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Single-grain two-fragment method for dating terrace deposits using red thermoluminescence from quartz

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Abstract
We undertook red thermoluminescence (RTL) dating of terrace deposits in northern Japan using an improved method that requires the analysis of only a single quartz grain. In previous studies, two components (the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e) after exposure to artificial light) were evaluated from separate multiple-grain aliquots to obtain the age of deposits by RTL and other luminescence techniques.

We developed a new method, called the "single-grain two-fragment" (SGTF) method, to determine the values of Ap-D_e and Rl-D_e. In this method, two fragments are prepared by breaking a single quartz grain using a hammer and nail. The use of recently developed, highly sensitive RTL measurement apparatus enabled the successful detection of RTL signals from single quartz grains (300–500 μm in diameter) with doses as low as 20 Gy. The performance of the SGTF method was assessed by a case study involving the analysis of two samples of terrace deposits. Two separate RTL age groupings (241±20 and 113±17 ka) were determined as minimum equivalent doses (D_e), as deduced from Ap-D_e and Rl-D_e using the single aliquot regeneration (SAR) method. These ages are in good agreement with estimates of the age of the terraces based on geological criteria (OIS 5e and OIS 7).

Introduction
Studies of red thermoluminescence (RTL), based on emissions from 600–650 nm at around 360–390°C from volcanic quartz grains, were pioneered in the 1980s and 1990s (Hashimoto et al., 1986, 1987; Miallier et al., 1991). A series of studies has confirmed the potential of the RTL dating method in evaluating ages over 1 Ma, as trapped electrons are held for periods longer than 10^9 years at ambient temperature (Hashimoto et al., 1987, 1993; Fattahi and Stokes, 2000a). The reliability of these ages is also indicated by the absence of anomalous fading and the excellent RTL reproducibility obtained for repeated irradiations (Fattahi and Stokes, 2003). Using this method, RTL dating has been undertaken using multiple aliquots for volcanic products ranging in age from 2 ka to 1.2 Ma (Fattahi and Stokes, 2003). The advantages of the RTL method also raise the possibility of dating Pleistocene volcanic products and sedimentary deposits.

The application of RTL dating to Pleistocene sedimentary deposits remains limited by problems regarding the bleachability of the RTL signal in quartz. The RTL peak at 360–390°C is not completely bleached under daylight exposure (Miallier et al., 1994; Scholefield and Prescott, 1999; Lai and Murray, 2006). To overcome this problem, previous studies have examined the possible use of other peaks (e.g. 270, 305, and 325°C) because these RTL signals are more rapidly bleached than the 380–390°C peak (Scholefield and Prescott, 1999; Franklin et al., 2000). However, Lai and Murray (2006) reported that residual signals for the peaks at 300–370 and 370–420°C remained at significant levels for quartz from Chinese loess exposed to sunlight for 1260 minutes. To compensate for this residual level, previous studies have evaluated two components (the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e) after exposure to artificial light) from separate multiple-grain aliquots and obtained the age of the deposits by using the difference between the Ap-D_e and Rl-D_e (Tanaka et al., 1997).

The multiple-grain or multiple-aliquot methods used in previous studies of RTL dating are hampered by another problem; the sample aliquot may contain grains derived from several sources, of different ages, and transported by multiple processes from source areas. The selection of multiple grain aliquots results in different equivalent dose values for each aliquot, depending on the mixture of grains of different origins. To resolve this problem as much as possible, we propose a new dating method based upon the

analysis of single grains of quartz. Such an approach is complex since two parameters need to be measured (Ap-D_e and Rl-D_e). Huot and Lamothe (2003) had a similar challenge in their analysis of feldspars, and overcame this by splitting individual grains into two fragments. We have used a similar approach here. Our method is referred to as the single-grain two-fragment (SGTF) method. In this approach, two fragments from the same quartz grain are utilized to obtain the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e), using the single aliquot regeneration (SAR) method.

Sampling sites and sample preparation

For RTL dating, we analyzed samples NKW-U (NKW-U1, NKW-U2, and NKW-U3) (40°45'39"N, 141°15'40"E) and Fs-U (40°48'0"N, 141°17'58"E) collected from lower and upper terraces upon the Kamikita Plain, located along the Pacific coast of northern Japan (Fig. 1). NKW-U and Fs-U were used for dating by the single-grain two-fragment method (SGTF), and NKW-U1, NKW-U2, and NKW-U3 for multiple-grain dating. The ages of NKW-U and Fs-U are thought to correspond to OIS 7 (186–242 ka) and OIS 5e (127–105 ka), respectively, based on geological criteria (Kuwabara, 2004), comparisons of terrace altitudes, and the ages of two tephra layers (100 and 240 ka) intercalated in the terrace deposits. Both samples contain a reasonable amount of medium-sand-sized quartz grains, presumably supplied from early to middle Pleistocene volcanoes (Hakkodo Volcano and Towada Caldera) located to the west of the sampling sites.

The samples were collected from about 50 cm depth within the selected outcrops to ensure shielding from exposure to daylight. The water content of the samples (water weight/dry sample weight) was measured after drying at 110°C for 1 week. After washing and drying, the grains were sieved into two size fractions (250–350 and 700–900 μm) and treated with 5 M NaOH and 5 M HCl to dissolve glass shards. After magnetic separation, the surfaces of quartz grains were etched with 24% HF for 2 hours to remove glass shards. The purified quartz grains were finally re-sieved into the above two grain-size fractions.

RTL apparatus

RTL emissions from volcanic quartz yield a strong peak at around 360–380°C in the glow curve. To measure weak RTL signals, it is important to completely remove interference by black-body radiation emitted from the heating device at temperatures above 300°C (Fattahi and Stokes, 2000a, b; Stokes and Fattahi, 2003).

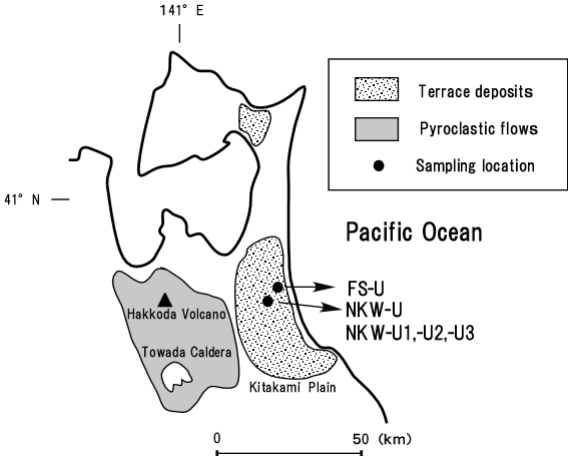

Figure 1: *Location map of the Kamikita Plain, northern Japan, showing the sampling sites for RTL dating.*

Various studies have sought to construct high-performance RTL measurement systems and have examined the optimum combination of filters (Fattahi and Stokes, 2000b; Yawata and Hashimoto, 2004, 2007; Ganzawa et al., 2005; Hashimoto, 2008). We assembled a highly sensitive RTL apparatus equipped with a multi-alkali photomultiplier tube (PMT) (R649s; Hamamatsu Photonics, Hamamatsu city, Japan) encapsulated in a thermoelectric refrigeration chamber (C2761; Hamamatsu Photonics) to minimize the noise signal of the PMT. The heating device, covered by a silver vessel, is able to heat the sample up to 600°C at a rate of 1 or 2°C.s^{-1}. A sample aliquot on a silver plate can also serve to reduce the amount of black-body radiation. The combined use of Hoya O60 (3 mm) and Schott BG39 (3 mm) optical filters enables the optimal detection of RTL at around 600 nm. The use of these components yielded a successful RTL readout, resulting in reliable RTL signals even from a 0.3 mm diameter single grain of quartz. A small X-ray source (VF-50JF; Variant) installed in the present apparatus can supply a dose rate of 6.1 Gy.min^{-1} to the irradiation site at a power of 50 W and 0.1 mA. The use of this X-ray source enables us to apply the SAR method (Murray and Wintle, 2000a, b) as modified by Ganzawa et al. (2005). To eliminate lower-energy X-rays, a 200-μm-thick aluminum absorber was placed in front of the radiation port of the X-ray source (Hashimoto et al., 2002; Hashimoto, 2008). All components of the RTL apparatus were controlled by a personal computer, which enables the successive readout of 10 aliquots for SAR dating.

Prior to the RTL readouts, all quartz grains were screened by 852nm IR laser to eliminate RTL from

feldspar inclusions. The preheating condition was set to 220°C for 3 min. All RTL readouts in the experiments were obtained over the range 100 to 450°C at a heating rate of 1°C.s^{-1}. The minimum detection dose of the present RTL apparatus was estimated to be about 20 Gy when analyzing a single grain of 400 μm diameter. For bleaching tests, we used a solar simulator (XC-100B; SERIC) at a power of 100 W. Bleaching of the RTL signal from quartz grains from sample NKW-U reached a stable residual level of approximately 30–50% after an 8-hour bleaching period at a distance of 50 cm from the simulator, corresponding to the power of daylight bleaching for approximately 80–100 hours at a latitude of 45°N.

Experimental methods

1. Multiple-grain method

The multiple-grain method was applied to samples NKW-U1, -U2, and -U3 to evaluate the reproducibility and reliability of the dating method. The resulting ages were then compared with the ages obtained using the SGTF method. The value of D_e for the multiple-grain method was evaluated from two parameters: Ap-D_e determined from an aliquot (approximately 30 grains in each aliquot) using the SAR method, and Rl-D_e was also evaluated by the SAR method after bleaching of quartz grains by the solar simulator (see Figs. 2 and 3). The average Rl-D_e assessed from five aliquots was used for D_e evaluation. Dose response curves were obtained using the integrated signal between 340 and 360°C of the RTL glow curves. Several values of D_e were independently determined from different sets of samples.

