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The luminescence dating laboratory at the University of Bonn: equipment and procedures

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Abstract: This paper documents the facilities and procedures used for luminescence dating in the laboratory of the Department of Geography at the University of Bonn. We focus on optically stimulated luminescence (OSL) dating of fine-sand quartz samples and infrared stimulated luminescence (IR-OSL) dating of polymineral fine-silt samples.

Introduction

The luminescence dating laboratory at the University of Bonn is situated in the Department of Geography where it is part of the working group focusing on geomorphology and Quaternary geology. The major task of the laboratory is to provide age data for internal research projects and to contribute to luminescence dating research. Moreover, it is expected to conduct dating for external research projects. The laboratory is able to carry out dating of aeolian deposits, fluvial and colluvial deposits and coastal deposits using fine-sand quartz and feldspar grains (100-250 µm, commonly called "coarse grained") or polymineral fine-silt (4-11 µm, commonly called "fine grained") extracted from the sample.

A number of laboratories with similar capacities and objectives exist. Essential procedures used in these laboratories are mostly reported together with the scientific results. It is the purpose of this paper to present the facilities, equipment, essential parameters, sample procedures, tests for D_e reliability and assessments of uncertainties used in our laboratory and in doing so, to push forward standardisation in luminescence dating.

Facilities and equipment

Laboratory light

Quartz samples are treated under subdued yellow or red light. Yellow light is provided by a low pressure Na-lamp (Osram MW 18W, monochromatic sodium doublet at 589 nm, Spooner et al., 2000) filtered with a Schott OG530/3mm filter to cut the weak blue emission of the lamp. Red light is provided by red light emitting diodes (LED type Siemens LS5421-Q) filtered with a Hoya R62/3mm filter (Figure 1, Schilles, 1998). Feldspar and fine-silt samples are treated under subdued yellow or green light; the latter

is provided by one LED (type Nichia NSPG-500) filtered with a Schott OG 515 filter (Figure 1, Schilles, 1998). Routinely, light does not directly fall onto the sample being prepared. All light sources were tested using two fine-silt loess samples from Germany and Pakistan (laboratory codes: BN181 and BN80, respectively, prepared as indicated in section 3), and a fine-sand quartz sample from the Sahara (laboratory code BN202, prepared as indicated in section 3). The quartz sample was subsequently manually powdered to grains < 15 µm and was settled onto aluminium discs. In Figure 2 the results of the tests of the LED-lamps and the Na-lamp are shown: none of the light sources are able to bleach the sample within 20h.

Beta-sources

The laboratory has two beta sources at its disposal:
1) a stand-alone Amersham $^{90}Sr/^{90}Y$ β-source (3.7 GBq) mounted in a Littlemore device delivering 7.5±0.4 Gy min^{-1} (calibrated on "date", i.e. value related to the calibration datum) to fine-silt sample material on 5 mm thick aluminium discs.
2) a Amersham $^{90}Sr/^{90}Y$ β-source (1.5 GBq) mounted in the luminescence reader delivering 5.9±0.09 Gy min^{-1} (calibrated on "date") to 90-300 µm quartz grains mounted on 5 mm thick stainless steel discs. The strength of the source can be reduced to a minimum dose rate of ~0.76 Gy min^{-1} by fitting aluminium discs of different thickness underneath the source.

Both β-sources were calibrated using loess and quartz material respectively, irradiated by the ^{137}Cs γ-source of the Risø Laboratory (Denmark). This γ-source delivers an accurately known dose rate of about 0.10 Gy h^{-1} in air (A.S. Murray, pers. com.). The calibration dose of the quartz sample is 4.59±0.07 Gy, that of the loess sample is 8.51±0.13 Gy.
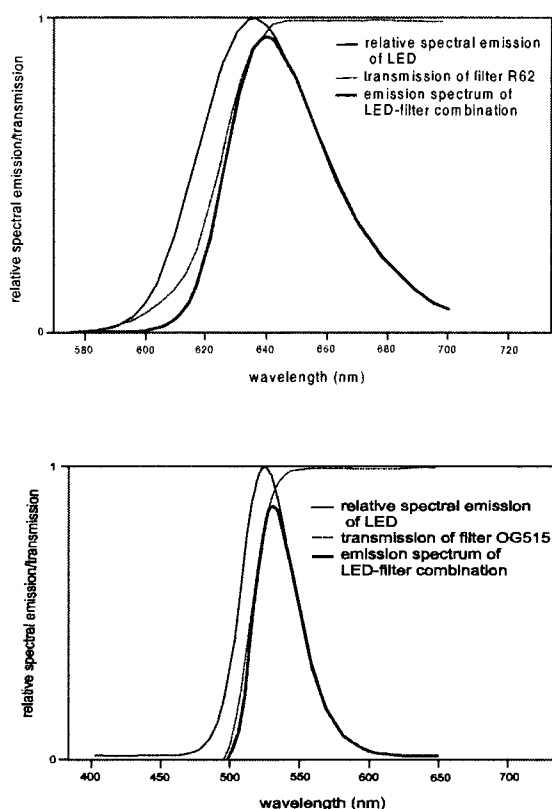
Figure 1.

