

## The partial heat – longest plateau technique: Testing TL dating of Middle and Upper Quaternary volcanic eruptions in the Eifel Area, Germany

LUDWIG ZÖLLER & HENRIK BLANCHARD\*

**Abstract:** Middle and Upper Quaternary volcanic events are often difficult to date, in particular when minerals suitable for  $^{40}\text{Ar}/^{39}\text{Ar}$ -dating are missing. Here, we present first tests of a newly developed technique to use the thermoluminescence (TL) of maar tephra and crustal xenoliths for dating the eruption event. We take into consideration that resetting of the latent geological TL of country rock fragments during eruption may be incomplete. We therefore develop the “partial heat – longest plateau” (PHLP) technique to approach the inherited TL signal at eruption. This technique can overcome TL age overestimates due to incomplete zeroing, as is demonstrated for some eruptions in the Quaternary Eifel Volcanic Field, Germany, in the time range from 11 to 300 ka old. Although we avoid the often observed strong anomalous fading of volcanic feldspars and other volcanic minerals by using heated country rock xenoliths, we still face the problem of longer-term anomalous fading which may be responsible for apparent age underestimates. The orange-red TL emissions (RTL) of pure fine-grained quartz extracts from crustal xenoliths are also tested from two samples. This approach needs, however, further systematic investigations into the TL characteristics of RTL.

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**Kurzfassung:** Vulkanische Ereignisse des mittleren und oberen Quartär sind schwer zu datieren, insbesondere, wenn geeignete Minerale für eine  $^{40}\text{Ar}/^{39}\text{Ar}$ -Datierung fehlen. Mit diesem Artikel werden erste Tests einer neuen Anwendungsmöglichkeit der TL-Datierung an Maar-Tephra und Xenolithen präsentiert, um derartige Eruptionsvorgänge zu datieren. Hierbei wird berücksichtigt, dass ein Rückstellen des latenten geologischen TL-Signals der Gesteinsfragmente während des Eruptionsvorganges eventuell nur unvollständig erfolgte. Aus diesem Grund wurde die „Partial heat- longest plateau-Technik (PHLP) entwickelt, um genau diesem Problem Rechnung zu tragen. Die neu entwickelte Technik kann helfen, eine Überschätzung von TL-Datierungen infolge der unvollständigen Rücksetzung des TL-Signals zu vermeiden. Dies wird für vulkanische Ablagerungen im Eifel-Vulkanfeld demonstriert, die eine Zeitspanne von 11 bis 300 ka umfassen. Obwohl das Problem starken anomalen Ausheilens des TL Signals vulkanischer Feldspäte und anderer vulkanischer Minerale umgangen wurde, existiert immer noch das Problem einer Unterschätzung geologischer Alter als Folge eines langfristigen anomalen Ausheilens im TL Signal. Die orange-roten TL-Emissionen (RTL) reiner feinkörniger Quarzproben aus Xenolithen wurden an zwei Proben getestet. Dieser Datierungsansatz verlangt jedoch noch weitere systematische Untersuchungen.

Keywords: thermoluminescence, anomalous fading, maar tephra, xenoliths, volcanic eruptions, dating, Eifel Volcanic Field.

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## 1 Introduction

Thermoluminescence (TL) dating has often been tested for volcanic minerals (see, e.g., FATAHI & STOKES 2003) but faces the problems of strong anomalous fading of volcanic feldspars (WINTLE 1973) or of very low TL signals from other volcanic minerals except for quartz. The latter is, however, not present in mafic volcanic rocks unless as xenoliths derived from older country-rock in the direct vicinity of the vent. In order to circumvent these problems, we tried to use xenoliths (cf. CHEN et al. 2001) and maar tephra derived from country-rock to date their last resetting during the volcanic activity by TL. This approach, however, raises new problems, in

particular the degree of heating during the eruption (cf. GONZÁLEZ et al. 2006 and comments by DULLER 2006) and the question if, besides heating, other events may reset the parent “geological” TL signal of the dated minerals.

We sampled xenoliths derived from Lower Devonian silicoclastic country-rock (partly affected by metamorphism) and maar tephra from several volcanic eruptions in the Quaternary Eifel Volcanic Field, Germany (Fig. 1), separated in two NW-SE oriented sub-fields, the West and the East Eifel Field (MEYER 1986, SCHMINCKE 2000, BÜCHEL et al. 2000). In the West Eifel Field phonolitic eruptions are missing. Hydroclastic maar eruptions occurred in both fields but are more typical for the West

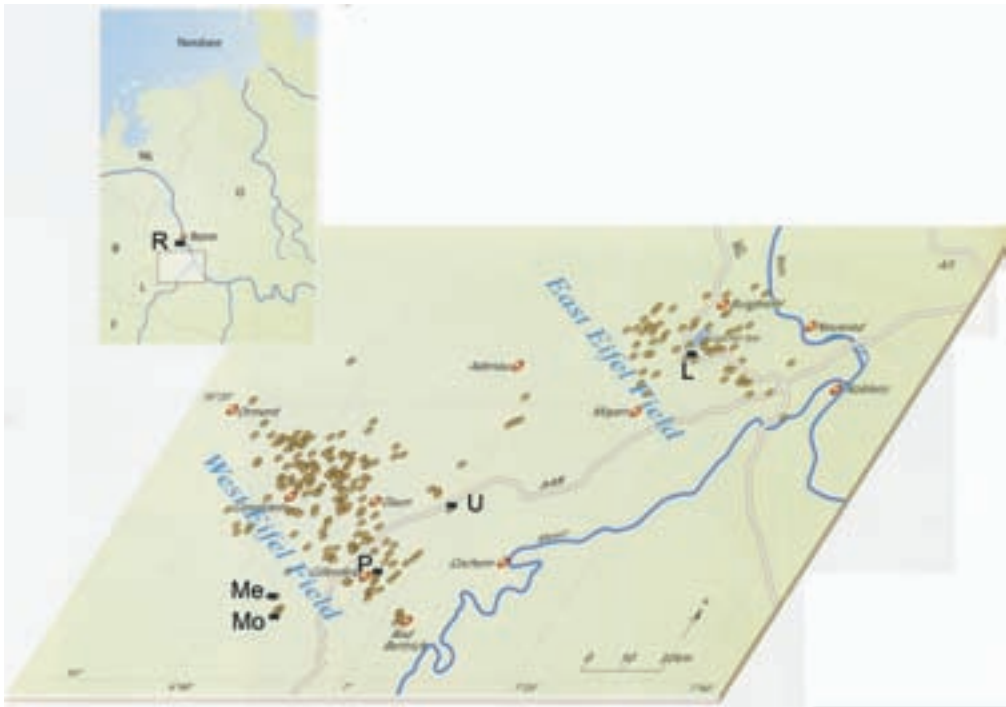


Fig. 1: The Quaternary West and East Eifel Volcanic Fields in the Rhenish Massif, Germany (adopted from SCHMINCKE, 2000). Studied localities shown as black rectangles: Me=Meerfelder Maar (Deudesfeld site), Mo=Mosenberg scoria cone (southernmost cone), L=Lake Maria Laach (Wingertsberg site), P=Pulvermaar (near Gillenfeld), R=Rodderberg, south of Bonn, U=Ulmener Maar.

Abb. 1: Quartäre Vulkanfelder der West- und der Osteifel, Rheinisches Schiefergebirge, Deutschland (bearbeitet nach SCHMINCKE, 2000). Schwarze Rechtecke markieren bearbeitete Lokalitäten: Me=Meerfelder Maar (Tuffgrube Deudesfeld), M=Mosenberg-Schlackenkegel (südlichster Kegel), L=Laacher See (Wingertsberg-Wand), P=Pulvermaar (bei Gillenfeld), R=Rodderberg südlich Bonn, U=Ulmener Maar.

Eifel Field where maar tephra consists of up to >90% of country rock clasts (mainly Lower Devonian slates, siltstones and quartzites or quartzitic sandstones). In some maar tephra (e.g., Meerfelder Maar) also peridotite xenoliths from the upper mantle with diameters up to ca. 20 cm can be found which, however, were not used for dating.

