LUMINESCENCE DATING OF QUATERNARY SEDIMENTS – SOME PRACTICAL ASPECTS

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Abstract:

Luminescence dating is based mainly on the dosimetric properties of quartz and feldspar. These minerals are among the most popular found on Earth, resulting in the possibility of using luminescence methods in practically any environment. Currently, quartz remains the best recognized mineral in terms of dosimetric properties, particularly with regards to results obtained for quartz grains, which are regarded as being the most reliable in luminescence dating. Supporters of luminescence methods are constantly growing, however, these groups do not always have sufficient knowledge to avoid even the most basic of issues that may be encountered overall – from the process of sampling through to the awareness of what a single luminescence result represents. The present paper provides an overview of several practical aspects of luminescence dating such as correct sampling procedures and all necessary information regarding the calculation of the dose rate and equivalent dose with particular reference to potential problems that occur when the age of the sample is being determined. All these aspects are crucial for obtaining a reliable dating result, on the other hand, they remain a potential source of uncertainty.

Key words: luminescence dating, dose rate, equivalent dose

INTRODUCTION

Luminescence is the emission of electromagnetic radiation in the visible range caused by the heating or illumination of a substance previously excited by ionizing radiation. Light emission occurs as a result of the release of electrons from trap levels and their recombination with luminescence centres. Only crystalline material with a specific chemical composition called phosphors may be excited. In Pleistocene sediments, such substances are primarily quartz and feldspar. The sources of ionizing radiation are the isotopes of radioactive elements present in the grains of these minerals or in their immediate environment. The most important are ^{40}K and isotopes: ^{232}Th, ^{238}U and ^{235}U. Luminescence dating is based on specific properties of quartz and feldspar that depend on the existence of defects within mineral crystals and the interaction of electrons with these defects (Duller, 2004). These materials are therefore dosimeters, recording the amount of radiation to which they have been exposed (Murray and Olley, 2002).

As the half-life decay of ^{40}K, ^{232}Th and ^{238}U isotopes are comparable to the time of Earth's existence the amount of natural radioactive isotopes in the environment in a given place undergoes only slight changes, even on a geological time scale. Therefore, we can assume that the lon-ger the material stays in a given place, the larger the luminescence signal will be accumulated in the luminescence 'clock'. The process of resetting the luminescence 'clock' is the last exposure of quartz or feldspar mineral grains to daylight (Fig. 1). For aeolian sediments, sufficient exposure to daylight to reset the luminescence signal usually occurs while sediments are eroded, transported and deposited (Hilgers *et al.*, 2001).

As a result, the time elapsed since the sediment grains were buried can be determined by measuring both the luminescence signal and sensitivity from a sample of sediment, and by estimating the intensity of the ionising radiation to which it has been exposed since burial. Establishing the luminescence age of deposition requires different types of measurements. The dose rate and equivalent dose need to be calculated. The final age is calculated using a simple equation:

$$age\,(years) = \frac{equivalent\ dose\,(Gy)}{dose\ rate\left(\dfrac{Gy}{year}\right)}$$