Spectral curves of LED laboratory light (Schilles, 1998). A: red LEDs; B: green-yellow LEDs

Alpha-sources

Six ^{241}Am sources (3.7 MeV) are mounted in a self-made unit controlled by a self-made electronic box. The sources have been calibrated using TLD 200 and cross-checked with α-sources of the same type in the Forschungsstelle Archaeometrie at Heidelberg. The mean source strength was 0.2018 µm^{-2} min which resulted in 2.62 Gy min^{-1} being delivered to fine-silt (4-11 µm) TLD 200 deposited on aluminium discs as a monolayer (Bürgi, 1992). Irradiation is administered at 10 mm distance to the discs under vacuum (10^{-4} mbar).

Luminescence readers

1) Daybreak 1100 automated TL/IR-OSL System equipped with 10 IR-LEDs (845Δ20 nm), and a 300 W Xe arc lamp. This is used for fine-silt sediments.

2) Risø automated TL/OSL System, DA-15 equipped with a β-source, a 1W IR-laser diode (830Δ20 nm, delivering ~250 mW cm^{-2}) and 42 blue LEDs (470□20 nm, delivering ~50 mW cm^{-2} when running at full power). This is used for sand-sized quartz and feldspars.

Gamma-spectrometer

The low level γ-spectrometer is characterised by the following data:

- DSG coaxial HPGe detector, n-type
- Detector size: 58.2 mm diameter and 57.8 mm high
- Entrance window: Mylar, at ~5mm distance from the Ge-crystal
- Energy resolution: FWHM at 122 keV : 0.87 keV, FWHM at 133 MeV: 1.86 keV
- Efficiency: 33.2%
- Peak to Compton: 58.4:1.0

The detector is shielded with 5 mm perspex, 15 mm copper and 15cm low activity Pb. A perspex bar guarantees exact positioning of the sample and the reference material onto the detector (Figure 3).

Alpha-counter

The laboratory has a Littlemore Low Level Alpha Counter 7286 equipped with 2 photomultiplier tubes (EMI type 6097). This device is based on scintillator techniques where light is produced by an α-particle interacting with a zinc sulphide screen. The light is collected by a photomultiplier tube.

Beta-counter

Kind collaboration allows us to use occasionally the β-counter in the Department of Geography at the University of Cologne. The equipment is a Risø low level GM 25-5 multicounter system equipped with five individual sample counters and a guard counter. The device is based on Geiger-Müller counting and anticoincidence techniques to reduce the cosmic ray background. Reference material is loess material (Table 1) for external beta dose determination and KCl granulat for internal beta dose determination.

Sample preparation

Fine-silt

The fine-silt preparation aims to separate 4 – 11 µm grains from the sample. The following steps are carried out:

1) sieving of the fraction < 63 µm; 2) H_2O_2 treatment to remove organic material starting with H_2O_2 10% in order to control heating of the sample and continuing with H_2O_2 35% after a weak alkaline pH (~8) is produced using NaOH-liquid; 3) settling in 0.01 N NH_3 in Atterberg cylinders to remove grains > 11 µm and grains < 4 µm using Stokes' Law. If the sample is rich in clay, it is shacken for around 12h before the separation process is started. The settling process is repeated as often as necessary to guarantee the completeness of the grain size separation; 4) dissolution of carbonate using CH_3COOH 20%; 5) settling the sample onto aluminium discs using the material suspended in water. During all preparation steps in which acids or alkaline solutions are involved, the pH is continuously monitored.
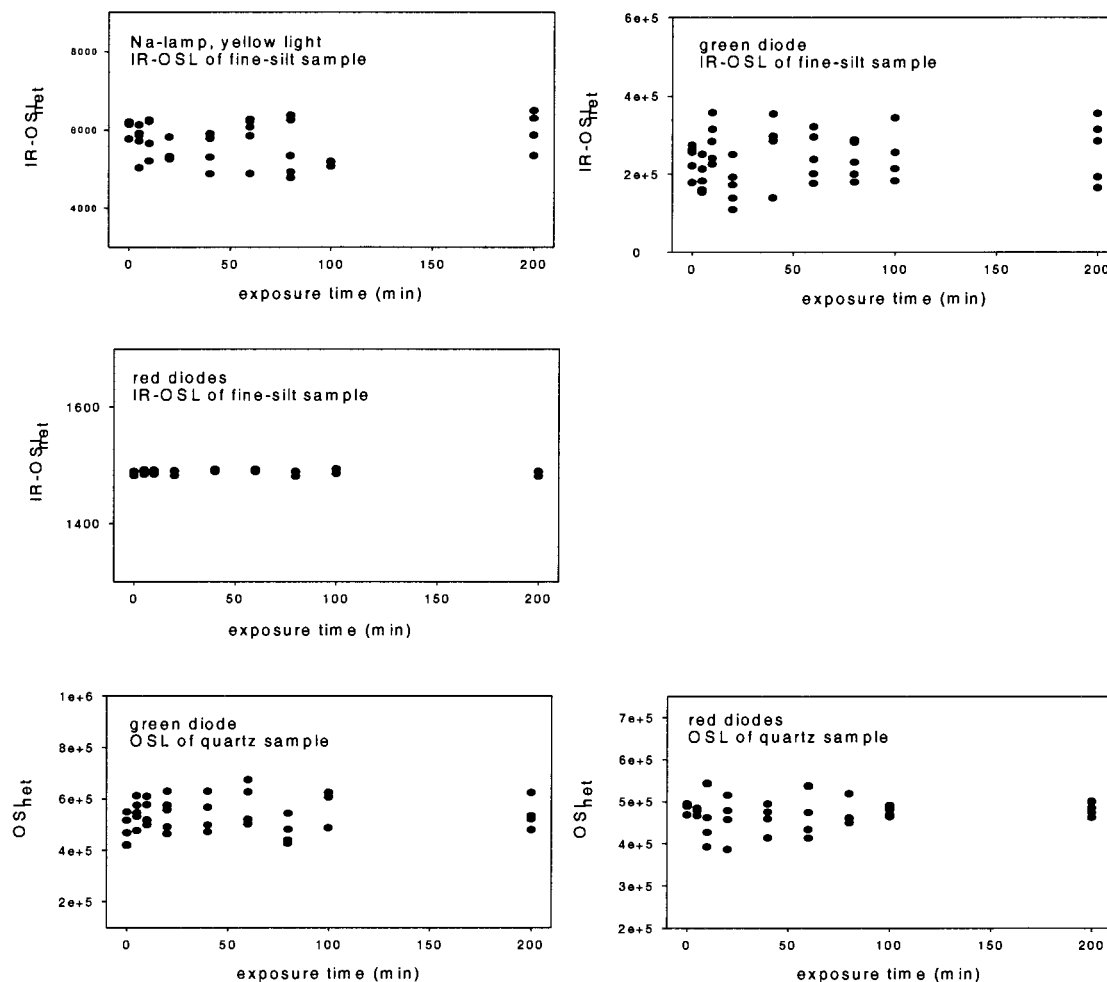
Figure 2.
Test of laboratory light sources. $IR\text{-}OSL_{net}$ = signal integral of the first 10 s subtracted from the background; OSL_{net} = signal integral of the first 1.2 s subtracted from the background. Loess samples: normalisation using 0.1 s natural IR-OSL before exposure to light; 60 s IR-OSL of the natural dose; Quartz sample: 46 Gy was added to the natural dose by laboratory irradiation, followed by a preheat of 260°C for 10 s and 0.1 s OSL before exposure to laboratory light. Distance between light source and sample and intensity of light was chosen according to routine working circumstances (~50 cm distance).
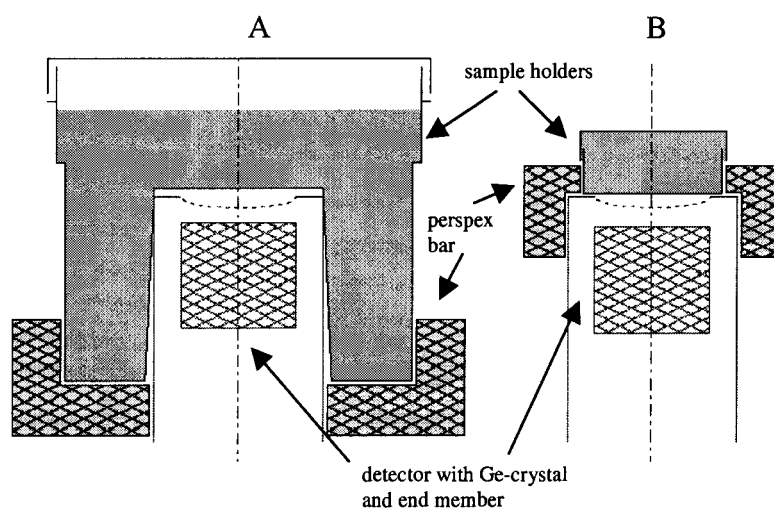

