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## Thermoluminescence dating of late Quaternary dune sand, Manawatu/Horowhenua area, New Zealand: a comparison with $^{14}\text{C}$ age determinations

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**Abstract** Samples of dune sand from two previously described sections in the southwestern North Island have been dated by the thermoluminescence method. At the first site, Koputaroa Phase dune sand immediately underlying Aokautere Ash (= Oruanui Ash  $^{14}\text{C}$  dated at c. 22 590 yr B.P.) was dated at  $24\,200 \pm 3700$  yr. At the second site, Holocene dune sand was dated at  $3000 \pm 500$  yr. Factors influencing the error limits of the thermoluminescence dates are discussed. The results confirm that coastal dunes were migrating inland during the last stadial of the Otira Glaciation and indicate that the thermoluminescence method could usefully be applied in the dating of upper Quaternary deposits in this region.

**Keywords** thermoluminescence dating; radiocarbon dating; dunes; eolian sediment; Pleistocene; Holocene; Manawatu; Horowhenua

### INTRODUCTION

During the past 30 years the application of new dating techniques in New Zealand has facilitated the understanding of late Quaternary environments, processes, and landscape evolution. Of particular significance has been the establishment of the broad chronological relationships between climatic and vegetation change, sea-level change, glacial fluctuations, loess deposition, and fluvial and marine terrace formation. Geological and geomorphological processes operate within a tectonic framework, and dating techniques have also provided information that has enabled deformation rates to be determined.

The technique of thermoluminescence (TL) dating is based upon the measurement of accumulated energy absorbed by mineral grains from the weak flux of nuclear radiation from surrounding sediment following deposition. It has been

developed and refined in recent years (Wintle & Huntley 1982; Aitken 1985) and is likely to form a valuable addition to the range of techniques presently available for the dating of Quaternary sediment in New Zealand. This paper describes the results of TL dating of dune sand at two sites in the Manawatu/Horowhenua area. As the objective was to assess the accuracy of the TL method for the dating of late Pleistocene and late Holocene eolian sediment, samples were collected from two sites where the sediment had previously been dated directly or indirectly by the  $^{14}\text{C}$  method.

### SITES

The sites from which samples were collected are described below:

#### 1. Paiaka Road, Horowhenua (grid ref. NZMS 260 S25/078683)

This exposure has been referred to by Cowie (1963, 1964). At the site, Koputaroa Phase dune sand of coastal origin (Shepherd 1985) overlies a marine terrace, known locally as the Tokomaru Marine Terrace, of probable Last Interglacial age (Hesp & Shepherd 1978). Interbedded with the dune sand is a tephra, locally named the Aokautere Ash (Cowie 1964). The bed resulted from the Oruanui eruption and has been widely used in the region as a time-stratigraphic marker. Although not dated at the site, Wilson et al. (1988) reported a mean age of  $22\,590 \pm 230$  conventional  $^{14}\text{C}$  yr B.P. from four samples of charcoal enclosed in Oruanui ignimbrite. The authors considered this to be a reliable  $^{14}\text{C}$  age for the eruption. Elsewhere in the Manawatu/Horowhenua region the tephra is interbedded within, but towards the base of, Ohakea Loess, which accumulated during the last stadial (oxygen isotope stage 2) of the Otira Glaciation (Milne & Smalley 1979). The sample of dune sand for TL dating (W836) was taken from 0.6 m below the tephra layer. As neither a paleosol nor a loess horizon occurs between the dune sand and the overlying tephra we consider that the age of deposition of the dune sand is likely to be similar to that of the tephra.