In the 1980s and the 1990s, the TL technique was applied in all luminescence laboratories in the world, however, no standard measurement protocol was ever developed for TL dating, and a variety of light sources, laboratory bleach-
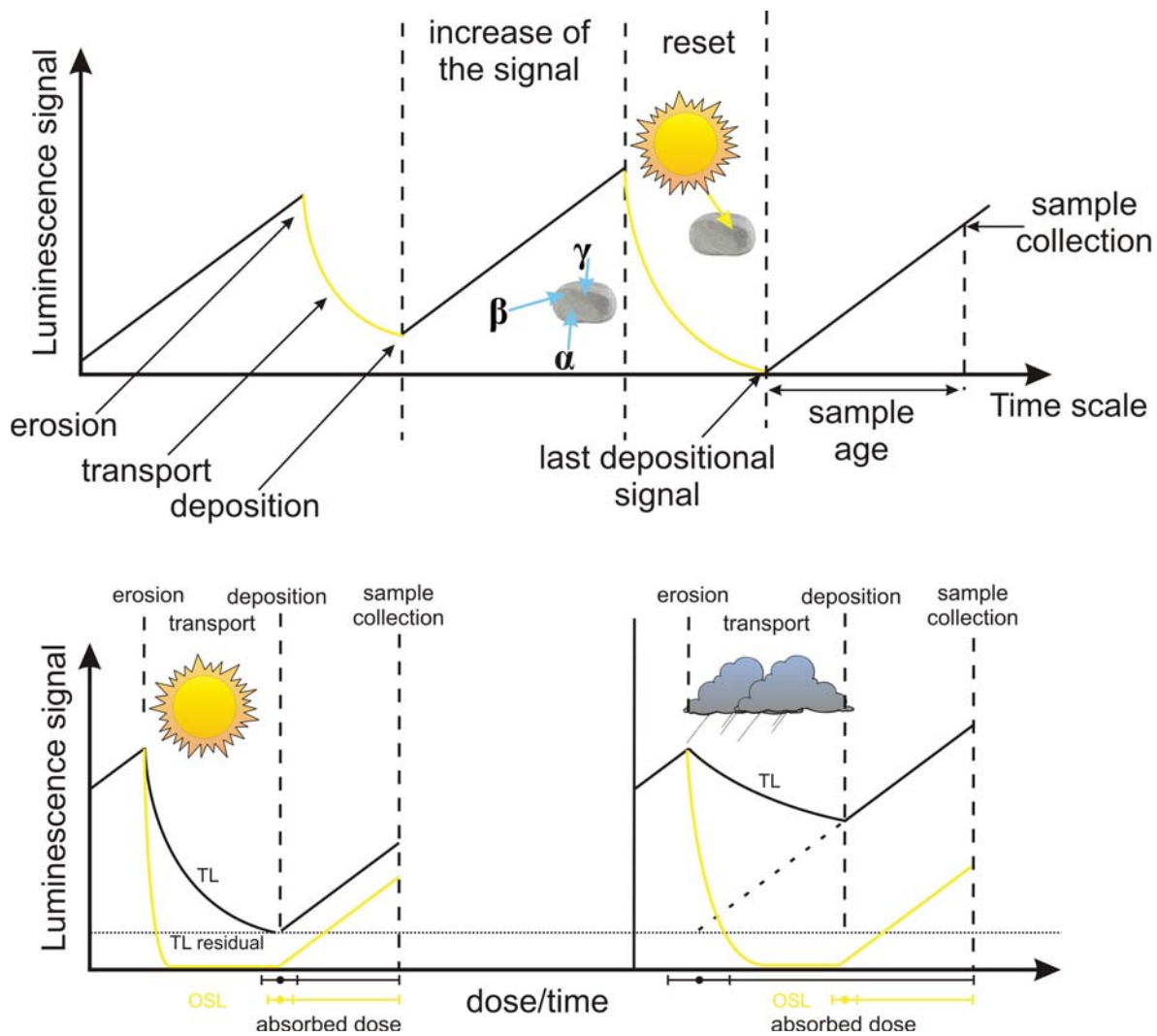
Fig. 1. Basic concepts of luminescence dating, reprinted from Moska and Bluszcz, 2013, with permission from Elsevier. Luminescence signal (number of stored electrons) versus sample history on the time scale and the main differences between the effectiveness of bleaching the OSL and TL signals in a natural environment (without heating to high temperatures).

ing times and preheat temperatures and durations were employed (Aitken, 1985; Wintle, 1990, 1997). The reporting of the exact measurement procedure and conditions is essential, however, if the quality of published ages is to be assessed and age determinations between different laboratories compared (Roberts, 2008). This underwent changes as facilities for optical dating became more widely available and the new single-aliquot regenerative dose protocol (SAR) for OSL dating was developed (Murray and Roberts, 1998; Murray and Wintle, 2000).

Studies using the OSL method are characterized by a wide variety of applications and are conducted for quartz of different fraction. OSL dating was applied to fine silt-sized (4÷11 μm) quartz (Watanuki *et al.*, 2003; Wang *et al.*, 2006; Timar *et al.*, 2010), coarse silt-sized (45÷63 μm) quartz (Roberts, 2006; Stevens *et al.*, 2007), fine sand-sized (63÷90 μm) quartz (Buylaert *et al.*, 2007, 2008) and coarse grains (Moska *et al.*, 2011, 2012). It is widely accepted that quartz

OSL dating using the SAR procedure provides reliable ages, but may suffer from saturation effects for sediments older than 100 ka (Buylaert *et al.*, 2007, 2008; Lowick *et al.*, 2010; Timar *et al.*, 2010; Timar-Gabor *et al.*, 2011).

The great popularity of this method resulted in the creation of the first luminescence laboratories in Poland in the 1980s. From a historical point of view, the first working luminescence laboratories were implemented by M. Proszyński at Warsaw University, J. Butrym at Lublin University, M. Pazdur at the Silesian University of Technology and S. Fedorowicz at Gdańsk University, while the first theoretical studies addressing key aspects related to using luminescence phenomena for dating appeared in the 1980s (Bluszcz, 1986a, b; Pazdur and Bluszcz, 1987a, b). At present, there are three luminescence laboratories in Poland which apply the OSL method: Nicolaus Copernicus University in Toruń, the Silesian University of Technology in Gliwice and Maria Curie-Skłodowska University in Lublin.

TL OR OSL METHOD

Despite the parallel existence of two luminescence methods (TL and OSL), today the OSL is mainly utilised. The OSL method has replaced the previously used TL method owing to the significant advantage which OSL has over TL. The widespread application of the OSL method is associated with a much greater reliability in terms of the obtained results, as the use of this method significantly increases the probability of meeting the basic premise on which the luminescence methods are based, i.e. resetting the luminescence signal during sediment redeposition. Fig. 2 presents results of the renowned experiment showing the process of resetting the luminescence signal for the same quartz material using different method of stimulation (Godfrey-Smith *et al.*, 1988).

