Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Galloway, R. and Napier, H., 1994. *The influence of sample treatment on feldspar dose response.* Ancient TL 12(2): 43-49. https://doi.org/10.26034/la.atl.1994.228

The influence of sample treatment on feldspar dose response

R. B. Galloway & H. J. Napier

Department of Physics and Astronomy, University of Edinburgh, Mayfield Road, Edinburgh EH9 3JZ, Scotland.

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.

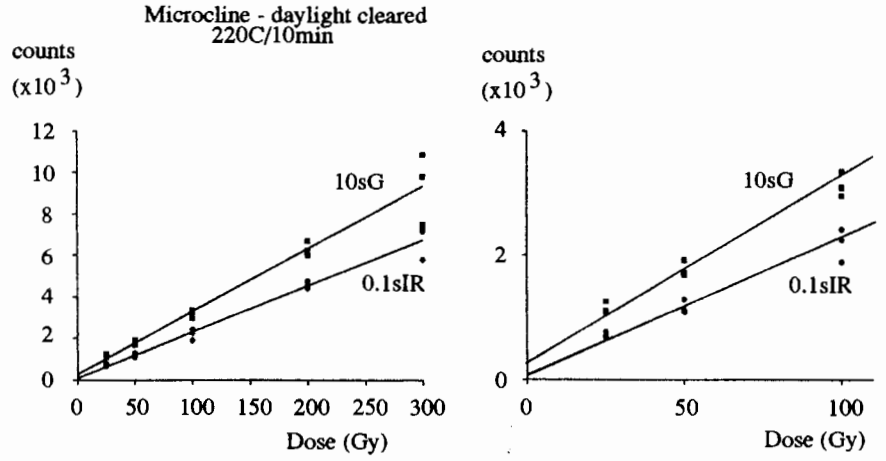

Figure 1 .
(a) The luminescence dependence on dose for daylight bleached microcline feldspar preheated at 220°C for 10 mins and stimulated by infra red and by green light. Measurements were made on 15 aliquots. The counts axis indicates the number of photon counts recorded in 0.1 s of infra red stimulation 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.

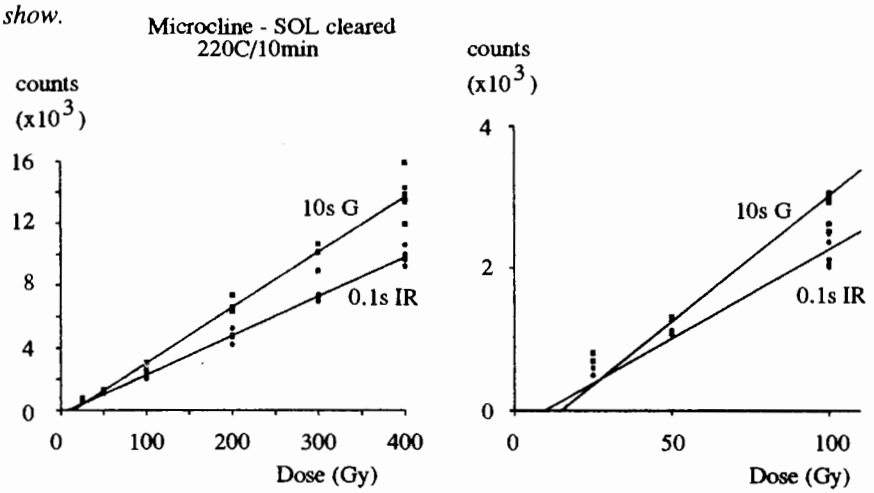

Figure 2.
(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator, based on 23 aliquots and in a similar format to Fig. 1. Preheating was at 220°C for 10 mins. Plot (b) of the low dose region shows that the response for both infra red and for green stimulation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

