An automated sample changer for Bruker ESR spectrometers

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***Abstract** : We have developed an automated sample changer which allows the measurement of up to 40 samples without operator attendance. It had not been possible to adapt commercially available robotic arms or auto-loaders. A performance test on enamel samples from a single tooth showed that (i) the changer worked reliably and was not an additional source of error and (ii) when using Fourier transformation to eliminate high frequency noise of the spectra, the uncertainty in the measurement of the ESR intensity was in the range of 1%. Furthermore, there was no difference in the uncertainty of an ESR measurement between aliquots that were weighed as precisely as possible to 40 mg before measurement and subsamples (ranging between about 33 and 50 mg) whose weight was used after measurement for spectrum weight normalisation.*

Equipment

The equipment consists of three major components: (i) a sample holder, (ii) a transfer arm and (iii) a microprocessor control unit. The components were constructed to allow maximum flexibility.

The sample holder is a barrel chain designed to hold the common 5 mm Wilmad quartz tubes. These have been shortened by the manufacturer to 100 mm (which makes them about 30% cheaper than the standard 178 mm tubes). In principle, the chain can be extended to accommodate any number of samples. The transfer arm was constructed from reinforced composite graphite which is non-magnetic, light weight and rigid. The 15 kG magnet of the ANU spectrometer requires a vertical transfer of about 500 mm. The arm can be optimised for any insertion depth. The sample tube is picked up by the use of a vacuum supplied by a small pump. The sample positioning in the cavity can be adjusted with an insertion vial (Figure 1). The system is controlled by a programmable micro-processor unit (Little Star, ZWorld Engineering) which is interfaced with the Bruker spectrometer.

After loading the sample tubes into the holder and switching the unit on, the first sample position is homed-in under the transfer arm. When the system is activated, the arm picks up the first tube and lifts it out of its barrel. The carriage holding the chain swings forward allowing the transfer arm to descend towards the cavity. The arm stops when mechanical resistance occurs. The optimal positioning of the samples in the cavity is ensured by the insertion vial (Figure 1). A scale at the top can be used to adjust the correct height for samples with different dimensions. The sample remains in the cavity until the unit receives a pulse from the spectrometer upon which the sample is lifted up and re-inserted into the holder. Provided another tube is in the next barrel, this tube will be transferred to the cavity. If the next position is empty, the micro-processor will recognise that no vacuum could be successfully applied and the unit will switch to stand-by mode.

Measurement

The timing of a sample change is controlled by the spectrometer. The recording of an ESR spectrum requires two files, the first is saved with the spectrum of the sample, whilst the second one is only used to send a pulse to the sample changer after the measurement has finished (called PULSE in step 2 of the automation routine). Before starting an automation routine, a representative sample is used to tune the spectrometer and work out the best measurement conditions. These will be applied to all following spectra. It seems advisable that the samples of one run have approximately the same volume and spectrum characteristics. The following automation routine will measure 40 samples, save spectra and show the measured spectra on 40 consecutive pages. The first page has been used to determine the measurement parameters.

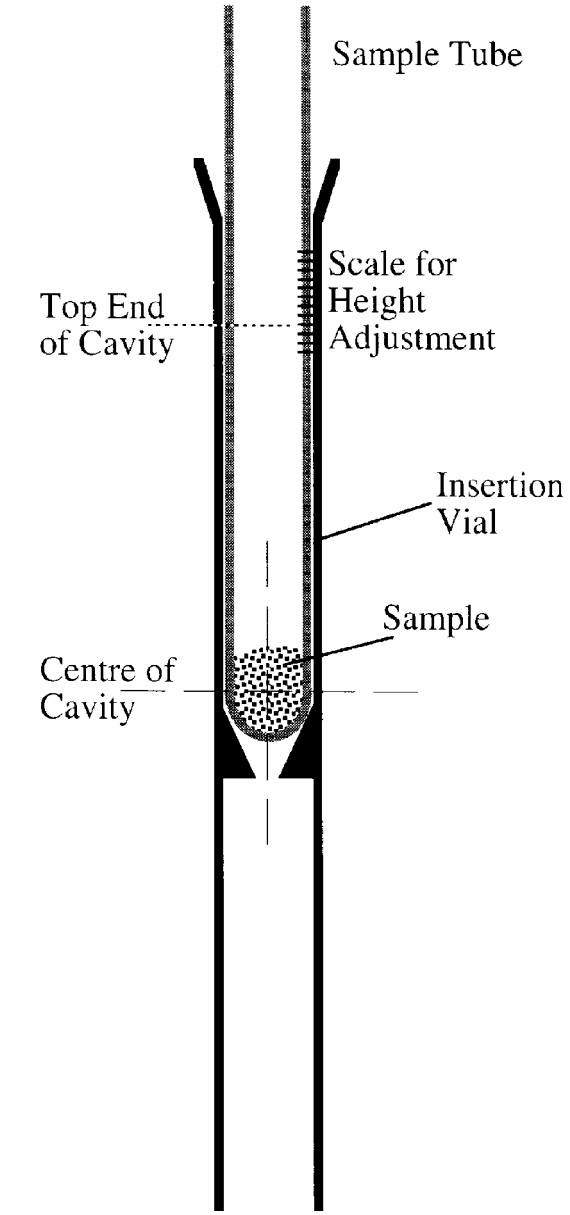

***Figure 1**: Schematic drawing of the insertion vial (not to scale).*

Example of an automation routine:

1> PG INC 1 'increase the page number by 1
2> READP PULSE 'read file that contains the pulse sequence (make sure it is in the selected directory)
3> SETP 'set parameters to page
4> TP INC 1 'copy parameters to next page (for next run)
5> DEL 'delete any previously stored spectra
6> RACQ 'measure (for sending pulse, the recorded spectrum will be over-written in the next run)
7> PG DEC 1 'decrease page number by one
8> SETP 'set parameters to page
9> WAIT 90 s 'time required to remove old sample and insert a new one
10> MTU 'fine tune
11> DEL 'delete any previously recorded spectra
12> RACQ 'measure sample
13> WRITE RUN1 'save recorded spectrum with the nam

When the sample is removed from the cavity, the spectrometer automatically resets the microwave power to 0.44 mW. During an automation routine it is not possible to switch the spectrometer to stand-by mode or to activate a complete auto-tuning cycle for each sample. The fine-tuning (step 10) optimises diode current and frequency. As long as the samples have similar ESR characteristics, the fine-tuning is sufficient to optimise the spectrometer. The sample changer is started during the first wait period. If a position is empty, no further sample will be re-inserted into the cavity. This has the disadvantage that in the subsequent fine tuning step, the spectrometer will be completely mis-tuned. It seems therefore advisable that the loop number (in step 16) corresponds to the number of samples to be measured, in which case the last sample stays in the cavity and the spectrometer remains tuned.

Performance

In order to test the performance of the sample changer, two sample sets were measured. About 3 g of tooth enamel was extracted from a hippopotamus tooth from the archaeological site of Florisbad. Two size fractions were prepared, 250-150 µm and < 150 µm. The first fraction was etched with acetic acid to remove any dust attached to the surfaces of the grains. It was thought that grains would be ideally suited for single aliquot dating (Grün, 1995) because the weight-loss should be minimal during the transfer of the sample from the measuring tube to the vial in which it is irradiated (and vice versa). The second fraction is used routinely for dating in this laboratory. Both sets were divided into 40 subsamples. Aliquots of the grains were weighed as closely as possible to 40 mg with a weighing error in the range of 0.2 mg. The powder samples were in the range of about 33 to 50 mg and were precisely weighed after they were filled into the sample tubes.

The weighing error of these samples is in the range

of < 0.05 mg. The measured powder ESR spectra were subsequently normalised on the weight. The measurement conditions were the ones that are routinely used for enamel samples with relatively large signals: accumulation of 20 scans with 1.015 Gpp modulation amplitude, 10.24 ms conversion factor, 20.48 ms time constant, 2048 bit spectrum resolution (resulting in a total sweep time of 20.972 s), 120 G sweep width and 2 mW microwave power. Each set was measured ten times in the course of about two weeks. The spectrometer had to be turned off several times between the runs. No standard was measured. As shown in Figure 2A, the spectra were quite noisy. In order to address this problem, the spectra were evaluated in two ways:

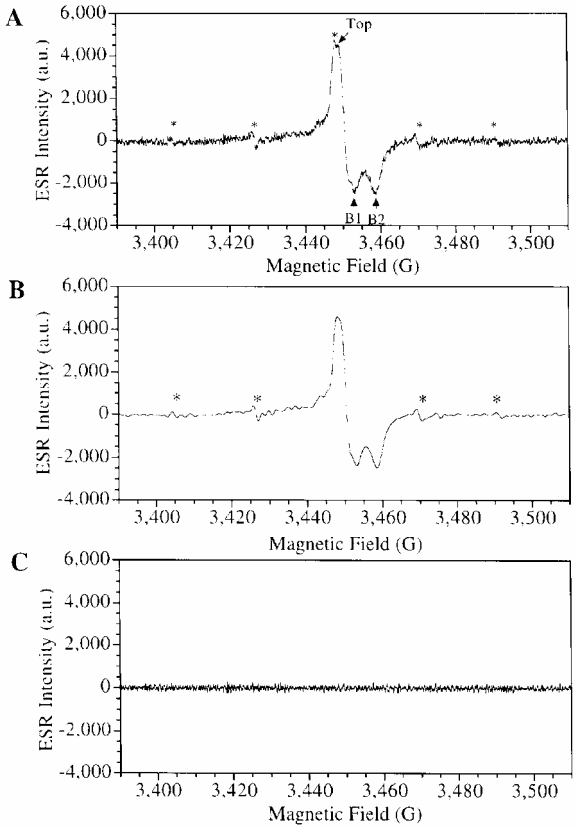

Figure 2
A: Raw spectrum of a grain sample. The asterisks indicate a quintet that has been ascribed to dimethyl (Bouchez et al. 1988).
B:Fourier-transform manipulated spectrum. Note that the split at the top of the wide signal has disappeared whilst the other four signals are still clearly resolved.
C: High frequencies that were stripped off the raw spectrum by the Fourier transformation procedure.