It is evident that exposure of quartz to sunlight for 20 hours using the TL method is synonymous with only a few seconds' exposure using the OSL method. In addition, the exposure of quartz to sunlight for approximately 30 minutes is equivalent to the virtually complete bleaching of the accumulated luminescence signal. In fact, it is very common to encounter geological material containing a mixture of grains with varying degrees of bleaching, in which not all quartz grains had been exposed long enough to sunlight. Therefore, it is relatively often possible to not be able to fulfil the basic premises for the OSL method. This also illustrates the low suitability of the TL method for this type of sediment owing to the much longer time needed to reset the natural luminescence signal. This does not mean that the TL method is irrelevant, as it may still be applied in situations in which the material in question has been heated to approximately 500°C. These situations result in the complete resetting of the luminescence signal (TL and OSL) thus deeming both methods satisfactory for the dating of archaeological objects such as bricks or ceramics (Chruścińska *et al.*, 2008).

Resetting the OSL signal

The key question that should be posed by a potential user of luminescence methods is: What is the probability that the material that I would like to date fulfills the basic luminescence premise: how long could it be exposed to sunlight during redeposition?

While in the case of pure quartz extracted from sediment, it takes a few seconds of light exposure to remove the luminescence signal, however, the chance of experiencing such quartz grains in the natural environment is practically reserved for only desert and semi-arid areas. Quartz grains in natural settings usually have iron and manganese oxide surface coatings, and they may have adhering clay grains. The transport medium is rarely transparent; even during aeolian transport (usually considered to be the most ideal bleaching condition) other grains can attenuate light.

Fluvial transport may also meet the premise for the OSL method, however in this case the probability of uneven
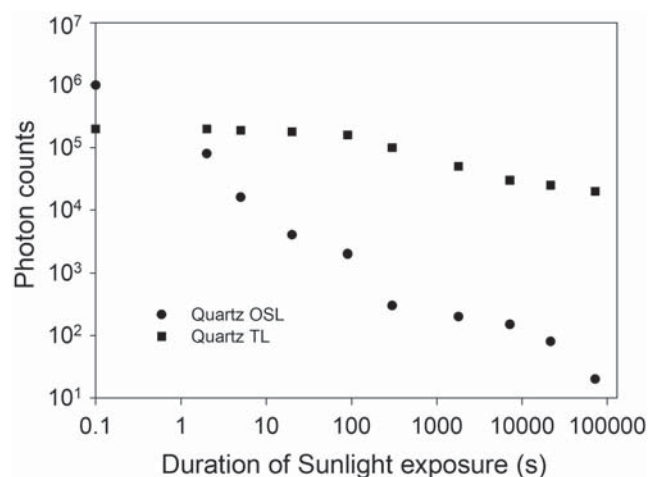

Fig. 2. Loss of luminescence signal as a result of exposure to sunlight, according to experimental measurements (Godfrey-Smith *et al.*, 1988).

bleaching of the signal increases significantly. This may be due to the fact that the medium which is river water is very often not non-transparent, thus making it difficult for the sun's rays to reach more than a few centimeters below the water level.

Oliver (1990) showed that for lowland areas where rivers are characterized by laminar flow, theoretically during which there is a higher probability for longer exposure of quartz grains being transported by the river stream to daylight, all sunlight is absorbed up to 1 metre below the water's surface-level. Thus, in real terms, the highest probability of adequate exposure of quartz grains to sunlight is fulfilled by such rivers, which are characterized by shallow and calm currents in combination with clean water.

As a result of these various processes, the bleaching history of every grain will be different, and only when all grains have had more than sufficient light exposure will a sediment sample be completely bleached. This is usually assumed to be true for aeolian transported material, but it is in the very least questionable for sediments transported in other manners. Incomplete bleaching corresponds most often with multimodal equivalent dose distribution responsible for a statistically significant increase in the obtained equivalent dose, causing the overestimation of the final age.

An example of this erroneous overestimation can be illustrated in the work of the Gliwice Luminescence Dating Laboratory. It concerns an extremely young sample which was collected from colluviums on loess areas located in southern Poland in the Miechów Upland. The sample was collected only several centimeters below the surface and according to geological recognition, this material was deposited on a layer of leaves that had remained from the previous autumn. This meant that over the course of one year there was a rapid flow of material in this area, and during which the luminescence signal was not removed from the quartz. The resulting age distribution is shown in Fig. 3. For this sample it is easy to calculate the final age at approximately
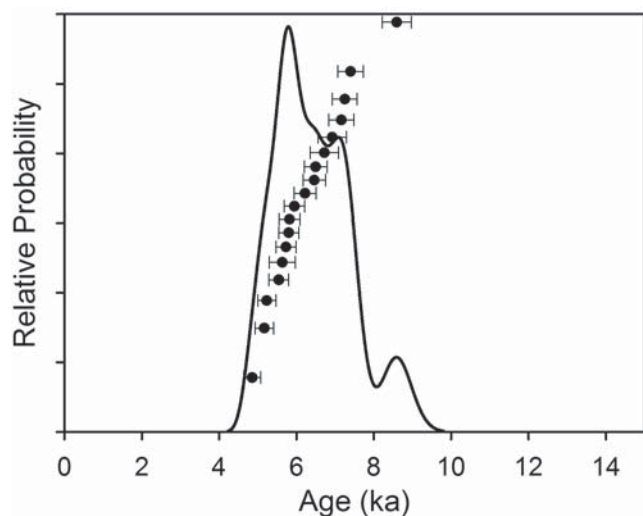
Fig. 3. Example of age distribution for colluvium material which was not bleached during redeposition.

