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User's guide for paleointensity experiments: an update of the Guidebook "Guida all'uso del forno ASC TD-48SC per la stima della paleointensità assoluta con il metodo Thellier-Thellier modificato"

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User's guide for paleointensity experiments: an update of the Guidebook "Guida all'uso del forno ASC TD-48SC per la stima della paleointensità assoluta con il metodo Thellier-Thellier modificato"

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Cover ASC Model TD48 Thermal Demagnetizer (picture by https://www.ascscientific.com/) | *In copertina* ASC Model TD48 Thermal Demagnetizer (foto da https://www.ascscientific.com/)

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Abstract

The understanding of the ancient Earth's Magnetic Field strength, or paleointensity, is fundamental for several disciplines of Earth Sciences. Following the growing interest in this field in the last decades, the number of paleointensity studies has been increasing and the Paleomagnetic Laboratory of the Istituto Nazionale di Geofisica e Vulcanologia (INGV) in Rome, Italy, expanded the instrument park with a new ASC TD-48SC oven for paleomagnetic and paleointensity measurements. This manual represents an updated version of the original manual "Guida all'uso del forno ASC TD-48SC per la stima della paleointensità assoluta con il metodo Thellier-Thellier modificato" [Di Chiara, 2014]. The aim is to provide a practical guide for paleointensity measurements, using the IZZI- Thellier experiment and the software Thellier_GUI for the processing of the geomagnetic field strength or paleointensity data.

Keywords Geomagnetic field strength or paleointensity; IZZI-Thellier method; PmagPy software

Introduction

The estimate of the absolute geomagnetic field strength, or palaeointensity, is fundamental for several Earth science disciplines (geophysics, geology, volcanology, archaeology, etc.) and can be obtained by applying different laboratory protocols to suitable rock samples. Several methods exist [Tauxe, 2010]: the double-heating Thellier-Thellier [Konigsberger, 1938; Thellier and Thellier, 1959] based methods, the multispecimen [Dekker e Böhnel, 2006], the non-heating methods [Shaw, 1974], microwave [Walton et al., 1992], among others. Because all methods are based on ideal single domains magnetite particles from the Neels theory, which may fail at describing the complexities found in natural samples, the percentage of success in the experiments are usually low, between 20 and 30%. Moreover, for the heating-based methods, it is required that the samples do not alter during the experimental procedures and the reciprocity between blocking and unblocking Temperature spectra is verified. While no agreement exists among the most reliable method to obtain absolute paleointensities, the most widely accepted is the Thellier and Thellier [1959]. The method is based on the step-wise thermal demagnetization of the sample, both in the absence (zero-field) and presence of a laboratory magnetic field (in-field steps). The strength of the ancient field is calculated as the ratio between laboratory field acquired and the Natural Remanent Magnetization lost during the heating/cooling protocol. The aim of this manual is to guide the user through the procedure to obtain absolute paleointensities.
This manual is an updated version of the original "Guida all'uso del forno ASC TD48SC per la stima della paleointensità assoluta con il metodo ThellierThellier modificato" ([Di Chiara, 2014], hosted at the Paleomagnetic Laboratory of the INGV). The ASC TD-48SC oven allows to set a constant laboratory field for the desired time, therefore all versions of the classical Thellier method can be performed with the same oven. The guide also introduces some theoretical elements about the methods and practical guide to the software for the interpretation and visualization of the data.
Compared with the first version of Di Chiara [2014], the new manual is written in in English in order to be accessible by a larger audience. This new version provides additional details about the sample preparation and the use of the *PmagPy* software [Tauxe et al., 2016] for data processing, updated since 2014, together with a new section for archeointensity studies (the investigation of the absolute paleointensity on archaeological artifacts, Section 7) dedicated to the anisotropy and cooling rate corrections. The section dedicated to the oven's operation and measurement protocol has not been modified from the first version.

1. The IZZI protocol

This guide adopts the IZZI protocol as an example for measuring absolute paleointensities. The IZZI was first introduced by Yu et al. [2004] and Tauxe e Staudigel [2004] and embeds two variants of the Thellier method, the IZ [Aitken et al., 1988], when a in-field step (I) is followed by a zero-field step (Z), and the ZI [Coe, 1967] where order of the steps is reversed, and a partial thermal remanent magnetization step (pTRM). For further details about the method the reader is referred to the papers by Yu et al. [2004] and Tauxe and Staudigel [2004].

