

# Ancient TL

[www.ancienttl.org](http://www.ancienttl.org) · ISSN: 2693-0935

Issue 38(2) - December 2020  
<https://doi.org/10.26034/la.atl.v38.i2>

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# Ancient TL

A periodical devoted to Luminescence and ESR dating

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<http://ancienttl.org>

**December 2020, Volume 38 No.2**

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# Ancient TL

Started by the late David Zimmerman in 1977

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## Ancient TL

# ‘gamma’: An R Package for Dose Rate Estimation from In-Situ Gamma-Ray Spectrometry Measurements

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Received: September 25, 2020; in final form: October 28, 2020

### Abstract

**In situ gamma spectrometry is a useful technique used by the Luminescence and ESR dating community to improve the representativity of dose rate measurements in the context of gamma dose rate heterogeneities around dated material. This paper presents ‘gamma’, a new R package and its graphical user interface ‘gammaShiny’, allowing a reliable and reproducible workflow for *in situ* gamma spectrometry data analysis in the context of luminescence and ESR dating.**

**Keywords:** Archaeology, Geochronology, Luminescence dating, Gamma spectrometry

### 1. Introduction

*In situ* gamma spectrometry is a powerful tool used in luminescence dating to tackle gamma dose rate heterogeneity (Aitken, 1998; Klusoň, 2010). A gamma spectrometer probe records gamma rays emitted within a sphere of radius equal to 30–50 cm (Figure 1 in Guérin & Mercier, 2011) whereas a classic BeGe laboratory gamma spectrometer with planar or coaxial geometry only characterizes samples of a few dozen cubic centimeters, thus greatly improving the representativity of the measurement (Gilmore, 2008).

Different techniques are currently used in luminescence and ESR dating field to determine the gamma dose rate from *in situ* gamma spectrometry measurements:

- The ‘window technique’, that consists in selecting different parts of the spectra to quantify the U and Th-series and K radioelements contents in the sediment, around the probe.
- The ‘threshold techniques’, that allow a direct determination of the gamma dose rate without individual determination of K, U and Th series radioelements. Murray et al. (1978) have shown that the gamma dose rate can be estimated using the proportionality existing between the number of counts measured above a certain energy and the gamma dose rate. Mercier & Falguères (2007) developed this approach for a NaI(Tl) probe. They conclude that the measurement time (and consequently the risk of changing temperature during the measurement) is significantly reduced in comparison with the ‘window technique’.

There are two methods based on the ‘threshold technique’: in counts and in energy. In both cases, Guérin & Mercier (2011) have shown that the environmental gamma dose rate determined in this way does not depend on water presence, nor on U-series disequilibrium. They have also shown few dependence on the gamma dose rate with the nature of the sediment.

The majority of *in situ* gamma-ray spectrometers produce energy shifted and spectrally distorted spectra. This could be due to the fact that detector crystals (usually LaBr or NaI) are often used in unstable temperature conditions (Casanovas et al., 2012). As a result, spectrum processing is required before conversion from a spectrum to gamma dose rate.

A typical workflow for energy shifting correction requires manual identification of reference peaks. This is usually

