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SAR TL DATING OF NEOLITHIC AND MEDIEVAL CERAMICS FROM LAMEZIA, CALABRIA (SOUTH ITALY): A CASE STUDY

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ABSTRACT

In this work some ceramic fragments from the neolithic and medieval period have been studied by Thermoluminescence (TL). Six samples of "*Stentinello*" ceramics found in the area of Acconia, in the "*Piana di Curinga*" (Lamezia Terme, Cz, South Italy) dating back to the VI-IV millenium B.C. and four samples of *Nicastro Castle and St. Eufemia Abbey* (Lamezia Terme, Cz, South Italy) dating back to the 1500 A.D. The samples were analysed by TL using the Single Aliquot Regenerative (SAR) dose protocol.

The so obtained results provided reliable age estimates for the following samples (the Italian labels refer to different archaeological sites in the same valley): Neolithic samples: "*Terravecchia*", age range 2700-6500 ± 500-1200 B.C.; "*Suveretta*", 4100 ± 700 B.C.; "*Romatisi*", 5100 ± 400 B.C.. Medieval samples: *Nicastro Castle*, 1690 ± 60 A.D. ; St. *Eufemia Abbey* 1600 ± 50 A.D..

The dating results are consistent with the typological characterization of the samples and in agreement with the historical period to which the fragments are supposed to belong.

KEYWORDS: Thermoluminescence, Equivalent dose, SAR protocol, Neolithic period, Medieval period.

INTRODUCTION

To classify and explain the historical context of archaeological artifacts when observation of the morphological and stylistic characteristics are uncertain, the Thermoluminescence (TL) dating of clay artifacts is used

Clay is a natural material. It comes from sedimentary rocks and debris of variable mineralogical composition, always formed by clay minerals and non-clay minerals, which consist of quartz, feldspar, calcium carbonates, oxides and hydroxides of iron and other minors as well as in organic matter and various random materials (Cuomo di Caprio, 2007).

TL is a technique that allows us to date pottery or terracotta in general, since their crystalline components, such as quartz or feldspars, are provided with intense luminescence signals (Aitken, M.J.,1985). Some crystals, like for example the quartz included in ceramics, when heated, are able to emit light in a manner proportional to the dose of ionizing radiation accumulated over time by the crystals themselves. Since their formation the clay components accumulate a "Geological dose" that they lose when heated at a temperature sufficient to empty all the electron traps. Once the object is deposited or is used, it begins again to accumulate a certain amount of absorbed dose *("Equivalent dose" or "Archaeological dose"* or *Paleodose")* due to the natural radiation coming from the ceramic itself (U, Th, K-40) and from the surrounding environmental gamma rays plus cosmic radiation. The subsequent heating to high temperatures in the laboratory, results in the release of the light corresponding to the dose accumulated over time. By reading the light emitted, it is possible to calculate the time elapsed since the original heating in the oven, according to the well known equation age (Aitken, 1985; Furetta and Gonzàlez Martinez, 2007) expressed in years:

Age = Paleodose (Gy) / Dose Year (Gy / year)

Where, the *"Dose Year"* is the annual natural radiation. Here we describe the research phases and the methodological criteria of TL dating of ceramics. The Neolithic and Medieval artifacts (see Figs. 1 and 2) are dated by a SAR protocol (Single Aliquot Regenerative-dose), and the obtained results will be compared with the historical data, to confirm the authenticity and classify the findings.

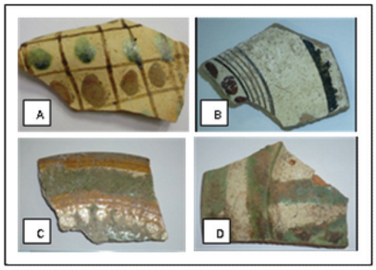

Figure 1. Medieval Samples: Sample CAST n°6 (A), found in *Nicastro Castle* and ABB n°7 (B), 8 (C) and 9 (D) found in *St. Mary of St. Eufemia Abbey*, Lamezia Terme (Cz).

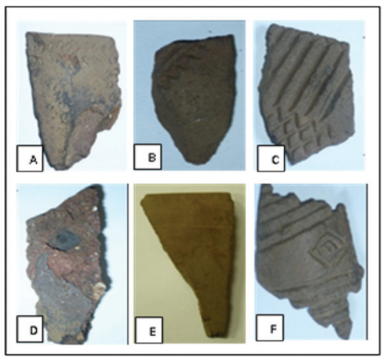

Figure 2. Neolithic Samples: found in different areas such as: *Terravecchia* (Samples TERn°1 (A), 2 (B), 3 (C) and 4 (E)); *Suveretta* (Sample SUVn°5 (D)); *Romatisi* district (Sample ROMn°6 (F)), Curinga (Cz).