1.1 The PmagPy software

The PmagPy software [Tauxe et al., 2016], based on the Python language-based Anaconda platform, can be used to both plan the experimental protocol and process the final data. The software package and the instructions for installations (both for Mac and PC) are available at the link: https://earthref.org/PmagPy/cookbook/. On the same link, there is a troubleshooting section and one to report bugs and contact the developers for help and questions. Since the program is constantly updated, a frequent consultation of the website and update of the program is recommended. Once successfully installed, one can proceed to plan the experiment (Section 2).

2. Planning the experiment

2.1 Planning the demagnetization steps for the experiment

In order to plan the spreadsheet for the experiment, you need to open the command prompt (or terminal for Mac) and launch the program *chartmaker.py*. *Please note that to run the "chartmaker.py" program, one needs to use DOS commands to enter in the PmagPy folder which contains the code.* The program allows to input the steps and the frequency of the steps (for instance a frequency of 100° C from 0 to 300° C, every 50° up to 450° C and every 10° up to 600°, Figure 1). The output of the program is a file, chart.txt, in which Tx.0, Tx.1 and Tx.2 and additional columns are displayed (Table 1). The Tx.0 represent the step at a given temperature T with no laboratory field switched on (zero-field), Tx.1 represent the step during which a constant field is switched (in-field), and Tx.2 represent an in-field step as a check the stability of the magnetic mineralogy (pTRM check). The reversibility of the Tx.2 pTRM check steps (triangles on the Arai plot of Figure 7C) ensures that the heating steps are not causing mineralogical alteration, which would invalidate the entire experiment.

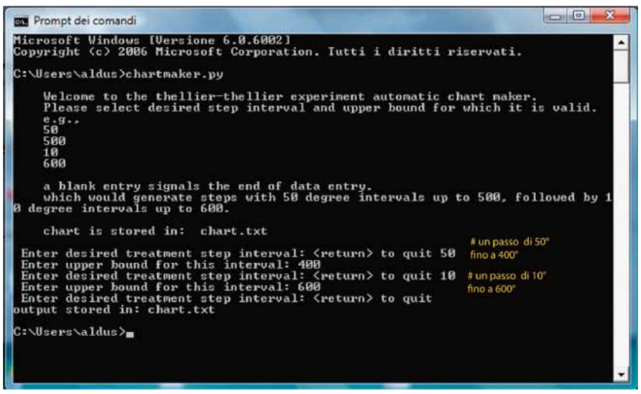

Figure 1 An example of the chartmaker.py program to plan the absolute paleointensity experiment using the IZZI Thellier modified protocol. The program outputs a table, "chart.txt", located in the same folder as the program. NB: to run the "chartmaker.py" program, one needs to use DOS commands to enter in the PmagPy folder which contains the code.

As a practical note, the laboratory field value is chosen arbitrarily. It is common practice that the value is picked to be close to the expected value of the geomagnetic field intensity for the given age of the samples.

It is advised to select the frequency of the steps as a function of the material analysed, thus its magnetic mineralogy (unblocking temperature spectra), and in general, to intensify the number of steps after 350° C and even more after 500° C. An example is shown in Figure 1, but a much detailed temperature steps is recommended (ca. 30-50 steps).

Table 1 **Example of the chart.txt table** where you can note the time at the beginning of the step (start), the time at which the set point temperature is reached (set point or sp) and the time at which the cooling of the step starts (cool). The set point will be reached at increasing time intervals with increasing temperature steps. It is recommended to keep the time at set temperature constant (20 or 30 minutes). Zone I, II e III refer to the magnetic field measured at three zones of the oven (e.g. close to the oven's door, the middle of the heating chamber and the end of the heating chamber. Reporting of such information is optional but it helps keeping a record of the stability of the field at the beginning of the in-field steps as well as how accurately zeroed is the field at the beginning of each zero-field step.

	date	run#	zone I	zone II	zone III	start	sp	cool
	0.0							
Z	100.0							
I	100.1							
I	200.1							
Z	200.0							
Z	300.0							
I	300.1							
I	400.1							
Z	400.0							
Z	450.0							
P	300.2							
I	450.1							
I	500.1							
Z	500.0							
Z	550.0							
P	450.2							
I	550.1							
I	600.1							

3. Sample preparation

The sample preparation depends on the kind of investigated materials.