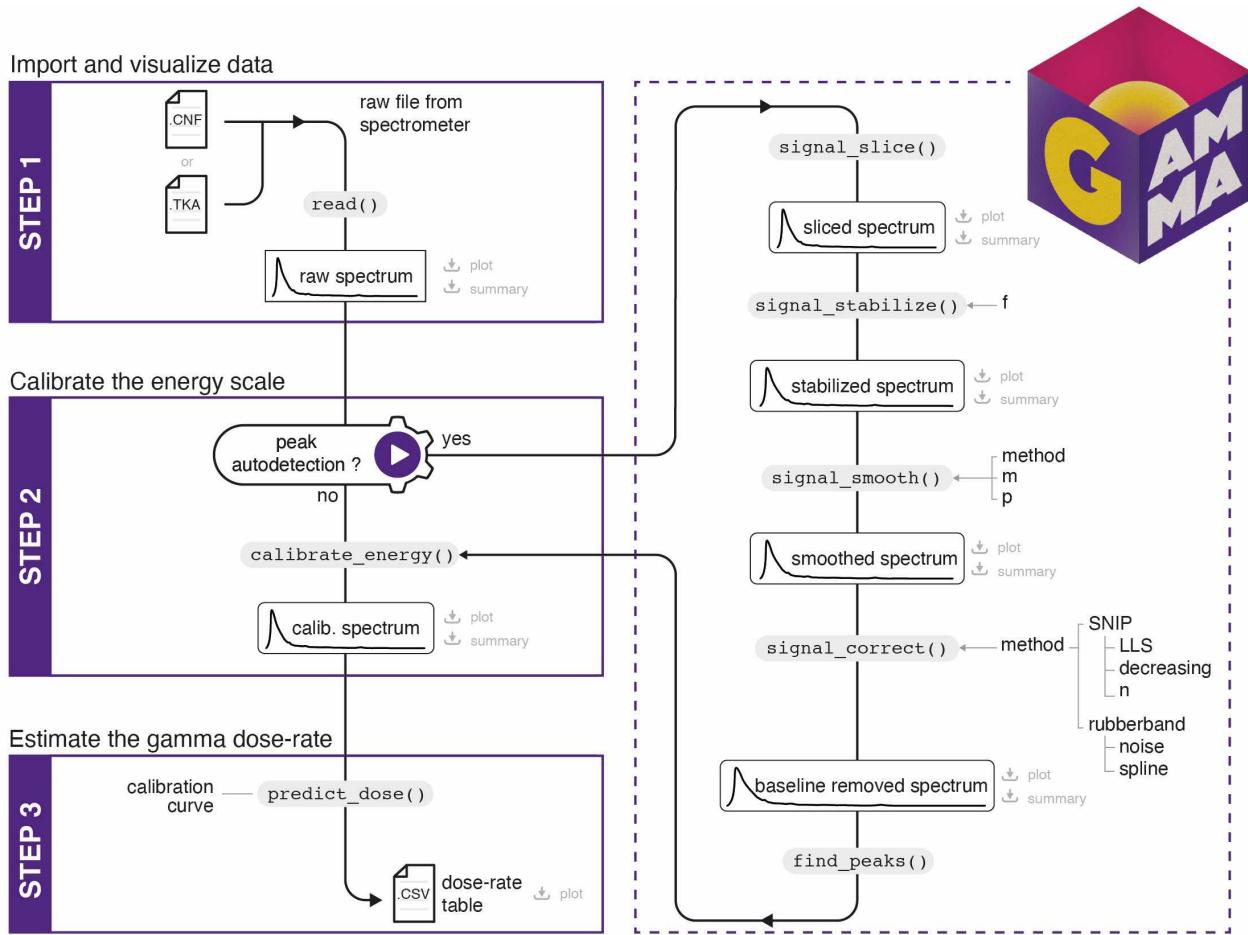


Figure 1. A typical workflow within the 'gamma' package.

performed using proprietary software such as Genie2000® (Canberra division of Mirion technologies, Windows OS only). Once reference peaks are identified, a new channel-energy curve is generated, taking into account the apparent shift between the observed and theoretical energies of these peaks. To this day, there is no turnkey solution for these steps and personal or laboratory-based solutions are implemented, e.g. Visual Basic Advance® macros within Microsoft Excel®.

There are two challenging issues to these existing approaches. The first one is the lack of transparency: details of spectrum processing are not presented in the publications and researcher's internally developed solutions are not made available to the community, greatly limiting reproducibility and reliability of the measurements (see Kreutzer et al., 2017 for a broader discussion in the field of luminescence dating). Secondly, the use of proprietary software limits their access by the community, hindering their maintenance and evolution.

To address these different issues, we present an integrated solution to analyse *in situ* gamma spectrometry data, from raw spectrum processing to gamma dose-rate estimation: the R package 'gamma'. The R programming language (R Core Team, 2020) has many advantages, starting

with its ease of learning and use. R is available on all computer platforms and its community offers efficient support. R is distributed under the GNU General Public License (therefore free of charge), ensuring transparency and modularity. Finally, several R packages dedicated to geochronology already exist (such as the 'Luminescence' package; Kreutzer et al., 2012).

## 2. The R package 'gamma'

The 'gamma' package allows the user to import, inspect and correct the energy scale of one or multiple spectra. It provides methods for estimating the gamma dose rate by the use of built-in or custom calibration curves. A graphical user interface (GUI) is also provided through the dedicated R package 'gammaShiny', using the 'shiny' package (Chang et al., 2020).

### 2.1. Workflow overview

This section presents a typical workflow within the 'gamma' package (Figure 1). A walk-through guide is also provided in the package manual. The following examples can be reproduced in any integrated development environ-

ment (like RStudio<sup>TM</sup> Desktop; <http://rstudio.org>) or in the **R** terminal.

Once the ‘gamma’ package is loaded with `library(gamma)`, processing a spectrum is achieved in three steps: import, energy calibration and dose-rate estimation which are detailed hereafter.

All of these steps can be performed through the GUI, which can be launched with `launch_app()` after loading the package with `library(gammaShiny)`. Note that we chose to develop a GUI to allow the use of ‘gamma’ for users who might not feel comfortable with the use of the command line. However, only the full publication of scripts along with the results guarantees the reproducibility of published studies.