The incipient observation for this study was the finding of a few cm to several dm thick beds of very well sorted, fine-grained maar tephra in the ramparts of Lake Ulmener Maar and Lake Pulvermaar in the West Eifel Field. These tephra beds consist almost entirely of fine-ground Lower Devonian slate and siltstone clasts with a clear maximum in the silt fraction and very low clay fraction. The grinding of the hard country-rock down to these fine fractions can be explained by the theory of hydroclastic eruptions as presented by LORENZ & ZIMANOWSKI (2000). Preliminary TL measurements from the polymineral fine-grained fraction (4–11  $\mu\text{m}$ ) using the “multiple aliquot additive dose” (MAAD) protocol (WINTLE 1998, VANDENBERGHE et al. 2004) showed that the TL and the infra-red stimulated luminescence (IRSL) were totally zeroed at deposition of the two studied maar tephra (ZÖLLER et al. 2009). This result is, however, somewhat surprising as zeroing by light exposure in a base surge is certainly insufficient and it must be doubted if thermal zeroing in a hydroclastic eruption is able to completely erase the parent TL. Heat transfer in the eruption chamber of a maar during subsurface grinding of the country-rock is probably low in a dry gas-solid mixture, although the presence of water under high pressure conditions, however, may increase heat transfer (pers. comm. M. Zimanowski 2006). Nevertheless, we explored an additional possible zeroing mechanism: hydrostatic pressure, based on the description of “mechanoluminescence” by BANERJEE et al. (1999) (see also SINGHVI et al. 1994). The results are published elsewhere (ZÖLLER et al. 2009) and will not be further reported here. It was shown that hydrostatic pressure (1 GPa for 19 h) at room temperature did not significantly reset the natural TL (NTL) of a Lower Devonian

slate fragment whereas hydrostatic pressure at elevated temperature (1 GPa at 150°C for 19 h) partially reset the NTL beyond the thermal draining. In the following chapters we try to develop a laboratory technique to overcome the problem of partial resetting of maar tephra or crustal xenoliths independent from the question what caused the resetting (heat transfer, frictional heating or hydrostatic pressure).

## 2 Samples and sample preparation

Three kinds of samples were collected for testing TL dating,

- a) only slightly consolidated maar tephra-beds,
- b) crustal xenoliths extracted from maar tephra or scoria and ash cones, and
- c) a piece from Lower Devonian slate (sample no. 5) originating from the same epoch (Upper Siegenium) as country-rock outcropping at Lake Puvermaar (for dose recovery experiments; the sampling locality at the northern end of the Middle Rhine Valley at Bonn-Friesdorf is far away from possible thermal overprint by Quaternary volcanism).

All samples were processed under subdued red light (diodes, 620 nm) after removal of the outer minimum 2 mm rim using a handsaw. Hard rock samples were carefully crushed in a bench vice and with an agate mortar and sieved. Fine-grain samples were prepared according to ZIMMERMAN (1971) and quartz coarse grain samples according to MEJDAHL (1985a) and AITKEN (1998). From two samples (13RTL and 14 RTL) fine-grained quartz separates were obtained by etching in  $\text{H}_2\text{SiF}_6$  (FUCHS et al. 2005). More detailed information about the samples is given in Table 1.

## 3 Experimental

TL and IRSL measurements were executed using a Daybreak 1150 TL/IRSL reader equipped with infra-red diodes (870 $\pm$ 30 nm), an EMI 9586Q photomultiplier (PM) and a combination of four detection filters (BG-3, GG-400, BG-3, BG-39, “blue combination” in Table 1) in front of it. TL readout occurred at a ramp rate

Table 1: Samples, sample preparation and experimental details.

Tabelle 1: Proben, Probenaufbereitung und Messparameter.

Sample	Locality	Material	Expected age	Method	IRSL	Preheat	Ramp	Filter
1b/a	Ulmener Maar	fine-grain maar tephra, dark grey	11 ka	PM FG BTL-ADD	max. ca. 72 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
2	Pulvermaar	fine-grain maar tephra, grey	18...24 ka	PM FG BTL-ADD	max. ca. 75 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
2	125...250µm			PM 125...250µm BTL-ADD		stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
3	Roddeberg "initial"	fine-grain maar tephra, grey, gravel	240...300 / 550 Ka	PM FG BTL-ADD	max. ca. 25 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
4a	Roddeberg "final"	fine-grain maar tephra, grey, bedded	240...300 ka	PM FG BTL-ADD	max. ca. 120 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
5	Devon Friesdorf	siltstone, Lower Devonian	400 Ma, saturated	PM FG BTL	max. ca. 650 ds / sec	-	5 °C / sec.to.450 °C	blue combination
10a	Meerfelder Maar	fine-grain maar tephra, initial, dark grey	40...120 ka	PM FG BTL-ADD	max. ca. 240 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
10b	Meerfelder Maar	Xenolith in cauliflower bomb	40...120 ka	PM FG BTL-ADD	max. ca. 3500 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
13	Mosenberg	Xenolith, brick-red	40...80 ka	PM FG BTL-ADD	max. ca. 15 ds / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
13 RTL				<b>Qz FG RTL REG</b>	-	<b>stage / cont. 250 °C, 15 sec</b>	<b>2 °C / sec.to.450 °C</b>	<b>D630/60 + D620/75</b>
14a	Lake Maria Laach	Devonian siltstone xenolith, grey	12.9 ka	PM FG BTL-ADD	ca. 45 cts / sec	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
14a RTL				<b>Qz FG RTL REG</b>		<b>stage / cont. 250 °C, 15 sec</b>	<b>2 °C / sec.to.450 °C</b>	<b>D630/60 + D620/75</b>
PBLP-experim.	(simulated eruption)	siltstone of sample 5	(50.0 Gy)	PM FG BTL-ADD	-	stage / cont. 220 °C, 120 sec	5 °C / sec.to.450 °C	blue combination
				(PM=polymineral FG=fine grains BTL=blue TL RTL=red TL ADD=additive dose REG=regeneration)				

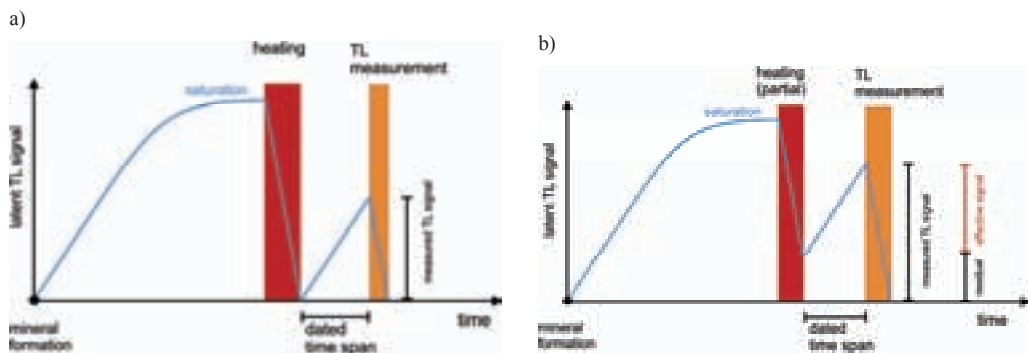


Fig 2: Principles of TL dating assuming total (a) and partial resetting (b) of the parent TL during the event to be dated. The parent TL signal acquired during the geological history of the mineral grain (geological TL) is saturated and then entirely annealed during heating (a). Subsequently, the parent TL signal accumulates again with time due to natural radioactivity of the sample and its environment. The intensity of the natural TL signal (NTL) recorded in the laboratory is a measure of the time elapsed since the last heating. If, however, the heating event did not completely erase the acquired geological TL a residual TL remains at the heating event. The NTL signal measured in the laboratory consists of TL accumulated after the heating event plus an inherited TL signal (b).

Abb. 2: Grundlagen der TL-Datierung unter Annahme vollständiger (a) und partieller Rückstellung (b) des latenten TL-Signals während des datierten Ereignisses. Das latente TL-Signal, welches im Laufe der geologischen Geschichte des Mineralkorns akquiriert wurde, ist in Sättigung und wird dann während der Erhitzung völlig gelöscht (a). Danach wächst das latente TL-Signal wieder infolge der natürlichen Radioaktivität der Probe und ihrer Umgebung. Die Intensität des natürlichen TL-Signals (NTL), welches im Labor gemessen wird, ist ein Maß für die Zeit seit der letzten Erhitzung. Wurde jedoch während des Ereignisses der Erhitzung die akquirierte geologische TL nicht vollständig gelöscht, verblieb ein residuales (ererbtes) TL-Signal. Das im Labor gemessene NTL-Signal setzt sich dann aus dem seit der Erhitzung akquirierten Signal plus dem ererbten Signal zusammen (b).

of 5°C (for blue TL) and 2°C per second (for orange-red TL, see below), respectively (see Table 1) to a maximum temperature of 450°C, whereas IRSL was recorded at room temperature. For blue TL preheat of 220°C for 2 minutes was applied (“stage and continue”, i. e. the aliquot is heated to 220°C and held there for 2 minutes, before heating continues to maximum temperature); deviating protocols for orange-red TL are mentioned in Table 1. For sub-sample 13RTL (Mosenberg volcano) the orange-red TL (RTL) of a pure quartz separate was also measured using a detection filter combination (Oriel D630/60 + D620/75) with transmission from 570 to 685 nm (maximum from 600-660 nm; based on manufacturer’s data). The quantum efficiency of the used PM in this wavelength range is, however, only ca. 1%. Laboratory irradiation

was performed using a  $^{90}\text{Sr}/^{90}\text{Y}$  beta-source delivering ca. 9.96 Gy/min.

