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U/Th dating of pre-Holocene calcareous tufa deposits in the Umbria-Marche Apennine (Italy)

Abstract: Cilla G., Dramis F., Fubelli G., Maceroni D., Malocco S., Materazzi M., Remigio M., Soligo M., *U/Th dating of pre-Holocene calcareous tufa deposits in the Umbria-Marche Apennine (Italy)*. (IT ISSN 0391-9838, 2025). This note describes the characteristics of two calcareous tufa (freshwater travertine) deposits found in the Umbria-Marche Apennine. The first has grown in the Laverinello Valley, a tributary of the upper Potenza River, along the road from the village of Poggio Sorifa; the second deposit originated in Val di Sasso (Sasso Valley), a tributary of the upper Esino River, on the edge of a waterfall, currently inactive and disconnected from the present drainage network. The U/Th dating of samples taken from the investigated tufa deposits (55 ± 6 ka BP - 58 ± 6 ka BP, 65 ± 8 ka BP - 67 ± 5 ka BP, respectively) places them in the Upper Pleistocene, corresponding to sharp warming peaks within cooling periods. These dates seem to support the genetic model of freshwater travertine deposition proposed by Dramis *et al.* (1999), based on the control exerted by the formation in the aquifer of a reversed thermal gradient in correspondence with climate warming.

Key words: Calcareous tufa, Ground thermal gradient, Climate change, Pleistocene, Umbria-Marche Apennine, Italy.

Riassunto: Cilla G., Dramis F., Fubelli G., Maceroni D., Malocco S., Materazzi M., Remigio M., Soligo M., *Datazione U/Th di depositi pre-olocenici di tufa calcarea nell'Appennino Umbro-Marchigiano*. (IT ISSN 0391-9838, 2025). In questa nota sono descritte le caratteristiche di due depositi di “tufa calcarea” (travertino d’acqua dolce) rinvenuti nell’Appennino umbro-marchigiano. Il primo si è sviluppato nella Valle di Laverinello, affluente del Fiume Potenza, lungo la strada che parte dal paese di Poggio Sorifa; il secondo deposito ha avuto origine nella Val di Sasso, affluente del Fiume Esino, lungo una cascata, oggi inattiva e disconnessa dall’attuale rete idrografica. La datazione U/Th di campioni prelevati dai depositi di travertino investigati (rispettivamente 55 ± 6 ka BP - 58 ± 6 ka BP, 65 ± 8 ka BP - 67 ± 5 ka BP) ne colloca la formazione nel Pleistocene superiore, in corrispondenza di picchi di riscaldamento improvvisi nel corso di periodi freddi. Queste attribuzioni temporali sembrano supportare il modello genetico del travertino di acqua dolce proposto da Dramis *et al.* (1999) e basato sul controllo esercitato dalla formazione di un gradiente termico inverso nella falda acquifera indotto dal riscaldamento climatico.

Termini chiave: Travertino, Gradiente geotermico, Cambiamento climatico, Pleistocene, Appennino Umbro-Marchigiano, Italia.

INTRODUCTION

Calcareous tufa, also known as meteogene or freshwater travertine (Pedley, 1990; Ford and Pedley, 1996; Dramis *et al.*, 1999; Pentecost, 2005; Gandin and Capezzuoli, 2008), is typically found in stream valleys that cross limestone

landscapes. It generally consists of massive bodies of phytothermal travertine, formed over stream knickpoints and waterfalls through CaCO_3 precipitation due to CO_2 degassing from water spraying and the photosynthesis of algae and mosses. These bodies typically block the watercourses, resulting in dams behind which swampy-lacustrine basins form, featuring phytoclastic travertine layers on their bottom (Pentecost, 2005). The growth of tufa dams occurs where the deposition rate of calcium carbonate is high enough to balance the speed of streamflow erosion (Fubelli *et al.*, 2013).

Calcareous tufa is distinct from thermogene travertine (or, simply, *travertine*) precipitated by thermal waters rising from deep-reaching aquifers along fractures and faults (Ford and Pedley, 1996; Pentecost, 2005; Gandin and Capezzuoli, 2008).

