatter of the data points around this best fit may be used to assess the random errors that are

Figure 1. (Right)

Alpha irradiator for ESR samples: the alpha source (see Fig. 2) is mounted in the upper half of the stainless steel tube. The samples and collimator (Fig. 3) are placed on the adjustable plate. The chamber can be evacuated (top valve) and flushed with argon (side valve).

Figure 2. (lower right)

Alpha source and source holder (top: side view, below: frontal view). The source has a diameter of 100 mm and is constructed from four smoke detector foils.

Figure 3.(lower left)

Sample holder (top and below) and collimator (top). Dimensions are in mm. The two smaller holes in the sample holder are for fixing the positions of the samples.
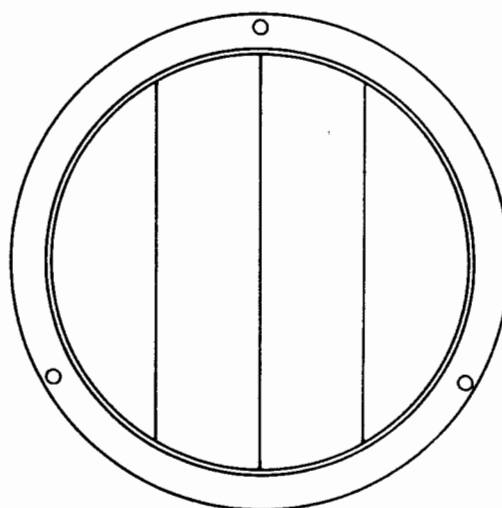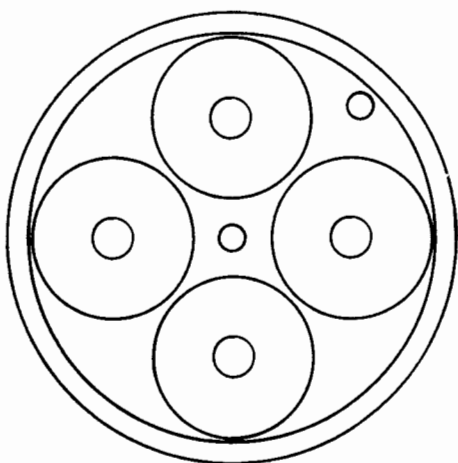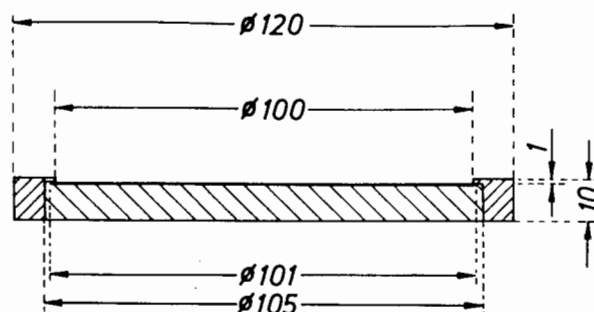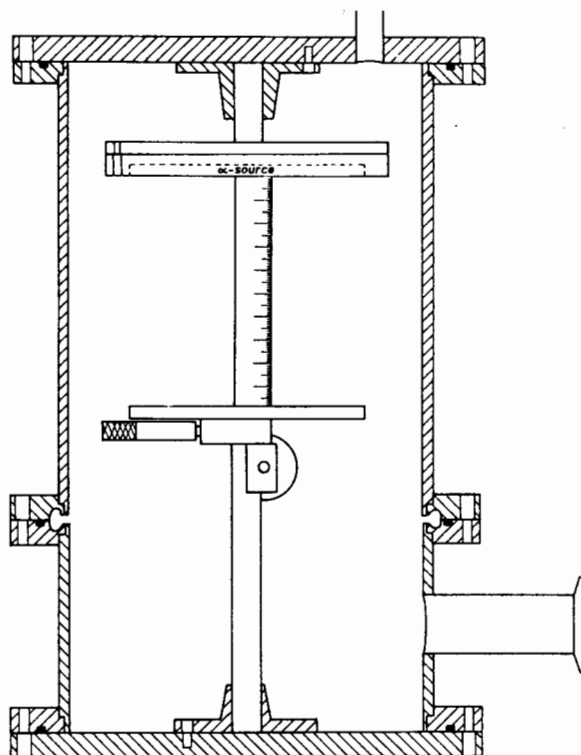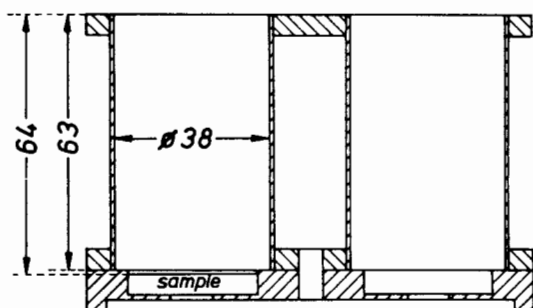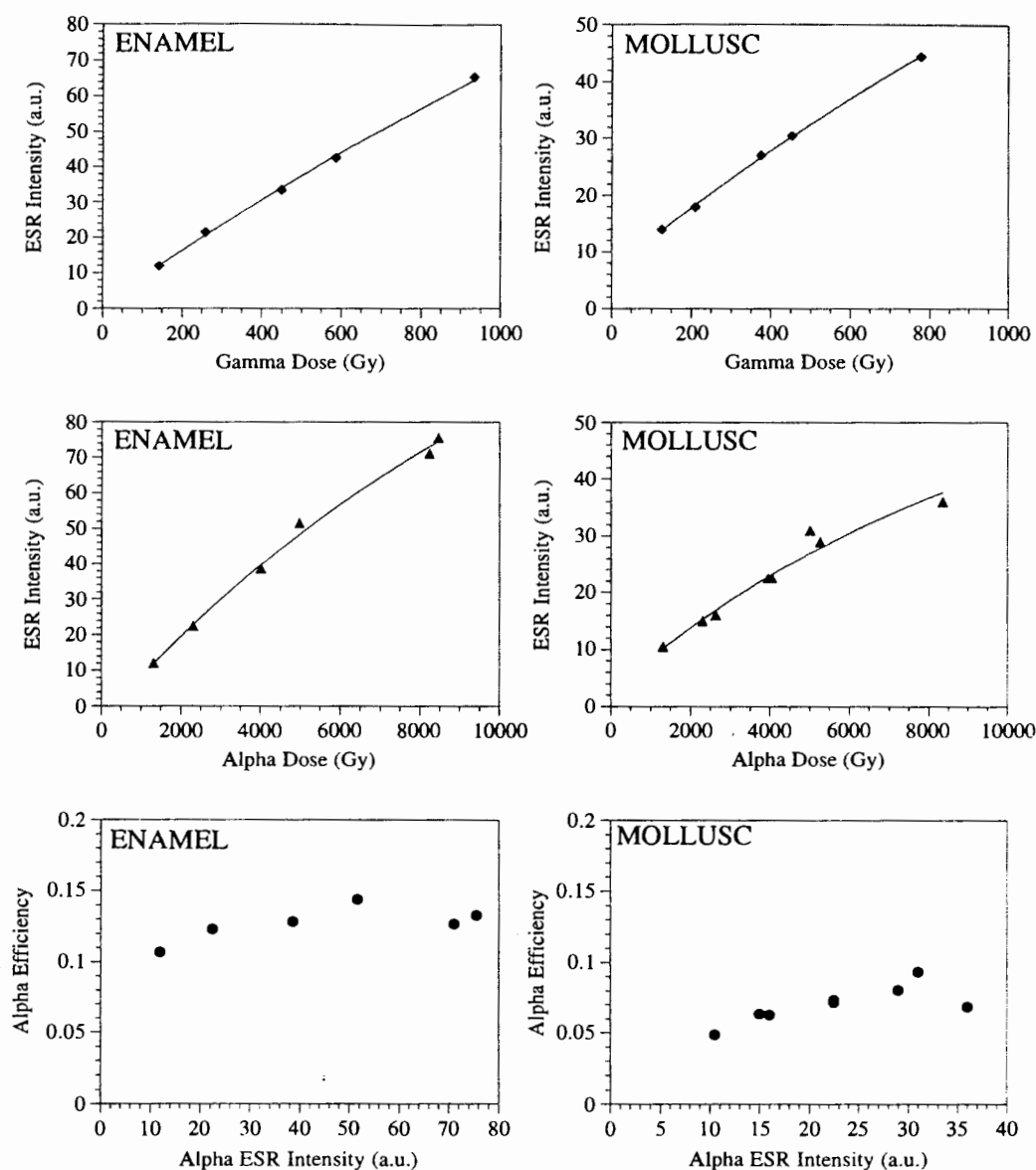
Figure 4.