1) the raw spectra were manually evaluated (by setting markers and retrieving the corresponding intensities from the software of the spectrometer) from the top of the wide peak (as indicated in Figure 2A) to the base of the first trough (Top-B1 in Figure 3) as well as to the base of the second trough (Top-B2);
2) the spectra were Fourier transformed, the higher frequencies zeroed and the lower frequencies back-transformed. Figure 2B shows the manipulated spectrum. The peak intensities were then automatically evaluated by the software of the spectrometer.

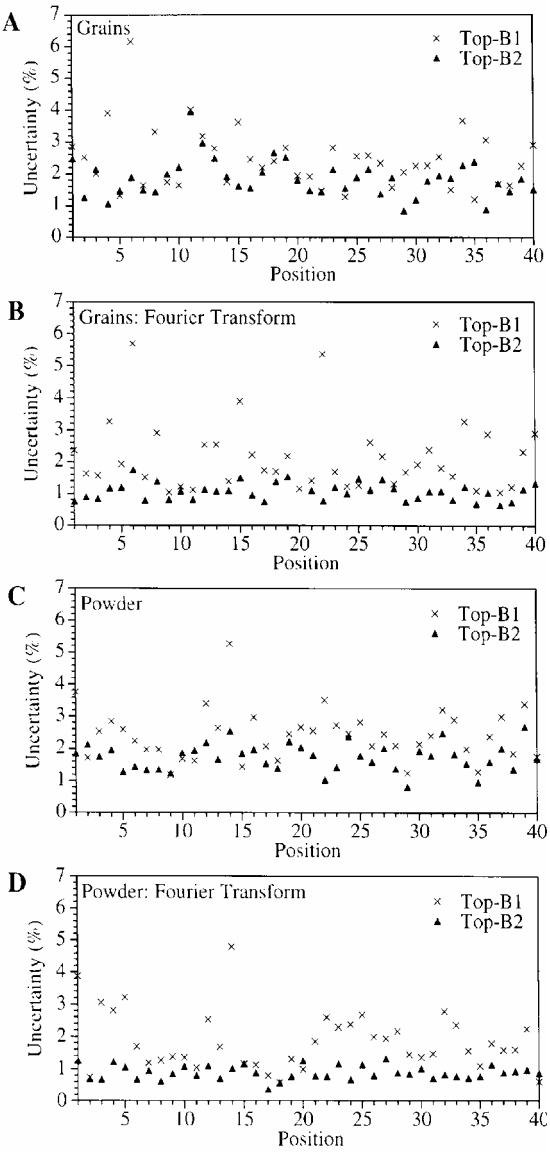

Figure 3*: Uncertainties involved in the repeated measurement of a specific sample. The two signal heights (Top-B1 and Top-B2) are indicated in Figure 2A.*

Figure 2C shows the higher frequencies that were stripped off the raw spectrum. This spectrum does not seem to contain any distinctive peaks. One particular advantage of the Fourier transform, henceforth FT, manipulation was that the dip at the top of the wide peak disappeared giving a more straight forward definition of the top-intensity. The other lines which are thought to be part of the same quintet (marked with asterisks) were smoothed but still resolved (compare Figure 2A and 2B). The difference between the average measured peak intensities of the raw and the FT manipulated spectra was less than 1%. The uncertainty values discussed below are the standard deviations expressed as percentage of the mean.

The performance test was used to address three questions:

1) Determination of the uncertainty of an ESR measurement of a particular sample. This has bearing on the estimation of the number of data points and distribution of dose steps required for the establishment of satisfactory dose response curves (Grün and Rhodes 1991, 1992) as well as the assessment of errors in dose determination (Grün and Brumby, 1994).
2) Determination of the over-all uncertainty of a run of the sample changer. This can be used to decide whether subsamples used for a dose response curve can be measured in the same run or are better measured in the same sample tube in subsequent runs.

3) Is there is a difference between weighing out aliquots before measurement and post-measurement spectrum normalisation? This can be used for minimising the efforts in sample preparation.

Figure 3 shows the uncertainties of the ten repeated measurements of each sample. These uncertainties arose from random measurement errors, equipment stability and positioning of the sample tube in the cavity. It is noteworthy that the signal intensity Top-B1 (crosses in Figure 3) showed nearly always larger uncertainties than the signal intensity Top-B2. The raw spectra of the grains and powders (Figure 3A and C) had uncertainties in the range 2 to 4%, whilst the FT manipulated spectra of the two sets showed uncertainties of around 2% for Top-B1 and about 1% for Top-B2.

Ten repeated measurements of a single sample without removing it from the cavity resulted in uncertainties of around 2% for the raw spectra. The uncertainty of the FT manipulated spectra Top-B2 was 0.89% which falls well into the respective ranges of the grains (1.07±0.27%) and powders (0.88±0.21%). Apart from the absence of mechanically removing the sample, the measurements of this sample were carried out within about two hours, minimising potential long term stability problems. One can conclude from these data that sample changing over longer periods of time did not introduce any significant errors.

