

# Dating the Quaternary: progress in luminescence dating of sediments

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Received 9 November 2005; accepted 15 November 2005

## Abstract

Luminescence dating comprises a collection of numerical-age techniques that are among the most significant chronological tools currently used in Quaternary research. This paper briefly reviews the key historical developments in luminescence dating, from its roots in thermoluminescence dating of heated minerals to the development of optical dating methods for sunlight-exposed sediments. We describe the principles and practicalities of the various techniques commonly used in luminescence dating, including multiple-aliquot, single-aliquot and single-grain procedures, and we discuss some of the latest approaches to recognising and minimising potential errors in age estimation (e.g., by means of component analysis and dose distribution methods in optical dating). The overview also introduces the other papers in this special issue of *Quaternary Science Reviews*, which address a selection of important issues in basic research, technique development and application of luminescence dating to critical questions in the geological and archaeological sciences, illustrated with examples from around the world and the last million years of Earth history.

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## 1. Introduction

The Quaternary Period was, and is, arguably one of the most important intervals in Earth's history. It has been not only a time of extreme climatic fluctuation, but also an interval during which modern humans first appeared, the first civilisations formed and farming began, and humans began to have a profound effect on the natural environment. This significant period has, however, been difficult to put into an absolute temporal context despite the development of several dating techniques (Fig. 1). This is because most techniques require the presence of a specific, often uncommon, material that has to occur in the relevant context. Moreover, many dating methods are only useful over short time scales, the calculated age does not directly date the desired event, and/or complex age calibration may be necessary which can significantly increase the uncertainty in the final result.

The most commonly used geochronological technique has been radiocarbon dating, which was also one of the first Quaternary dating methods to be invented and to be

studied in detail. Radiocarbon dating is relatively affordable and this has undoubtedly assisted with its popularity. Although this method has contributed greatly to our understanding of Earth history, its use is nevertheless limited. In most circumstances, it is useful only from a few hundred years to 60,000 years; it usually requires the presence of fossil vegetation, and this is often detrital; and if radiocarbon ages are to be compared to calendar years, then detailed knowledge about past variations in the concentration of radiocarbon in the atmosphere is needed. Other methods such as potassium–argon, argon–argon, uranium-series and fission track dating, for example, require the presence of in situ volcanic or carbonate deposits, while dendrochronology, amino acid racemisation and palaeomagnetic techniques give only relative ages unless age patterns can be matched with independently dated sequences. Recent developments in cosmogenic nuclide exposure dating have shown good promise for this technique, but a better understanding of some of the key physical processes is still needed and, in many geomorphic settings, there still remains the problem of age uncertainties as a result of periodic surface cover. Clearly, the most useful dating method would be one that directly dates the material of interest, can be applied to the most ubiquitous

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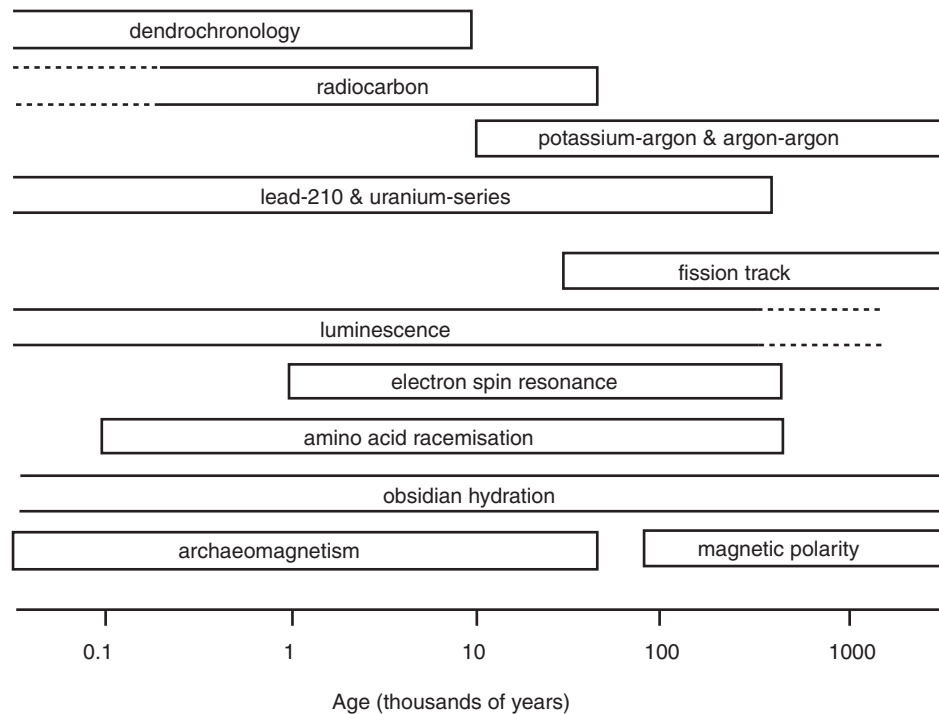


Fig. 1. Age ranges of some of the various dating methods commonly used in Quaternary research. The age limits are only approximations as they are dictated not only by the time-dependent biological, chemical and physical processes that regulate these geochronometers, but also by the nature of the material dated and, in some cases, by ambient conditions. For luminescence dating, for example, the amount of U, Th, and K in the material immediately surrounding the sample will, to a large degree, govern the upper and lower age limits of the method (modified from Fig. P.1 of Aitken, 1998).

materials, works over a long time range, and gives calendar ages without complex calibration. The only method that comes close to satisfying all of these conditions is luminescence dating.

Luminescence dating is used to date the time since last exposure of quartz or feldspar grains (the two most common minerals on Earth) to sunlight or heat. It is, therefore, commonly used to date the time of formation, and alteration, of sedimentary landforms; this is significant, as sedimentary landforms are direct proxies for environmental (climatic) change. Luminescence dating can also be used to put into temporal context any artefact or fossil buried within a sedimentary deposit, and, if an object has been affected by sufficient heat (e.g., fired pottery or a hearth stone), then it may be dated directly. Luminescence dating has been shown to work for samples as young as a few years, or as old as several hundreds of thousands of years (the upper theoretical age limit is thought to be a few million years), and it gives calendar ages directly.

One of the most significant aspects of luminescence dating is its inherently experimental nature. It consists of a family of techniques, and when one technique is not suited to a particular circumstance, then often another one may be. Moreover, because of the variability in the luminescence properties of natural quartz and feldspar, and the variations in sedimentary depositional processes, the technique has to be tested, and suitable measurement conditions validated, each time it is applied. This has required that workers not only give a detailed account of

their methodology each time it is used, but a thorough discussion of the reliability of the ages and supporting data has to be reported also. This has led to a degree of quality control rarely seen with other dating methods. This practice of reporting experimental detail, and therefore the sharing of methodology, has also resulted in the rapid advance of the technique, with the recognition of procedural pitfalls and new avenues for enquiry. Indeed, luminescence dating has, in the last decade, advanced at a rate rarely matched by other dating methods. As many of the techniques have become more established, however, some workers are now failing to report the necessary details—despite guidance offered by some practitioners (e.g., Prescott and Robertson, 1997, p. 918)—and this is of concern.

This special issue of *Quaternary Science Reviews* was inspired by a conference at the South Australian Museum in May 2004. The conference was in celebration of the 80th birthday of one of luminescence dating's most respected researchers, Professor John R. Prescott (see the dedication section at the end of this paper). The presentations reflected the current state of the science, and how recent developments in luminescence dating techniques have allowed new problems to be addressed in Quaternary research. Some of the papers presented at this conference, and others, are included in this special issue. Here we introduce the papers in this issue, but first set the stage by giving a brief account of what luminescence is, and how luminescence dating was developed. This introduction is intended to give general

Quaternary scientists an appreciation of the importance of luminescence dating to Quaternary research. Other recent reviews, with differing emphases, include Troja and Roberts (2000), Lian and Huntley (2001), Lian et al. (2002), Murray and Olley (2002), Wallinga (2002), Feathers (2003), Fattahi and Stokes (2003), Duller (2004) and Roberts et al. (2005).

## 2. The luminescence process

Luminescence refers to the light emitted by some materials in response to some external stimulus, such as heat (resulting in thermoluminescence, TL), pressure (triboluminescence), a chemical reaction (chemiluminescence), electromagnetic radiation (radioluminescence), or ionising radiation (photoluminescence). In the latter case, the term “optical dating” is used when the stimulation is either by visible light (commonly referred to as optically stimulated luminescence, OSL) or near-infrared radiation (infrared stimulated luminescence, IRSL). TL should not be confused with incandescence, which is the light observed when, for example, a filament light bulb is switched on or a stove element is heated to a high temperature. In the case of TL, OSL and IRSL, a further exposure to stimulation will not result in the production of more light. What is needed to stimulate further luminescence is a subsequent exposure to a source of ionisation, such as nuclear radiation.

The occurrence of luminescence has been known about for at least 450 years: the first scientific publication describing observations of the phenomenon was probably that of Gesner (1555), and the first luminescence experiments were likely those of Boyle (1664), who studied TL. A comprehensive account of the history of luminescence research was published by Harvey (1957), while summaries relevant to dating are provided by Aitken (1985, 1998).

For luminescence dating, the materials of interest are natural crystals, specifically quartz and feldspar sediment, although other minerals can be used in principle. Minerals contain structural defects and chemical impurities, some of which can act as traps for unbound (free) electrons. Free electrons are produced when the mineral grains of interest are exposed to ionising radiation: alpha and beta particles, and gamma rays, emitted by the decay of radioactive elements within the minerals and their immediate surroundings, and from cosmic rays originating from unknown sources in the universe. If irradiation continues, then electron traps will fill until a further dose of radiation will not result in more luminescence being measured; this condition is referred to as “saturation”.

Put simply, luminescence results when some external stimulation ejects electrons from traps. The evicted electrons diffuse around the crystal lattice; some of these eventually become lodged at a similar type of trap or return directly to the ground state, while others find themselves at a different kind of site, referred to as a recombination centre. When recombination occurs, excess energy is given

off either as heat (lattice vibrations or phonons) or as luminescence (photons); the latter process is referred to as radiative recombination. This process of electron eviction and recombination is nearly instantaneous. In any given crystal, there are many kinds of electron trap. Some of these traps can be emptied very easily when the grains are stimulated by light, but, for others, exposure to light has little or no effect. Most traps, however, can be emptied by exposure to sufficient heat ( $\sim 500^\circ\text{C}$ ). Moreover, at ambient temperature ( $\sim 20^\circ\text{C}$ ), some traps are only able to hold electrons for a few days or less, while others can hold electrons for millions of years or more. The latter types of trap are referred to as deep traps, and these have thermal lifetimes that are sufficiently long for Quaternary dating.