2. Single-grain two-fragment method (SGTF)

It is impossible to obtain both Ap-D_e and Rl-D_e from a single grain, because each result must be separately measured using different experimental procedures. To overcome this difficulty, we propose the SGTF method to evaluate Ap-D_e and Rl-D_e individually from two quartz fragments split from a single quartz grain using a hammer and small nail. The SAR method was applied to one of the two quartz fragments to determine Ap-D_e. Rl-D_e was calculated using the SAR method from the second fragment after full bleaching, as employed in the multiple-grain method described above. Details of the SAR experimental method are shown in Fig. 2; regenerative doses were given from 50 to 600 Gy and a test dose of 50 Gy was used. A dose recovery test (204 Gy) was carried out in the final run of the SAR method (Fig. 2). The equivalent dose (D_e) from the original single grain can then be evaluated by subtracting Rl-D_e from Ap-D_e, as calculated independently from the two fragments.

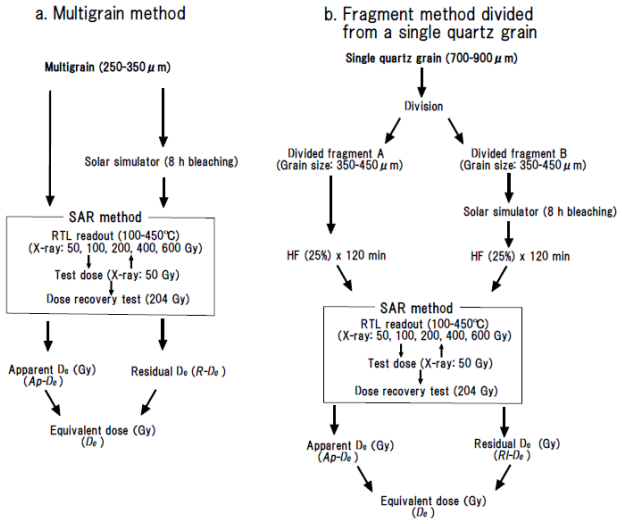

Figure 2: *Experimental procedures followed in determining D_e using (a) the multiple-grain method and (b) the single-grain two-fragment (SGTF) method.*

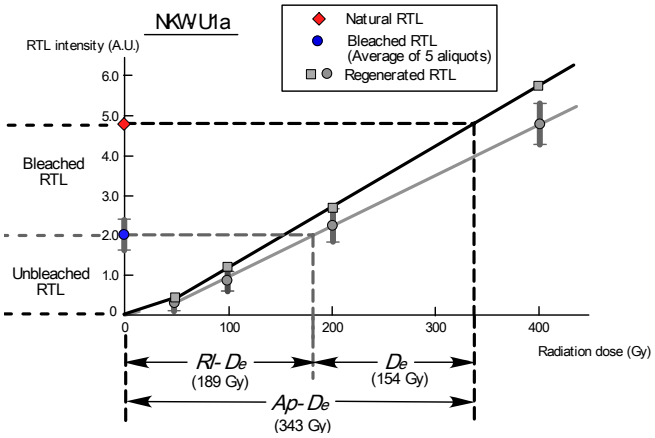

Figure. 3: *D_e evaluation of multi-grain method (Sample code, NKW-U1a; See Fig.2 and Table 1). D_e of an aliquot (30 grains in an aliquot) was determined from the natural sample using the SAR method. Rl-D_e, averaged from five aliquots containing about 30 grains in each, was deduced from bleached RTL intensities after 8-hour bleaching period using the growth curve of NKW-U1a. Ap-D_e was calculated by subtraction of Rl-D_e from D_e.*

3. Annual dose rate

The concentration of uranium and thorium was measured by neutron activation analysis (NAA) at the Inter-University Laboratory for Joint Use of Japan Atomic Energy Research Institute facilities in Japan. An ICP-mass spectrometer was used for analyses of potassium (Ganzawa et al., 2005). The average water

Sample	Ap-D_e (Gy)	Rl-D_e (Gy)	D_e (Gy)	D_a (mGy.a^{-1})	Age (ka)
NKW-U1					
NKW-U1a	343±17	189±19	154±17	0.57±0.1	261±29
NKW-U1b	384±19	212±17	172±16	0.57±0.1	292±28
NKW-U1c	342±14	188±17	154±15	0.57±0.1	261±26
NKW-U1d	356±21	196±14	160±15	0.57±0.1	271±25
NKW-U2					
NKW-U2a	323±16	248±20	75±7	0.42±0.1	169±16
NKW-U2b	303±12	232±26	71±8	0.42±0.1	179±21
NKW-U3					
NKW-U3a	345±17	152±20	193±27	0.50±0.1	386±54
NKW-U3b	332±10	146±23	186±30	0.50±0.1	372±61
NKW-U3c	324±13	143±21	181±28	0.50±0.1	362±56
NKW-U3d	340±20	150±15	190±22	0.50±0.1	380±44

Table 1: *RTL ages calculated using the multiple-grain method. D_e: equivalent dose, Ap-D_e: apparent equivalent dose, Rl-D_e: residual level dose, D_a: annual dose rate, Age: deposit age. Uncertainties in the ages are total errors, including both random and systematic uncertainties.*

content of the samples (18–20 wt%) was measured from three samples collected during three different seasons in a single year. The cosmic doses of 0.11, 0.03 and 0.12 mGy.a^{-1} for NKW-U, NKW-L and FS were calibrated using the standard cosmic ray intensity (0.185 mGy.a^{-1}) at a latitude of 40°N (Prescott and Hutton, 1994). Annual dose rates were estimated from the U, Th, and K contents using conversion data and recently developed conversion factors (Aitken, 1985; Adamiec and Aitken, 1998). Additionally, beta attenuation ratios for 300 μm size quartz grains of 0.81, 0.75 and 0.90 for U, Th and K, were also used for annual dose rates. Thus, the total dose rates at the sampling sites were evaluated to be 0.42–0.57 mGy.a^{-1}.

Results and discussion

1. RTL ages obtained using the multiple-grain method

Analysis of sample NKW-U1 (collected from the uppermost site on the upper terrace) using the multiple-grain method yielded ages in the range 261±29 to 292±28 ka (Table 1). These ages are older than the assigned age of OIS 7 (186–242 ka), as indicated by geological evidence (Kuwabara, 2004). Multiple-grain RTL ages of 169±16 to 179±21 ka and 362±56 to 386±54 ka were determined for NKW-U2 and NKW-U3 at levels located 3 and 5 m below NKW-U1, respectively, again in disagreement with the age of OIS7 (Table 1). These ages are also inconsistent with the known stratigraphy at the sites of the three samples (Kuwabara, 2004).

One possible explanation of the conflicting ages is the mixing in the aliquot of grains derived from several early to middle Pleistocene volcanic sources in the area to the west of the sampling sites (Fig. 1). An additional uncertainty is the bleachability of volcanic quartz, which is strongly resistant against sun-bleaching.

2. Variations in glow curves for single quartz grains

To understand the results from the multiple-grain aliquots, the natural RTL count rate emitted from individual grains was assessed. Figure 4 shows the cumulative sum of the RTL signals from 48 single grains of NKW-U, arranged from brightest to dimmest. The two brightest grains contribute 20% of the total natural RTL counts of the 48 grains, and the nine brightest grains contribute more than 50%, indicating that the value of D_e evaluated using the multiple-grain method is strongly dependent on a small number of the brightest grains.

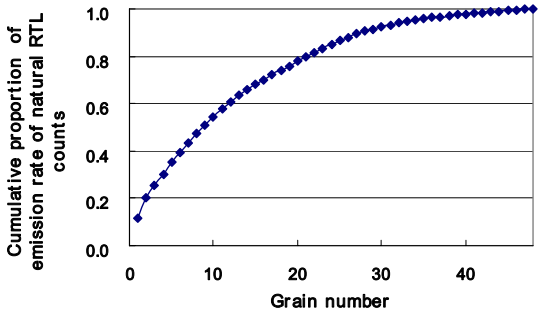

Figure 4: *Cumulative proportion of natural RTL counts for 48 grains. The grains are arranged in the figure from the brightest (grain number 1) to the dimmest (grain number 48).*

We also examined the pattern of RTL glow curves obtained for single grains. Figure 5 shows three representative natural RTL glow curves obtained for single grains of NKW-U, classified into end-member patterns, type-A and type-B, and an intermediate pattern, type-C. Type-A shows a single pronounced peak at around 360°C, whereas type-B and -C show broad patterns, with a mixing of low- and high-temperature (300 and 360°C, respectively) peaks. The different RTL patterns evident in Fig. 5 possibly reflect the diversity of volcanic quartz grains, probably related to magma temperature and component, volcanic age, bleachability and so on. The analytical result of RTL patterns of individual grains showed that NKW-U1 was composed of 11 grains for type-A, 11 grains for Type-B and 2 grains for type-C. The above results indicate that NKW-U consists of various types of quartz grains; consequently, the RTL multiple-grain method is not appropriate for dating this deposit.

3. The effect of hammering on the natural RTL of grains

The SGTF method proposed here involves hitting the single grain with a hammer and nail. This might have affected the RTL signal intensity. Therefore, we assessed the effect of hammering on the natural RTL intensity by examining the SAR D_e and the natural RTL intensity (normalized by a test dose of 50 Gy) in an experiment on a 100 ka (corresponding to 200 Gy) sample of volcanic quartz grains. The effect of hammering was evaluated by comparing the 14 impacted fragments from 7 single grains with 56 impact-free single grains. In Fig. 6 pairs of fragments extracted from the same grain are shown using the same symbol (e.g., gray inverted triangles), and 56 single grains are represented by white diamonds with a blue outline. The fragment pairs show similar D_e values and similar normalized RTL intensities, and values obtained for fragment pairs lie within the field of values obtained for the 56 intact single grains.

