Manual coring equipment for the collection of stratified samples from dry sand dunes

Olaf Bubenzer

University of Cologne, Department of Geography,
Albertus-Magnus-Platz, D-50923 Köln, Germany,
e-mail: olaf.bubenzer@uni-koeln.de,
Fax.: 0049-(0)-221-470-5124, Tel.: 0049-(0)-221-470-3966

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Introduction

The analysis of the sandy layers of dunes, especially from draas (or megadunes), are suitable for dating palaeo-processes and the reconstruction of palaeo-windsystems (Besler, 1998). The sand layers in dunes are created by different wind directions and/or velocities and often show different grain sizes and colours. Thus, many dunes show a clear structure and their separate analysis is important for a large range of sedimentological, geomorphological, climatological and archaeological studies. Dry, non-fixed sand with no observable cohesion or pedogenetic developments behave in some way similar to liquids (Haynes and Johnson, 1984). Therefore, it is difficult to collect samples by drilling or digging because the sand flows immediately back into the hole. Until now it was only possible to reach depths of 1-2 m with manual drilling methods (Besler and Gut, 1997). Drilling to greater depths required heavy machines and large water supplies, which is not feasible for sampling in remote areas. Other problems arise from the fact that samples for luminescence dating have to be undisturbed and not be exposed to light. Furthermore, sample sizes in the range of about 500g are required. Wallinga and van der Staay (1999) described a suction corer, which is suitable for collecting samples from waterlogged sands. This paper describes a lightweight manual coring equipment for dry sands to reach deep sediment layers and to collect undisturbed samples. It was developed within the Collaborative Research Center ACACIA (Arid Climate, Adaptation and Cultural Innovation in Africa, SFB 389, University of Cologne) - section A „Holocene Environment and Cultural History in Northeastern Africa.

Equipment, drilling and sampling method

Figure 1 shows a schematic drawing of the drilling and sampling equipment, which can be used for sample collection to depths of up to 6 m. It weighs about 25 kg for the basic tools and the sampling equipment and 6 kg/m for the tubing. It is available from Eijkelkamp Co., P.O. Box 4, 6987 ZG Giesbeek, The Netherlands (www.eijkelkamp.com). The water consumption is about 1-2 l/m.

The coring equipment can be used in two configurations:

(i) Collection of mixed samples

The plastic liner tube (1) is placed on the sand surface. A light drilling-rod with a bayonet connection (2) and an Edelman sand auger (3) is inserted and used to core below the liner. Movable casing tube clamps (4) are used to hold the liner in place and to press it down whilst lifting the auger. For the first 1 to 2 m no water supply is necessary because it takes only a very short time to lift the auger. At greater depths, about 200 ml water is needed for one auger scoop of 10 to 20 cm. The full auger produces about 250 cm^3 sand. The coring works by cutting, therefore, the auger samples are mixed over the average coring depth of 10-20 cm.

(ii) Collection of undisturbed, stratified samples

After the liner has been driven to a given depth, it is possible to collect stratified samples with diameters of 6.5 cm and lengths of 40 cm. A removable stainless steel tube (6) with a core catcher (7) on the head of a rod with a conical

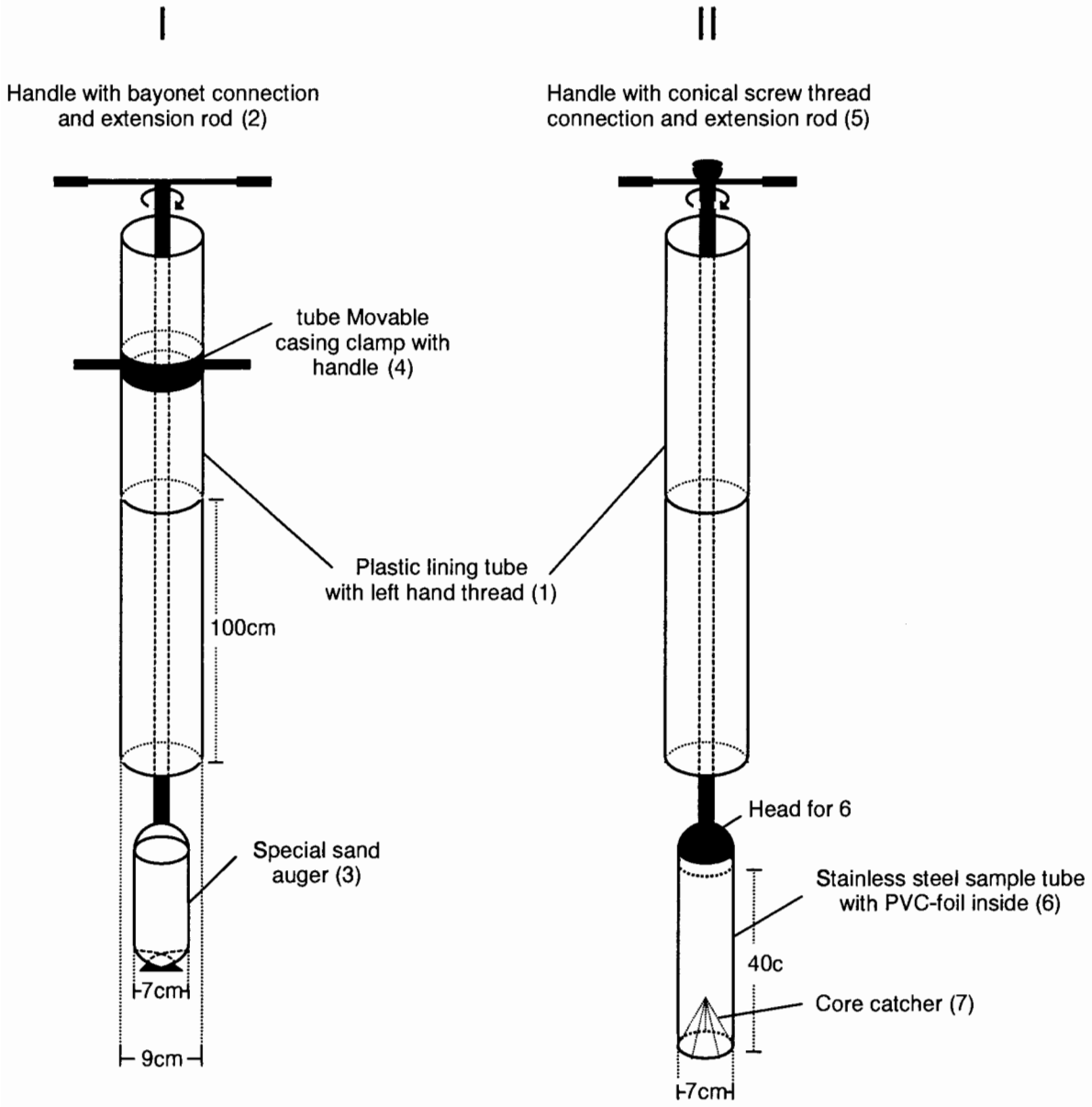

Fig. 1: *Schematic drawing of the coring equipment*

screw thread connection (5) is pushed with a percussion-free hammer into the sediment, which is watered prior to the insertion of the equipment. The watering in conjunction with core catcher ensure that the sediment is hauled as a complete sample core. The inner PVC-foil ensured that the sediment cores stays intact. The stainless steel tubes can be disconnected from the rod and taken to the laboratory where the sample is extracted in darkroom conditions. The maximum sediment volume in a sample tube is 1.400 cm^3. Two persons will require about 2 to 3 hours to reach a depth of 3 m and collect one sample.

Depending on grain size and sediment density it is possible - in our experience - to reach maximum depths of about 6 m. The equipment is maintenance-free and can easily be carried a few hundred meters without a car.

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Reviewer

Rainer Grün

Dose evaluation on a fossil tooth using multiple and single powder aliquots as well as a fragment

Rainer Grün, Steve Robertson
Research School of Earth Sciences
The Australian National University
Canberra ACT 0200, Australia

Matthew Gordon
Daramalan College
Canberra ACT 2602

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Introduction

The use of single aliquots for ESR dating of tooth enamel has the advantage that small samples can be analysed (5 to 30 mg), requiring less sample preparation time, and that sample inhomogeneity becomes insignificant. Single aliquots dose measurements can be carried out on powders as well as fragments. The analysis of the latter is necessary when working on valuable samples, such as fossil human teeth. Thus far, no experiments have been carried out to validate single aliquot measurements on powders nor fragments.

