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The use of an image intensifier to study the TL intensity variability of individual grains

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When using 100μm quartz grains in TL dating a difficulty commonly experienced is poor disc-to-disc reproducibility, a major cause of which is intrinsic sample variability. Because this occurs when there are several hundred grains per disc, it can be deduced that most of the TL must arise from only a few per cent of the grains.

In order to test this deduction we have constructed a simple apparatus that gives a semi-quantitative light intensity distribution of a large number of grains and permits sorting the grains for further study.

The apparatus is shown in Figure 1. The sample chamber was an Oxford-style glow oven with a Wild BG-38 filter (pass band 320-630nm) to remove the incadescence emission. Above this an f1.4 camera lens was mounted with a reverse adapter on the bottom of a bellows unit. At the top of the bellows an image intensifier was placed and its position adjusted so that an image of the sample was focused on its input. The glowing grains were readily visible at the upper phosphor surface of the intensifier and could easily be photographed with the camera shown in the upper part of the figure.

The image intensifier used was a Varo model 3603 from Varo Inc. 2203 W. Walnut St., P.O. Box 469014, Garland, Texas 75046-9014, U.S.A. It had a gain of $> 10^5$ from 400-800nm, operated on two 1.5V cells and cost ~US$2000.

Small chips of CaF_2:Dy dosemeter crystals were used for set-up and focusing. After being given a large dose (> 1kGy) their phosphorescence was visible for over two weeks.

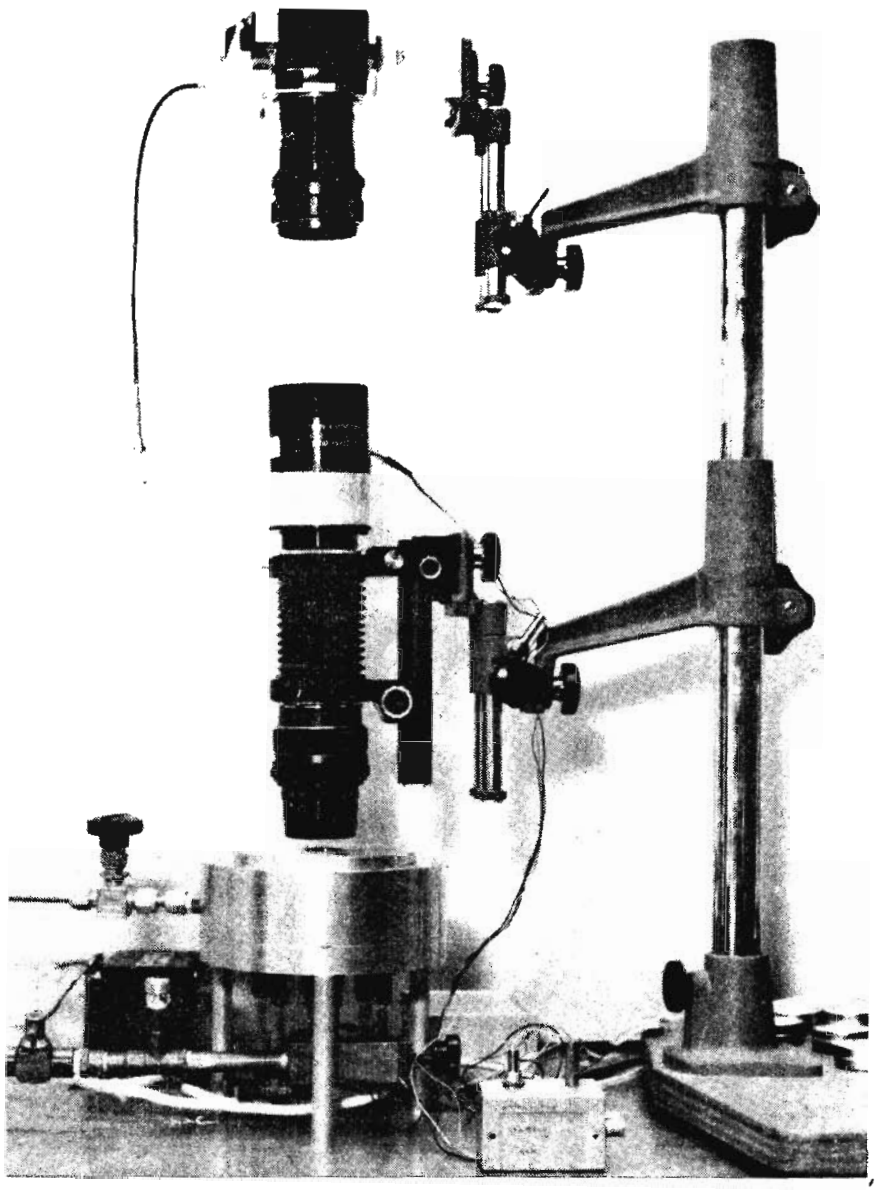

Figure 1

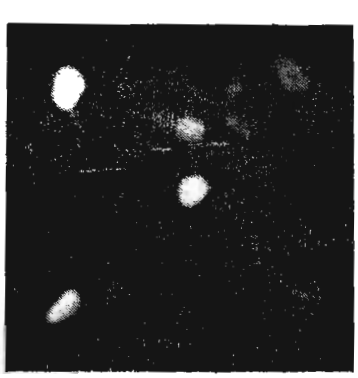

Figure 2a

Figure 2b

Figures 2a and 2b are photographs of the intensified natural TL of 25 quartz grains, 0.5-1mm in diameter, extracted from a 120ka South Australian beach dune. Figure 2A is the TL from 250-400°C and Figure 2b the TL from 400-450°C. Although many grains are visible, most of the light is coming from 2 or 3 grains. It is also interesting to note that the brightest grain (upper left) in the first picture is barely visible in the second one. Photographs of discs containing several hundred 100μ quartz grains of the same sample also indicate that the TL is dominated by a few bright grains.

We know of only two previous attempts to determine an intensity distribution, those of McKerrell and Mejdahl (1981) and Benko (1983) who measured the TL of individual grains. Because of the small number that they measured they could have missed the important bright grains. The idea that only a small percentage of the grains contribute significantly to the measured TL was put forth several years ago by D. Zimmerman (D. Stoneham, private communication, 1983) yet has received little discussion in the literature. The cause and implications of this variability remain to be investigated.

A sensitive apparatus for photographing TL has been described previously by Debenham and Walton (1982); our system is much cheaper, simpler to construct and easier to use.

Acknowledgements

We thank A. Walton for assisting with a pilot experiment using his image intensifier in Oxford in 1983, M. L. W. Thewalt for the loan of the Varo image intensifier, and P. M. Hobler for the loan of some of the photographic equipment. The work was supported by the Natural Sciences and Engineering Research Council.

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Reviewer's Comments (M. J. Aitken)

This follow-up of the pilot work with Alan Walton shows an important new facet. This is that grains which are bright in one temperature range may be dim in another range. This presumably explains why Zero Glow Monitoring (ZGM) using the 110° peak is often found to be unsatisfactory as a way of normalizing the high temperature TL of different portions of 100-micron quartz grains.

Another aspect of single grain investigations is grain-to-grain variability in anomalous fading. Relevant reports have been made by Hoyt et al. (1972), Sutton and Zimmerman (1976, 1979) and Templer (1985).

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Preliminary study of the thermoluminescence behaviour of quartz from a Dutch cover sand

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Introduction

The object of this study was to determine wheter it is possible to obtain reproducible and meaningful TL measurements on quartz grains from cover sands before embarking on a dating study. Therefore we examined the bleaching characteristics of quartz. A similar study was presented at the Fourth Specialist Seminar on TL and ESR dating by Jungner (1985) on post-glacial sand dunes.

The sample discussed here is a well sorted wind-blown sand with a mean grain size of 150-210 micron taken at a depth of 2.45 m at Loozerheide, near Weert in the southern part of The Netherlands and covered by several paleopodzols developed during the Holocene in aeolian sands (fig. 1). A radiocarbon date of the lower soil, approximately 60 cm above the sample, will be available soon, but is expected to be around 7,000 years. Based on lithostratigraphic criteria the underlying aeolian sand is considered to be part of the Younger Cover sand II deposit of post Allerod age. The expected age of the sample therefore is in the range of 7-11,000 years. For a preliminary study the 125-150 micron grain size fraction was treated with 30% H_2O_2 for 24 hours, although hardly any organic matter was present, and subsequently with 40% HF for 1 hour at room temperature to remove any feldspars as well as to etch the quartz grains. The resulting grains were washed thoroughly and dried. Aliquots of 4-6 mg were weighed and evenly spread onto 1 cm

aluminium discs, which had to be sprayed with a silicone-base spray to prevent sample removal during measurement. The glow curves were weight-normalised for a 4 mg aliquot.