The results reveal that the hammering involved in breaking multiple fragments from a single grain using a hammer and small nail results in little change to the original natural RTL intensity of the quartz fragments. Consequently, there was no need to correct the natural RTL intensity for the effect of hitting impact.

4. RTL age of single grains

The effective D_e value required for dating sedimentary deposits can be calculated from SAR measurements of Ap-D_e and Rl-D_e obtained from two quartz fragments from a single grain. All dose recovery tests (204 Gy) showed satisfactory values within a range between 194 Gy and 222 Gy. The

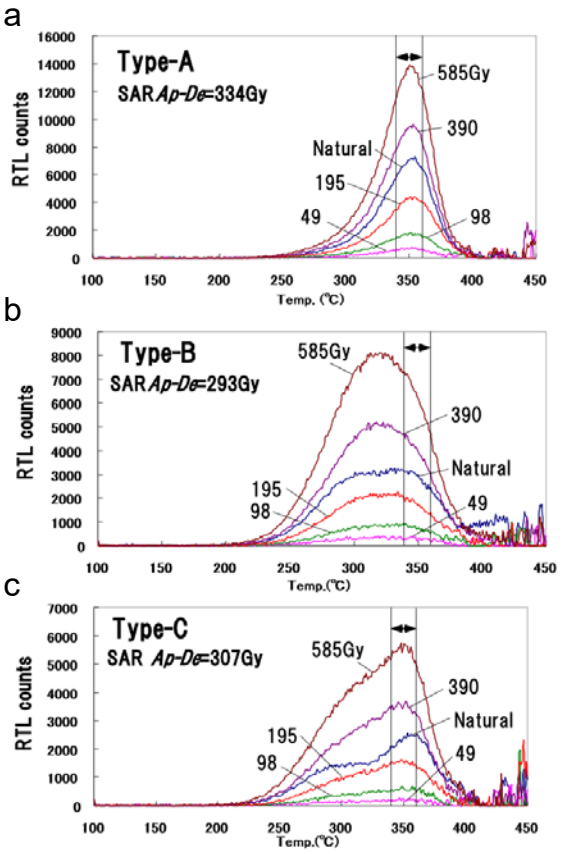

Figure 5: *Three representative glow patterns of the natural RTL of single grains. The obtained patterns were classified as either a mono-peak (a, type-A), a broad peak (b, type-B), or an intermediate double-peak (c, type-C). The RTL intensity was evaluated by the integrated counts for the range 340 to 360℃ in the glow curves (see double-headed arrows). Ap-D_e values evaluated by the SAR method, for regenerated doses of irradiation from 98 to 585 Gy, were 334, 293, and 307 Gy for Type-A, -B, and -C, respectively.*

value of D_e obtained from many replicate grains by subtracting Rl-D_e from Ap-D_e yields a range, with low values obtained from well-bleached grains and higher values obtained from incompletely bleached grains. In terms of the variation in D_e among grains, the minimum value of D_e is concordant with the effective D_e, providing the best estimate of the age of the deposition; this arises because D_e consists of the pure RTL signal accumulated since the quartz grain was finally fixed in the sedimentary deposit. Radial plot analysis provides an accurate means of showing the range of D_e values.

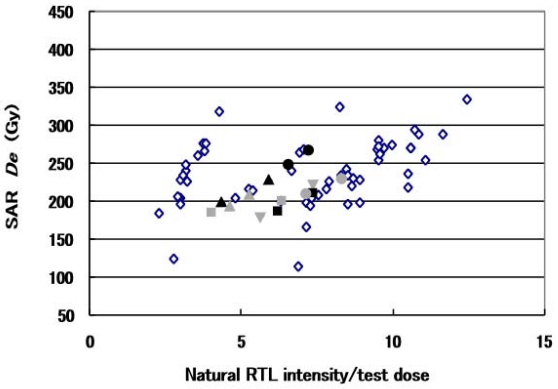

Figure 6: *Comparison of SAR D_e and normalized natural RTL intensity for 14 pairs of quartz fragments derived from individual grains and 56 single grains. The same symbol (e.g., black triangles) is used for each pair of fragments, and the 56 non-divided single grains are represented by white diamonds with blue borders.*

The D_e values obtained for NKW-U1 range from 118 to 408 Gy (Fig. 7a). The eight grains (filled circles) distributed in the 2σ range represent the minimum D_e, and can be regarded as a well-bleached grain mass. The eight grains also passed the χ2-test showing a ratio of 81.4. The RTL pattern of glow curves of these grains within the range of the minimum D_e are composed of three grains of type-A, four grains of type-B and one grain of type -C. This result suggests a wide variation of bleach-process and bleached degree of individual quartz grains. The D_e range of the mass is 118 to 165 Gy, yielding an average age of 241±20 ka based on an annual dose of 0.57 mGy.a^{-1}. This age agrees well with the estimated age range of OIS 7 (186–242 ka; Kuwabara, 2004).

In an additional test using sample Fs-U, the D_e distribution also showed a wide range, between 62 to 676 Gy. The average age, evaluated from the minimum D_e of five grains between 62 to 112 Gy in the 2σ range, which showed a ratio of 54.2 for χ2-test, was 113±17 ka based on an annual dose of 0.86 mGy (Fig. 7b). This age is also in agreement with the estimated era of OIS 5 (71–127 ka).

These two ages obtained using the SGTF method demonstrate the validity and applicability of RTL dating of sedimentary deposits based on analyses of single grains of quartz.

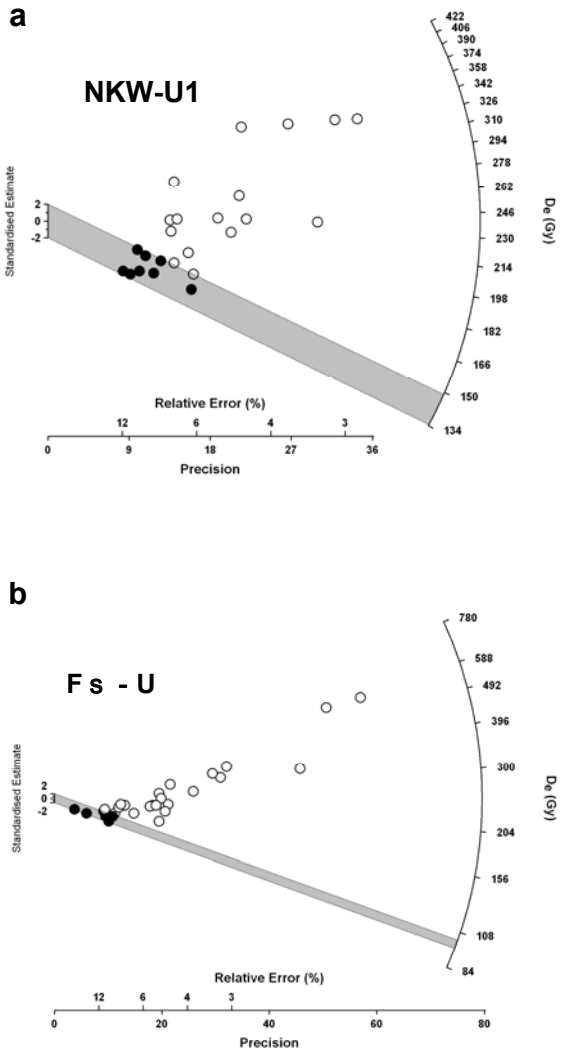

Figure 7: *D_e values of the terrace deposits (corresponding to the deposition age) of NKW-U1 (a) and Fs-U (b), as calculated from divided fragments from single grains (filled circles) within the 2σ range of the radial plot.*

Conclusions

1. We developed an improved RTL apparatus, comprising a multi-alkali PMT, an electric cooling system, and an X-ray irradiator, for the SAR method of dating single quartz grains. The minimum detection performance of this apparatus is about 20 Gy when analyzing a single quartz grain of 0.3 mm in size.

2. An RTL emission study of 48 individual grains revealed that the two brightest grains contribute 20%

of the total natural RTL intensity. This result shows that a small number of the brightest grains dominate the equivalent dose of a deposit when the multi-grain method is applied.

3. The SGTF method was tested based on an analysis of two terrace deposits in northern Japan. The single-grain ages obtained for the two tested samples, distributed within the minimum 2σ range in a D_e radial plot, are in agreement with the geological age and correspond to the timing of transgression in an Oxygen Isotopic stage diagram.

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Reviewer

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A method for retrospectively calculating the water content for silt-dominated desiccated core samples

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Introduction

When dating sediments by luminescence the water content is one of the most important values needed for age estimation and often places the largest uncertainty on dose rate calculation. If sediments are sampled in situ it is a straightforward process to measure their present day water content directly, although more consideration is required to assess what this figure would have been over geological time. For example, hydrological conditions may have experienced important changes in the past, as is often the case of fluvial sediments (Tanaka et al., 1997). This paper addresses the problem of retrospectively estimating the water content of core samples, and describes the procedure applied to a sedimentary core that was completely dry prior to sampling undertaken for luminescence dating. Following desiccation, the coarser, more sandy sediments are prone to disaggregation and so are unsuitable for luminescence dating as it is likely that many of the grains will have been exposed to light. Sampling was therefore restricted to the consolidated silty sediments where it was clear that the inner parts of the core had not been exposed to light subsequent to extraction. As the samples remained below the water table throughout burial it was assumed that all pore space in the sediment was originally filled with water. As the water evaporated during storage the sediment contracted and calculating the amount of this shrinkage is a relatively simple procedure. The amount of shrinkage can then be converted to pore space available for filling by water (Aitken, 1998, p. 82), but this does not take account of the pore space still remaining in the sample and which is also required to make an accurate assessment of original water content. This can be obtained by measuring the amount of water required to saturate a known volume of sample, but as the sediment under consideration very quickly disaggregated in water, it made a reliable measurement impossible. Instead, an alternative method to identify pore space volume, and subsequently water content, was used and is described here. The results were then evaluated by applying to fresh core samples and comparing the results with the conventional method for calculating water content.