Physical models used to explain the luminescence process are complex, and many of the observations have not yet been explained in detail. Reviews that are applicable to dating are provided by McKeever and Chen (1997), Bailey (2001) and Bøtter-Jensen et al. (2003), while more general accounts can be found in Aitken (1985, 1998).

In practice, sediments are collected in the field while being kept in the dark. This can be accomplished in daylight by collecting and sealing samples in opaque cylinders, excavating sediment blocks, or by sampling loose material at night with the aid of suitably filtered light. General guidelines on sample collection procedures can be found in Aitken (1985, 1998), Lian and Huntley (2001) and Wallinga (2002). In the laboratory, under special dim red or orange lighting, the mineral grains of interest are separated. Then the so-called “natural” luminescence signal is evoked by exposing the grains to heat (for TL dating) or to photons of a specific energy or energy range (for optical dating). In the case of TL dating, a “glow curve” of TL intensity versus stimulation temperature is obtained (Fig. 2a), whereas a “shine-down” (or decay) curve of OSL or IRSL intensity versus stimulation time is obtained in optical dating (Fig. 4). Using a calibrated laboratory radiation source, the way the mineral grains respond to increasing doses of radiation is measured. From these measurements, and the natural signal, the dose the sample had absorbed in nature since burial (or since exposure to heat) is estimated—the palaeodose estimate. Since laboratory radiation sources typically only expose the sample to beta or gamma radiation, which is different from the condition in the natural environment where the sample is exposed to alpha, beta, gamma and cosmic-ray radiation, the palaeodose estimate is commonly referred to as the “equivalent dose”.

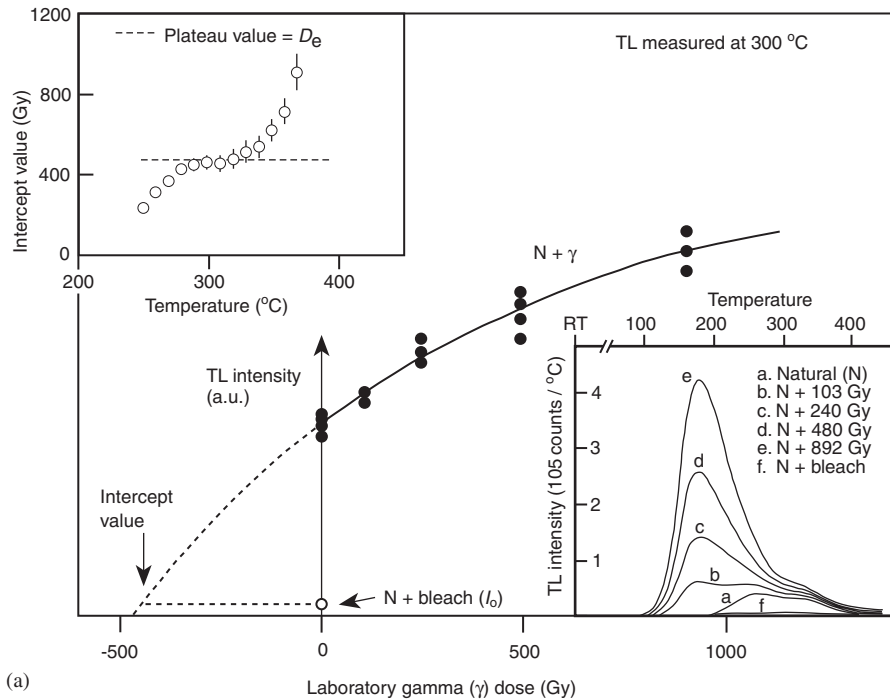
A subsample of the sediment used to determine the equivalent dose is analysed for U, Th, and K, and the concentrations of these radioactive elements, and their daughter products, together with an estimate of the cosmic-ray intensity at the sample site, are used to calculate the rate at which ionising radiation energy is absorbed by the mineral grains of interest. A number of factors can complicate this assessment, such as the need to estimate

the average water content over the entire period of sample burial or since the last heating (as pore water absorbs or attenuates radiation), and to establish whether or not, at any time since this event, the U-series decay chain has been in a state of disequilibrium. Some forms of disequilibrium are time-dependent and should be taken into account when determining the dose rate (e.g., Wintle and Huntley, 1979a, 1980; Readhead, 1987; Prescott and Hutton, 1995; Olley et al., 1996, 1997, 2004b; Stokes et al., 2003). The luminescence age is then calculated as the equivalent dose divided by the dose rate.

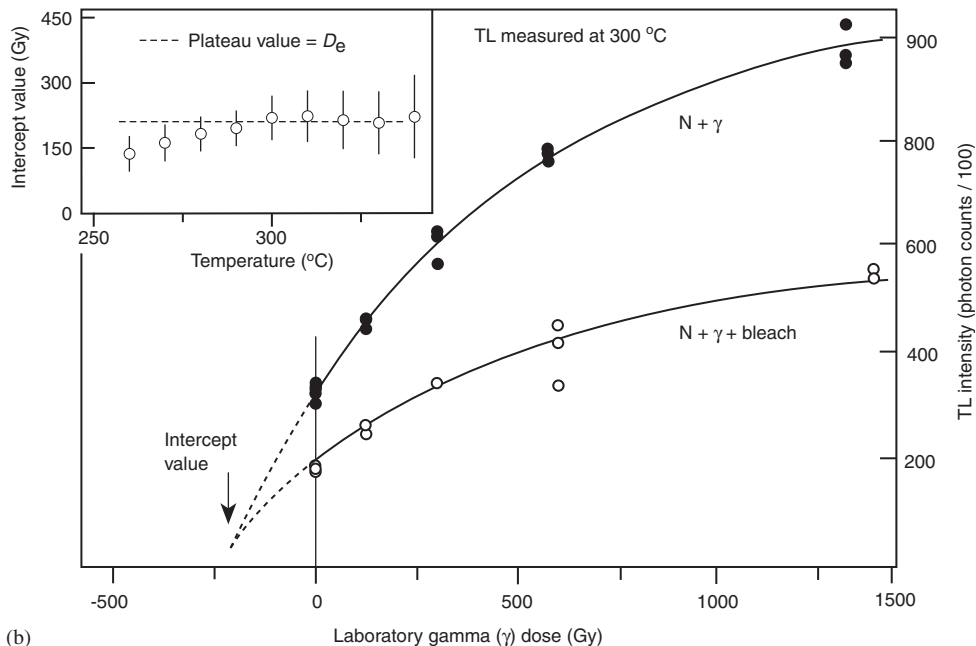
### 3. Luminescence dating of sediments—historical background

As mentioned above, the calculation of a luminescence age requires that the sample's dose-response be modelled, which leads to an estimate of the palaeodose referred to as the equivalent dose. This, together with a measure of the environmental dose rate, is used to calculate a luminescence age.

The techniques used to determine the dose rate (dosimetry) have changed little since the first luminescence



(a)



(b)

ages were determined  $\sim 40$  years ago, although improvements in analytical methods (instrumentation) have increased efficiency, precision and, in some cases, accuracy—the latter through identification of past and present disequilibria in the U-series. Notable advances include: (i) gaining a better understanding of the contribution of cosmic rays to the dose rate, specifically the details on how this changes with altitude and latitude, with depth beneath the ground surface, and with fluctuations in Earth's magnetic field (Prescott and Hutton, 1988, 1994); (ii) the implementation of portable gamma-ray spectrometers in the 1980s as a replacement for the cumbersome and time-consuming practice of using TL dosimetry capsules for in situ measurements; and (iii) the greater accessibility to high-resolution alpha and gamma spectrometry facilities to investigate the state of equilibrium of the U and Th decay chains (e.g., Olley et al., 1996). More substantial advances, however, have been made in obtaining reliable estimates of the equivalent dose, which we discuss below.

Following the landmark publication by Daniels et al. (1953) on the potential geological and archaeological applications of TL, there were several attempts at dating rocks. But the details of how radiation energy is absorbed by the materials being dated was never completely worked out, and this, together with other complications, resulted in a slowing down of research on dating of geological samples by the mid-1960s, although progress in TL dating of fired pottery and other heated materials continued apace (e.g., Aitken et al., 1964, 1968; see review by Roberts, 1997). The first published research on TL dating of sediments came from the former Soviet Union and included those of Shelkopyas and Morozov (1965), Morozov (1968) and Shelkopyas (1971), but these results were not accessible to Western workers until the summary paper by Dreimanis et al. (1978). Moreover, the Soviet papers did not provide the detail needed to fully evaluate the results, but, on the basis of what was published, there was reason to believe that the protocols used by these workers were invalid, insufficient, or inadequately described (Wintle and Huntley, 1982).

In the West, meanwhile, independent studies by Johnson and Blanchard (1967) and Bothner and Johnson (1969) had shown that the TL emitted from carbonate (and, hence, the absorbed dose) in sea cores increased with depth below the seabed. A similar result was published for radiolaria extracted from an ocean core by Huntley and Johnson (1976), but it was soon discovered that the TL signal was not coming from the radiolaria, but from the mineral sediment adhering to them (Wintle and Huntley, 1979a). During this time, some advances were also being made in Hungary, Poland, and China (see detailed accounts in Wintle and Huntley, 1982), but it was not until the work of Wintle and Huntley (1979a, b, 1980) that a suitable technique was developed for TL dating of sediments.

The simplest technique developed to determine the equivalent dose was the additive-dose method, for which it is assumed that the number of electrons in the traps of interest is negligible for a sediment sample at the time of burial. In reality, this assumption is only valid for heated material, as it has been shown that even a prolonged exposure to direct sunlight does not empty all of the electron traps sampled during measurement. This led to a model in which the measured TL( $I$ ) is considered to consist of two components, such that  $I = I_0 + I_d$ , where  $I_0$  arises from traps that are insensitive to light and  $I_d$  comes from light-sensitive traps (Wintle and Huntley, 1979a; see also Singhvi et al., 1982 and Smith et al., 1982). Such a division of traps is clearly an approximation, but this generalisation seems to hold in practice. In the laboratory,  $I_0$  is approximated by exposing some of the samples to an extended period (several hours) of sunlight. This protocol is referred to as the “total bleach” method and the procedure is explained in Fig. 2a.