3.1 Mini-cylinders

If the investigated material are lavas, intrusive rocks, coherent materials, one can use a desk drill with small drill bits, to produce mini-cylinders of 9.8 cm^3 volume (Figure 2) which need to be marked with an arbitrary azimuth and the specimen ID, with heat resistant paint.

3.2 Fragments

If the specimens need to be taken from previously collected samples (and/ or is not possible to drill mini-cylinders), like volcanic glass or archaeomagnetic material as ceramics, bricks, etc., one needs to gather the following items (Figure 2):

1. Glass tubes; in this example we are using glass tube "Vials, Shell, article number 609311 12, sized 12 x 35 e 0.5 DRAM", by Kimble Chase.
2. Microfiber glass paper; for instance, "Glass microfiber filters GF/D, 25 mm of diameter, Cat. No. 1823-025", by Whatman.
3. Mini-drill (e.g. Dremel) with thin metal bit or laser printer to mark the specimens and a non-magnetic marker to inscribe a reference azimuth line.
4. Kasil glue (kaolin + Sodium silicate).

Figure 2 Standard paleomagnetic sample (cylinders with 2.5 cm in diameter and 2.2 cm high), fragments cut from it, glass filters and vials (not marked in the picture).

It's recommended to pick 0.5 to 2 g fragments (using a high-precision balance and noting the weight), and check that the value of the Natural Remanent Magnetization (NRM) is high enough be measured with the 2G cryogenic magnetometer. To prepare the samples, follow this workflow:

1. Clean the empty glass vials using alcohol, and/or heating them at 600° C, and/or inducing an Isothermal Remanent Magnetization (IRM) and demagnetising them.
2. Along the long side of the vials, use the non-magnetic square and pen to mark the azimuth, and with the drill draw the identification number/name of the sample.
3. Prepare the Kasil.
4. Place one glass filter at the bottom of each vial, add some glue, place the sample fragment

(depending on the size it can be wrapped in a filter to ensure additional stability), place an additional filter (or two) to fix the sample in place and add glue. Please note that using an excessive amount of glue will not ensure that the sample will not move as the glue will melt and overflow while heating. Finally, the samples are kept in place mainly by the glass filter hardened by the glue, therefore it is better to wait overnight before starting the experiment to let the glue dry. During the experiment and especially at high temperature steps, one may add drops of glue to reduce the risk of cracks and movement of the specimen.

4. Use of the ASC TD-48SC Oven

The ASC TD-48SC oven (Cover image) is magnetically shielded, in order to reduce the ambient field disturbances, which would induce a bias in the final results, when the solenoid inside the oven is not active. It is a single chamber oven, where both the heating and cooling phases occur. Three thermocouples control the temperatures, connected with three controllers (Watlow brand, Figure 4), which regulate and maintain a constant temperature during the heating phase. The oven is equipped with two fans: one remains on for the entire operation of the oven while the second one turns on automatically when the delay timer sets off. It is recommended to turn off the oven, by the oven commutator, during the cooling phase. At the end of every heating/cooling cycle is recommended to reset the oven's timer by pushing the knob for three seconds. Because the set temperature is reached in a different time depending to the set temperature itself (more time is required to reach higher temperatures), it is recommended to initially set the timer of the fan for 60 minutes and then set the timer at 30 minutes once the desired temperature is reached; such procedure guarantee that the fan does not turn on during heating. To operate the oven please proceed as follows:

1. Load the samples in the sample holder (with the open side of the vials toward the gate of the oven so that while cooling the fan will not move them). During the in-field steps it is recommended to orient the azimuth of the vials parallel to the z axis of the oven.
2. Connect the thermocouple to the holder.
3. Turn on the electric switch of the oven (power) and regulate the desired temperature (setpoint adjustment, Figure 4).
4. Turn on the oven and the cooling fan, regulating the timer at a set time (e.g. 3 hours). Please note that the fan switching is controlled by the timer, therefore if the timer is regulated to 0 the fan will not turn on (useful for the 'cooling rate correction' on archaeomagnetic experiments, Section 7.2).
5. Once the oven (central control) reached the desired temperature, regulate the timer at desired time at the set point (for instance 20 or 30 minutes), thus triggering the countdown to turn on the cooling fan.
6. Once the fan is turned on, proceed to switch off the oven.
7. When the temperature is close to the room temperature, disconnect the thermocouple from the holder, remove the samples and measure the NRM with a magnetometer.