Gamma (top) and alpha (middle) dose response curves for enamel (left) and mollusc shells (right). The lowermost diagrams show the alpha efficiencies by projecting the measured alpha intensity values onto the gamma dose response curves and calculating the ratio of projected gamma dose over alpha dose.

involved in the sample preparation and irradiation procedures. For molluscs the data points show a scatter of about 1.2% and 5.8% around the best fit for gamma and alpha irradiation (using the same sample pretreatment procedures), respectively. For enamel the corresponding values are 2.2% and 4.9%. This shows that the alpha irradiation causes an additional error compared to gamma irradiation which may be attributed to agglomeration of the fine grains. Considering a random error of up to 5% in the calibration plus a

possible systematic calibration error of 5%, the precision of this configuration is in the range of 7% and the accuracy for dose measurements is probably in the range of 12%.

The lowermost diagrams of Figure 4 show the calculated alpha efficiencies for the measured samples. The corresponding gamma doses were calculated by projecting the measured alpha intensities onto the fitted gamma dose response curve. The efficiency value

results from the ratio of projected gamma dose over alpha dose. Both data sets imply a more or less constant alpha efficiency over the whole dose range although the lowermost intensity values seems to display slightly lower efficiency values than the higher ones. This may be due to a somewhat larger signal to noise ratio. The average efficiency values are 0.127 ± 0.011 for enamel and 0.070 ± 0.012 for mollusc shells. The alpha efficiency values of corals presented by Radtke and Grün (1988) and Grün et al. (1990) of 0.06 ± 0.01 and 0.05 ± 0.01, respectively, were also obtained with this source.

Experiments with alternating alpha and gamma doses (Katzenberger, 1989) on mollusc shells do not show changes in the gamma sensitivity which implies that damage by alpha rays does not create additional trap sites in this material.

The alpha efficiencies of secondary carbonates measured with this source and obtained by others are all below 0.1 (for a compilation see Grün, 1992). This means that the contribution of the alpha dose rate to the total dose rate is relatively small and therefore uncertainties in the assessment of the total dose rate introduced by an unknown alpha efficiency (e.g. 0.07 ± 0.03) are also relatively small (see also Lyons and Brennan, 1989; 1991). However, more measurements should be carried out before such a low alpha efficiency value can be assumed for all ESR signals in secondary carbonates.

The alpha efficiency of tooth enamel which has been repeatedly measured to be in the range of 0.11 and 0.15 is not negligible, particularly for samples with high uranium concentrations.

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PR Albrecht Wieser

A simple semi-automatic TL apparatus of new design

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General principles

Sixteen small cups lined up at regular intervals along the circumference of a polished metallic round plate (supporting platform; see Fig. 1) sit within the somewhat larger circular holes of an overhanging perforated metallic turntable (Figs. 1 and 2). As the turntable rotates it pushes all the cups along a circular path and placing them one after another on top of a titanium hot plate imbedded in the support and level with it. As each cup is centered over the hot plate the rotation stops, heating is applied and the TL measured. To make it less likely that the turntable interferes with the controlled temperature rise of the cup during the TL measurements two precautions are taken:

1. The hot plate is separated from the turntable by a fraction of a millimeter;
2. Once a cup sits on the hot plate the turntable reverses direction for a fraction of a second breaking physical contact with the cup.

Key Components

The apparatus consists of three major components.
1. A DC motor driving the turntable through a drive belt;
2. An underlying co-axial circular plate (slotted wheel) with equidistant slits along the circumference, one for each turntable hole;
3. Slotted opto-switch which turns off the motor when triggered by the passage of a slit (in the underlying co-axial circular plate) indicating that the cup is centered over the hot plate.

Construction

We modelled the apparatus on the equipment currently in use in our Laboratory, except that the 16 titanium cups (density 4.5 g/cm^3) have an external diameter of 13 mm and can accommodate about 10 mg of 100-160 μm size grains. The cups were polished with an alumina jet to help spread a monolayer of grains evenly over their surface.

The heating system is similar to one described in several publications (Bøtter-Jensen, 1988; Galloway, 1990). The temperature is measured with a chromel-alumel thermocouple in a stainless steel tube buried in the 1.5 mm thick hot plate. A 12 volt, 3 Watt, TAA-Crouzet motor rotates the turntable via a Binder-magnetic synchroflex drive belt (type M449C) at the rate of 4 RPM. The motor is powered through an integrated circuit UDN2953B which allows for forward and reverse motion. The arrival of cups on the hot plate is signalled by the plate slots which are detected by an Optoschmitt Honeywell HOA2001 slotted optoswitch.

Results

A run of 16 measurements with 12 mm cups containing about 10 mg of flint grains each yielded a standard deviation of 1.7%. An X-Y plot of the measurements is reproduced in Figure 3. The scatter of mean temperatures from 16 curves ($\pm 2°C$) provides an indirect test of reproducibility of the temperature rise. In practice, when the paleodose of burnt flint is determined by the additive dose method only four cups per dose are used which yield TL glow curves with a precision of about $\pm 1\%$.

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PR Ed Haskell

The described reader offers significant simplifications over other designs currently in use. Its main advantage lies in the elimination of additional hardware normally required to move the sample disk into contact with the heating plate. This, in principle, results in better vacuum integrity, and more precise alignment of the sample under the PMT over long periods of time. The design is also amenable to the addition of a beta source which would be useful for pre-dose measurements. the use of a 1.5 mm titanium heating plate may also minimize plate movement and warping during heating.

