DETERMINING WHEN A SEDIMENT WAS LAST EXPOSED TO SUNLIGHT BY OPTICAL DATING

by

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Abstract

Optical dating has now been established as a method that can be used to determine when quartz or feldspar grains in sediment were last exposed to sunlight. In this review we outline the processes that occur in these minerals while in the environment, and the laboratory techniques that are used to obtain an age estimate. The questions "are optical ages right" and "what can go wrong" are addressed. Examples are provided of cases where optical ages are in agreement with independent ages, and examples are given for which unexpected ages were obtained, and possibilities for resolutions are discussed. Emphasis is placed on the necessity of sufficient sunlight exposure prior to burial, and the degree to which this depends on the environmental circumstances. The examples chosen are primarily from our work in western Canada and they are intended to show what a geoscientist can and cannot expect of optical dating.

INTRODUCTION

Optical dating is a method of determining the time elapsed since mineral sediment was last exposed to sufficient sunlight, the term sunlight being used here to encompass any form of daylight, whether it be direct, or through clouds or water. Such a sunlight exposure may occur between the time of erosion of sand or silt grains and the time of their subsequent deposition in a sedimentary deposit. Some examples of processes in which such a sunlight exposure can occur are loess deposition following deglaciation, the formation of a dune during a dry climatic event, and the formation of a beach dune at a high sea stand. Failure to recognize that the last sunlight exposure is the event being dated is a common cause of difficulty that a geoscientist may have in interpreting optical ages since this event may not coincide with the event of geological interest.

Optical dating is based on specific properties of quartz and feldspars that depend on the existence of defects within mineral crystals and the interaction of electrons with these defects. When a mineral is subjected to ionizing radiation, some electrons are ejected from their usual states and some of these subsequently become lodged at specific defects. Such defects are referred to as traps. This is a metastable situation, but it is possible for the lifetime of a trapped electron at ambient temperatures to exceed 10⁸ years. The larger the dose of radiation, the larger the number of electrons trapped at these defects. Traps fill until all the traps are full or until some other process leads to a state of equilibrium, or until a sunlight exposure or heating empties them.

The task of the physicist is to deduce from the trapped electrons what the radiation dose was that put them there, and from this, in combination with the radiation dose rate, to calculate an age.

The age of the sample is calculated from:

Optical dating is one of three techniques based on this principle; the other two are thermoluminescence dating and electron-spin-resonance dating. They do not overlap as much as one might imagine because each method generally makes use of different defects.

Radiation dose is strictly defined as the amount of radiation energy that is absorbed per unit mass of the mineral; the S.I. unit is the gray, with 1 Gy = $1 \text{ J} \cdot \text{kg}^{-1}$. However, the actual radiation dose is not what is determined in any of these dating techniques. This is because the radiation dose arises from cosmic rays, and from a, b and g radiation from the decay of potassium, thorium and uranium, and their daughter products within the sediment and its surroundings, and because the number of trapped electrons depends not only on the dose but on the type of radiation. a-particles are only about one-tenth as efficient on a perunit-of-absorbed-energy basis as the other forms of radiation. To deal with this the term 'equivalent dose' is introduced; it means the radiation dose of b or g radiation that would produce the same effect as the actual radiation dose (i.e. that received in the natural environment). It is commonly abbreviated D_e or D_{eq} .

One can distinguish conceptually between the following quantities: a sequence of dose intercepts on graphs made from data obtained in the laboratory, an estimate of the equivalent dose deduced from them which is used to calculate the age, and the actual equivalent dose. There is no agreed terminology that distinguishes the three, and the term equivalent dose is often used to refer to any of them.

Quartz and feldspars are used for optical dating because of their ubiquity, ease of separation from sediment, and because they yield ample luminescence for measurement. Other minerals can be used in principle but there has been little research on them.

Any sediment that contains quartz or feldspar and has been exposed to sufficient sunlight prior to burial is suitable for optical dating. What is "sufficient" sunlight exposure is discussed in some detail later. Depositional environments most likely to satisfy the requirement are aeolian, such as sand dunes and loess, and some low-energy water

environments, such as peat bogs. In order of decreasing likelihood we would add lacustrine, fluvial, and glacial environments. Such a listing may be misleading for there is loess that is not suitable, and there are some glacial deposits that are; the key is the amount of light exposure to the grains. The established age range of optical dating is from about 1,000 years to 100,000 years. What can be achieved depends very much on the minerals present in the sample and their behaviour. Ages of 100 years or less can readily be obtained, and may be credible if proper techniques are used. For some samples, both quartz and feldspar have sufficient sensitivity to produce ages of the order of one year, but there are presently no reliable techniques for achieving such an age. "Ages" of the order of 1,000,000 years have also been obtained, but again reliable techniques for obtaining accurate ages of this order have not been developed.