Figure 3.
Design of the γ-spectrometer detection unit. A Marinelli beaker, B plastic sample holder

Fine-sand

This procedure aims to separate a monomineralic quartz or K-rich feldspar subsample in the grain size 100 – 250 µm. The following steps are carried out:
1) sieving out the fine sand fraction according to the grain size composition of the sample. We prefer restricted fractions (e.g. 160-200 µm); 2) H_2O_2 treatment to remove organic material starting with H_2O_2 10% in order to control heating of the sample and continuing with H_2O_2 35% after a weak alkaline pH (~8) is produced using NaOH-liquid; 3) dissolution of carbonate using CH_3COOH 20% or HCl; 4) density separation using sodium polytungstate to eliminate heavy minerals and to separate quartz (2.70 - 2.62 g cm^{-3}) from K-rich feldspars (< 2.58 g cm^{-3}); 5) etching the quartz grains in 40% HF for 80 min while stirring the grains. Etched grains are treated subsequently in 10% HCl and sieved to > 100 µm to remove broken grains; 6) spreading grains onto stainless steel discs; the amount of material is chosen according to the bleaching history of the sediment and the brightness of the mineral.

Detection filters

Generally, IR-OSL of feldspars is detected in the blue wavelength range using Schott 2xBG3/3 mm, BG39/3 mm, GG400/3 mm filters (Krbetschek et al., 1996). The filter combination transmits 390-450 nm and thus encompasses the 410 nm-emission. OSL of quartz is detected in the UV wavelength range using Hoya U340/7.5mm filter glass transmitting between 290 and 380 nm.

D_e determination

Fine-sand K-rich feldspar and polymineral fine-silt samples

Routinely, these samples are measured using a multiple aliquot additive dose protocol. We prepare 80 aliquots of a sample: 40 aliquots are used to determine De_β (10 aliquots for L_N and 6x5 aliquots for increasing $L_N+\beta$,.e.g. Figure 4) 3x3 aliquots receive an increasing additive α-dose and 10 to 25 aliquots are reserved for anomalous fading test A and test B (see section 6). All samples are normalised before any treatment using a 0.1 to 0.2 s stimulation time according to the brightness of the dosimeter. All samples are stored at room temperature for at least 30 d after irradiation.

Fine-sand quartz

These samples are measured using a single-aliquot regenerative-dose (SAR) protocol. If possible (e.g. enough material available), a preheat test is
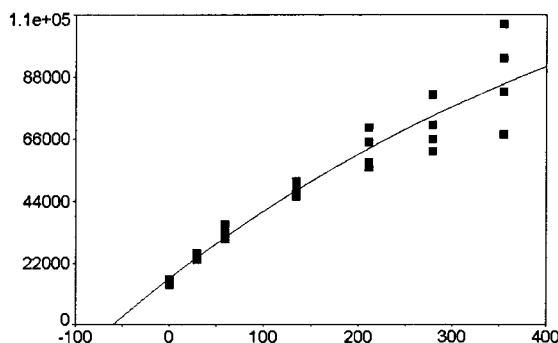

Figure 4.
Dose response curve and D_e determination of the loess sample (BN181) using multiple aliquot additive dose protocol. The sample was stored 40 d after irradiation, the preheat was 280°C for 10 s; IR-OSL_{net} = signal integral of the first 10 s subtracted from the background

conducted between 200°C and 300°C for 10 s to determine the optimal preheat procedure. Routinely, a preheat of 260°C for 10 s is used for L_x and 220°C for 10 s for T_x. The amount of recuperation is calculated from $\dfrac{L_N}{T_N} / \dfrac{L_0}{T_0}$, where L_N is the natural OSL and L_0 is the zero OSL of the SAR protocol (Murray and Wintle 2000, e.g. Table 2); The recycling ratio is determined from the ratio of the first regenerated dose and the repeated first regenerated dose at the end of the SAR-run (e.g. Table 2). Sensitivity change during a SAR run is corrected by a test dose OSL (T_x). The reliability of this correction is checked by plotting L_x versus T_x of a SAR protocol in which an equal beta-dose is administered for 6 times (Figure 5C).This run is conducted with aliquots used for D_e-determination without treating them further. The L_x/T_x relationship is expressed as Pearson's correlation coefficient (e.g. Table 2) and should be linear to zero. Figure 5C shows the robustness of SAR even if the test dose does not lay in the optimal dose range. If possible, $D_e(t)$-plots are produced over the entire stimulation time and the reliability of D_e estimation is checked according to the stimulation time chosen for D_e estimation (e.g. Table 2, Figure 5D). If necessary, a dose recovery is conducted, where a given dose similar to the estimated D_e is recovered. The same aliquots and the same SAR protocol used for D_e determination are used and the aliquots are not treated before administering the laboratory dose (e.g. Table 2).