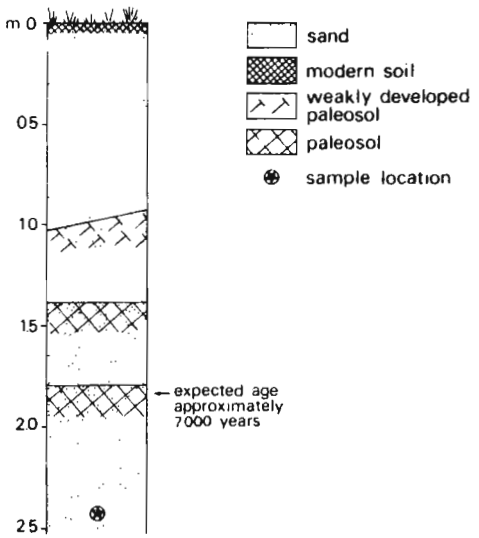

Figure 1 *Schematic diagram of the section at Loozerheide.*

TL Measurements

Measurements were made at $5^{o}C/s$ using an EMI 9635 photomultiplier tube and a Corning 5-58 filter to enhance the quartz signal. Wakefield thermal compound was found to be essential for thermal contact between the disc and the heating plate at this heating rate, otherwise disc-to-disc shifts of up to $20^{o}C$ occurred.

Curve 2 in figure 2A is the natural TL (N.B. all black body signals have been subtracted) and curve 1 shows the effect of 28.3 Gy additional dose. These are similar to the graphs of Jungner (1985). Curve 3 is the TL that is measured after the grains containing their natural TL have been exposed to an unfiltered mercury sunlamp for 60 hours at 35 cm distance. A phototransfer peak occurs at $170^{o}C$. Comparison of the glow curves at $360^{o}C$ indicates that the natural TL is equivalent to the TL induced by some 30 Gy which is at least an order of magnitude greater than expected for this sample. This is caused by laboratory overbleaching with wavelengths not present in the original bleaching spectrum. This was supported by attempts to measure the ED by the R - beta method using a 30 minute partial bleach which resulted in the intersection of the two growth curves occurring at a negative TL intensity value.

Using a Black Light lamp from Philips, Jungner (1985) found that the natural TL bleached faster for quartz than a similarly treated feldspar sample. However, his use of natural TL would have incorporated a UV bleachable component not bleached at the time of deposition. We felt that it was more appropriate to obtain the bleaching characteristics for quartz which has been well bleached and given a known laboratory radiation dose (in this case 60 hours UV exposure followed by 31.4 Gy dose). Weight normalization gave rise to less scatter than second glow normalization (as used by Jungner) but reproducibility better than about 10% could not be achieved. The results are given in figures 2B and 3.

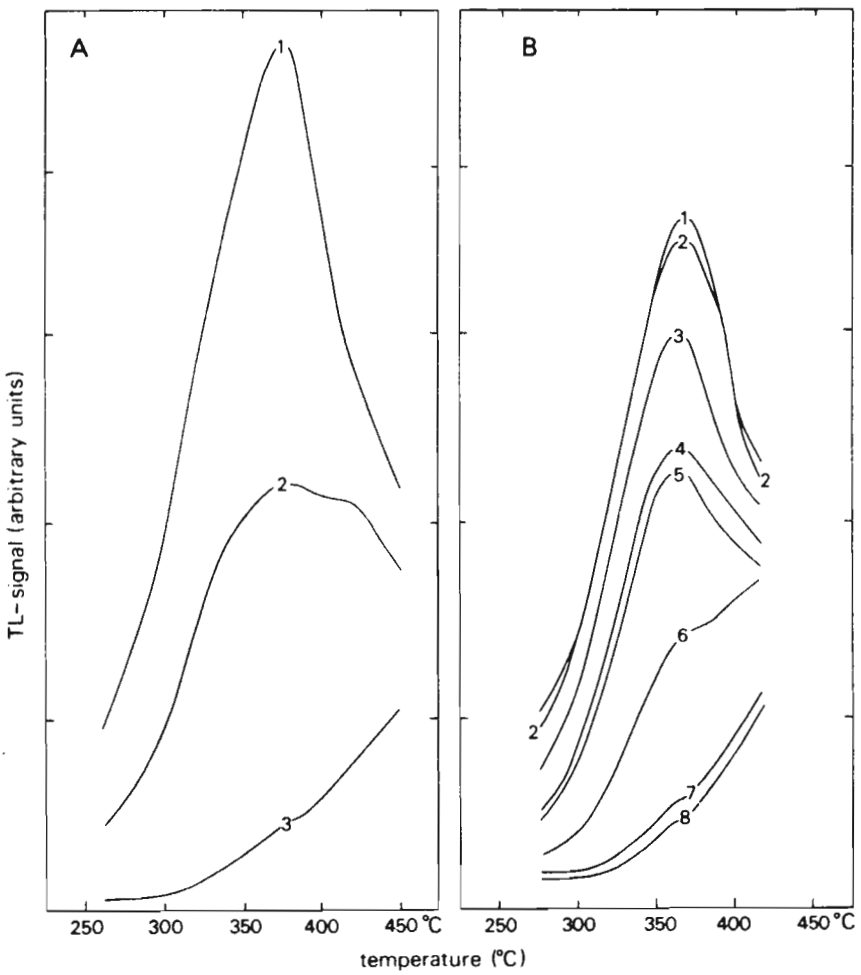

Figure 2: (A) Glow curves of the 360°C quartz peak for 1. Natural TL + 28.3 Gy beta dose, 2. Natural TL, 3. Natural TL + 60 hours bleaching.

(B) Glow curves (scale is identical to that of (A)) of the radiation induced 360°C quartz peak with the following subsequent bleaching times: 1) 0 minutes, 2) 5 minutes, 3) 15 minutes, 4) 30 minutes, 5) 1 hour, 6) 2 hours, 7) 8 hours and 8) 20 hours.

In figure 3 the percentage of bleachable TL as a function of bleaching time (the 0% level is the TL as shown in fig. 2A, curve 3) is compared with that obtained for the 330°C TL of a separated feldspar (Kronborg, 1983) and that obtained for 300°C TL of polymineral fine grained sediment (Wintle and Huntley, 1979). Kronborg (1983) found that if the data were plotted on a log-log scale, an almost straight line could be drawn through them, but this is not the case for the present data (fig. 4). We found some 40% reduction in the radiation induced signal is attained after about 30 minutes exposure and this would be a reasonable exposure time to select for the partial bleach (R - beta) method as long as the original spectrum was similar. Much slower bleaching was achieved for this sample with a Corning 3-67 filter directly in front of the discs during the 60 hours sunlamp exposure as suggested by Huntley et al. (1983). Moreover no phototransfer peak

occurred when this filter was used. Other bleaching curves have been obtained with an ultraviolet lamp for natural and gamma induced TL from sand (Vlasov et al., 1979); they concluded that the TL is reduced to less than 1% of its initial value by a light exposure of 70 kcal/cm, equivalent to 1 polar day.