However, the most universally applicable model developed at this time was the “partial bleach” method (Wintle and Huntley, 1980), which could be used in situations where the sediment grains in a sample had received only limited sunlight exposure. In practice,  $I_0$  and  $I_d$  are distinguished by giving some subsamples (“aliquots”) a

Fig. 2. (a) Illustration of the total bleach method. The data shown are for K-feldspar medium sand extracted from sediment deposited in an intertidal beach environment that yielded an apparent TL age in the range 240–290 ka (modified from Fig. 3 of Balescu and Lamothe, 1993). Several aliquots are prepared for the total bleach method, some are left as is (the “naturals”, N), while the others are given various radiation doses (N + gamma). Some of the N aliquots are then given a prolonged exposure to light to empty all of the light-sensitive traps, leaving electrons only in the traps responsible for  $I_0$  (see text). All of the aliquots are then heated to a high temperature, resulting in “glow curves” of TL intensity versus stimulation temperature; the inset graph (lower right) shows representative glow curves for a natural aliquot (curve a), for aliquots that have received various laboratory doses (curves b–e), and for a natural aliquot that has received prolonged exposure to light: 8 h of natural sunlight in this case (curve f). The laboratory dosed aliquots (b–e) also emit TL below  $\sim 200$  °C that has resulted from the filling of traps that are not thermally stable for geological lengths of time. The dose-axis intercept value is determined as shown in the main graph (the dashed lines indicated the portions of the fitted curves which are extrapolated), and is plotted as a function of glow curve temperature, as shown in the other inset graph (upper left); the equivalent dose ( $D_e$ ) is usually taken as the dose at which the intercept values become constant—i.e., where they form a plateau. In the example, a plateau occurs between  $\sim 300$  and 330 °C, and the  $D_e$  is estimated from it to be  $\sim 480$  Gy (a slightly higher value was estimated by Balescu and Lamothe, 1993). If the dose-axis intercept values fail to form a plateau, then the method fails and an age cannot be calculated. (b) Illustration of the partial bleach method (sometimes called the  $R$ -gamma, or  $R$ - $I$  method if gamma radiation is used, or  $R$ -beta method if beta radiation is used) for TL dating. The data shown are for TL emitted from K-feldspar in fine silt extracted from a  $\sim 110$  ka fossil peat bed (modified from Fig. 4d of Lian et al., 1995). Typically, 40–50 aliquots of a sample are prepared, some are not dosed (the N aliquots), while the others are given various radiation doses, in this case from a  $^{60}\text{Co}$  gamma ( $\gamma$ ) source. Roughly half of the set are then given a short exposure to light, thus emptying only the most light-sensitive traps. These aliquots form the N +  $\gamma$  + bleach data set; the other aliquots form the N +  $\gamma$  data set. Curves are fitted to the two data sets, and they are extrapolated (dashed lines) to where they intersect each other above the dose axis. As with the total bleach method, these intercept values are determined as a function of glow curve temperature (inset graph) and the dose at which they become constant is taken as the  $D_e$ .

short sunlight exposure, and provided this exposure is less than that received in the environment immediately prior to burial, then the method will give the correct equivalent dose (Fig. 2b). After the establishment of the partial bleach protocol, research continued into fine-tuning of the

method, which involved investigating TL emission spectra of quartz and feldspar (and some other minerals), their bleaching characteristics, and the thermal stability of the electron traps responsible for the measured luminescence. There was also much work done on comparing calculated TL ages with independent age information, which led to the development of various field-sampling strategies. Good reviews are those of Berger (1988, 1995). A variant of the partial bleach method, called the “selective bleach” method, was developed for the TL dating of quartz sediments (Prescott and Mojarrabi, 1993). In this procedure, the equivalent dose is estimated for only the most light-sensitive TL signal in quartz, which is separated from the other TL signals by selectively bleaching the sample using photons of green and longer wavelengths (Franklin and Hornyak, 1990; Prescott and Fox, 1990).

Later still, a third TL dating procedure received widespread application. This method involved two sets of aliquots that are used together to estimate the equivalent dose: the first set is given the conventional additive-dose treatment, while the second set is given a prolonged bleach to get as close as possible to  $I_0$ , followed by a series of regenerative doses. Ideally the dose responses of both data sets are identical except for a shift along the dose axis; therefore a single function is fitted to both data sets, with the shift being a parameter in the fit. The magnitude of the shift is equal to the equivalent dose after correction for incomplete laboratory bleaching (Fig. 3). This technique, sometimes called the “Australian slide” method because of its application to the million-year-old coastal dune sequence in South Australia (Prescott et al., 1993; Huntley et al., 1993b, 1994; Huntley and Prescott, 2001), and other Australian sediments (Readhead, 1982, 1988), was devised to overcome the uncertainty involved with extrapolation of the additive-dose growth curve to determine the dose-intercept—as is required for both the total bleach and partial bleach methods shown in Fig. 2. This problem is especially acute for old samples, because a large extrapolation is required (e.g., as shown by the dashed lines in Figs. 2a and b). The second virtue of the Australian slide technique is that any sensitivity changes induced by the initial bleach of the regenerated aliquots can be identified

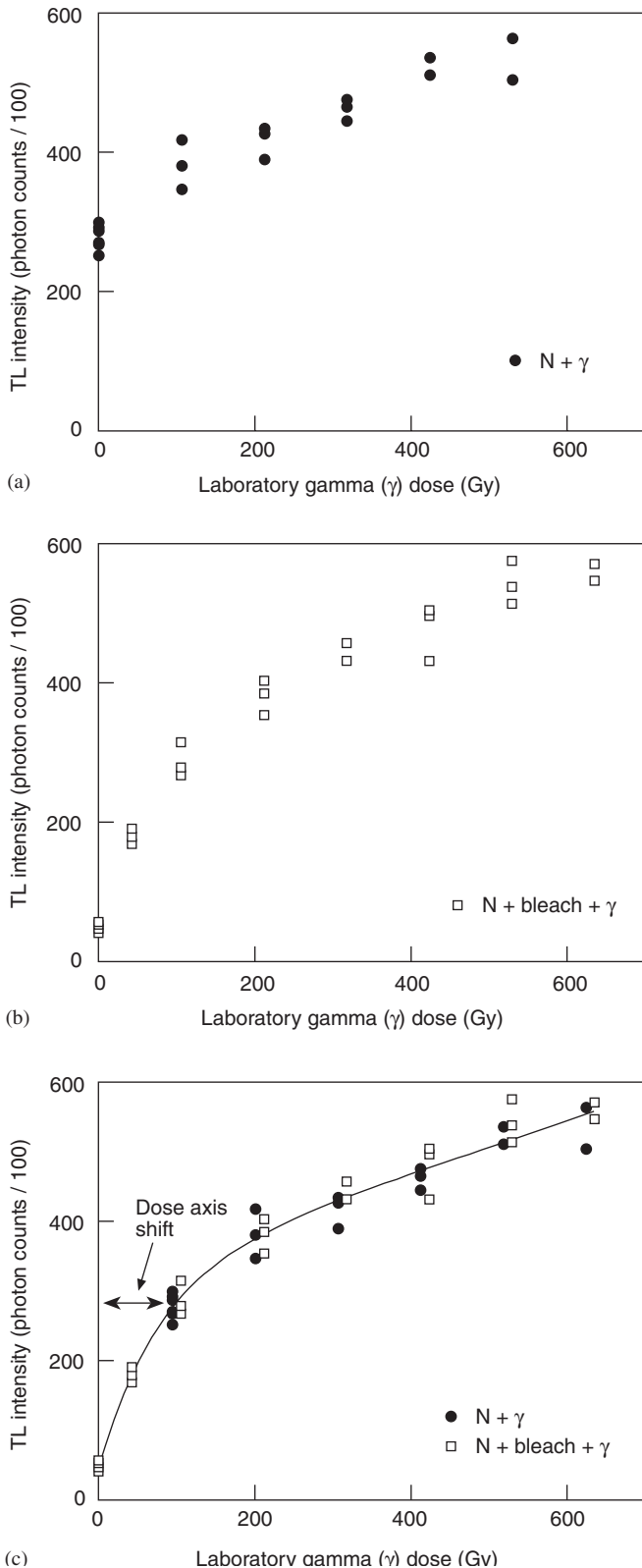


Fig. 3. Illustration of the Australian slide method, a combined additive- and regenerative-dose technique used for TL dating as well as for optical dating. Several aliquots are prepared; roughly half of them are given various laboratory doses ( $N + \gamma$  set) to construct the sample's dose-response (a), while the remaining aliquots are given a prolonged exposure to light to empty all of the light-sensitive traps, and are then given various regenerative doses ( $N + \text{bleach} + \gamma$  set), as in (b). The doses given to the regenerative set of aliquots are chosen so as to overlap most of the range of doses given to the additive-dose set. (c) The  $N + \gamma$  set is then shifted along the dose axis to where it coincides with the  $N + \text{bleach} + \gamma$  set, the magnitude of the shift being equal to the  $D_e$  (98 Gy in this case) after a correction for incomplete laboratory bleaching. The example shown is for sand-sized grains of quartz extracted from a relict beach dune at Policeman's Point on the Coorong in South Australia (sample SESA-121) that gave an apparent TL age of  $\sim 95$  ka; the data shown are from the TL measured at  $360^\circ\text{C}$  (Huntley and Prescott, 2001).

by including an intensity-scaling parameter in the curve-fitting algorithm. If a change in sensitivity is detected, then an equivalent dose (and age) cannot be reliably determined from the data.

The next leap forward in the field of luminescence dating came with the introduction of optical dating (Huntley et al., 1985). Optical dating, also referred to as optically stimulated luminescence (OSL) dating, is similar to TL dating in that the basic mechanism responsible for the generation of luminescence is thought to be the same. The major difference, however, is that light of a specific wavelength or narrow wavelength range—instead of heat—is used to excite electrons from traps (Fig. 4). The profound implication of this is that only the most light-sensitive traps are sampled, which means that the “luminescence clock” is much more easily reset by daylight. As a consequence, there is a smaller chance that a given sample has retained electrons in the relevant traps (and, hence, an inherited age) at burial, and this in turn means that younger samples are better suited to optical dating than to TL dating.