### 2.1.1 Step 1: import and inspect a gamma spectrum

The ‘gamma’ package supports the most common type of spectrum files: Canberra CAM (.CNF) and Programmers Toolkit files (.TKA). This prevents spending time on data transformation and preserves file metadata. The `read()` function allows to import a CNF-file with the path of the file as a single argument. When the path leads to a directory instead of a single file, all spectra included in the directory are imported.

```

1 ## Get an example file
2 cnf <- system.file(
3   "extdata/LaBr.CNF",
4   package = "gamma"
5 )
6 ## Import data
7 spc <- read(cnf)
8 spc
```

Spectra are stored as instances of an S4 class. Many mutator methods and group generic functions are available for basic operations and data wrangling. The `plot(spc)` method allows to quickly graphically inspect the data.

### 2.1.2 Step 2: adjust the energy scale

The energy calibration of a spectrum is a challenging part and must be performed before dose rate estimation. The solution presented here requires the user to specify the position (channel) of at least three observed peaks and their corresponding theoretical energies (in keV). A second order polynomial model is fitted on these energy *versus* channel values, then used to predict the new energy scale of the spectrum.

The package allows the user to provide the channel-energy pairs to be used. However, the spectrum can be noisy so it is difficult to properly identify the peak channel. In this case, a better approach may be to pre-process the spectrum (variance-stabilization, smoothing and baseline correction) and perform a peak detection (Figure 2). Once the peak detection is satisfactory, the user can set the corresponding energy values (in keV) and use these lines to calibrate the energy scale of the spectrum. All signal processing methods are prefixed with `signal_`.

**Cleaning** Several channels can be dropped to retain only part of the spectrum with `signal_slice()`. If no specific value is provided, an attempt is made to define the number of channels to skip at the beginning of the spectrum. This drops all channels before the highest count maximum. This is intended to deal with the artefact produced by the rapid growth of random background noise towards low energies.

**Stabilization** The stabilization step aims at improving the identification of peaks with a low signal-to-noise ratio. This particularly targets higher energy peaks. To perform this step, a function is applied to all the intensity values of the spectrum with `signal_stabilize()`. The example below uses a square root transformation but any user defined function can be used.

**Smoothing** Counting artefacts can be removed by smoothing intensities with `signal_smooth()`. Several methods are implemented here such as (weighted) sliding-average and Savitzky-Golay filter (Savitzky & Golay, 1964; Gorry, 1990). The example below uses the Savitzky-Golay filter as this method usually provides better results, at the expense of a longer computing time.

**Baseline correction** Spectrum baseline can be estimated and removed with `signal_correct()` using specialized SNIP algorithm (Ryan et al., 1988; Morháč et al., 1997; Morháč & Matoušek, 2008).

**Peak detection** `find_peaks()` allows automatic peak position detection on a baseline-corrected spectrum. For a successful detection, a local maximum has to be the highest one in a given window and has to be higher than  $k$  times the noise (estimated as the Median Absolute Deviation) to be recognized as a peak.

**Energy calibration** The energy scale calibration needs the energy values of detected peaks to be set. `calibrate_energy()` then fits a second order polynomial model to predict the new spectrum energy scale.

```

1 library(magrittr)
2 ## Signal processing
3 tmp <- spc %>%
4   signal_slice() %>%
5   signal_stabilize(f = sqrt) %>%
6   signal_smooth(method = "savitzky", m = 21)
7   ↪ %>%
8   signal_correct(method = "SNIP")
9 ## Peak detection
10 pks <- peaks_find(tmp)
11 set_energy(pks) <- c(238, NA, NA, NA,
12                      1461, NA, NA, 2615)
13 ## Energy scale calibration
14 spc2 <- energy_calibrate(spc, pks)
```

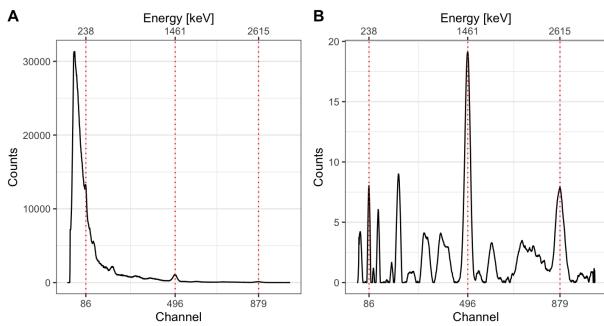


Figure 2. (A) Raw gamma ray spectrum from *in situ* measurement; (B) Processed spectrum used for peak finding along with the detected peaks.

### 2.1.3 Step 3: gamma dose-rate estimation

To estimate the gamma dose rate, one of the calibration curves distributed with this package can be used (Figure 3). These built-in curves are in use in several luminescence dating laboratories and can be used to replicate published results. As these curves are instrument specific, the user may have to build its own curve. The ‘gamma’ package provides a set of functions to build custom calibration curves.

```

1 ## Load the calibration curve
2 data("BDX_LaBr_1", package = "gamma")
3 ## Estimate the gamma dose rate
4 doses <- dose_predict(BDX_LaBr_1, spc2)
5 doses

```

The construction of a calibration curve with `dose_fit()` requires a set of reference spectra for which the gamma dose rate is known (see Miallier et al., 2009 for the reference values in use at the CRP2A laboratory) and a background noise measurement. First, each reference spectrum is integrated over a given interval, then normalized to active time and corrected for background noise. The dose rate is finally modelled by the integrated signal value used as a linear predictor (York et al., 2004). `dose_predict()` returns the predicted dose rate value with both the count and energy threshold approaches.