Potential problems that can occur with optical dating are discussed in detail later. Here we note that, as with other dating methods, one should put little faith in an isolated optical age. Instead one should have a suite of samples which are part of a detailed research program, and that one should consider the optical ages in the context of the overall picture. This is because one's intuition about the adequacy of sunlight exposure at the time of deposition is often wrong, and the laboratory techniques in use are not always appropriate.

Earlier reviews of optical dating have been published by Aitken (1992, 1994) and Berger (1995). Sediments can also be dated using thermoluminescence (Berger, 1988, 1995). In most cases optical dating is simpler and superior. The one notable exception is that quartz that is too old for optical dating can sometimes be dated using thermoluminescence dating. Optical dating is simpler because heating in a controlled atmosphere is replaced by a light exposure in air. It is superior because less sunlight exposure before burial is required, the traps that are most easily emptied by sunlight are sampled preferentially (leading to the ability to obtain much younger ages), and single-aliquot methods that make use of much less sample are feasible. In addition, advantage can be taken of the ability to select the energies of the excitation photons to select particular minerals or defects..

Some of the terminology used varies from author to author so we include an appendix in which we have described the meanings of some of the more common terms found in the literature.

MEASUREMENTS

Sunlight ejects electrons from traps, and environmental radiation after burial slowly puts electrons in again. Exposure to light in the laboratory ejects the electrons which, having an excess of energy, may lose it by emitting light. The last process will occur at a particular

type of defect that is referred to as a luminescence or recombination centre. The intensity of this light emission (luminescence) is thus a measure of the number of electrons trapped since the last sunlight exposure. The longer the mineral grains remain shielded from sunlight, the more light they will emit when measured.

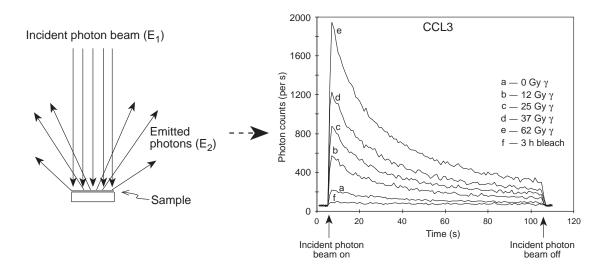


Figure 1. The basic measurement in optical dating. A beam of photons of energy E_1 is shone on grains separated from the sample; these grains respond by emitting photons of energy E_2 . The longer the sample has been buried, the greater the number of emitted photons. On the right is shown the decrease in luminescence intensity with time, as the traps are emptied by the laboratory excitation. The different curves are for aliquots given the radiation doses shown, and are for 4–11 mm grains from a 2.4-2.7 ka loess. Each curve is an average of several. More data for this sample are shown in Figure 2 (cf. CCL3 in Table 2).

The measurement procedure is simply to shine light on mineral grains separated from the sample, and to measure the light that is emitted in response, as shown in Figure 1. In each case the light should be thought of as a stream of photons, their energies being E_1 for the incident beam, and E_2 for the emitted photons. The photons of the incident beam need not necessarily all have identical energies, but for practical reasons only a small range is used. The emitted photons may have a relatively large range of energies spread over one or more emission bands that depend on the nature of the minerals present. Two of the most common combinations of E_1 and E_2 that are used in practice are shown in Table 1. The emitted photons are counted using a photomultiplier tube, the output of which is an electrical pulse for each photon detected, and some electronics that counts these pulses. Figure 1 shows typical curves of count rate (luminscence intensity) \underline{vs} time, the decrease in luminescence occurring as the traps are emptied.

Table 1. Commonly used incident and measured photon energies; wavelengths and colours
are given in parentheses.

Mineral	Incident photon energy	Measured photon energy	
	$\mathbf{E_1}$	$\mathbf{E_2}$	
Quartz	2.2 –2.4 eV ^a (510-560 nm,	3.35 eV (370 nm, ultraviolet)	
	green)		
K-feldspar	1.4 eV (880 nm, infrared)	3.1 eV (400 nm, violet)	
a 1 eV (electron-volt) = 1.6	6 ¥ 10 ⁻¹⁹ joules.		

Because a large number of the incident photons are simply scattered by the grains of the sample and other processes lead to emission of photons at different energies (ordinary photoluminescence and Raman scattering in the grains), one needs to choose to measure only photons of significantly higher energy than that of the incident ones, as is shown in the examples given in Table 1. Optical filters are used in the measuring system to accomplish this, thus a sample that has just been well exposed to sunlight will not scatter or emit any photons in the energy range that is detected. In the case of quartz the little information available indicates there is little choice of energy and filter combinations, there being only one emission band, and incident photons of different energies all emptying the same traps (Huntley et al., 1996). Feldspars, on the other hand, offer more options because there are several emission bands and traps. However, the potential for using these options has not yet been fully explored. There is also the option of employing inclusions, probably feldspar, within quartz grains (Huntley et al., 1993). Indeed, any given sample may present several options for optical dating.