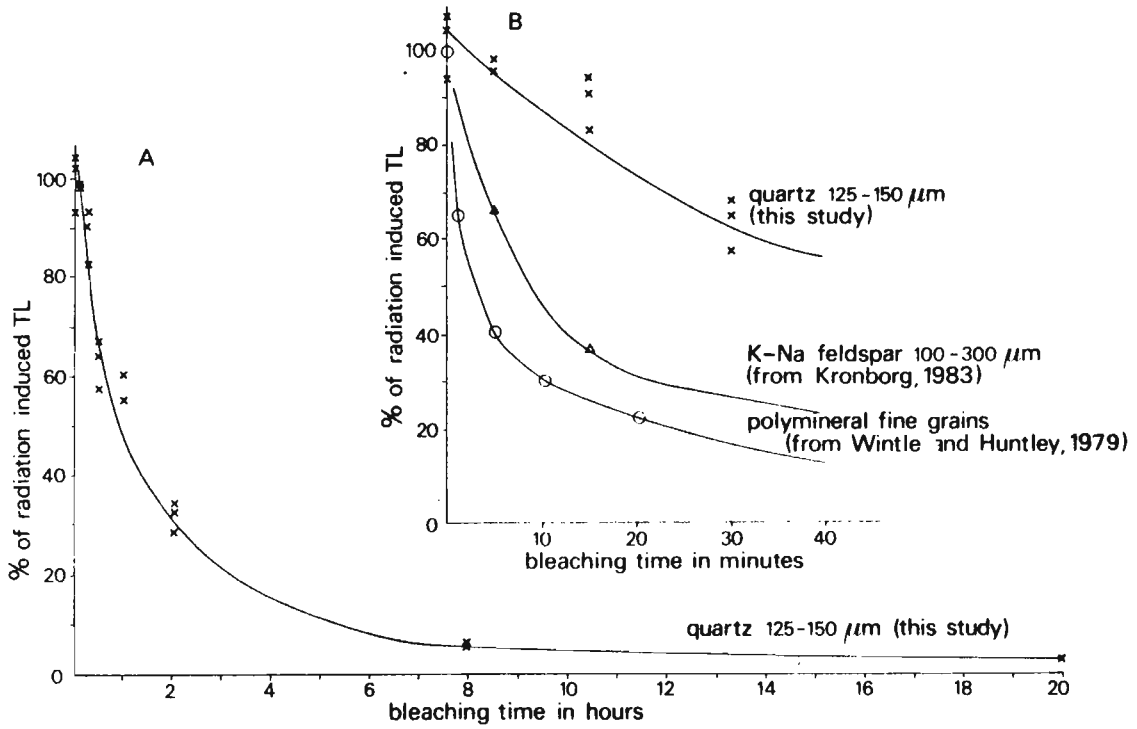

Figure 3: (A) Bleaching curve of the 360°C quartz peak showing the loss of radiation induced TL with increasing bleaching time. To obtain radiation induced TL all samples were bleached for 60 hours (0% level) and were subsequently given a 31.4 Gy beta dose (100% level).

(B) Same bleaching curve as (A) compared with other studies.

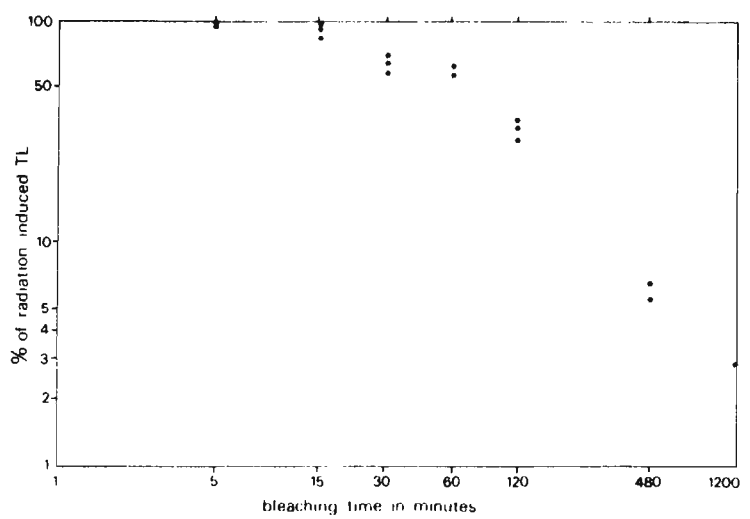

Figure 4: As figure 3 (A) but plotted on a log-log scale.

Conclusions

(A) Most of the 360°C quartz TL signal is bleachable provided that long enough exposure times are used with a broad spectrum source.

(B) Use of an unfiltered medium pressure mercury lamp is unsuitable for the regeneration, total bleach and R - beta methods for the quartz from this kind of sediment, because it induces overbleaching. It should be noted that this problem was not encountered by Singhvi, et al. (1983) who studied quartz extracts from sand dune in Rajasthan, India.

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Reviewer's comments (V. Mejdahl)

An important problem in TL dating of sediments is to define those types that are suitable for dating. Considerable success has been achieved in dating loess (Debenham, 1985 and Wintle et al., 1984), and aeolian sand deposits such as dune and cover sands also appear promising.

The present study of quartz from cover sand brings out one of the difficulties in dating quartz from a young sediment, namely, that it has been deposited with a residual TL level that constitutes a large proportion of the present natural level. Determination of the residual level by prolonged laboratory sunlamp bleaching (or sun bleaching) therefore results in severe overbleaching.

I have two specific comments on the paper:

1. The discussion related to Figure 2(A) is unclear. It is true that the natural TL level corresponds to about 30 Gy as estimated from the figure, but the laboratory overbleaching must relate to the TL accumulated since deposition, that is the difference between curves 2 and 3, which corresponds to about 21 Gy. Assuming a dose rate of 1.2 Gy/ka, 21 Gy yields an age of 18 ka. If the expected age, 7-11 ka, is correct, then the true residual TL would be roughly 3 times that in curve 3 or about 60% of the natural level. This agrees well with the residual levels that I found for quartz from cover sand from Jutland (Mejdahl, 1985). Considering this high residual level, it is uncertain whether quartz from such young sediments can be dated with sufficient accuracy. However, it can be used together with K feldspar to determine the much lower residual level in feldspar (Mejdahl, 1985).

2. Bleaching experiments. It is important to note that the bleaching rate depends strongly on the initial TL level when the residual level is expressed as percentage of the initial level. Therefore, a comparison of bleaching rates is meaningful only when the minerals have the same initial level. It should further be stressed (and the authors are well aware of this) that laboratory bleaching using an unfiltered sunlamp does not reflect correctly the bleaching taking place in nature; this is particularly true for quartz.

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An indication of universal linear variation of K_2O percentage with beta dose rates in ceramics : preliminary results

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Introduction

In geology it has been repeatedly documented that there is an almost universal tendency for an increase in the concentration of uranium and thorium in proportion to the concentration of later members - potassium, for example - in any "igneous differentiation series" (the migration and concentration of elements during metamorphism so as to produce an inhomogeneous rock from an originally homogeneous one). (Peterman, 1963; Hamilton, 1959; Gottfried, 1959; and Heier and Rogers, 1978, in Handbook of Geochemistry, Vol. II, 5 (1978), ed. K. H. Wedepohl.) Accordingly it was thought interesting to look for such a relationship in archaeological clay artifacts.

When thorium ppm values are plotted against those of their counterpart potassium, the above-mentioned tendency is illustrated (see Fig. 1a). These data were derived from geological materials, from Skaergaard intrusion Greenland, Dillsburg sill, Pennsylvania and Duluth Complex, Minnesota (Peterman, 1963), and from archaeolgical materials from Greece (Liritzis, 1979).

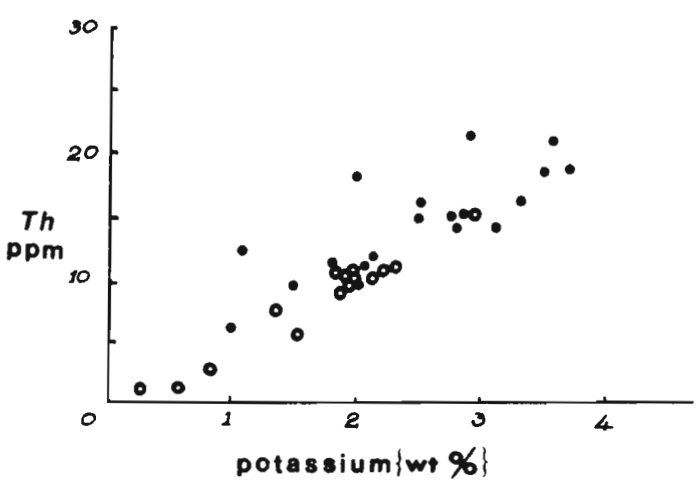

Figure 1a: Thorium (ppm) vs K for geological materials; opens circles (after Peterman, 1963) and from present studies (black dots).

As a result of this observation, it was thought of interest to extend such a study to learn if the K_2O percentages from the samples, plotted against their beta dose-rates (derived from their respective uranium, thorium, and K_2O values), are in proportion. It was considered worthwhile to plot the potassium content of each pottery and well-fired clay sample used in a TL-dating project (from materials throughout Greece), against its respective beta dose-rate values (mrads/yr) (Liritzis, 1979; Liritzis and Galloway, 1980; Liritzis, 1981). The beta dose-rates were measured by both the alpha-counting and potassium determination (α + K) as well as the TLD methods. But before discussing these plottings, some geochemical aspects of the distribution/correlation of uranium, thorium, and potassium in nature will follow.