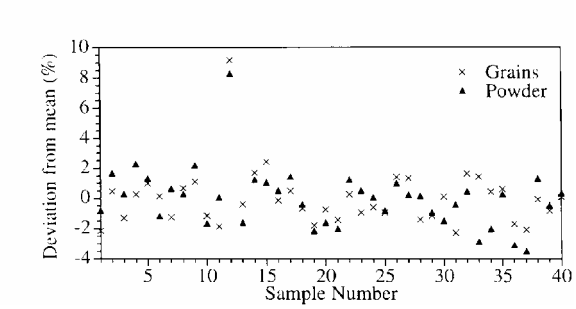

***Figure 4**: Deviation of the mean intensity at a particular position from the mean of all measurements.*

Additional sources for the uncertainty of the 40 measurements of a run were weighing errors, sample inhomogeneity and differences in glassware. Figure 4 shows the deviation of the mean intensity at each of the 40 positions from the mean of all measurements. Surprisingly, position 12 showed a very large increase of the intensity for both grains and powder. This was most probably due to the characteristics of the sample tube. The other positions showed random scattering in the range of ± 2%. Sample position 12 was excluded from the following evaluation. The uncertainties for a complete run were for the grains: 2.84±0.43% (Top-B1) and 2.15±0.32% (Top-B2) for the raw spectrum evaluation and 2.76±0.54% (Top-B1) and 1.55±0.15% (Top-B2) for the FT manipulated spectra. The values for the powder set were for raw spectrum evaluation: 3.37±.49 (Top-B1) and 2.24±0.28% (Top-B2); FT manipulated spectra: 3.05±0.39% (Top-B1) and 1.70±0.27% (Top-B2).

The uncertainties of the intensity measurement of complete runs (FT manipulated spectra, Top-B2) are nearly twice as high as the uncertainties of the repeated measurement of specific samples. This implies that subsamples used for the construction of a dose response curve ought to be measured in the same sample tube. Furthermore, single aliquot measurements minimise the problem of sample

inhomogeneity. However, so far it has not been demonstrated that the single aliquot technique is applicable to materials other than enamel.

The basic difference between the two sample sets is that the ESR spectra of the grains did not have to be weight-normalised. The uncertainties for the complete runs showed no quantifiable differences between the grain aliquots and the powders. Because precise weighing of aliquots requires more time and concentration than the precise weighing of subsamples whose absolute weight may scatter by up to about 15%, it seems preferable to use post-measurement weight normalisation.

It was thought that grains were particularly suitable for single aliquot measurements (Grün 1995) as weight-loss between measurements ought to be negligible. However, electrostatic problems made complete recovery impossible and up to 10% of the samples were lost. The weight-loss of powders can be kept below 1%.

Conclusions

The sample changer allows the measurement of up to 40 samples without operator attendance. Performance tests showed that the changing process introduced negligible uncertainties that were not quantifiable. The performance tests also showed that:

- the uncertainties in the ESR intensity estimation of relatively noisy spectra of tooth enamel were in the range of about 2%;
- FT manipulation could be successfully applied for the reduction spectrum noise and optimisation of the reproducibility, the uncertainties improving to about 1%;
- inhomogeneity of the samples and differences in the properties of the sample tubes introduced additional errors of about 1.3% which means that a single aliquot technique and measurement in the same sample tube ought to give best results for the construction of dose response curves;
- weight normalisation of spectra did not introduce additional errors for samples with weights between about 33 to 50 mg compared to pre-weighed 40 mg aliquots;
- it is more difficult to recover grains than powders from the sample tubes due to electrostatic problems.

Acknowledgment

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Reviewer
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Procedures used for optically and Infrared Stimulated Luminescence Dating of Sediments in Heidelberg

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Abstract : *This paper describes the procedures used for Optically Stimulated Luminescence (OSL) and Infrared Stimulated Luminescence (IRSL) in sediment dating at the Forschungsstelle Archäometrie in Heidelberg, Germany. The topics discussed include sampling, sample preparation, luminescence measurement, equivalent dose estimation and determination of dose rate.*

Introduction

Since the findings of Huntley *et al.* (1985) and Hütt *et al.* (1988) OSL and IRSL dating has been frequently applied and has become a method of high demand in geoscientific research of late Quaternary sediments. Nevertheless, some methodical problems involving experimental parameters, such as laboratory illumination, mineral separation, preheat procedures, stimulation and detection wavelengths, equivalent-dose (D_E) determination and dose rate determination, are still to be optimised and are subject to lively discussions and controversies. Depending on the combination of these parameters significantly different age results may be obtained.

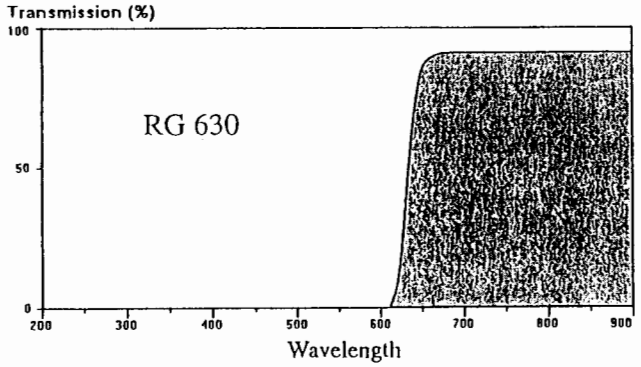
One of our research topics is to find out for which types of sediments optical dating may be applied successfully. In order to test the hypothesis that most waterlain sediments from Middle Europe can be dated, we use an empirical approach, i. e., dating of sediments for which independent age control is available (Lang and Wagner, 1997). This approach requires the specification of the experimental procedures applied. In this paper, we describe the techniques used for OSL and IRSL dating of sediments at our laboratory, particularly sampling, sample preparation, luminescence measurement, equivalent dose estimation and determination of dose rate, carried out on aeolian, alluvial, colluvial, limnic and littoral sediments with expected ages of less than 50 ka. Discussion of dating techniques may be found in Aitken (1992,1994), Wintle (1994), Mejdahl and Christiansen (1994) or Lang and Wagner (1996).