Figure 1.
Vertical cross section showing the principal components of the apparatus.
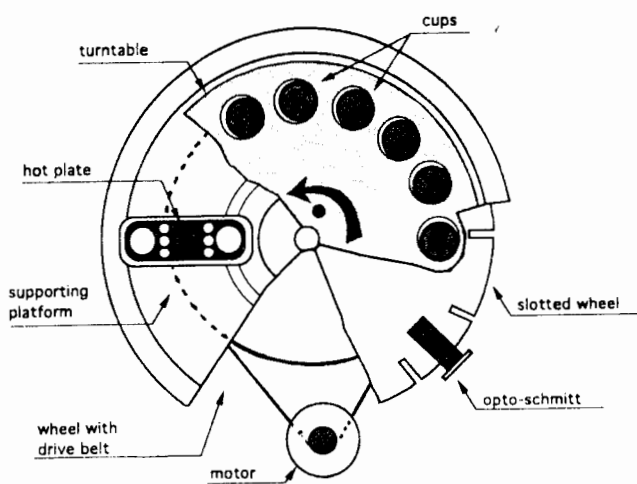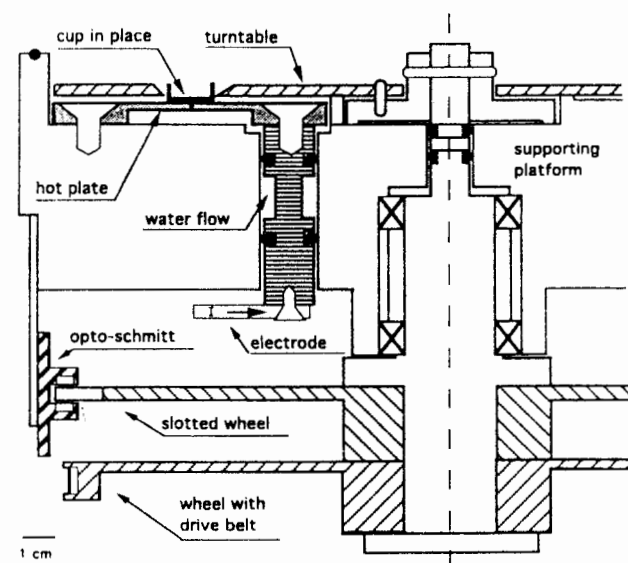

Figure 2.
Plan view of the apparatus showing the rigid supporting platform and the three synchronous rotating wheels (turntable, slotted wheel, and the wheel pulled by the drive belt). For the sake of clarity, the Optoschmitt is shown in the plane of the figure.
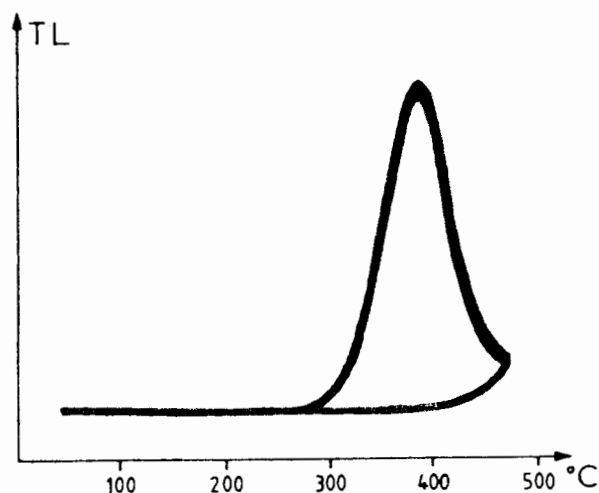

Figure 3.
TL curves of 16 cups containing 10mg aliquots of crushed burnt flint each. The standard deviation was ± 1.7%.

Thick-source alpha counting of peat

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Some years ago we showed the potential for dating peat by thermoluminescence (Huntley et al. 1983), the details of which may be found in Divigalpitiya (1982). With renewed interest in the subject here I have been concerned with the question of whether or not thick-source alpha counting of peat gives the results expected of it. It is possible, for example, that the organic material may not be homogeneously distributed within the sample placed on the ZnS, and hence lead to an incorrect evaluation of the dose rate.

Divigalpitiya showed that the way to evaluate the dose rate when both water and organic matter were present was to consider the sample to consist of three components: mineral (m), water (w) and organic matter (o). He defined A values for water and organic matter by

$$\Delta^w = M^w/M^m \text{ and } \Delta^\circ = M^\circ/M^m \qquad (1)$$

where the M's are the masses of the three components.

The correction factor for the alpha dose rate then becomes

$$\frac{1}{1 + H_\alpha^w \Delta^w + H_\alpha^o \Delta^\circ} \qquad (2)$$

where H_α^o is the ratio of the stopping power in the organic material to that in the mineral, for alpha particles. H_α^w is the same ratio but for water to mineral and is what is in common use and denoted by H_α. This correction factor is to be applied to the dose rate that would exist in the absence of both organic matter and water.

Similar expressions are used to correct the beta and gamma dose rates. Divigalpitiya calculated the following values of the H^o values:

$$H_\alpha^o = 1.37 \pm 0.06, H_\beta^o = 1.20 \pm 0.03$$
$$\text{and}$$
$$H_\gamma^o = 0.948 \pm 0.08 \qquad (3)$$

With the arguments of Aitken and Xie (1990) it seems likely that this value for H_γ^o is probably too small and a value of 1.1 would be more appropriate.

In order to make the corrections it is necessary to determine the value of Δ° for each sample. We do this by drying the sample, weighing it, putting it in an oven at 500°C overnight to burn off the organic material, and weighing again. Δ° is calculated as the weight loss divided by the final weight. If storage of the dried peat is necessary, this is done in a desiccator.

Expression (2) is just the factor by which the rate at which alpha-track-length delivered to mineral matter is reduced by the presence of water and organic matter. The same factor pertains to the expected reduction of the thick-source alpha count rate. In the absence of water the reduction factor is $1 + H_\alpha^o \Delta^\circ$.

The experiment was designed to test this. The peat samples used were from Lynn Canyon, North Vancouver (LCP5), Double Bluff, Whidbey Is., Washington (WFP2), and Salmon Springs, Sumner, Washington (SSP2). Each was dried at ~100°C and milled well in a ring mill; this is the material called "dry peat" in the table. A subsample of this was heated overnight at 500°C to determine Δ° and the resulting material is called "ash" in the table.