The actual apparatus used for the measurements can be quite simple, such as that used in the initial work (Huntley et al., 1985). The measurements lend themselves readily to automation and there are at least three commercial instruments available; one is described by Bøtter-Jensen and Duller (1992) and Markey et al. (1997). Such instruments can be very useful for routine work, but they sometimes lack the flexibility needed for more fundamental research and their "black-box" nature allows novice users a broad scope for making errors. For a sample that yields little luminescence it is worthwhile measuring as many of the emitted photons as possible, and at present the most efficient apparatus in this respect is the design of Baril (1997). Instruments for field use are possible (Poolton et al., 1994) and may be valuable for comparative work, but they should not be expected to provide ages.

DETERMINATION OF THE EQUIVALENT DOSE

The central problem here is to determine the laboratory radiation dose that produces the same population of trapped electrons as did the environmental radiation dose. The methods used can be divided into two groups: additive dose and regeneration.

The additive dose method is illustrated in Figure 2. Here, aliquots consisting of grains separated from the sample are given different laboratory radiation doses, heated, stored, and then their luminescence measured; a line is then fitted to the luminescence intensity vs. laboratory dose data. The line is commonly extrapolated to zero intensity, and the dose intercept taken to be the equivalent dose; however, for reasons described below this procedure is not correct, though for relatively old samples the error introduced may not be significant. The heating is required because the laboratory irradiation produces some thermally-unstable trapped electrons that are not present after an environmental radiation dose, which occurred over a much longer time; the heating gets rid of them. There is no universally agreed prescription for this treatment, but 5 minutes at 220 °C, 16 hours at 120 °C, or 7 days at 140 °C are typical; often what is chosen is based on convenience as much as science. It is clear that the temperature and duration used may make a difference and in at least one instance there is a strong difference of opinion on the matter (Roberts et al., 1994; Stokes and Gaylord, 1994).

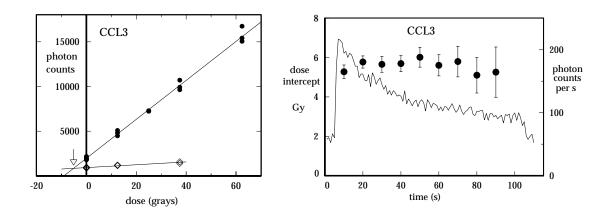


Figure 2. (a) Illustration of the additive dose method with the thermal transfer correction. The intensity of the luminscence resulting from optical excitation is plotted as a function of laboratory radiation dose for two sets of aliquots. The solid circles are "additive dose" data from aliquots irradiated and heated. The open diamonds are from aliquots irradiated, given a light exposure and heated, and are used to determine the amount of thermal transfer. The equivalent dose is taken to be the radiation dose where the two lines intersect as marked by the arrow, with a small correction for decay during the normalization.

(b) Equivalent dose, determined as in (a), as a function of illumination time. Also shown is the intensity <u>vs</u> time curve for an aliquot that has only been preheated in order to show the time scale.

Unfortunately, this heating has an unwanted effect. This is most readily observed by taking grains that have just been exposed to sunlight, heating them, and then measuring them. Whereas without the heating no luminescence is measured, with it a substantial amount may be measured. The reason for this is that at high temperatures, say at 160 °C, electrons are emptied from traps that are not emptied at the environmental temperature of \sim 20 °C, even over a long time period. Some of these electrons are subsequently retrapped in traps being sampled during the measurement process. This is referred to as 'thermal transfer' and it can be corrected for as shown in Figure 2. The number of electrons in the source traps for the thermal transfer depends on the radiation dose, thus it is necessary to make the thermal transfer measurement as a function of dose. Hence, a number of the aliquots are exposed to laboratory light to empty the traps of interest, and then they are heated along with the ones that received no laboratory light. The luminescence measured from the former defines the lower data set shown in the figure, and the dose intercept of the two lines is taken to be the equivalent dose. If this correction is not made then a substantially incorrect age can be obtained, particularly for young samples. In the example (Fig. 2), we show data obtained for loess directly overlying Bridge River tephra, which is known to have been deposited 2.4 - 2.7 ka ago (Clague et al. 1995; Leonard, 1995). The optical age obtained using the thermal transfer correction, 2.5 ± 0.3 ka, is in excellent agreement. Had the dose axis intercept been used an erroneous age of 4 ka would have been obtained instead. This method of making the thermal transfer correction also allows for the dark count rate from the photomultiplier tube and scattered excitation photons that are counted. In the example of Figure 2 all three effects were significant.

We have found a number of quartz samples for which the luminescence from thermal transfer so overwhelms what we are trying to measure that it has defeated our attempts at optical dating. For some feldspar samples that are older than ~1 ka we have evidence that the above method of correcting for thermal transfer is not quite right, but the reason for this is not yet understood. We have also found that the actual laboratory light exposure used to make the thermal transfer correction can be important (Huntley and Clague, 1996).