Experimental procedure

A sedimentary core, drilled four years earlier in Azzano, northeastern Italy, was sampled for luminescence dating in 2006. The core was 260 m long, with the top lying only 9 m above sea level (a.s.l.), and composed of sand, silt and clay sediments, of which only the consolidated silty sediments were sampled (Fig. 1). As it had been exposed to air during storage almost all the water originally present had evaporated.

Figure 1: *Azzano Decimo core, samples ADC18 and 19. Samples were between 8 and 20 cm in length, and only taken from those parts that remained completely consolidated.*

When measuring the water content of fresh samples, they are usually weighed before and after oven drying, and water content (w) is normally expressed as % by weight using the equation

$$w = \frac{M_w - M_d}{M_d} \text{ x } 100, \qquad (1)$$

where M_w and M_d are the weights of the wet sediment and dry sediment respectively (terms used in this paper are listed in the Appendix). In order to identify an upper limit for water content, the fraction of the total volume that can be occupied by water, the pore space volume (V_p), is required (Aitken, 1998, p 63); this is a simple task when the original bulk volume (V) of the sample is known, and can be calculated using the equation

$$V_p = V - \frac{M_d}{\rho_d} \qquad (2)$$

where ρ_d is the density of the particles within the sediment. Water content can then be calculated by modifying Equation 1 to read

$$w = \frac{\left(\frac{V_p}{\rho_w}\right)}{M_d} \times 100, \qquad (3)$$

where ρ_w is the density of water. As the core samples had remained saturated during burial it was the original pore space volume of the sediment that was required to identify water content, and this required a little more effort to establish their original bulk volume; the samples were taken from one half of the core and it could not be assumed they had been cut exactly in half. In order to identify the original water content, two unknown values were required; the pore space volume of the completely dry samples ($V_p(d)$), together with the amount of shrinkage that the samples had undergone. To determine $V_p(d)$, pieces between 5-18 cm^3 of each sample were cut and, to ensure that only sediment grains and pore space were present, samples were first air dried, and then oven-dried overnight at 105°C. The samples were then weighed (M_d) and their bulk volume (V_d) determined using mercury porosimetry (Rootare, 1970). As a non-wetting liquid, mercury will not penetrate the sediment under ambient pressure and instead, forms an envelope around the material to be measured. Samples were submerged in mercury, and measurement of the displaced liquid was used to calculate V_d. The density of the grains (ρ_d) making up individual samples was measured on additional portions of disaggregated oven-dried sediment, using a gas pycnometer, after which present pore space ($V_p(d)$) of the sample was calculated using the equation

$$V_p(d) = V_d - \left(\frac{M_d}{\rho_d}\right). \qquad (4)$$

Determination of the amount of shrinkage that had occurred was conducted on the bulk samples (Fig. 1) following oven drying. The entire length of the core had been split in half and samples taken from one half. The present diameter of the samples (d_d) and of the coring equipment (d_o) was identified, and used to estimate the original bulk volume (V) using the equation

$$V = V_d\left(\frac{d_o^{\,3}}{d_d^{\,3}}\right). \qquad (5)$$

The linear values for diameter were cubed ($d_d^3$and d_o^3) to ensure that the ratio between them reflected a change in volume, rather than just length which would result in an underestimation. This assumes that shrinkage occurs to the same magnitude in all dimensions which may not exactly be the case, but was considered the most appropriate approach when the sample length was often not too much longer than its diameter, as illustrated in Fig. 1. To identify the original pore space volume (V_p), a similar correction was also made to present pore space ($V_p(d)$) of the sample using the equation

$$V_p = V_p(d)\left(\frac{d_o^{\,3}}{d_d^{\,3}}\right), \qquad (6)$$

after which Equation 3 could then be solved to identify the original water content (as % by weight).

Testing the method

In order to check the validity of the pore space volume approach in evaluating water content, samples were taken from three further cores. One was 41 m long and drilled one year earlier from Lake Fimon (FM1-12), northeastern Italy, and had subsequently been stored in a plastic liner. This sediment was higher in both organic and clay components than the Azzano samples. Two more, 12 and 40 m long cores, were taken from Niederweningen, northern Switzerland (NWG1/1-11 and NWG2/1-12) and were sampled shortly after coring. While most of these were from silty sediments, they also included some more sandy samples as well. Once again only the consolidated sediments were sampled. While it is possible that each of the cores may have lost some moisture following storage, they were considered to be in a similar condition to those samples taken normally for luminescence from fresh exposures. Two sets of samples were taken from each of the cores. Water content was determined on one set using the conventional method where samples were weighed

before and after drying at 105°C (w_o). The second set were air dried and then oven-dried overnight at 105°C to simulate the Azzano samples; water content was determined using mercury porosimetry (w_p) and shrinkage of the samples was also measured. Particle density was only measured for all samples in NWG1 (n = 12) and averaged 2.62 g cm^{-3}. This value was applied to the samples that were more sandy at the top of NWG2, but for the lower silt/clay samples a value of 2.75 g cm^{-3} was applied; this was the average value for the original Azzano samples to which the fine sediments of NWG2 were far more similar. For the Fimon samples, a value of 2.65 g cm^{-3} was applied as, although these were very silty, they were known to also contain an organic component.

Water content (w_{LOI}) of the Fimon core, at 25-50 cm intervals, had already been measured prior to loss on ignition (LOI) measurements. While this is done in exactly the same way as the conventional oven dry method, samples for LOI are approximately 1 cm^3, and so between 5 and 18 times smaller than those used for the former method.

As core samples may often remain exposed to air for several hours when first opened and during cataloguing of the samples, the effect of this on measured water content was investigated. Three different sizes of fresh sample (NWG2/6) were cut and weighed, with the smallest sample being similar in size to those taken for w_{LOI}. They were then weighed intermittently as they stood exposed in the luminescence laboratory at room temperature over the space of 3 hours; these measurements were used to identify water content as a function of time.

Results

Fig. 2(a) compares the water content calculated using both the oven dry and pore space volume methods for NWG1, 2 and Fimon. NWG1 and 2 record water contents of up to 30 % using the w_o, and values calculated using w_p are within 20 % of these, which would result in a variation in age of ± 4%. The Fimon samples were all higher in water content than both NWG1 and 2, and w_p underestimates the oven dry method by more than 40 % in some cases, which would lead to much larger age underestimations. For those more sandy samples that disaggregated on drying it was not possible to derive water content values using w_p. Fig. 2(b) compares the water content calculated for the Fimon samples using w_{LOI} and w_o values. These show a systematic underestimation using the w_{LOI} measurements although for almost all samples, this is no more than 10% below that derived by w_o.

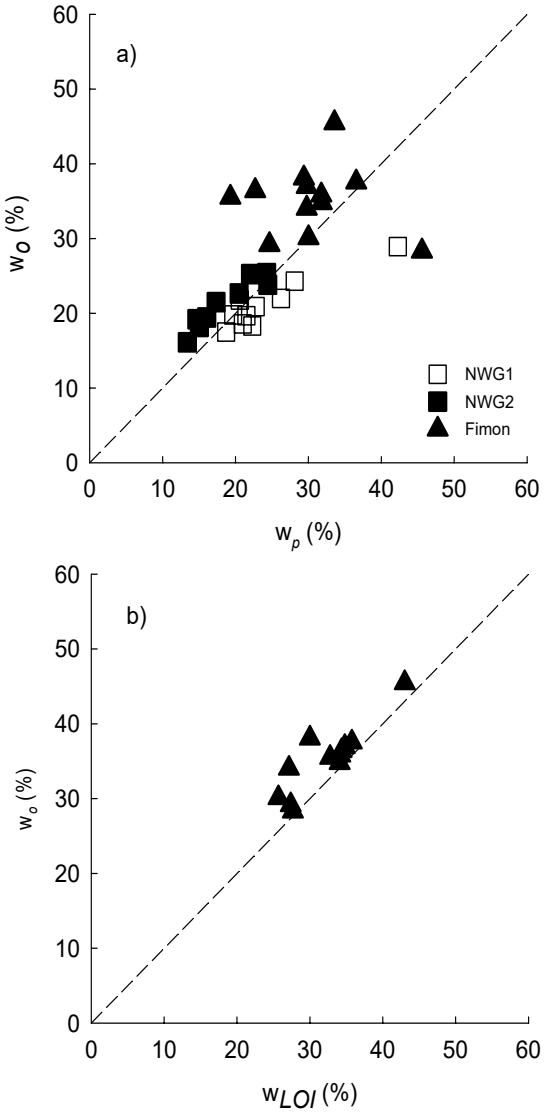

Figure 2: *(a)Comparison of pore space volume (w_p) and conventional oven dry(w_o) methods used to identify water content (expressed as % by weight). (b) Comparison of oven dry (w_o) and preliminary LOI (w_{LOI}) measurements made to determine water content for the Fimon samples. Dashed lines signify unity.*

The measured water content of samples as a function of time while exposed in the laboratory is plotted in Fig. 3; this shows that the smallest samples, which have the largest surface area relative to volume, exhibit the greatest loss of water. The largest piece, with a volume of approximately 32 cm^3 and a surface area of around 60 cm^2, lost 19% of its water content after 3 hours. The smallest piece with a volume of approximately 1 cm^3 and 6 cm^2 surface area, had lost 80% of its water content in the same time.