The table shows the alpha count rates for the peats and ashes, their ratios, the Δ° values and the theoretically expected ratios. Because dry peat was typically dark brown and the ashed samples were much lighter in

Table: Thick-source alpha count rate

Sample	Δ *	dry peat $ks^{-1}cm^{-2}$	ash $ks^{-1}cm^{-2}$	measured ratio	reflectivity - corrected ratio	expected ratio $1 + H_\alpha^0\Delta$ *
LCP5-S	0.33	0.164±0.004	0.259±0.005	1.58±0.05	1.44±0.05	1.45±0.03
SSP2-6	1.20	0.086±0.003	0.264±0.007	3.07±0.14	2.73±0.14	2.64±0.08
SSP2	2.06	0.052±0.002	0.217±0.004	4.17±0.17	3.72±0.17	3.82±0.14
WFP2-6	6.90	0.037±0.002	0.361±0.004	9.76±0.54	9.00±0.50	10.45±0.42

colour, one can expect a significant effect of the different sample reflectivities on the measured ratio. Reflectivities were therefore measured, and the count rates corrected for them as outlined in the appendix. The ratios of the corrected count rates are also shown in the table. (An alternative to the reflectivity correction is the placement of thin aluminized mylar between sample and ZnS to eliminate the effect of reflectivity variations as outlined by Martini, 1981).

It is seen that there is satisfactory agreement between expected and corrected ratios. I conclude then that all is well, but note that this method, while suitable in principle suitable for determining H_α^0, does not yet have sufficient precision to give a value better than the calculated one. It is perhaps worthwhile reiterating here the necessity of milling or glassing most sediment samples for alpha counting because of non-uniform distribution of U and Th (Jensen & Prescott, 1983).

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Appendix

Here I discuss the correction to the alpha count rate needed when the reflectivity of the sample is different from that of the standard used to set the discriminator on the alpha counter. The model used is that of Huntley and Wintle (1978) (or see Huntley, 1978) in which the size of the light pulse incident on the photocathode of the photomultiplier is proportional to $(1+\rho)$ where ρ is the sample reflectivity. The discriminator voltage setting required to pass a specific fraction of these pulses is thus also proportional to $(1+\rho)$.

With this assumption one can show that the count rate measured for a sample should be divided by

$$\frac{(1+\rho_s) + (\frac{\rho-\rho_s}{0.82})}{1+\rho}$$

where ρ is the reflectivity of the sample, and ρ_s is the reflectivity of the standard used to set the discriminator. The number 0.82 is appropriate for a uranium standard and should be changed to 0.85 for a thorium standard.

In the present work a glass standard was used and its reflectivity determined to be 0.5. Reflectivities of the dried peat samples were all under 0.1 and those of the ashes were in the range 0.35 to 0.63.

The influence of sample treatment on feldspar dose response

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Introduction

There are occasions when it is desirable to make measurements on samples which have been bleached of their natural luminescence, both to study of aspects of the luminescence process and for regeneration dating. Changes in sensitivity with certain bleaching methods have often been reported, e.g. Duller (1991) in relation to infra-red stimulated luminescence, Fleming (1979) in relation to thermoluminescence. Sensitivity change and supralinearity of dose response are closely related in thermoluminescence (TL), e.g. McKeever (1985). We have looked at how the linearity of dose response is influenced by bleaching and by preheating for feldspar samples stimulated by infra red and by green light.

Method of measurement

Fine grain samples of a Norwegian microcline feldspar and of an orthoclase feldspar were prepared by sedimentation on stainless steel discs. The samples were bleached either by exposure to daylight for 4 weeks, by Henley SOL 2 solar simulator for 24 hours or by heating to 500°C . The radiation dose was then applied using a ^{90}Sr beta source.

Infra red stimulation used LEDs with an emission maximum at 950 nm wavelength and the luminescence was measured through BG39 and 7-59 filters (Galloway, 1991). Green stimulation used LEDs with an emission maximum at 565 nm wavelength and the luminescence was measured through a combination of BG39, UG11, 7-59 and 7-60 filters (Galloway, 1992; 1993). Some TL measurements were made for comparison using the equipment described by Galloway (1990) and with HA3, UG11 and 7-59 filters in front of the photomultiplier. The heating rate was 5°C/s.

Each determination of the dose dependence of luminescence used a set of aliquots, of between 11 and 23 in number. After exposure to the beta source and preheating the aliquots were measured under infra red stimulation for 0.1 s, then with green stimulation for 10 s after which either a TL glow curve was recorded up to 500°C or the aliquots were simply heated to 500°C. All aliquots in the set were then given an equal beta dose and the measurement sequence repeated to provide normalisation against mass fluctuations between the aliquots. The infra red dose response was corrected using the infra red normalisation measurements, the green response by the green normalisation measurements and the TL response by the TL normalisation measurements. Tests showed that the 0.1 s infra red exposure made no detectable diminution of the subsequent green stimulated luminescence and that the 0.1 s infra red and 10 s green stimulations made no detectable difference to the TL glow curve, in accord with previous investigations of the inter-relation between these luminescence processes in feldspars e.g.. Duller and Bøtter-Jensen (1993); Galloway (1994).

Dose responses

The response of microcline feldspar which had been bleached by daylight, with preheating at 220°C for 10 mins, stimulated by infra red and by green light is shown in Fig. 1. The response is linear and extrapolation to the counts axis indicates a small positive value as would be expected since for zero dose at least photomultiplier noise and a little scattered light is inevitably counted with the possibility of a small recuperated signal also (Rees-Jones and Tite, 1994). The agreement between extrapolated and measured counts for zero dose is shown in Table 1.
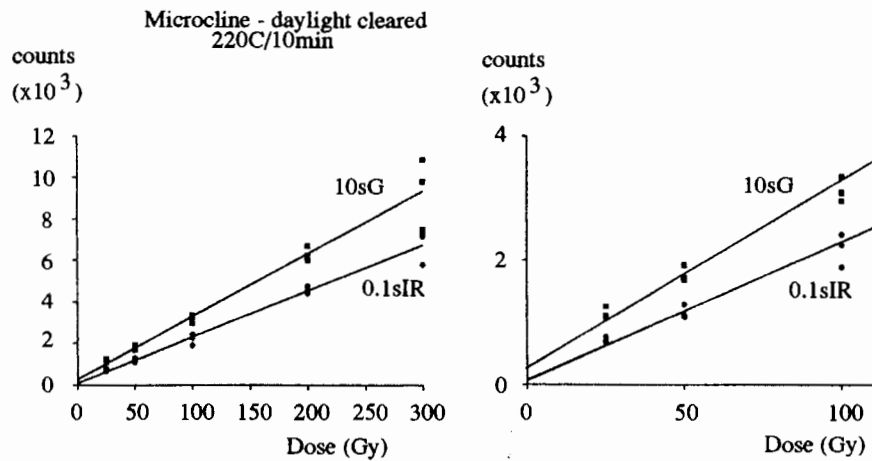
Figure 1 .