We used the SAR TL method because it allows a more accurate estimate of the Paleodose (Equivalent Dose, *ED*) and a proper correction to problems arising from sample sensitivity (Gattuso et al, 2012; Murray and Roberts, 1998). Normally, in addition to TL, we use also the OSL methods to compare the results of the two techniques. In this particular case we use only the TL technique because we don't know exactly the history of the samples regarding their exposition to light.

SAMPLING AND HISTORICAL BACKGROUND

The *«Museo Archeologico Lametino»* is into the monumental complex of San *Domenico*, Lamezia (Catanzaro).

The exposure is divided in three different sections:

- Prehistory: Lamezia Valley *(Casella di Maida, Acconia, San Pietro Lametino);*
- Classical period;
- Middle Age: *Nicastro Castle, St. Eufemia Abbey.*

In the Prehistory section there are the most ancient tools used by Paleolithic hunters which lived in the region. Furthermore we can find also artifacts that prove the presence of Neolithic farmers in the Lamezia Plain *(Casella di Maida, Acconia, S.Pietro Lametino),* since 7500 years ago.

Neolithic Samples are found in different places in Curinga (CZ) such as: *Terravecchia* (the samples are termed as TER n°1, 2, 3 and 4 hereafter); Suveretta (the sample is termed as SUV n°5 hereafter); *Romatisi* district (the sample is termed as ROM n°6 hereafter).

The Neolithic findings (Fig. 2) are all impressed ceramics and, in the first analysis, they have a dark brown mixture and a kind of clay which more or less purified.

The six samples (Fig. 2) of "*Stentinello*" ceramics have been found in the area of *Acconia*, in the *"Piana di Curinga"* (Lamezia Terme, Cz, South Italy) (see, Fig. 3A), dating back to the V millenium B.C.. This culture begun with the Neolithic civilization, and it is characterized by the presence of imprinted decorations on the artifacts, which are hand-made on the external surface or by other instruments (shells, pieces of wood, bones, flint stones, etc.). Such decorations are peculiar of the neolithic culture as studied in the Neolithic archeological sites nearby Syracuse (Sicily). The *"Stentinello"* culture represents an evolution of these decorative techniques, during the diffusion of the Neolithic in the Southern Italy (Purri, 2007; Purri, 2011).

The Medieval section, is dedicated to the *Nicastro Castle* (Fig. 3B) and the findings of the *St. Mary of St. Eufemia Abbey* (Fig.3C) have been found during the archaeological excavations, run by the Archaeological Superintendence of Calabria since 1993. In particular the Medieval Samples are: sample CAST n°6, found in the *Nicastro Castle* and samples ABB n°7, 8 and 9 found in the St. Mary of St. Eufemia Abbey, Lamezia Terme (Cz) (Fig. 4).

Figure 3. (A) Area of *Acconia*, in the *"Piana di Curinga"* (Lamezia Terme, Cz, South Italy); (B) *Nicastro Castle;* (C) *St. Mary of St. Eufemia Abbey*

Figure 4. Map of Calabria region (Italy) and archaeological location on the map.

The mediaeval findings are polychrome glazed pottery and glazed pottery on white background, they have a mixture that mostly consists of purified clay, the color varies from light beige to pink. They are samples of fragments of different kinds of pottery.

The *Nicastro Castle,* built in Norman (1130-1189) probably on the site of a Byzantine fort, was an emblem, together with the *St. Eufemia Abbey* of the political and economic power of this period. The same architectural structure of the castle gives the opportunity to observe the different historical phases that modified its appearance. From the second half of the XVI century begins the slow decline of the castle, the earthquake of 1638 damaged it considerably. Later used as a prison, it was finally abandoned after the earthquake of 1783.

The *St. Mary of St. Eufemia Abbey* is located in the municipality of Lamezia Terme (Catanzaro), in the village of *St. Eufemia Vetere* in place *Terravecchia*. One of the first ecclesiastical foundations established officially by the Normans in Calabria has been the *St. Eufemia Abbey*, refounded on an earlier Byzantine monastery between 1062 and 1065 by *Roberto il Guiscardo* as an Abbey of the Latin rite (Cingari, 1987; Arias, 1988).