Tests for contamination and for anomalous fading

Several tests are carried out to control the D_e result:

laboratory	technique	U [µg/g]	Th [µg/g]	K [wt%]	$\dot{D}_\alpha$	$\dot{D}_\beta$	$\dot{D}_\gamma$	D_e
FSA	low level γ-spec	3.32±0.24	8.21±0.85	1.20±0.09	12.25±0.74	1.67±0.08	1.10±0.06	62.4±5.4
MPI	NAA	3.00±0.11	8.09±0.09	1.19±0.03	11.48±0.25	1.61±0.03	1.05±0.02	-
MPI	NAA	2.80±0.08	8.27±0.09	1.19±0.04	11.16±0.18	1.59±0.03	1.04±0.01	-
MPI	NAA	2.84±0.17	8.09±0.24	1.18±0.05	11.13±0.40	1.58±0.05	1.03±0.03	-
SAW	low level γ-spec	3.00±0.10	7.42±0.32	1.07±0.06	11.07±0.29	1.50±0.05	0.99±0.02	-
	low level γ-spec	2.99±0.40	7.50±1.20	1.64±0.28	11.10±0.14	1.95±0.23	1.13±0.10	-
Bonn	low level γ-spec	3.12±0.05	8.50±0.10	1.09±0.04	12.0±0.13	1.56±0.03	1.02±0.01	59.8±14.6

Table 1.

Comparison of analytical data for the loess sample (BN181). Sample material used in this study has been collected subsequent to previous analysis and the sample was collected at a position only a few decimetres from the previous sampling position. Data from the other laboratories are unpublished.

FSA = Forschungsstelle Archaeometrie der Heidelberger Akademie der Wissenschaften, data provided by A. Lang (1997).

MPI = Max-Planck-Institut für Kernphysik in Heidelberg, data provided by E. Pernicka (1995).

SAW = Saechsische Akademie der Wissenschaften, data provided by J. Heinecke (1996).

Table 2.

Analytical data of BN169. Moisture factor is the measured field moisture normalised to the dry mass and corrected for fluctuation of water content; n indicates the number of measured aliquots; Recuperation is given in % of $\frac{L_0}{T_0}/\frac{L_N}{T_N}$; recycling ratio is the ratio of the repeated first regenerated dose at the end of the SAR protocol and first regenerated dose; Sensitivity change is a check of the correction procedure during SAR by the relationship of L_x and T_x after equal beta-dose for 6 times. The term is expressed in correlation coefficient. Dose recovery is given as recovered dose/laboratory dose. $D_e(t)$ indicates the stimulation time range (s), where D_e is onstant (1σ). IR-sensitivity indicates a feldspar component of the quartz dosimeter by the ratio $\frac{L_p}{T_p}/\frac{L}{T}$. Age is indicated with 1 σ total uncertainty.

For further explanations see text.