6 ka. So, the final result is overestimated to about 6 ka and only thanks to the appropriate recognition in the field. This is possible to surmise because the distribution itself did not suggest it. Broader discussion of the interpretation problems of luminescence dating in Holocene colluvium was presented by Poręba and coauthors last year (Poręba *et al.*, 2018). Certainly in nature there are many cases of this type that we are not able to predict and at the same time which may be responsible for erroneous environmental interpretations.

SAMPLING AND METHOD

Sampling

One of the most important issues in luminescence dating is the appropriate selection of places from which the material for research is collected. Samples for luminescence dating should be collected from a clean vertical section using thin-walled steel pipes (to protect collected material from sunlight). At the same time as the sample collection, approximately 1kg of sediment should be collected into plastic bags from around the tubes for HPGe gamma spectrometry (which is the most common technique used for dose rate determination). A portable scintillation gamma spectrometer can be used in the specific place from which the sample for luminescence dating is collected. Thus, there is more information which, in turn, allows researchers to obtain the distribution of dose power in the sediment and examine the effect on the dose of sediment layers lying in the vicinity of the point from which the sediment was collected for testing in the laboratory.

Fig. 4 presents a simple example showing proper and less appropriate collection points for luminescence dating. Green points indicate appropriate places for sampling, red points indicate a problematic places.

Why are these places problematic from the point of view of luminescence dating?

Modern soil – the main issue is due to bioturbation, and there is always some evidence that bioturbation has occurred. This process can affect the obtained results as bioturbated samples may contain grains providing both anomalously low and high equivalent dose values;

Thin layers of (1–4 cm), for example, sand in another type of sediment (e.g. loess) – the main issue is owing to the calculation of the correct dose-rate value. Sand usually contains much fewer radionuclide particles than other sediments such as soils or loess. This means that by measuring the dose rate only from a sample from this layer, the results will be consistently underestimated and the final age will be overestimated final. In order to avoid these issues, a portable scintillation spectrometer for recording energy spectra of ionizing radiation in field conditions at sampling points may be used.

A similar situation to the previous one is observed when sampling point is located too close to objects which are not representative for deposits in which it is located (larger fragments of rocks, meteorites, erratic boulders, and so on). This situation is also problematic due to correct dose rate calculations, which is related to highly different concentrations of radionuclides between these objects.

The upper part of the fossil soil can be problematic due to dose rate calculations, and from this point of view samples should be collected about 15–20 cm below the border between different kinds of sediments. Obviously soil sediments still can be affected by the bioturbation process, which will manifest itself from wider spreads of equivalent dose distributions.

In summary, in terms of sample collection, some places, such as boundaries between different sediments or places
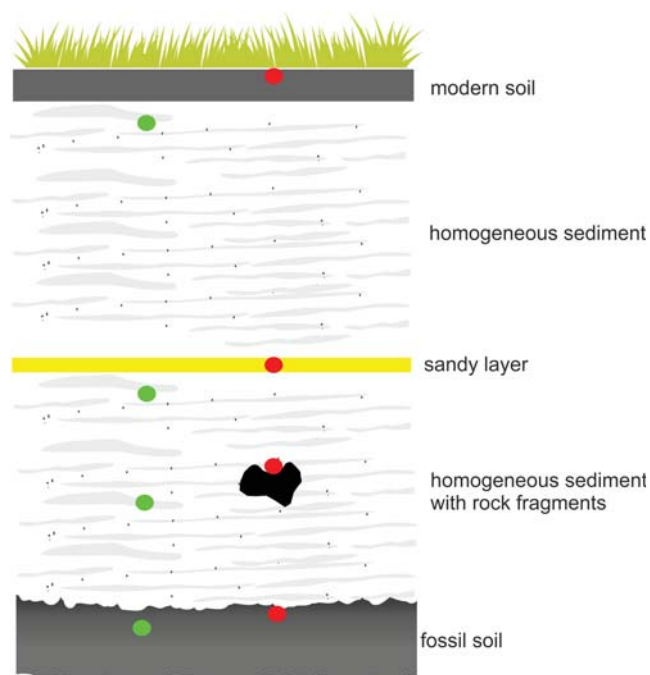
Fig. 4. Examples of correct and incorrect selection of sampling points.

near which there are objects for which significantly different content of radioisotopes are suspected, should be avoided.

Dose rate calculation

From the point of view of assuming the maximum precision possible to obtain during luminescence analyses in the laboratory, a crucial element is to determine the dose rate of sediment from which material for further research is collected. Correct determination of the dose rate is just as important as the equivalent dose value, however, in the literature, problems related to the correct determination of the annual dose are marginal (Murray *et al.*, 2018).

The dose rate can be determined based on radioisotope content measurements. In the natural environment, the dose rate comes mainly from the radioactive nuclides of uranium (^{238}U), thorium (^{232}Th) and potassium (^{40}K), which are natural components of the Earth's crust and are the source of ionizing radiation. Currently, a number of different methods are used to determine the dose rate: mass spectrometry (ICP-MS), gamma ray spectrometry (both with scintillation detectors and HPGe), alpha counting (TSAC), beta particle count (TSBC).