In the 1638 a devastating earthquake that struck the whole of Calabria, causing many victims, upsets the urban and rural area of Lamezia, the Abbey was destroyed, never rebuilt and the Knights were limited to build a small church for the few survivors.

INSTRUMENTATION AND ANALYSIS

The diagnostic plan includes a first phase for the cognitive context of the findings and then the Thermoluminescence analysis to date the artifact using the Single Aliquot Regenerative-dose (SAR protocol).

The dating has been obtained via thermoluminescence measurements using a TL reader from Risø, by applying the SAR protocol. For the artificial doses we use a beta source (90Sr) characterized by a dose rate of 0.1 Gy/sec. Generally we used a heating rate of 5 °C/s up to a temperature of 450 ° C and the samples were irradiated with seven or ten regeneration doses.

The essential components of the Risø TL/OSL reader (model TL/OSL-DA-20) are:

- light detection system
- luminescence stimulation system (thermal and optical)
- irradiation source

The standard PMT in the Risø TL/OSL reader is a bialkali EMI 9235QB PMT, which has maximum detection efficiency between 200 and 400 nm, making it suitable for detection of luminescence from both quartz and feldspar. The Risø TL/OSL reader used with the following detection filter: Hoya U-340 (7.5 mm thick, diameter Ø = 45 mm).

It is known in literature that the conventional regeneration procedure produces changes in sensitivity of the quartz grains during repeated TL measurements (Nakagawa, 2003; Przegietka et al, 2005), so that one must take into account these changes in sensitivity in order to get a reliable estimate of the equivalent dose. In this work the modified SAR protocol has been adopted, where the thermal treatment is excluded with respect to the original SAR protocol (Hong, et al., 2006; Barrandon, 2001). The sequence of measurements is summarized in Table 1, the identification labels used throughout the text and figures are also reported for each measure.

The Annual Dose (AD) has been determined by measuring the natural radiation coming from the ceramic itself due to their content in radioactive elements (U, Th, K-40), adding then gamma and cosmic radiation from the surrounding. As reported in Table 2, data on radioactive content of K, Th and U are obtained by Laser Ablation-Inductively Coupled

Table 1: Sequence illustrating an example of the procedure for S.A.R. measurements adopted in this study.

Step	Sequence		label
1	NTL 450°C,5°C/s		NTL
2	Irrβ 1Gy + TL	Test dose	Test NTL
3	Irrβ 2Gy + TL	First Regenerative Dose	First TL 2Gy
4	Irrβ 1Gy + TL	Test dose	Test TL 2Gy
…	…	…	…
x	Irrβ x Gy + TL	"x" Regenerative Dose	Test TL x Gy
…	Irrβ 1Gy + TL	Test dose	Test TL x Gy
…	…	…	…
…	Irrβ 2Gy + TL	Last Regenerative Dose	Last TL 2Gy
…	Irrβ 1Gy + TL	Test dose	Test TL 2Gy

Plasma-Mass Spectrometry (LA-ICP-MS) and converted to infinite matrix dose using the conversion factors by Adamiec and Aitken (Adamiec, & Aitken, 1998) [NB: most recent re-evaluation data (Liritzis et al., 2012) show a few % differences but within the total age error bars]. Contributions from cosmic rays were included using the equations given by Prescott and Hutton (Prescott and Hutton, 1988, 1994). The contribute of cosmic rays is in agreement with that obtained by Bianca et al. (Bianca M., et al., 2011) for the same geographical area. The natural annual dose rate AD has been corrected by the following factors: 1) using, for the moisture rate (W), the water content attenuation factor given by Zimmerman (Zimmerman, 1971) and the size attenuation factors of Mejdahl (i.e. 1.25 for beta and 1.14 for gamma contributions) (Mejdahl., 1979); 2) using an attenuation factor of 0.90 for beta contribution except in the case of Rb for which a factor of 0.75 has been used to account for lower penetration (Adamiec & Aitken, 1998); 3) finally a value of 20% for the escape of radon, in the case of the U-238 series, has been included (Aitken,1985).

The LA-ICP-MS analyses, to determine the natural radiation, were carried out using an ElanDRCe instrument (Perkin Elmer/SCIEX), connected to a New Wave UP213 solid-state Nd-YAG laser probe (213 nm). Samples were ablated by a laser beam in a cell and the ablated material was then flushed in a continuous flow of an argon and helium mixture to the ICP system, where it was atomized and ionized for quantification in the mass spectrometer (Barca D. et al., 2010).