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Paper published on the 25th anniversary of AIGeo, the Italian Association of Physical Geography and Geomorphology. GFDQ vol. 48, Guest Editors: Pappalardo M., Rotigliano E., Ferrando A.

Scientists generally agree in relating the deposition/erosion of calcareous tufa to climate controls (Gullentops and Mullenders, 1972; Vaudour, 1986; Pedley, 1990; Baker *et al.*, 1993; Andrews *et al.*, 1994; Pentecost, 2005). Warm climates are believed to favor tufa deposition because of: (1) higher levels of biogenic CO₂ in soil layers, resulting in higher rates of limestone dissolution (Thraillkill, 1968; Atkinson, 1977); (2) higher air temperature at the springs favoring the outgassing of CO₂ dissolved in water; and (3) absorption of CO₂ by aquatic plants. Conversely, cold climates are considered to be less favorable due to: (1) reduction of biological activity in soils; (2) lower air temperatures at the springs; and (3) lesser development of aquatic plants.

Humid-arid climate variations are also believed to control the deposition and erosion of tufa deposits. Wet climates increase the abundance of surface water, favoring biogenic processes in soils and the development of aquatic vegetation, which is disadvantaged in dry climates.

A further model to explain the increase-decrease in tufa deposition rates refers to anomalies in thermal gradients between the ground surface and the limestone aquifer induced by climate change (Dramis *et al.*, 1999). Because of the low thermal conductivity of rocks (Vasseur *et al.*, 1983) and the related slow penetration of thermal changes into the ground, reversed thermal gradients with differences of temperature up to several degrees may be induced between the surface and the underlying bedrock over timescales ranging from years to thousands of years depending on the thermal change duration and magnitude, and the aquifer thickness (Williams and Smith, 1989).

With climatic changes to warmer conditions, water percolating through progressively colder layers in the vadose belt and the phreatic zone undergoes a progressive enrichment in dissolved CaCO₃. At emergence, the spring water becomes oversaturated with CaCO₃, inducing tufa deposition due to higher surface temperatures. Opposite effects, such as the deposition of dissolved carbonate in warmer bedrock fractures and the emergence of “aggressive” spring waters undersaturated with CaCO₃, should be expected as the climate shifts to colder conditions.

The growth of tufa dams with changes to warmer temperatures was largely observed during the Holocene (Dramis *et al.*, 2003; Fubelli *et al.*, 2013; Dramis *et al.*, 2014; Dramis and Fubelli, 2015) but also in the previous cold stages (MIS 2-3-4) (Fubelli *et al.*, 2021; Fubelli and Dramis, 2023) providing support to the above model.

This note presents the U/Th dating results of two pre-Holocene tufa deposits found in the upper basins of the Potenza and Esino rivers (Umbria-Marche Apennine).

GEOLOGY OF THE TUFA DEPOSITS OCCURRENCE AREAS

The upper basins of the Potenza and Esino rivers are located on the eastern side of an NNW-SSE trending anticlinorium ridge (Umbria-Marche Ridge) involving massive and stratified limestone, marly-limestone, and marls, Jurassic to Oligocene in age, and representing the outcrops of an east-verging thrust structure emplaced during the main tectogenetic phase (Late Miocene - Early Pliocene) of the Umbria-Marche Apennine (Bally *et al.*, 1986; Calamita *et al.*, 1991).

The geo-lithological units outcropping in the areas where the investigated tufa deposits occur are, from youngest to oldest, the following (Centamore *et al.*, 1979; Cresta *et al.*, 1989):