## 3. Discussion

The ‘gamma’ package provides a convenient and reproducible toolkit for *in situ* gamma spectrometry data analysis. Along with the dedicated ‘gammaShiny’ package (GUI), it offers a useful set of functions for spectra processing, calibration curve building and dose rate estimation.

The ‘gamma’ package allows batch processing of spectra. To ensure the reliability of the produced results, manual verification is strongly recommended since automatic detection may sometimes produce unreliable results, especially when the processed spectra display poor signal-to-noise ratios. In the future, improvement of the automatic peak detection process is to be expected (Guilhem Paradol, pers. comm.).

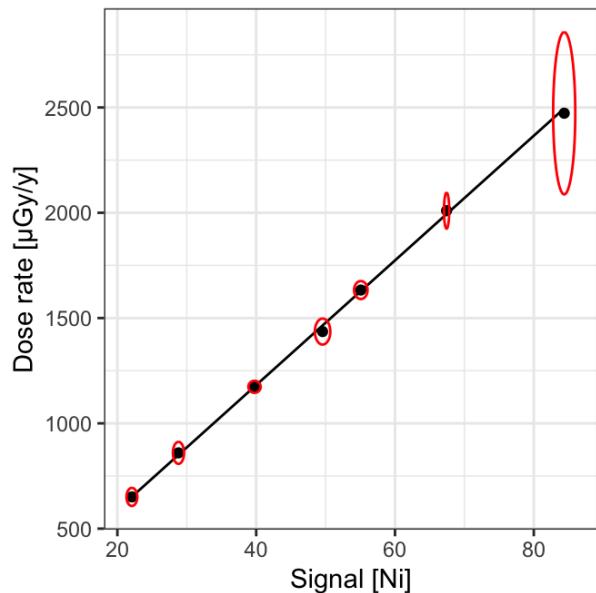


Figure 3. Built-in LaBr calibration curve in used at the CRP2A laboratory (Bordeaux).

## 4. Conclusions

The ‘gamma’ package is distributed over the Comprehensive R Archive Network (CRAN; <http://cran.r-project.org>). It is provided under the General Public Licence (GNU GPL3) conditions: the code is open and anyone can review it. Source code and installation instructions are available on GitHub at <https://github.com/crp2a/gamma> and <https://github.com/crp2a/gammaShiny>. Users are invited to contribute, share feedback, request new features or report bugs via the GitHub platform. As for the ‘Luminescence’ package (Kreutzer et al., 2012; Fuchs et al., 2015), future improvements to the ‘gamma’ package and associated GUI are intended to be community driven.

## 5. Acknowledgements

This work received a state financial support managed by the Agence Nationale de la Recherche (France) through the program *Investissements d’avenir* ref. 10-LABX-0052. The project leading to this publication has received funding from the Excellence Initiative of Aix-Marseille University - A\*MIDEX, a French ‘Investissements d’Avenir’ programme ref. 11-IDEX-0001. BL thanks Didier Miallier (Laboratoire de Physique de Clermont, UMR 6533) for his help during the calibration of the NaI probe.

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Martin Autzen

## **Thesis Abstracts**

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### ***Eslem Ben Arous***

#### **Chronology of human populations in North-western Africa during the Upper Pleistocene: chronological multi-methods approach (ESR/US, OSL and C-14) applied to Rabat-Témara sites**

*December 2019*

Museum national d'Histoire naturelle –  
UMR 7194 HNHP, Paris, France

*Degree: Ph.D.*

*Supervisors: Christophe Falguères and Roland Nespolouet*

During the last decades, research in Morocco has greatly highlighted the importance to consider North-western Africa in the study of the dispersal of early *Homo sapiens* within and out of Africa over the Upper Pleistocene. For these issues, the Rabat-Témara region is a rare example for having preserved from the last 120,000 years human stratified occupations in six caves. These human occupations are attributed to the Middle Stone Age (MSA) and the Later Stone Age (LSA).

However, the current chronological framework in Témara is subject to many questions, both geochronological/methodological and cultural. In order to solve them, a new geochronological campaign was undertaken at El Harhoura 2 and El Mnasra. These two caves were recognized as essential for two main issues: (1) the early *Homo sapiens* coastal occupations linking the earliest examples of cognitive complexity evidence and exploitation of the marine resources during the MSA and (2) the cultural and populations shift traduced by the transition from the MSA to the LSA.