In our work, ablation was performed with spots of 80 μm with a constant laser repetition rate of 10 Hz and fluence of ~20 J/cm^2. All procedures for data acquisition were those routinely used (Scarciglia., et al., 2009; Barca., et al., 2011; Miriello et al., 2012; Barberio et al., 2011). The external calibration of LA-ICP-MS was performed using the NIST612-50 ppm and NIST610–500 ppm glass reference materials (Fryer et al., 1995), internal standardization to correct instrumental instability and drift was achieved with SiO_2 concentrations (Pearce, et al., 1997) from SEM-EDX analyses. Accuracy was evaluated on BCR 2G glass reference material and the resulting element concentrations were compared with reference values from literature (Gao et al., 2002; Barca et al., 2007). The relative difference from reference values was always better than 10%.

SAMPLE PREPARATION

Sample preparation took place in condition of darkness with the aid of a single soft red light (> 600 nm). We have chosen the *Quartz inclusion* technique, since with this method we obtain grains of larger size which can then be submitted to acid solution to eliminate surface carbonates (Fleming, 1979; Leute, 1987).

The first step of preparation involved sampling (using a low speed drill) in the sample bulk, in order to avoid external contamination, such as, sources of light

Table 2. Equivalent Dose and TL dating of the samples as obtained using TL data based on the S.A.R. protocol. The label "n°" indicates the number of measurements for which the sensitivity correction has been successful; "r" indicates the Linear Correlation Coefficient.

Sample		ED (Gy)	n°	r	Th (ppm)	U (ppm)	K (%)	Rb (ppm)	W (%)	D_β (mGy/a)	$D_{\gamma+cosm}$ (mGy/a)	AD (mGy/a)	Age (a)	TL Dating Samples	Place of discovery	Site Historical Dating
Middle Age	ABB n° 7	1,6 ± 0,2	1	0,98	5,8 ± 0,2	2,2 ± 0,1	1,9 ± 0,4	508 ± 9	0,4	1,9 ± 0,3	1,1 ± 0,1	3,0 ± 0,3	533 ± 85	1480 ± 90 A.D.	Sant'Eufemia Abbey	In 1062 founded; 1638 destroyed
	ABB n° 8	1,4 ± 0,1	3	0,99	10,5 ± 0,3	3,4 ± 0,1	1,6 ± 0,3	307 ± 5	0,8	1,9 ± 0,2	1,4 ± 0,1	3,2 ± 0,2	438 ± 42	1570 ± 40 A.D.		
	ABB n° 9	1,2 ± 0,1	7	0,98	15,4 ± 0,5	6,3 ± 0,3	2,2 ± 0,6	378 ± 8	0,5	2,8 ± 0,4	2,0 ± 0,2	4,8 ± 0,4	250 ± 29	1760 ± 30 A.D.		
	CAST n° 6	1,2 ± 0,2	3	0,98	10,9 ± 0,3	2,2 ± 0,1	2,3 ± 0,1	298 ± 5	0,5	2,2 ± 0,1	1,48 ± 0,03	3,7 ± 0,1	324 ± 55	1690 ± 60 A.D.	Nicastro Castle	In XI-XII sec. founded; in 1783 definitively
Neolithic	ROM n° 6	20 ± 1	9	0,99	13,7 ± 0,4	1,7 ± 0,1	1,4 ± 0,1	219 ± 5	3,9	1,6 ± 0,1	1,35 ± 0,03	2,8 ± 0,1	7143 ± 439	5100 ± 400 B.C.	Curinga Valley - Romatisi Locality	«Impressa» Ceram Culture - VI-V millennium B.C.
	SUV n° 5	19 ± 2	1	0,99	13,7 ± 0,4	2,2 ± 0,1	1,7 ± 0,1	243 ± 5	5,1	1,9 ± 0,1	1,47 ± 0,03	3,1 ± 0,1	6129 ± 675	4100 ± 700 B.C.	Curinga Valley -Suveretta Locality	"Cultura di Stentinello" - V millennium B.C.
	TER n° 1	19 ± 3	4	0,98	27,6 ± 1,1	5,8 ± 0,3	1,0 ± 0,1	285 ± 10	9,4	2,1 ± 0,1	2,3 ± 0,1	4,0 ± 0,1	4750 ± 759	2700 ± 800 B.C.	Curinga Valley -Terrevecchia Locality	"Cultura di Stentinello" - IV millennium B.C.
	TER n° 2	16 ± 2	1	0,98	9,6 ± 0,6	3,6 ± 0,2	1,1 ± 0,1	222 ± 10	4,3	1,5 ± 0,1	1,26 ± 0,04	2,6 ± 0,1	6154 ± 805	4100 ± 800 B.C.		
	TER n° 3	14 ± 2	1	0,98	8,9 ± 0,4	2,1 ± 0,1	0,53 ± 0,04	111 ± 4	9,7	0,87 ± 0,03	0,95 ± 0,02	1,65 ± 0,04	8485 ± 1229	6500 ± 1200 B.C.		
	TER n° 4	12 ± 1	3	0,99	11,1 ± 0,6	2,3 ± 0,1	0,9 ± 0,1	369 ± 16	5,1	1,3 ± 0,1	1,20 ± 0,04	2,3 ± 0,1	5217 ± 490	3200 ± 500 B.C.		