Sampling and sample preparation

In the field, sediments from outcrops are sampled with steel cylinders (100 cm^3, 200 cm^3 or 500 cm^3), which are hammered into the sediment. When recovering, both ends of the cylinder are immediately covered tightly with light-proof plastic lids. An additional sample of the sediment is watertight sealed in a plastic bag for determination of the actual interstitial water content and for radiometric analysis. In the laboratory's dark room, both ends of the sediment core, which may have been exposed to light, are removed. The sediment remaining in the steel cylinder is processed for luminescence analysis. If outcrops are absent, sampling is carried out by means of bore-cores. The core is split in half in the dark room and samples are taken from the inner part of the core. From fine grained sediments a minimum of 200 g is collected and for coarser grained sediments a minimum of 500 g.

Special attention is paid to optimal laboratory illumination. All lamps (halogen lamps with optical filters of 5 cm diameter as front window) are equipped with dimmers and are never focused directly onto the sample. Hence, during handling the samples are exposed at most to diffused light with very low intensity. Otherwise, the samples are strictly protected from light. During the separation procedure of quartz, red light of > 630 nm (Fig. 1) is used. The preparation of coarse grained feldspars and fine grained polymineral fractions is carried out under green-yellow light of wavelengths between ~ 500 and 600 nm (Fig. 1). This allows for minimum signal loss but high sensitivity of the human eye

OSL



IRSL

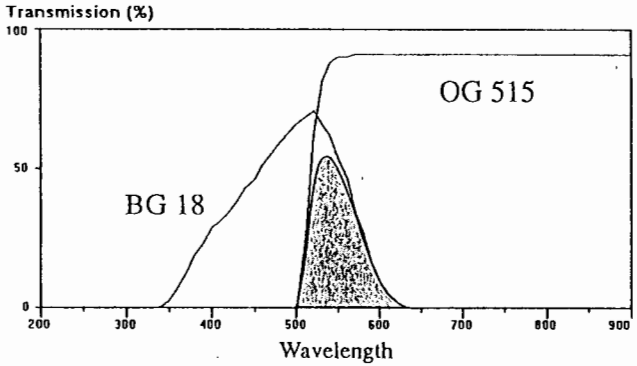
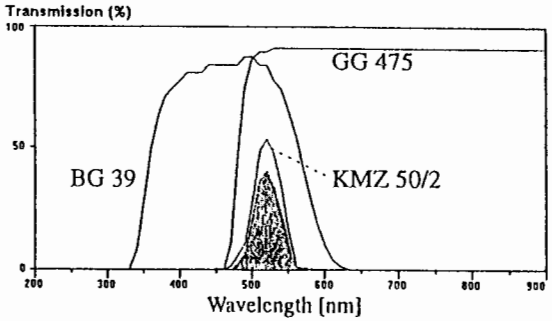


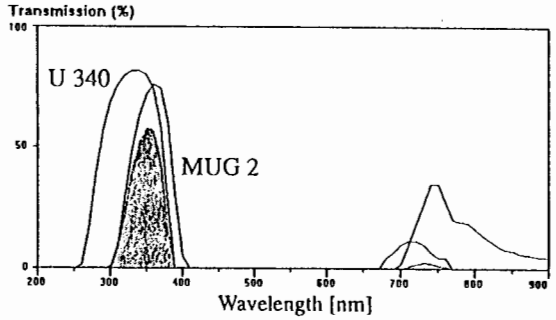
Figure 1 *Wavelength regions used for laboratory illumination. Transmission in % is plotted versus wavelength for the filters put in front of the lamps during sample preparation for IRSL and OSL measurements. The total transmission of the filter combinations is marked as shaded area. Filter used for OSL sample preparation: RG 630. Filters used for IRSL sample preparation: BG 18 and OG 515 (Appendix A).*

OSL

Stimulation:



Detection:



IRSL

Detection:

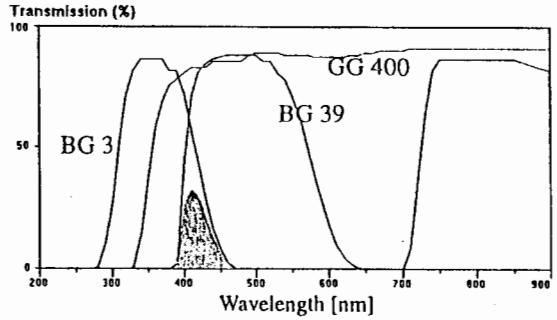


Figure 2 *Wavelength regions used for stimulation and detection. Transmission in % is plotted versus wavelength for the filters used for stimulation and detection of OSL (stimulation: BG 39-, GG 475- and KMZ 50/2 filters; detection: MUG 2- and U 340 filters) and for detection of IRSL (BG 39, 2 BG 3 and GG 400 filters) (Krbetscheck, et al., 1996). The total transmission of the filter combinations is marked as shaded area (Appendix A).*

when adapted to darkness (Wiggenhorn, 1995; Appendix B).