Experimental

We have selected sample 698, a relatively large bovid molar for the archaeological site of La Quina, which allowed the preparation of a series of sub-samples. The following procedures for dose estimation were tested (for the relative location of the sub-samples see Figure 1):

1) conventional multiple aliquots, one set (M2) heated for 2h at 90 °C after irradiation, then stored for at least 1 day at ambient temperatures and humidity following the recommendations of Hayes et al. (2000), the other set (M1) not heat treated;
2) single powder aliquots, not heated treated (S1 to S4);
3) single powder aliquots, heated for 2h at 90 °C and equilibrated after each irradiation step (H1 to H4);
4) a fragment, not heat treated (F2 was measured only).

The enamel of the fragments and single aliquots had an average thickness of about 1200 µm, whilst the enamel of the multiple aliquots was thinner (800 to 1000 µm).

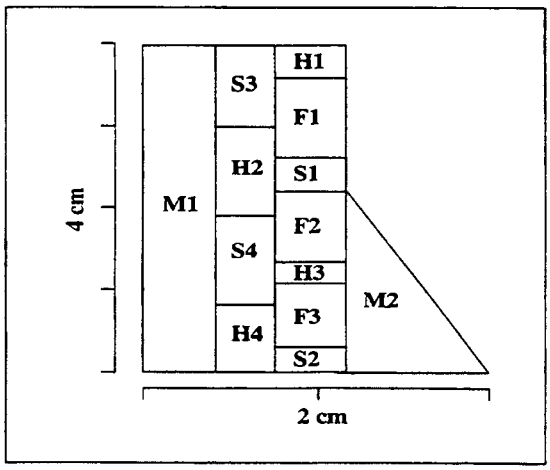

Figure 1.
Schematic drawing of the positions of the samples. M: multiple aliquots, S: single aliquots, not preheated; H: single aliquots, preheated. The enamel in the area of M2 was thinner than for the other samples.

Samples were irradiated with 0, 10.2 20.4, 40.8, 81.6, 132.6, 163.2 and 204 Gy using a ^{137}Cs source and measured using routine measurement conditions. The fragment was mounted in a quartz holder such that the outer and inner surfaces of F2 were perpendicular to the rotational axis. The sample was measured in 10° intervals with a programmable goniometer. For spectrum evaluation of the powders, we used a series of well described procedures (Grün 2000), plus natural spectrum fitting (scaling the

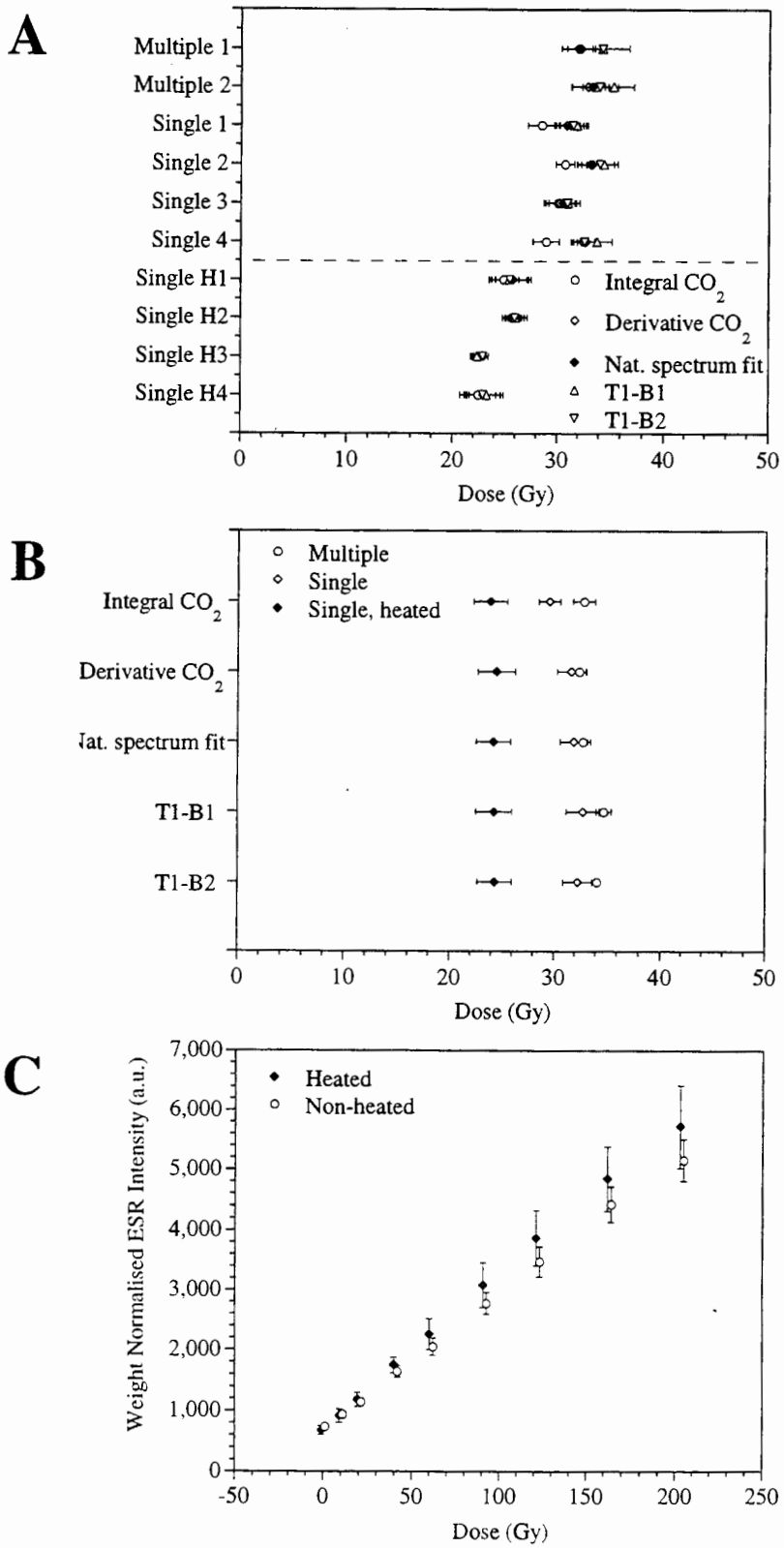

Figure 2.

(A) Dose values for each sample using a variety of evaluation methods
(B) Averaged dose values for the sample groups for a variety of evaluation methods
(C) Average, weight normalised ESR intensities of the single aliquots, non-heated and heated. The data points are slightly offset for clarity

natural spectrum into the irradiated spectra, for more details see Grün, submitted). The latter study concluded that natural spectrum fitting ought be the most reliable method for spectrum evaluation because it was least dependent on interferences. For the fragment, spectrum deconvolution (using four Gaussian peaks: see Robertson and Grün 2000) as well as natural spectrum fitting was applied. The dose values were obtained by linearisation of the single saturating exponential dose response function and Monte Carlo error calculation (Grün and Brumby 1994).

Results and Discussion

Figure 2 presents the results on the powder samples. The dose results for each sub-sample are not dependent on the method of spectrum evaluation (Figure 2A): the dose values of a given sub-sample agree within error (there may be some tendency for lower dose estimation when using integral deconvolution for samples S1, S2 and S4). The dose values within each group of samples (i.e. multiple aliquots, single aliquots: non heated, single aliquots: heated) also agree within error. There is also no quantifiable difference between M1 (not heated) and M2 where each aliquot was heated after irradiation.

Figure 2B shows the average dose values for each sample group. Again, the dose values show little dependence on the method of spectrum evaluation. The dose results of the multiple aliquots and the single, non-heated aliquots agree within error. The small systematic offset between the means of the multiple aliquots and the non-heated single aliquots can be attributed to the difference in enamel thickness. However, it seems clear that repeated preheating leads to significant smaller doses when using single aliquots (Figure 2B). The reason for the dose differences lie in changes in the natural, as well as the dosed samples. The heating decreases the natural intensity by about 9% whereas the intensity of the highest dose point increases by about 11% (Figure 2C). The effect could be explained by the presence of a less stable component (or indeed part of the stable component, depending on the kinetics) which is removed from the natural sample and that some other charges (not present in the natural sample) are re-distributed after irradiation (see also Brik et al. 2000, Hayes and Haskell 2000). Heating experiments by Scherbina and Brik (2000) indicate some charge transfer between the CO_2^- radicals with other "unobservable" centres at temperatures above 80°C. Alternatively, the heating could lead to the generation of additional precursors similar to those observed in speleothems (Yokoyama et al. 1985), mollusc shells (Brumby and Yoshida 1994) and corals (Yoshida and Brumby 1999)

Unfortunately, spectrum analysis does not show an unambiguously identifiable component that is apparently re-distributed in the spectra. However, the results clearly demonstrate that the prescribed heating procedure does not simulate geological aging, because the overall effect on the irradiated spectra ought be qualitatively the same as for the natural spectra (i.e. an overall decrease in the ESR intensity should occur). It should be noted here that experiments by Skinner et al. (2000) showed an increase of the dose value of a non-heated sample over 6 months. They concluded that this process could be simulated by preheating the aliquots for 3 days at 90 °C. On the other hand, Rhodes and Grün (1991) did not detect any changes in the dose values of two non-heated samples 2 years after the initial measurement. Whilst it is at present not possible to state that preheating is generally to be avoided, the setting chosen in this study is clearly inappropriate for single aliquots of geological samples. The experience from a large number of studies implies that ESR results obtained on non-heated samples does not yield systematic age overestimations. Indeed, samples from La Quina have caused consternation because they seem to yield ages, which are systematically too young (see Grün et al. 1997).