The one disadvantage that optical dating has when compared to TL dating is that, during measurement, there is no way of separating the luminescence derived from thermally stable traps (deep traps) from that derived from thermally unstable traps; the latter are filled during laboratory irradiation but do not remain filled for geological lengths of time. To overcome this, the sample is heated in an oven prior to the measurement. This “preheat” is designed to empty the thermally unstable traps, while leaving most of the thermally stable traps filled. In the initial trials of Huntley et al. (1985), the equivalent dose was estimated from quartz using green (514.5 nm) light from an argon-ion laser for stimulation, a preheat of 250 °C, and a protocol similar to the total bleach method used for TL dating. Three samples were analysed. The first sample was one that had been independently dated to ~60 ka and gave an optical age that was consistent with this. The other two samples were selected because they could not be dated successfully using TL, and it was hoped that the new optical dating method would yield better results. One of these samples consisted of quartz grains extracted from a soil Ah horizon. It gave an equivalent dose consistent with that found previously using TL, which was too high by a factor of two. The other sample consisted of quartz grains from a modern beach sand. The equivalent dose for this sample was 8 Gy, and not zero as expected, but it was nevertheless much lower than that determined previously using TL. These overestimates were attributed mainly to insufficient sunlight exposure prior to burial.

The next significant advance was made by Hütt et al. (1988), who showed that low-intensity infrared (IR) stimulation can result in relatively intense luminescence from feldspars, but not quartz, due to the radiative recombination of electrons evicted from traps that are thermally stable at environmental temperatures over geological time. This finding was surprising, as the basic

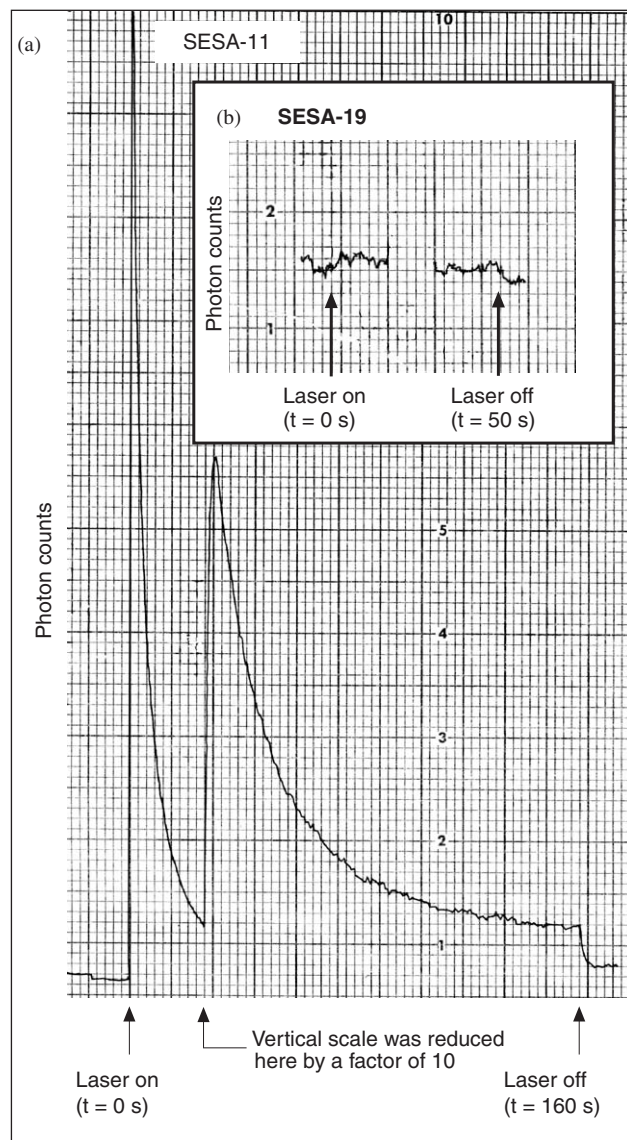


Fig. 4. Optically stimulated luminescence intensity versus time, or “shine-down” curve for two quartz samples. The stimulating 514.5 nm green light was from an argon-ion laser and was filtered by a 520 nm interference filter and Corning 3–70 and 3–71 glass filters; the laser intensity at the sample was  $50 \text{ mW cm}^{-2}$ . Luminescence was detected by an EMI 9635 photomultiplier tube behind several Corning 7–59 glass filters and a Corning 5–58 glass filter. The images shown were scanned from the original chart recorder paper and show data collected during the very first optical dating experiments, which were conducted by D.J. Huntley, D.I. Godfrey-Smith and M.L.W. Thewalt in the Physics Department at Simon Fraser University on May 7, 1984. The main graph (a) shows the shine-down curve for quartz sample SESA-11, collected from a >780 ka raised beach dune at East Naracoorte, South Australia. The vertical scale was reduced during the measurement after ~28 s by a factor of 10 to better resolve the decay. The inset (b) shows the shine-down curve for sample SESA-19, collected from a modern beach dune in the same region of South Australia (15 s of data from the middle of the curve is missing); the OSL intensity from this sample is indistinguishable from background. The data from these two samples together suggested that optical dating was likely to be feasible, and led to the results presented in the seminal paper by Huntley et al. (1985). Note that the shine-down curve for sample SESA-11 is not a single exponential decay, but consists of the sum of several such functions, each reflecting a population of electron traps with a different sensitivity to light. When the laser is switched on, the most light-sensitive traps are emptied first.

physical models available at the time indicated that IR photons were not sufficiently energetic to directly eject electrons from deep traps. Nevertheless, many comparisons with independently dated samples showed the method to be valid, and this, in turn, indicated that a more complex mechanism was responsible for electron eviction and recombination, as was borne out by later detailed studies (e.g., Spooner, 1994). The consequence of Hütt et al.'s discovery was that optical dating could be done using affordable light-emitting IR diodes instead of expensive lasers, and, if appropriate optical filters were used, that feldspars could be preferentially measured in samples consisting of mineral mixtures.

During the initial trials of optical dating, one of the concerns was that the preheat used to empty electrons from the thermally unstable traps filled during laboratory irradiation would also result in unwanted “thermal transfer”: the transfer of electrons from light-insensitive, thermally stable traps to the light-sensitive traps sampled during measurement. If this transfer were significant, then the equivalent dose would be overestimated, and hence the age calculated from it would be too old. To alleviate this problem, Huntley et al. (1993a) introduced the additive-dose with thermal-transfer correction (ADTT) method (Fig. 5). The ADTT method also accounts for unwanted signal arising from detector background counts and Raman-scattered photons. These components can also be corrected for by using the late-light subtraction method (Aitken and Xie, 1992), but this technique does not correct for thermal transfer. The Australian slide method described earlier for TL dating can also be used for optical dating in cases where extrapolation of the additive-dose curve to the dose axis is expected to be large. Moreover, because both the additive- and regenerative-dose sets of aliquots are preheated together, a separate correction for thermal transfer is not needed.

The initial TL and optical dating protocols both required that many aliquots (typically 20–50), each consisting of many thousands of sediment grains, be used to construct a sample's dose response (e.g., as in Figs. 2 and 5). With these methods, much of the uncertainty in the equivalent dose results from the inherent variability of the sensitivities of the many grains that make up each aliquot, and the fact that only a small percentage (typically <10%) of the grains in any aliquot are responsible for the measured luminescence. This problem is overcome to a certain extent by normalisation (Fig. 5), but it is never eliminated. Moreover, it is difficult (but not impossible, e.g., Huntley and Berger, 1995; Ollerhead et al., 2001) using multiple-aliquot techniques to produce accurate ages in environments where the sediment grains have not all received sufficient sunlight exposure prior to final burial. This difficulty led workers to consider other ways to model the dose response.

One of the advantages that optical dating has over TL dating is that, in most cases, an easily measurable quantity of luminescence can be generated by sampling only a small proportion of the relevant electron traps. This led Huntley

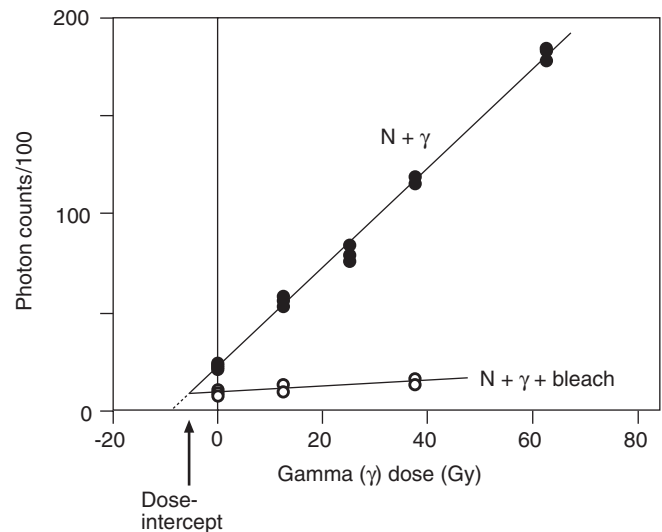


Fig. 5. Illustration of the additive-dose with thermal-transfer correction (ADTT) method used for optical dating of sediments (based on Fig. 7 of Lian and Huntley, 1999). The data are from the violet luminescence emitted by fine-silt K-feldspar from a 2.4 ka aeolian deposit (sample CCL4) when excited by IR radiation. Several aliquots were prepared, and were normalised to reduce the scatter in the luminescence that results from the intrinsic sensitivity differences between the aliquots. Normalisation is accomplished by briefly exposing the aliquots to the stimulating light source, and the recorded luminescence intensity is divided by the average value for all of the aliquots in the set, thus producing normalisation factors that are used to reduce the scatter in the final measurements. After normalisation, some aliquots are left undosed (N), while the others are given various radiation doses (from a gamma source in this case). Some of these aliquots are left as is (N + γ set), and others are given a light exposure to empty the light-sensitive traps while leaving most of the light-insensitive traps full (N + γ + bleach set). All of the aliquots are then preheated and, after a delay (usually several days), are measured together and curves fitted to the data. The equivalent dose is taken as the point where the two curves intercept above the dose axis (the dose-intercept). Dose intercepts are usually determined as a function of measurement time. If dose-intercept values are found to rise with measurement time, then it may mean that some of the grains in the sample have not been adequately bleached in the environment (e.g., Lian and Huntley, 2001; Lian et al., 2002), although this interpretation is questionable for quartz when multiple-aliquot techniques are used to determine the equivalent dose (Roberts et al., 1999; Bailey, 2000).

et al. (1985) to propose that a sample's dose response could be constructed using just a single aliquot of sediment. This prompted several researchers to develop and test single-aliquot dating protocols. The first published results of dating single multi-grain aliquots of K-rich feldspar were those of Duller (1991, 1994, 1995), while dating individual grains of this mineral was first investigated by Lamothe et al. (1994) and Lamothe and Auclair (1997). Comparable studies of single multi-grain aliquots of quartz were first reported by Liritzis et al. (1994) and Stokes (1994), while Murray and Roberts (1997) and Roberts et al. (1997) experimented with dating single grains of quartz.