5. Controlling the laboratory field

The "BK precision" (model 1735A) needs to e connected with the solenoid terminals of the ASC TD-48SC oven (Figure 4), using it as current generator (red led turned on); if the desired field is for instance 40 µT, the generator need to produce 0.4 A current, bearing in mind that in this case (according the oven's manual) a current of 1 A is needed to produce a magnetic field of 1 G which (in vacuum) corresponds to 1 Oe = 1 G = 10^{-4}T.

To produce the magnetic field inside the oven, one need to turn on the current generator and adjust the current with the 'coarse' know first and then 'fine' knob (Figure 4). The user is recommended to check and adjust the value of the field during the entire experiment an especially during cooling when the field inside the oven tends to change slightly.
The correspondence between the current and the magnetic field produced inside the oven can be checked with a triaxial fluxgate, setting its measurement range at 1000 (thus the maximum measurable range is 999 mOe for each vector component). A 0.4 Oe axial field (parallel to the long horizontal axis of the oven, Figure 3) can be obtained by reading 400 mOe on *x* axis of the fluxgate and the component of the *y* and *z* axis is negligible.

5.1 In-field steps (Tx.1 and Tx.2)

a. Regulate the field using the "coarse" e "fine" knobs. Usually, the laboratory field is chosen between 10 and 70 µT, close to the expected field intensity for the age of the samples.
b. It is good practice to check that during the heating and cooling phase, the field doesn't vary from the chosen value. If it does, adjust the voltage value using the "fine" knob.

5.2 Zero-field step (Tx.0)

a. Before each zero-field step, check with the fluxgate that the magnetic field inside the oven is zero, especially along the long axis following an in-field step.
b. If the field is not zero, turn on the variac, insert the wooden stick with the coil connected to the variac, slowly increase up to 15 V and then decrease the field to zero. **Do not increase the value above 15 V**. Repeat the step 2 or 3 times and then check again using the fluxgate that the field inside the oven is finally zeroed.

6. Sample's measurement

After each step, samples can be measured as in the standard thermal demagnetization cleaning, with the 2G cryogenic magnetometer. The difference between the paleointensity experiments and the standard thermal demagnetization is that for each infield step one needs to manually add xx.1 e xx.2 decimal value according to the measured step.During the measurements, it is good practice to minimize the offset of the X, Y and Z SQUIDS to <|0.00050|, allowing to detect flux jumps and re-measure the sample. Indeed, the risk of a flux jump is greater at high temperature steps, because the difference between the remaining NRM and the new Thermal Remanent Magnetization (TRM) along the z-axis (parallel to the direction of the field in the oven) progressively increases.

7. Anisotropy and cooling rate corrections

When analysing archaeological samples for archaeointensity analyses, once the Thellier experiment is completed, the corrections of the anisotropy (ATRM correction) and for the cooling rate need to be applied.

7.1 ATRM correction

Multiple protocols exist to perform the ATRM correction. They are all based on the measure of the TRM along the three axis (x, y and z) rotated with respect to a constant laboratory field (Figure 3), at constant temperature chosen depending on the dominant magnetic mineralogy (for instance 585° C when magnetite dominates).

Before starting with the experiment, it is recommended to measure a step (step 0) at zero field to erase some residual signal. The example in Figure 3 shows the 7 steps for the ATRM correction, all at a constant field and at constant temperature. In the first three steps the x, y and z axis of the sample are oriented parallel to the field (note that the third step will be at the same orientation as the IZZI experiment). In the next three steps, the samples will be oriented opposite to the first three steps. The last step consists in repeating the step number 1. At the end of each thermal step, the TRM needs to be measured with the 2G cryogenic magnetometer and each step needs to me marked with a progressive decimal number following the temperature step (for instance "585.1" will represent the step number 1). It is suggested to add some glue at the end of each step to avoid the excessive drying and potential movement of sample inside the glass vials.