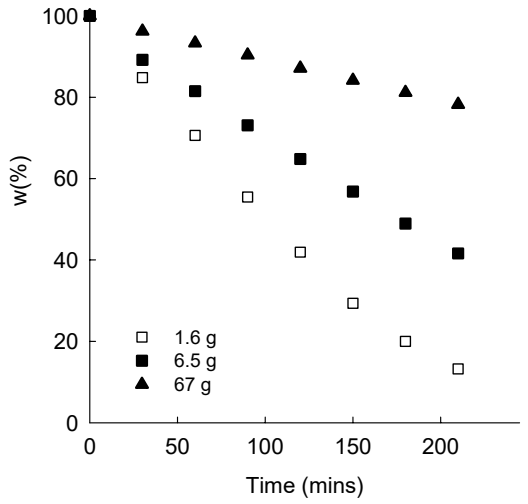

Figure 3: *Water content as a function of time during exposure at room temperature in the luminescence laboratory. This shows a correlation between relative surface area and moisture loss, with the greatest loss occurring in the smallest sample.*

Discussion

NWG1 and 2 samples show a good correlation between the two methods and suggest that the pore space volume method of calculating water content works well for these samples, while the Fimon samples on the other hand, show far more spread in the values. This is believed to stem primarily from the difficulty in measuring shrinkage of those samples that were less 'blocky' and subsequently distorted on drying, and also highlights that it is the reliable measurement of shrinkage on which estimating original water content relies. For example, NWG1/4 shrank from 4.0 cm to 3.7 cm, and a water content of 26% was subsequently calculated. Shrinkage of 0.1 cm less would produce a dry water content value of 21 %, and go on to produce a ~5 % lower age. The Azzano samples appear to have retained their original shape well and shrunk in a uniform manner. These samples remained undisturbed while they dried slowly and without heat, and their much larger volume (some pieces up to 20 cm long) led to a much more regular contraction. The possibility that cubing a linear measurement of shrinkage to convert it to volume may overestimate the amount of shrinkage, has already been mentioned, although it appears to have been appropriate in this study. It may be unrealistic to assume cores are always cut exactly in half, and if this were the case then it is not possible to estimate the true diameter of dry samples. For this reason a more accurate method of measuring the shrinkage was to lay each piece on circular templates of varying diameters, which provided a more reliable measurement and also confirmed that they had contracted uniformly. The original core was 8.89 cm in diameter and all samples were found to have shrunk to between 8.2-8.4 cm; an indication of the very similar sediments that were sampled throughout the core.

The need to determine individual particle densities for all samples was considered. The Azzano samples had particle densities between 2.65 and 2.85 g cm^{-3}, and applying a mean value of 2.65 g cm^{-3} only altered age calculations by ± 2 %. Although this figure is small, the value increases as sediment becomes finer and particle density increases suggesting that its actual determination may be preferable. It should also be mentioned that the comparison of the texture and appearance of unmeasured samples with a reference set of core sediments with a range of known particle densities, proved successful in the estimation of appropriate values.

It is important to be aware of possible moisture loss that may have occurred before water content measurements are made. Although the influence of compaction during coring needs to be considered for modern soft sediments (Zheng et al., 2002), the effect on the samples in this study, should be no more than that applicable to any sediments taken using a sampling cylinder. It has also been suggested that a small amount of water may be lost during core retrieval and extraction (Forman et al., 2007). A new core may also be exposed for several hours on first opening and cataloguing, and this can result in further water loss. While neither of these losses would influence water content estimation obtained using the pore space volume method, the oven dry method would be unable to identify the original water content.

Conclusions

While immediate measurement of fresh samples remains preferable, measurement of porosity offers a useful alternative in retrospectively estimating the water content of core sediment. Determining the present day pore space volume is simple, and quick to achieve, but using this to calculate the in situ water content is only possible when the subsequent shrinkage to samples can be accurately assessed. This is most likely where samples have remained undisturbed and dried slowly, resulting in a uniform shrinkage. It is highly unlikely that a core has been cut exactly in half, and so matching the curvature of cores to templates, rather than measuring the diameter, will provide more reliable results and also identify any distortion that may have occurred during drying.

Acknowledgements
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Appendix

d_d	Diameter of dried core sample (cm)
d_o	Diameter of coring equipment (cm)
M_d	Weight of dry sediment (g)
M_w	Weight of wet sediment (g)
ρ_d	Density of sediment grains (g cm^{-3})
ρ_w	Density of water (g cm^{-3})
V	Original bulk volume (cm^3)
V_d	Bulk volume of dried sample (cm^3)
$V_p(d)$	Pore space volume of dried sample (cm^3)
V_p	Original pore space volume (cm^3)
w	Water content (% by weight)
w_{LOI}	Water content determined prior to LOI measurements (samples ~ 1 cm^3)
w_o	Water content determined using weight of samples before and after drying oven drying at 105°C (% by weight)
w_p	Water content determined using mercury porosimetry following air drying and oven drying at 105°C (% by weight)

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Reviewer
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Can temperature assisted hydrostatic pressure reset the ambient TL of rocks? – A note on the TL of partially heated country rock from volcanic eruptions

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Abstract
In the West Eifel Vocanic field of Germany, extensive fine grained tephra beds exist. These are derived from fragmentation of Lower Devonian siltstone. Analysis of fine grain TL and the IRSL of fine-grained tephra beds from independently dated tephra layers of 11 ka and ca. 20 ka suggested that their geological luminescence was reduced to near zero residual value during the eruption. This was despite the absence of any field evidence of heating above 400°C .

We examined possible resetting mechanisms and suggest that thermally assisted hydrostatic pressure can reset the latent geological TL of country rock fragments during phreato-magmatic maar eruptions. We also provide first evidence of the suitability for TL dating of such material. As anomalous fading experiments have not been completed so far, we cannot give definitive TL ages here, but as the apparent ages are underestimated as compared to the control ages, complete zeroing is inferred. However a fortuitous combination of incomplete resetting and anomalous fading, that yields the expected D_e, though unlikely in view of long D_e plateaus arguing for complete resetting, cannot be entirely ruled out.

Introduction
Thermoluminescence (TL) dating has often been tested for volcanic minerals (e.g. Fattahi and Stokes, 2003) but may face the problems of anomalous fading of luminescence of volcanic feldspars (Wintle, 1973) or of low TL sensitivity of other volcanic minerals, with the exception of quartz. Quartz does not occur in mafic volcanic rocks except in situations when xenoliths derived from older country rock in the direct vicinity of the vent are also present. We examined the TL of fine-grained silicoclastic country rock occurring in maar tephra beds to understand if, besides heating, other processes can reset the parent "geological" TL signal of such minerals.

Hydroclastic maar eruptions occurred in the entire Eifel Volcanic Field (Schmincke, 2000). In the West Eifel Volcanic Field maar tephra has >90% of country rock clasts (mainly Lower Devonian slates, siltstones and quartzites or quartzitic sandstones) and their TL is expected to be in saturation or in thermal equilibrium with corresponding palaeodoses in the range 2000 to 3000 Gy (Wagner, 1998, Fig. 79). Maar lakes are excellent archives of past climates (Negendank and Zolitschka 1993; Zolitschka et al., 2000; Sirocko et al., 2005) and this makes reliable dating of maar eruptions key for major advances in regional palaeoclimatology.

In the Eifel Volcanic Field, the youngest, well dated maar eruption is the Ulmen Maar dated to 11,000 varve years (before 1950 AD, Zolitschka 2000). Proximal Pulvermaar eruption has not yet been radiometrically dated, however the presence of a post-eruptive ice-wedge cast in the tephra suggests its antiquity to be at least the last glacial maximum (LGM; 21.5 ka calendar years; Büchel et al., 2000). Fine-grains (4-11 μm) extracted from cm to dm thick silty - sandy tephra beds in the exposed ramparts of these two maar lakes had a natural TL intensity (blue, 420±60 nm) that was far below saturation of the dose response function (Figs. 1-3). For such a deposit, optical bleaching during eruption can not be the resetting mechanism as big blocks of country rock embedded in tephra suggested a deposition by base surges (Lorenz and Zimanowski, 2000). Thermal resetting during the fragmentation phase just prior to the eruption cannot be ruled out but, the field evidence does not carry any signatures of heating above 400°C. We therefore examined another possible resetting mechanism through hydrostatic pressure, following earlier mechanoluminescence studies by Banerjee et al. (1999; see also Singhvi et al., 1994 and Porat et al., 2007).

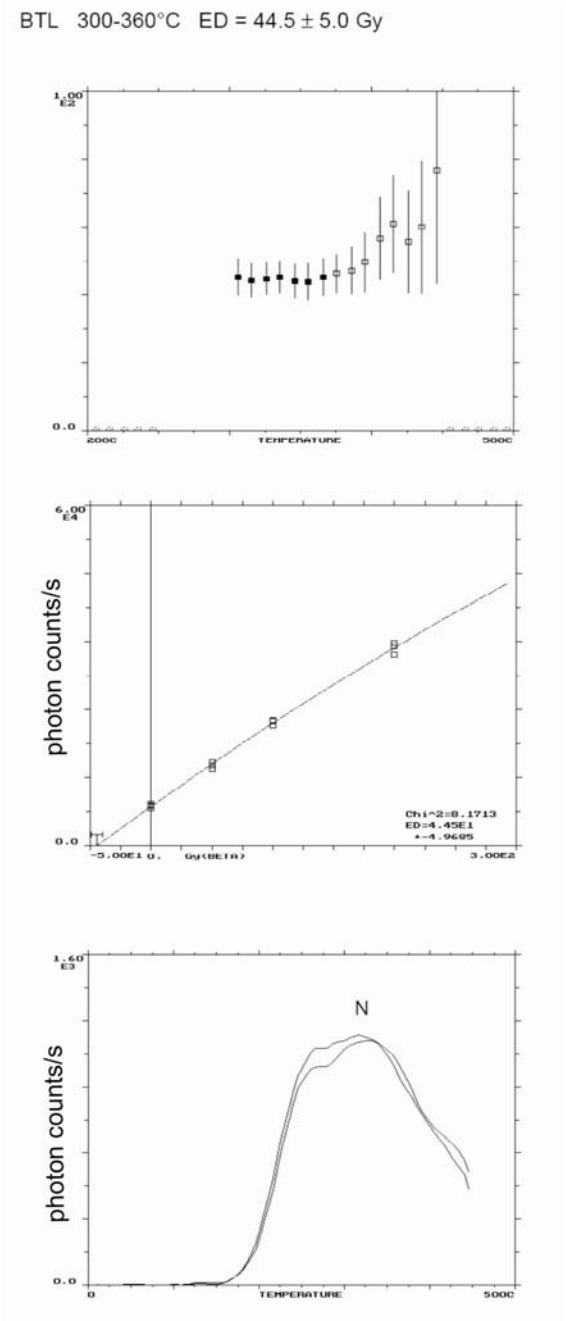

Figure 1: *D_e plateau test (top), TL growth curve 300-360°C (middle, saturating exponential fit, not corrected for anomalous fading) and two NTL glow-curves of #1 Ulmener Maar (bottom).*