mostly, heat or radiation to which the sample surfaces may have been exposed after the discovery.

Grain size 63-125 μm was sieved and treated with 10% HCl to eliminate any present carbonates and bath with 30% H_2O_2 to eliminate organic material. The so obtained different crystalline fractions were separated by using a heavy liquid solution. The quartz grains were then extracted, dried and used for the dating process. These grains were fixed with silicone oil on stainless steel discs of 9.7 mm in diameter and then loaded on the spectrometer.

RESULTS AND DISCUSSION

The luminescence signal in quartz crystals, related to the Equivalent Dose (ED) in the age equation, can be released from traps by both thermal stimulation (Thermo Luminescence, TL) or Optically Stimulated Luminescence (OSL).

Regarding the determination of the Equivalent Dose, the traditional method involves normalization procedures due to various irradiation rates or to difference in sample masses, or to sample sensitivity problems which render such methods more complicated and time consuming. Instead the procedure based on a Single Aliquot Regenerative-dose (SAR) has the advantage, over the traditional methods (MAAD, Multiple Aliquot Additive Dose), of avoiding standardization or sensitivity problems and reduces the effort required in sample preparation.

The SAR protocol has been discussed by Murray and Wintle (2000) who proposed a modified regeneration method with respect to the conventional one. In our work we decided to use the protocol proposed by Hong et al (Hong et al., 2006; Gattuso et al., 2012) and we performed several measurements for each sample using the SAR-TL protocol (Table 1). The determined Equivalent Doses of the Neolithic and of the Medieval samples are reported in Tables 2

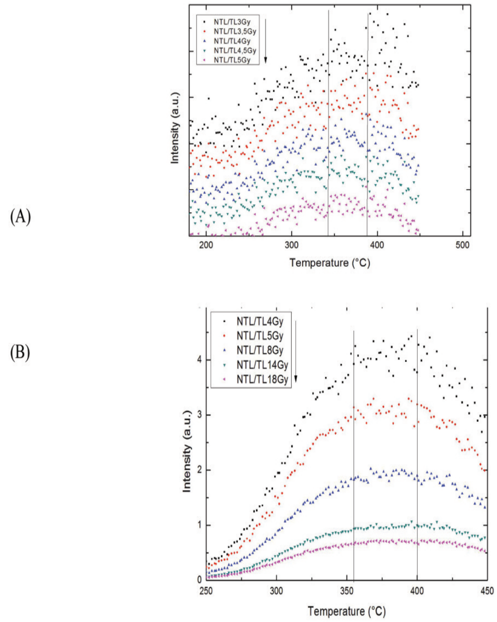

Figure 5. (A) Plateau test of Medieval sample (the graphs have been vertically shifted) and (B) of Neolithic sample.

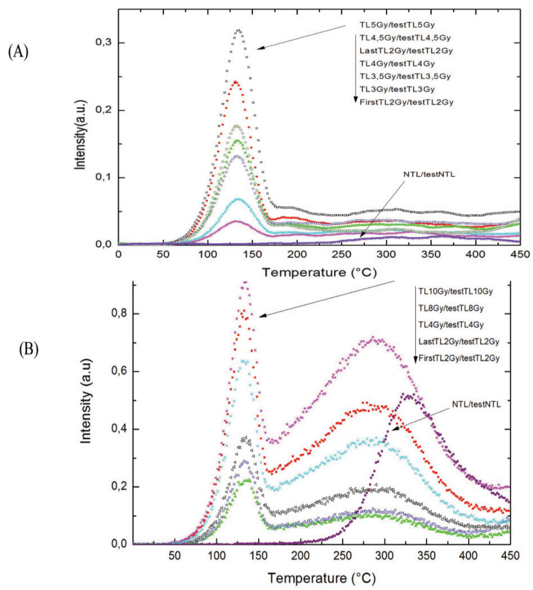

Figure 6. Example of Glow Curves of Medieval sample (A) and Neolithic sample (B) with correction for sensitivity as described in the text. The TL response to the first and the final regenerative doses (labeled "First 2Gy" and "Last 2Gy") are almost the same, indicating that the correction is successful.

