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# Recent archaeomagnetic studies in Slovakia: Comparison of methodological approaches

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**Abstract:** We review the recent archaeomagnetic studies carried out on the territory of Slovakia, focusing on the comparison of methodological approaches, discussing pros and cons of the individual applied methods from the perspective of our experience. The most widely used methods for the determination of intensity and direction of the archaeomegnetic field by demagnetisation of the sample material are the alternating field (AF) demagnetisation and the Thellier double heating method. These methods are used not only for archaeomagnetic studies but also help to solve some geological problems. The two methods were applied to samples collected recently at several sites of Slovakia, where archaeological prospection invoked by earthwork or reconstruction work of developing projects demanded archaeomagnetic dating. Then we discuss advantages and weaknesses of the investigated methods from different perspectives based on several examples and our recent experience.

**Key words:** archaeomagnetism, Thellier method, stepwise demagnetization

## 1. Introduction

The typical archaeomagnetic material used for dating is heated clay like ceramics, bricks, oven's cores and so on. This material had been heated in the past to high temperatures, which upon subsequent cooling gave rise to the record of the ancient magnetic field of the Earth in this material. To obtain relevant information about past magnetic field, different techniques were developed. The first who studied the magnetic behaviour of archaeological materials was Thellier (*Thellier and Thellier, 1959*). The Thellier-Thellier methodology was established for determination of paleointensity and pale-

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odirections. Over time the methodology went through some changes and improvements. Some time later a methodology based on applying alternating fields to samples to get paleodirecitions came to practise. These methods are used for processing both archeomagnetic and geological materials (*Biggin and Thomas, 2003; Riisager and Riisager, 2001; Paterson et al., 2015; Chauvin et al., 2005; Dunlop et al., 2005; Schnepp, 2003; Dunlop, 2009; Shaar et al., 2011; Dunlop and Özdemir, 2001; Dunlop, 2011*).

For a comprehensive comparison of the benefits and yields of these two methodological approaches field samples from sites Bratislava, Čierne Kľačany, Nitra, Šurany and Triblavina were collected and studied in laboratory. The main objectives and requirements of archaeologists were dating of the last firing at these objects (in case of ovens), or the last exposure to fire (in case of damage and destruction by fire). The need for our archaeomagnetic research (dating) at the discussed study sites arose due to legislative archaeological research invoked by earthwork or reconstruction work at several construction sites: Bratislava, Čierne Kľačany, Šurany, Nitra, and Triblavina. The stepwise AF demagnetisation measurements were performed on samples from all localities, while the Thellier method of double heating only on samples from localities Bratislava and Triblavina.

## 2. Sites and samples

*Bratislava*

The site of our field work in Bratislava is situated on the western side of the northern castle courtyard of the Bratislava Castle. Legislative archaeological research was invoked by the intended construction of underground garages. Many burnt objects were discovered, a wall, a warehouse and one oven composed of bricks. Even carbonized pieces of cereals were discovered. Blocks of samples were taken from these objects, as well as four entire bricks from the oven. Each block was independently oriented. Samples from the burnt wall were damaged during cutting. Seven samples were selected, samples cut from the burnt warehouse. They were subjected to stepwise AF demagnetisation measurements. Measurements were performed from 0 to 16 mT with a step of 2 mT, then to 50 mT with step 5 mT and to 90 mT with step 20 mT. Six samples from bricks of the oven were selected for thermal demagnetisation using the Thellier method of double heating.

Measurements started at room temperature, and proceeded with heatings to 150 °C, 195 °C, 235 °C, 270 °C and subsequently continuously with a step of 40 °C. The archaeological expectation for the dating of this site was Late Stone Age.

*Čierne Kľačany*

Legislative archaeological research was situated in the cadastral territory of the village Čierne Kľačany. During excavation a house oven was discovered. Four oriented sample blocks were taken from the oven. They were cut into 22 samples. All samples were used for stepwise AF demagnetisation measurements. Measurements were performed from 0 to 27 mT with step 3 mT, then to 50 mT with step 5 mT and to 90 mT with step 20 mT. The archaeological expectation for the dating of this site was around 4000 BC.