These caves have been the subject of optically stimulated luminescence (OSL) dating, published in 2012 and, preliminary combined US-ESR dating published in 2012. In this thesis, we re-investigated their chronological framework, using a multi-dating chronological approach based on the inter-comparisons of various results: OSL on quartz grain samples, combined US-ESR on teeth enamel and radiocarbon on seed and charcoal from archaeological hearths.

The inter-comparisons of the new 39 dates obtained in this work with previous dating work has shown the close corre-

spondence between the radiocarbon and the US-ESR ages and has revealed: (1) a systematic discrepancy between all US-ESR ages and all OSL ages (OSL ages are systematically 25-50% older than US-ESR ages) and (2) discrepancy between OSL ages in different studies that can be as high as 40%. Reasons for these discrepancies are not yet fully understood and remain ambiguous because they can be explained with multiple causes (post-depositional mixing grains, the effect of variations in the beta dose rate to individual grains, incomplete bleaching,...).

At El Harhoura 2, these ages are enabled to date the end of the MSA around 40,000 years ago, either 15,000 years younger than previous dates. For the first time, we propose LSA presence from 12,000 years to 8,000 years, covering Marine Isotopic Stage (MIS) 1. New dates allow us to identify a chronological hiatus of about 30,000 years between the end of the MSA and the LSA (formerly 45,000 years before this work). This absence of ages extends from the end of MIS 3 to MIS 2. As a result of this work, we built the first chronological model of the Rabat-Témara region at the North African scale, showing that new dating results have clear and direct implications to interpret human settlement dynamics: the absence of human populations from the end of the MIS 3 until the MIS 1 in this region is associated to a major environmental and climatic change.

At El Mnasra cave, the most intensive MSA human occupations which yielded evidence of marine shells exploitation (food and ornaments purposes) have been dated between 65,000 and 100,000 years, about 10,000 to 15,000 years younger than the OSL ages published in 2012. While previous single-grain ages of these levels associated them with periods of high sea levels, the new results bring more nuance. Indeed, the large analytical errors associated with the ages (this study and previous studies) shows that the chronological resolution of these occupations is not precise enough to validate this model.

A PDF of this thesis can be requested from the author at [eslem.ben-arous@mnhn.fr](mailto:eslem.ben-arous@mnhn.fr) or at [ben-arous@shh.mpg.de](mailto:ben-arous@shh.mpg.de)

### ***Lucas Sátiro do Carmo***

#### **Nuclear Radiation Importance on Archeological and Geological Dating – Application To Sediments Dating From Cabo Frio**

*April 2020*

Institute for Nuclear and Energy Research , São Paulo, Brazil

*Degree: Ph.D.*

*Supervisor: Shigaeo Watanabe*

In this work, a dunefield known as Dama Branca

(Brazil) has been dated using the following techniques: Optically Stimulated Luminescence (OSL), Thermoluminescence (TL) and Electronic Spin Resonance (ESR). Sediments have been collected from several points to study age distribution throughout Dama Branca. These ages are related to events of sediment transportation and stabilization. For Dama Branca specifically, variables related to weather such as rainfall and wind power are suspected to be responsible for its formation. OSL results were obtained applying the SAR protocol. TL and ESR results were obtained using the Multiple Aliquot Additive Dose protocol (MAAD). With respect to ESR measurements, the Ti-Li center was chosen for dating since it can be completely bleached by sun light exposure, which makes it suitable for aeolian sediment dating. The Ti-Li center is strongly dependent upon preheat, its stability has been assessed and a preheat temperature of 180 °C was selected. OSL ages are within  $0.05 \times 10^3$  and  $2.05 \times 10^3$  years. TL ages agree with OSL ages for samples collected from the dune base, however there are discrepancies between OSL and TL ages for the DBM2BASE sample, suggesting that it underwent a quicker burial process. ESR results are satisfactory for two samples, 2DB10 and 2DB11 (they follow OSL and TL results). The ages were compared to a simplified morphological study. In recent works about the weather in the Cabo Frio region it has been seen that the studied areas were formed under influence of arid conditions and cold water, variables that control sediment transportation in the region.

A PDF of this thesis can be downloaded from:  
<https://doi.org/10.11606/T.85.2020.tde-17092020-152716>

**Ştefana-Mădălina Groza-Săcaciu**  
**Revisiting optically stimulated luminescence chronologies on loess-paleosol master sections from Europe and beyond**

*November 2020*

Faculty of Environmental Science and Engineering, Babeş-Bolyai University, Cluj-Napoca, Romania

*Degree: Ph.D.  
Supervisor: Alida Timar-Gabor*

Optically stimulated luminescence (OSL) is linked to Quaternary research, especially in what regards loess-paleosol sections of paleoenvironmental and archeological importance. At Mircea Vodă, an interlaboratory exercise on a new set of samples confirmed previous discrepancies between quartz fractions as well as the age underestimations for older samples. The Pleistocene/Holocene samples yielded an earlier age discrepancy ( $> \sim 20$  ka). Mircea Vodă was further investigated alongside Râmnicu Sărat for OSL and magnetic susceptibility analysis in order to assess the Pleistocene/Holocene transition. It was concluded that the time variation of the MS record across the transition is rather synchronous for these two sites and two other regional sites