An additive dose method is quite suitable when the luminescence vs. dose data is linear, or nearly so, and an accurate extrapolation can be made. When this is not the case then a regeneration method should be seriously considered. Figure 3 shows an example. Here one group of aliquots is given a variety of radiation doses, while a second group is exposed to laboratory light to empty the traps, and then given similar radiation doses. The second group, after the light exposure, resemble hypothetical aliquots prepared immediately after burial. Both sets of aliquots are then heated and measured. The data from the second

group of aliquots are referred to by the term regeneration, and may be thought of as defining the missing portion of the additive dose curve that would otherwise have to be extrapolated. The equivalent dose is taken to be the shift along the dose axis that brings the two data sets into alignment, with a correction for the incomplete effect of the laboratory light exposure. There is some concern that the laboratory light exposure may have some unwanted effects, such as a change in the dose response of the sample, and in practice some test should be made to determine whether or not the two data sets do actually define a single curve. Here the duration and spectrum of the laboratory light used may well be important. Sunlight is often used because that was what was present at the time of deposition and one wishes to reproduce the same condition. However, this argument does not withstand scrutiny as the original sample condition may not be obtained, and may not even be attainable. For K-feldspars we have been using an infrared exposure which typically reduces the measured 1.4 eV-excited luminescence intensity to about 3% of the original with no apparent detrimental effect.

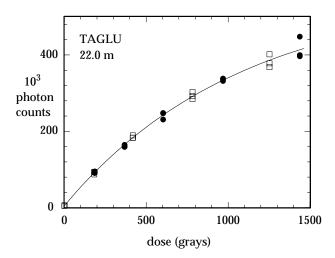


Figure 3. Illustration of the regeneration method. The two data sets are an additive-dose set (solid circles) and a regeneration set (open squares). The latter are for aliquots that have been given a light exposure, a laboratory radiation dose and then heated. The additive dose data have been shifted horizontally to give the best fit. The equivalent dose is taken as the shift, with corrections for incomplete trap emptying by the light exposure and decay resulting from the normalization measurement. The data are for a sediment from the TAGLU core, Mackenzie River delta (Huntley, 1997).

With either the additive-dose or regeneration method the equivalent dose should be determined as a function of illumination time. It should be constant, as shown in the example in Figure 2b. If it is not constant there is cause for concern, but this effect is not yet understood though it may arise from inadequate sunlight exposure before burial.

With both the additive dose and regeneration methods scatter results from the fact that all the aliquots prepared from a sample are not identical. This is due primarily to the grains being a highly inhomogeneous collection, with most of the measured luminescence arising from a small fraction of them. A method that is in common use for alleviating this problem is to initially make a brief measurement of each aliquot and to subsequently use this luminescence to normalize the data. If this measurement empties a significant portion of the traps, say 5%, then it must be corrected for when the equivalent dose is calculated.

The uncertainty in the equivalent dose is obtained using proper statistical techniques in fitting the data; it is usually the dominant contribution to the uncertainty in the final age.

DOSIMETRY

The radiation dose arises from the radioactive decay of K, U, Th and Rb and their decay products, both from within the sediment grains, and from the surroundings, up to about 50 cm away. There is also a contribution from cosmic rays. All of these have to be evaluated. The various contributions to the dose rate (strictly the equivalent dose rate) are shown in Figure 4 for a typical K-feldspar grain from a sample collected from a sand dune.

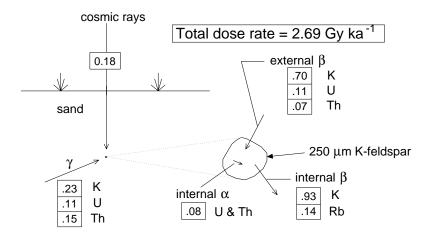


Figure 4. Illustration of the various contributions to the dose rate. The example is for a typical 250 mm diameter K-feldspar grain at 1 m depth in a sand dune consisting of homogeneous material containing 1% K, 1 mg.g⁻¹ U, 3 mg.g⁻¹ Th and 5% water. The K-feldspar grain has 13% K, 250 mg.g⁻¹ Rb, 0.2 mg.g⁻¹ U and 0.4 mg.g⁻¹ Th.

Dose rates are typically in the range 2-3 Gy·ka⁻¹, but can be up to five times smaller or larger. Because the dose rate is the sum of several contributions, what might normally be thought to be a large uncertainty in one of them, say 10%, will lead to a much smaller uncertainty in the final age. There is little merit in striving for high accuracy in any

one term. There is one exception to this: it is the evaluation of the b dose rate from K and Rb within K-feldspar grains. The uncertainty of the K content of the K-feldspar grains that contribute to the luminescence being measured may well be 20%, and this can lead to an uncertainty in the age in the range 2–10%.