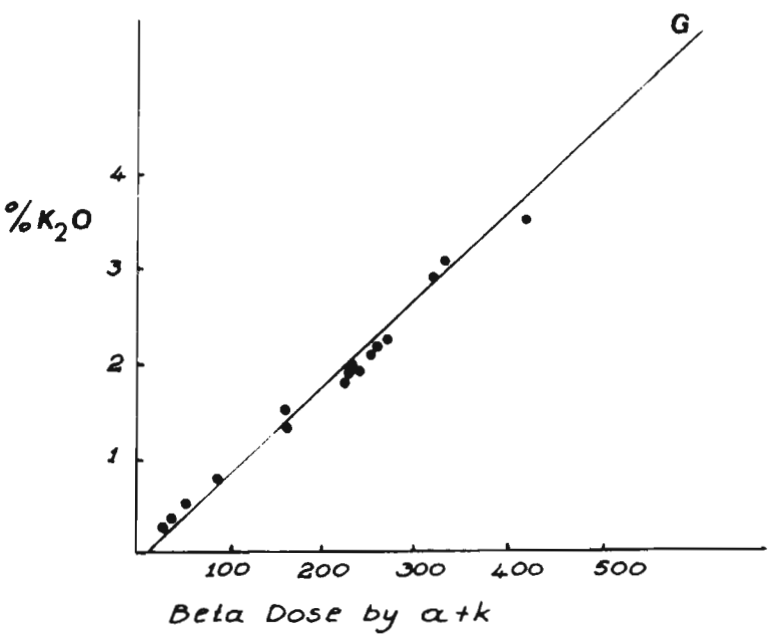

Figure 1b: β dose-rate and K_2O for geological materials.

Uranium, Thorium and Potassium Distribution/Correlation in Nature

Few studies directly examine the behaviour of thorium, uranium, and potassium during weathering and alteration. It is, however, known that the tendency of uranium to be oxidized to the comparatively soluble uranyl ion (UO_2^{++}) permits uranium to be mobilized easily in surficial processes. This oxidation is primarily responsible for the wide variations in Th/U ratios shown in surficial material - e.g. clays, sands, soils. The comparatively insoluable thorium is concentrated in resistate minerals or is adsborbed on clays, whereas uranium is redistributed in surface and ground waters.

The almost universal tendency for the concentration of uranium to increase toward the later members of an igneous differentiation series has been documented mainly from plots of uranium content against some type of differentiation index such as the potassium content. The consistent trends are shown by most extrusive series than by instrusive rocks (plutonic, i.e., deeply buried igneous structures).

Similar observations have been made for the K/Th ratio, which has been found to have a nearly constant value in a large variety of igneious rocks, sediments, and surficial materials. Although the precise location of uranium and thorium in rock-forming minerals - e.g. allanite, monazite, feldspars, epidote and zircon is yet uncertain, for geochemical reasons these three radio-elements are highly correlated in these rock-forming minerals. The weathering that follows diagenesis, transportation, mobilization/ differentiation of radioisotopes and sedimentation presumably controls the abundance of these radioactive elements, and one may expect a correlation of their distribution/concentration with climatological variations.

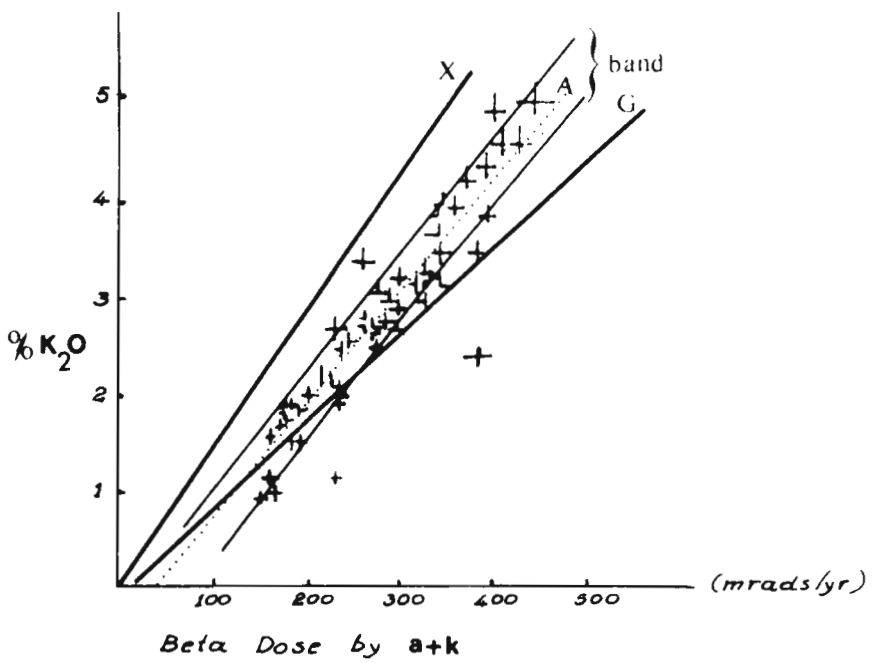

Figure 2: Plotting β-dose rate (α + K) vs K_2O.

Discussion

Fig. 2 plots the K_2O percentages against the beta dose-rates - derived by (α + K) - and exhibits a linear distribution, as most of the points with their standard errors lie along curve A (drawn from the least square method). There appears to be a higher scatter in the case of the beta dose-rates measured by TLD - that is, curve T, Fig. 3. For curves A and T, however, a quadratic fit is better than a linear - that is, the chi-square is lower. Nevertheless an exponential initial rise with subsequent linear response is more realistic in view of the shape of the curve, due mainly to the lack of points of low K_2O percentages. Scattered points might imply systematic errors due to some prior assumptions regarding the method (Liritzis and Galloway, 1981).

If there were no thorium and uranium present in a sherd, the X-curve - that is, the beta dose derived from ^{40}K against K_2O% - of Figs. 2 and 3 would indicate the lower linear boundary of the expected uniform distribution. With the presence of thorium and uranium, the corresponding distribution would incline toward higher beta dose values. There is no reason to suppose that typical clay

minerals have depleted thorium and uranium and NAA has shown the opposite for clays, worldwide. Thus there should be no scattering of points to the left - that is, the lower part of the distribution curves - but only toward the right, or upper, parts. For excessive thorium and uranium, the points, then, would be expected to lie even further from the line of the "band" - that is, the curves A and T plus their associated error bars.

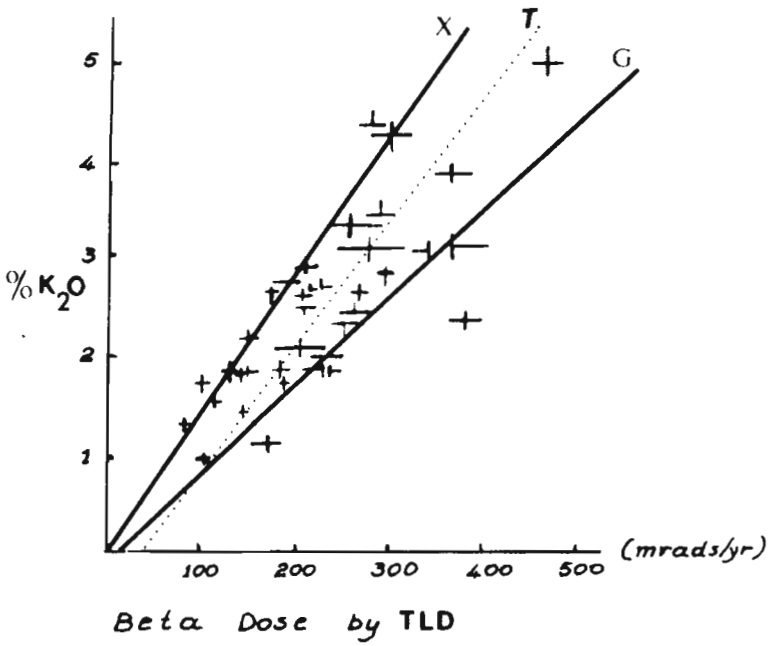

Figure 3: Plotting of β-dose rate (TLD) vs K_2O.