(SAR protocol). The value of the equivalent dose is the mean value over all the measurements for which the sensitivity correction has been successful. The correlation indexes "r", which are rather good, indicate that we don't have supralinearity problems.

In Fig.6 we report two examples of TL glow curves (sample ABB n° 9 (A) and sample ROM n°6 (B)) corrected by using the sensitivity correction procedure described by Hong, D.G. et al. (2006). The correction for sensitivity changes was made by monitoring the luminescence response of the sample to a subsequent test dose of 1 Gy, given to the sample after measuring the natural dose and after each regeneration step, as resumed in Tab. 1. The correction procedure consists in normalizing each measurement to the intensity of the relative test dose. Because the first regeneration dose equals the last regeneration dose, the TL intensities for these two regeneration doses should be identical if the correction is successful. As shown in Fig. 6 the TL responses for the first and the last regeneration doses are similar.

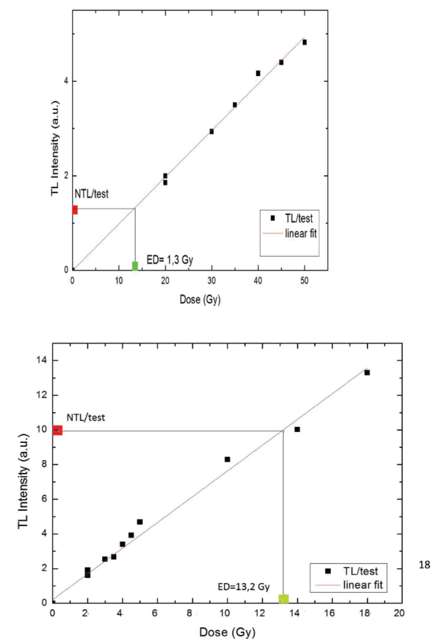

Figure 7. Determination of the Equivalent Dose, ED, of Medieval sample (A) and Neolithic sample (B), based on the Single Aliquot Regenerative-dose (SAR) protocol, using sensitivity corrected TL signals from quartz. The experimental points are fitted by a linear function. Note that at 2Gy the two experimental points, representing the success of sensitivity corrections, coincide.

Fig. 7 shows examples (ABB n°7, medieval sample (A) and TER n°3, neolithic sample (B)) of the sensitivity-corrected TL signal versus radiation dose. The data of the growth curve, corrected for sensitivity, were fitted by a linear function. The equivalent dose was calculated from the growth curve extracting its value at the corrected NTL (Natural Thermoluminescence).

So, the age of the findings from our results we can argue that this ranges around the V millennium B.C., for the Neolithic, and around 1500 A.D., for the Middle Age.

The results obtained are perfectly compatible with the location and the historical background of the site they come from, the site of *Curinga* for samples of Neolithic, and the *Nicastro Castle* and *St. Eufemia Abbey,* for samples of the Middle Age.

CONCLUSIONS

This work represents the experimental efforts for dating artifacts from Lamezia Terme, Calabria.

The results, obtained by TL-SAR method (as mentioned in the introduction we don't use here the OSL-SAR protocol) , provided reliable age estimates for the following samples (the Italian labels refer to different archaeological sites in the same valley): "*Terravecchia*", age range 2700-6500 ± 500-1200 B.C. (see table 2 for details); "*Suveretta*", 4100 ± 700 B.C.; "*Romatisi*", 5100 ± 400 B.C.

Comparing the historical-artistic framework of the samples with the dating results, it is possible to affirm that they are in agreement with the historical period to which the fragments belong, attributed by archaeologists to the V millennium B.C.

A similar conclusion can be drawn for the medieval samples, for which the obtained dating agrees with the respective historical context of *St. Eufemia Abbey and Nicastro Castle.*

The correction obtained by the TL-SAR method allows a more accurate estimate of the ED and avoids problems arising from sample sensitivity, which, by this method are highlighted and thus corrected. The overall conclusion is that the dating results are in agreement with the historical framework described by the archeologists.

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