As part of our project, we plan to use:
– mainly HPGe high resolution spectrometry. This method allows for the simultaneous determination of the content of both the uranium and thorium series of isotopes, as well as the content of in radioactive potassium (^{40}K).
– TSAC method which will allow the measurement of the uranium and thorium contribution in smaller samples.
– hybrid system μDose (Tudyka *et al.*, 2018) which combines the advantages of TSAC measurement techniques, TSBC with additional radioactive identification capabilities and ^{40}K The device allows for the measurement of small samples and verification of the results of an independent measuring method.
– portable scintillation spectrometer manufactured by Canberra for recording energy spectra of ionizing radiation in field conditions at sampling points.
– a new generation of dosemeters, based on the scintillators LaBr$_3$, CeBr$_3$ and SrI$_2$, read out with conventional photomultipliers, to be used in the field of environmental gamma-radiation monitoring (Kessler *et al.*, 2018).

These methods allow to determine the content of radioactive isotopes in the sediment with maximum precision. However, there are several factors that may lead to the misrepresentation of the activity results regarding the radioactivity measurements, which perhaps may be based on radioactive imbalance. Until now, in most studies conducted worldwide, in the case of luminescence dating, it was assumed that radioactive isotopes are in a state of radioactive equilibrium. In the case of aeolian land sediments, this is an acceptable assumption. However, in the case of aquatic environments, this equilibrium may be violated.

The final dose rates are calculated using the conversion factors of Guerin *et al.* (2011) and includes corrections for

the cosmic ray contribution is determined as described by Prescott and Hutton (1994) and the humidity of the sediments according to Aitken (1985).

Luminescence procedures and De (equivalent dose) calculation

The following procedures are designed to ascertain the quality of the luminescence signal for use during single-aliquot regenerative-dose (SAR) measurement procedures.

A preheat plateau test is usually performed to establish the most appropriate preheat temperature. Preheat temperatures are usually increased from 180°C to 300°C in 20°C intervals. The luminescence signal from an example sample presented (Fig. 5) has no systematic variation in De with preheat temperature. This provides positive evidence that thermal transfer from incompletely emptied traps is not an issue in this sample, and therefore a 260°C preheat can be used for all subsequent measurements.

In the SAR protocol, sensitivity changes which may occur from one measurement cycle to another are measured by the OSL response to a small test dose (Murray and Wintle, 2000). The corrected OSL ratio (regenerated OSL response/OSL response to the fixed test dose) should be independent of prior dose or thermal treatment. This is tested by repeating a particular regenerative dose after various larger values have been used, and comparing the ratio of the two regenerated sensitivity-corrected OSL responses (known as a recycling ratio); this ratio should ideally be close to unity (Murray and Wintle, 2000). The recycling ratios for all measured samples should be close to unity (from 0.90 to 1.10). Preheating the sample can also cause recuperation of the OSL signal (Aitken, 1998). To test this, a 0 Gy regenerative dose step is incorporated into the SAR protocol (Murray and Wintle, 2000). The luminescence signal should then be zero (this is known as the 'recuperated' luminescence signal). Any aliquots with grains for which this sensitivity-corrected recuperated signal is >5%
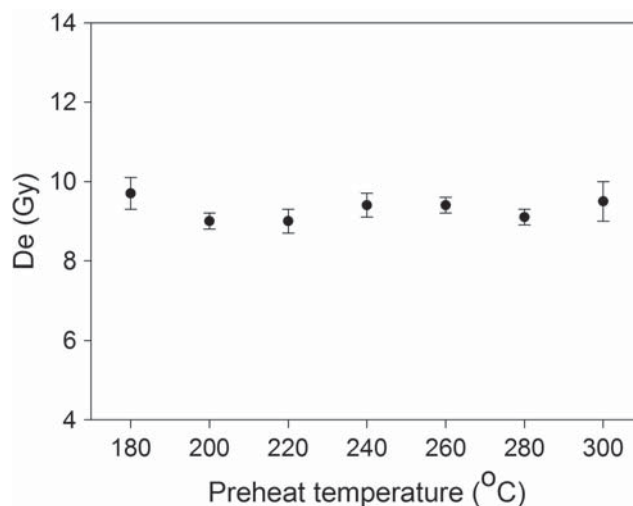

Fig. 5. Example of preheat plateau graph.

of the corresponding natural signal are rejected (Murray and Olley, 2002).

The dose recovery test is a basic requirement to determine the suitability of the SAR protocol. This is done by administering a known dose in laboratory conditions and retrieving it using the same measurement procedures as for dose estimates (Wallinga *et al.*, 2000). During the dose recovery test, first the natural signal is optically erased using a solar simulator (a lamp with a light spectrum from about 300 to 800 nm for 1–2 hours) and then a known dose is admitted to the sample. The ratios between the given and measured doses for the example sample are shown in Fig. 6 as a function of preheat temperature. The measured dose reproduces the known given dose at all preheat temperatures from 180°C to 300°C (average measure to given dose ratio of 1.02 ± 0.03). If the protocol works correctly, the ratio between the measured and the given dose should be between the values from 0.90 to 1.10.