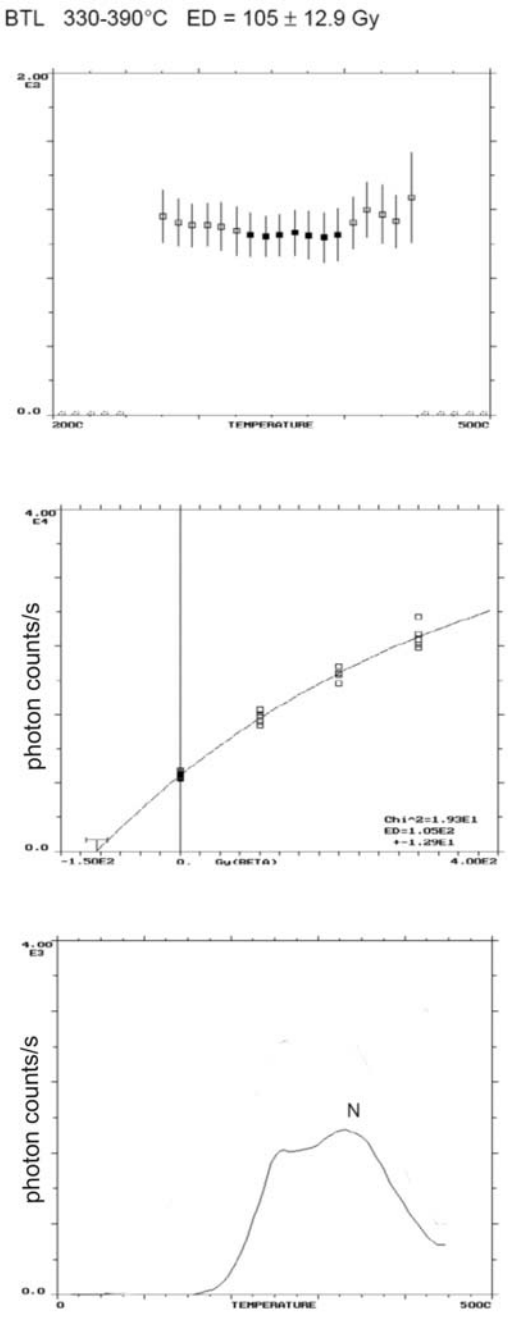

Figure 2: *D_e plateau test (top), TL growth curve 300-360°C (middle, saturating exponential fit, not corrected for anomalous fading) and NTL glow-curve of #2 Pulvermaar, bottom.*

Figure 3: *Road cut in the village of Ulmen, dark fine-grained maar tephra bed (hammer, sample 1) in the basal part of the rampart of the Ulmener Maar, topped by a diaclastic base-surge layer. Sample #1 was extracted at the tip of the hammer.*

Figure 4: *Exposure of tephra in the rampart of Pulvermaar. In the upper part, a 5-10 cm thick, light, fine-grained and more consolidated bed (A) sticks a bit out of the wall. This bed was sampled for TL and IRSL analysis (sample #2). In the lower part antidunes (B) and "swimming blocks" witness the deposition by base surges. The height of the exposure is 10 to 11 m.*

Samples

Samples were taken from the ramparts of two maar lakes in the West Eifel Volcanic Field, Germany, in the village of Ulmen (Ulmen Maar, Fig. 3), near the village of Gillenfeld (Pulvermaar, Fig. 4), and in the southern part of the city of Bonn in the Middle Rhine Valley (for details see below).

Two kinds of samples were collected, weakly consolidated fine-grained maar tephra-bed at Ulmen Maar, #1, (road cut in the village of Ulmen, 50°12' 38.10''N, 06°58'48.10''E, Fig. 3), and Pulvermaar, #2,(exposure 50°07'49.40''N, 06°55'05.40''E, Fig. 4), and a piece from Lower Devonian slate (sample #5) originating from the same epoch (Upper Siegenium) as country rock outcropping at Lake Pulvermaar. This was to test for zeroing experiments as the locality at the northern end of the Middle Rhine Valley at Bonn-Friesdorf is far away from any thermal overprint by volcanic activity during the Quaternary.

Experimental

Sample Preparation

The samples were processed under subdued red light (diodes, 620 nm) after removal of a minimum 2 mm outer rim using a knife or a handsaw. Sample #5 was carefully crushed in a bench vice and with an agate mortar and sieved (no natural TL below 200°C glow temperature indicative of triboluminescence was seen). Fine-grain samples were prepared using standard procedures (Zimmerman, 1971). From sample #2, the fraction 125-250µm was also extracted by wet sieving. Table 1 summarizes the experimental protocols for each sample. The fine-grain extracts from the Devonian slate (Bonn-Friesdorf, #5) were loaded in a silver tube of 3 mm diameter and 9 mm height and closed by a silver lid for subjecting it to high pressures.

High pressure and grinding experiments

The fine grains from sample #5 loaded in a silver tube were subjected to a static pressure of 1 GPa (10 kbar) for 19 h using the piston cylinder facilities of Bayerisches Geoinstitut (BGI) at Bayreuth. This static pressure corresponds to pressure conditions at the continental crust/mantle boundary. Two different runs of the experiment were conducted as follows:

Room temperature

a) natural sample held at 1 GPa for 19 h at room temperature;

b) natural sub-sample as received (without pressure).

Elevated temperature

a) natural sample held at 150°C for 19 h under 1 GPa pressure, and

b) natural sample held at 150°C for 19 h without pressure.

Furthermore, a sub-sample of rock sample #5 was homogenously spread between two steel plates. Strong hammer blows with a lump hammer (ca. 1 kg) were then administered to the upper plate for 3 minutes. Subsequently, the rock fractions were vigorously ground using an agate mortar for 15

Sample	Locality	Material	Expected age	Method	IRSL	Preheat
1	Ulmener Maar	fine-grain maar tephra, dark grey	11 ka	PM FG BTL ADD	max. ca. 72 cts/s	stage / cont. 220°C, 120 sec
2	Pulvermaar	fine-grain maar tephra, grey	18-24 ka	PM FG BTL ADD	max. ca. 75 cts/s	stage / cont. 220 °C, 120 sec
2	As above	As above	As above	PM 125-250um BTL ADD		stage / cont. 220 °C, 120 sec
5	Devonian Friesdorf	siltstone, Lower Devonian	400 Ma	PM FG BTL	max. ca. 650 cts/s	-

PM = polymineralic, FG = fine grains 4-11 μm, BTL = blue TL, ADD = additive dose (MAAD)

Table 1: *Description of samples and experimental details. For all samples a heating rate of 5°C/s was used, with measurements up to a maximum of 450°C. A filter combination of two BG-3 filters, a GG-400 and a BG-39 was used.*

minutes to see the effects of dynamic pressure in reducing the latent TL signal.

Luminescence and dosimetry measurements and data processing

TL and IRSL measurements were made using a Daybreak 1150 TL/IRSL reader equipped with infra-red diodes (870±30 nm), an EMI 9586Q photomultiplier (PM) coupled to a combination of detections filters (bottom to top: BG-3, GG-400, BG-3, BG-39, "blue combination" in Table 1). TL readout was at a heating rate of 5°C/s, to a maximum temperature of 450°C. The IRSL of sample #2 was recorded at room temperature.

For TL, preheat was done by holding the temperature at 220°C for 2 min followed by ramp heating to 450°C at 5°C/s. Laboratory irradiations were performed using a $^{90}Sr/^{90}Y$ beta source delivering ca. 0.17 Gy/s. Figs. 1-2, 5-6 provide the dose response curves using the MAAD protocol (Wintle, 1998). Owing to low IRSL sensitivity, no normalization beyond weight normalization (2 mg per disc) of aliquots was applied. Five aliquots per dose were measured. Tests for short term anomalous fading (7 days at 70°C, see Zöller, 1995) were carried out later to check for the occurrence of anomalous fading, but are not considered here as longer lasting fading tests at room temperature (required for correct estimation of burial dose) have not been executed so far. Estimation of the notional equivalent dose D_e (fading-uncorrected) required identification of the plateau range, (i.e. the glow curve temperature range exhibiting identical D_e within a given experimental variability of ca. 5%). As the growth curves were non-linear we used the D_e plateau test only.

Although definite TL ages were not the aim of this study, a check for significant age overestimates owing to incomplete TL resetting required dose rate calculations. Effective internal α- and ß-dose-rates for the samples were calculated using thick source alpha-counting of fine-ground bulk samples (Aitken, 1985; Zöller and Pernicka, 1989) for U and Th decay chains, and by ICP-MS and AAS measurements for K. Dose conversion factors of Adamiec and Aitken (1998) were applied. The γ dose-rate was calculated from U, Th and K concentrations of the maar tephras assuming homogenous 4π geometry. The cosmic dose rate was estimated with respect to sample depth below surface using Prescott and Hutton (1994). Present day interstitial water content was measured for samples #1 and #2 with due consciousness that this value may not be representative for the entire burial time. The a-value (alpha efficiency, see Aitken, 1985) was taken as 0.08±0.02 for polymineral fine-grain samples (representative value from Zöller, 1995). A secular equilibrium of U decay chains was assumed. In the absence of anomalous fading corrections, the notional ages of samples #1 and #2 do not provide the true eruption ages, but do provide an indication if these ages accord with the expected ages or, if these are overestimated resulting from incomplete zeroing. Underestimates of the apparent ages, of course, may result from anomalous fading, but this questions lies outside of the scope of the present study.