The age of a sample can - simplified - be calculated as the radiation dose accumulated since the dated event,  $D_E$  [Gy], divided by the effective dose-rate,  $D$  [Gy/a]. To determine the equivalent dose  $D_E$  it is necessary to define the plateau range, i.e. the glow curve temperature range exhibiting identical  $D_E$ . In case of non-linear dose-response a simple plot of  $I_{(N+B)}/I_{(N)}$  versus glow temperature (e.g., AITKEN 1985, Fig. 1.3), with  $I_{(N+B)}$  denoting the TL intensity of an additively dosed aliquot and  $I_{(N)}$  the TL intensity of a natural aliquot, respectively, is not justified (e.g., BERGER 1991, 1994, BERGER & ANDERSON 1994). We therefore used the  $D_E$  plateau test only. Longer-term anomalous fading tests and corrections (according to AUCLAIR et al. 2003, LA-

MOTHE et al. 2003) were not yet executed due to limited laboratory time for this pilot study. Instead, an accelerated short term fading test (7 days storage at 70°C in the oven, see BERGER 1985, 1987, 1988; ZÖLLER 1995) was performed on polymineral fine grain samples and apparent equivalent doses were corrected for short term fading: TL intensities of the highest additive dose ( $N+\beta_{\max}$ ) were measured immediately after irradiation and after seven days delay at 70°C. In case anomalous fading was detected and calculated as

$$\frac{[I_{(N+\beta_{\max})} - I_N] / [I_{(N+\beta_{\max}+\text{delay})} - I_N],$$

with  $I$  = TL-intensity in the ED plateau range, the portion  $I_{(N+\beta_i)} - I_N$  of all additively dosed aliquots was recalculated by subtraction of the fading percentage, and the corrected intensities of  $I_{(N+\beta_i)}$  were used for refitting the additive dose response curve. Although there may be doubts about the general validity of extrapolated long term fading tests this discussion lies outside of the scope of the present study. We are aware, however, that further studies should also include longer-term fading tests. So far, all apparent TL-ages obtained from the polymineral fine grain fraction must be regarded as minimum ages!

Effective internal  $\alpha$ - and  $\beta$ -dose-rates for the dated samples were calculated using thick source alpha-counting of fine-ground bulk samples (AITKEN 1985, ZÖLLER & PERNICKA 1989) for U and Th decay chains, and by ICP-MS and AAS measurements for K. Dose conversion factors given by ADAMIEC & AITKEN (1998) were applied. The  $\gamma$  dose-rates were calculated from U, Th and K concentrations of maar tephtras if homogeneity for  $4\pi$  geometry could be assumed by field evidence. For xenolith samples not fulfilling the requirements of  $4\pi$  geometry the  $\gamma$  dose-rate was measured on-site using a portable NaI 4-channel gamma spectrometer (Harwell). The very small contribution of cosmic dose-rate was estimated with respect to sample depth below surface using the formula given by PRESCOTT

& HUTTON (1994). The  $\alpha$ -value (alpha efficiency, see AITKEN 1985) was taken as  $0.08 \pm 0.02$  for polymineral fine-grain samples and  $0.03 \pm 0.01$  for fine-grain quartz samples. Secular equilibrium of U decay-chains was assumed.

## 4 The partial heat – longest plateau (PHLP) technique in TL dating

### 4.1 Partial resetting of parent TL – basic considerations

Luminescence dating is a paleodosimetric dating method based on the absorption of the energy of ionizing radiation by the sample with time (WAGNER 1998). A basic assumption of luminescence (TL, OSL, IRSL) dating is the complete resetting (zeroing) of the latent luminescence signal at the event to be dated. Heat and light exposure are the most prominent resetting mechanisms allowing the dating of the last heating event of a sample or the last exposure of its mineral grains to daylight (e.g., sedimentation). Incomplete resetting results in an over-estimation of  $D_E$ , and, thus, an age over-estimation. It is, therefore, essential to ascertain a complete resetting at the event to be dated or to find applicable corrections for the inherited residual luminescence value (Fig. 2).

For TL dating of sediments with an inherited signal at deposition, MEJDAHL (1985b, 1988) presents several so-called “partial bleach” techniques. One of them is called the “longest plateau” technique (MEJDAHL 1988). It relies on the observation that the natural TL of quartz or feldspars consists of components exhibiting different sensitivity to daylight. Assuming that all mineral grains of a sample saw the same light sum prior to deposition, the correct  $D_E$  can be found if for different glow temperatures within the thermally stable part of the glow-curve the obtained  $D_E$ s are identical ( $D_E$ -plateau). This can be achieved by trying the residual TL of glow-curves from natural TL (NTL) aliquots following various light exposures. Since the advent of optical dating the longest plateau

technique after MEJDAHL, however, has been more or less abandoned.

When a sample having acquired an amount of NTL is subjected to insufficient (partial) heating (e.g., 200 to 300°C) its TL is not totally zeroed. Glow peaks lower than or equal to the maximum heating temperature are drained completely, whereas those above the heating temperature are partially erased or even remain unaffected, depending on their peak temperature and the duration of heating. A residual TL glow curve is obtained when the sample is glow to maximum temperature (450 or 500 °C, respectively). The shape of this residual TL glow curve is in some aspects similar to the residual TL after optical bleaching (see Fig. 6b) in so far as the percentage residual TL, referred to the NTL, increases with glow temperature. In contrast to partial optical bleaching, no TL peaks for glow temperatures below the partial heating temperature are recorded. This can

even be considered as an advantage over partial optical bleaching. Thus, MEJDAHL's approach can be modified to the TL dating of partially heated rocks, sediments or soils.

#### 4.2 A new laboratory protocol for partially heated rocks

We tried to optimize the longest plateau technique for partially heated volcanogenic material such as crustal xenoliths and maar tephra. We so far developed the following protocol:

1. Construction of a multiple aliquot additive dose-response curve (MAAD). In case a long  $D_E$  plateau is observed by extrapolation to zero TL level complete resetting of the latent (geological) TL at eruption is proved and the following steps are not necessary (Fig. 2a).
2. A number of zeroed aliquots of the sample to be dated (here: 18 aliquots) are regenerated by a laboratory dose up to the estimated pre-

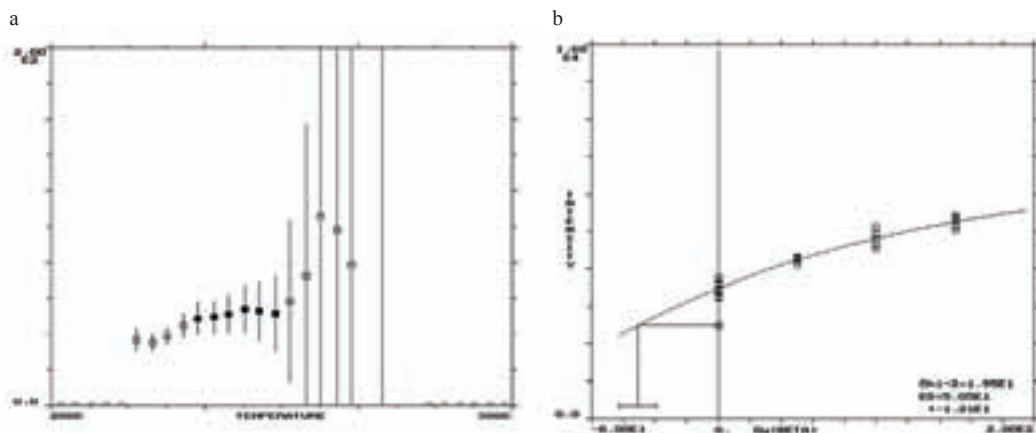


Fig. 3 a:  $D_E$ -plateau of the dose recovery test. The sample 5 with geological TL in saturation was partially heated (15 min at 300°C) and then laboratory-dosed with 50 Gy. The PHLP technique recovers a dose of  $50.5 \pm 12.1$  Gy (see b).

b : Results of the PHLP technique applied to sample 5 dosed with 50 Gy after partial heating for 15 min at 300°C. The PHLP technique recovers a dose of  $50.5 \pm 12.1$  Gy. The longest  $D_E$ -plateau is seen at the correct equivalent temperature.