For the samples, equivalent doses are determined using the SAR protocol (Murray and Wintle, 2000). The OSL SAR protocol contains the following steps:

1. Irradiation with the regenerative beta dose Di
2. Preheat at a temperature of 260°C for 10s
3. Blue light stimulation at a temperature of 125°C for 100s
4. Irradiation with the test dose Dt (10% of the natural dose, but not less than 1 Gy)
5. Cut-heat at a temperature of 220°C
6. Blue light stimulation at a temperature of 125°C for 100s.

For the SAR protocol, in order to determine De the dose response curves must be built using a single saturating exponential function. A standard procedure provides the ability to use three or four regeneration doses bracketing the De. In the measurements, the first second of the decay is used as a measure of the signal, while the background is estimated using the last 10 seconds of the OSL decay. Examples of growth and decay curves are presented on the Fig. 7.

Final D_e values are usually calculated using the Central Age Model (CAM) (Galbraith *et al.*, 1999). The overdispersion parameter (σ_{OD} refers to the spread in D_e values remaining after all measurement uncertainties have been taken into account) of these distributions is always calculated using the Galbraith's approach (Galbraith *et al.*, 2005), and usually varies from only a few percentage points to about 20%. The sources of overdispersion are both intrinsic (e.g. counting statistics as described by Adamiec *et al.*, 2012) and extrinsic (e.g. beta microdosimetry or incomplete bleaching; Duller, 2008). The effects of microdosimetry or bioturbation are likely to be reduced due to the size of the aliquots. Arnold and Roberts (2009) performed the most detailed analysis of stochastic modelling of multi-grain equivalent doses. They examined all the possible number of sources of variation contributing to the commonly observed scatter in equivalent dose distributions.

The De distribution can be presented in terms of relative probability density functions (Berger, 2010). Statistical analysis using CAM is not a problem if the typical uni-
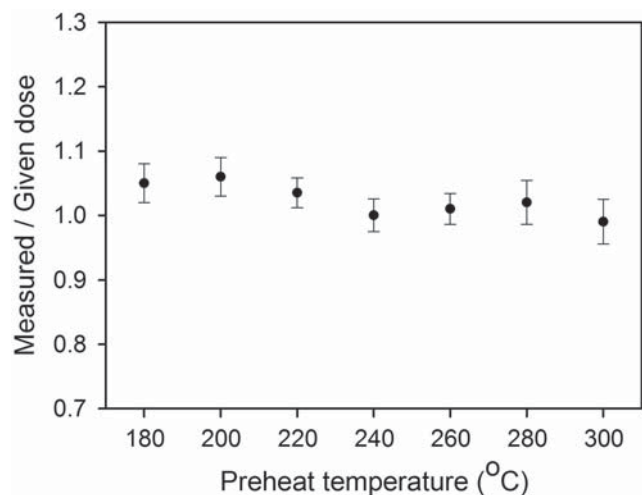

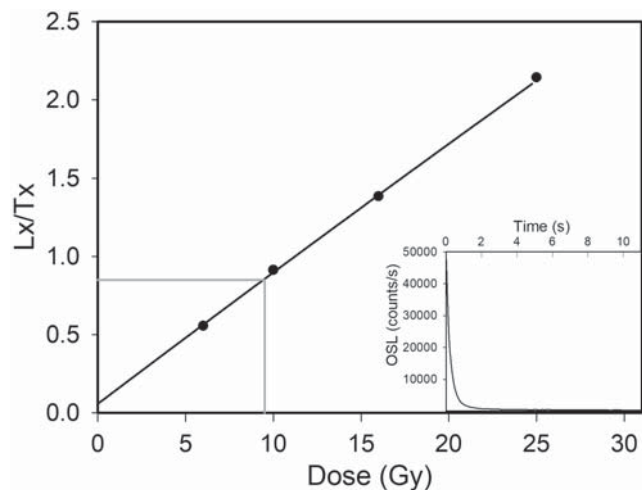**Fig. 6.** Example of dose recovery test.

Fig. 7. Example of growth and decay curves.

modal distribution is presented (example of this kind of distribution is presented in Fig. 8). This group of adequately bleached samples is characterised by lower overall overdispersion, typically about 10% or less (Arnold *et al.*, 2007).

Very often it is possible to witness distributions which have overdispersion parameters ranging between 10 and 20%. In those cases the typical unimodal shape of the distribution is not observed (see Fig. 9). This does not change the fact that CAM for all aliquots remains the most suitable in order to estimate the final value of the D_e. Usually, fluvial samples are representative of this group and in this case some asymmetry in the distribution (i.e. not statistically significant) may be observed (Arnold *et al.*, 2007).