To prepare polymineral fine grains (4-11μm), the sample is treated with H_2O_2, then sieved and settled in 0.01 n NH_4OH. Subsequently, the sample is treated with HCl (10%), rinsed several times and dispersed in acetone. Finally, between 80 and 100 aliquots of each sample are prepared by settling in acetone on aluminium discs. Each disc carries equally 1 to 2 mg of silt. For preparing coarse grains, the preferred 90 to 125 μm fraction (if not available then 90 to 200 μm or 200 to 315 μm) is achieved by sieving. The sieved grains are treated with H_2O_2 and HCl. The quartz (using densities of 2.62 and 2.75 g/cm^3) or a K-feldspar-rich fraction (2.53 to 2.58 g/cm^3) are obtained by density separation. The quartz fraction is etched in 40% HF for 40 min and subsequently treated with HCl. As for the fine grain aliquots, 80 to 100 aliquots are prepared but in this case the weight is 2 to 4 mg. Steel discs or rhodium plated bronze cups are used as sample carriers and the grains are fixed on the discs with silicon spray. Because of the different response of quartz and feldspar to the wavelengths of the laboratory light it is advisable to obtain both mineral fractions in seperate sieving runs.

Equivalent-dose (D_E) determination

Routinely, the multiple aliquot additive dose methpd is used. Irradiation is carried out at room temperature using $^{90}Sr/^{90}Y$-β- and ^{241}Am-α-sources. 5 β-irradiator systems are available (dose rates between ~ 1 and ~ 10 Gy/min). Feldspar and polymineral aliquots are stored after irradiation for at least 4 weeks at room temperature. Quartz samples are stored at least 1 week at room temperature. Preheating of 220°C for 5 min (Smith *et al.*, 1986; Godfrey-Smith, 1994; Lang, 1996) is usually carried out on all samples. For determination of the growth curve 10 'natural' discs and 6 groups of artificially dosed discs (5 each) are measured. Additive doses are chosen subsequent to initial tests of the sample's dose response, in order to obtain at least a threefold increase in luminescence intensity.

Shine-down curves of 60 s are measured at room temperature using a 'Risø reader TL-DA-12' (Bøtter Jensen *et al.* 1991) equipped with an EMI 9635Q photomultiplier and a Xe-lamp (Kuhn, 1993). The light of the Xe-lamp filtered by a combination of BG 39-, GG 475- and KMZ 50/2 filters (transmission 500 - 550 nm, Fig. 2, Appendix A) is used for OSL stimulation of quartz. The OSL is detected with MUG 2- and U 340 filters (transmission 300 - 380 nm, Fig. 2, Appendix A). TEMT 484 diodes are used for IR stimulation (880 Δ 80 nm) of feldspars and IRSL is detected with a filter combination of BG 39, 2 BG 3 and GG 400 (transmission 390 - 450 nm, Fig. 2, Appendix A) (Krbetscheck, *et al.*, 1996). By restricting the detection to the 410 nm IRSL emission it is ensured that the sampled signal is more stable than the signal sampled by the broad detection window of a BG 39 filter only (Lang and Wagner, 1996).

Data handling is done using G. Duller's ANALYSE software. R. Grün's SIMPLEX ROUTINE is used for D_E calculation applying error weighting proportional to variance. For IRSL analysis, the subtraction technique is applied (Aitken and Xie, 1992), using the mean IRSL-intensity of the interval 50 s to 60 s as the 'late light'. This is necessary because the 410 nm emission seems to have a dose dependent hard-to-bleach-component (Lang and Wagner, 1997).

Fading tests are carried out for all polymineral fine grain and potassium-feldspar-rich fractions. An additional set of natural and maximum dosed subsamples, which have been irradiated and preheated contemporary to the discs used for the growth curve fixing, are measured after 3 months storage. The ratios of 'natural' intensities to 'artificially irradiated' intensities for the first and second measurement are compared to check for fading.

Dose rate determination

α-counting (Littlemore ELSEC 7286), β-counting (Risø GM-25-5) and low-level-γ-spectrometry (Ge-detectors) are routinely applied. Whenever possible, *in situ*-measurements are carried out using portable γ-spectrometry (NaI-detectors). α- and β-dose rates are calculated from α- and β-counting results respectively and from the U-, Th- and K-contents determined with low-level-γ-spectrometry and using the dose conversion factors given by Nambi and Aitken (1986). Although α- and β-counting are more precise than low-level-γ-spectrometry, systematic errors such as under- or overcounting may occur. Therefore, α- and β-counting are only used for age calculation if they do not differ significantly from the γ-spectrometric results. The α-dose rate for fine grains is calculated using the a-value system (Aitken and Bowman, 1975). The external α-dose rate for coarse grained feldspars is estimated as discussed in Appendix C. β-counting is used to determine the K-content of the potassium rich feldspar fraction. β-attenuation factors for dose rate determination of coarse grains are taken from Mejdahl (1979). To check for radioactive equilibrium, low-level-γ-spectrometry is used. If no *in situ*-measurements are

available, the γ-dose rate is calculated from the radionuclide contents derived from low-level-γ-spectrometry. Additionally, these radionuclide contents are used for modelling the dose rate if the sample exhibits radioactive disequilibrium. The contribution of cosmic rays to the dose rate is calculated following Prescott and Hutton (1994). All errors quoted are standard errors. Errors are calculated using the Gaussian error propagation law.