#### 2. Manawatu River section (Barber's Farm, NZMS 260 S24/147814)

At this site, described by Shepherd & Lees (1987), two Foxton phase dunes (Cowie 1963) of Holocene age have migrated onto the floodplain of the Manawatu River. The veneer of sand deposited by the older dune, which was sampled for TL dating (W835), overlies a peaty paleosol with trees in growth position, presumed to have been buried by the advancing dune. The outermost rings of one tree were dated at  $2380 \pm 110$  yr B.P. (NZ5220C), which is considered to approximate the age in calendar years of the sampled dune sand. An overlying peat bed dated at  $1860 \pm 65$  yr B.P. (NZ5221C) provides a minimum age for the sampled bed.

## METHOD

### Sample collection

Sediment samples were collected from the two exposures by driving a 6 cm diameter PVC tube approximately 60 cm into the outcropping sedimentary unit to be sampled. At the same time, surface exposures were collected with adhesive tape. The tubes were wrapped in black plastic and taken to a darkroom where the innermost 30 cm of tube was removed and wrapped in black plastic for postage, together with the surface samples, to the dating facility at Wollongong University, N.S.W., Australia. The dating laboratory was provided with no indication of the likely age of the samples.

### Thermoluminescence dating

TL dating of sediments depends upon the acquisition and long term stable storage of TL energy by crystalline minerals contained within a sedimentary unit. Prior to the final depositional episode it is necessary that any previous geologically acquired TL is removed by exposure to sunlight. After burial the TL energy begins to build up again at a rate dependent upon the radiation flux delivered by the long-lived isotopes of uranium, thorium, and potassium. The presence of rubidium and cosmic radiation play a lesser but contributory role, and the total radiation dose delivered to the TL phosphor is modified by the presence of water. The period since deposition of the sediment is therefore measured by determining the total amount of stored TL energy, the paleodose ( $P$ ), and the rate at which this energy is acquired, the annual dose ( $AD$ ).

$$\text{TL Age} = \frac{\text{Paleodose } (P)}{\text{Annual dose } (AD)}$$

### Evaluation of the paleodose

The TL dating method used here is essentially the regenerative method as modified by Readhead (1984, 1988). The 90–125  $\mu\text{m}$  quartz fraction was separated by sieving, chemically cleansed in HCl and etched in HF to remove the oxidised coating and the outer layer of diffused silica. This etching process also removes nearly all of the TL produced by short range alpha irradiation. Following heavy liquid separation, impurities were not detectable by either XRD or IR spectroscopy. A portion of the sample was subjected to a minimum of 48 h bleaching under an ultraviolet lamp (Philips MLU 300W), so reducing its stored TL to a minimum level, the so-called unbleachable level. Aliquots of this material were deposited as a monolayer onto 14 aluminium discs which were incrementally irradiated using a calibrated  $^{90}\text{Sr}$   $\beta$  plaque source. These discs were stored for a minimum period of 12 h and then heated to a temperature of 500°C at 5°C/s in a high purity nitrogen atmosphere. The TL output emitted was detected by an E.M.I. 9635QB photomultiplier fitted with a Chance Pilkington heat filter and a Corning 7-57 blue transmitting filter. Photon counting was achieved with Ortec NIM modules. The laboratory-induced TL levels were recorded and a growth curve relating TL and radiation dose constructed (Fig. 1).

Unbleached quartz was deposited on 14 discs; 8 were glow out as described and the remaining 6 were incrementally irradiated. The mean of the TL output as measured from the discs containing unirradiated and unbleached material was computer fitted to the TL growth curve and the equivalent radiation dose ( $ED_N$ ) thus derived. The value of  $ED_S$ , the