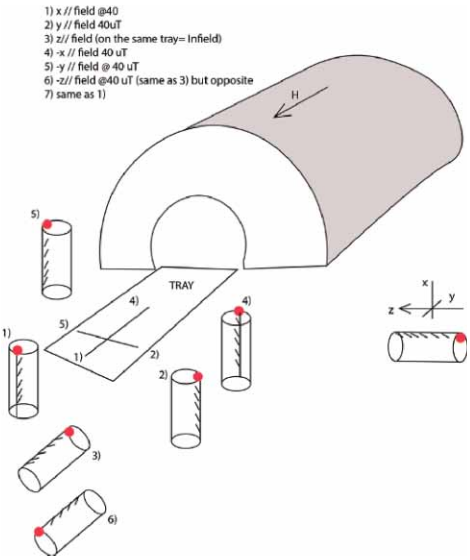

Figure 3 he 6 phases of the ATRM correction protocol where the azimuth of the sample is marked and highlighted with a red dot. The orientation of the field inside the oven is marked by the orientation of H.

7.2 Cooling rate correction

The cooling rate correction protocol consists in four steps, all at constant temperature (for instance 585° C) and some at constant field and the same orientation of the samples with respect to the laboratory field:

1. Step 0 at zero field and constant temperature (585° C).
2. Step 1, at a constant field and cooling using the fan.
3. Step 2 at a constant field and cooling, cooling without switching on the fan. This step may require up to 12 hours to reach room temperature.
4. Step 3 is the same as *step* 1.

At the end of each step, the TRM needs to be measured with the 2G cryogenic magnetometer, marking corresponding step with a progressive decimal number (for instance "585.0" will represent the step number 1).

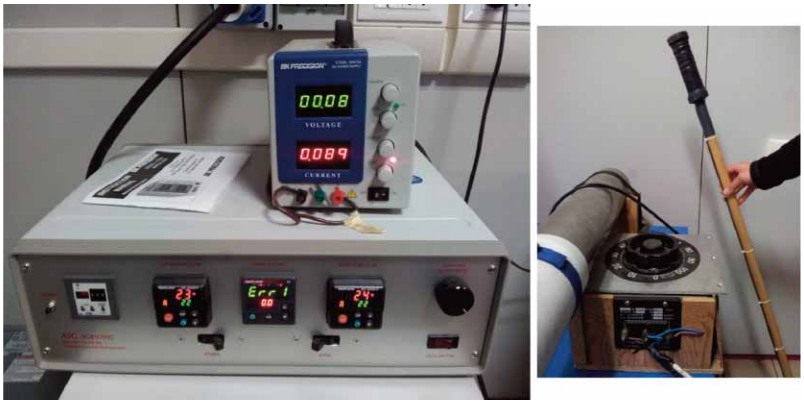

Figure 4 To the left, command panel to switch on the oven, control the temperature, regulate the timing for the fan. On top of the command box, there is the controller for the magnetic field inside the oven. To the right: variac to erase the residual field inside the oven. This step needs to be performed after each in-field step, Tx.1 o Tx.2, before a zero-field step, Tx.0.

8. Data processing

Once the measurement cycle is completed, the output measurement file needs to be converted in a readable format using the Thellier_GUI.py program [Shaar and Tauxe, 2013] (Figure 5) freely available to download both as a stand-alone and as part of the PmagPy software package. All updates are available at https://github.com/PmagPy/PmagPy. The same site contains several Jupyter Notebooks with useful examples on how to process and visualise paleointensity and paleodirectional data.
Thellier_GUI.py allows to import, convert, visualize, interpret and export the data. The YouTube channel contains a video for the installation step by step of the Pmag_GUI package, which allows to import and interpret all kind of paleomagnetic data. The package includes the Thellier_GUI for processing the paleointensity data (https://www.youtube.com/watch?v=vRDiIXCm-sY&t=4s).
Once the software is successfully installed, one can proceed as follow:

1. Launch *Pmag_GUI.py* using the *command prompt* or *terminal*, and select the working directory (figure 5).
2. Convert the measurement file ".dat" in a format readable by the program (Figure 6), populate the table with metadata including the kind of experiment (IZZI), laboratory field used, and naming convention (such as number of characters indicating the site/sample/specimen). For instance, SLM0101A can represent the location SLM, site 01, sample 01, specimen A.
3. Once populated, always select "OK" before "Go to next step".
4. Once the measurement file is converted, data can be processed.

Thellier_GUI.py (Figure 7) visualizes the data with an Arai plot (Figure 7C, natural TRM vs laboratory TRM normalized for the initial NRM), Zjiderveld diagram, Stereonet, Magnetization *vs* Temperature, the site-level statistics (Figure 7D), where only the specimens passing the selection criteria are shown.
The reliability of the data is assessed by a set of selection criteria, or *acceptance criteria*, which need to be chosen from the menu Analysis > Acceptance criteria. For an overview of all the parameters available the reader is recommended to consult the online book http://earthref.org/PmagPy/cookbook/ and the paper by Paterson [2011].