Conclusions

A large number of sediments ranging from aeolian sands, loess, limnic sediments, salt marshes, some fluvial sands to colluvial sediments and anthropogenic deposits have been dated successfully using the procedures described. The ages obtained range from less then 200 a up to 70 000 a with precisions from 7% to 13%.

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Appendix A: optical filters

All optical filters mentioned are 3 mm thick, except the KMZ 50-2 which is 4 mm thick. They are all produced by Schott except the U 340 which is supplied from Hoya.

Addresses:

Schott Glaswerke, Hattenbergstr. 10, D-55122 Mainz, Germany.

Hoya Corporation, optics sales division, 3-3-1 Musashino, Akishima-Shi, Tokyo, 196, Japan.

Appendix B: green-yellow light for IRSL sample preparation

The use of wavelength shorter than red for IRSL sample preparation goes back to personal communications with N. A. Spooner, G. Hütt and U. Rieser. Consequently, Wiggenhorn (1995) weighted the curve of spectral efficiency of the human eye (Gobrecht, 1977) with the spectral curves of energy required for bleaching the blue IRSL-emission of different types of feldspars (microcline and sanidine) as given by Spooner (1993 and 1994). The calculated curves show minimum signal loss but maximum sensitivity of the human eye when adapted to darkness between 500 and 550 nm, depending on feldspar type. The visibility (especially for contrasts) is higher than it is under longer wavelength. Consequently, sample handling can be carried out under fairly low light intensity. Following Wiggenhorn (1995) the laboratory lamps were equipped with dimmers and the filter combination shown in Fig.1. We checked for potential occurrence of accidental bleaching: Sample aliquots were split in two groups: Group 1 aliquots accompanied samples in preparation during the whole preparation procedures. Group 2 aliquots were kept in the dark. After finishing the sample preparation, the mean IRSL-intensities of the two groups were compared. In between statistical errors (3% to 6%) no signal loss occurred. The test is repeated from time to time.

Appendix C: external α-dose rate for coarse grained feldspars

Contrary to quartz, the surface of a feldspar grain is not regularly etched by HF (Duller, 1992). Etching occurs preferentially along crystal plains. Therefore, it is unlikely that the outer α-irradiated layer will be removed totally during etching. The luminescence signal of a HF-treated feldspar grain may still contain luminescence induced by α-particles - an amount which can hardly be determined. We think that estimating the external α-dose rate is more precise, even when the estimate is only rough.

For this rough estimate the percentage (x) of a grain's volume influenced by α-particles is calculated, assuming spherical grains, homogeneous irradiation of the grains, an α-penetration depth of 10 ± 5 μm and using the mean diameter of the grain size fraction separated. The α-dose rate for x is calculated as for fine grains, using an a-value of 0.1 ± 0.05. This procedure has the advantage that as soon as a precise method is available, the α-dose rate can be recalculated.

Reviewers

Martin Aitken, Rainer Grün

Comments

This detailed account of laboratory procedures is greatly welcomed; it is in line with one of the original objectives of Ancient TL. My only comment is in respect of Appendix C where the optimistic impression is given that there are no hidden problems associated with the HF etching of quartz grains. Investigations by Bell & Zimmerman (1978) showed there to be similar uncertainties with quartz grains; this was for grains extracted from baked clay; maybe the situation for grains from sediment is better. The failure of 45 minutes of HF treatment (at 40°C) to eliminate completely the alpha dose from quartz grains has been demonstrated by Valladas & Valladas (1982); they propose that evaluation is to be preferred and describe an appropriate experimental technique.

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M. Aitken

Thesis Abstract

Thesis title : Statistical analysis of thermoluminescence experiments for sedimentary dating.
Author : W.Chandanie Wijayalatha Perera
date : July 1996
degree : Doctor of philosophy - Simon Fraser University
speciality : Mathematics and Statistics.

Abstract:
Sediment (or other buried material) when heated gently glows with light called thermoluminescence. The amount of light given off depends on the material and on the amount of the radiation impinging on the sample while buried. Comparison of the equivalent dose (a known laboratory dose required to produce the same amount of luminescence as the original untreated sample) with historical radiation rates permits estimation of the age (duration of burial) of the sample, a process called thermoluminescence dating.

We study statistical techniques for estimating the equivalent dose from the data collected for thermoluminescence dating. Physical models are used to motivate generalized non-linear models for the data and to justify assumptions about the distribution of errors in these models. Maximun likelihood, quasi-likelihood and least squares estimators are compared by examining their statistical propreties. Formulae are provided for the biases and the mean squared errors of these estimators valid in the limit of small measurement errors.

In thermoluminescence studies, data are collected on a single sample at a series of temperatures. Consequently, observations collected at different temperatures are correlated. We propose a generalized estimating equations procedure for estimating the equivalent dose from the correlated data. Large sample asymptotic properties of the proposed estimate are examined and a formula is provided for estimating the error of the estimate. We propose symmetric confidence intervals for the equivalent dose with a *t* quantile; a formula is provided for the approximate degrees of freedom of the suggested *t* quantile, valid in the limit of small measurement errors. Finite sample performance of the asymptotic results is examined by Monte Carlo.