Figure 3 shows the powder spectra of M1 (Figure 3A) and the spectra for the enamel fragment F2 at 120° (Figure 3B) and at 220° (Figure 3C). These two angles show the largest spectral differences. Because of the angular distortions caused by the anisotropic crystal magnetic field of the hydroxyapatite fragment, the resulting spectra cannot be fitted with an ideal powder CO_2^- spectrum. The spectra of the fragment were analysed using spectrum deconvolution with four Gaussian lines (for more details see Robertson and Grün 2000), where Ax1 and Ax2 present the low and higher field component of the central, apparently axial signal. Peak-fitting with the natural spectrum was applied by optimising for the "whole peak" between S and E (see Figure 3A) as well as selected partial regions: T1 (between half height T1 and zero passing between T1 and B1), B1 (zero passing to half height between B1 and T2), B2 (half height between T2 and B2 and B2 to zero), the combinations of T1-B1, T1-B2 as well as T1-B2 width (S to T1 and B1 to E) and peak width (S to half height of T1 region and half height B2 to E). The latter two fitting methods are less dependent on interferences within the central peak region. Peak-to-peak methods were used for T1, B1, B2, T1-B1 and

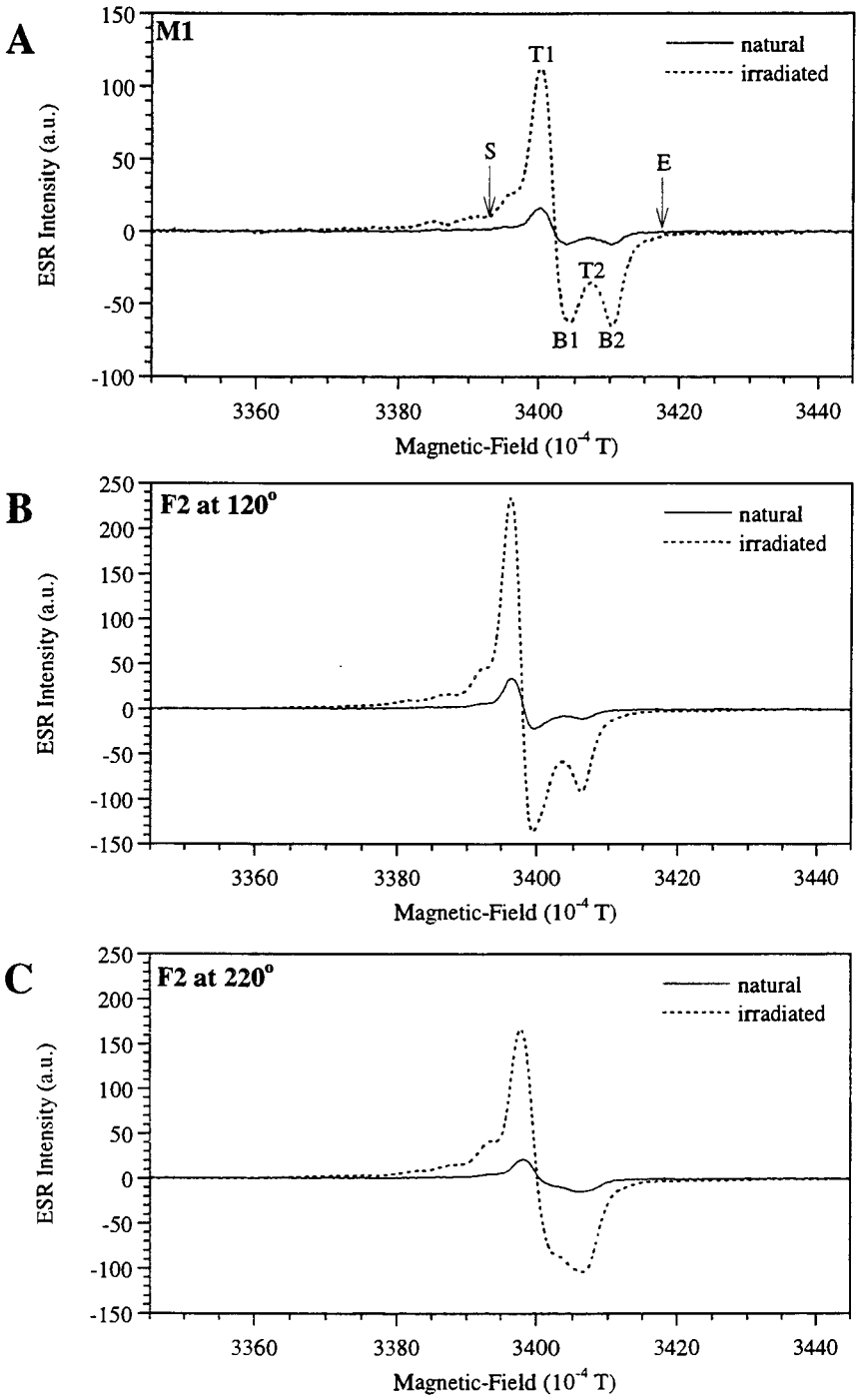

Figure 3.

ESR spectra of a powder (A) and the fragment measured at angles of 120° (B) and 220° (C). The positions S, T1, B1, T2, B2 and E denote positions used for peak-to-peak measurements and to define regions for natural spectrum fitting.

T1-B2. The dose values of all spectrum evaluation methods show angular dependencies (Figure 4). At about 120°, the T1-B1 peak is largest and the dose values derived from Ax1, Ax1 + Ax2 as well as natural spectrum fitting are about the highest. At 220°, where B2 is largest, doses derived from Ax2 are highest, those of Ax1, Ax1 + Ax2 as well as natural spectrum fitting about lowest, respectively. Natural spectrum fitting yields average dose results that are close to the average dose of the single, non-heated aliquots, whereas all dose results from the deconvolution (Ax1+Ax2) lie below this value.

Figure 5 shows the average dose values for the various methods of spectrum evaluation for the fragment.. The errors bars represent the magnitude of the angular dose dependency. Spectrum deconvolution (Ax1 + Ax2) shows the least angular dependency, but also yields the smallest dose value as well as the largest deviation from the average dose value of the single aliquot powders (if sample selection and spectrum evaluation were ideal, the dose value of the fragment should be equal to the dose value of the single aliquot powder). All regions of interest of the natural fitting as well as all peak-to-peak dose evaluations, albeit associated with larger errors, agree with the average dose value of the single aliquot powders. The reason why spectrum deconvolution of the absorption spectrum yielded the largest deviation from the average dose value of the powders most likely lies in the correct placement of the "wide" line in the deconvolution process (for more details see Robertson and Grün 2000).

When the dose response curves are normalised on the natural intensity, it can be observed that the angular dose dependency is caused by an angular sensitivity change. Similar observations were made by Brik et al. (2000) who attributed these interferences to a chaotic CO_2^- radical. Alternative explanations involve phase sensitive interferences or some anisotropic, non-radiation sensitive peaks. At the moment, however, we are not able to unambiguously identify these interferences or provide a plausible explanation for them.

Summary

Dose evaluations on multiple and single aliquots of powders yield consistent results. However, post irradiation heating causes severe dose underestimations. Although is it difficult to pinpoint the interferences which cause the intensity changes in the heated samples, it is clear that the prescribed heating does not simulate the geological ageing process, at least for this sample. We find that the average dose value of the single aliquot powders lie within the dose range provided from a variety of spectrum methods on the spectra of the fragment. Spectrum deconvolution, though being by far the most time intensive method, yielded the largest deviation between the dose values of the fragment and the powders, respectively.

It is clear that more experiments are required to pinpoint (and eliminate) any interfering signals in the ESR spectra of powders and fragments and that systematic fading tests ought be carried out. At seems at this stage, that pre-heating procedures should be treated with suspicion.