(a) The luminescence dependence on dose for daylight bleached microcline feldspar preheated at 220°C for 10 mins and stimulated by infra red and by green light. Measurements were made on 15 aliquots. The counts axis indicates the number of photon counts recorded in 0.1 s of infra red stimulation and in 10 s of green stimulation. The lines are least squares fits to the data points. Plot (b) is of the low dose region on an enlarged scale where any indication of supralinearity would show.
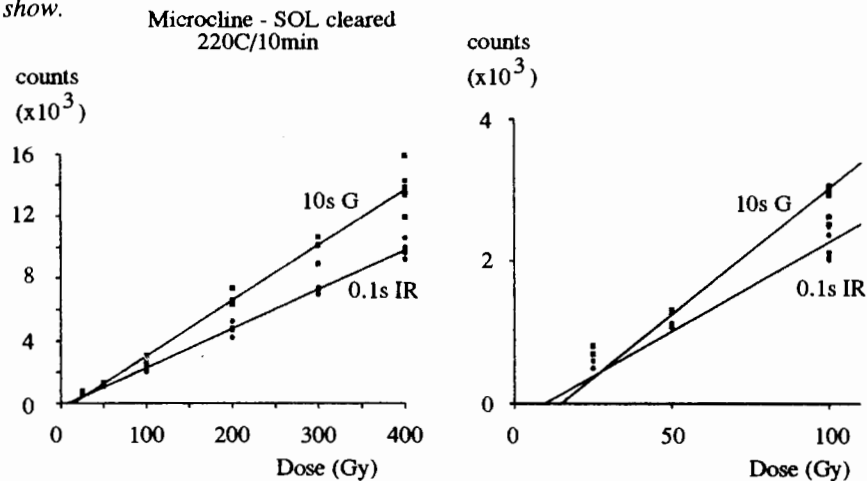

Figure 2.

(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator, based on 23 aliquots and in a similar format to Fig. 1. Preheating was at 220°C for 10 mins. Plot (b) of the low dose region shows that the response for both infra red and for green stimulation is supralinear below 50 Gy.
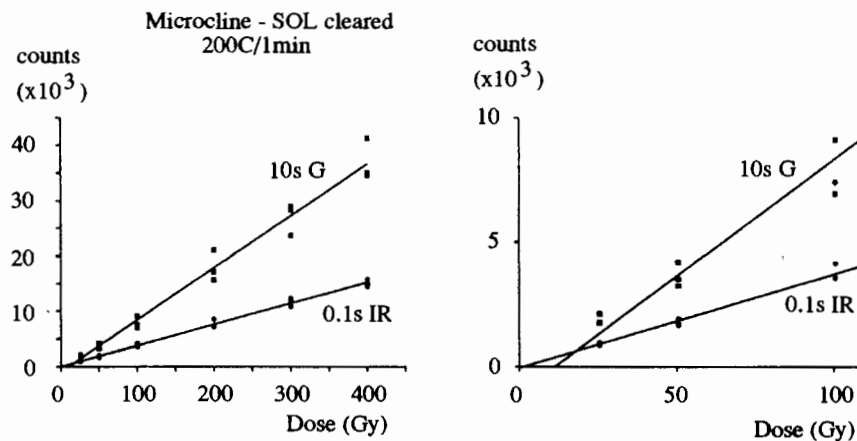

Figure 3.

(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator with preheating reduced to 200°C for 1 min.. 18 aliquots were used. The low dose plot (b) shows that supralinearity persists for the green stimulation.
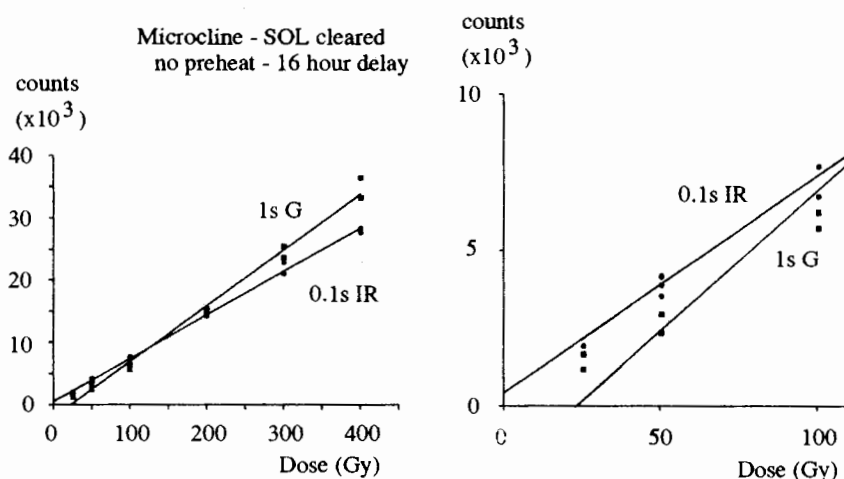
Figure 4 .

(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator and with no preheating but with a delay of 16 hours between dosing and reading. Green stimulation was for 1 s in this case and 13 aliquots were used. The low dose plot (b) shows supralinearity for green stimulation but not for infra red.
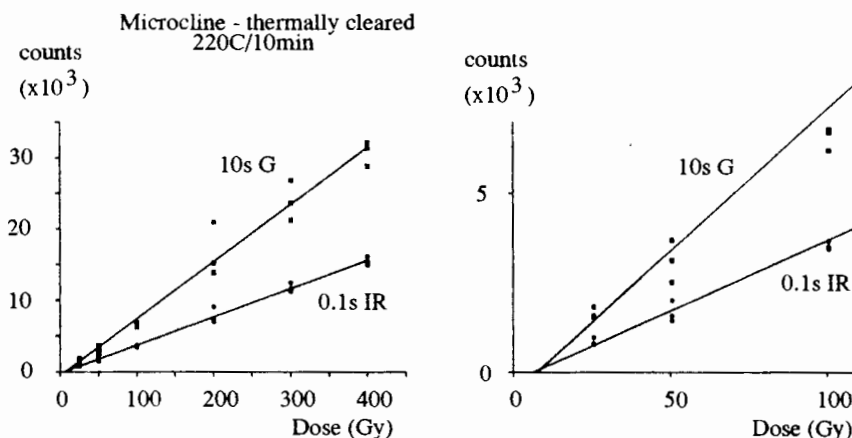

Figure 5 .

(a) The luminescence dependence on dose for microcline feldspar bleached by heating to 500°C and with preheating after beta dose at 220°C for 10 mins. 18 aliquots were used. The enlarged low dose plot (b) shows the possibility of a small degree of supralinearity.
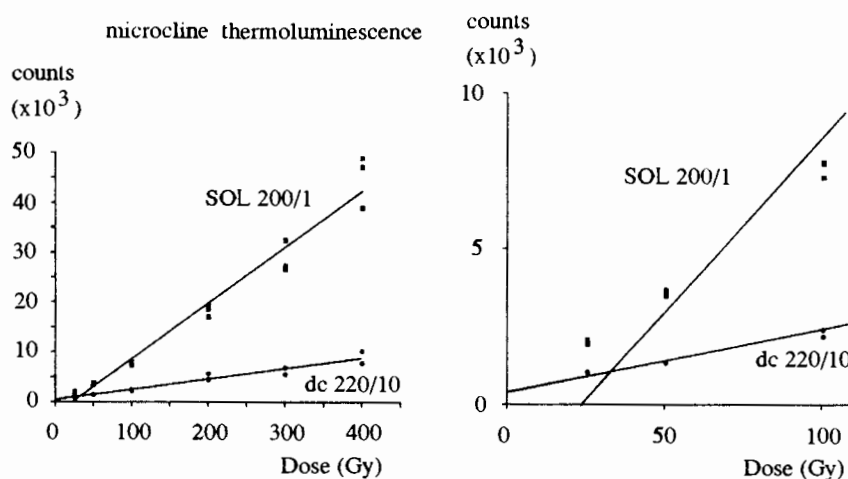

Figure 6.