*Nitra*

The studied site is situated on the castle hill. The legislative archaeological research was conducted because of the intended construction of a restaurant. Three ovens were discovered. From these, independently oriented blocks were taken and cut into 36 samples. All samples were used for stepwise AF demagnetisation measurements. Measurements were performed with similar demagnetisation steps like samples from locality Čierne Kľačany. The archaeological expectation for the dating of this site was within the interval 11$^{th}$ to 14$^{th}$ century.

*Šurany*

Legislative archaeological research was situated in the cadastral territory of the village Šurany. A house oven was unearthed there, from which one oriented block sample was extracted and cut into eight samples. All samples were used for stepwise AF demagnetisation performed with similar demagnetisation parameters like samples from locality Čierne Kľačany. The archaeological expectation for the dating of this site was around 200 AD.

*Triblavina*

During legislative archaeological research along highway D1 Bratislava–Trnava, associated with earthwork for the express highway bypass of the capital city Bratislava, two house ovens were unearthed at the cross-section Triblavina. Independently oriented blocks were extracted from both ovens and subsequently cut into 52 samples. Of these, 12 samples were selected for measurements of the Thellier double heating method, 6 per each oven. In addition, 40 samples were prepared for stepwise AF demagnetisation. The demagnetisation measurements were performed with similar values like samples from locality Čierne Kľačany. The thermal demagnetisation procedure parameters were the same as those used for locality Bratislava. The archaeological expectation for the dating of this site was within the interval $13^{\text{th}}$ to $14^{\text{th}}$ century.

## 3. Methods

### 3.1. Stepwise demagnetisation

During demagnetisation, or in other words during magnetic cleaning, the sample is exposed to an increasing magnetic field in field-free space, i.e. space shielded from the natural ambient magnetic field. The magnetisation of the sample is remeasured after each step of the demagnetization. An alternating field with peak value of magnetic induction $B$ demagnetises all grains with microscopic coercive force:

$$B_C < B \cos\theta \,,$$

where $\theta$ is an angle between $B$ and the coercive force ($B_C$) of a microscopic grain.

The results of stepwise AF demagnetisation can be visualised by Remasoft 3.0 software (*Hrouda and Chadima, 2006*). The example of Fig. 1 shows a sample with single domain behaviour with one dominant direction. Fig. 2 shows a sample with multidomain behaviour with more possible directions. Additional information (*Tauxe, 2002*) is necessary for unique specification. The examined specimens are from archaeological excavation from Slovak sites in Bratislava (Fig. 2, left), Čierne Kľačany (Fig. 2, right), Nitra (Fig. 1, left) and Šurany (Fig. 1, right).