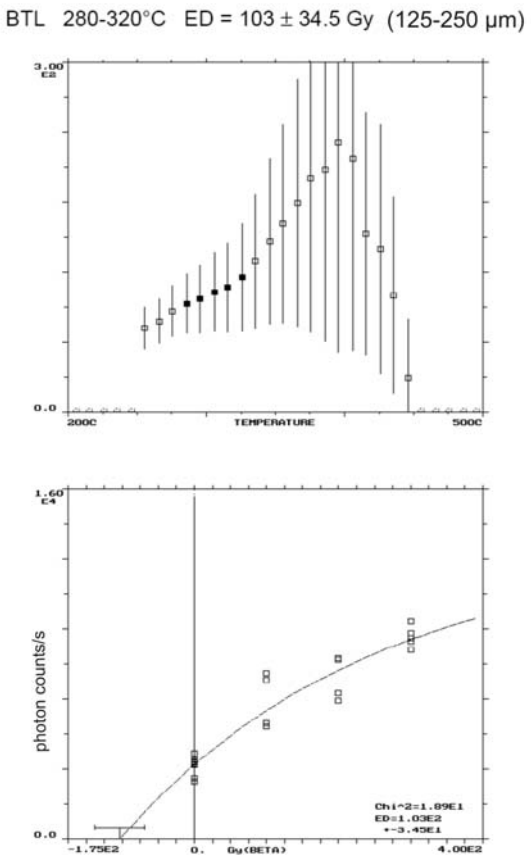

Figure 5: *D_e plateau test and TL growth curve of #2 Pulvermaar, 125-250 µm, 280-320°C (saturating exponential fit), not corrected for anomalous fading.*

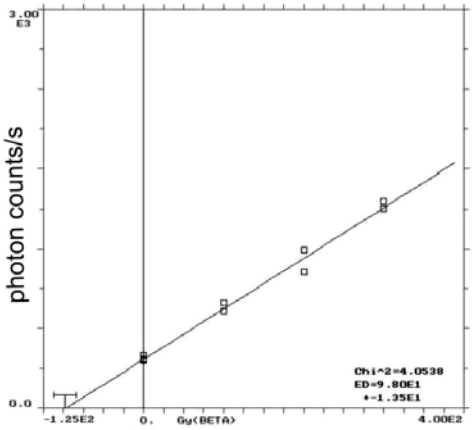

Figure 6: *IRSL MAAD #2 Pulvermaar, fine grains, linear fit, not corrected for anomalous fading. Within error bars, the D_e (98±13.5 Gy) is identical with the D_e from TL measurements (see Fig. 2)*

Results

Results of high hydrostatic pressure and grinding experiments

The aliquot to aliquot reproducibility of TL glow-curves from the first experiment (high pressure at room temperature) was ± ca. 25% compared to ± ca. 10% in the second experiment (high pressure at 150°C). However, loss of TL intensity after high pressure at room temperature (not shown) was not detected. The ratio of intensities was not significantly different from 1. The grinding experiment increased the natural TL via triboluminescence by ca. 7% rather than draining it.

High pressure at elevated temperature, however, resulted in a partial decay of the natural TL beyond thermal draining (Fig. 7). The TL signal loss affected the entire natural glow peaks and up to 36% of the signal was lost when compared to the natural TL after an identical thermal wash of 19 h at 150°C. This suggests that high hydrostatic pressure at elevated temperature is able to at least partially reset the latent TL and thermal assistance enhances the effect. Such conditions of moderately elevated temperature (ca. 150°C or more) and high pressure may exceptionally occur at shallow crustal depths typically <3 km in a maar explosion chamber during the fragmentation stage. For such a rock of Lower Devonian age, the geological dose is computed to be 2 MGy.

TL results from fine-grained maar tephra

Sample #1a (TL from Ulmener Maar tephra) yielded a long D_e plateau which was not expected a priori due to absence of any field evidence of heating that could erase the geological luminescence. The dose response was almost linear up to 30 Gy of additive dose (Fig. 1). This suggested a near complete resetting of TL (of fine grains) at eruption in the D_e plateau range (up to 360°C) as partial resetting would have implied finite initial dose and hence chances of nonlinear growth increases (as is evident from Fig. 2 and from TL growth curves of older maar tephra beds not mentioned here, onset of sublinearity can be seen from 200-300 Gy on). Supra linearity intercept (Aitken 1985) was not determined as its magnitude was expected to lie within the error bars of D_e.

The apparent TL age for Ulmener Maar was calculated to be 7.3 ± 1.0 ka (not corrected for observed short term fading of ca. 13% within 7 days at 70°C). The apparent TL age is significantly lower than the independent age (11 ka, Zolitschka et al., 2000), but as it is not higher than the expected age, a plausible inference of zeroing could be made. Age overestimation would be expected in the case of incomplete TL zeroing during eruption. As the apparent TL age of sample #1, calculated from the D_e

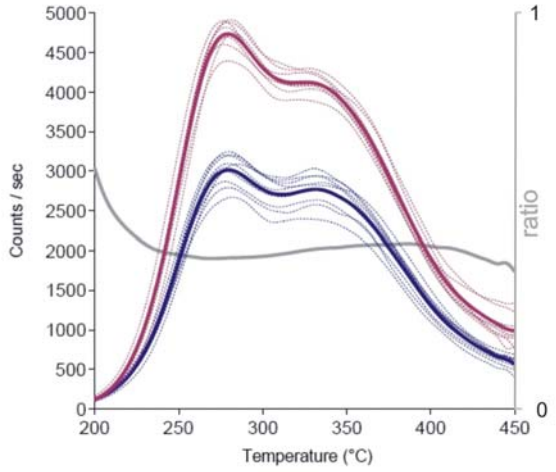

Figure 7: *TL glow-curves of sample 5 (Devonian siltstone). Upper curves (thick line: mean) NTL after 150°C preheat for 19 h only (T). Lower curves: (thick line: mean) NTL after 150°C preheat for 19 h and simultaneous 1 GPa pressure (P+T). Grey line: ratio of (P+T)/T.*

corrected for short term fading (8.4±1.1 ka) would still underestimate the known age (11 ka) significantly it is unlikely that a more rigorous fading correction would result in a significant age overestimate.

The dose response curve of #2 (fine grains) shows a sub-linear shape but also stays far below saturation. A fit to a saturating exponential curve yielded a D_e plateau extending over 80°C (Fig. 2). The D_e found by the IRSL MAAD protocol (Fig. 6) is identical within error bars, thus arguing for a total resetting of the IRSL at eruption as well. A good shine plateau (not shown here) was obtained.

The notional TL age of Pulvermaar (19.2 ± 2.7 ka, not corrected for short term fading of ca. 9% within 7 days at 70°C) lies within the LGM as expected (Lorenz and Zimanowski, 2000). Nevertheless, the true age is expected to be older due to observed anomalous fading. If corrected for short term fading the apparent age would be 21±3 ka which still lies within the LGM. But again, there is no evidence for TL age overestimation.

From Pulvermaar we also used the TL of the 125-250 µm fractions to check for possible grain size-dependant zeroing. This fraction consists of platy slate clasts. As shown in Fig. 5, a D_e-plateau is not obtained using extrapolation of the dose response curve to zero level. This suggests that TL-zeroing of this fraction during the fragmentation phase in the explosion chamber of the maar was not as complete as for the fine silt fraction (see Discussion).

These preliminary observations suggest that fine-grained maar tephra derived by hydroclastic fragmentation of country rock may be dated reliably by TL. Strategies to achieve this are discussed below. Coarser grains from maar tephra, however, may suffer from incomplete resetting at eruption and are easily subject to age-overestimation.

Discussion

The observed total or partial resetting of TL glow peaks in maar tephras derived from non-volcanic country rock may have several reasons: (a) thermal only zeroing (questionable, but in addition to hydroclastically frictioned rocks), (b) thermodynamic friction by dynamic pressure (frictional heating), and (c) hydrostatic high pressure at elevated temperatures.

High pressure experiments gave no evidence for resetting of the latent TL at room temperature; whereas high pressure at elevated temperature (150°C) partially drained the latent TL in addition to thermal bleaching. This thermally assisted resetting due to hydrostatic pressure is similar to the phonon-assisted bleaching of infra-red stimulated luminescence of feldspars (Hütt et al., 1988; Aitken, 1998). Our grinding-by-hand experiment was not able to reset the latent TL signal, but recently Takeuchi et al. (2006) reported successful resetting of the TL in milled quartz grains clearly showing a grain-size effect. The TL of smaller grains (<500 nm), corresponding to the surface disordered layer, was the more effectively reset. This coincides with our findings from the Pulvermaar tephra bed (sample 2: 4-11µm fraction totally reset; 125-250 µm fraction partially reset). Mere thermal resetting of the TL during or prior to maar eruption due to heat transfer from the rising magma into the adjacent non-volcanic rock is expected to affect all rock clasts. Our results, however, provide evidence for grain size dependent resetting in maar tephra, arguing for a resetting by thermally assisted hydrostatic pressure, or both, rather than merely thermal resetting by heat transfer.