Abb. 3a:  $D_E$ -Plateau des Dosis-Wiederherstellungstests. Die Probe 5 mit geologischer TL in Sättigung wurde partiell ausgeheizt (15 min bei 300°C) und erhielt dann im Labor eine Dosis von 50 Gy. Die PHLP-Technik findet eine Dosis von  $50,5 \pm 12,1$  Gy wieder (siehe b).

b : Ergebnisse der PHLP-Technik für Probe 5 mit einer Labordosis von 50 Gy im Anschluss an ein 15-minütiges partielles Ausheizen bei 300°C. Die PHLP-Technik findet eine Dosis von  $50,5 \pm 12,1$  Gy wieder. Das längste  $D_E$ -Plateau stellt sich bei der korrekten Äquivalenttemperatur ein.

Table 2: Summarized TL dating results from three samples of the Rodderberg volcanic complex.

Tabelle 2: Zusammengefasste TL-Datierungsergebnisse von drei Proben des Rodderberg-Vulkankomplexes.

Sample	Material	Stratigraphic position	$D_E$ (Gy)	Dose rate (Gy/ka)	Apparent age (ka)
<b>3</b>	hydroclastic maar tephra	initial series	1108 ± 355	3.70 ± 0.25	<b>300 ± 98</b>
<b>4a</b>	hydroclastic maar tephra	final series	957 ± 161	2.87 ± 0.21	<b>334 ± 61</b>
<b>R4-D-1</b>	slate xenolith	strombolian series	1403 ± 141	4.37 ± 0.24	<b>321 ± 37</b>

eruptive dose-level. This step is not used for regenerative dating but only to produce aliquots suited for step 3. In the case of Lower Devonian basement the aliquots must be irradiated up to the saturation dose level (minimum 2,000 Gy; a saturation dose of 2,000 to 5,000 Gy was found for our samples). To minimize irradiator time additive dosing on top of the natural dose is advised.

- The dosed aliquots are split in 6 groups. The aliquots of each group are subjected to thermal pre-treatment on the hotplate of the TL reader for 5 min with temperature increments of 10K. Temperatures for one group each are held at 250, 260, 270, 280, 290 and 300 °C, respectively. By this way, 6 different residual TL levels are produced representing partial annealing of the pre-eruption TL level.
- Extrapolation of the MAAD dose-response curve (step 1) to the various residual TL levels to obtain the parent  $D_E$  for each glow temperature in the range from 280 to ca. 400°C and for each residual TL level (cf. Figs. 5, 6a). Ideally, the plateau is expected to be the longest if the thermal pre-treatment in the laboratory matches exactly the natural conditions during and after (cooling down-period) the eruption. This will hardly be the case, and, thus, the laboratory preheat temperature yielding the longest plateau is called “equivalent temperature” (Fig. 2b). Only for an equivalent temperature delivering a residual TL resembling best or very closely the residual TL at eruption an acceptable  $D_E$ -plateau is obtained.
- Ideally, dating results from two or more xenoliths of the same eruption but yielding different equivalent temperatures and/or dose-

rates are identical within error bars and can, thus, validate the eruption age (see section “results”).

We designed the following dose recovery experiment to prove the validity of the protocol:

The partial heating of a crustal xenolith during a volcanic eruption was simulated by partial heating of a piece of sample 5 (Lower Devonian basement, TL in saturation) in an oven. To apply a geological “age” to the sample after the artificial “eruption”, a defined test-dose of 50.0 Gy was administered in the laboratory following the partial heating.

Detailed protocol of the PHLP dose recovery experiment:

- Preparation of the polymineral fine grain fraction (4-11µm) from a Lower Devonian basement slate (50 aliquots).
- Simulation of partial heating during a volcanic eruption by heating the fine grain fraction in an oven at 300°C for 15 min.
- Storage of the aliquots for >24h at room temperature to allow for possible charge redistributions.
- Additive  $\beta$ -irradiation (50 Gy) of all aliquots to simulate a geological age of the “eruption”.
- Storage at 160°C for 15min in an oven to remove thermally unstable TL-peaks, then at room temperature for >24h to allow for charge redistribution.
- Determination of the apparent  $D_E$  using the MAAD protocol applying the PHLP technique (Fig. 3 a, b).
- Using the same fine grain material as produced in the 1<sup>st</sup> step to produce the residual TL levels after incremental preheat (as de-



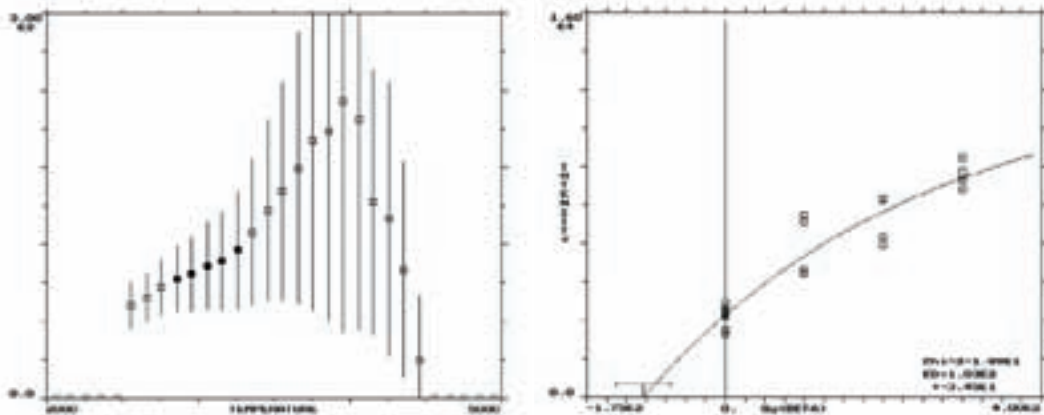


Fig. 4:  $D_E$ -plateau function (top) and dose response curve (bottom) for the 125-250 $\mu$ m fraction of sample 2 (Pulvermaar). A plateau is not obtained if total zeroing of the TL at deposition is assumed. Note that the  $D_E$  shown in the figure is not corrected for anomalous fading.

Abb. 4:  $D_E$ -Plateaufunktion (oben) und Dosis-Wirkungs-Kurve (unten) für die 125-250 $\mu$ m-Fraktion der Probe 2 (Pulvermaar). Bei Annahme vollständiger Rückstellung der TL bei Ablagerung ergibt sich kein Plateau. Man beachte, dass der  $D_E$ -Wert in der Abbildung nicht um den Betrag des anomalen Ausheilens korrigiert wurde.

scribed above). Laboratory regenerative irradiation up to the pre-eruptive dose-level is not necessary in this case as dose-saturated material without natural thermal overprint is used.

## 5 Results

### 5.1 Results of the dose recovery experiment

By applying the PHLP technique as described above, the artificial laboratory dose was reproduced correctly ( $50.5 \pm 12.1$  Gy, see Fig. 3) even if within large error bars. The experiment also confirmed the hypothesis that the extrapolation of the dose response-curve to the correct residual results in a sufficiently long  $D_E$  plateau, whereas extrapolations to wrong residuals cannot yield a good plateau and result in wrong apparent  $D_E$  values. The plateau must, however, cover the temperature range of the glow-curve peak relevant for dating to serve as a reliable check for the correct  $D_E$ .

The newly developed PHLP technique can, thus, be expected to reliably date the eruption of partially heated crustal xenoliths. The tech-

nique was tested using maar tephra and crustal xenoliths from some Middle and Upper Quaternary eruptions in the West and East Eifel Volcanic Field.

### 5.2 Preliminary results from selected volcanic eruptions in the Eifel Volcanic Field

We first studied *maar tephra beds* with good to moderate age control to check if the PHLP technique delivers satisfying results for such material. The eruption age of *Ulmener Maar* (sample 1, Photo 1) is well constrained to ca. 11,000 cal BP by  $^{14}\text{C}$ -AMS dating and varve chronology (ZOLITSCHKA et al. 2000). From *Pulvermaar* (sample 2, Photo 2) a physical dating is not yet available, but from geomorphologic evidence its age is assumed to fall within the last glacial maximum (LGM; LORENZ & ZIMANOWSKI 2000).