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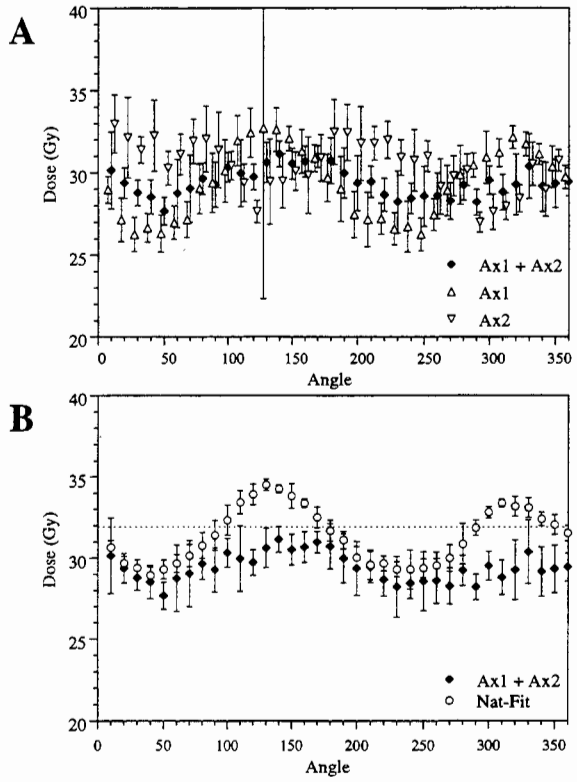

Figure 4.

(A) Dose values of the fragment F1 derived from spectrum deconvolution (for details see Robertson and Grün, 2000)
(B) Comparison of the dose values of the fragment obtained by spectrum deconvolution and natural spectrum fitting

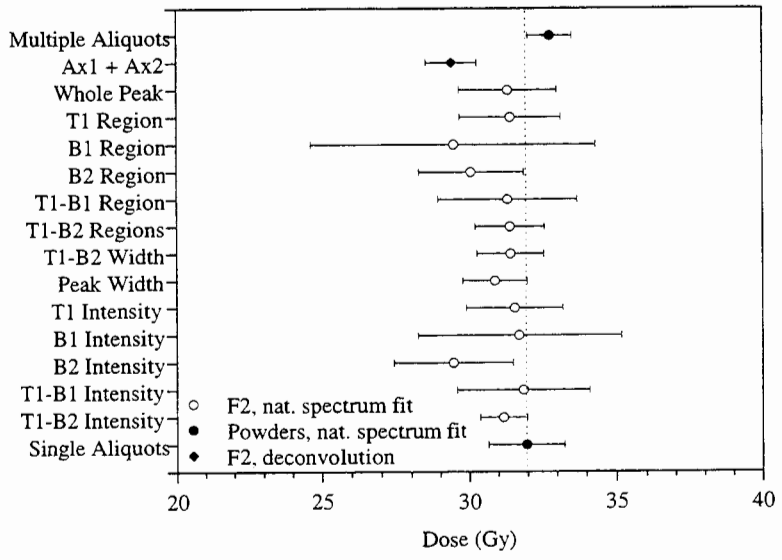

Figure 5.

Summary of dose results. If there were no interferences, the dose of the fragment ought be the same as of the non-heated, single aliquots (dotted line). All evaluation strategies on the fragment yield dose values that are statistically indistinguishable from the single aliquots. There seems a tendency towards smaller dose values in the B2 region (see values for B2 region and B2 intensity).

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Reviewer
H. Schwarcz

Light transmittance through dry, sieved sand: some test results

Jeff Ollerhead

Department of Geography, Mount Allison University, 144 Main St., Sackville, New Brunswick, Canada E4L 1A7

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Abstract: *a series of measurements was made at different light wavelengths of transmittance through various thicknesses of dry sand grains from a variety of size classes. Transmittance was found to decrease approximately exponentially with increasing sand layer thickness and with decreasing grain size. Transmittance did not vary significantly with incident light wavelength. It is concluded that for any dry sand, a layer with a thickness of 7 mm, and possibly much less, is capable of reducing the transmittance of incident light to 0.05 % or less. This result should be considered when collecting a surface sample as a modern analogue for a sample to be optically dated.*

Introduction

It is now generally accepted that optical dating techniques can be used to determine the length of time that has elapsed since a mineral was last exposed to daylight (Aitken, 1998; Huntley and Lian, 1999). However, as Wintle and Huntley (1982) noted almost two decades ago, a necessary criterion for a valid luminescence age is that a modern sample in the same context yield an age of zero. Thus, in an 'ideal world', a modern sample (usually a surface sample) from an environment under study yields an optical age of zero. However, this is not always the case. For example, Wintle *et al.* (1994) obtained an optical age of 40 ± 15 years for a surface sand sample. In my own work, I have obtained optical ages significantly greater than zero for modern samples. An age greater than zero for a modern sample can occur because: (i) the sample is not well bleached prior to collection, (ii) there is a deficiency in the dating technique used (*e.g.*, an inappropriate laboratory bleaching procedure, such as one which uses photons not present in nature, is used), or (iii) both (i) and (ii). The focus here is on one factor related to bleaching: overlying sediment that might block light penetration.

Modern samples should normally be collected as analogues for older samples being dated. If modern samples are found to be inadequately bleached, this raises questions about whether older samples of interest were adequately bleached prior to deposition. An understanding of the environment from which a sample is collected is clearly needed to address the question of whether or not one should expect mineral grains in a modern sample to be bleached sufficiently to yield an optical age of zero. In my own work, I have usually collected the top 0.5-1.0 cm of sediment from a site as a modern sample. This is done on the assumption that this layer of grains is well bleached. In an active environment (*e.g.*, on a beach) this is probably a fair assumption as the grains are usually in motion every few days or weeks. Nevertheless, it became apparent that a more concrete understanding of the magnitude of light attenuation through a given thickness of sediment is desirable.

A search of available literature revealed little data to aid in this understanding. Others have studied the bleaching process (*e.g.*, Huntley, 1985; Berger, 1990; Gemmell, 1994; Huntley and Clague, 1996) and most review papers on the subject (*e.g.*, Berger, 1995; Huntley and Lian, 1999) suggest various mechanisms for incomplete bleaching (*e.g.*, overlying water, rapid burial, coatings on grains, etc.). However, the only paper found directly related to this topic was that of Ditlefsen (1992) who investigated the bleaching of K-feldspars in turbid water suspensions. His approach was to bleach samples in different suspensions and then measure their luminescence and compare it to that of unbleached grains. He showed that in dense suspensions (> 0.05 g/l) little bleaching took place. This led him to consider the limitations of using optical dating techniques to obtain ages for water-laid sediments.

From Ditlefsen's (1992) results one can infer that light attenuation through dry sediments should be very rapid. The purpose of this study was to test this idea. The key variables were assumed to be: (i)

sediment thickness, (ii) grain size, and (iii) light wavelength. Transmission of light through sand can happen in two ways: (i) transmission through grains, and (ii) transmission through spaces between grains. It was assumed that most of the light would be transmitted through spaces between grains although this was not tested. It was expected that light transmission would decrease exponentially with sand thickness. It was also expected that light transmission would decrease with decreasing grain size because, although the pore volume would be unchanged, there would be more reflections at grain surfaces and during each reflection a significant fraction of the intensity is not reflected. Given that the sand tested contained different minerals (*e.g.*, quartz, K-feldspar, etc.), a variation in light transmittance with wavelength was anticipated although the nature of such an effect was unknown. In order to test the hypotheses, a series of measurements were made at different light wavelengths of transmittance through various thicknesses of sand grains from a variety of size classes.

Sample collection, preparation and measurement

A large (~ 1 kg) sand sample was collected at Cape Jourimain, New Brunswick. In the laboratory it was rinsed with distilled water and then dried for 24 hours at 105°C. It was then sieved, in batches, to extract the following size fractions: -2.0, -1.0, -0.5, 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5 and 4.0 ϕ. In each case the size fraction value is that of the smallest grains in a fraction (*e.g*, the 1.0 ϕ fraction contains grains that fit through a sieve with 0.5 ϕ openings but not through a sieve with 1.0 ϕ openings). The mm equivalents for each ϕ size are provided in Table 1.

A custom holder was machined to hold a pair of standard microscope slides (25 mm x 75 mm x 1 mm thick) at the following spacings: 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0, 7.0, 9.0, and 11.0 mm. To establish a reference prior to each measurement, transmittance for a given spacing with no sample was measured from 320 to 820 nm at 2 nm increments using a Hewlett Packard 8452A diode-array spectrophotometer. Sand grains from a given size class were then poured into the space between the two microscope slides and transmittance measured from 320 to 820 nm at 2 nm increments. The instrument used is a single-beam, microprocessor controlled, UV/VIS spectrophotometer with collimating optics. The beam is 6 mm x 8 mm and its intensity is approximately 28 $mW \cdot cm^{-2}$. Wavelength accuracy is ±2 nm and wavelength reproducibility is ±0.05 nm. The lower limit of measurable transmittance is 0.05 %. Some grain-size classes could not be measured at some spacings because the grains were larger than the space between slides. All results presented are for the mean of two independent trials.