(a) The TL dependence on dose for microcline feldspar, comparing daylight bleached material preheated at 220°C for 10 mins (12 aliquots) with SOL 2 bleached material preheated at 200°C for 1 minute (18 aliquots). The enlarged low dose plot (b) emphasises the acceptable linearity of the daylight bleached material and the supralinearity of the SOL 2 bleached material. The counts axis indicates counts recorded in 0.4 s.
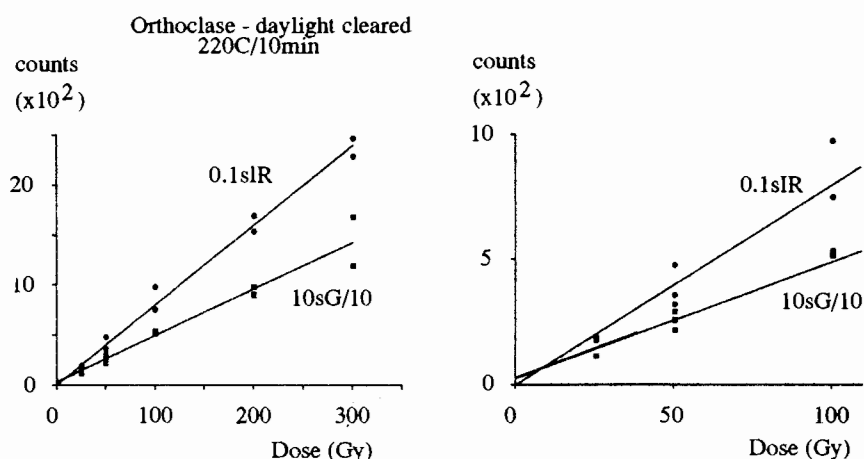
Orthoclase - daylight cleared
220C/10min

counts
$(x10^2)$

counts
$(x10^2)$

Figure 7.

(a) and (b). For orthoclase feldspar, daylight bleached and preheated at 220°C, the luminescence response to dose for both infra red and green stimulation is linear. The counts axis for the green stimulation indicates counts per second for the orthoclase material. 12 aliquots were used.
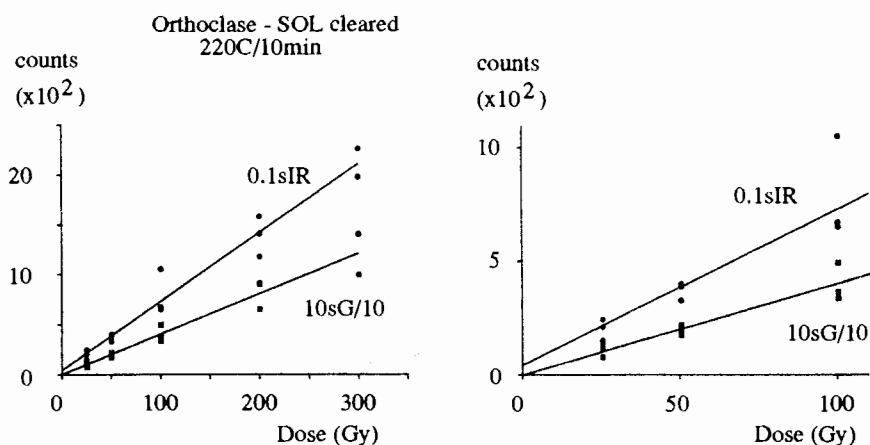
Orthoclase - SOL cleared
220C/10min

counts
$(x10^2)$

counts
$(x10^2)$

Figure 8.

(a) and (b). For orthoclase feldspar, bleached in the SOL 2 and preheated at 220°C for 10 mins, the luminescence response to dose for both infra red and green stimulation is linear, in contrast to the microcline response in figure 2. (14 aliquots were used).

orthoclase thermoluminescence
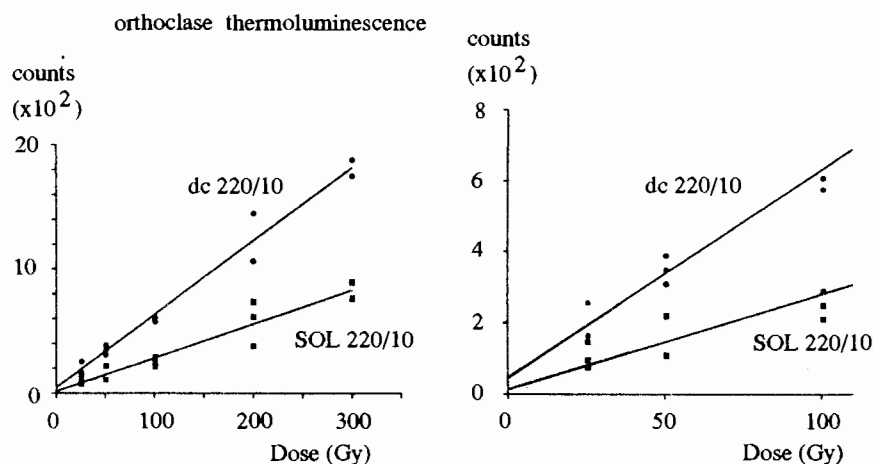
counts
$(x10^2)$

counts
$(x10^2)$

Figure 9.

(a) and (b). For orthoclase feldspar, the TL response for daylight bleached and SOL 2 bleached material compared. 11 and 14 aliquots respectively were used, with preheating at 220°C for 10 mins in both cases.

Table 1.
Comparison of measured and extrapolated counts for zero dose from daylight bleached material with preheating at 220°C for 10 mins.

		0.1s IR	10s Green	TL 330°C peak
microcline	Extrapolated	70±140 Fig. 1	250±280	370±330 Fig.6
	Measured	90±10	270± 20	350±20
orthoclase	Extrapolated	0±40 Fig. 7	300 ± 500 Fig. 7	45±45 Fig. 9
	Measured	16±4	220±15	130±12

Table 2.
Comparison, for different sample treatments, of extrapolated counts for zero dose, which should be a small positive number for linear response and becomes negative for supralinear response.

	bleach	preheat	0.1s IR	10s Green	TL (330°C peak)
Microcline					
	Daylight	220/10	70±140	250±280	370±330
	SOL 2	220/10	-250±120	-550±290	
	SOL 2	200/1	-60±180	-1100±700	-2600±1100
	SOL 2	None	390±260	2100±600	
	500°C	220/10	-270±200	-600±700	
Orthoclase					
	daylight	220/10	0±40	300±500	45±45
	SOL 2	220/10	40±60	0±450	10±40

Notes: Preheat is given as temperature °C /time mins; No preheat measurements had a 16 hour delay between application of beta dose and measurement.