The apparent TL age for *Ulmener Maar* was calculated to  $8.36 \pm 1.11$  ka (corrected for short term fading of ca. 13%). The TL age significantly underestimates the independent age (11 ka, ZOLITSCHKA et al. 2000), but there is no evi-

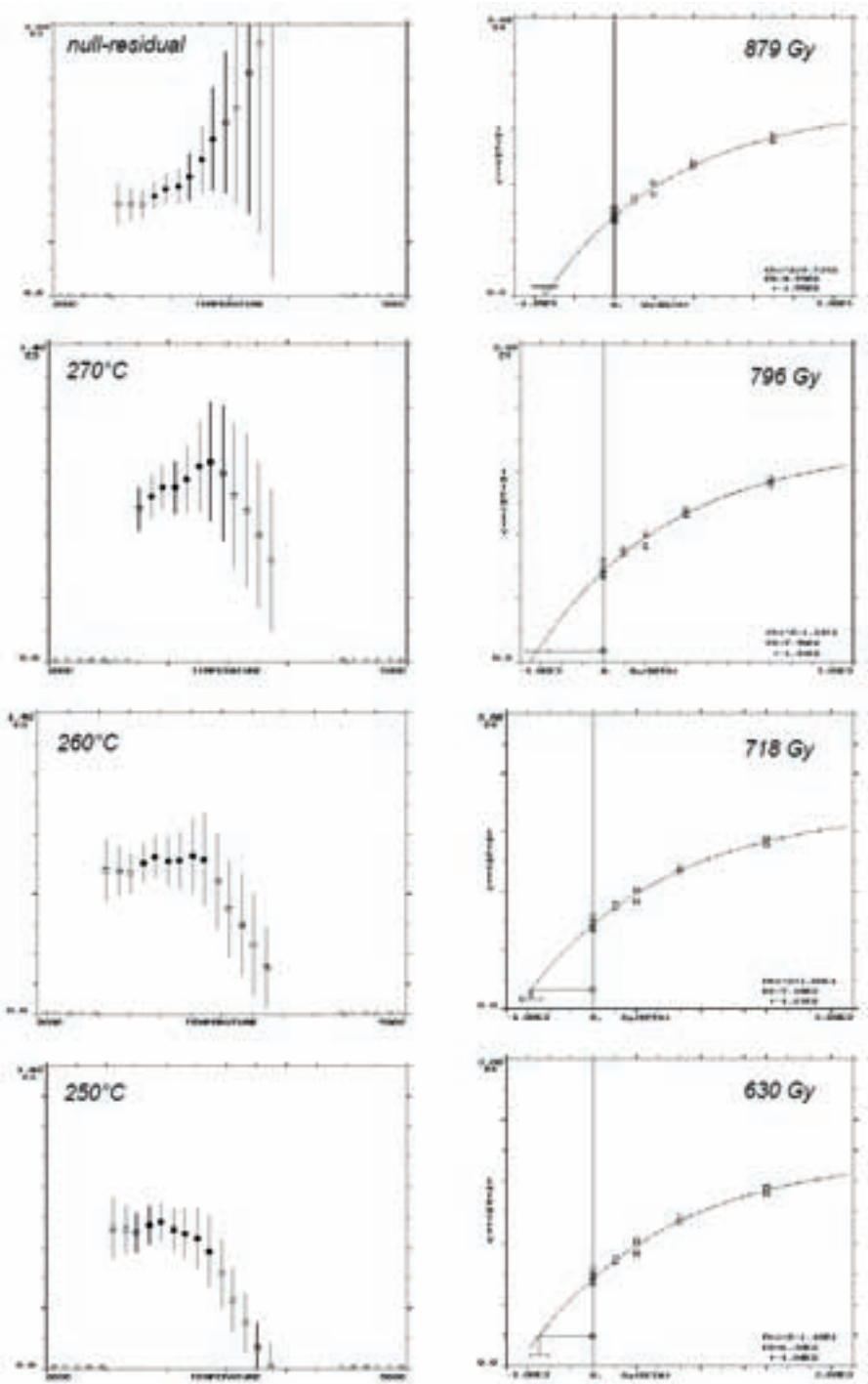
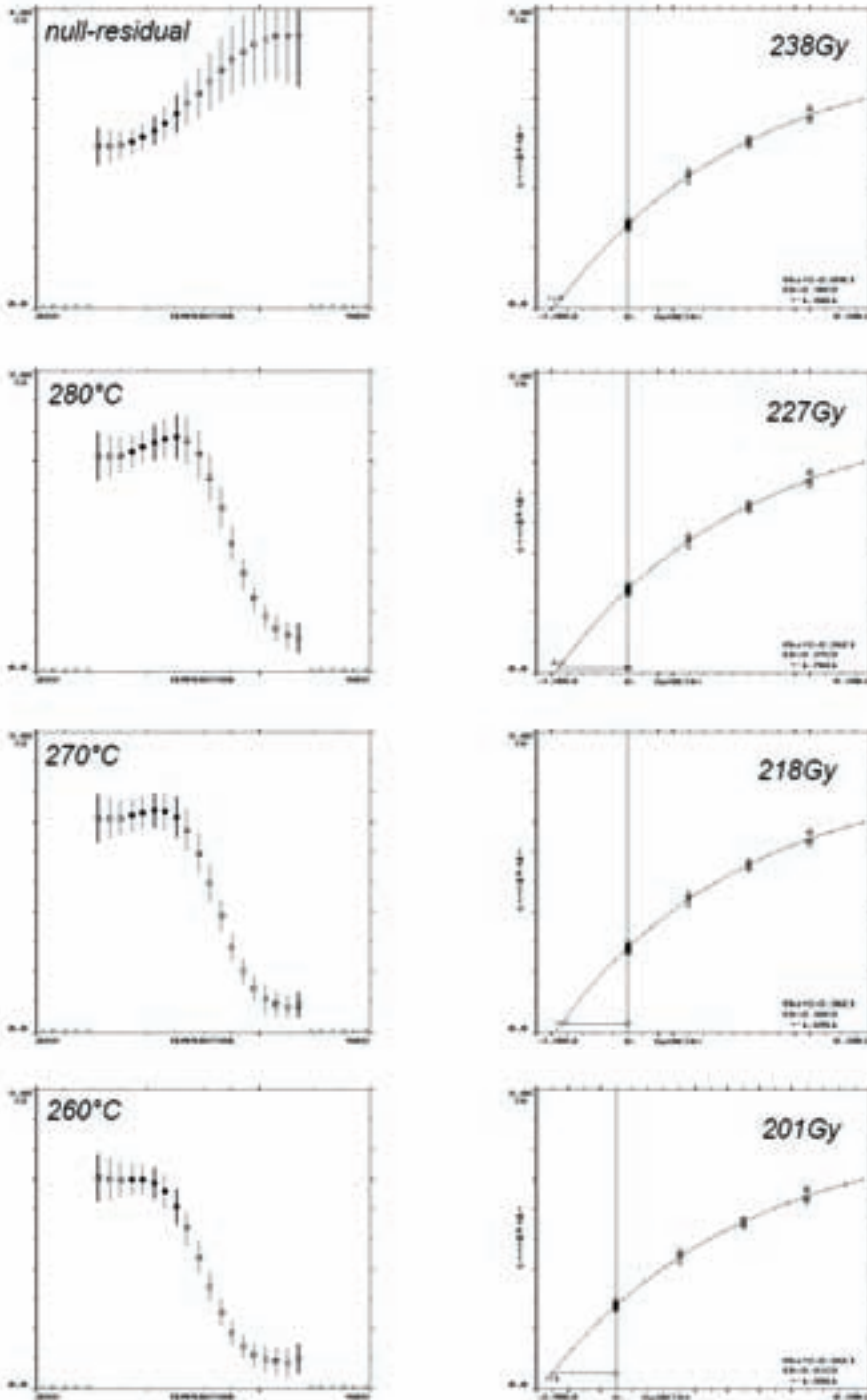


Fig. 5: Results of the PHLP technique for maar tephra (sample 4) from the Rodderberg volcanic complex, apparently ca. 300 ka old. The plots demonstrate that PHLP works even for high accumulated doses of ca. 1,000 Gy (corrected) or more.

Abb. 5: Ergebnisse der PHLP-Technik für die Maartephra (Probe 4) des Rodderberg Vulkan-Komplexes mit einem offensichtlichen Alter von ca. 300 ka. Die Diagramme zeigen, dass PHLP selbst für hohe akkumulierte Dosen von ca. 1 000 Gy (korrigiert) oder darüber funktioniert.