In general, these early trials used additive-dose procedures, where the equivalent dose was estimated by extrapolation to the dose axis of the dose–response curve attained from a single aliquot or grain; these protocols

became known as single-aliquot additive-dose (SAAD) procedures. One of the problems with using a single aliquot to model a sample's dose response is that the aliquot must be repeatedly dosed, preheated, and optically stimulated, and this commonly results in luminescence sensitivity changes that have to be corrected for. For that reason, early single-aliquot protocols often relied on the employment of a second aliquot to monitor sensitivity change. More than one aliquot is also required for the single-aliquot regeneration and added dose (SARA) protocol of Mejdahl and Bøtter-Jensen (1994). In principle, this offers a means of correcting for sensitivity changes, but it suffers from the same handicap as any other multiple-aliquot technique in assuming that the equivalent dose for all aliquots in a sample is the same. This assumption is not generally valid for sediments, which may be buried with substantially different numbers of electrons remaining in traps, so the application of SARA has been mostly restricted to heated archaeological materials.

Galloway (1996) was the first to develop a SAAD protocol that required only one aliquot of feldspar to obtain an estimate of the equivalent dose, and Murray et al. (1997) extended this approach to single aliquots of quartz. However, SAAD protocols are still vulnerable to progressive changes in luminescence sensitivity arising from the repeated application of preheats, and this led to the development of single-aliquot regenerative-dose (SAR) protocols. Early SAR protocols, developed for quartz, monitored changes in sensitivity using the 110 °C TL response measured during preheating of each of the natural and regenerative doses (Murray and Roberts, 1997, 1998; Roberts et al., 1997), whereas subsequent variants (e.g., Roberts et al., 1998a; Galbraith et al., 1999; Murray and Mejdahl, 1999; Murray and Wintle, 2000, 2003; Jacobs et al., 2003a; Olley et al., 2004a; Wintle and Murray, 2006) account for sensitivity changes by monitoring the sample's response to a small dose (a "test dose") administered to the same aliquot immediately after the luminescence from the natural dose and each regenerative dose has been measured (Fig. 6). The effect of thermal transfer is observed from the change in equivalent dose (if any) as a function of preheat temperature, and from the luminescence intensity observed after a "zero" regenerative dose is given. An internal check on the effectiveness of the sensitivity correction is made by repeating at least one of the data points in the dose-response curve (Fig. 6), and it is also recommended that a "dose recovery" test be performed on each sample. In this test, fresh aliquots are bleached at room temperature (either by filtered laboratory light or by natural sunlight) to eliminate the light-sensitive natural signal, and then given a known laboratory dose. This surrogate natural dose is measured using the SAR protocol to check that the correct (given) dose can be recovered.

SAR protocols were originally devised for quartz and have since been extended, with mixed success, to feldspars. They currently are the most robust procedures available for determining the equivalent dose of sediments from a

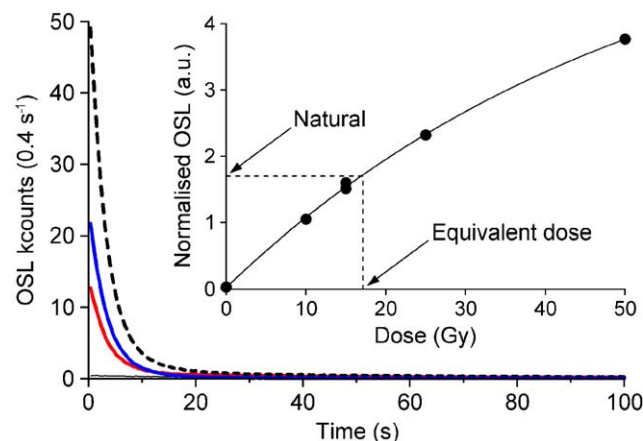


Fig. 6. Examples of continuous wave (CW) OSL shine-down curves for a single aliquot of sand-sized quartz (from sample M3T 58.0m, Lake Mungo, southeastern Australia), with the corresponding dose-response curve shown in the inset graph (Bowler et al., 2003). The main graph shows the CW-OSL curves resulting from the "natural" dose (blue line), a test dose of 10 Gy (red line), and regenerative doses of 50 Gy (dashed black line) and 0 Gy (thin black line, just above the x-axis). Additional regenerative and test doses were also given as part of the SAR protocol. The aliquot was optically stimulated using blue (470 nm) light-emitting diodes set at constant intensity for the 100 s duration of illumination, and all OSL signals were calculated from the initial 3 s of data, with the final 30 s being used to estimate the background count rates. The resultant dose-response curve (inset graph) has two features of note. First, the duplicate dose point at 15 Gy has a "recycling ratio" consistent with unity ( $1.06 \pm 0.03$ ), indicating that the test dose sensitivity-correction procedure has performed satisfactorily; and second, the zero-dose intensity is only 1.6% of the natural intensity, indicating that thermal transfer (induced by the preheat given immediately before optical stimulation) is negligible in this sample.

variety of depositional environments, and their ability to measure the dose response of individual grains has enabled very small samples to be investigated. But the greatest benefit for Quaternary scientists is that the distribution of equivalent doses determined from a sample can be analysed, thereby allowing issues of sample contamination to be addressed, such as the identification of grains that have been insufficiently bleached before burial and the recognition of sediments that have been adversely affected by post-depositional mixing. Extraction of the desired equivalent dose (usually that representing the true burial age) from the measured dose distribution is one of the major current challenges in luminescence dating.

All of the above optical dating protocols have been used primarily to isolate the most light-sensitive signal from the luminescence emission, with the intensity of the stimulating light source held constant (referred to as "continuous wave" or CW excitation). In the seminal paper (Huntley et al., 1985), it was noted that the observed decrease of luminescence with stimulation time (the shine-down curve) did not follow a single exponential, but appeared to be made up of several exponentials (Fig. 4), each with a different rate of decay, suggesting that the measured luminescence originated from more than one type of trap. Smith and Rhodes (1994) deduced that a

shine-down curve for quartz consists of three main components (see also Bailey et al., 1997), commonly referred to as “fast”, “medium” and “slow” in decreasing order of sensitivity to illumination. This, in turn, inspired the development of linear modulation (LM) techniques, in which luminescence is measured while the power of the stimulating light source is ramped from zero to some maximum value (Bulur, 1996). Using the LM technique, the various components can be separated by sampling different parts of the shine-down curve. At least 7 components have been recognised in quartz (Bulur et al., 2000; Jain et al., 2003; Singarayer and Bailey, 2003), although not all of them occur in every grain (Bulur et al., 2002; Yoshida et al., 2003) (Fig. 7).

While the methods used to estimate the equivalent dose were being perfected, there was also considerable research into understanding the physical mechanisms related to optical dating. For example, Baril and Huntley (2003a, b) have shed new light on the process responsible for IR-excited luminescence emitted from feldspars. Much of the work on physical mechanisms has been summarised in special issues of *Radiation Measurements* [e.g., from 1997 (Vol. 27, No. 2), 2000 (Vol. 32, Nos. 5–6) and 2003 (Vol. 37, Nos. 4–5)] and in Bøtter-Jensen et al. (2003). The journal *Ancient TL* also publishes papers on various experimental and theoretical aspects of luminescence

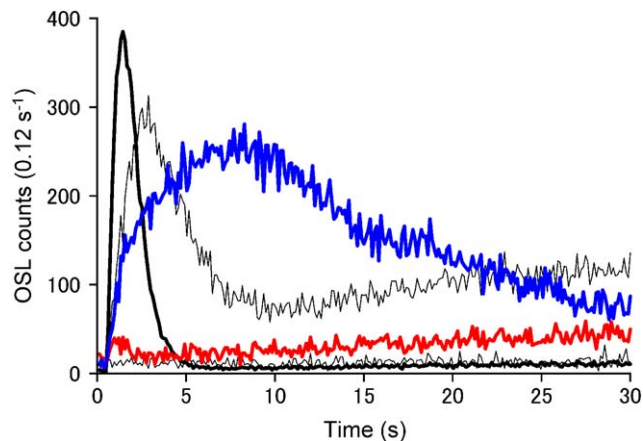


Fig. 7. Examples of linear modulation (LM) OSL response curves for four individual sand-sized grains of quartz from sample M3T 58.0 m, Lake Mungo, southeastern Australia (Bowler et al., 2003). The electron traps in the “natural” aliquot were excited using a focused green (532 nm) laser, steadily increasing the laser power from zero to 90% (i.e., from zero to  $45 \text{ W cm}^{-2}$  at a single grain) over the 30 s period of illumination. The typical background LM-OSL signal for the instrument is shown by the thin black line, which plots just above the  $x$ -axis. The other thin black line represents a grain that contains traps associated with more than one OSL component, whereas the thick black line shows the response of a grain for which the measured luminescence (multiplied by 0.2 for purposes of presentation) is dominated by that originating from traps that are the easiest to bleach—i.e., the “fast” component. The curves shown in blue and red are for grains that contain larger relative amounts of “medium” and “slow” components, respectively. For this sample, the total LM-OSL output for a collection of grains over the first 5 s of stimulation is dominated by grains with an intense fast component, with 55% of the total light sum being contributed by just 10% of the grains.

(and electron spin resonance, ESR) dating, and these are available free online at [www.aber.ac.uk/ancient-tl/](http://www.aber.ac.uk/ancient-tl/).

As the techniques used to model the equivalent dose have advanced, so too has the instrumentation. Early luminescence readers consisted of devices for which only one aliquot of sample could be measured at a time, aliquots had to be changed manually, and irradiations and preheating were performed using separate irradiators and ovens (e.g., as in Fig. 1.2 of Aitken, 1985, and Fig. 1 of Huntley et al., 1985). These instruments were soon replaced by automated machines, in which up to 64 aliquots could be analysed in sequence, and irradiations and preheating were performed on board; the first of these became commercially available in 1983 (see review by Bøtter-Jensen, 1997). The latest advances to automated readers include attachments that allow hundreds of individual sand-sized grains of sediment to be measured quickly in a single run using light from a relatively cheap semiconductor laser as the stimulation source (Duller et al., 1999), and an instrument that can sort grains on the basis of luminescence intensity has also been developed (McCoy et al., 2000). There are also attachments available for commercial automated readers to measure radioluminescence (the prompt luminescence emitted during exposure to ionising radiation), to irradiate samples at elevated temperatures, and to enable an X-ray generator to be used instead of a beta source for laboratory irradiations (Hashimoto et al., 2002; Bøtter-Jensen et al., 2003). Probably the biggest single advance in instrumentation was the introduction of affordable light sources for stimulation, the most efficient of these being high-powered light-emitting diodes that replaced expensive lasers.