Grain-size class		Sand thickness required to reduce T to ≤ 0.05 %
(ϕ)	(mm)	(mm)
-2.0	4.00	7
-1.0	2.00	5
-0.5	1.41	5
0	1.00	4
0.5	0.71	3
1.0	0.50	2
1.5	0.35	1.5
2.0	0.25	1
2.5	0.18	< 1
3.0	0.13	< 1
3.5	0.09	< 1
4.0	0.06	< 1

Table 1.

Thickness of sand required (in mm) to reduce the transmittance of incident light to 0.05 % or less (the lower limit of the instrument used is 0.05 %) for the grain-size classes tested for incident light wavelengths from 320 to 820 nm. Transmittance was found to be independent of wavelength so this variable is not included in the table.

Measurement results

Representative examples of the results obtained are presented in Figures 1 and 2. As expected, transmittance was found in all cases to decrease approximately exponentially with increasing sand layer thickness. For example, Figure 1(a) shows transmittance of about 7 % for a 1.5 mm thick layer of grains from the 1.4 mm size class; transmittance drops to about 1 % for a 3.0 mm thick layer of the same grains. Figure 1(b) shows that transmittance is only about 1 % for a 1.5 mm thick layer of grains from the 0.7 mm size class. It was also found that transmittance decreases approximately exponentially with decreasing grain size. For example, Figure 2 shows the rapid decrease in transmittance with decreasing grain size for a layer of sand 1 mm thick. As Figures 1 and 2 also indicate, no observable dependence on incident light wavelength was found.

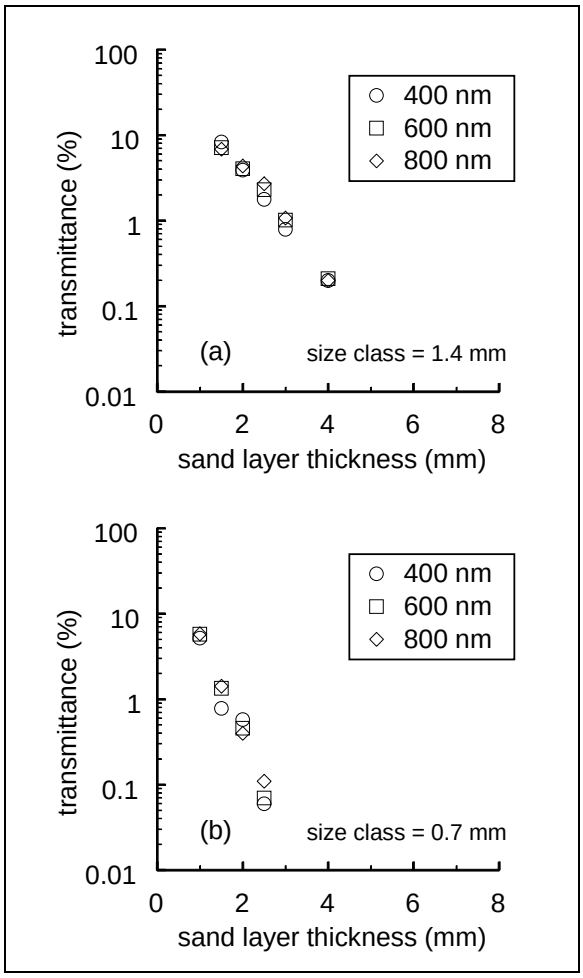

Figure 1.
Relationship between light transmittance and sand layer thickness for light at 400, 600, and 800 nm for the (a) 1.4 mm, and (b) 0.7 mm grain-size classes.

Table 1 shows the thickness of sand required (in mm) to reduce the transmittance of incident light to 0.05 % or less for the grain-size classes tested. These data show that a sand layer thickness of 7 mm is sufficient to reduce the transmittance of incident light to 0.05 % or less at all wavelengths tested for any grains smaller than 4 mm in diameter. They also show that a sand layer thickness of 1 mm is sufficient to reduce the transmittance of incident light to 0.05 % or less for all wavelengths tested for any grains smaller than 0.25 mm in diameter.

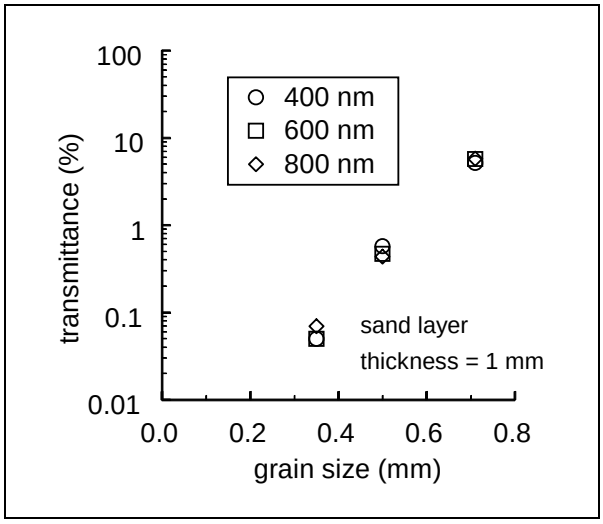

Figure 2.
Relationship between light transmittance and grain size for light at 400, 600, and 800 nm for a layer of sand 1 mm thick.

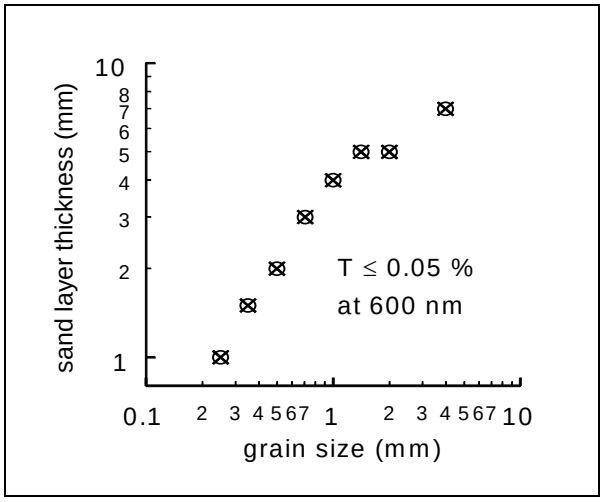

Figure 3.
Sand layer thickness required to reduce the transmittance of 600 nm incident light to 0.05 % or less (the lower limit of the instrument used is 0.05 %) for the 8 largest grain-size classes tested.

Figure 3 summarizes the results presented in Table 1 for 600 nm incident light. It shows the sand layer thickness required to reduce transmittance to 0.05 % or less for a given grain-size class. Although there is scatter in the data, primarily due to the fact that discrete sand layer thicknesses were used, the nature of the relationship is apparent.

Discussion and conclusions

The results show that light transmittance decreases approximately exponentially with increasing sand layer thickness and with decreasing grain size. The greatest amount of scatter in the data was for the larger grain sizes. This may be due to much greater variability in the packing of the larger grains which tend to be more angular than their smaller diameter counterparts. In supplemental testing, tapping the microscope slides to consolidate grains in the larger size classes (≥ 2 mm) resulted in reductions of transmittance by a factor of two or three. For grains from the smaller size classes (≤ 1 mm), tapping the microscope slides had no measurable effect on transmittance. Note that 'natural sands' are likely to be well packed and include a variety of grain sizes and thus transmittance through them is likely to be much less than what is reported here for sands with a very limited grain-size range.

No relationship between light transmittance and incident light wavelength was observed. This does not mean that a relationship does not exist but it does suggest that light wavelength is not an important variable in predicting rate of light attenuation through dry sand where more than a monolayer is present. It may also be an indication that, as assumed, most of the light is being transmitted around the grains rather than through them.

The results indicate that for any sand, a layer with a thickness of 7 mm, and possibly much less, is capable of reducing the transmittance of incident light to 0.05 % or less. This should be taken into account when collecting a surface sample as a modern analogue for a sample to be optically dated. As suggested previously, collecting the top 10 mm of a sand in an active environment is probably reasonable since one would expect most of the grains to be in motion every few days or weeks and thus be well bleached. In a less active environment (*e.g.*, the bed of a sheltered lake), making this assumption is probably not wise. One should try to collect only the actual surface grains as a modern analogue in this case. One technique for doing this is by using adhesive tape as first described by Readhead (1984).

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Reviewer

J.R. Prescott

Comments

This is a useful piece of laboratory work in support of field measurements and is helpful in interpreting them. It is surprising how little direct laboratory work of this kind has been reported. Since it also good physics, it appeals to me. My generation would probably ask, "What did Rayleigh say about it?" The exponential absorption relation and its eventual breakdown would come as no surprise to him. For clean quartz, no wavelength dependence is expected for particles in this size range. In the wider luminescence dating world, almost everyone one who has tried it, finds that the surface layer 0.5-1.0 cm has a small but non-zero age, and/or that quartz bleached in layers in the laboratory reaches a lower level than surface quartz collected in the field. The present paper throws some light on this, if the play on words may be forgiven.