Water in the sample matrix reduces the dose rate. If the water content is known its effect is readily calculated. What can be difficult is estimating the history of the water content over the existence of the deposit; an estimate has to be made by the geoscientist. Sometimes the best that can be done is to use two extreme possibilities and use them for the 95% confidence range. In cases where a sediment has been water-saturated for a portion of the time and wet to dry for the rest, the uncertainty in the final age may be dominated by the uncertainty in the proper average water content to use, and may be as high as 10%.

Evaluation of the dose rate can be made using laboratory analyses for K, U, Th and Rb contents, thick-source alpha counting, beta counting, or high-resolution alpha or gamma spectrometry; *in-situ* gamma spectrometry and thermoluminescence dosimetry may also be used to determine the gamma dose rate. It is desirable to make use of more than one set of measurements if high accuracy is desired or if the sediment is inhomogeneous within 50 cm of the sample; in this way difficulties associated with inhomogeneities or radioactive disequilibrium can be identified and allowed for. Finally we should point out the necessity for vigilance; 4 of the 5 laboratories which we have employed to do analyses have on occasion provided us with significantly incorrect values.

SAMPLE COLLECTION

There are three things that must be kept in mind during sample collection:

- (a) There must be no light exposure to the grains that are to be measured.
- (b) If the sediment is not homogeneous within 50 cm of the sample in all directions, then samples of differing material will be required and the geometry recorded; *in-situ* dosimetry is an alternative and often preferable.
- (c) Estimates of present and past water contents will be needed.

Sample collection is best done by someone from the optical dating laboratory. If this is not feasible, close collaboration is essential. If accuracy is a major concern then *insitu* gamma spectrometry should be performed and laboratory personnel may be able to do this.

About 1 kg of material is usually more than adequate, and should be collected if it is available. If the sediment is loose then a fresh face should be exposed and the sample put into a 1 litre lined can and sealed; this is best done at night, although it can be done quickly under a black cloth in the daytime. For material that can be carved into a block, such a

block can be collected in daylight, wrapped in foil and sealed. The outer material is subsequently removed from the block in the laboratory and discarded. Samples can also be collected by pushing a tube into the sediment.

There are occasions when 1 kg is not adequate and there are cases where it may be possible to date as little as 1 g. The details are numerous and will not be discussed here. The potential user should provide the laboratory with information about the composition of the material, such as grain size distribution, organic content and carbonate content, and find out from the laboratory what it is possible to do.

In general sediment that has not been collected with optical dating in mind will not be suitable. There are exceptions, but they are rare and reduced accuracy should be expected.

ARE OPTICAL AGES RIGHT?

This is not an easy question to answer. The basic physics is understood in outline and a number of tests have given ages in agreement with independent ages. On the other hand, the physics is not understood in detail and even the identities of most of the relevant defects are not known. Moreover, there are effects observed that have yet to be explained and incorporated into the methodology. This is further complicated by the fact that the mineral grains being measured are usually a highly inhomogeneous collection, even within a single mineral type from the same source area.

The first recourse is to perform independent tests of the validity of optical ages. It is highly desirable in any suite of samples to have some checks, and this should include a modern, i.e., a zero-age analogue (admittedly one can never be sure that one has sampled a true modern analogue); if the age obtained for it is not zero then there is something amiss. Other checks may be provided by radiocarbon or other dating methods, or by volcanic ash or other known-age markers. Paleoclimatic information can be used when it can be related to an oxygen-isotope interval of known age. Table 2 shows some selected successful comparisons of optical ages determined at the SFU laboratory with independent ages. Other comparisons can be found in Smith et al. (1990), Stokes and Gaylord (1993), and Berger (1995).

Table 2. Selected successful comparisons of optical ages with independent ages

Excitation and	Sample name	Material	•	Independe nt age	optical	Ref
mineral				(ka) [†]		•
1.4 eV fg	HCP1 LCP5 MPS1 CCL3,4	Peat* Peat* Organic-ric floodplain Loess		$ \begin{array}{r} 18 - 19 \\ 25 - 32 \\ 116 - 132 \end{array} $ $ \begin{array}{r} 2.4 - 2.7 \end{array} $	$16.4 \pm 1.$ 5 $31.2 \pm 2.$ 6 119 ± 9	1 1 1
1.4 eV Kf	SAW94-62 CBTS2	Loess Tsunami-la	nid sand	12.8 – 13.6 1.03 – 1.10	2.5 ± 0.3 $14.3 \pm 0.$ 7 $1.28 \pm 0.$ 10	3 4
1.4 eV qi	SESA-71 SESA-55	Dune sand Dune sand		116 - 132 400	116 ± 16 360 ± 33	5 5
2.4 eV qz	SESA-61 SESA-71	Dune sand Dune sand		116 – 132 116 – 132		6 6
fg = silt grains, 4 – 11 μm, polymineral. Kf = K-feldspar sand-sized grains. qi = feldspar(?) inclusions within sand-sized quartz grains. qz = quartz sand-sized grains. * = the inorganic silt fraction was dated. † = calendar ages before AD 1995 1 = Lian et al (1995). 2 = Lian (1997) 3 = Vreeken et al. (in prep.) 4 = Huntley and Clague (1996). 5 = Huntley et al. (1993) 6 = Huntley et al. (1996)						