The response of microcline feldspar which differed only by being bleached in the SOL 2 is shown in Fig. 2, with clear indication of supralinearity from both infra red and green stimulation. A convenient indication that the response is supralinear is given by the fact that the value for counts at zero dose extrapolated using the least squares fitted straight line is negative, Table 2. Staying with SOL 2 bleached microcline feldspar, but reducing the preheating to 200°C for 1 min. led to the response in Fig. 3, clearly supralinear for green stimulation but possibly linear for infra red stimulation as indicated numerically in Table 2. Moving to no preheating with a 16 hour delay between dosing and reading showed again a linear response to infra red and a supralinear response to green, Fig. 4 and Table 2.

Microcline feldspar which was bleached by heating to 500°C and which was preheated at 220°C for 10 mins is the subject of Fig. 5. Visual inspection suggests a small degree of supralinearity although the relevant standard deviations in Table 2, if taken at face value, suggest that the supralinearity may not be statistically significant. However it should be realised that the standard deviations in Table 2 can only be taken at face value if the data is linear. If the data is supralinear, not only will the extrapolated counts at zero dose be negative but the supralinearity will cause a deviation of measured points from the fitted straight line which will be reflected in an increased standard deviation compared with that obtained from data of similar quality which is linear.

Two measurements of TL dose response were made on the peak at 330°C for comparison with the infra red and green light induced responses discussed so far. Daylight bleached material preheated at 220°C for 10 mins showed a linear response, Fig. 6, and extrapolated zero dose counts in agreement with direct measurement on an undosed aliquot, Table 1. However for material

bleached in the SOL 2 and preheated at 200°C for 1 minute the response is clearly supralinear, Fig. 6 and Table 2.

Measurements were also made on orthoclase feldspar for comparison with the microcline feldspar results. These orthoclase measurements all involved preheating at 220°C for 10 mins and concerned daylight bleached material stimulated by infra red and green, Fig. 7 and SOL 2 bleached material, Fig. 8, with TL responses in Fig. 9. In all these cases the orthoclase response appears linear (see also Tables 1 and 2).

Discussion

With the microcline feldspar there is clear indication of the relationship between luminescence and dose being influenced by the method of laboratory bleaching of the sample. For infra red stimulation the linear response of daylight bleached material was replaced by a supralinear response for SOL 2 bleaching with preheating at 220°C for 10 mins but the response became linear for less or no preheating. A preheat associated nonlinearity of response to optical stimulation of some feldspar samples when adding to a natural dose has been reported by Godfrey-Smith (1993). For green stimulation the daylight bleached material also shows a linear response while the supralinearity associated with SOL 2 bleaching becomes worse as the preheating is reduced in contrast to the behaviour for infra red stimulation, Table 2. TL too shows a linear response for daylight bleached material and supralinearity for SOL 2 bleaching with 200°C for 1 min. preheating, resembling the green response rather than the infra red. That the linearity of response for infra red stimulation and for green stimulation depend differently on bleaching and preheating is qualitatively in accord with other evidence that the trap populations probed by the different methods of stimulation are not identical (Duller and Bøtter-Jensen, 1993; Galloway, 1994). In relation to models of the luminescence process, there are calculations from which the inducing of supralinear response may be related to sensitivity change (McKeever, 1985; 1991) and the model of Li and Wintle (1992; 1993) which relates sensitivity change of infra red stimulated luminescence to the duration of bleaching.

However treatments that produced significant supralinearity of response from the microcline feldspar studied elicited a linear response from the orthoclase.

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PR Ann Wintle

Reviewers Comments

This paper is a further example of the complex responses of optically stimulated luminescence (OSL) signals from the two commonest types of potassium feldspar. Although using museum specimens guarantees the uniformity of the sample aliquots, compared to those extracted from sediments by settling in heavy liquids, it has the drawback that the natural OSL signals (IR and green stimulated) cannot be used for aliquot-to-aliquot normalisation. In this study the responses after heating to 500°C were used and there is the possibility that a dose dependent sensitivity change could have occurred; one which decreases with dose would have resulted in the corrected curve being supralinear. On the assumption that this is not the case, and that the normalisations are valid for all data sets, it suggests that errors of 20 Gy may occur in the course of laboratory dating procedures when microcline is the dominant potassium feldspar.

Authors' response:

Fortunately it is possible to give some further information on the possibility of dose dependent sensitivity change introducing supralinearity through the normalisation of the data. Sedimentation of the sample material onto the discs used for measurement was carried out on trays containing from 20 to over 100 discs. Some batches of simultaneously deposited sample discs were markedly more uniform than others. The discs used for the microcline SOL bleached measurements were uniform within about ±5% according to the normalisation measurements, which was regarded as an insignificant variation. Consequently no normalisation was in fact applied to this data which provided figures 2, 3 and 4 and the case of the infra red stimulation show clear examples of supralinearity (figure 4). On the other hand quite significant normalisation was required with the orthoclase material, figures 7, 8 and 9 with apparently linear responses for all treatments tested.

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A cautionary note: use of 'water content' and 'depth for cosmic ray dose rate' in AGE and DATA programs

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The AGE program was written by me several years ago for calculating TL ages. A similar program, DATA, has been used for calculating ESR ages. In a recent discussion, Prof. Prescott brought to my attention that the conventional water content of soil is defined as the ratio of the water mass over the dry mass of a soil sample (e.g. Foth 1984). This makes particular sense as all other analyses are subsequently carried out on the dry fraction of the soil sample (e.g. elemental composition, alpha, beta, and gamma activity, etc).

In the AGE and DATA programs, however, the sediment water content is defined as water mass over the mass of the collected, wet soil sample. For example, if the weight of a 100 g soil sample is reduced by heating at 105°C to a constant mass of 75 g, the present program input, X, would be 25%. The program then calculates water attenuation coefficients, W_β, according to the following formula (adapted from Bowman, 1976 and Aitken, 1985: section 4.2.3.):

$$W_{\alpha,\beta,\gamma} = \left\{ 1 + H_{\alpha;\beta;\gamma} \frac{X}{100-X} \right\}^{-1}$$

where $H_\alpha = 1.49$, $H_\beta = 1.25$, and $H_\gamma = 1.14$.

However, using water/dry mass the water content would be 33% (25 g water over 75 g oven dry soil). The use of this larger value will lead to overestimations of the attenuation factors, if inserted in the present version of the program.

The Table below shows the differences resulting from this problem.

In other words, if the water content in its conventional definition is 10% and this value was used in the program, the calculated beta and gamma dose rates would be about 1.2% smaller than the correct dose rates. The resulting age may be up to 1.2% older than a correctly calculated one. The problem becomes significant for higher soil moisture contents. At 40% water content, the program will calculate dose rates that are nearly 20% smaller than the correct ones.