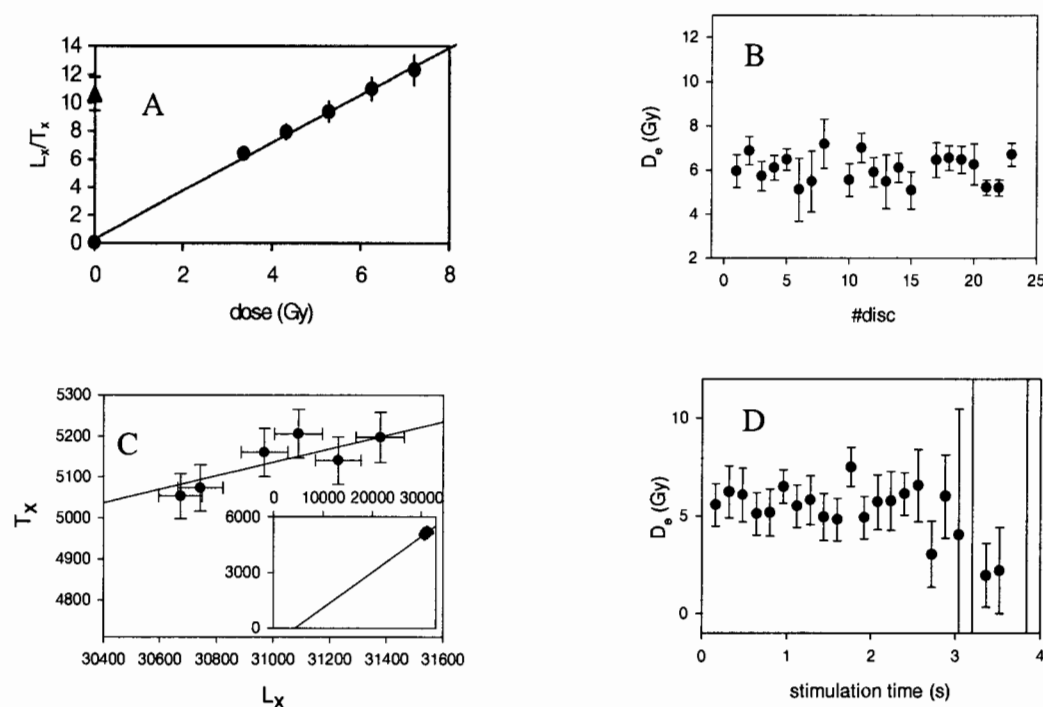| Moisture factor	1.15±0.10
grain size (µm)	160-200
U (µg g⁻¹)	0.28±0.026
Th (µg g⁻¹)	0.69±0.07
K (wt %)	0.44±0.01
$\dot{D}_{cosm}$ (Gy ka⁻¹)	0.18±0.009
$\dot{D}_{effective}$ (Gy ka⁻¹)	0.65±0.04
D_e (Gy)	6.05±0.17
n	21
recuperation (%)	0.11±0.02
recycling ratio	1.000±0.002
sensitivity change correction	0.70±0.04
dose recovery	1.03±0.03
$D_e(t)$ (s)	0 - 2
IR sensitivity	1.02±0.10
age (±1σ, ka)	9.30±0.68

Figure 5.
D_e determination and D_e reliability tests of the quartz sample BN169 using SAR protocol. A: dose response curve with circle=regenerated dose response using a single exponential saturation function, triangle= natural OSL. Data points are calculated as mean and standard deviation of 21 aliquots; B: dose distribution of 21 aliquots; C: plot of L_x versus T_x as a control of the sensitivity change correction procedure in SAR. Test dose was around 10% of the expected D_e (~0.48 Gy). Inset shows the same data set extrapolated to the x-axis; D: $D_e(t)$ plot of aliquot 22.

energy (keV)	nuclide	group and parent
583.2	^{208}Tl	
2614.5	^{208}Tl	group I: ^{232}Th
238.6	^{212}Pb	
911.2	^{228}Ac	
338.3	^{228}Ac	group II: ^{232}Th
969.0	^{228}Ac	
609.3	^{214}Bi	
1120.0	^{214}Bi	group I: ^{238}U
1764.5	^{214}Bi	
295.2	^{214}Pb	
351.9	^{214}Pb	
63.3	^{234}Th	group II: ^{238}U
46.5	^{210}Pb	
1460.8	^{40}K	^{40}K

Table 3.
Energy lines and daughter nuclides in secular equilibrium used to determine weighted mean of equivalent total U- and Th activity and ^{40}K activity. Data set and calibration of the spectrometer is based on the Nuclear Science Reference file.

Feldspar contamination of quartz:
Before conducting a SAR sequence for D_e-determination around 15 aliquots used for other tests are given a laboratory dose equivalent to the expected D_e and the OSL normalised to a test dose is measured ($\frac{L}{T}$). Subsequently the same aliquots are given the same laboratory dose as before and the post-IR OSL normalised to a test dose is measured ($\frac{Lp}{Tp}$). The ratio $\frac{L}{T}/\frac{Lp}{Tp} > 1$ indicates a feldspar component in the OSL signal (Henshilwood et al., 2002).