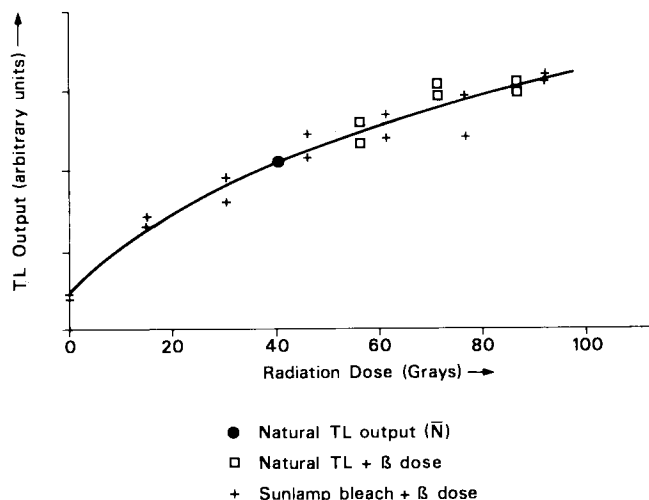


Fig. 1 Thermoluminescence growth curve at 375°C for Koputaroa Phase dune sand (sample W386).

equivalent radiation dose measured from a similarly treated sample collected from a modern surface was similarly derived. Thus the sample paleodose becomes:

$$\text{Paleodose } (P) = (ED_N - ED_S)$$

The TL outputs measured from the natural TL plus beta irradiated discs were plotted on the TL growth curve with reference to the mean ( $N$ ) value only. If there has been no change in TL sensitivity due to the different bleaching methods (sunlight and UV lamp) of the two portions of the sample, these values should coincide with the growth curve constructed using laboratory bleached and irradiated sample material. In this case an  $ED_N$  value computed from the ( $N$ ) and the ( $N + \beta$ ) should agree with that computed from the fitting of ( $N$ ) to the regenerated TL growth curve. Essentially this is a combination of both the regenerative and the additive TL test methods (Aitken 1985). The TL output of all discs was normalised using a standard irradiation/second glow procedure. This corrected for any disc-to-disc variation.

### Data analysis

The TL output of all discs was read at 25°C intervals from continuous glow curves recorded between ambient temperature and 500°C. The mean natural TL values were compared to those exhibited by a pair of UV bleached and laboratory irradiated discs selected to give a TL ratio of about unity. If the natural and the regenerated TL glow curves are the same shape then comparison of one with the other provides a plateau region which is indicative of electron trap stability. In practice this lies between 300 and 500°C (Fig. 2), although the length and position may vary from sample to sample. The existence of a well-defined TL plateau also indicates that results are not affected by the storage of laboratory irradiated samples for only 12 h prior to glowout. In the final TL computation it can be demonstrated that the sample paleodose, and consequently the specimen age, follows this plateau characteristic (Nansen et al. in press). Thus, if the ratio of the natural TL to the laboratory-induced TL remains constant, the TL age will also remain constant. Indeed, analysis at temperatures at which the rate of change is large can lead to error due to the difficulty in measurement and the problem of temperature matching between glow curves.

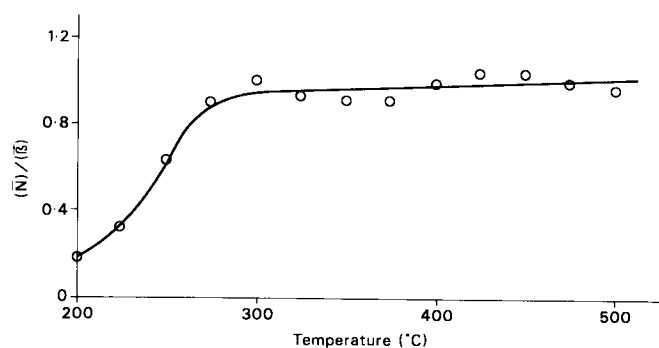


Fig. 2 Plateau test for dune sand from the Manawatu River site (W835).

#### Determination of the annual radiation dose

This determination was made by means of thick source alpha counting and therefore assumed secular equilibrium of the uranium and thorium decay chains. Samples were prepared by finely crushing approximately 100 g of oven-dried sample to a grain size less than the range of the alpha particles, thus ensuring that all such radiation was subsequently detected. Approximately 3 g of this material was lightly pressed onto a 42 mm ZnS scintillation screen placed within a sealable alpha counting cell. After a period of 21 days the alpha activity of the specimen was measured using a previously calibrated photomultiplier system with suitable Ortec NIM modular electronics. A check of the two alpha counting systems used in the Wollongong laboratory with a well-documented appropriate standard (USGGSS-G2) resulted in a 0.6% and 2.8% discrepancy from the anticipated specific activity value. These results were well within the acceptable experimental error. Finally, the potassium and rubidium levels of the samples were measured by means of XRF and the radiation dose received annually computed using equations derived by Readhead (1984). A full account of this procedure is also given by Aitken (1985).