#### 4. Current research and development

In this section, we discuss some of the recent developments in luminescence dating and give examples of the application of the various luminescence dating techniques to solving problems in Quaternary research, with specific reference to the papers presented in this special issue.

The development of the SAR technique for measuring single aliquots has shown that different equivalent doses are obtained from different aliquots of the same sample. This has introduced the challenge of how to distinguish between variations in equivalent dose due to the bleaching history of the sample and those due to local dosimetry influences and intrinsic differences in the luminescence sensitivity of the aliquots measured. Several methods of analysing equivalent dose distributions from single aliquots have been proposed (e.g., Olley et al., 1998; Stokes et al., 2001; Lepper and McKeever, 2002), and of these the most sophisticated methods, based on well-established statistical principles, are those of Galbraith et al. (1999). The latter authors describe some general statistical models, developed originally for analysis of fission track data (see Galbraith, 2005), that may be applicable to luminescence samples from a wide range of geomorphic settings.

One such model, known as the “central age model”, calculates the weighted-mean equivalent dose for a set of single-aliquot or single-grain estimates, taking into account the extra spread (“overdispersion”) above and beyond that associated with the measurement uncertainties. Even samples that had been well-bleached at the time of burial can exhibit substantial overdispersion (e.g., up to 20%) in their equivalent dose values (e.g., Roberts et al., 1998b, 2000; Jacobs et al., 2003a, b, 2006; Olley et al., 2004a, b; Galbraith et al., 2005); the exact causes of this overdispersion are not fully understood.

Another model described by Galbraith et al. (1999) is known as the “minimum age model”, which provides an estimate of the sample equivalent dose for the lowest-dose population of aliquots or grains. This model has been tested in simulations and provides correct dose estimates for young fluvial samples of known age (Olley et al., 2004a)—one the most troublesome types of deposit for luminescence dating, because incomplete bleaching prior to deposition will have the greatest effect on the measured equivalent dose. With the minimum age model, some estimate of the underlying overdispersion should also be taken into consideration, preferably from a sample of well-bleached grains derived from the same source (Galbraith et al., 2005).

Statistical models should, of course, be applied in tandem with consideration of sedimentary process and other lines of evidence about the bleaching and burial history of the deposit. For example, the minimum age model may not be appropriate for sediments that have experienced post-depositional mixing or for deposits in which the spread in equivalent dose is due to factors other than partial bleaching (e.g., large spatial variations in the beta dose rate, which would result in substantial differences in the dose absorbed by individual grains at the scale of a few millimetres). In some circumstances, a “maximum age model” may be appropriate (see Olley et al., 2006—this issue), while another potentially useful model is the “finite mixture model” (Galbraith, 2005), which can be applied to sediment mixtures to identify discrete dose components (e.g., Roberts et al., 2001; Rodnight et al., 2005; Jacobs et al., 2006). The latter model requires an accurate estimate of the inherent dose overdispersion in the sample to deduce the correct number of components and their corresponding doses, together with the correct relative proportions of these components (Roberts et al., 2000), although a Bayesian approach may be less sensitive to “outliers” in the data (Sivia et al., 2004).

A related aspect to these statistical models is the issue of data presentation. A feature common to single-grain and some (multi-grain) small-aliquot data is that equivalent dose values are measured with a wide range of precisions, the most luminescent grains providing the best counting statistics and, hence, the highest measurement precisions. (The latter, however, should be treated with caution, as the measurement uncertainties associated with the equivalent doses of individual, well-bleached aliquots are commonly much smaller than the variation in equivalent dose between these same aliquots (Galbraith et al., 2005); hence the need

to explicitly account for dose overdispersion when calculating the sample equivalent dose.) A standard frequency distribution (histogram) is not well suited to displaying

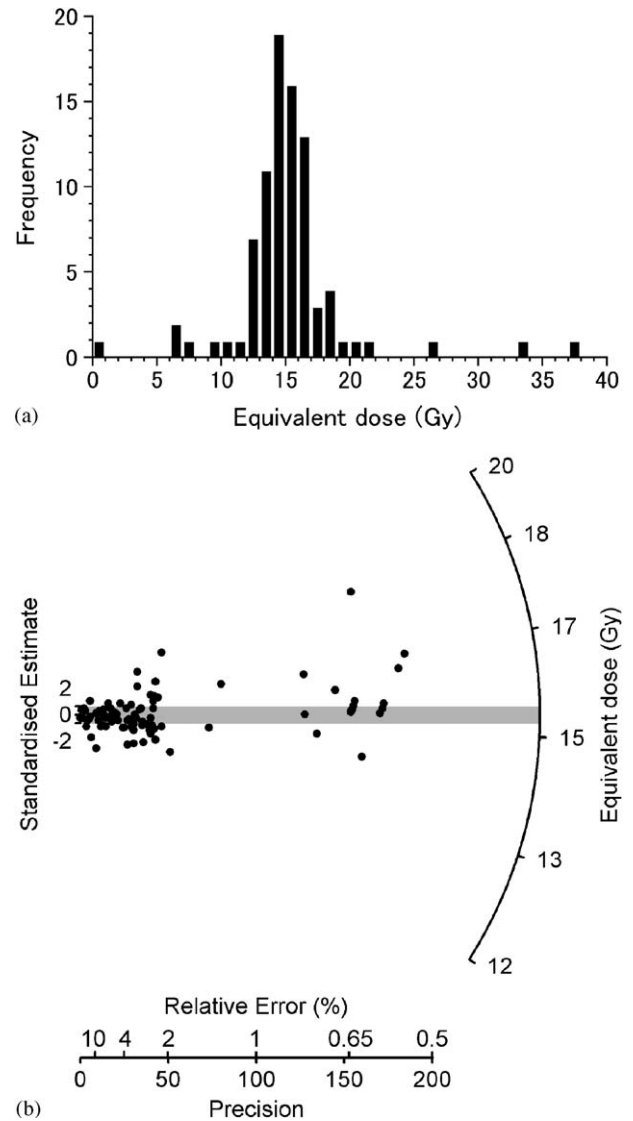


Fig. 8. (a) Frequency distribution (histogram) of the equivalent doses obtained for 86 small aliquots of sand-sized quartz from sample M3T 58.0 m, Lake Mungo, southeastern Australia (Bowler et al., 2003). (b) Radial plot of the same data as displayed in (a), where the shaded band is centred on an equivalent dose value of 15.3 Gy, which is the weighted-mean value of all 86 independent estimates. The histogram shows several equivalent doses of  $>20$  Gy, but some of these are measured with poor precision, which is not apparent using this type of data display. With the radial plot, however, each estimate of equivalent dose is shown together with its associated precision: imprecise values fall to the left of the radial plot, whereas values measured with the greatest precision lie farthest to the right. For this sample, equivalent doses measured with relative errors of  $<1\%$  are distributed over a narrow dose range (14–18 Gy). A further point of interest in the radial plot is the large number of values, of both high and low precision, that falls outside the shaded band. This indicates visually that the data are distributed more broadly than can be accounted for solely on the basis of the measurement uncertainties, and the amount of overdispersion can be determined numerically using the central age model. In this instance, the data are overdispersed by  $13.9 \pm 1.2\%$ , which is a value that falls within the range reported for several other samples of well-bleached quartz. See text for details.

such data. Fig. 8a shows a histogram of 86 single-aliquot estimates of equivalent dose for sample M3T 58.0 m, which was collected from the same stratigraphic layer as that which contained the Lake Mungo III skeleton (Bowler et al., 2003; Olley et al., 2006—this issue). The majority of doses are centred around a value of  $\sim 15$  Gy, but there are a few higher and lower doses. From the histogram, the high-dose values might be interpreted as representing aliquots composed of a few poorly bleached grains, but the measurement uncertainties on the two highest values ( $37 \pm 28$  and  $33 \pm 15$  Gy) are too large to support this inference. While “probability density plots” or “weighted histograms” may appear to avoid this problem, they are not necessarily straightforward to interpret and can be quite misleading (Galbraith, 2005).

To circumvent the ambiguities inherent in histograms, Galbraith et al. (1999) advocated the “radial plot” for the presentation of single-aliquot and single-grain data. Fig. 8b is a radial plot of the same data as those displayed in Fig. 8a. The radial plot displays not only the equivalent dose value for an aliquot (found by drawing a line from the  $y$ -axis (“standardised estimate”) origin through the dose point of interest until it intersects the radial axis, at the value of the equivalent dose, on the right-hand side) but also its measurement uncertainty (expressed as the standard error, found by extending a line vertically to intersect the  $x$ -axis). The latter is displayed as both “relative error” and as its reciprocal (“precision”), so that the dose estimates measured with the highest precision plot farthest to the right. Fig. 8b shows clearly that the largest equivalent dose measured with high precision (relative error of  $< 1\%$ ) is  $\sim 18$  Gy, which falls within the main cluster of doses shown in the histogram. A further feature of the radial plot is that any dose overdispersion can be readily identified, as 95% of the dose estimates should lie within  $\pm 2$  units of a common value on the standardised estimate axis. Clearly, this is not the case with the single-aliquot data for sample M3T 58.0 m, which are overdispersed by 14%, a value typical for small aliquots and single grains of well-bleached quartz (Galbraith et al., 2005).

Despite these improvements to the analysis and display of single-aliquot and single-grain data, a most pressing challenge remains that of understanding the nature of the distribution of equivalent doses that often occurs in a single sample, and how to determine an accurate age from an equivalent dose distribution. This problem is addressed in this issue by Bailey and Arnold (2006). They model the response of the quartz OSL to various irradiation and bleaching conditions to test the fidelity of current techniques used to extract the “true” equivalent dose (and age) from a sample composed of grain populations, each with a different equivalent dose. The main finding of their study is that no single method of analysis is applicable to all samples (depositional environments), but that it is possible to choose the most appropriate model on the basis of the shape of the equivalent dose distribution. Bailey and

Arnold also suggest a guide that can be used to help decide which is the most appropriate data-analysis technique to use on a case-by-case basis.

One of the other challenges that face luminescence dating researchers is perfecting the use of feldspars as geochronometers. Despite the fact that feldspars have properties that make them, in many cases, superior to quartz (e.g., see Huntley and Lamothe, 2001; Lian et al., 2002), most of the recent techniques of modelling equivalent dose (e.g., the SAR protocol) have only been comprehensively tested on the latter mineral. The use of feldspars is, however, important because (i) in most instances, feldspars emit much more luminescence than does quartz, which means that they can be used to date younger samples; (ii) the luminescence from feldspars usually saturates at higher doses compared to that from quartz, therefore older samples can be dated; and (iii) there are cases where quartz does not emit any measurable luminescence at all.