Anomalous fading

Tests for anomalous fading are performed on all feldspar samples. Two test protocols are used in our laboratory: Test A is routinely applied to all samples. Test B is performed, if test A shows a ratio of < 0.9.
Test A: This test is incorporated in the multiple aliquot additive dose protocol. A set of 5 aliquots with the natural dose (L_N) and 5 aliquots containing the maximum additive dose ($L_N+\beta_{max}$) used for D_e-determination are preheated at 280°C for 10 s and subsequently stored at room temperature for around three months (t~2200h). The amount of anomalous fading is then given by the ratio:

$$\left(\frac{L_{N+\beta\,max}}{L_N}\right)stored / \left(\frac{L_{N+\beta\,max}}{L_N}\right)unstored \qquad (1)$$

which is the ratio between the aliquots stored for ~2200 h and the aliquots stored for ~720 h.
Test B: A set of 20 aliquots are used: 10 aliquots for L_N and 10 aliquots for $L_N+\beta_{max}$, and further 5 aliquots are used to correct for optical erosion of L_N and $L_N+\beta_{max}$. We use t_1=2d, t_2=storage time of aliquots used for D_e-determination (~30d), t_3=40d, t_4=50d, t_6=60d and plot $\frac{L_{N+\beta\,max}}{L_N}$ versus storage time. The slope of the regression line indicates the amount of signal loss over LN(t). Yet, we do not correct ages by fading rates determined (Huntley and Lamothe, 2001) but need more experiences to do so. The age of a sample showing a significant fading rate is indicated with its minimum age (a_{min}) given as a_{min}= a-2σ where 2σ is the 95% confidence interval.

Dose rate determination
The dose rate is produced by the activity of natural radionuclides in the sample and its immediate environment. Our laboratory determines the specific activity (Bq kg⁻¹) of a sample using low level γ-spectrometry. The specific activity ($A_s(t)$) is subsequently transformed into concentration ($A*_s$, µg g⁻¹ and wt%) following

$$A*_s(t) = \frac{1000\,m_a}{\lambda N_A} A_s(t) \qquad (2)$$

where N_A = Avogadro constant, m_a=atomic weight (n) and $\lambda = \tau^{-1}$ (decay constant) multiplied by the conversion factors of Adamiec and Aitken (1998) to obtain the dose rate for each isotopic chain. The calculated $\dot{D}_\alpha$ and $\dot{D}_\beta$ are occasionally checked by thick source α-counting and β-counting respectively. The accuracy of a dose rate value determined in this way, is largely dependent on the γ-spectrometer technique, the reference material and the activity of the sample. Our laboratory uses:

low level γ-spectrometry:
Samples are packed into air-tight plastic boxes holding around 100 g and stored for ~3 weeks in order to achieve secular equilibrium between ²²⁶Ra and ²²²Rn. Routine measurement time is ~70h. Energy lines of the γ-spectrum are selected according to (1) detector efficiency, (2) specific line intensities, (3) expected nuclide concentrations, (4) superposition of energy lines, (5) position of the Compton edge, (6) self-absorption of the sample. Selected energy lines are shown in Table 3. An experiment designed as a qualitative check showed that, in the low energy range, self-absorption may become significant. The increasing background in this energy range together with increasing self-absorption reduces the precision of energy lines <100 keV. We believe therefore, that secular disequilibrium can only be assessed qualitatively.
The reference material used is: BfS ST2.3/Boden/V-98. This is a soil material provided by the 'Physikalisch-Technische Bundesanstalt' (Schkade et al., 1998).

a-value

The a-value is determined using: $\frac{D_{e\beta}}{D_{e\alpha}}$ according to the a-value system of Aitken (1985), where

$$a_{3.7} = \frac{L}{Gy\alpha}/\frac{L}{Gy\beta} \qquad (3)$$

$a_{3.7}$ = a-value resulting from the ²⁴¹Am α-source delivering 3.7 MeV, $\frac{L}{Gy(\alpha,\beta)}$ = luminescence per Gy α– or β– irradiation. $D_{e\alpha}$ is determined using six ²⁴¹Am sources and administering 3 different additive

doses on a separate set of aliquots containing fine–silt material. The α-doses chosen encompass the $D_{e\beta}$ of the sample. Cosmic ray contribution ($\dot{D}_{cos}$) is estimated using of the average burial depth of the sample (Prescott and Hutton, 1994). The internal dose rate of K-rich feldspars ($\dot{D}_i$) is determined using β-counting of the grains used for D_e estimation.