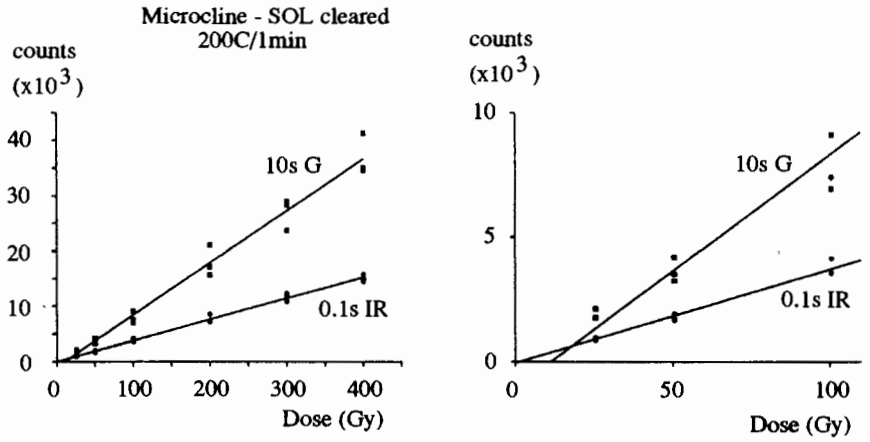

Figure 3.
(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator with preheating reduced to 200°C for 1 min.. 18 aliquots were used. The low dose plot (b) shows that supralinearity persists for the green stimulation.

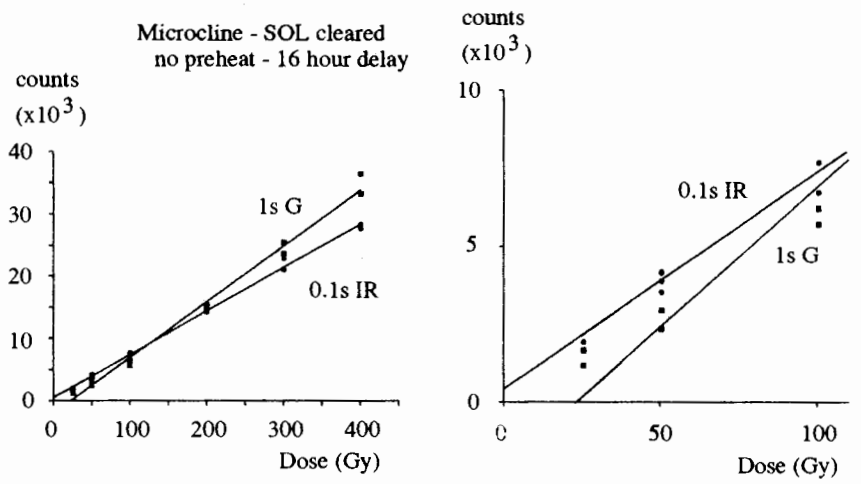

Figure 4 .
(a) The luminescence dependence on dose for microcline feldspar bleached in the SOL 2 solar simulator and with no preheating but with a delay of 16 hours between dosing and reading. Green stimulation was for 1 s in this case and 13 aliquots were used. The low dose plot (b) shows supralinearity for green stimulation but not for infra red.

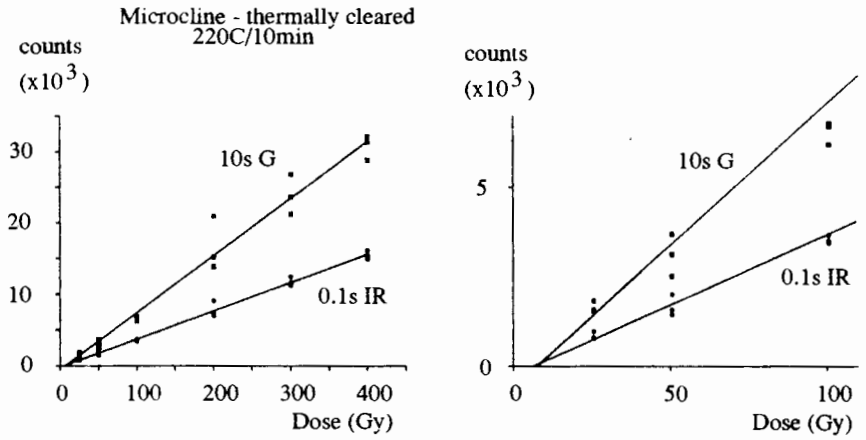

Figure 5 .
(a) The luminescence dependence on dose for microcline feldspar bleached by heating to 500°C and with preheating after beta dose at 220°C for 10 mins. 18 aliquots were used. The enlarged low dose plot (b) shows the possibility of a small degree of supralinearity.