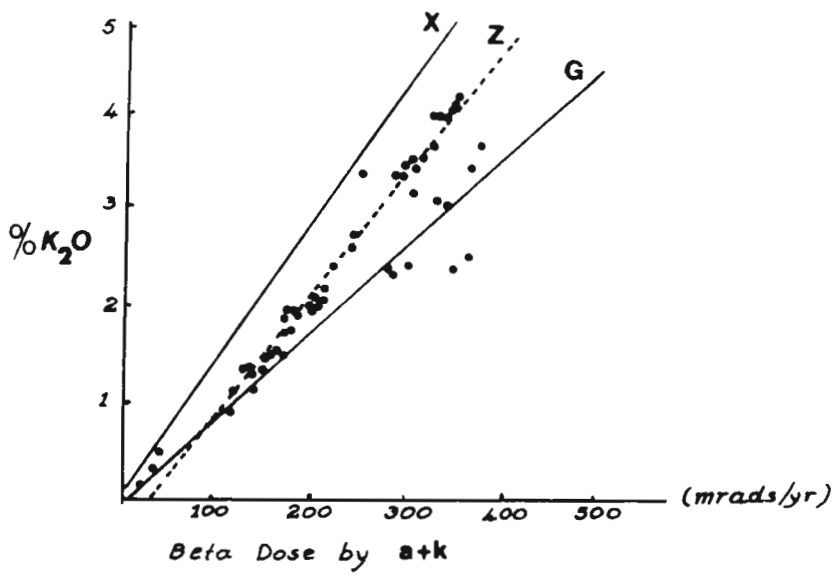

Figure 4 Plotting of β-dose rate (α + K) vs K_2O. Data taken from Zimmerman, Ph.D. thesis (1970).

In all of the above the line G is the best fit to the geological data of Fig. 1B.

Generally, the thorium and uranium correlate with the potassium in such a way that the result would be a curve with a slightly reduced slope compared to the X-curve, as the geological data (Fig. 1b) do not support a dose-rate from thorium and uranium which could be expressed as a curve parallel to X.

The above linear distributions were further substantiated by plotting the potassium contents of sixty-two sherds against their respective total beta doses (derived by α + K), the data being taken from Zimmerman (1970). His ceramics came from England, France, and Greece. For this the linear curve Z was obtained (Fig. 4). The geological curve (Fig. 1b) has a slightly lower slope that curves A, T, and Z, explainable as:

1. a result of the small number of geological samples analysed; or
2. because the geological analyses relate to variations of U, Th and K concentrations in three gabbro-granophyre sequences, which could obey different mechanisms than those in clays.

Overall, a linear distribution of K_2O percentage against beta dose-rates, with "narrow bands", could be considered as an indicator of their relationship, which can be used as a reference curve, in the sense that fairly satisfactory beta dose-rate values can be readily assessed by knowing only the potassium content of the sample. The novelty of this observation is that it can aid preliminary TL-dating by giving an approximation of the beta dose-values, thereby reducing the number of needed experimental measurements. (Note an unusually high beta value in Figs. 2 & 3.)

From the present preliminary results, besides the level of significance attached to each data point, the likely error resulting from use of the relationships of Table 1 can be evaluated. Thus for a value b of the true beta dose, the predicted one (at the 68% level of confidence) is given with a $\pm$ 4-5% error based on the error of the dose co-efficient of the relationships in Table 1. Drawing arbitrarily "narrow" bands around the A, T and Z curves that include most ($\geq$ 95%) of the experimental points, the errors in the beta-dose lie between approximately $\pm$ 6 to $\pm$ 15% for around 4% and 1% K_2O values respectively. Although this distribution has been approached by linear and by quadratic fitting (as well as for other function degrees for a more accurate functional representation), at present it is not possible to define a function that would take into account the lower regions of curve A.

Further data are, of course, needed to confirm such a correlation for the lower regions of the curve. At present this observation is further encouraging since the samples cover a wide range of provenance and they are not of local or limited origin.

Acknowledgements

I am thankful to Dr. J. Annand for supplying the computer program and also to the National Hellenic Research Foundation and the Royal Society, and to Dr. D. F. O. Russell of the Russell Trust for their financial assistance. I am grateful to Dr. M. J. Aitken for his useful comments.

Table 1

Data / Function	Geological	α + K	TLD	Zimmerman
Linear fit	$Y = 0.23 + 0.83\ x$	$Y = 0.604 + 0.76\ x$	$Y = 0.23 + 0.71\ x$	$Y = 0.36 + 0.80\ x$
	±0.10 ±0.038	±.01 ±.04	±.17 ±.08	±.1 ±.04
χ^2	0.658×10^0 (8.7)	0.103×10^3 (37)	0.15×10^2 (23.2)	0.87×10^3 (34)
Quadratic fit	$Y = -0.17 + 1.88x - 0.054x^2$ ±0.85 ±.058 ±.008	$Y = 0.57 + 0.79x - 0.005x^2$ ±.22 ±.18 ±.03	$Y = 0.43 + 0.52x + 0.04x^2$ ±.39 ±.34 ±.07	$Y = -0.086 + 1.27x - 0.1x^2$ ±.17 ±.15 ±.03
χ^2	0.159×10^0	0.105×10^2	0.154×10^2	0.75×10^3

Notes

The sets of points for the four figures were fitted to both linear and quadratic fits with the use of a program written in Fortran IV. The best fit amongst different degree polynomials was that of the second degree. The term x (β-dose, Dβ) is given as $10^{-2} \times D\beta$ and Y is given as % K_2O. Numbers in parenthesis are the critical χ^2 values at 95% confidence level. For $\chi^2_{calc.} < \chi^2_{crit.}$ the null hypothesis (i.e. calculated value close to a sample value) is not rejected, but the fitting function upon which the calculated values were obtained have a level of significance of 95%.

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P.R. Reviewed by M. J. Aitken and accepted after revisions.

Comments on extrapolation methods of dating sediments by TL

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In the last issue of Ancient TL, Mejdahl (1985) presented a TL age for a marine sediment from Kap Kobenhavn, Greenland, of 1.07 Ma. It was calculated from an equivalent dose (ED) obtained by extrapolating the first glow growth curve to zero TL. Since this old date exceeds the limiting value of 100 ka that I have observed in a study of N.W. European sediments (Debenham, 1985) he concludes that he is observing a TL signal which is more stable than the feldspar 400 nm emissions used by myself. In this contribution, I question whether the greater age of the Greenland sediment signifies greater signal stability, or merely results from the method of ED determination employed.

The assumption made when an ED is obtained from the first glow measurements alone is that the extrapolated curve retraces the original TL growth curve in antiquity. Mejdahl presents no evidence that his extrapolation does so. In general TL practice, the usual way of testing this assumption is to additionally determine the form of the second glow growth curve. In the case of sediments, this may be done by first bleaching out the removable part of the natural TL, and giving various beta or gamma doses. The resulting TL is referred to as a 'regenerated' growth curve. Comparison of the first glow and regenerated growth curves shows whether a TL sensitivity change has occurred on bleaching. In the series of N.W. European sediments that I have studied, it appeared that little or no such change occurred. If and only if this holds true, the ED is simply equal to the natural regeneration dose (NRD), i.e. the dose that, given to the bleached material, regenerates the intensity of the natural TL. This is the basis of the regeneration method for ED determination.

Figure 1 shows a selection of regenerated growth curves obtained by beta irradiation following a laboratory bleaching of the natural TL in five sediments. It is representative of the

variety of growth curve characteristics observed in over seventy sediments. In most cases, an initial non-linear portion is followed by a rising linear section, while, for a few samples, no initial non-linearity was evident. They cannot be fitted by curves of the simple saturating exponential type. Note in particular that data in the high dose regions display little or no curvature. Only these sections of the growth curves are revealed by first glow measurements on old sediments. It is clear that extrapolations, whether by straight line or poly nomial, from these higher doses cannot be relied on to reproduce the form of the curves at relatively low doses where the curvature is, in most cases, much higher.

A specific example is worked out in figure 2, which gives measurements for the oldest of the N.W. European sediments I have studied. Here an extrapolated ED based on the first glow growth curve is compared with the natural regeneration dose (NRD) found by interpolation of the regenerated growth curve. The extrapolated ED is 2.3 times greater than the NRD. The slopes of the two curves at the intensity of the natural TL are closely similar (ratio S_R/S_N = 1.11 $\pm$ 0.44), suggesting that the laboratory bleaching which preceded the regenerating irradiations did not significantly alter the TL sensitivity of the material. A TL age of 130 $\pm$ 15 ka was computed using the regeneration technique, while use of the extrapolation method would yield an age of approximately 300 ka.