Other types of distributions are in cases where the use of CAM for all aliquots is not so obvious. Usually, distributions are characterized by overdispersion parameters above 20% and the width of the distributions increases noticeably (see Fig. 10) displaying clear asymmetry with a statistically
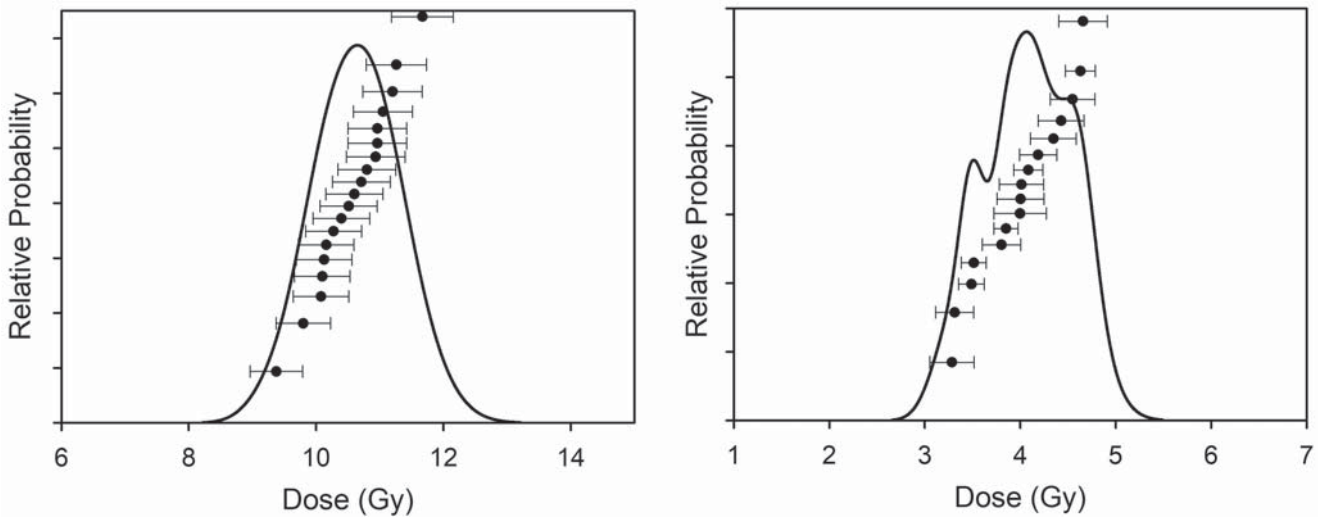
Fig. 8. Example of typical unimodal relative probability density function. Overdispersion for the left graph is 4% and for the right graph is 9%.
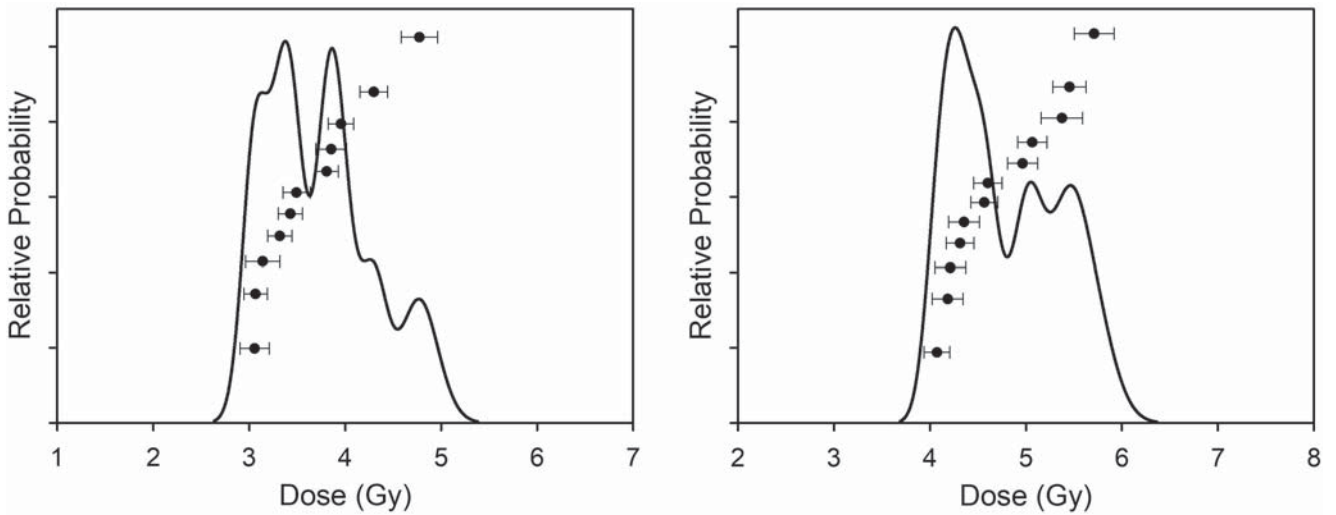