Figure 6 a



b

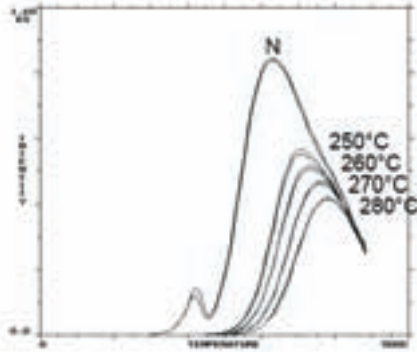


Fig. 6a: Additive dose response curve, residual TL value for 270°C equivalent temperature and  $D_E$  (longest plateau) from sample 10b (peperite xenolith from Meerfelder Maar tephra).

b: NTL glow curves and TL residuals after incremental preheat from sample 10b (peperite xenolith from Meerfelder Maar tephra).

Abb. 6a: Additive Dosis-Wirkungs-Kurve, residueller TL-Wert für eine Äquivalenttemperatur von 270°C und  $D_E$  (längstes Plateau) für Probe 10b (Peperit-Xenolith aus der Meerfelder Maar-Tephra).

b: NTL-Leuchtkurven und residuale TL nach stufenweisem Vorheizen der Probe 10b (Peperit-Xenolith aus der Meerfelder Maar-Tephra).

dence for age overestimation which would be expected in the case of incomplete TL-zeroing during eruption. The apparent TL age of Pulvermaar ( $21.0 \pm 2.95$  ka, corrected for short term fading of ca. 9%) lies within the LGM as expected (LORENZ & ZIMANOWSKI 2000).

From Pulvermaar we also used the TL of the 125-250  $\mu\text{m}$  fractions to check for possible grain size-dependant zeroing. This fraction consists of platy slate clasts. As shown in Fig. 4 (note that data were not corrected for fading) a  $D_E$ -plateau is not obtained using extrapolation of the dose response curve to zero. This suggests that TL-zeroing of this fraction during fragmentation in the explosion chamber of the maar was not as complete as for the fine silt fraction.

From the Rodderberg volcanic complex (RVC) south of the city of Bonn, in a previous study

one of us (BLANCHARD 2002) dated a well-heated brick-red xenolith (Lower Devonian siltstone) extracted from scoria and obtained an apparent TL-age of  $321 \pm 37$  ka (sample R4-D-1, data in PAULICK et al. 2008, Table 2). Tephra from the RVC was found in a nearby loess-paleosol section in loess attributed to the third last glaciation (BARTELS & HARD 1973, 1974), and the superposition of lapilli tephra and scoria on glacial loess argues for an eruption of the Strombolian series within a cold stage (probably MIS 8, ca. 250 to 310 ka). PAULICK et al. (2008) support an eruption age of the RVC of ca. 300 ka by geochemical and mineralogical methods and comparison with data given in BOGAARD & SCHMINCKE (1990). For the present study, we sampled two maar tephra beds (sample 3 from the initial maar tephra and sample 4 from the final maar tephra) from the RVC. Both tephra beds are much sandier than the maar tephra beds of samples 1 and 2.

The Rodderberg maar tephra layers both exhibit evidence of incomplete resetting by partial heating. Equivalent temperatures were found at 270°C (sample 3) and 260°C (sample 4a), respectively. Fig. 5 demonstrates that even in the high dose range PHLP yields satisfying results. Short-term anomalous fading was ca. 7% for sample 3 and ca. 25% for sample 4. Effective dose-rates differ significantly (3.70 and 2.87 Gy/ka, respectively), but apparent TL ages are consistent within error bars (Table 2). However, the apparent age of sample 4 in particular must be regarded with caution as the correction for short term anomalous fading may not account for the total amount of fading and related age underestimation. Our results so far argue for a volcanic activity phase of the Rodderberg complex ca. 300 ka ago, but a higher age (e.g., MIS 10) cannot be precluded.

At the base of the rampart of the Meerfelder Maar (West Eifel Volcanic Field) we sampled the initial fine grained maar tephra in the quarry near the village Deudesfeld (sample 10a).

Based on non-consistent radiocarbon ages from organic material, BÜCHEL & LORENZ (1984) interpreted the highest age ( $28750 \pm 2270 - 1760$  a BP) as a minimum age of the maar eruption.

The relatively small amount of preserved maar tephra with respect to the large crater (1700 m diameter), and the advanced aggradations of the maar lake argue for a significantly higher age (Negendank, pers. comm., 2006, see also LORENZ & ZIMANOWSKI 2000). Our PHLP TL-results yielded an equivalent temperature of 270°C. Short-term anomalous fading was ca. 10%. The apparent TL (minimum) age is  $69.2 \pm 14.5$  ka.

Our further studies concerned *crustal xenoliths* to verify the PHLP technique for material which was exposed to partial or even total zeroing of its parent TL. In the tephra originating from *Meerfelder Maar* some basaltic bombs containing peridotite (up to 20 cm diameter), pyroxene xenocrysts and light greyish “peperite” (LORENZ & ZIMANOWSKI 2000) with fragments of hydroclastic country rock occur. The latter bombs are also known as “cauliflower bombs”. A peperite extracted from a cauliflower bomb was dated (sample 10b) using the PHLP technique. The equivalent temperature was found at 270°C (Fig. 6 a). Natural TL glow curves and residuals after incremental preheat are shown in Fig. 6 b. Short term anomalous fading was ca. 5%. The apparent TL age of  $80.4 \pm 8.34$  ka is consistent with the apparent age of sample 10a within error bars despite significantly different effective dose-rates (2.85 Gy/ka and 4.54 Gy/ka, respectively). Our results thus confirm the much higher age of the maar eruption than previously thought: the eruption and, thus, the preserved paleovegetation remnants correlate to the Odderade interstadial (or an even older warm phase) rather than the Denekamp interstadial (BÜCHEL & LORENZ 1984).

Another crustal xenolith (Lower Devonian brick-red siltstone, sample 13) was extracted from the *Mosenberg* scoria cone from an exposure at the southwest foot of the volcanic hill. The *Mosenberg* volcanic complex (MVC) consists of several scoria cones and maars lying on a SE-NW oriented fault line. The nearby *Meerfelder Maar* is also believed to belong to this 3 km long volcanic complex with altogether seven eruptive centres (5 scoria cones and 2 maars) and 4 lava flows. It is believed so far

that volcanic activity shifted from SE to NW (LORENZ & ZIMANOWSKI 2000). Based on radiocarbon dating results from *Meerfelder Maar* and another radiocarbon age from *Hinkelsmaar* at the north-western end of the MVC (JUVIGNÉ et al. 1988), the minimum age of the complex is ca. 29 ka BP, but LORENZ & ZIMANOWSKI (2000) estimate it to be considerably older. ZÖLLER (1989) obtained a TL age of  $42 \pm 3$  ka from a brick-red slate xenolith found in the exposure mentioned above, but WODA (2000) dated  $87 \pm 8$  ka using ESR of a heated quartz pebble, and from volcanic glass LEYK & LIPPOLDT (1997) report a  $^{40}\text{Ar}/^{39}\text{Ar}$  age of  $81 \pm 23$  ka, which is not documented in detail, however. It is possible that the TL age by ZÖLLER (1989) dates a younger eruption, but undetected long-term

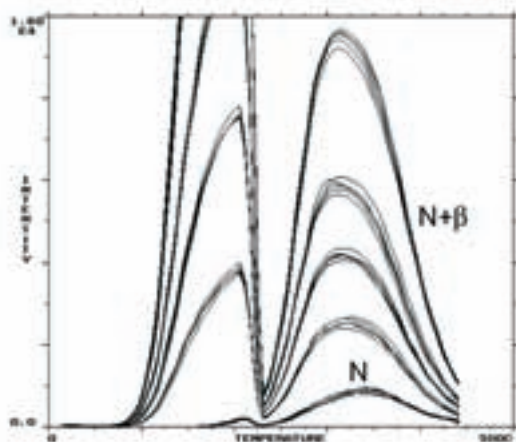


Fig. 7: TL glow-curves from natural and additively dosed aliquots of sample 13 (brick-red slate Xenolith from the *Mosenberg* scoria cone) after preheating at 220°C for 2 min. The peak-shift towards lower glow temperatures of remaining glow peaks (>220°C), referred to the NTL peak, indicates metastable TL of additively dosed aliquots still present after preheat.

Abb. 7: TL-Leuchtkurven natürlicher und additiv bestrahlter Teilproben der Probe 13 (ziegelroter Schiefer-Xenolith aus dem *Mosenberg*-Schlackenkegel) nach 2-minütigem Vorheizen bei 220°C. Die im Vergleich zur NTL sichtbare Verschiebung des Maximums (>220°C) additiv bestrahlter Teilproben nach Vorheizen zu niedrigeren Leuchtkurven-Temperaturen spricht für metastabile TL dieser Teilproben, die auch nach dem Vorheizen noch präsent ist.

anomalous fading may also account for the younger TL age.