It follows from the above that a TL date produced by means of first glow growth curve extrapolation may be much greater than that resulting from the regeneration method. Hence, TL ages exceeding the limiting value of 100 ka (Debenham, 1985) are to be expected when extrapolation is used, and they should not be taken as evidence of greater signal stability.

Finally, the following points regarding the regeneration technique should be emphasised. Comparison of the first glow and regenerated growth curves is considered an integral part of the method. The regenerated growth curve should be measured up to at least twice the NRD, and first glow points should be determined at various additive beta doses up to the same maximum TL intensity. This will give considerably better precision on slope ratio measurements than I have achieved so far. Any observed sensitivity change can be allowed for in the date calculation. Reproducibility of the TL measurements should be better than $\pm$ 5%, with good consistency of glow curve shapes. Anything worse than this probably indicates that the favourable feldspars 400 nm signal is suffering interference; quartz TL is usually to blame. While there can be no certainty about the validity of the regeneration method for dating sediments, it is clearly more justifiable to infer past TL acquisition from regenerated growth forms than from first glow extrapolation. A number of experiments have been carried out to further investigate the regeneration method. In these, samples recently bleached by sunlight have been irradiated to simulate an archaeological dose, artificially bleached, and then given regeneration doses. The samples were either sediments known to have been recently deposited and having very low natural TL, or were old samples bleached in sunlight while in suspension in water to low residual TL. Results show that the forms of the first glow growths were reproduced by the regenerated curves.

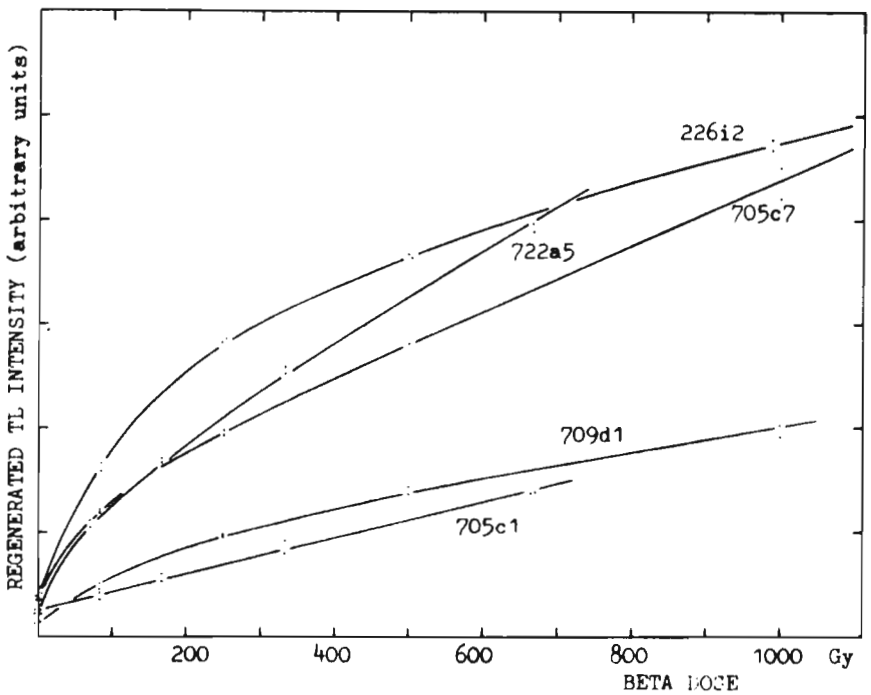

Figure 1: Growth curves at 300-310°C regenerated by beta irradiation after 16 hours bleaching under an Oriel simulated sunlight source. Polymineral fine grains samples on Al discs were observed with a quartz windowed EM1 9635 photomultiplier tube fitted with a Schott 2 mm thick UG11 filter. Heating rate was 2.5°C/s. Curves are labelled with sample identifiers (Oxford Lab. refs.); 226i2, Pontnewydd Cave, N. Wales, expected age > 200ka; 722a5, Achenheim, E. France, ~ 150ka; 705c7, Muleta Cave, Majorca, ~ 70ka; 709d1, Cagny-la-Garenne, N. France, ~ 400 ka; 705 c1, Muleta Cave, Majorca, ~ 50ka.

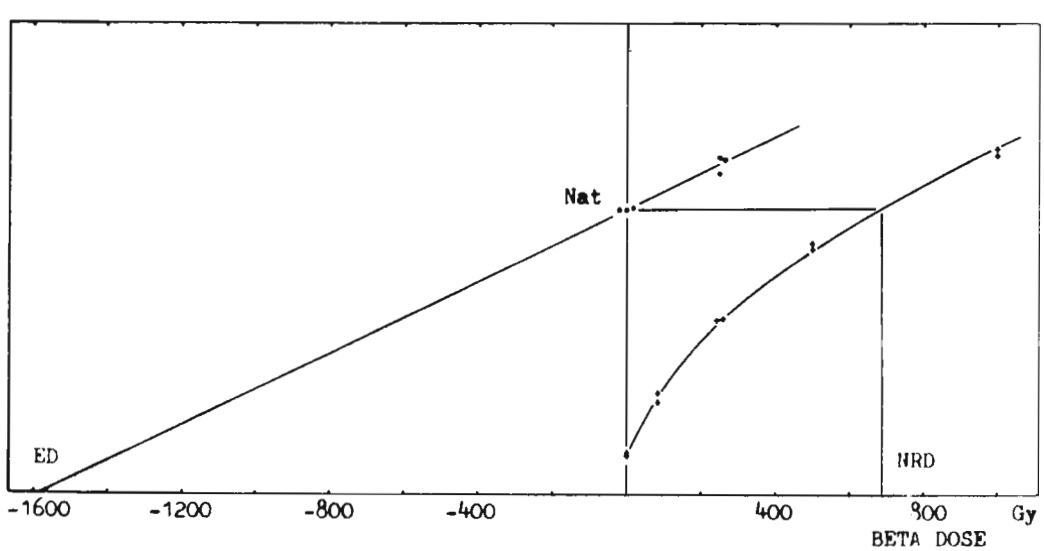

Figure 2: Comparison of regeneration and extrapolation techniques of ED determination as applied to a sediment (Oxford Lab. ref. 713f1) from Susterseel, W. Germany. Measurements derive from the temperatue interval 300-310°C.

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Reviewer's comments (A. G. Wintle)

The ongoing discussion concerning the method of ED determination for sediments is particularly relevant at this time since several papers containing TL dates have recently appeared. In particular, Buraczynski and Butrym (1984) have produced a series of TL dates ranging from 27,500 to 278,000 years BP for loess from Achenheim. They used the extrapolation of the additive first glow growth curve to obtain the ED. It is interesting to note that sample OxTL722a5 in Denbenham's paper is also from Achenheim and it shows considerable non-linearity in the regenerated response. Unfortunately Buraczynski and Butrym do not give any raw data, such as the ED obtained or how they converted from a LiF dose rate (from gammas only) to a SiO_2 dose rate (from alphas, betas and gammas to 50-56 micron polymineral grains). Also, lack of other age control means that their dates, and therefore their ED methodology, cannot be assessed.

The other papers of interest in this context are those from the December 1985 issue of the Canadian Journal of Earth Sciences by Berger and by Lamothe. In these the partial bleach method (R-I was used since the sediments being studied were waterlain and therefore likely to be 'overbleached' in the laboratory bleaching experiment. The examples given showed non-linearity for doses in excess of 400 Gy and for this reason quadratic fits were used for the extrapolation of the additive dose curve. The ages obtained were consistent with the geological evidence. However, it can be seen from their graphs that for samples older than those reported overestimation of the type described by Debenham would occur.

However, it should be stressed that it is not necessarily valid to apply the results obtained for polymineral fine grains from loess to data obtained for 100-300 micron pure potassium feldspar samples, as used by Mejdahl. On the other hand, it is clear that further studies using added doses on such feldspar samples should be attempted on sediments with greater age control in the period 100-300 ka where Debenham's proposed loss of luminescence centres should already be easily observed.

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Further comments on extrapolation methods of dating sediments

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Debenham (1985) questions the TL dating result that I obtained for K-feldspar (lab. no. R-841008) from a marine sediment from Cape Copenhagen, Greenland (Mejdahl 1985) on the grounds that the procedures used for extrapolating the first glow growth curve may not be valid. He further presents a series of growth curves which he finds cannot be fitted by saturating exponential functions.