Samples HCP1 and LCP5 are compressed peats deposited during the Port Moody interstade (d¹8O stage 2) and the Olympia non-glacial interval (d¹8O stage 3) respectively; sample MPS1 is an organic-rich floodplain silt deposited during d¹8O stage 5e; all three are from southwestern British Columbia. Samples CCL3 and 4 were taken immediately above and below a layer of Bridge River tephra in a post-glacial loess deposit in south-central British Columbia. Sample SAW94-62 is a post-glacial loess, containing Glacier Peak tephra, from the Cypress Hills, Alberta. Sample CBTS2 is a tsunami-laid sand from Cultus Bay, near Seattle, Washington State, U.S.A. The SESA samples are from beach dunes in south-east South Australia which formed during high sea stands.

Table 2 shows selected results, and should not be used as a general guide to the validity of any particular optical age. The Table does show, however, that valid optical ages can be obtained with appropriate techniques, and that an accuracy of 10% or better is attainable. It is quite possible to obtain a precision of 5% or better, but it is unlikely that the accuracy will be this good because of unknown changes in the radiation dose rate, mineral

behaviour that is not allowed for in the equivalent dose determination, and inadequate sunlight exposure prior to burial.

A second recourse is to obtain optical dates using different minerals or different excitation energies to sample different defects. The little work that has been done usually, but not always, shows agreement; examples can be found in Huntley et al., 1993 and 1996, Questiaux, 1991, Rees-Jones and Tite, 1997, and Wolfe et al., 1995. One example is given in Table 2.

WHAT CAN GO WRONG

Things that can go wrong can be divided into two areas, those that are in the domain of the field scientist, and those that are in the domain of the dating laboratory.

Probably the most important thing that can go wrong is that the sample was not adequately exposed to sunlight prior to the last burial. If the sample was not exposed at all then an age corresponding to an earlier exposure event will be obtained. If the sample was partially exposed then an intermediate age will be obtained. It is essential that the geoscientist has a clear picture of mode(s) of transport and deposition. Unfortunately knowledge on this point is often limited and intuition is often wrong. If there is error in this matter it is usually that there was much less sunlight exposure than envisaged. For this reason it is essential that the geoscientist should, if at all possible, collect a modern analogue, that is the same type of material (i.e., of similar mineral composition and derived from the same source area) very recently deposited under analogous conditions. The geoscientist is also responsible for providing information about the past water content, possible leaching or other changes in composition of the sediment with time, and the likelihood for disequilibrium in the uranium decay series, since these are required for the calculation of the dose rate. For example disequilibrium will occur if any of the isotopes in the ²³⁸U decay chain move out of, or are carried into, the sediment; some examples are given by Prescott and Hutton (1995).

There are many possibilities for error in the dating laboratory. The proper use of laboratory controls and alertness to potential problems are essential since optical dating cannot yet be considered a routine procedure. Different laboratories use different techniques for the evaluation of the equivalent dose; in some cases the differences are unimportant, but in others the technique used may be invalid. Even the most accomplished laboratories may produce incorrect values because of mineral behaviour that is not recognized as being anomalous. It is difficult for a non-expert to evaluate this, however some tests and considerations that one can look for are:

- (a) For relatively young samples a thermal transfer correction must be made; if it is not done, any age calculated is likely to be too old. It is difficult to be quantitative about this; one example has already been given; another is that of a 300 year old sample for which an age of 4500 years would have been calculated if the thermal transfer correction had not been included (Huntley and Clague, 1996).
- (b) If a regeneration method is used a test that the two dose response curves are the same should be used. If this is not or inadequately done, any age should be used with caution.
- (c) If feldspars are measured, anomalous fading may lead to an age that is too young. Tests can be made for the presence of anomalous fading and, by inserting a delay of weeks or months between irradiation and measurement, its effect can be reduced. There is, however, no known method of correcting for it. If no test results are reported one should assume that the age may be too young unless correct ages are obtained from sediments of similar material and age for which the ages have been well established.
- (d) If feldspars are measured and the sample is relatively old, i.e. > 100,000 years, there is evidence that the ages obtained may be too young, perhaps because the electrons do not remain in the traps at the environmental temperature. Any "old" optical ages should be viewed with this possibility in mind. For such samples, independent chronological information for some of them is highly desired.
- (e) Dose-intercept vs. illumination time plots should be shown and how equivalent doses were obtained from them should be explained.
- (f) An understanding of the scatter in the laboratory data is desirable; in some cases this can reveal inadequate sunlight exposure before burial.