The polymineral fine grain fraction of sample 13 was first measured using blue TL emissions. A long  $D_E$  plateau indicates that the xenolith was well heated. As was also found by ZÖLLER (1989), the dose response shows a linear growth up to high additive doses (1,000 Gy) and no detectable short-term anomalous fading. The apparent TL age is, however, only  $27.0 \pm 2.66$  ka, which is supposed to be due to metastable components of the laboratory induced TL as indicated by a peak shift towards lower glow temperatures after a preheat of  $220^\circ\text{C}$  for 2 min (Fig. 7), whereas ZÖLLER (1989) preheated at  $270^\circ\text{C}$  for 1 min. Because of the metastable components the apparent TL-age of  $27.0 \pm 2.66$  ka is discarded.

We then extracted pure fine grain quartz separates by etching in  $\text{H}_2\text{SiF}_6$  and measured the orange-red TL (sample 13RTL, 600-660 nm) using a regeneration protocol. Aliquot to aliquot reproducibility is substantially better than for blue TL. Anomalous fading has so far not been reported for the red TL (RTL) of quartz. RTL glow curves correspond to the shape described, e.g., by FATAHI & STOKES (2003) peaking at  $330^\circ\text{C}$ , and the  $D_E$ -plateau ranges from  $300$  to  $360^\circ\text{C}$ . The apparent age rises to  $53.1 \pm 1.83$  ka. The pronounced supra-linearity of the regenerated dose response-curve (Fig. 8) may, however, indicate TL sensitivity change due to heating which was not expected according to previous RTL dating studies from burnt flint (RICHTER & KRBETSCHKE 2006). We therefore regard this RTL age with caution until further studies can prove the reliability of the applied regeneration protocol. Nevertheless, the apparent RTL age supports the evidence for metastable blue TL components from polymineral fine grains of the sample.

From the well-dated (12.9 ka) trachytic *Lake Maria Laach* tephra a whitish crustal xenolith (quartzite?) was sampled at the famous “Wingertsberg” site (SCHMINCKE 2000). The polymineral fine grain (sample 14a) and the quartz fine grain fraction (14RTL, regeneration) were prepared, whereas a quartz coarse grains dat-

ing failed because of too small sample amount. The blue TL of sample 14a showed a long  $D_E$ -plateau from  $310$  to  $380^\circ\text{C}$ , but the apparent TL age ( $7.09 \pm 1.34$  ka) significantly underestimates the known age. Short-term anomalous fading could not be reliably determined due to very low signal to noise ratio, but anomalous fading is assumed to account for the age underestimation. An inhomogeneous natural radiation field of the xenolith and the surrounding trachytic pumice may also account for overestimation of the effective dose-rate. If anomalous fading can be corrected for or avoided by using RTL, equivalent xenolith samples should be datable by TL as heating during eruption appeared sufficient. RTL intensities of 14RTL were, unfortunately, very low, but the RTL ( $330$ - $360^\circ\text{C}$ ) around the peak temperature suggests an apparent age of  $10.8 \pm 0.63$  ka (linear fit) and  $10.4 \pm 0.72$  ka (exponential fit), respectively. The RTL regenerated after TL readout of the natural N-RTL does not start, however, at

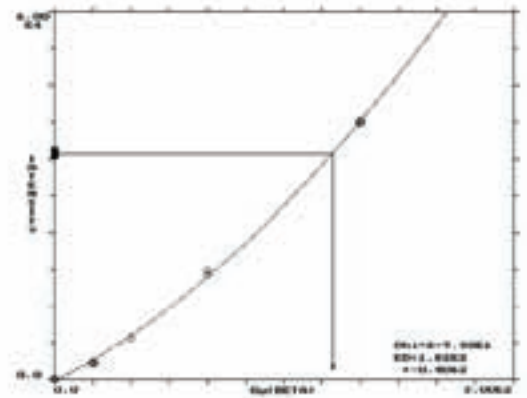


Fig. 8: Natural TL (600-660 nm emissions) of fine-grained quartz from sample 13RTL (brick-red slate Xenolith from the Mosenberg scoria cone) and regenerated dose response curve (quadratic curve fitting). Note the pronounced supra-linearity of the regenerated RTL.

Abb. 8: Natürliche TL (600-660 nm-Emission) von feinkörnigem Quarz der Probe 13RTL (ziegelroter Schiefer-Xenolith aus dem Mosenberg-Schlackenkegel) und regenerierte Dosis-Wirkungs-Kurve (quadratische Kurvenanpassung). Man beachte die ausgeprägte Supralinearität der regenerierten RTL.



Photo 1: Dark fine-grained maar tephra bed (hammer, sample 1) in the basal part of the rampart of the Ulmener Maar, topped by a diastrophic base-surge layer.

Foto 1: Dunkle feinkörnige Maartephra (Hammer, Probe 1) im basalen Teil des Tuffrings des Ulmener Maars, überlagert von diaklastischen Ablagerungen eines pyroklastischen Stromes („Base surge“).

zero for zero dose. It appears that some kind of recuperation has occurred after the first N-RTL readout which may also lead to an underestimate of  $D_E$ . Furthermore, the “through form” of the  $D_E$ -plateau may argue for TL sensitivity change after the first glow as the regenerated RTL peak exhibits a smaller half-width than the natural glow peak.

## 6 Discussion

The observed total or partial resetting of TL glow peaks in maar tephra and xenoliths derived from non-volcanic country rock may have several reasons:

a) merely thermal bleaching (questionable but not to be excluded for hydroclastically fragmented rocks),

b) thermodynamic fragmentation by dynamic pressure (frictional heating), and

c) hydrostatic high pressure at elevated temperatures (ZÖLLER et al. 2009).

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, found evidence for grain size-dependent resetting in maar tephra, arguing for a resetting by frictional heating or thermally assisted hydrostatic pressure, or both, rather than merely thermal resetting by heat transfer.

For crustal xenoliths, these two resetting mechanisms are unlikely and thermal resetting (heat transfer) is probably the most effective resetting mechanism. As rocks – in particular dry rocks – are poor heat-conductors, long-lasting



Photo 2: Exposure in the rampart of the Pulvermaar. The light fine-grained bed (sample 2) is visible in the upper third of the exposure (A). In the lower part antidunes and “swimming blocks” witness base surge deposition (B). In former years an ice-wedge cast was exposed in the uppermost part of the tephra, indicating harsh permafrost climate after the deposition of the rampart. The height of the exposure is 10 to 11 m.

Foto 2: Aufschluss im Tuffring des Pulvermaares. Die helle feinkörnige Lage (Probe 2) ist im oberen Drittel des Aufschlusses sichtbar (A). Im unteren Teil belegen Antidünen und „schwimmende Blöcke“ einen pyroklastischen Strom („Base surge“-Ablagerung) (B). In früheren Jahren war eine Eiskeil-Pseudomorphose im obersten Teil der Tephra aufgeschlossen, die raues Dauerfrostboden-Klima nach der Ablagerung des Tuffrings belegt. Die Höhe des Aufschlusses beträgt 10-11 m.

heating of the xenolith can be expected to be a prerequisite for total TL-resetting within the xenolith. Brick-red slate and siltstone xenoliths as well as light greyish to whitish altered xenoliths which experienced even higher temperatures appear to be suited for TL dating of their eruption. However, in the case of fast cooling of volcanic products as may be expected for volcanic bombs and for scoria falls, the heating time may not have been sufficient to generate a homogenous heat profile within a xenolith originating from a shallow depth (<1-3 km). For microdosimetry reasons (ranges and geo-

metry of  $\alpha$ -,  $\beta$ -, and  $\gamma$  radiation), probing the inner parts of a xenolith is desirable. The new PHLP technique proves its ability to overcome problems related to incomplete heating and related residual TL at the eruption. Our dating attempts using the PHLP technique so far demonstrate that its capability to date volcanic events is independent of the resetting mechanism.

Although one of the motivations to use crustal xenoliths for dating volcanic events was to circumvent the strong anomalous fading known from volcanic feldspars and other volcanic



minerals, anomalous fading still raises a major problem. Some samples not exhibiting short-term anomalous fading clearly underestimate the known age; whereas others with even strong (up to 25%) short term anomalous fading, after 7 days storage at 70°C allowing for settling of the fast component of the fading process, yield apparent TL ages in agreement with independent age estimates. This could be demonstrated, e.g., for our oldest dated samples from the Rodderberg volcanic complex, apparently ca. 300 ka old. The Rodderberg samples furthermore prove the applicability of the PHLP technique to Middle Pleistocene eruptions at least up to this age.