I agree that my work on the Greenland sample is still incomplete. The growth curve in question is given in Mejdahl (1985, Figure 1). Linear extrapolation based on three points gave an ED value of 4160 Gy while polynomial regression yielded ED = 3060 Gy. I have since studied the complete growth curve of a K-feldspar from a Swedish postglacial dune sand (lab. no. R-841611) by adding doses to the natural level (about 50 Gy). The growth curve (Figure 1) is approximately linear up to about 600 Gy after which it curves and approaches saturation. Polynomial regression gave a very poor fit to these points whereas a reasonably good fit was obtained by exponential regression as shown in Figure 1. The equation of the exponential is

$$y = 43.18\ (1 - \exp(-0.000650x)) \qquad ...(1)$$

The exponential seems to underestimate the response in the range 600-1000 Gy, but gives a good fit below and above this region, apart from the two outliers at 3000 and 4000 Gy. A similar exponential fit for a quartz growth curve (Danish ice wedge cast, lab. no. R-32104) is shown in Figure 2. The equation is

$$y = 42 + 45.24(1 - \exp(-0.0152x)) \qquad ...(2)$$

Good exponential fits with exponential terms very similar to those in eqs (1) and (2) have been obtained for K-feldspar (with higher natural doses than the one in Figure 1) and quartz from a number of sediments from Finland, Norway and Sweden. For some quartz growth curves the exponential term was smaller, around 0.005x.

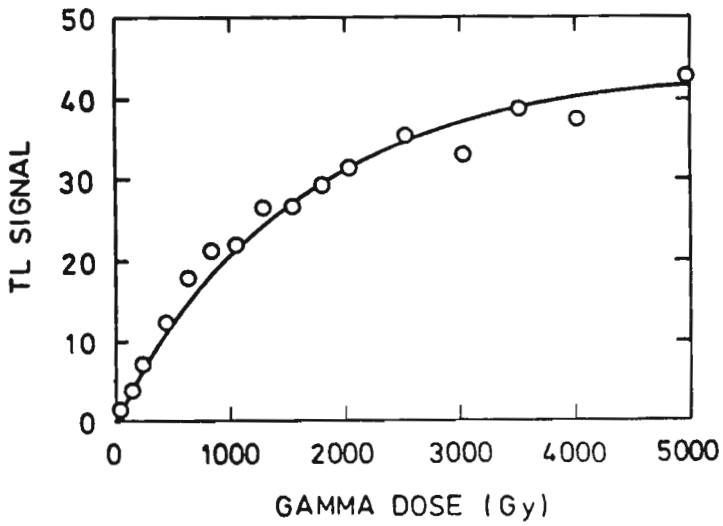

Figure 1: Growth curve for K-feldspar from a Swedish postglacial sand dune (lab. no. R-841611). Gamma doses were added to the natural level; about 50 Gy. A saturating exponential function has been fitted to the points.

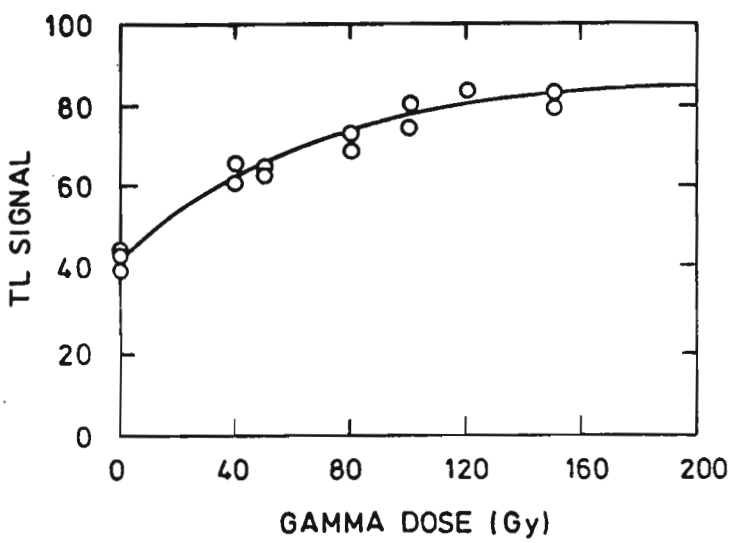

Figure 2: TL growth curve for quartz from a Danish ice wedge cast (lab. no. R-832104). Gamma doses were added to the natural level; about 40 Gy. A saturating exponential function has been fitted to the points.

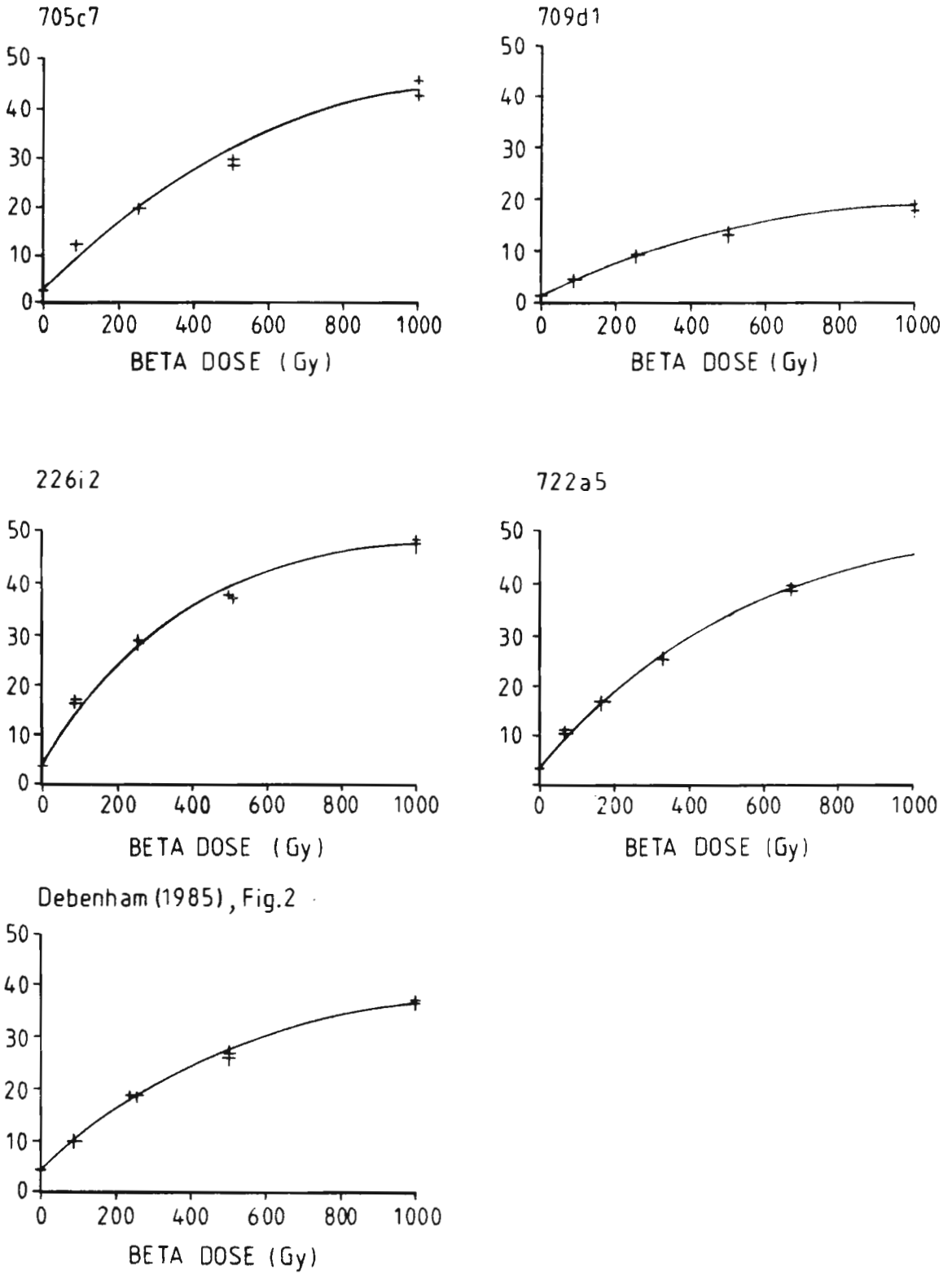

Figure 3: *Saturating exponential functions fitted to the TL growth curves given in Debenham (1985, Figs. 1 and 2).*

Editors note: *please note that curves were re-drawn free-hand by us from computer plots supplied by the author.*

The implication of these studies for the Greenland sample is that polynomial regression cannot be expected to give valid results, and linear regression will lead to an overestimate of the ED. Similarly, the linear extrapolation indicated by Debenham (1985, Figure 2) using only two groups of points will be invalid and his related discussion therefore unrealistic. Fitting an exponential to the growth curve of the Greenland sample (Mejdahl, 1985, Figure 1 plus a few more points, but none exceeding 2000 Gy) resulted in the following equation.

$$y = 85.4 + 48.15(1-\exp(-0.000818x)) \quad ...(3)$$

which has an exponential term very similar to the one in eq.(1). The corresponding ED value is 1250 Gy which, with a dose rate of 2.86 Gy/ka, yields an age of 437 ka. This is considerably smaller than those reported earlier but still well above Debenham's 100 ka limit. More work on the Greenland samples is in progress to justify the procedure and verify the results; this includes adding doses above 2000 Gy and regenerating growth curves up to saturation.