Total dose rate

The total dose rate of a quartz sample is calculated as the sum of $\dot{D}_\beta$, $\dot{D}_\gamma$ and $\dot{D}_{cos}$ assuming a negligible contribution of $\dot{D}_\alpha$ due to the HF-treatment of the grains. The total dose rate of a K-rich feldspar sample is the sum of $\dot{D}_\alpha$, $\dot{D}_\beta$, $\dot{D}_\gamma$, $\dot{D}_i$ and $\dot{D}_{cos}$ allowing for a $\dot{D}_\alpha$ contribution from 1/3 of the grain volume and an a-value assumed of 0.08±0.02. For both quartz and feldspar samples $\dot{D}_\beta$ is corrected with the attenuation factors for U, Th and K following Mejdahl (1979). The total dose rate of a fine-silt sample is the sum of $\dot{D}_\alpha$, $\dot{D}_\beta$, $\dot{D}_\gamma$ and $\dot{D}_{cos}$ The effective dose rate of all sample types described here results from correcting $\dot{D}_\alpha$, $\dot{D}_\beta$ and $\dot{D}_\gamma$ with the moisture factor (Aitken, 1985).

Uncertainties

The total uncertainty of an age determined is given by systematic and random errors. Sources of uncertainties are:

Water content: this value is estimated according to the field moisture monitored immediately after sampling, the pore volume estimated and the assessment of water content fluctuation over the time range to be determined. The latter is expressed by the uncertainty of the moisture factor.

γ-spectrometry: the uncertainty results from the fit (peak areas of the sample, of the reference material and of the background) and the effect of the background on the counting statistics of the sample. We assume similar and comparable sample geometry, chemical composition and mass between reference material and sample. The samples are measured as long as is needed to achieve a sufficiently low (<5%) statistical uncertainty in the peak areas. The value to be used for dose rate determination is calculated as the weighted mean of the energy lines that are in secular equilibrium (Table 3).

Beta source calibration: this uncertainty is calculated using Gaussian error progradation (equation 4) with the error of the reference source (here: Risø ^{137}Cs-source) and the error of the calibration measurement (counting statistics).

Internal activity of quartz: following Aitken (1985) a value of 0.03 Gy ka[-1] is added to the total dose rate uncertainty.

SAR: in the SAR protocol, uncertainties result from L_x, Bg_{Lx}, T_x, Bg_{Tx}, (Bg=background), calculated using $\sqrt{n}$ and each parameter of the regression analysis. Gaussian error propagation is used to determine the uncertainty of each aliquot. The uncertainty of D_e is then given as standard deviation of n measurements where n ≥ 24 including beta source calibration uncertainty.

Multiple aliquot: in the multiple aliquot additive dose protocol, the error of D_e is given by the disc-to-disc scatter, each parameter of the regression analysis and beta source calibration uncertainty.
Uncertainties are calculated using

$$\Delta F(x_1....x_n) = \pm \sqrt{\sum_{i=1}^{n} \left(\frac{\delta F(x1...xn)}{\delta xi}\Delta xi\right)^2} \quad (4):$$

where F = function, x = variable and Δx = absolute error of x.

Test of reliability of D_e determination in Bonn

Here, we show two examples of data sets used to control the laboratory procedures.

D_e of a polymineral fine-silt sample

The sample BN181 is a loess sample from the locality Nussloch (S-Germany). The sample was collected from calcareous loess overlying the Eltville Tephra. The age of the sample was expected to be younger than the Tephra, which has been dated to 22±3 ka (Zöller *et al.*, 1988, Hatté *et al.*, 1999), and older than ~15 ka, when loess deposition ended in this area. This sample has been analysed by a number of luminescence dating laboratories in Germany. From this comparison (unpublished) the D_e was expected to be ~60 Gy and IR-OSL age to be ~19 ka. Using multiple aliquot additive dose protocol and a preheat of 280°C for 10 s our laboratory determined 59.8±14.6 Gy (Table 1, Figure 4).

D_e of a fine-sand quartz sample

The quartz sample BN169 originates from a sandy dune layer in N-Germany, which was subject to podzolic alteration. This kind of soil forming process (dominantly vertical transport of Al and Fe in an acid

environment) should not alter the quartz grains, so that OSL is expected to date the time of dune sand deposition. The AMS ^{14}C age of a charcoal incorporated in this podzolic dune layer is 9.53±0.05 cal ka BP (Müller, 2000). According to the temporal relationship between sand deposition, soil formation and incorporation of the charcoal piece in the soil, the OSL age must be slightly lower than the ^{14}C age. Our laboratory determined 9.3±0.7 ka. All analytical data of the sample are given in Table 2.

Conclusion

It is shown, that the essential parameters in the luminescence laboratory of Bonn are accurate but that precision has to be improved.

We note here, that certificate reference material for α-counting, β-counting and γ-spectrometry is not available. Moreover, uncertainty calculations and fading tests and herein the time interval of observation need to be standardised. We feel, that reference material should be produced and more standards should be introduced in order to improve comparison in luminescence dating.

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