### 3.2. Thellier method

The Thellier method of double heating is one of the most widely used meth-

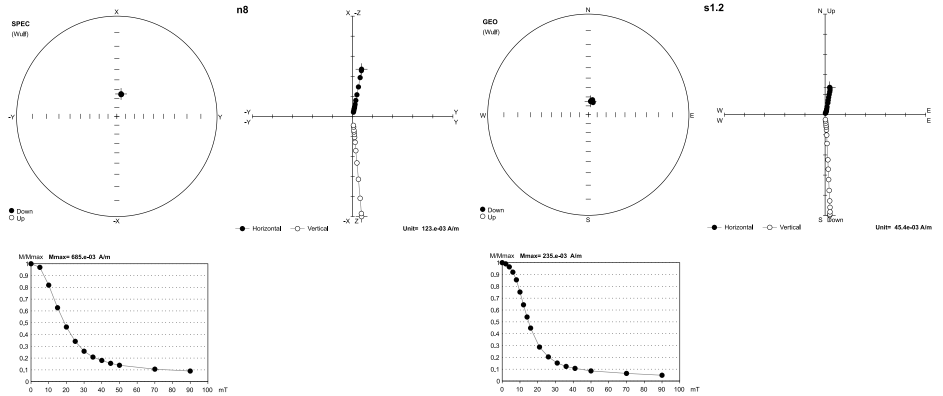


Fig. 1. Detailed record of stepwise AF demagnetization and principal component analysis of two samples. Top left – a stereographic projection of the natural remanent magnetization (NRM) of the sample in the natural state (crossed symbol) and after progressive AF demagnetization. Top right – Zijderveld diagram (*Zijderveld, 1967*), solid circles represent projection on the horizontal plane (XY), open circles represent projections on the north–south vertical plane (XZ). Bottom left – a graph of normalized values of the remanent magnetic moments versus demagnetizing fields; $M$ modulus of the remanent magnetic moment of a sample subjected to the AF demagnetization.

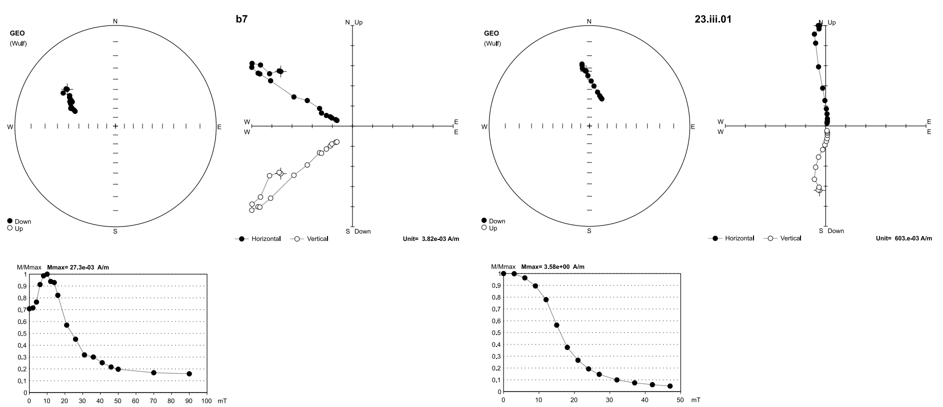


Fig. 2. Detailed record of stepwise AF demagnetization and principal component analysis of two samples. Top left – a stereographic projection of the NRM of the sample in the natural state (crossed symbol) and after progressive AF demagnetization. Top right – Zijderveld diagram (*Zijderveld, 1967*), solid circles represent projection on the horizontal plane (XY), open circles represent projections on the north–south vertical plane (XZ). Bottom left – a graph of normalized values of the remanent magnetic moments versus demagnetizing fields; $M$ modulus of the remanent magnetic moment of a sample subjected to the AF demagnetization.

ods at present for determination of magnetic paleointensity (*Thellier and Thellier, 1959*). Samples are heated up twice to the same temperature while they are oriented in two opposite directions: first to the magnetic south, and then to the magnetic north. Magnetic intensity, as well as direction, is measured at every temperature increment of the heating. The measuring starts at the temperature of about 25 °C (room temperature), and continues from 150 °C to the Curie temperature (700 °C, depending on the magnetic material of the sample) with a step of 50 °C. During the heating, the pTRM check needs be performed, too (see below). At temperature $T$ all grains with $Tb$ (blocking temperature) $T > Tb$ are demagnetised. Three components of magnetic intensity $\boldsymbol{I_s} = (x_s, y_s, z_s)$ are measured, when the sample is oriented to the magnetic south, and correspondingly $\boldsymbol{I_n} = (x_n, y_n, z_n)$ when the sample is oriented to the magnetic north. At each step of temperature and each orientation, also the magnetic susceptibility is measured. The dimensionless susceptibility is an indicator of phase transformations. For the magnetisation at the time of burning the material, referred to as the archeo-field, the following holds true:

$$J_A = \kappa H_A \,, \tag{1}$$

where $J_A$ is magnetization of the sample in the archeofield, $\kappa$ is magnetic susceptibility and $H_A$ is the intensity of the archaeomagnetic field. For magnetization at present we can write:

$$J_L = \kappa H_L \,, \tag{2}$$

where $J_L$ is magnetization of the sample in laboratory (actual) field, and $H_L$ is the intensity of the laboratory (actual) magnetic field. Formulae (1) and (2) are valid for every interval of temperature. $J$ and $H$ are scalars. The ratio of formulae (1) and (2) gives:

$$\frac{J_A}{J_L} = \frac{H_A}{H_L} \,. \tag{3}$$

From the graphical representation of the vector we get a formula for $J_A$:

$$J_A = \left| \frac{\boldsymbol{I_s} + \boldsymbol{I_j}}{2} \right| \,, \tag{4}$$

and for magnetization in laboratory field $J_L$ we get:

$$J_L = \left| \frac{\boldsymbol{I_s} - \boldsymbol{I_j}}{2} \right| .$$

The Thellier method is valid if:

- blocking temperature = unblocking temperature (law of reciprocity);
- the TRM acquired in the laboratory, i.e., is a linear function of the applied field (law of independence);
- the total TRM can be expressed of the sums of individual pTRMs (law of additivity).

The **pTRM check** is a control step in the Thellier method of double heating. It is similar to a normal step in the Thellier method. The step at lower temperature is repeated as the previous one. At higher temperature it is good to have partial termoremanent magnetisation (pTRM) check above the individual Curie temperatures of the individual minerals contained in the material. The NRM value of the pTRM check must stay at the last previous heating value, while the value of TRM must go back to the value of TRM of the same temperature. Graphical representation of regular and repeating control steps is shown in Fig. 3. A1 represents the first heating at temperature $T_2$ and A2 represents the second heating step at the same temperature. Below is the repeating control step at temperature $T_1 < T_2$, where B1 represents the first heating and B2 the second heating. The minus sign represents the change of magnetic field into opposite direction (*Spassov, 2016, personal communication*).

The Thellier-Thellier method of double heating was used by us on specimens from two ovens from locality Triblavina. The sample from the first oven shows stable values of susceptibility (Fig. 4 left, green). The demagnetisation curve has an expected descending character and the pTRM checks are correct. The acquisition curve has an ascending character and all pTRM checks, except for the last one, are in good position. The TRM is well acquired. The Arai plot is a graphical representation of the residual NRM in view of the partial TRM (*Nagata et al., 1963*). The Arai plot has a straight character, selected green points are the best for fitting (Fig. 4, right). The sample from the second oven probably had not been heated to a sufficiently high temperature (Fig. 5). We can see a change of the susceptibility value at about 300 °C. The acquisition of TRM has a higher value then the initial value of the NRM already at low temperatures. As early as the second