Although one of the motivations to use non-volcanic minerals for dating volcanic events was to circumvent the anomalous fading often observed from volcanic feldspars, anomalous fading still is a major problem. Corrections for anomalous fading as suggested by Auclair et al. (2003) and Lamothe et al. (2003) are needed. An alternative strategy may be TL from pure fine-grained quartz extracts, in particular if the orange-red emissions (much higher saturation

dose) are used despite their lower detection efficiency.

Conclusions and Outlook

High hydrostatic pressure and elevated temperatures (but too low for total annealing of the geological TL) may occur at low crustal depths (<2 km) in the root zone of hydroclastic maar eruptions during the fragmentation phase just prior to the opening of the maar eruption vent. In addition to frictional heating (Takeuchi et al., 2006) and to heat transfer from the rising magma to neighbouring country rock, we propose another reasonable mechanism for at least partial resetting of the geological TL (and IRSL), which may be termed phonon-assisted mechano-luminescence. Further high pressure experiments with varying pressure and temperature will be useful to better understand the preconditions and efficiency of this resetting mechanism.

Circumventing or correcting for anomalous fading may still be a limiting factor for reliable TL dating of volcanic feldspars (and other volcanic minerals). Fine-grained quartz extracts from fine-grained maar tephra beds are expected to overcome the fading problem (e.g. Richter and Krbetschek, 2006), the applicability of the resetting mechanism as discussed here for polymineralic fine grains, however, remains to be tested for pure quartz separates.

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Reviewer

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Thesis Abstract

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In the British and Irish Isles there has been considerable research effort expended in improving understanding of the expansion and retreat of the last ice-sheet (Marine Isotope Stage 2) but the chronological control for the Last Glacial Maximum (LGM) extent and subsequent retreat phases of the British and Irish Ice-sheet (BIIS) is poor. Based on results from extensive sedimentological and stratigraphic studies of fossil ice-marginal sandar at *Orrisdale* (Isle of Man), *Co. Wexford* (south-eastern Ireland) and *Porth Dinllaen* (north-western Wales), a suite of glaciofluvial lithofacies were identified and sampled for OSL analysis, with the ultimate aim of dating the retreat of the Irish Sea Ice-Stream (ISIS) during the last deglaciation of the Irish Sea basin. Complete bar-form fining-up sequences were targeted as they are typically indicative of waning or shallow water flow, allowing sufficient opportunity for bleaching of quartz grains within these glaciofluvial deposits.

Different grain size fractions of quartz were extracted and the SAR protocol applied using small aliquots (~30 grains) to identify which depositional environments and grain size fractions were best suited for optical dating. Equivalent dose (D_e) distributions for all samples showed wide and positively skewed characteristics with overdispersion values >40%, indicative of heterogeneous bleaching. The 'age' model decision-protocol of Bailey and Arnold (2006) was used to choose the most applicable 'age' model (i.e. Minimum Age Model; MAM, Central Age Model; CAM or Lowest 5% Model; L5%) by analysing the D_e distribution via weighted skewness, kurtosis and overdispersion. This enabled a statistically informed choice to be made as to which 'age' model was appropriate for burial dose estimation.

No observable difference was evident between the ages calculated for different grain size fractions of the same sample; agreement within 2σ errors was observed. Glaciofluvial sandur systems appear to act as an efficient 'mixer' of sediment grains, allowing some to be well-bleached and others to be poorly-bleached, with no bias towards preferential bleaching of a particular grain size fraction. However differences between ice-proximal and ice-distal depositional sub-environments were more evident. The ice-distal Orrisdale and Porth Dinllaen samples showed better bleaching characteristics (as observed from skewness and overdispersion parameters) than the ice-proximal Wexford samples. Age overestimation was observed for the Wexford samples, even using the MAM. The only obvious lithofacies difference observed was that of the trough cross-bedded sample (laterally extensive deep sandur palaeo-channel) taken from Orrisdale which showed very poor bleaching characteristics in comparison to the other samples taken from this field site, once again, resulting in significant age overestimation.

Ages of ~ 23 ka were calculated for Wexford samples, indicative of almost immediate retreat of the ice-stream margin from its LGM extent off the north coast of the Scilly Isles and the Celtic Sea to the present-day coastline of Co. Wexford. The OSL age from Porth Dinllaen is ~21 ka, indicative of continued northwards retreat, albeit in a slower and more oscillatory manner, due to the stabilisation of the ice-stream as it was constrained by the narrow corridor of land between Wales and Ireland. Ages in the range of 17-14 ka were calculated for the Orrisdale samples, coincident with the Heinrich Event 1/Killard Point stadial readvance in north-eastern Ireland. In combination with previously published ages, a detailed Bayesian model-derived retreat sequence for the ISIS was established which allowed estimated retreat rates to be calculated for different ice-marginal phases.

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Conference Announcements

6th New World Luminescence Dating and Dosimetry Workshop 2009

22-24th October 2009

The 6th New World Luminescence Dating and Dosimetry Workshop will be held 22-24 October 2009 on the campus of the University of Washington in Seattle, WA, USA. The workshop is being sponsored by the Quaternary Research Center and the Department of Anthropology at the UW and also is being supported by a generous contribution from the Earth and Planetary Sciences Department at the University of New Mexico. The schedule of the workshop is timed to follow the Geological Society of America meetings in Portland, Oregon, October 18-21. Portland is only 200 miles south of Seattle, so those attending the GSA meeting can easily travel to Seattle.

The workshop will consist of two days of presentations and discussions on current research in luminescence dating and related fields and a third day for a field trip to Whidbey Island in Puget Sound,. Whidbey Island contains dramatic exposures of glacial deposits as well as sweeping views (when it isn't raining) of the Cascade and Olympic Mountains. For those interested in additional sightseeing, Seattle provides access to the Cascades (including five volcanoes), San Juan Islands, the Olympic Peninsula, and the Pacific Ocean.

For additional information please contact James Feathers at the University of Washington.

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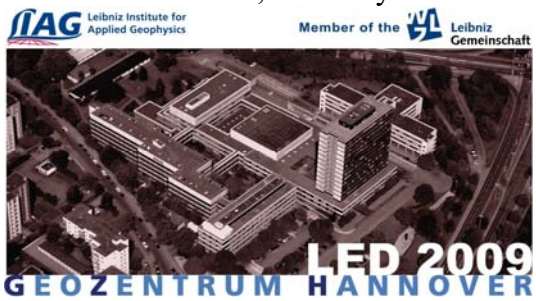
Conference Announcements

2009 German Meeting on Luminescence and Electron Spin Resonance Dating, German LED 09

Hannover, Germany



9-11th October 2009

We are pleased to invite you to the 2009 German Meeting on Luminescence and Electron Spin Resonance Dating, which will be held at the Leibniz Institute for Applied Geophysics (LIAG) in Hannover, Germany, 9-11th October. The aim of this meeting is to have scientific exchanges on the two dating methods and their applications. Participants are very welcome to introduce their works by poster or oral presentations. Topics may range from the progress in the methodology to the applications in Quaternary Science, Archaeology or Radiation Dosimetry.

The deadline for the abstract submission and registration will be on 10th August 2009. All general information about the meeting such as registration, accommodation, preliminary program, abstract submission, and the guideline for poster and oral presentation will be found in the web page: http://www.liag-hannover.de/de/aktuelles.html under "special events".

For further questions, please contact LED2009@liag-hannover.de. We are looking forward to welcoming you at our institute!

Manfred Frechen and Sumiko Tsukamoto
Section 3 Geochronology and Isotope Hydrology
Leibniz Institute for Applied Geophysics (LIAG)

Conference Announcements

The Second Asia-Pacific Conference on Luminescence and Electron Spin Resonance Dating (APLED-2)

The second Asia Pacific Conference on Luminescence and Electron Spin Resonance will be held during November 12-15, 2009, at the Physical Research Laboratory, Ahmedabad. This conference is in continuation of the 1st Asia-Pacific Conference on Luminescence Dating (APLD-2006) held at Hong Kong during October 2006. The conference will discuss the current research in the Methodological Aspects, Physics and Application of Luminescence and Electron Spin Resonance to Geological/ Archeological Dating with a particular emphasis on Problems/ Advancements in the applications of these methods in the Asian-Pacific contexts. The conference will also cover basic physics issues, new techniques, improvements in instrument-ation and protocols, geological, archeological and other dosimetric applications. It will also encourage contributions on the conjunctive use of luminescence and ESR methods with other techniques for better elucidation of geological processes and their rates. The conference will comprise invited reviews, oral and poster presentations. A workshop for practitioners and end users is also planned. Efforts are underway to publish the proceedings in international journals like Quaternary Geochronology, Radiation Measurements and Geochronometria.

The venue, Physical Research Laboratory (PRL) is a premier research institution of India, located in a 500 year old town with rich heritage and a vibrant present.

PRL has programmes in Theoretical Physics, Astronomy and Astrophysics, Solar Physics, Space and Atmospheric Sciences, Planetary and Geo-Sciences & Planetary Explorations. The Geo-science Group specializes in the use of isotopes in Earth and Planetary Sciences.

The accommodation is on the campus of the Indian Institute of Management, designed by famed architect, Le Corbusier and is about 5 minutes walk from PRL. The weather during the period will be mild with maximum day temperatures of 28-30°C.

Registration Fee

	Before Aug. 1	After Aug. 1
Professional	€ 200	€250
Student	€ 100	€150
Field trip	€ 50	
Accompanying Person	€ 100	

Fee covers all meals, conference dinner, airport pickups/ drops conference literature.

Important Deadlines

Expression of Interest for second Circular:	July 1, 2009
Request for financial assistance:	August 1, 2009
Early Registration:	August 1, 2009
Submission of abstract:	September 1, 2009
Reservation of Accommodation:	October 1, 2009

Details on the conference are available on **www.prl.res.in/~apled2**

The second circular will be sent to those responding to the first circular. In case of difficulty please send a e mail to ***apled2@prl.res.in***