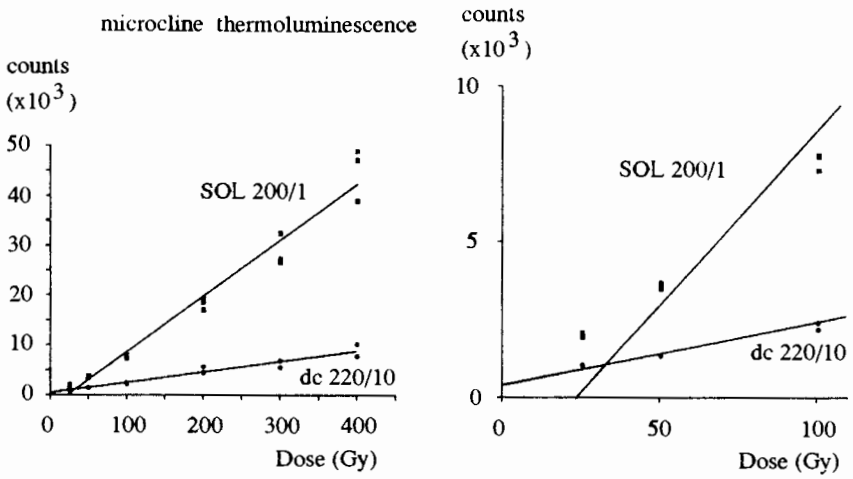

Figure 6.
(a) The TL dependence on dose for microcline feldspar, comparing daylight bleached material preheated at 220°C for 10 mins (12 aliquots) with SOL 2 bleached material preheated at 200°C for 1 minute (18 aliquots). The enlarged low dose plot (b) emphasises the acceptable linearity of the daylight bleached material and the supralinearity of the SOL 2 bleached material. The counts axis indicates counts recorded in 0.4 s.

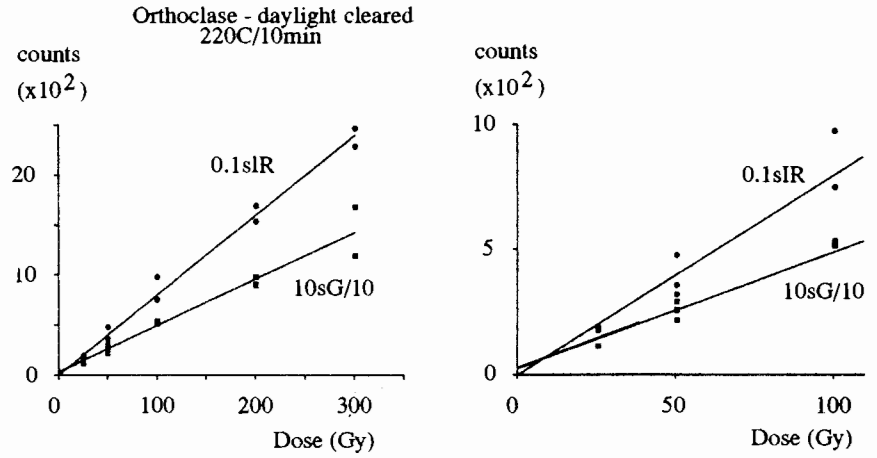

Figure 7.
(a) and (b). For orthoclase feldspar, daylight bleached and preheated at 220°C, the luminescence response to dose for both infra red and green stimulation is linear. The counts axis for the green stimulation indicates counts per second for the orthoclase material. 12 aliquots were used.

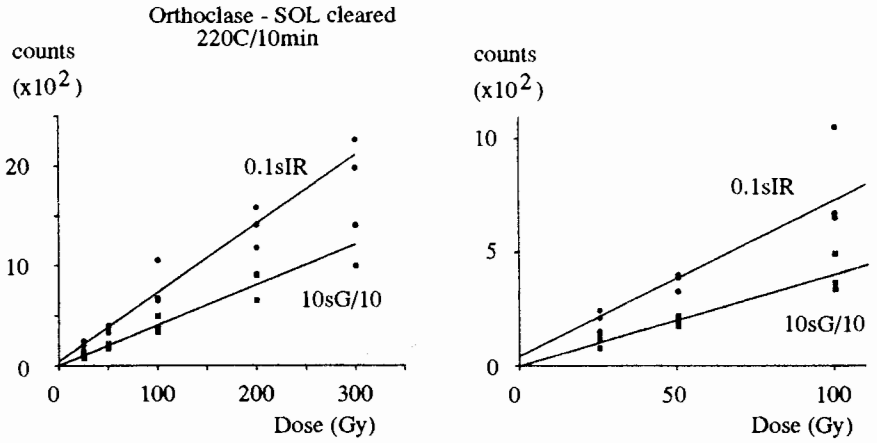

Figure 8.
(a) and (b). For orthoclase feldspar, bleached in the SOL 2 and preheated at 220°C for 10 mins, the luminescence response to dose for both infra red and green stimulation is linear, in contrast to the microcline response in figure 2. (14 aliquots were used).