Such measurements were carried out quite early in the application of luminescence techniques to sediments. Ollerhead refers to Readhead's thesis (1984). This relatively inaccessible reference contains an account of considerable laboratory work, which was also reported, in part, at the Cambridge LED meeting: Readhead, Quater. Sci. Rev. 7, 257-264 (1988). Historians may also care to look up the Helsingor LED Conference Proceedings at: Council of Europe Journal PACT 9, 505-512 (1983). Modesty forbids naming the author.

It is worth adding that there is a difference between TL and OSL in that some samples are never completely zeroed for TL, no matter how long they are exposed to sunlight.

Comparison of dose rate determination using high – resolution gamma spectrometry and inductively coupled plasma – mass spectrometry

Frank Preusser [1,2] and Haino Uwe Kasper [1]

[1] Geologisches Institut, Universität zu Köln, Zülpicher Strasse 49a, D-50674 Köln, Germany; preussef@uni-koeln.de; HU.Kasper@uni-koeln.de
[2] Geologisches Institut, Universität Bern, Baltzerstrasse 1, CH-3012 Bern, Switzerland.

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Introduction

Determination of dose rates for luminescence and electron spin resonance (ESR) dating is possible using a variety of different analytic methods. The investigations presented here were initiated by the need to determinate the concentration of dose rate relevant elements (K, Th, U) of sediment samples from drilling cores. High resolution (HR) gamma spectrometry, predominantly used in our laboratory, was not practical due to the limited amounts of sample material available. One major advantage of inductively coupled plasma – mass spectrometry (ICP–MS) is the possibility to analyse samples as small as 10 mg. The performance of sample preparation (homogeneity, digestion) and of the measurement itself can be verified by comparison with certified reference materials (CRM).

Disequilibria within the thorium and uranium decay series is a potential error source in luminescence and ESR dating. The most suitable approach to test for disequilibria is a combination of alpha spectrometry and low-level gamma spectrometry (Krbetschek et al., 1994; Olley et al., 1996). However, the low activities of ^{232}Th and ^{238}U make these measurements rather time consuming (some days per sample) precluding regular checks in routine dating procedures. An alternative method to allow evaluation of sample secular equilibrium is to measure the mother isotope at the head (ICP–MS) and the daughters at the end of a decay chain (HR gamma spectrometry). However, it is first necessary to exclude systematic differences between the two methods caused by difficulties with analytical procedures.

The results of ICP–MS and HR gamma spectrometry are compared for a variety of samples from different geological sites. These samples are likely to be in equilibrium state due to their depositional history. For one sample, repeated measurements were carried out to obtain more information concerning the reproducibility of the results. A brief description of samples used in this study is presented in Tab. 1.

Table 1.
*Description of samples analysed within this study. More information about the certified reference materials (CRM) is given by (*1) Govindaraju (1994) and (*2) Pszonicki et al. (2000), respectively*

Sample	Origin	Sediment type	Grain-size
AJS 1–3	Oman	aeolianite	sand
BBR	Germany	house standard (basalt)	
BCR–1	USA	CRM (basalt) *1	
BGR 1–11	Argentina	loess and dune sand	silt or sand
GSD–3	China	CRM (fluvial) *1	
GSD–4	China	CRM (fluvial) *1	
HAB 1–3	Switzerland	fluvial deposit	sand or silt
HÜW 1–3	Switzerland	overbank deposit	sand or silt
NUSSY	Germany	loess	silt
PGM 1–4	New Zealand	estuary deposit	sandy silt
PIC 5–7	New Zealand	coastal deposit	sand or silt
SOIL–7	Austria	CRM (soil) *2	
WIL 1–5	Oman	desert lake and dune sand	sand

High resolution (HR) gamma spectrometry

HR gamma spectrometry was carried out using a high–purity germanium detector (Canberra GC2019–7935.2, coaxial p/n–type, 20 % relative efficiency, 1.9 keV FWHM at 1.33 MeV). After drying the

samples at 50°C and determining natural water content, 800 g or 1600 g of sediment were filled into Marinelli beakers. Analyses were performed after one month of storage to build up equilibrium between radon and its daughters. Samples were measured for 20 hours. The influence of environmental radioactivity was minimised by a shielding of low–activity lead. Using large samples and measuring for a long time significantly reduces the effect of background variability. Since natural radioactivity is generally weak for the samples which had been analysed (0.5 – 2.0 % of K, 1 – 15 μg g^{-1} of Th, 1 – 4 μg g^{-1} of U), most of the thorium and uranium series γ–lines are close to the detection limit. Calibration was done by home standards containing known quantities of added radioactive elements (IAEA).

The results shown in Tab. 2 represent the average of 11 measurements of sample NUSSY, an Upper Weichselian loess from the Nußloch quarry near Heidelberg. All analyses were carried out during a period of two years using the same sealed container. This procedure provides accurate information of uncertainty due to peak matching and the error caused by background variability. The relative standard deviation (RSD) for different γ-lines ranges from approximately 1 % to 6 %. The poorer reproducibility is given for energy peaks where the signal/background ratio is low (e.g. for the peak of ^{212}Pb at 300.1 keV). The RSD of the average of all measurements and of all energy peaks is 2.1 % for K, 4.6 % for Th and 3.4 % for U.

Inductively coupled plasma – mass spectrometry (ICP-MS)

50 g of each sample were pulverised in a laboratory disc mill (tungsten carbide grinding barrel) and homogenised for analysis. Prior to ICP–MS analysis the sample material was dried overnight at 105°C (dry basis). All reagents used were Suprapur® grade (Merck).

Using conventional HF/HNO_3 decomposition, the recovery of Rb, Th and U will be severely restrained by selective coprecipitation with insoluble fluorides of major elements. The use of $HClO_4$ instead of HNO_3 enables an almost 100 % recovery of trace elements by completely suppressing fluoride precipitation (Yokoyama et al., 1999). Quantitative decomposition of samples, especially those containing resistant heavy minerals (e.g. Zircon), was considered using two different digestion protocols:

(1) *Conventional open system decomposition*
3 ml of 10 M $HClO_4$ and 3 ml of 22 M HF were added slowly to the weighed sample (15 ml Savilex PFA–vials, 100 mg) to prevent losses by violent carbonate reaction. After repeated sequential evaporation with 6 M HCl and 5.5 M HNO_3 the residue was dissolved with 0.5 M HNO_3, transferred to 50 ml volumetric flasks and stored in tightly closed HD-PE bottles. This procedure lasts for at least 2–3 days.

(2) *High pressure MULTIWAVE microwave digestion system*
This automated system is equipped with special designed 50 ml TFM-vessels in combination with an evaporation rotor (Anton–Paar/Perkin–Elmer 6EVAP) and an aspiration-evaporation device (Prolabo ASPIVAP) (Knapp et al., 1997). The p–T monitored microwave-powered closed system reaches maximum temperatures of 260° C (for 20 minutes) and a pressure of 70 bar. Reagents and sample quantity used are identical to the ones used in the conventional decomposition protocol. The main advantage of microwave digestion is the short time needed (ca. 7–8 hours) and a guaranteed quantitative sample dissolution (Kasper and Preusser, 2000).

The elements K, Rb, Th and U were analysed by inductively coupled plasma – quadrupole mass spectrometry (ICP–QMS, Sciex/Perkin-Elmer ELAN 6000). Special attention was focused on automatic and customised corrections for isotopic interference, interfering molecular species, specially customised resolution for K and internal standardisation.

The NUSSY sample was used for an inter–laboratory comparison and the results are shown in Tab. 3. For most of the measurements, the results are similar when the standard deviation is considered. However, the contents of U analysed with ICP–MS at the Cologne laboratory are slightly lower compared with the average results determined by the other laboratories. The concentrations of K and Th analysed by a commercial laboratory are slightly higher in comparison with the contents measured by the other laboratories.

Comparison of ICP-MS, HR-gamma spectrometry and CRM

Concentrations of dose rate relevant elements of 34 samples have been determined using ICP–MS and HR gamma spectrometry, respectively (Fig. 1–3).

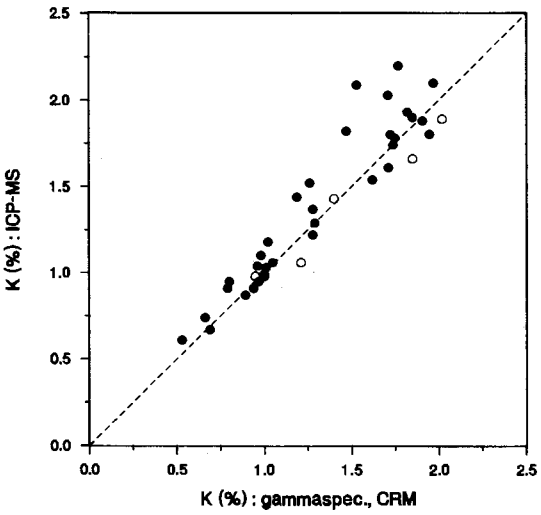

Figure 1.
Comparison of concentration of K determined by HR gamma spectrometry (closed circles) and CRM (open circles), respectively, versus ICP-MS.