## RESULTS

Full details of the results are provided in Table 1.

#### Surface residual TL correction

As the material comprising both samples was likely to have been transported at least 15 km by eolian processes it may be assumed that it has received considerable exposure to the sun's UV component. The surface samples, derived from sites relatively recently disturbed by human activities, probably received less exposure. This view is supported by work on alluvium in Northern Australia (Nanson et al. in press) and by Mejdahl (1988) who found that the subtraction of an incorrect surface residual leads to a poor plateau relationship. The Wollongong laboratory has found the effect to become increasingly noticeable in younger samples where the surface correction forms a larger proportion of the total TL signal exhibited by the sample. Sample W835 yields a satisfactory relationship (Fig. 2) indicating that there has been good exposure, and therefore removal of the geological component, during the transport phase. However, it is unlikely that this exposure would have been as intense as that derived from the laboratory UV lamp, and the ages shown must therefore be taken as maximum values.

#### COMPARISON BETWEEN THERMOLUMINESCENCE AND RADIOCARBON AGES AND ASSOCIATED ERRORS

With respect to the age of Koputaroa dune sand at the Paiaka Road site, the TL date of  $24\,200 \pm 3700$  yr would appear to correspond closely to the date of  $22\,590 \pm 230$  yr B.P. obtained by the  $^{14}\text{C}$  dating method, after allowing for the fact that in the 20 000–25 000 yr age range conventional  $^{14}\text{C}$  dates may be younger than true ages by 1000–2000 yr as discussed by Wilson et al. (1988).

The TL date of  $3000 \pm 500$  yr for the sample from the Manawatu site is older than the calibrated  $^{14}\text{C}$  age of  $2380 \pm$

Table 1 Thermoluminescence analyses: measured values with associated errors for samples collected from the Paiaka Road and Manawatu River sites.

	Koputaroa dune sand	Holocene dune sand
Lab. no.	W836	W835
Plateau region (°C)	300–500	300–500
Analysis temp. (°C)	375	375
Paleodose (Grays)	41.3 $\pm$ 6.1	5.5 $\pm$ 0.9
K content (%) - note 1	0.91 $\pm$ 4.6	1.50 $\pm$ 5.6
Rb content (ppm) - note 1	45.8 $\pm$ 4.6	55.7 $\pm$ 5.6
Cosmic contribution ( $\mu\text{Gy/yr}$ )	150 $\pm$ 50	150 $\pm$ 50
H <sub>2</sub> O content (%) - note 2	2.4 $\pm$ 3	26.4 $\pm$ 3
Specific alpha activity (Bq/kg) - note 3	29.8 $\pm$ 4	30.1 $\pm$ 4
Annual radiation dose ( $\mu\text{Gy/yr}$ ) - note 4	1704 $\pm$ 75	1829 $\pm$ 59
<b>TL age (years) - note 5</b>	<b>24 000 <math>\pm</math> 3700</b>	<b>3000 <math>\pm</math> 500</b>

<sup>1</sup> Potassium and rubidium levels were determined by X-ray fluorescence.

<sup>2</sup> Corrections made for moisture content as measured from the samples.

<sup>3</sup> The specific alpha activity of the specimen was measured using "thick source" alpha counting over a 42 mm scintillation screen and represents both the U and the Th decay chains.

<sup>4</sup> The annual radiation dose was computed assuming there to be no disequilibrium in the U and Th decay chains.