Our dating results from Rodderberg and from Meerfelder Maar – apart from the pending fading problem - support the overall validity of our TL dating protocols as different materials (maar tephra, crustal xenoliths) yield identical apparent TL ages within error bars. From volcanic events with well-based known age no significant age overestimates are obtained: the Rodderberg volcanic complex must be younger than ca. 500 ka as the complex rests on a River Rhine terrace ( $t_{R6}$ ) supposed to be ca. 500 ka old (6<sup>th</sup>-to-the-last glacial, see BIBUS 1980). Apparent TL ages from Lake Maria Laach tephra and Ulmener Maar tephra underestimate the known ages, most probably due to long-term anomalous fading (see above). Our apparent age from the Pulvermaar supports the geomorphologically based age estimate (LGM), but other sound age information is not available so far. Our age estimates from the Meerfelder Maar corroborate the geomorphologically much higher age estimate than suggested from radiocarbon minimum ages. The age obtained from the slate xenolith extracted from the Mosenberg scoria cone using blue TL is rejected due to metastable TL signals (see above), whereas the apparent RTL age (quartz fine grain fraction) from the same xenolith is closer to the ESR and  $^{40}\text{Ar}/^{39}\text{Ar}$  age estimates of the underlying lava flow. If it holds true that the MVC including Meerfelder Maar erupted within a short time period as suggested by previous authors (see review by LORENZ & ZI-

MANOWSKI 2000) the entire complex could have been formed some 80 ka ago, but rejuvenated activity of the Mosenberg volcano cannot be precluded at present.

As maar lakes are most valuable geoarchives often containing long records of annually varved sediments (ZOLITSCHKA et al. 2000, NEGENDANK & ZOLITSCHKA 1993), reliable dating of maar eruptions serves as an essential tool to envisage the maximum time span covered by those high resolution geoarchives (see also SIROCKO et al. 2005). Reliable dating of alternating volcanic activity phases in the two spatially distinct Eifel Volcanic Fields (and other Central European areas with Quaternary volcanic activities) will also be crucial to validate the model of pulsed fingers of the “Eifel Plume” as presented by RITTER et al. (2001) and to further investigate its dynamics during the Middle and Upper Pleistocene. For the past 200 to 300 ka, in particular, TL dating using the PHLP technique can prove very valuable and complementary to fill dating gaps, as K/Ar or  $^{40}\text{Ar}/^{39}\text{Ar}$  dating is difficult if sanidines are lacking and radiocarbon dating is hampered by the lack of suitable datable material or its limited time range.

## 7 Conclusions and Outlook

High hydrostatic pressure and elevated temperatures (but too low for total annealing of the geological TL) may occur at shallow 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. This may cause partial resetting of the latent TL (and IRSL) in addition to heat transfer from the rising magma to neighbouring country rock and to frictional heating (TAKEUCHI et al. 2006). Recognizing and estimating the amount of partial resetting is essential to correctly date volcanic eruptions by luminescence and to circumvent age overestimates.

We demonstrated that the PHLP technique of TL dating applied to maar tephra or crustal xenoliths is suited to obtain meaningful eruption ages back to a minimum of 300 ka. The PLHP technique involves an intrinsic check

for incomplete resetting of the latent TL signal at eruption and determination of the inherited natural TL intensity. Thus, overestimates of TL eruption ages are avoided. Age underestimates attributed to long-term anomalous fading were rather observed from some samples of known-age eruptions. For this reason apparent TL ages presented in this study have to be regarded as minimum ages. Circumventing or correcting for anomalous fading as a limiting factor for reliable TL dating remains a challenge, even if volcanic feldspars (and other volcanic minerals) may be more prone to anomalous fading than mainly non-volcanic minerals or mineral assemblages from maar tephra and crustal xenoliths used in our experiments. As this pilot study was designed to develop and test the PHLP technique rather than to produce definite ages and as laboratory time was limited, long-term fading tests following AUCLAIR et al. (2003) have not been executed so far, but will be a major aim of further studies. Instead, an accelerated test for short-term anomalous fading after BERGER (1987) was applied for correction of equivalent doses, but correction factors and, thus, correct equivalent doses may still be underestimated. Our data give some evidence, however, that short-term anomalous fading observable in the laboratory and mid- or long-term anomalous fading *sensu* XIE & AITKEN (1991) not directly observable may be different and non-proportional processes (see also ZÖLLER 1995, for TL ages from loess). In this case, even longer observation of anomalous fading in the laboratory may not necessarily result in correct ages.

Therefore, other strategies to circumvent anomalous fading should be designed. One approach which has already been tested successfully (see review by FATAHI & STOKES 2003) is the use of orange-red TL (RTL) emissions from quartz. It remains to be investigated, however, if the PHLP technique is applicable to the RTL from quartz. This may be even doubted as the RTL glow peak used for dating normally is symmetric and has a much smaller half-width than the blue TL (BTL) from quartz and feldspars in the relevant glow temperature range.

Nevertheless, RTL-PHLP measurements on samples for which partial resetting were detected by BTL-PHLP is required in future studies. Another problem with RTL from quartz, the pronounced supralinerarity of the (regenerated) dose-response curve observed from our sample 13RTL, needs further systematic investigations. In general, further investigations into the reliability of the regeneration method for RTL appear inevitable as TL sensitivity change after the first TL-readout may turn out to be a more significant problem than for the RTL of burnt flint (RICHTER & KRBETSCHKE 2006). The still significant RTL age-underestimate of our sample 14RTL may underline this problem.

Isothermal decay of TL (JAIN et al. 2005) at temperatures below or equal to the temperature experienced by the sample during eruption may be considered as an alternative as well. One problem with application to partially heated rocks will be to anneal thermally unstable laboratory-induced TL (dose-rate of laboratory irradiation is up to ca.  $10^9$ -fold of the natural dose-rate) in a way equivalent to the annealing of naturally induced TL at ambient temperatures. ZÖLLER & WAGNER (1989) report experiments using the “partial thermal washing technique” (preheat only applied to laboratory dosed subsamples) for TL-dating of loess which demonstrate that exact matching of the glow peaks is rarely achieved. Alternatively, “plateau heating” as established for fission track dating to isolate thermally stable track lengths from both natural and induced fission tracks (WESTGATE 1988, WAGNER 1998) could be applied prior to isothermal TL, but the necessary annealing temperatures may be as high as or even higher than equivalent temperatures during eruption and erase most or all of the TL signal relevant for dating. The dating approach using isothermal TL of partially heated materials may therefore not look very promising; nevertheless it deserves experimental testing. Anomalous fading of polymineral samples is expected to raise similar problems as with the PHLP technique. Another “partial bleach”-technique originally developed for TL dating of sediments (WINTLE & HUNTLEY 1980) called R- $\beta$  or R- $\gamma$  technique

(see also AITKEN 1985, BERGER 1988, ZÖLLER 1995), may be adopted and tested for TL dating of partially heated rocks as well. Similar as for partial optical bleaching of the TL of sediments, the ratio of resetting during partial heating referred to the natural TL is glow temperature-dependent. By replacing laboratory optical bleaching using incremental bleach times with laboratory partial heating using incremental temperatures as shown for the PHLP technique, the correct  $D_E$  accumulated since the eruption may be expected. This technique anticipated here may be preliminarily called "PHR- $\beta$ " technique. The validity of this approach has, however, to be tested in laboratory experiments which, to our knowledge, have not been tried so far.

The present study may open new perspectives for TL dating of Middle and Upper Quaternary volcanic events by the evolution of a complementary physics-based dating method applicable to a wide range of materials and eruption types. Although TL dating of heated materials has by now been employed since almost 50 years (see GRÖGLER et al. 1960) we stand at the beginning of dating partially heated volcanogenic eruptives, and more fundamental research may be stimulated by this contribution.

### Acknowledgements

The present pilot study was sponsored by the Deutsche Forschungsgemeinschaft (German Research Foundation) DFG, project number Zo 51/28-1. We thank Dipl.-Inf. (FH) Manfred Fischer for support during laboratory work in the luminescence dating laboratory at the University of Bayreuth, Dr. Catherine McCammon (BGI, Bayreuth) for improving the English of our manuscript, and PD Dr. Daniel Richter (MPI for Evolutionary Anthropology Leipzig) for critical and helpful reviewing.

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