Fig. 9. Example of atypical unimodal distribution of relative probability density function. Overdispersion for the left graph is 13% and for the right graph is 12%.
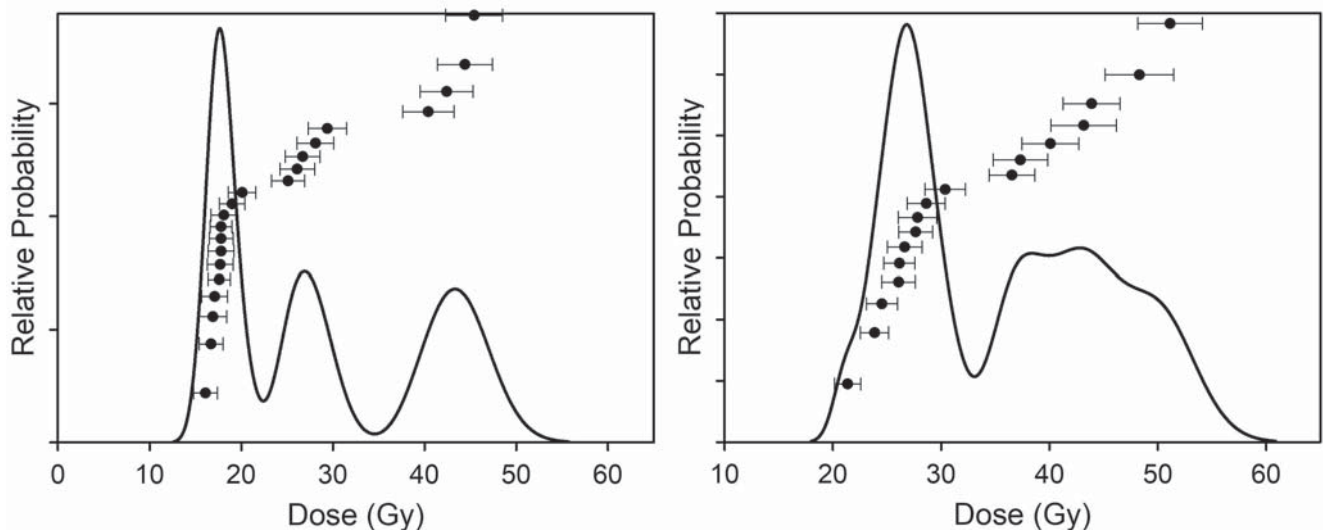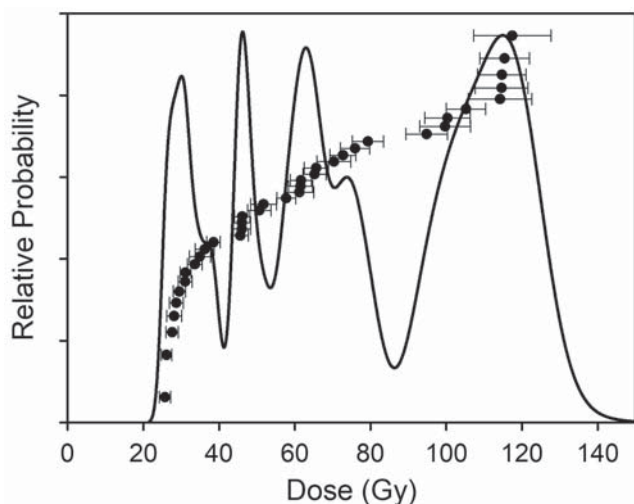

Fig. 10. Example of multimodal relative probability density function. Overdispersion for the left graph is 35% and for the right graph is 26%.

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Fig. 11. Example of extremely bad relative probability density function. Overdispersion for this sample is 50%.

significant positive skew. This means that one sample has a mixture of grains with different ages or alternatively, there are issues with insufficient bleaching (some of grains were exposed to sunlight for too short a time period). This type of single aliquot D_e distribution exhibits characteristics pertaining to heterogeneously bleached forms (Arnold *et al.*, 2007). In these cases, we define the final D_e value based on the first main mode (for which we can assume the highest probability of correct bleaching quartz grains during redeposition in nature).

The rarest group of samples that pass through luminescence laboratories are those that were assumed to be wrongly selected for future investigation. The material of these samples does not fulfil the basic premise of the OSL method. An example of this kind is presented in Fig. 11. This graph presents a sample taken from flood sediments from mountain river terraces. It is easy to see that there is an extremely wide distribution of density probability function and in this case it is impossible to obtain a reliable final OSL dating result. In this case only the age of the youngest quartz grains (preferably bleached) are able to determined. It is also worth mentioning that interpretation of subsequent modes on this kind of relative probability density function should be avoided, as their presence does not specify the age of any group of grains (it may well be a mixture of very young and older grains and it is impossible to resolve using standard luminescence methods (Arnold and Roberts, 2009)).

SUMMARY AND OUTLOOK

Luminescence is a powerful technique that complements other dating methods. Luminescence dating covers the time range from a few years or decades up to several hundred-thousand years and represents a suite of related

techniques, all of which provide an estimate of radiation history due to an accumulation of trapped charges in mineral lattices. OSL dating continues to undergo rapid development, although the quartz SAR protocol, in particular, provides reliable age estimates for sediment deposition in many contexts and is now widely applied. Recent research focuses on improved and innovative procedures of both equivalent dose (Arnold and Roberts, 2009) and dose rate determination (Tudyka *et al.*, 2018; Kesser *et al.*, 2018). For equivalent dose determination, it is necessary to better understand the sources of variation observed for individual palaeodose estimates determined for the same sample, especially to quantify the effect of microdosimetry. Calculating luminescence ages involves the measurement of many parameters, so the final results should be presented not only as the final result but also should contain other data from luminescence protocols such as relative probability density function graphs, it is essential to incorporate sufficient data to enable readers to judge the quality of the age.

Attention should also be paid to raising awareness of all users of luminescence methods with regards to the fact that today, luminescence dating it is not only a matter of final numbers in terms of ages and uncertainties. Given the complex nature of luminescence dating it is generally recommended that research should be undertaken in conjunction with luminescence laboratories involved with the measurements in order for them to provide expert input into the presentation of the data.

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