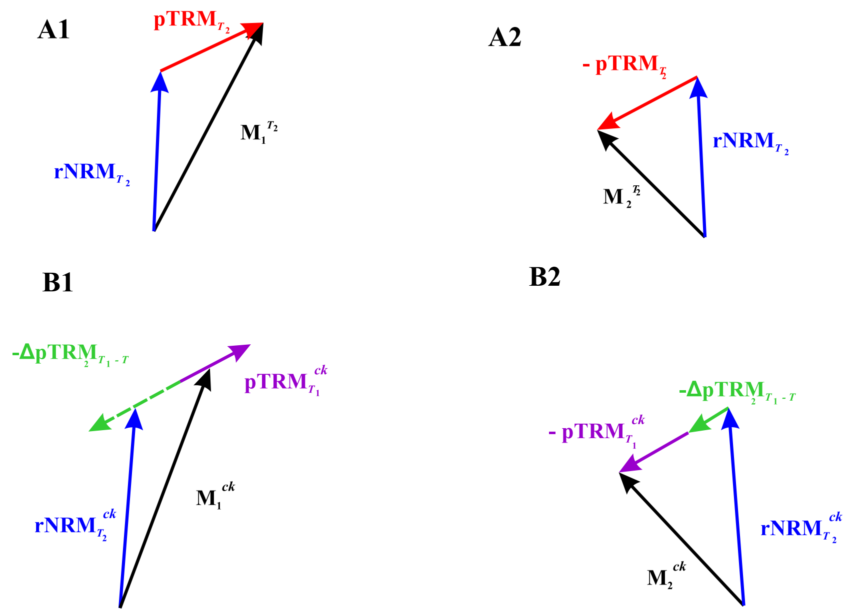


Fig. 3. Top: Regular step (A1, A2) at temperature $T_2$ by using Thellier-Thellier method. A1 is the first heating and A2 is the second heating step. $\mathrm{M}_1^{T_2}, \mathrm{M}_2^{T_2}$ are the measured vectors. The rNRM is a corresponding residual natural remanent magnetisation and pTRM is the corresponding partial thermoremanent magnetisation. Bottom: Repeating control step (B1, B2) at temperature $T_1 < T_2$ by using Thellier-Thellier method. B1 is the first heating and B2 is the second heating step. $\mathrm{M}_1^{ck}, \mathrm{M}_2^{ck}$ are the measured vectors. The ΔpTRM is a partial thermoremanent magnetisation as a residual component caused by heating to a lower temperature like the temperature of the last heating step.

pTRM, the check has a high value of TRM. The Arai plot does not have a straight character. It is possible to select three points for fitting, which is not enough.

## 4. Results

*Bratislava*

Both investigation (dating) approaches were applied to samples of each examined object from locality Bratislava. The behaviour of samples was inconsistent comparing the two approaches. Samples used for the Thellier

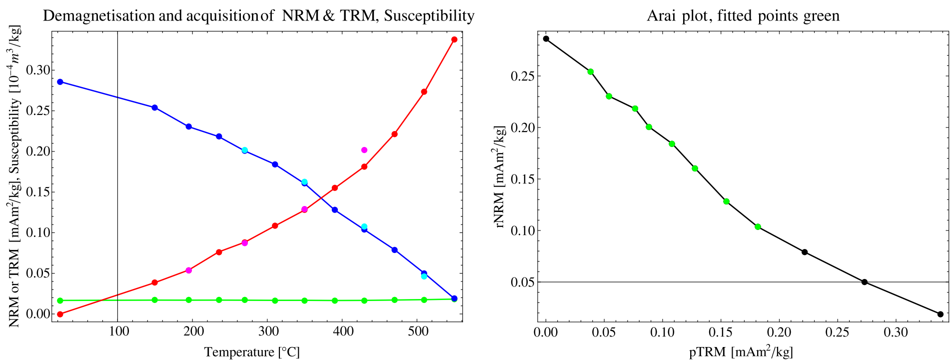


Fig. 4. Left: Demagnetisation of the NRM (blue), acquisition of the TRM (red) and susceptibility (green) measurements during the Thellier-Thellier double heating method. The pTRM control step of the demagnetisation of NRM (light blue) and the pTRM control step of the acquisition of TRM (pink). Right: The Arai plot. Green points are selected for fitting and evaluation.

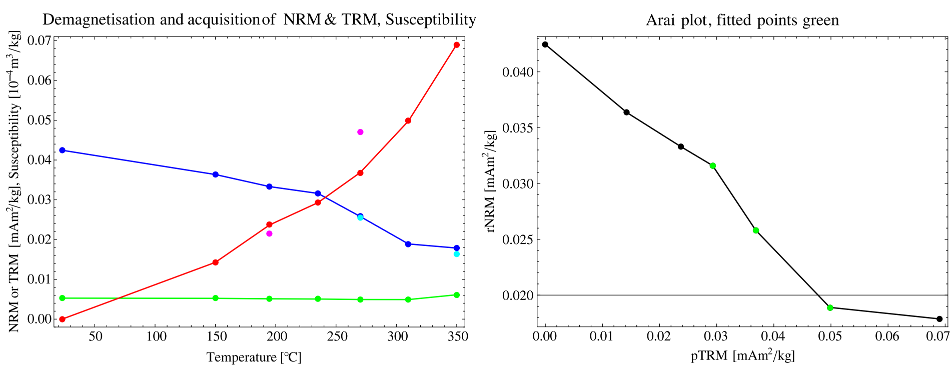


Fig. 5. Left: The demagnetisation of the NRM (blue), acquisition of the TRM (red) and susceptibility (green) measurements during the Thellier-Thellier double heating method. The pTRM control step of the demagnetisation of NRM (light blue) and the pTRM control step of the acquisition of TRM (pink). Right: The Arai plot. Green points are selected for fitting and evaluation.

method did not display a consistent behaviour and the results are meaningless. From seven samples investigated by the stepwise AF demagnetisation only two samples displayed a stable direction. The remaining samples exhibited an inconsistent and unpredictable behaviour.