HOW MUCH SUNLIGHT EXPOSURE IS SUFFICIENT?

The answer to this question depends on the situation, thus the question is best addressed by discussing examples. Figure 5 shows typical effects of sunlight exposure on quartz and feldspar grains cleaned and separated from deposits of ages ~500 ka and 70 ka, respectively. If the 70 ka deposit was eroded, exposed to sunlight, redeposited and buried, with *all* the feldspar grains receiving 500 seconds of the same sunlight as was used to obtain the data of Figure 5, and if a measurement were made of this material immediately after burial, the luminesence measured would be about 1% of that that would be measured had the sunlight exposure not occurred. If the dose response is linear, it would mean that an optical dating determination would yield an age of 700 years, not zero as one would wish. If sediment were to be buried for 1000 years, an optical age of 1700 years would be obtained, significantly in error. If the burial period were 50,000 years, the optical age

would be 50,700 years, which might be considered not to be significantly wrong, especially when analytical errors are taken into account.

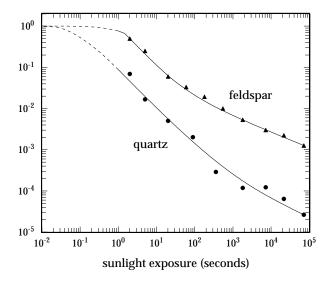


Figure 5. Illustration of the reduction of the 2.4 eV-excited luminescence caused by a prior sunlight exposure. Data are shown for quartz separated from a ~ 500 ka dune sand and K-feldspar separated from a ~ 70 ka alluvial sand (based on Godfrey-Smith et al., 1988). Note the logarithmic axes.

Alternatively, if the 70 ka deposit was eroded and redeposited with 1% of the grains receiving no sunlight exposure at all and the other 99% receiving a long sunlight exposure, optical dating would again give an age 700 years too large.

Clearly then the details of the sunlight exposure are important and in particular whether or not a particular sunlight exposure before burial is sufficient depends on the age of the deposit dated and the age of the deposit from which the sediment was eroded. In some cases the history of sunlight exposure and radiation dose before the last deposition will be relevant.

There are additional considerations. The intensity and spectrum of sunlight or daylight are highly variable, the grains may have (or had) mineral or organic coatings on them, or the grains may have been exposed through turbid water. Thus a range of possibilities of effective sunlight exposure to the actual grains can be expected.

This leads to the question of whether or not it is possible in the laboratory to determine whether or not the sunlight exposure before burial was sufficient. There are currently two possibilities here. The first follows from the fact that within a grain there is a variety of traps; these range from those which are readily emptied by sunlight to those which are emptied with only a very long sunlight exposure. It is this range that gives rise to

the non-exponential behaviour shown in Figure 5. During the laboratory measurement, after the excitation is switched on the readily-emptied traps are emptied first, and the traps that are harder-to-empty are emptied more slowly. The measured luminescence reflects this change, and it can be seen if an equivalent dose vs. time plot is obtained. If the equivalent dose is constant then all is well, but if it is rising this could be due to sampling less readily-emptied traps that were not emptied before burial such as in the example above of the 70 ka feldspar exposed to 500 s of sunlight.

The second method is useful if the grains were not uniformly exposed to sunlight. That is, some grains were well exposed and others were not, as in the second case above. If this has occurred then it may give rise to a recognizable form of scatter in the data.

The data of Huntley and Berger (1995) illustrate both methods, but more experience has to be gained before these techniques can be considered reliable. An alternate method is to make measurements on a large number of individual grains. If the relative effect of a laboratory dose given shows too wide a variation one can conclude that the sunlight exposure was insufficient (Lamothe, 1996).

UNEXPECTED AGES

It is inevitable that a new dating method will produce some unexpected results. Here we describe some examples of optical ages that were very different from what were anticipated, two of which were collected as part of the Palliser Triangle project. The first example is a Holocene cliff-top dune above Onetree Creek in Alberta. The optical age obtained was $112 \pm 15\,$ ka, indicating that the sediment was clearly not exposed to sufficient sunlight during the Holocene. The source of the sediment was pre-last glacial material eroded during incision of the creek. On the basis of modern analogues it seems likely that the sediment was transported upslope rapidly by strong winds. Since there are other examples of insufficient sunlight exposure for cliff-top dunes (Huntley et al. 1983, Lamothe and Auclair, 1997) optical dating should be used on such sediments with caution.

The second example is a loess from the Cypress Hills, Alberta. Until now this has been thought to predate the last glacial maximum (Vreeken, 1986), but our optical ages of 12.6 ± 1.4 ka for silt-sized grains and 14.0 ± 0.5 ka for sand-sized K-feldspar grains are leading to a reinterpretation of the geology (Vreeken et al., in prep.).