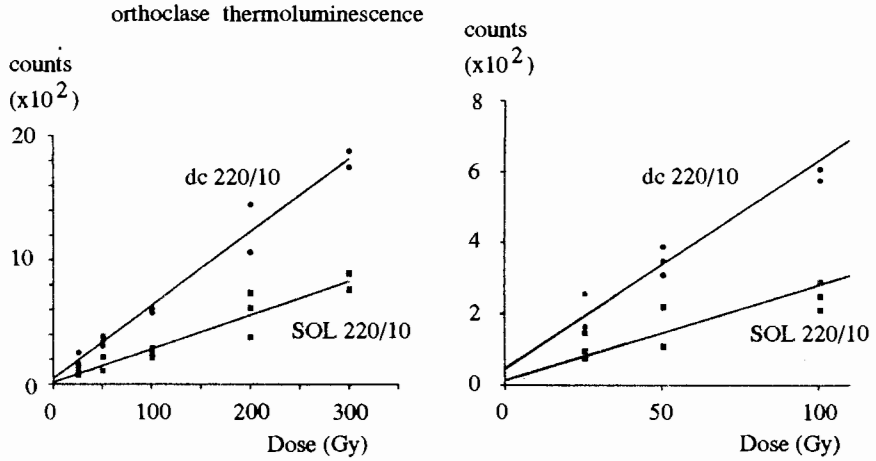

Figure 9.
(a) and (b). For orthoclase feldspar, the TL response for daylight bleached and SOL 2 bleached material compared. 11 and 14 aliquots respectively were used, with preheating at 220°C for 10 mins in both cases.

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.

References

Duller, G. A. T. (1991) Equivalent dose determination using single aliquots. *Nucl. Tracks Radiat. Meas.* **18**, 371-378.

Duller, G. A. T. and Bøtter-Jensen, L. (1993) Luminescence from feldspars stimulated by infrared and green light. *Radiat. Prot. Dosim.* **47**, 683-688.

Fleming, S. (1979) Thermoluminescence techniques in Archaeology (Clarendon Press, Oxford) pp 66-69.

Galloway, R. B. (1990) Notes on a recently constructed TL system. *Ancient TL* **8**, 10-11.

Galloway, R. B. (1991) A versatile 40-sample system for TL and OSL investigations. *Nucl. Tracks Radiat. Meas.* **18**, 265-271.

Galloway, R. B. (1992) Towards the use of green light emitting diodes for the optically stimulated luminescence dating of quartz and feldspar. *Meas. Sci. Technol.* **3**, 330-335.

Galloway, R. B. (1993) Stimulation of luminescence using green light emitting diodes. *Radiat. Prot. Dosim.* **47**, 679-682.

Galloway, R. B. (1994) Comparison of the green and infra red stimulated luminescence of feldspar. *Radiation Measurements* **23**, 617-620.

Godfrey-Smith, D. I. (1993) Thermal effects in optically stimulated luminescence. *7th Int. Specialist Seminar of Thermoluminescence and Electron Spin Resonance.*

Li, S-H. and Wintle, A. G. (1992) Luminescence sensitivity change due to bleaching of sediments. Nucl. Tracks *Radiat. Meas.* **20**, 567-573.

Li, S-H. and Wintle, A. G. (1993) A model for sensitivity change of IRSL signals. *Ancient TL* **11**, 33-35.

McKeever, S. W. S. (1985) Thermoluminescence of solids (Cambridge University Press) Ch. 3.

McKeever, S. W. S. (1991) Mechanisms of thermoluminescence production: some problems and a few answers? *Nucl. Tracks Radiat. Meas.* **18**, 5-12.

Rees-Jones, J. and Tite, M.S. (1994) Recuperation of IRSL after bleaching and consequences for dating young sediment. *Radiation Measurements* **23**, 569-574

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.