(Roxolany-Ukraine and Mošorin – Serbia), with the onset of increase of the magnetic susceptibility signal dated through OSL starting closer to the 17-ka transition from the benthic  $\delta^{18}\text{O}$  record. High-resolution luminescence dating on the Pleistocene/Holocene transition was performed at Luochuan, China, to check for hiatuses due to erosional events. No hiatuses were observed but the OSL ages were highly variable and showed multiple inversions, most probably due to significant vertical mixing. At Krems-Wachtberg, a well-known archeological site from Austria, the average from CW-OSL and POSL ages for the horizons bracketing the archaeological layer is  $32.4 \pm 1.5$  ka, thus agreeing within error limits with the previous chronological results. Establishing a robust chronology at the LGM site Kammern-Grubgraben managed to clarify certain aspects regarding the complex stratigraphy and geochronology.

**Maryam Heydari**  
**Applying Bayesian models to improve luminescence-based chronologies of Middle to Upper Palaeolithic sites in Iran**

*August 2020*

Université Bordeaux Montaigne, Pessac, France

*Degree: Ph.D.  
Supervisors: Chantal Tribolo, Pierre Guibert, Guillaume Guérin*

Statistical data analysis is a fundamental aspect of luminescence dating. For decades, data processing predominantly employed the frequentist (classic) school of thinking. Only recently, the development of Bayesian modelling specifically for luminescence dating in R environment, has provided an alternative. This thesis aims firstly to discuss the benefits of applying Bayesian models over the frequentist approach, and secondly to provide the first luminescence-based chronologies for Middle-Upper Palaeolithic key sites in Iran. Due to its location at the intersection between Africa, Europe and Central Asia, the Iranian plateau is of importance in tracking human dispersal over time. Despite this, there are very few chronologies spanning the Palaeolithic period in Iran. This thesis focuses on three key sites: Mirak, located at the margin of the central Iranian desert, the site of Ghār-e Boof in the southern Zagros Mountains, and Bawa Yawan in the central Zagros Mountains. Bayesian modelling of chronologies produced for the site of Mirak, date Upper, Intermediate and Middle Palaeolithic assemblages to 21–28 ka, 26–33 ka and 43–55 ka (95% CI) respectively. For the site of Ghār-e Boof, the Upper Palaeolithic culture was dated to 37–42 ka (95% CI) and the Middle Palaeolithic culture to 44–84 ka (95% CI). Elsewhere in Bawa Yawan, ages spanning 56–90 ka (95% CI) were obtained for assemblages attributed to the Middle Palaeolithic. The chronological study also revealed an age range of 12–16 ka for a unit attributed to the Epipalaeolithic culture.

Based on the study of these sites, this thesis explores the essential aspect of applying Bayesian methods in luminescence dating. The ideas which were particularly discussed were the benefits of Bayesian models to address systematic shared errors between samples and the ability to include independent chronological information, such as stratigraphic constraints or radiocarbon ages. This has helped to improve the precision of the Palaeolithic chronologies for Iran. Furthermore, this thesis tests the ability of Bayesian models to obtain an accurate central dose for well-bleached samples compared to one frequentist model, when quartz signals are close to saturation or when beta-dose rate heterogeneity in the surrounding sediment is high.

A PDF version of this thesis is available on request the author at [mariheyd@gmail.com](mailto:mariheyd@gmail.com).

**Maike Nowatzki**

**Aeolian Landforms as Palaeo Wind Indicators - Application of Geoinformatics and Optically Stimulated Luminescence Methods to Analyse Dunefields in the Ili-Balkhash Region (Kazakhstan)**

*June 2020*

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*Degree: M.Sc.*

*Supervisors: Hans-Joachim Rosner (Department of Geography, University of Tübingen), Kathryn Fitzsimmons (Max Planck Institute for Chemistry, Mainz)*

Drylands currently cover almost half of the earth's land surface and are predicted to extend further due to human and climate change induced desertification. A significant part of the world's population will thus be affected by land degradation and other dryland hazards such as sand and dust storms or dune encroachments. The uncertainty of future developments render dryland research essential in order to provide scientific input for policy making. Palaeoclimatological studies in dryland settings as well as the examination of fundamental processes in dryland systems such as dunefields are therefore crucial. This study's research area, the Ili-Balkhash region, provides an optimal setting for such studies due to its richness in aeolian dryland forms. Additionally, Central Asia is predicted to be particularly challenged by the effects of climate change.