The reason that feldspars are used less than quartz for optical dating is that they have some malign properties. One of the difficulties with K-feldspars when using the SAR protocol is that changes in luminescence sensitivity cannot, in many cases (e.g., Wallinga et al., 2000), be adequately corrected for using current routines (e.g., by application of a test dose). However, the most significant problem with feldspars is so-called “anomalous fading”, which refers to the loss of electrons from traps that are thermally stable at ambient temperatures over geological time; this effect is thought to be due to quantum-mechanical tunnelling of electrons from thermally stable traps to other defects and centres. The phenomenon was probably first observed by Wintle (1973) for feldspars, and much of the research on the mechanism responsible for it has been done by R. Visocekas (see review by Visocekas, 2002). In the feldspar family of minerals, the dominant violet emission from some K-rich (alkali) feldspars has been observed to fade less than in other feldspars (e.g., Spooner, 1994), and that finding, together with the fact that the dose rate from  $^{40}\text{K}$  within the grains is well defined, has resulted in them becoming the feldspars of choice for optical and TL dating.

One way to circumvent the effects of anomalous fading is to select plagioclase feldspars and measure the ultraviolet TL emitted at high temperatures (Guérin and Valladas, 1980) or to use the red TL emitted from most feldspars at high temperatures, as it has been shown not to fade for the samples studied (Zink and Visocekas, 1997). However, it can be difficult to measure these signals because they are dim, and the red TL is largely masked by the red-hot incandescent glow; moreover, the red TL signal bleaches more slowly than the IR-stimulated violet luminescence from this mineral (see review by Fattahi and Stokes, 2003). Another option is to measure the IR-stimulated red emissions from feldspars, which early trials (reviewed by Stokes and Fattahi, 2003) indicated do not suffer badly (if at all in the far-red) from anomalous fading. Further

work will clarify the optical dating potential of these far-red IRSL signals (e.g., Arnold et al., 2003).

A third option is to develop a method to correct violet-emission optical ages for anomalous fading. Huntley and Lamothe (2001) did just that, but their method is only valid for samples that are within the linear part of their dose response. In this issue, Huntley and Lian (2006) present some further insights into the nature of anomalous fading. These authors found that, for laboratory-irradiated K-feldspar samples, there is a clear link between the anomalous fading rate of the IR-excited violet luminescence and how close the sample was to saturation when collected from the field. This paper also presents insights into the thermal lifetime of the traps in K-feldspar relevant to IRSL dating, and information on the identities of the elemental impurities responsible for anomalous fading in plagioclase feldspars. Interestingly, all of the 77 feldspar samples studied in this research (comprising sand-sized natural sediment and slices from crystals collected from various parts of the world) exhibited measurable anomalous fading, which gives further support to the notion of Huntley and Lamothe (2001) that anomalous fading of the dominant violet emission in K-feldspars is ubiquitous. By contrast, many fine-silt K-feldspar samples analysed in other studies give IRSL ages consistent with independent chronological information (e.g., Lian and Huntley, 1999; Lian and Brooks, 2004), which suggests that grain size, or the mineral's resistance to weathering, may correlate with the degree of anomalous fading. The observations of Huntley and Lian (2006) should help workers gain a better understanding of the nature of anomalous fading, and accelerate progress toward a general model that can be used to correct for it.

As mentioned above, one of the instances where feldspar is preferred over quartz is when quartz does not emit measurable OSL. The reason for this is not understood, but it has been observed that quartz sediment derived from volcanic rock tends to emit only weakly in the blue and strongly in the red, in contrast to quartz derived from rocks of plutonic origin. In such cases, and if using feldspar is not an option, one has to devise an alternative approach to conventional optical dating, especially if single-aliquot dating is desired. In this issue, Westaway and Roberts (2006) report a novel means of isolating the light-sensitive red TL signal in volcanic quartz collected from Indonesia. They separate the light-sensitive red TL signal from the other TL signals using a modified SAR protocol on two aliquots of the same sample, and derive age estimates for Late Pleistocene cave and river terrace deposits in and around the important archaeological site of Liang Bua on the island of Flores, which recently yielded the remains of a new species of human, *Homo floresiensis* (Brown et al., 2004; Morwood et al., 2004, 2005). They assess the validity of this dual-aliquot protocol by comparing the TL ages with those obtained by radiocarbon dating of charcoal, U-series dating of flowstone and coupled ESR/U-series dating of tooth enamel. They also investigate various

properties of the red TL signal that are relevant to dating, and note that the dual-aliquot protocol has broader application to red TL emissions from other quartz samples.

There are also situations where quartz is lacking in a convenient grain size, where sediments are expected to be older than the typical ~150 ka limit of quartz, or where the quartz is of uncertain abundance (such as occurs in deep-sea sediments) and feldspars are preferred for dating. This is demonstrated by Berger (2006—this issue), who investigated dating protocols for fine-grained (silt) sediments extracted from cores taken from across some basins and ridges of the Arctic Ocean. Berger dated the K-feldspar fraction in polymineral aliquots using multiple-aliquot TL and IRSL techniques, and found that several of the calculated ages were broadly consistent with independent age information up to ~100 ka. For many samples, age overestimates were thought to reflect the complex processes of sedimentation associated with Arctic conditions (e.g., ice-rafting of debris and bottom-current reworking). However, residual ages from core-top sediments were relatively low at some sites, indicating that there is a high likelihood of much older samples yielding ages of acceptable accuracy. This study also discusses which sites (and depositional environments) are thought to be best suited to future luminescence dating work, and recommends the application of SAR protocols to quartz (both as individual sand-sized grains, and as multi-grain aliquots of silt-sized material).

An application of optical dating to the terrestrial Arctic environment is also illustrated in this special issue. Bateman and Murton (2006) present a study of the chronology of sand sheets and sand dunes situated in Tuktoyaktuk Coastlands of the Arctic Coastal Plain in Canada that span the last ~40 ka. Using quartz and the standard SAR technique, Bateman and Murton are able to make important links between the nature and timing of sediment supply, various styles of sedimentation, landform stability, and what is already known about fluctuations in past climate. Of particular note is that the sites sampled by these workers laterally bracket the limit of the Laurentide Ice Sheet, so valuable information about the timing of its demise can be inferred: their data clearly show that glaciofluvial activity in the area was replaced by aeolian deposition by 12 ka and, hence, the last advance of the ice sheet must have occurred between this time and ~21 ka.

Most applications in optical dating now employ the SAR technique, and the dose-response is modelled by sampling the luminescence emitted during the first few seconds (or less) of stimulation. This is to select the luminescence resulting from the most light-sensitive traps, thereby minimising any residual signal. There are instances, however, where one might want to sample both the latter traps and one or more of the less bleachable (i.e., slower) components of the shine-down curve. Such an approach would offer a means of detecting partial bleaching in multi-grain single aliquots and, in principle, single grains of quartz extracted from fluvial sediments (e.g., Bailey, 2003;

Bailey et al., 2003; Singarayer et al., 2005). The existence of incomplete bleaching can be recognised from an increase in equivalent dose with illumination time, whereas the existence of a “shine plateau” in equivalent dose values indicates that the hard-to-bleach traps had been emptied to the same extent as the easy-to-bleach traps; hence, the grain(s) must have been fully bleached at deposition.

A good example of this approach in a different context is given in this issue by Olley et al. (2006), who investigate the potential of dating archived samples of quartz-bearing sediment that once encased human fossils. In this instance, resin-impregnated blocks of grave-fill made during excavations some 30 years previously, and since stored in room light, were disaggregated and the dose-response of individual grains was measured using both CW and LM techniques. Application of a maximum age model to the equivalent doses obtained from the fast-component CW signal produced ages in accordance with expectations, indicating that at least some of the grains embedded in the resin block had remained light-safe. They then estimated the equivalent doses for the fast and slow LM signals, and found that some grains yielded the same equivalent dose from both signals—confirming that some of the embedded grains had been sufficiently shielded from light during storage—and that these grains yielded an optical age consistent with independent chronological information. This dual-signal LM-OSL approach has previously been applied to individual grains from archaeological and deep-sea sediments (Yoshida et al., 2003; Olley et al. 2004b), the latter being one of the original subjects of TL dating research (Huntley and Johnson, 1976; Wintle and Huntley, 1979a, 1980) as well as more recent investigations using optical dating (Stokes et al., 2003). The potential of this approach to identify well-bleached grains in mixed-age deposits has obvious applications to commonly archived sediments (e.g., lake and marine cores) and samples afflicted by partial bleaching and post-depositional disturbance (young fluvial deposits, soils and archaeological sediments being foremost among these), with the possibility of tracking the movement of individual grains in sediment mixtures.

Another significant observation that has come out of the SAR technique is that not all aliquots from a single sample show the same dose-response (e.g. Roberts et al., 1999, Jacobs et al., 2003b). Indeed, there are grains for which the dose-response saturates at doses much higher than is typical of other grains in the sample. These were referred to as “supergrains” by Yoshida et al. (2000). In this issue, Rhodes et al. (2006) further examine the issue of how far back optical dating can be applied, in a study of shallow marine and associated aeolian sands in northwestern Africa. The Casablanca site was first investigated more than 80 years ago, and has yielded abundant archaeological evidence (including a *Homo erectus* jaw and Acheulian stone tools) in addition to a sedimentary record of sea-level fluctuations. Rhodes and colleagues used a variety of optical dating protocols, and concluded that multiple-

aliquot additive-dose methods were ill-suited to the oldest of the Casablanca deposits. By contrast, SAR protocols produced ages that were more precise, internally consistent—a formal test of consistency being made using a Bayesian approach—and in good accord (for most of the samples) with independent age constraints.

The oldest sample analysed by Rhodes et al. was collected from palaeomagnetically reversed sediments, and yielded optical ages of >780 ka from both the fast component (measured under CW stimulation) and one of the three slow components measured by the LM technique. These results support the findings of the TL dating studies of quartz from coastal dunes in southeastern Australia (Huntley et al., 1993b, 1994; Huntley and Prescott, 2001), and optical dating of supergrains (Yoshida et al., 2000) and single aliquots (Banerjee et al., 2003) from the same deposits, which achieved ages approaching 1 Ma. Routine dating of Early and Middle Pleistocene sediments has not yet been demonstrated, however, and several approaches have been made to extend the age-range of luminescence dating methods beyond 1 Ma for quartz. Recent examples include the use of red TL emissions (Fattahi and Stokes, 2000), the OSL slow component (e.g., Singarayer et al., 2000), the isothermal blue TL signal (e.g., Jain et al., 2005) and the recuperated OSL signal (Wang and Lu, 2005).