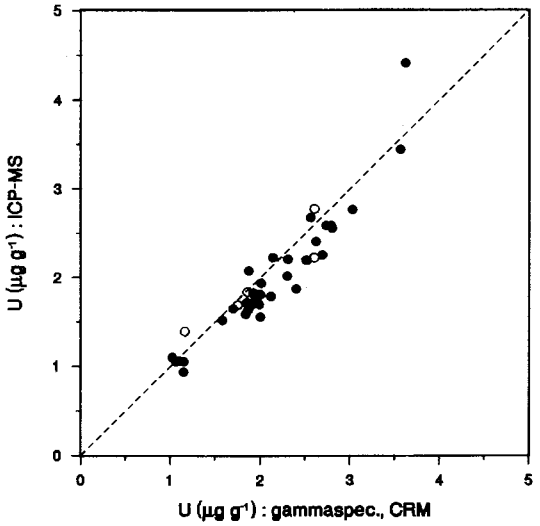

Figure 3.
Comparison of concentration of U determined by HR gamma spectrometry (closed circles) and CRM values (open circles), respectively, versus ICP-MS.

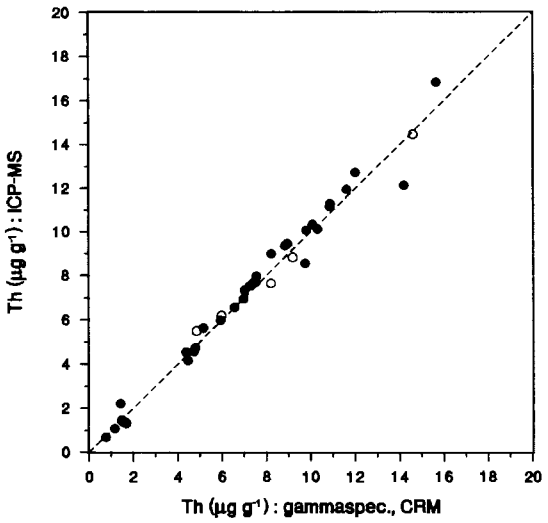

Figure 2.
Comparison of concentration of Th determined by HR gamma spectrometry (closed circles) and CRM (open circles), respectively, versus ICP-MS.

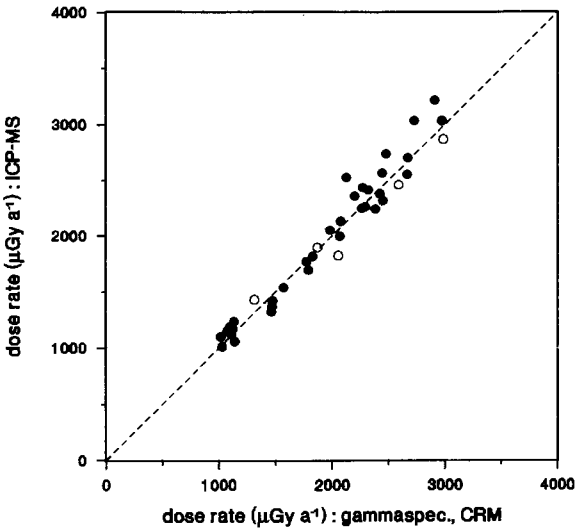

Figure 4.
Plot of dose rates calculated for HR gamma spectrometry (closed circles) and CRM values (open circles), respectively, versus dose rates determined using the ICP-MS results.

Table 2.

HR gamma spectrometry results for the different energy peaks of potassium and of isotopes from the thorium and uranium series of the NUSSY sample. The averages (n = 11) of the deduced contents of dose rate relevant elements, standard deviation (SD) and relative standard deviation (RSD), and the signal to background (BG) ratio with standard deviation are shown for repeated measurements.

Element	Peak (keV)	Content	SD	RSD	signal/BG
Potassium (%)					
K–40	1460.0	0.96	0.02	2.1	26.3 ± 1.9
Thorium (μg g^{-1})					
Tl–208	583.2	7.29	0.18	2.5	3.5 ± 0.2
Tl–208	2614.5	7.25	0.27	3.7	40.2 ± 7.3
Pb–212	238.6	7.37	0.08	1.1	3.8 ± 0.1
Pb–212	300.1	7.43	0.47	6.3	0.37 ± 0.03
Bi–212	727.3	7.30	0.44	6.0	1.0 ± 0.1
Ac–228	338.3	7.67	0.35	4.6	1.3 ± 0.2
Ac–228	911.1	7.58	0.25	3.3	4.2 ± 0.2
Mean	–	7.41	0.34	4.6	–
Uranium (μg g^{-1})					
Pb–214	295.2	2.67	0.06	2.2	2.3 ± 0.1
Pb–214	351.9	2.70	0.04	1.5	4.6 ± 0.4
Bi–214	609.3	2.70	0.03	1.1	6.3 ± 0.6
Bi–214	1120.3	2.69	0.09	3.3	2.0 ± 0.2
Ra–226, U–235	186.0	2.61	0.16	6.1	0.53 ± 0.04
Mean	–	2.68	0.09	3.4	–

Table 3.

Comparison of analyses determining the dose relevant elements (RSD) for the NUSSY sample carried out by different laboratories using ICP–MS and HR gamma spectrometry, respectively.

Laboratory		K (%)	Rb (μg g^{-1})	Th (μg g^{-1})	U (μg g^{-1})
Cologne (γ–spec.)	(n = 11)	0.96 (2.1)	–	7.4 (4.6)	2.7 (3.4)
Cologne (ICP–MS)	(n = 10)	1.04 (8.9)	51.8 (2.3)	7.7 (2.1)	2.3 (4.4)
GFZ (ICP–MS)	(n = 3)	–	51.6 (1.5)	7.7 (5.3)	2.6 (0.8)
Commercial (ICP–MS)	(n =1)	1.14	–	8.8	2.7

Generally, the results for both methods are in good agreement.

For some samples significantly higher K contents were determined using ICP–MS (Fig. 1). This is due to the atomic weight of K and hence the massive isotopic interference of Argon from the plasma source. With respect to reproducibility and accuracy of the ICP–MS results, we intend to carry out further tests using alternative analytical methods such as atomic absorption spectrometry (AAS) or flame emission spectrometry (FES). Additionally, the remarkable covariance of Rb and K might be used for cross-checking the results.

Slightly lower U contents in comparison with HR gamma spectrometry were measured using ICP–MS (Fig. 3) as it has already been shown in the laboratory inter–comparison test (Tab. 3). It is possible that the

conventional digestion protocol might not always guarantee complete dissolution of samples due to chemically resistant minerals such as Zircon. Thus, we favour the use of high–pressure microwave digestion in future research.

A comparison of dose rates calculated from the concentration of radioactive elements as determined by the two different methods is shown in Fig. 4. Dose rates have been calculated for 150 μm quartz grains assuming an alpha efficiency of 0.1 and a sediment moisture of 20 %. Neither cosmic rays nor internal dose rate contribution have been considered.

Calculations were carried out using the AGE program developed by R. Grün which uses the conversion factors of Nambi and Aitken (1986). The differences in dose rates of the two different methods is generally less than 10 % but it has to be considered that some of the ICP–MS results may be affected by methodological difficulties.

Conclusions

Determination of the concentration of dose rate relevant elements analysed by ICP–MS and HR gamma spectrometry yield consistent results for most of the samples. The ICP–MS results are in good agreement with values of analysed CRM. However, digestion of samples and the custom resolution technique used for the determination of K have to be considered carefully. Methodological improvements may further increase the accuracy of ICP–MS analyses, in particular for K. It is emphasised that a combination of ICP–MS and HR gamma spectrometry may reduce the uncertainties in dose rate determination by the ability to routinely check for secular disequilibria within the thorium and uranium decay series.

Acknowledgements

The NUSSY sample was homogenised and provided by J. Heinicke, A. Lang, U. Rieser and L. Zöller. P. Dulski, GeoForschungs Zentrum (GFZ) Potsdam, contributed with three ICP–MS analyses of the NUSSY sample. M. Bateman provided results determined by a commercial ICP–MS laboratory. We want to express our special thanks to N. Willems who initially calibrated the HR gamma spectrometer used in this study and to M. Kelly for improving the English. M. Krbetschek is greatly thanked for reviewing the manuscript which benefited from his constructive comments.