This thesis attempts to tackle the reconstruction of wind directions from dune orientations using remote sensing and GIS (Geographic Information Systems) methodology. A semi-automated algorithm for dune mapping and the quantification of dune orientations is presented and fully applied to two, partly applied to four dunefields in the Ili-Balkhash region. The algorithms are implemented using a combination of JavaScript and Python scripts run on the Google Earth Engine and pyQGIS, respectively. Dune mapping is conducted applying object-based image analysis (OBIA). Differ-

ent types of primary data (optical satellite imagery and a digital elevation model) as well as secondary data derived thereof (filtered satellite imagery, dune height, slope data, and a vegetation index) are used to perform image segmentation and subsequent supervised classification. The calculation of dune orientations is exercised applying oriented bounding boxes around the dune features that have resulted from the image classification.

In order to connect dune orientations to (palaeo) wind behaviour, modern wind data is used to test for its compatibility with dune orientation patterns in three dunefields. Bedform trends are predicted from wind data based on the maximum gross bedform-normal transport rule (MGBTNT). Additionally, Optically Stimulated Luminescence (OSL) analyses are conducted to date selected dunes and examine if OSL behaviour can be used to draw conclusions about sediment provenance.

The bedform trend prediction yields partial consensus with the observed dune orientations, suggesting that modern wind regimes might not exclusively be responsible for the present dune morphologies. Given the Middle Holocene ages that were established for two dunes, a difference between dune-forming wind regimes and the modern wind regime appears possible. However, further research taking into account other parameters influencing dune orientation (e.g. sediment supply) needs to be conducted to confirm this hypothesis.

A pdf of this thesis can be acquired by contacting the author ([maike.nowatzki@ouce.ox.ac.uk](mailto:maike.nowatzki@ouce.ox.ac.uk))

**Miriam Saleh**

**TL, OSL and IRSL dating on ancient ceramics in the context of the European project nEU-MED**

*October 2020*

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*Degree: M.Sc.*

*Supervisors: Anna Galli, Letizia Bonizzoni*

The aim of this thesis was to compare the data obtained with OSL and IRSL dating on ceramic materials with those achieved with the well-established thermoluminescence (TL) dating protocols. The work was carried out at LAMBDA Laboratory (Laboratory of Milano Bicocca University for Dating and Archaeometry) of Material Science Department of Milano-Bicocca University.

In detail, the main goal of nEU-MED European project is evaluating the economic development from the 7<sup>th</sup> to the 9<sup>th</sup> century between Northern Europe, and the territories of southern Europe still influenced by the late Roman domination. To understand difference among these situations nEU-MED's archaeologists spent on the study of ceramics artefacts. The area chosen for the analysis was the northern Maremma (Tuscany).

Twelve samples belonging to three different archaeological sites, were prepared with the “fine-grain” technique, 30 aliquots each one. TL dating was performed using a home-made system developed in LAMBDA Laboratory and based on the photon counting technique with a photomultiplier tube (EMI 9635QB) coupled to blue filters (Corning BG12). For OSL and IRSL measurement has been used the Risø TL / OSL system DA-20. Artificial irradiations were carried out by a 1400 MBq  $^{90}\text{Sr}$  -  $^{90}\text{Y}$  beta source and a 37 MBq  $^{241}\text{Am}$  alpha source. For the dose rate evaluation, internal annual alpha and beta dose-rates were obtained by total alpha counting with ZnS scintillator discs and flame photometry analysis. For the measurement of paleodose with thermoluminescence, we applied the dose additive protocol (beta imparted dose: 3.51, 7.02 and 10.53 Gy). Moreover, OSL and IRSL measurements were carried out by applying the SAR protocol. The pre-heat value was experimentally derived based on the results of a dose recovery pre-heat plateau test.

By applying the recycling ratio, recuperation, and dose recovery tests, it was evident that not all samples showed the minimal criteria of acceptability. The main problem associated with OSL and IRSL dating was a low natural signal, especially in OSL measurements, perhaps due to low signal intensity emitted by quartz. As well as some TL measures are afflicted by low reproducibility of natural glow peak and saturation of signal.

Afterwards, we identified five samples datable by at least two of three dating techniques. The thermoluminescence results cover a large time span (7<sup>th</sup> – 11<sup>th</sup> centuries): two samples are assumed to be 7<sup>th</sup>-century potteries ( $618 \pm 61$  AD;  $665 \pm 66$  AD), while three ceramics belong to the 10<sup>th</sup>-century ( $999 \pm 63$  AD;  $976 \pm 95$  AD;  $1114 \pm 100$  AD). These two conclusions are in very good agreement with historical records and age hypothesized in advance by archaeologists, who assume in the analysed area a preponderant human presence between the 8<sup>th</sup> and 11<sup>th</sup> centuries. Furthermore, the IRSL results are to be considered good compared to poor results received from the OSL analysis. In fact, IRSL dating corresponds to the age given by the thermoluminescence, considered the error range. The potteries dated by TL at 7<sup>th</sup> century, give the same results with IRSL dating ( $687 \pm 16$  AD;  $574 \pm 25$  AD). One sample of the already mentioned group of three samples confirms the previous TL result ( $939 \pm 29$  AD), the last two datable samples appear to be younger on the IRSL analysis ( $1311 \pm 65$  AD,  $1514 \pm 74$  AD). On the other hand, the OSL measures, in addition to being affected by high errors, do not coincide with the results of the TL and IRSL measures.