Test based on the empirical distribution function (EDF tests) of the standardized residuals are proposed for testing the distributional assumptions on the random errors in two situations : without assuming the fitted model is correct and assuming the fitted model is correct. We propose a recurrence formula for evaluating the cumulative distribution function of two fitted standardized residuals needed in the proposed EDF tests. Weak convergence properties of the related empirical processes are examined. Finite sample performance of the suggested EDF tests is examined by Monte Carlo.

Letters

Comment on ' Non linear approach of TL response to dose: polynomial approximation. Ancient TL, 14, 7-14. (1996) Guibert, P., Vartanian, E., Bechtel, F., and Schvoerer, M. '

G. W. Berger
Quaternary Sciences Center - Desert Research Institute - Reno, NV 89506-0220, USA.

The objective reader might be interested to learn of earlier development and application of polynomials to TL dose-response curves than those cited in the recent review and explanatory note by Guibert et al. (1996). These authors provide a useful outline of the data-processing procedures used by the laboratory in Bordeaux, including their version of the "Australian-slide method" (though not cited, as pointed out by the reviewer), but they fail to give proper credit to much earlier, and more original, work by Berger et al. (1987) on the use of both quadratic and cubic polynomials for many of the same purposes (except the slide method). My own development of the use of polynomials grew out of the efforts of Divigalpitiya (1982) to employ quadratic polynomials.

In the review part of their note, Guibert et al. also fail to mention any of the much earlier accurate applications by Berger of quadratic polynomials to both sublinear and supralinear TL dose responses in sediments (e.g., Berger, 1985; Berger and Mahaney, 1990; Berger et al., 1991). Furthermore, they fail to mention any of the much earlier applications of single-saturating exponentials of Berger, and of cubic polynomials (Berger, 1987; Berger et al., 1987). Finally, they fail to mention the earlier description and application of the single-saturating-exponential-plus-linear function, with error analysis (Berger, 1990, 1991), developed contemporaneously with the different approach of Grün (1990). Such shortcomings in accreditation of at least representative prior scientific literature are not appropriate in scientific publication.

In closing, the use of polynomials (especially of order three or higher) was discouraged by Berger et al. (1987), precisely because they seem at present to have no apparent basis in physical processes underlying luminescence dose responses, at least for sublinear responses. The introductory rationalizations of Guibert et al. (page 9) for their use of polynomials may therefore be questioned. For example, there appears to be no practical need to have a single function for representing all dose-response regions simultaneously. If the dose-response curve is saturating, then the error introduced by ignoring any low-dose departure from an exponential (or exponential-plus-line) model appears to be insignificant (as has been demonstrated in citations above). On the other hand, for very young samples (e.g., archeological material or geological samples less than 1 ka), there appears to be no need to reconstruct the high-dose region. For this reason, with such "young" samples, second-order polynomials appear to be acceptable under certain empirical conditions (e.g., relatively small extrapolations, or for supralinear fits), as demonstrated in the citations above (and others).

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Notices

Cheltenham Geochronology Laboratories

The Centre of Environmental Change & Quaternary Research at the Department of Geography & Geology invites you to the opening of the Geochronology Laboratories, to be held at Francis Close Hall (wine reception and presentation of the laboratories) 26 March 1997. Afterwards there will be a lecture by Dr. Manfred Frechen at The Park:.

"The Terrestrial Record of the Last Interglacial/Glacial Cycle in Eurasia"

This lecture will include a discussion of state of the art TL and OSL dating methods and their application to the dating of eolian sedimentary sequences in Europe and Asia.
For further information, please contact:

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Centre for Environmental Change & Quaternary Research
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FIRST ANNOUNCEMENT AND CALL FOR PAPERS
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Gliwice, Poland

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SCOPE AND OBJECTIVES

The Institute of Physics of the Silesian Technical University invites you to the 3rd International Symposium on Luminescent Detectors and Transformers of Ionizing Radiation, LUMDETR '97, to be held in Ustron, Poland, 6-10th October, 1997.

The 3rd International Symposium continuous traditions of conferences held first in the Soviet Union and since 1991 as an international symposium held in Baltic states: Latvia and Estonia.

LUMDETR '97 will bring together specialists around the world in the fields of luminescence processes, physics and chemistry of luminescent phosphors, dosimetry and transformation of ionizing radiation.

Opening lectures read by invited scholars will introduce the main topics of the symposium. Most of the time will be reserved for the scientific and technical exchange on current work in the form of oral presentations. The poster presentations will be briefly introduced by the authors at the beginning of poster sessions.

Oral and poster sessions during the Symposium will be devoted to the following topics:

- fundamental problems of radioluminescence, photo- and thermoluminescence processes in solids,
- physics and chemistry of luminescent materials used for detectors and transformers of ionizing radiation,
- scintillators, dosimeters and transformers of ionizing radiation and their application,
- natural luminescent minerals and their applications in dosimetry and paleodosimetry

SYMPOSIUM ORGANIZER : Andrzej Bluszcz

CONFERENCE SECRETARIAT

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