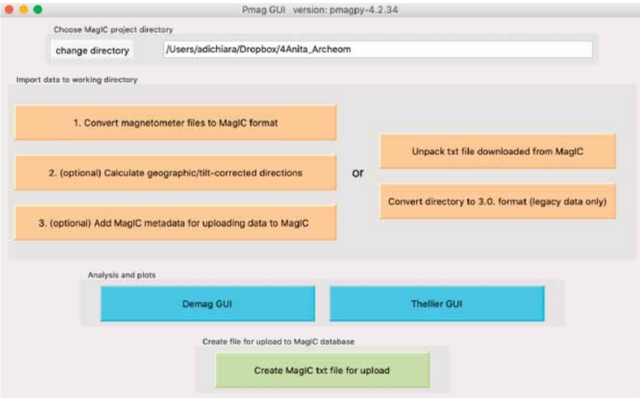

Figure 5 Example of the Pmag_GUI.py program, where one can change the working directory, import new data, convert them in a readable file and interpret them with Demag_GUI.py (for directional data) and Thellier_GUI.py (for intensity data).

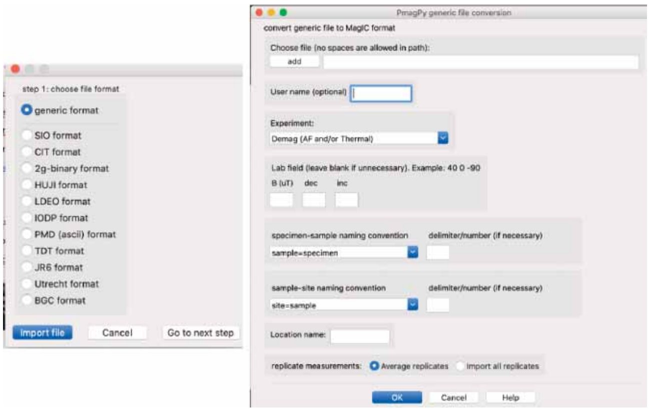

Figure 6 An example of the dialog window to import the data format (for instance 2g binary format) and go to the next step in which one can populate the metadata table with the kind of experiment, laboratory field (for paleointensity experiments). It is advised to fill all fields, insert zeros if not used.

In order to interpret the data one can manually select the temperature interval (Figure 7B) which best represent a straight line interpolating the data shown on the Arai Plot (Figure 7C). Another option is to run the Auto-interpreter tool from the main menu bar. At the end of the automatic interpretation, data are saved by default in the folder "thellier interpreter", located in the working directory. At the end of the analyses, data need to be saved as a Thellier.redo file (Analyses>save current interpretation to a "redo" file) and all results are saved as three separate files *sites.txt*, *samples.txt e specimens.txt* (File>Save MagIC tables). One can export the results as PDF file images (File>Save plot>...).

Figure 7 Example of the Thellier_GUI.py program [Shaar and Tauxe, 2013]. The menu bar allows to interpret and save the data. Specimen name and the Thellier experiment steps are shown (A). One can select a temperature step (B) and visualize the Arai plot (C). If the specimen passes the selection criteria, the specimen's statistic boxes will be marked in green (otherwise, they will be marked in red). All specimens passing the selection criteria contribute to the site level statistics (D).

Conclusions

The manual aims to guide the user throughout the process of paleointensity measurement using the ASC ASC TD-48SC paleointensity oven hosted at the INGV in Rome (Italy), by using as an example the IZZI protocol as described by Yu et al. [2004], Tauxe and Staudigel [2004] and Yu and Tauxe [2005]. The manual represents also a practical guide to absolute paleointensity data processing using the PmagPy package by Tauxe et al. [2016] and the Thellier_GUI software by Shaar and Tauxe [2013]. PmagPy is constantly updated and improved, therefore the user is recommended to consult the website http://earthref.org/PmagPy/cookbook/, both for additional details and for troubleshooting.

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ADC is grateful to the training received by the one and only Professor Lisa Tauxe and for her constant work together with her MagIC team to keep the PmagPy software updated, free and available for everyone.

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