<sup>5</sup> The errors shown represent one standard deviation in the measurements made. In these determinations a surface residual TL correction corresponding to the level attained following exposure to an intense laboratory UV sunlamp was applied.

110 yr B.P. but by little more than one standard deviation. The disparity between the dates would have been reduced had it been possible to apply the surface residual TL correction as opposed to the subtraction of the TL level measured following exposure to UV radiation in the laboratory. UV radiation from the sunlamp is likely to have exceeded that received during transport prior to deposition and thus the TL age must represent a maximum value. The age difference would have been further reduced if the average environmental water content of the sample since deposition had been less than that measured at the time of collection. For example, if there had been an average water content of 10%, as opposed to the measured value of 26.4%, a TL age of 2600 yr B.P. would have resulted, owing to the greater proportion of the radiation reaching the sample from its immediate environment (Aitken 1985).

The standard deviation of the TL dates is much greater than for the  $^{14}\text{C}$  dates, but whereas the TL errors reflect the total experimental and statistical uncertainty assigned to each TL parameter, the  $^{14}\text{C}$  errors merely reflect the statistical uncertainty. Experience at the University of Wollongong TL dating laboratory suggests that dates from similar stratigraphic units tend to cluster well within the stated error band. The standard deviation of a given TL date may be reduced by increasing the number of data points and measurements made upon any one sample. Alternatively, more than one sample may be taken from the unit of interest and the uncertainty reduced in that manner.

Although not clearly demonstrated here with only two samples being dated, in general, percentage errors become larger for younger samples because the measurement errors remain much the same and therefore become a greater proportion of the final computed age. The error introduced in not applying the surface residual TL correction also becomes larger for younger samples as it forms a greater proportion of the measured paleodose. As discussed above, if such a correction is not applied, a maximum age results, although, with respect to the two samples described in this paper, the TL characteristics indicate that the samples had been well bleached prior to deposition.

The age range of TL dating is a function of the total absorbed radiation dose, the upper limit being governed by the saturation of the available electron traps (McKeever 1985) and the lower level by the TL sensitivity of the sample material. These limits may be modified by the rate at which the radiation energy is supplied, which, in turn, governs the rate at which the TL traps are filled. Thus a sample receiving a high radiation level will reach saturation (i.e. maximum determinable TL age) sooner than the same sample receiving a lesser annual radiation dose. Given favourable conditions it is possible to determine the age of sedimentary quartz grains in the range 1000–400 000 yr.

A major constraint on the use of TL dating is that the quartz grains must be bleached by sunlight prior to burial and, therefore, dune sediment is particularly appropriate for its application. However, some fluvial sediment has also been dated successfully (e.g., Nanson & Young 1987).

#### SIGNIFICANCE OF THE AGE OF THE KOPUTAROA PHASE DUNE SAND

The TL age obtained for the Koputaroa Phase dune sand appears to confirm the suggestion by Cowie (1963) and Fleming (1972) that the dunes were active during the last stadial of the Otira Glaciation. However, whereas those authors favoured a

fluvial origin for the sand, Shepherd (1985) provided evidence that some, if not all, of the dunes, including the one that was TL dated, were of coastal origin. This suggests that the prevailing northwesterly winds were at least as intense as at present, and demonstrates that it should not be assumed that the presence of coastal dune sand within the cover beds of coastal terraces in the southwest of the North Island is necessarily indicative of interglacial or interstadial conditions.

#### CONCLUSION

The two dates reported in this paper give reason for confidence that the TL method can successfully be applied in this region, and may prove suitable for dating upper Quaternary deposits elsewhere in New Zealand. The age obtained for Koputaroa Phase dune sand confirms that coastal dunes were migrating inland during the last stadial of the Otira Glaciation. The technique is likely to be particularly useful for dating sediment which is either too old to be dated by the radiocarbon method, or which is unable to be dated by other methods. It may also serve as a cross-check where contamination of radiocarbon dated samples is suspected.

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