*Čierne Kľačany*

Stepwise AF demagnetisation was applied to all samples. Samples were stable and consistent. Some samples have a small overprint that was usually removed with the step of 5mT. Only one sample exhibited an unstable direction, which was excluded from the results. This archaeomagnetic dating has a good correlation with the archaeological expectations (estimates).

*Nitra*

Stepwise AF demagnetisation was applied to all samples from locality Nitra. Ninety percent of the samples have a stable direction usually with a small overprint. Other samples had more than one component, or were unstable, and had to be excluded from further processing. Our archaeomagnetic dating of all three ovens has a good correlation with the archaeological expectations.

*Šurany*

Eight samples from locality Šurany were used for stepwise AF demagnetisation. All samples could be used for further processing. They had a stable direction, single domain behaviour and consistent results. The archaeomagnetic dating of the oven shows a bit later time interval than that of the archaeological estimation.

*Triblavina*

Samples from two ovens were used for both the stepwise AF and the thermal demagnetisatios. Results from the Thellier method of double heating show that the first of the two ovens had not been heated sufficiently enough. The second oven exhibited consistent results for each sample. The stepwise demagnetisation of samples from the first oven gave stable and consistent results for all nine investigated samples. The stepwise demagnetisation was applied to all 31 samples of the second oven. Two samples had an unstable direction and had to be excluded. The remaining samples were consistent and stable with very good precision. Our archaeomagnetic dating of these ovens yielded coincidence with the archaeological estimation based on the identification and classification of excavated ceramics. Results from both methods are consistent, but the Thellier method appears less accurate.

## 5. Discussion and conclusions

Methodical comparison was performed on samples from archaeological excavation of legislative archaeological research in Bratislava, Čierne Kľačany, Nitra, Šurany a Triblavina. During excavation ovens and burnt buildings were discovered that were suitable for archaeomagnetic research. From these objects samples were taken mainly for estimation of the time of the last firing of (at) these objects. The Thellier method of double heatings was performed on samples from sites Bratislava and Triblavina. Only results from site Triblavina were suitable for dating. Results from locality Bratislava were ambiguous. Stepwise AF demagnetisation was performed on samples from all sites. All sites except for Bratislava exhibited consistent results in most cases during the stepwise demagnetisation measurements. Our archaeomagnetic dating gave results comparable with the archaeological expectations. The ambiguity of the results from the Bratislava Castle could be caused by the continuing occupation of the locality. During this time several diverse heatings could have occurred, caused by different reasons (fire, blaze, rebuilding, ...).

To study paleointensity (archaeointensity) and paleodirections (archaeodirections) of the archaeomagnetic field, two methods were applied: the AF demagnetization and the Thellier-Thellier double heating method, in order to compare the pros and cons of both methods.

The Thellier method of double heating is used for estimation of paleointensity and paleodirections from archeological material. This method is time-consuming and results could be uncertain. Usually the heating exceeds the Curie temperature of the investigated sample. It can cause phase transitions that change the magnetic properties of the sample. For this reason it is necessary to perform control steps, in terms of pTRM checks. If the studied sample was not very well (sufficiently) heated in the past, at its origination of the archeofield, the results at higher temperatures become meaningless.

The stepwise AF demagnetisation is faster for the determination of paleodirections, than the Thellier-Thellier method. Samples are not heated, therefore the risk of phase transitions caused by heating is eliminated. The drawback can arise if a sample has a multidomain behaviour, in which case more than one stable direction can appear. Paleodirections could be separated with additional information and including additional methods and techniques (*Aidona et al., 2001; Batler, 2006; Roberts, 1997; Li et al., 2008*).

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