A new version of AGE is available that uses water content expressed as % water over dry mass. The old version can still be used provided water content is expressed as water/wet mass.

water/oven dry soil (wd) %	water/wet soil (ww) %	$\dot{D}_{\beta\text{-wd}}/\dot{D}_{\beta\text{-ww}}$ %	$\dot{D}_{\gamma\text{-wd}}/\dot{D}_{\gamma\text{-ww}}$ %
10	9.1	98.8	98.9
20	16.7	95.2	95.8
30	23.1	89.6	90.1
40	28.6	81.2	82.9

Some newer versions of AGE and DATA have a depth, d, input for the cosmic ray dose rate. The program assumes an average sediment density of 2 g cm^{-3} and applies the formula of Prescott & Hutton (1988):

$$\dot{D} = 210 \exp(-0.07x + 0.0005x^2)$$

The dose rate is expressed in μGy a^{-1} and x is in units of 100 g cm^{-2}. This formula as used in the programs is only valid between 0.75 and 25 m. The current programs do not consider the soft component of the cosmic dose rate which is of relevance for depths less than 0.75 m. The input of "0" returns a zero cosmic ray dose rate. A version of the program is available on request which allows the user to insert the total cosmic ray dose rate directly.

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PR John Prescott

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Compiled by Ann Wintle

Thesis Abstracts

On the suggestion of Dave Huntley, Simon Fraser University, abstracts for two recent theses are included below and it is hoped that other laboratories will wish to keep the field informed of new and recent theses by sending further details to the Editor.

• Thesis title: Luminescence Dating of Quartzite from Diring Yuriakh
Awarded by: Simon Fraser University
Author: M. P. Richards
Date: 1994   Degree: MSc

Abstract
The lower cultural stratum (stratum 5) at the Diring Yuriakh archaeological site, in Siberia, Russia, contains crude stone tools, which, it has been suggested, were made before the currently accepted earliest occupation of Siberia in the Upper Palaeolithic (ca. 35,000 years ago). The stratum 5 artifacts are mostly quartzites, which contain a great deal of quartz. The artifacts lie on a deflation surface, and show evidence of wind abrasion, so it is likely that they were exposed to sunlight for a period of time before being buried by the overlying sediments. Experiments were undertaken to see if these quartzites could be dated using luminescence dating techniques similar to those that have been used to date the last time quartz grains extracted from sediments were exposed to sunlight. A method was developed to extract quartz grains from the quartzites, layer by layer, using successive 30 minute treatments of 50% hydrofluoric (HF) acid. By comparing the luminescence signal of quartz grains from each layer of quartzite recently exposed to sunlight, it was found possible to determine how deep into the quartzite the sunlight penetrated sufficiently to reduce the luminescence to zero. This allows selection of quartz grains for luminescence dating that should have been exposed to sufficient sunlight in the past. Dating attempts were made on quartzite samples from stratum 5 and stratum 2 (a deeper noncultural stratum), and it was found that the traps in the quartz grains were in saturation. After determining the radiation dose required to saturate the traps the dose-rate was calculated for each sample and then minimum age limits were determined. A stratum 2 quartzite was found, using thermoluminescence, to be last exposed to sunlight more than 150 ka ago. A stratum 2 control sample, which was expected to be in saturation on the basis of its supposed age, was found not to be in saturation, and yielded an equivalent dose of 440 ± 90 Gy, using 1.4 eV excitation. The evidence presented for a stratum 5 quartzite suggests it was last exposed to sunlight over 74 ka ago.

• Thesis title: Infrared Optical Dating of Organic-rich Sediments
Awarded by: Simon Fraser University
Author: Jinsheng Hu
Date: 1994   Degree: MSc

Abstract
The dating of sediments using infrared stimulated luminescence has drawn very much attention in recent years, especially because of the simplicity of the apparatus by which such luminescence can be measured. On the other hand, the applicability of the technique for age determination has not yet been demonstrated satisfactorily so far. This work is an attempt to investigate whether or not the infrared optical dating technique could produce correct ages for organic-rich sediments. Five of the seven samples studied yield satisfactory ages ranging from 0 up to about 100 ka. Two of the samples are of well-established ages while another three yield ages which are consistent with other evidence. It was also found that inhomogeneities in this type of sediment can be large enough to destroy the validity of the optical age. For the oldest sample studied (the age of which is between 0.78 and 1.06 Ma), the optical age was found to be 706 ± 98 ka.

Additionally, a possible lower limit of 290 ± 30 ka was obtained for the age of a sand sample collected from the Diring Yuriakh stone tool site.

Notices

UK
Luminescence Dating
Meeting
19-20 December 1995
Sussex University

For further details please contact
Professor Helen Rendell
Geography Laboratory, University of Sussex

● **Luminescence Spectra of Minerals by B.S. Gorobets**

From John Prescott, University of Adelaide
I have recently acquired a copy of a very rare publiation in Russian:
B.S. Gorobets 1981, Spektry Lyuminestsentsii Mineralov , Moskva, Ministersvo Geologii SSSR
[B.S. Gorobets 1981 Luminescence Spectra of Minerals Moscow, Ministry of Geology]

It is a compendium, mostly of work in the then USSR, based on observations on over 200 minerals. The bibliography covers references from 1970 to 1982. It appears to have been intended for internal use because the print run was only 400 copies and it is now out of print. An English translation of the Table of Contents and the Summary follows. Lest there be any misunderstanding, this is the only part of the document available in English translation.

I would be happy to provide copies of the Russian original for postage for the cost of copying and airmail postage: Australian dollars $20 paid in advance.
J.R. Prescott, Physics/ Mathematical Physics, University of Adelaide, Australia 5005.

ABSTRACT

B.S. Gorobets, Luminescence spectra of minerals.

(Technical recommendations of the scientific committee for the study of minerals. VIMS MINGEO USSR) 1981

10 folios [pp 153], 220 figures, 24 tables.

This is a summary of technical knowledge on studies of the luminescence spectra of minerals, produced with the aim of expediting diagnostic use and analysis of typomorphic features development of rational schemes for radio metric classification. It begins with a short note on the physical basis for the occurrence luminescence in minerals, its dependence on the crystal chemistry the latter, and on geological-genetic factors in mineral formation. Apparatus and methods for recording photo luminescence (PL) spectra and radio luminescence spectra (RL) are described. A method of interpreting spectra is based on a "standardised" form of the PL and RL spectra, obtained at VIMS or found in the literature. Altogether there are about 700 spectra, representing roughly 400 specimens of 240 varieties of minerals, at 77K and 300K; luminescence bands are attributed to specific luminogenic impurities or to intrinsic luminescence. Seventeen tables contain summarised data on the frequency of occurrence of different luminescence centres in minerals. The tabulation describes the differing frequency of occurrence of luminescence centres (rare earths and manganese) in apatite, fluorite and calcite, as well as scheelite from deposits of different genetic origins. These data allow decisions on the further question of the qualitative nature of oxidising-reducing, acid-basic conditions and the concentration of rare and dispersed elements in the process of mineral formation. The technical recommendations are intended for a wide circle of mineralogists and for specialists in radiometric ore processing.

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