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Reviewer

M. Krbetschek

Thesis abstract

Thesis title: Studies of quartz luminescence sensitivity relevant to dating and dosimetry
Author: Chen Geng
Date: May 2000
Supervisor: Dr. S.H. Li ; Examiners : Prof. A.G. Wintle and Dr. J.K.C. leung
University: The university of Hong Kong

Quartz luminescence sensitivities, both OSL and 110°C TL, to irradiation dose were studied. The sensitivity is expressed as the ratio of luminescence centres (L) to non luminescence centres (R), and is explained using a model in respect of its changes with dose, heating and UV exposure. A modified model is introduced involving multiple R hole centres. It is found that only one type of R centre is involved in the OSL sensitivity change, and it is shared with the 110°C TL. However, more hole centres are involved in production of the 110°C TL.

Experiments involving annealing at various temperatures indicate that sensitivity changes of both OSL and 110°C TL signals are temperature dependent. At temperatures below 500°C, different filling and emptying of R and L centres is the dominant process and governs the sensitivity changes. There is a linear relationship between the OSL sensitivity and 110°C TL sensitivity with regeneration dose. Most holes can be transferred from R centres to L centres by annealing at 500°C for 10 minutes. Above 500°C, the sensitivity change results mainly from the destruction of R centres. The initial destruction of R centres happens at the first phase change of quartz at 573°C. At the second phase change of quartz at 870°C, the destruction of L centres occurs.

Isothermal decay experiments and quick heat experiments are applied to obtain the parameters of the OSL trap and the R centres. The lifetime of the OSL trap is found to be in a range from 10^8 to 10^9 years at 20°C. It is suggested that the OSL signal is stable enough for the dating of one million-year old samples. The OSL 325°C trap contributes an overwhelming majority of he OSL signal used in the OSL dating process. The lifetime of the R center is found to lie in the range from 1 to 30 ka at 20°C. The decay of R centres results in OSL sensitivity changes. The effect of sensitivity change must be considered in dating and dosimetry measurements.

Competition for electron between the OSL trap (325°C TL trap) and other traps is observed. The electron traps shallower than OSL trap compete with the OSL trap, but the traps deeper than the OSL trap do not.

Thesis abstract

Thesis title: Development and application of luminescence dating to quaternary sediments from China
Author: Jiafu Zhang
Date: August 2000
Supervisor: Dr. S.H. Li and Dr. M-Y.W. Tso ; examiners : Prof. T.M. Chen and Dr. D.D. Zhang
University: The university of Hong Kong

A three-stage single-aliquot additive-dose protocol (3-SAAD) for the equivalent dose (ED) determination of potassium feldspar is proposed. This procedure consists of 3 stages: preheat/measurement, dose/preheat/measurement and preheat/measurement; all the measurements for the equivalent dose determination are made on a single aliquot of feldspar. 3-SAAD provides a simple and efficient way of checking the appropriateness of preheat conditions during the ED estimation; the effects of the differences in mineralogy and luminescence properties from aliquot to aliquot are minimized. This procedure is also suitable for ED measurements on individual grains of feldspar.

An isochron technique is proposed, based on the EDs of grains (or aliquots) as a function of internal dose rate. This technique uses both quartz and feldspars grains (of the same size range), and takes advantage of the range in internal dose rates in these minerals to overcome uncertainties in the external dose rate. Even in the absence of independent age controls, the validity of the isochron ages can be assessed using the isochron equation. A significant improvement in dating uncertainty is demonstrated.

Assessment of the adequacy of bleaching of optical signals is very important for optical dating of sediments. A new method of assessing the bleaching of sedimentary feldspar grains is proposed, based on the difference in the bleaching rate between IRSL and TL signals. The size and scatter of the bleaching factors (normalized ratio of IRSL to TL) of grains (or aliquots) from a sample indicate the degree of bleaching. Well bleached grains can be identified by a relatively small bleaching factor, and relatively small scatter. All measurements required to estimate the ED and the degree of bleaching are carried out on the same grain (or aliquot). HF etching is necessary in optical dating of feldspar. Feldspars from different samples have different etching characteristics. For sedimentary feldspars from Hong Kong, it is found that 40 min etching with 10% HF at room temperature is appropriate.

Finally, three types of sediment from different areas are systemically dated using the optical techniques. They are colluvium sediments from Sham Wat debris lobe, Hong Kong, coastal deposits from area offshore of Hong Kong and dune sand samples from Horqin sandyland of northeastern China. Potential difficulties in optical dating of such sediments are discussed.

Note

Some notes on language

The language of science is supposedly precise. In practice it is often not so, sometimes through ignorance or carelessness, and sometimes because precision requires so much to be said as to be impracticable. With this in mind, I hope these notes will help curtail some of the misuses of terms that have crept into the literature. I write these notes knowing full well that I am sometimes guilty of comparable offences against the language.

Age or Date? A date refers to a specific year in the past, for example AD1666 or 326 BC. An age refers to the amount of time of past existence, for example an age of 350 years means the thing referred to has existed for 350 years. The word "date" is often used erroneously when "age" is meant. "Date lists" are often really "age lists".

Black-body radiation. This is an idealized concept. Imagine a closed cavity in thermodynamic equilibrium, then the electromagnetic radiation within the cavity is a function only of the temperature, and is referred to as black-body radiation. If a tiny hole is made in the cavity then the radiation that comes out of the hole is a close approximation to black-body radiation. In contrast the radiation that is emitted by something that is hot is NOT blackbody radiation unless it is perfectly "black" and no such material exists. Such radiation is of less intensity than black-body radiation, and the spectrum will be different. The word incandescence is used to describe the emission from a tungsten light bulb or any other visibly hot object.

Dose.
The term "radiation dose" has a well-defined meaning; it is the amount of radiation energy absorbed per unit mass of matter. Thus if 1 joule of radiation energy is absorbed in 1 kilogram of matter, the radiation dose is 1 $J.kg^{-1}$ or 1 Gy. The key words here are 'energy' and 'absorbed' . What happens to the energy after it is absorbed is completely irrelevant to the evaluation of the dose. In practice, most of the energy will end up in the form of heat and leave the matter in question by thermal conduction, convection or electromagnetic radiation (there is an exception to this is if the matter is thermally isolated from the surroundings). A small fraction of the energy absorbed will end up stored by electrons and atoms in excited states. It would be wrong to talk about the absorbed radiation energy contained in something. Thus it would be equally wrong to talk about a dose contained in something and the phrases "dose in", "stored dose", "contained dose", acquired dose" and such should not be used.

Dose rate or Annual Dose? The annual dose is the radiation dose that occurs in one year and the SI unit for it is the gray (Gy). The dose rate is the radiation dose per unit time, and the practical unit for it is $Gy.ka^{-1}$. The term "annual dose rate" should not be used.

Luminescence. Luminescence is the light emitted by a substance in response to a stimulus. The stimulus may be heat, light, sound, shock, a beam of electrons, or anything else. The terms thermoluminescence, photoluminescence, sono-luminescence etc are used to refer to the light emitted in the cases of different stimuli. The term "optical luminescence" that is sometimes used does not make any sense since luminescence is already an optical phenomenon. Luminescence is not something that can be stored, removed, reset or set to zero.

Palaeodose. This word refers to a past radiation dose. The difficulty I have with current use is that in our work it is not the actual past radiation dose that is determined, but the beta or gamma dose that results in the same luminescence intensity during thermal or optical excitation. In the past, the term equivalent dose was introduced to deal with this difference. The introduction of the term was necessary because there can be a large difference between the actual radiation dose and the equivalent dose. This difference arises mainly because alpha particles produce different effects than do betas or gammas on a per unit-of-absorbed-energy basis. Someone reading our literature could easily be misled into thinking that palaeodose meant the actual radiation dose. For this reason I think we should be using the term palaeodose equivalent, Pe or Peq. For the same reason we should not be using the term 'dose rate' but be using 'equivalent dose rate', since what we evaluate is not the true dose rate.

Hz and Bq. Herz (Hz) is the unit for the frequency of a periodic signal. This is one that repeats on a regular basis, such as the sound of a tuning fork or the electrical voltage at a wall socket in a house. 1 Hz is 1 cycle per second. Becquerel (Bq) is the SI unit of activity of a radioactive source. The decays occur randomly, not periodically, in time. An activity of 1 Bq means the decays occur at an average rate of 1 per second. It is not appropriate to use either of these for the rate at which photons are emitted from a sample, or the rate at which photons are counted. Photon emission is a random process, not periodic, thus precluding the use of Hz. It is not radioactive decay, thus precluding the use of Bq. Hence one should use counts per second or something similar as the unit for the photon count rate.

D.J.Huntley
Physics Dept.,
Simon Fraser University,
Burnaby, B. C., V5A 1S6,
Canada

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