Although the obtained data are very preliminary, it was still possible to provide further information to complete the historical information concerning nEU-MED project aim. In addition, the good results of the IRSL analysis suggest that the ceramic body is dominated by feldspar minerals instead of the quartz. Moreover, we can consider this thesis a preliminary assessment of the comparison between the techniques used.

**W.M. (Marijn) van der Meij**

**Co-evolution of soils and landscapes in the Anthropocene – from natural to intensively managed landscapes**

*November 2020*

Department of Environmental Sciences, Wageningen University and Research, Wageningen, The Netherlands

Degree: Ph.D.

Supervisors: Jakob Wallinga, Michael Sommer, Arnaud J.A.M. Temme

The aim of my thesis is to identify and quantify how soils and landscape have evolved and possibly co-evolved during the transition from natural land cover to intensive land management in the Anthropocene.

As study site I use the landscape laboratory CarboZALF D. CarboZALF D is a kettle-hole catchment of 4 ha with elevation differences up to 8 meters, located in north-eastern Germany. The colluvium in the closed kettle hole catchment provides a complete geo-archive of landscape change. We reconstruct the paleosurface of study site Carbo-ZALF-D prior to the anthropogenic erosion. We used an extensive dataset of soil descriptions, which enabled a detailed spatial estimate of erosion and deposition by estimating erosion based on soil profile truncations and deposition based on colluvium thickness. We reconstruct the rates of deposition in Carbo-ZALF-D using Optically Stimulated Luminescence (OSL) dating. The reworking of colluvial sediments by tillage causes two challenges for OSL dating: grains with different depositional ages become mixed and grains become exposed to daylight even long after they are deposited. We present a novel methodology where we combine OSL dating with advanced age modelling and an archaeological reconstruction of historical land use to correct for this post-depositional bleaching. Our results show a 100-fold increase in deposition rates, starting around 5000 years ago. The kettle hole shows a complex spatiotemporal pattern of colluvial infilling and landscape evolution, which we were only able to reconstruct using a high OSL sampling density and extensive soil geomorphic research.

To simulate the evolution of soils and landscapes under varying climatic and anthropogenic forcing, we review the role of water as dominant driver in natural soil and landscape evolution and its potential as driver in simulations with soil-landscape evolution models (SLEMs). The co-evolution of soils, topography and the hydrological system is essential for understanding the response of soils and landscapes to changes in climate. However, this co-evolution can currently not be simulated over long timescales with SLEMs due to several conceptual and methodological challenges. We provide partial solutions for these challenges to develop our SLEM HydroLorica. HydroLorica simulates soil and landscape evolution with various dynamic drivers such as water flow, vegetation type and land use. We included additional essential processes such as tree throw, soil creep and tillage. We use HydroLorica to simulate the evolution of soils

and landscape under various rainfall and land-use scenarios for an artificial undulating landscape. The results show that cultivation of natural landscapes increases soil heterogeneity, but also increases correlations between soil and terrain properties. Our results confirm that humans have become the dominant soil forming factor in intensively managed landscapes.

The development of HydroLorica, with water flow as explicit driver and with increased process coverage, is a big step forward in soil-landscape evolution modelling. A combination of reconstruction and simulation methods is essential for developing and testing hypotheses of soil-landscape co-evolution. Soil-landscape evolution in natural and intensively managed landscapes have different characteristics due to different driving forces and dominant processes. In intensively managed landscapes, disturbance rates are much higher than in natural settings. As a consequence, slowly developing soil properties degrade, while fast-developing soil properties can form a new equilibrium. As a consequence, co-evolution does not occur in the sense that it does in natural settings, because interactions between landscape components are missing. However, the management of soils and landscapes is often adapted to counteract unintended changes to soils and landscapes under earlier management. In intensively managed landscapes, land management may thus co-evolve with the rest of the landscape.

A PDF of this thesis can be downloaded from: <https://www.researchgate.net/publication/341670234>

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Compiled by Sébastien Huot

From 1st June 2020 to 31st November 2020

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# Ancient TL

ISSN 2693-0935

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Ancient TL is a journal devoted to Luminescence dating, Electron Spin Resonance (ESR) dating, and related techniques. It aims to publish papers dealing with experimental and theoretical results in this field, with a minimum of delay between submission and publication. Ancient TL also publishes a current bibliography, thesis abstracts, letters, and miscellaneous information, e.g., announcements for meetings.

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