The third example is post-glacial loess (mostly silt-sized material) from the Fraser Plateau in south central British Columbia; ages ranging from 22 to 99 ka were obtained. Detailed investigation showed that the likely reason for the incorrect ages to be a combination of the presence of carbonate-cemented grain clusters that were apparently transported intact from the source material to the loess deposit, and a short (< 100 m)

transport distance. In the laboratory grains that had remained shielded from sunlight within the clusters were liberated during routine sample preparation procedures (Lian, 1997).

RECENT DEVELOPMENTS AND PROSPECTS FOR THE FUTURE

One of the attractive features of optical dating is that measurable luminescence from a very small amount of sample, in some cases as little as a single grain of sand, can be obtained. This opens up some new opportunities. The techniques described above involve preparing 30-60 aliquots, each holding 1000 or more of the separated grains. The use of a single aliquot for all the measurements has been shown to be feasible for both feldspars (Duller, 1995; Mejdahl and Bøtter-Jensen, 1994) and quartz (Murray et al., 1997). Any results from such measurements should at present be regarded with caution since corrections are required because of the repeated irradiations and heating, and it is not always clear that these are done appropriately. In addition, attention to the thermal transfer correction still needs to be made. Data from a single aliquot show very little scatter leading to a high precision for the equivalent dose. However, this can be misleading since scatter that results from natural variability between grains is hidden unless several aliquots are measured. Measurements on several aliquots may also permit detection of inadequate sunlight exposure before burial (Duller, 1995).

Extension of single aliquot techniques to measurements on single grains may allow the relaxation of the requirement that all the grains were well exposed to sunlight at deposition. As long as some of the grains were well exposed, and providing that one can determine which ones they were, then all one has to do is to determine the average equivalent dose for these. Progress in this direction has been made by Lamothe et al. (1994) for feldspars, and Murray and Roberts (1997) for quartz. Measurements on single grains may also be used to determine whether or not a sample received sufficient sunlight exposure for dating (Lamothe and Auclair, 1997).

The time elasped since the surface of a rock was last exposed to sunlight is also something that, in principle, one can determine with optical dating. This should be a fertile field for further research if suitable material is chosen. The only work reported so far is that of Richards (1994) on quartzite pebbles from an archaeological site in Russia.

Anomalous fading is generally observed in feldspars (Spooner, 1992). There are two questions for which answers are needed. The first is that, since anomalous fading is observed in most museum feldspar specimens, why can correct ages be obtained for deposits from which feldspars are measured? The second question is how can one correct for anomalous fading in the laboratory?

Finally, a detailed understanding of the physics of the defects, trapping, and excitation mechanisms in quartz and feldspars is needed.

CONCLUDING REMARKS

Optical dating may now be considered to be a valuable technique that can give correct ages under the right circumstances. The researcher must be aware that the event dated is the last exposure to sufficient sunlight. The researcher should also be aware that the uncertainty quoted with an age may only represent analytical uncertainties since uncertainties in the appropriateness of the methodology and the adequacy of sunlight exposure prior to burial cannot be evaluated.

One should be wary of an isolated optical age with no supporting independent information. It is highly preferable that a suite of samples, including a modern analogue (zero age) of the source material, be measured, and that there are checks that the right ages are being obtained. Intuition or experience is often not a good guide for deciding whether or not there was sufficient sunlight exposure before burial for optical dating to give the correct age.

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APPENDIX

The literature is rife with synonyms, acronyms and specialized phrases. Here is a list of the more common terms and their meanings.

OSL: optically stimulated luminescence, which is the luminescence emitted when light is shone on a sample, the emitted photons having more energy than the incident photons.

IRSL or **IR-OSL:** the same when the incident light is infrared (IR), specifically 1.4 eV photons.

GLSL or **G-OSL**: the same when the incident light is green, the photons having energies of about 2.2 - 2.4 eV.

Anomalous fading: The phenomenon that the measured luminescence decreases with time after irradiation and heating even though simple theoretical calculations predict that it should not.

Bleach: A light exposure, either from sunlight or a laboratory light source.

Equivalent dose: The laboratory b or g radiation dose that leads to the same amount of measured luminescence as does the environmental dose.

Palaeodose: Often incorrectly used instead of equivalent dose; palaeodose equivalent would be appropriate.

Preheat: The heating of a set of aliquots before the final measurement.

Recuperation: Increase of measured optically stimulated luminescence with time or heating after bleaching.

Saturation: The phenomenon whereby at high enough radiation doses further irradiation does not lead to an increase in luminescence resulting from optical excitation.

Shine down curve: Luminescence intensity vs time, as in figure 1, for example.

Stimulation: An alternative term to excitation.

Thermal transfer: Recuperation that results from laboratory heating and that would not occur in nature during burial.

Zeroing: The phenomenon whereby exposure to sunlight reduces the subsequently-measured luminescence resulting from optical excitation. The latter is zero if the sample has been truly "zeroed".

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