A range of techniques for measuring the equivalent dose is also shown by the other applications papers in this issue. For the archaeological and human burial site of Roonka in South Australia, Robertson and Prescott (2006) synthesise some of the earliest TL dating studies of dune sediments conducted anywhere in the world (Prescott, 1983) with unpublished TL dating results and a suite of optical ages obtained using a modified SAR protocol. Most significantly, they obtain an age bracket of 20–16 ka for human remains on the east bank of the Murray River, making them among the few securely dated Pleistocene burials in Australia. Also working in the Murray Basin, Cupper (2006) undertook a study to assess the viability of using optical dating of quartz as a geochronometer for Holocene playa lake deposits, which are inherently difficult to date, but are extremely important as proxies for climate change. Cupper extracted sand-sized grains, interpreted to have arrived at the sites via aeolian processes, from four playas. Equivalent doses were determined for aliquots composed of only 5–10 grains using the standard SAR technique. The dose estimates were combined using the central age model to obtain the sample equivalent doses from which the optical ages were calculated, and these data were then compared to the radiocarbon ages of bulk organic carbon collected from the same sample sites. Half of the 10 samples dated showed consistency at the 95% confidence interval between the radiocarbon and optical ages, with discrepancies being attributed to the presence of “dead” carbon in the radiocarbon samples. Of importance is the fact that the optical dating chronology is supportive of existing models of hydrological change in the area.

To the southeast, in northeastern Tasmania, [Duller and Augustinus \(2006\)](#) present a reanalysis of quartz samples from stabilised linear dunes using the SAR protocol; the authors had previously dated some of the same samples using multiple-aliquot techniques. The ages found during the previous study suggested that the dunes had remained inactive during the last glacial maximum (LGM), a result that contradicted palaeoenvironmental information from mainland Australia. However, when the samples were reanalysed using the SAR protocol, the new age data supported dune activity during, or immediately after, the LGM, as anticipated. Duller and Augustinus attribute the discrepancy between the SAR and multiple-aliquot age-data sets to uncorrected sensitivity changes in the dose-response data for the latter. Indeed, when uncorrected SAR ages were calculated, many of them were consistent with the previous multiple-aliquot ages. The authors therefore advise that, in general, unless there is independent age information for comparison, representative subsets of quartz samples dated using multiple-aliquot techniques should be tested using the SAR protocol for the severity of sensitivity change before accepting the multiple-aliquot ages.

On the Indian subcontinent, [Williams et al. \(2006a\)](#) use luminescence dating methods to examine the response of rivers in north central India to changes in the strength of the summer monsoon during the Late Pleistocene. Optical ages were obtained from silt- and sand-sized feldspars using multiple-aliquot additive-dose methods. These revealed that successive phases of river incision and aggradation occurred from 90 ka to the mid-Holocene, which they relate to periods of higher or lower river discharge and the greater or lesser availability of moisture delivered by the summer monsoon. The most marked of the phases of weak monsoon activity extended from ~39 to 16 ka, and coincided with the transition from Middle to Upper Palaeolithic cultures. Intriguingly, shifts from stronger and reliable summer monsoon to weaker, more variable summer rainfall also appear to coincide with two other major archaeological transitions (Lower to Middle Palaeolithic, and Mesolithic to Neolithic) in this important region for Indian prehistoric archaeology.

Farther west in India, on the southern margin of the Thar Desert, [Juyal et al. \(2006—this issue\)](#) applied optical dating to fluvial and aeolian sediments to infer the strength of the southwest monsoon over the last 130 ka. In their study, sand-sized quartz grains were analysed using the SAR protocol and, because of the likelihood of incomplete bleaching before sediment deposition, ages were obtained using an approach akin to the minimum age model. Evidence was found for alternating phases of enhanced and reduced monsoonal activity, with a weak monsoon between ~30 and 11 ka: a similar time interval to that documented further east, in north central India ([Williams et al., 2006a—this issue](#)). Juyal et al. then compare the continental Indian sedimentary records with those preserved to the west, in the Arabian Sea and on the Arabian

Peninsula, where correlations are not so straightforward. They assess the similarities and differences in the intensity and timing of monsoonal activity, and offer an explanation for the lack of correspondence between these sedimentary archives.

Farther west again, [Williams et al. \(2006b—this issue\)](#) explore the timing of the return of the summer monsoon to the climatically sensitive and archaeologically important region of northeastern Africa. They show, using a combination of radiocarbon and optical dating, that the monsoon returned abruptly at ~15 ka, resulting in a megala- lake, 400 km in length, along the valley of the White Nile. This wet event appears to be broadly synchronous with enhanced monsoonal activity in India ([Juyal et al., 2006—this issue](#); [Williams et al., 2006a—this issue](#)) and, as [Williams et al. \(2006b\)](#) discuss, it coincides also with the increased discharge of other major tropical rivers in Africa, the sudden return to warmer and wetter conditions in many inter-tropical parts of the world, and the abrupt warming recorded in the Greenland icecap. As regards optical dating techniques, [Williams et al. \(2006b\)](#) estimated the equivalent dose for sand-sized quartz grains using the multiple-aliquot Australian slide method, which was developed originally for TL dating. The optical ages of three samples were in correct stratigraphic order and in good agreement with the radiocarbon chronology.

The final paper in this special issue ([Lang and Mauz, 2006](#)) involves a study undertaken in central Europe to see whether gully formation can be dated by analysing the sediments that have filled them. Relict gullies are a prominent geomorphological feature on the landscape in central Europe and are an important proxy for past climatic change. Lang and Mauz used the SAR technique, multi-grain aliquots, the minimum age model, and various grain sizes of quartz to date these features. They found that if the larger sand-sized fraction was dated, then optical ages consistent with independent age information could be attained for many samples. An important finding of this study was that all of the gullies were formed during the Holocene, and not in the Late Pleistocene as previously thought. The authors suggest that their chronology could be improved by employing the single-grain SAR technique, but that this may be problematic due to the low luminescence intensity observed in the quartz from this region.

In conclusion, the papers in this issue provide a representative selection of key challenges and new developments in the field of luminescence dating, together with a collection of Earth science and archaeological applications from around the world. The wide geographic range of topics, spanning the last 1 Ma, illustrates the flexibility of luminescence dating methods and their ability to solve long-standing, as well as new, problems in Quaternary research.

## 5. Dedication

This special issue of *Quaternary Science Reviews* was inspired by a conference held in Adelaide, Australia, in

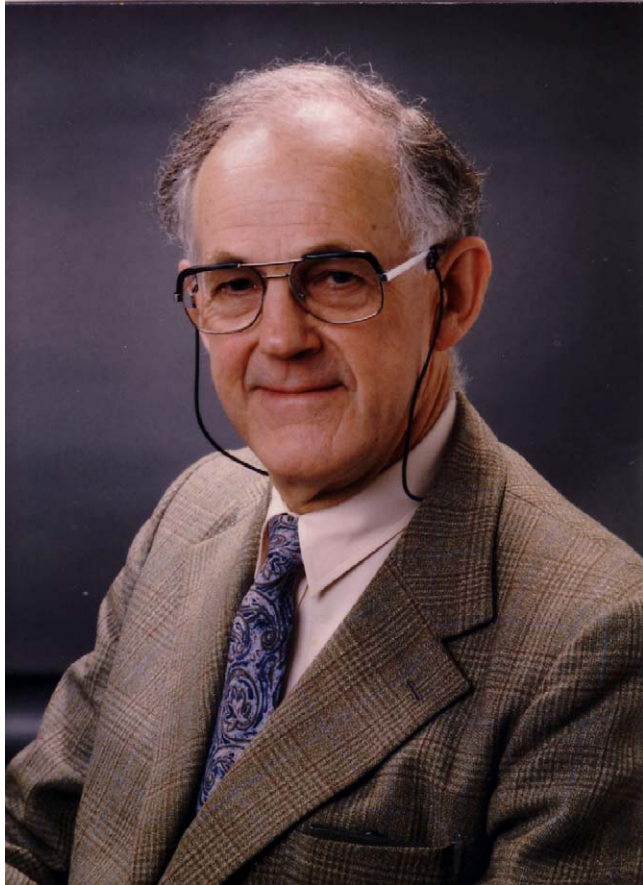


Fig. 9. Professor Emeritus John R. Prescott.

May 2004 to celebrate the 80th birthday of one of the discipline's pioneers and most significant contributors: Professor Emeritus John Prescott (Fig. 9). Professor Prescott, like most of the early developers of luminescence dating, is a physicist who later decided to lend his skills to archaeology and the Earth sciences. John's main research focus in physics had been on cosmic-ray radiation, so it is not surprising that one of his most significant contributions to luminescence dating has concerned the improved estimation of the cosmic-ray dose rate. His principal papers on the subject (Prescott and Jensen, 1978; Prescott and Stephan, 1982; Prescott and Hutton, 1988, 1994) are among the most highly cited in the luminescence dating literature. Some of John's other major contributions include research on the emission spectra of quartz and feldspar (e.g., Prescott and Fox, 1990; Prescott et al., 1994; Scholefield and Prescott, 1999; Scholefield et al., 1994; Franklin et al., 1995, 2000), his work with D.J. Huntley and J.T. Hutton on testing the accuracy of TL dating of quartz (Huntley et al., 1993a,b, 1994; Huntley and Prescott, 2001), and his application of luminescence dating methods to Australian and Pacific archaeology (e.g., Prescott et al., 1982; Prescott, 1983; Smith et al., 1997; Bowler et al., 2003). He also developed the selective bleach method for TL dating of quartz (Prescott and Mojarrabi, 1993), and has made significant geoscience applications in

fields as diverse as palaeoseismology (Hutton et al., 1994), palaeohydrology (e.g., Williams et al., 2001) and, most recently, the dating of meteorite impact craters in Saudi Arabia (Prescott et al., 2004). It is with great appreciation for John's efforts and contributions to the field of luminescence dating that we dedicate to him this special issue of *Quaternary Science Reviews*.

### Acknowledgements

We thank T.F. Flannery, Director of the South Australian Museum, for making available the museum facilities to host the conference held in honour of Professor J.R. Prescott in May 2004. R.G.R. acknowledges the support of the Australian Research Council through the award of a Senior Research Fellowship, and O.B.L. the support of the Natural Science and Engineering Research Council of Canada. H. Yoshida is thanked for drafting some of the figures, and F. Williams for assisting with organising the conference and for providing some of the information on J.R. Prescott. We also thank D.J. Huntley for reviewing a draft of the manuscript.

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