Concerning Debenham's second point, viz., the shape of the growth curves presented in his Figures 1 and 2 (Debenham 1985), I can not agree on his interpretation that an initial non-linear portion is followed by a linear one. Except for 705 c1, which is linear throughout, nowhere can one find more than two groups of points that lie on a straight line. In my opinion all curves show a saturating behaviour which would be even more pronounced if they were continued to higher doses. In Figure 3 below I have fitted saturating exponentials to all of Debenham's curves except 705c1. The fit is not quite satisfactory for 226i2 and 705c7 but excellent for 722a5, 709d1 and Debenham's Figure 2. It is interesting to note that the deviating points in the two samples deviate in the same way; this shows that the deviation is not random. The exponential equations are given in Table 1.

It is interesting to note the similarity of the exponential term for all samples. The saturation behaviour appears to be intermediate between what I find for K-feldspar and quartz. In Table 2 I have compared this behaviour, taking the saturation dose to be that for which 90% saturation is reached. An average exponential factor of 0.002 and an x-axis intercept of 30 Gy have been assumed for the samples in Table 1 and an x-axis intercept of 50 Gy has been included for the quartz sample.

Debenham does not state what his samples are, but it is evident that their TL growth is different from that of either of the K-feldspar and quartz samples discussed above. There seems to be no basis, therefore, for assuming that dating limitations found for his samples would automatically apply to samples from other regions. On the other hand, it must be admitted that as yet there is no definitive proof that this 100 ka barrier can be exceeded.

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Debenham, N. C. (1985) Comments on extrapolation methods of dating sediments by TL. Ancient TL 3 (2), 17-20.

Mejdahl, V. (1985) Thermoluminescence dating of loess deposition in Normandy. Ancient TL 3 (1), 14-16.

Table 1. Saturating exponential functions fitted to the growth curves given in Debenham (1985, Figures 1 and 2)

Sample No.	Equation
705c7	$y = 2.35 + 48.83\ (1 - \exp(-\ 0.001783x))$
709d1	$y = 1.35 + 21.46\ (1 - \exp(-\ 0.001886x))$
226i2	$y = 3.65 + 45.72\ (1 - \exp(-\ 0.002936x))$
722a5	$y = 3.65 + 48.55\ (1 - \exp(-\ 0.001938x))$
Fig. 2	$y = 8.30 + 75.51\ (1 - \exp(-\ 0.001895x))$

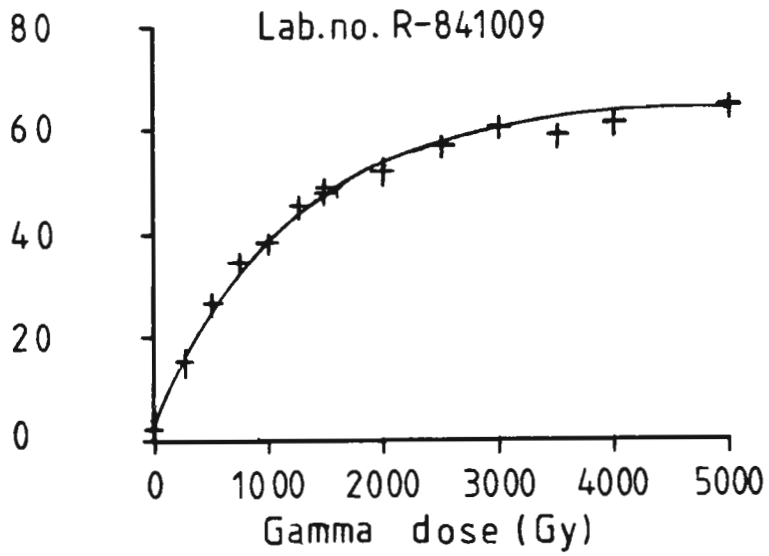
Table 2. Comparison of saturating behaviour of growth curves from Debenham (1985, Figures 1 and 2) and K-feldspar and quartz from Sweden and Denmark assuming exponential regression.

Sample	Lab no.	Exponential term	Dose for 90% saturation (Gy)
K-feldspar[1]	R-841611	$0.000650x$	3500
Quartz	R-832104	$0.0153(x - 50)$	200
Debenham	see Table 1	$0.0020(x - 30)$	1180

1. Quartz contamination less than 5%. Signal from quartz negligible.

Postscript

I have since completed regeneration measurements for a K-feldspar from a Greenland sample (lab. no. R-841009) taken from the same locality but 25 m above that represented in eq (3). The sample was bleached by exposure to sunlight for two days. The resulting regenerated growth curve is shown below.



Regeneration curve for K-feldspar from a Greenland sample, lab. no. R-841009. Bleaching was achieved by exposing the sample to sunlight for two days. An exponential function has been fitted to the points. The equation of this exponential is

$$y = 2.44 + 62.8(1 - \exp(-0.000822x))$$

The exponential term, and thereby the shape of the curve, is identical with that in eq. 3. This indicates that the extrapolation based on eq. 3 and described in the paper is a valid approach

Reviewer's comments (A. G. Wintle)

It seems that much more experimental work on a wide range of samples needs to be done to look at the non-linearity of different types of feldspars and quartz and fine grain mixtures of these minerals.

Also, these questions could be answered in part by further studies on a range of known age material, greater than 100 ka. However, such samples are very hard to find.

We must ask again whether there are any laboratory experiments which might prove, or disprove, Debenham's hypothesis that the problem is caused by loss of luminescence centres with time.

Letters to the editor

From: K. S. V. Nambi, Bhabha Atomic Research Centre, Bombay.

(i) **TL and ESR Terminology : k Factor**

Henry Schwarcz has suggested that an inequality of TL and ESR k-factors may arise out of the opacity of samples which could affect k_{TL} evaluation but not k_{ESR} (Ancient TL, Vol. 3, No. 1, 1985). As evaluation of k_{TL} is always done by comparing the TL outputs due to alpha and beta irradiations using samples thin enough that alpha particles pass entirely through, the opacity changes are not likely to affect k_{TL} evaluations. In any case such contributions can be readily checked by monitoring the TL output from a third sample given additive alpha, beta irradiations to the same individual doses as during k_{TL} evaluations i.e. by checking if the TL outputs satisfy the relations,

$$(NTL + \alpha) + (NTL + \beta) = (NTL + \alpha + \beta) + NTL$$

(ii) **Plateaux and Preheating**

In response to D. Huntley's query (Ancient TL, Vol. 3, No. 1, 1985) I would like to point out that, almost simultaneously with G. Valladas, we had announced the desirability of preheat treatment to separate 280/330°C peaks in limestone samples (N. Jb. Miner. Abh. 133, 1978, 215); no specific mention was however made on the plateau test. It has been our experience that preheat treatments are necessary to obtain good plateau especially in geological materials.

Nambi, K.S.V. + Mitra, S. Thermoluminescence investigations of old carbonate sedimentary rocks.

Notices

8th International Conference on Solid State Dosimetry, organised by the National Radiological Protection Board, U.K., 26th-29th August, 1986, St. Catharine's College, Oxford.

Further details from: Miss L. Ashby, National Radiological Protection Board, Chilton, Didcot, Oxfordshire, U.K.

The **'first international symposium on ESR Dating'** will be held in Ube, Japan, from 1st-4th September, 1985.

Further details may be obtained from Professor M. Ikeya, Technical College, Yamaguchi University, Tokiwadei, Ube 755, Japan.

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