- *Scaglia Variegata e Cinerea* (Lutetian *p.p.* - Aquitanian *p.p.*), 20-40 m thick marly limestone and calcareous marl in thin layers, varying in color from pink to gray-greenish (*Scaglia Variegata*), followed by around 100 m thick marly limestone and gray marl (*Scaglia Cinerea*);
- *Scaglia Bianca e Rosata* (Upper Cenomanian *p.p.* - Lutetian *p.p.*), around 50 m thick stratified limestone with reddish flint at the base (*Scaglia Bianca*), and pink to reddish limestone stratified with flint and marly intercalations for a total thickness of 200-300 m;
- *Marne a Fucoidi* (Lower Aptian *p.p.* - Cenomanian *p.p.*), comprising a marly member at the base followed by a varicolored clayey one for a total thickness of 100 m;
- *Maiolica* (Titonian *p.p.* - Lower Aptian *p.p.*), a sequence of stratified whitish limestone with white-gray flint intercalations of variable thickness, from less than 100 m to over 500 m;
- *Calcari Diasprini Umbro-Marchigiani* (Bajocian - Tithonian *p.p.*), limestones with flint strips and nodules, flint limestones and flint in thin layers (4-10 cm) that become irregular with swellings or abrupt lens-shaped terminations where siliceous sediments prevail, especially in the middle part of the unit; the overall colour is predominantly grey-greenish, often passing to brightly polychrome zones, especially in correspondence with the most siliceous lithotypes. The thickness varies between 60 and 150 m (Centamore *et al.*, 1979).

The stratigraphic succession continues at depth for over 250 m of thickness with the calcareous marly and marly units of the *Bosso Formation* (Toarcian *p.p.*-Bathonian) and the *Marne del Sentino* (Pliensbachian *p.p.*-Toarcian *p.p.*), heteropic and discontinuous, the *Corniola limestones* (Sinemurian *p.p.*-Pliensbachian *p.p.*), and the *Calcare Massiccio* (Hettangian – Sinemurian *p.p.*), stratified in pluri-metric banks and several hundred meters thick. At the base of this succession is the evaporitic formation of *Anidriti di Burano* (Triassic), recognized from a deep-drilled well in the Burano Valley (Martinis and Pieri, 1964).

In both areas, near-surface units (Middle-Upper Pleistocene to Holocene) consist of gravelly alluvial deposits on the riverbeds and terraced at different heights, as well as loose or stratified slope deposits (Coltorti and Dramis, 1987; Cilla *et al.*, 1994). In addition to folds and thrusts, the structural setting of the investigated areas includes faults of various types, mainly transverse to the thrust structures, as well as more recent west-dipping normal faults, parallel to them (Servizio Geologico d'Italia, 1979; Regione Marche, 2004).

The stratigraphic succession described, made of permeable calcareous units (aquifers) alternating with marly levels (aquicludes) and heavily affected by faults, has favored: (1) the formation of large, deep-reaching aquifers comprising more than one of the permeable units; (2) the circulation of ground waters that emerge from various types of springs located at different altitudes on the slopes and in the valley floors (Dramis, 1969).

Some of these waters have been responsible for the deposition of calcareous tufa in the Holocene (Calderoni *et al.*, 1996).

In other parts of the Umbria-Marche Apennine, calcareous tufa deposits dated to the Holocene and Pleistocene have also been found (Carrara *et al.*, 1995; Calderoni *et al.*, 1996; Carrara, 1998; Farabollini *et al.*, 2004; Carrara *et al.*, 2006; Dramis *et al.*, 2008; Fubelli *et al.*, 2013).

THE INVESTIGATED TUFA DEPOSITS

The calcareous tufa outcrops and their upstream areas (fig. 1) underwent a detailed field survey, focusing on the genetic context and stratigraphy of the deposits, as well as the presence of springs that may have contributed to their formation.

The first deposit has grown over the *Scaglia Bianca e Rossa* limestone (fig. 2) at about 600 m a.s.l. in the Laverinello Valley (upper Potenza River basin) along the road from the village of Poggio Sorifa (location in fig. 1). The outcropping section, partially covered by vegetation, extends approximately 180 m in length and 30 m in height, above and below the road. From top to bottom, the following levels are visible (fig. 3):

1. between 0 and -1.50 m, slope debris is present, consisting of a few cm clasts;
2. from -1.50 to -4.50 m, there is compact and crystalline travertine of light color (fig. 4);
3. between -4.50 and -8.80 m, the first phytothermal structures appear associated with trunks and branches around which the travertine has grown;
4. from -8.80 to -11.90 m, there are layers of less compact travertine material with a thickness of a few centimeters;

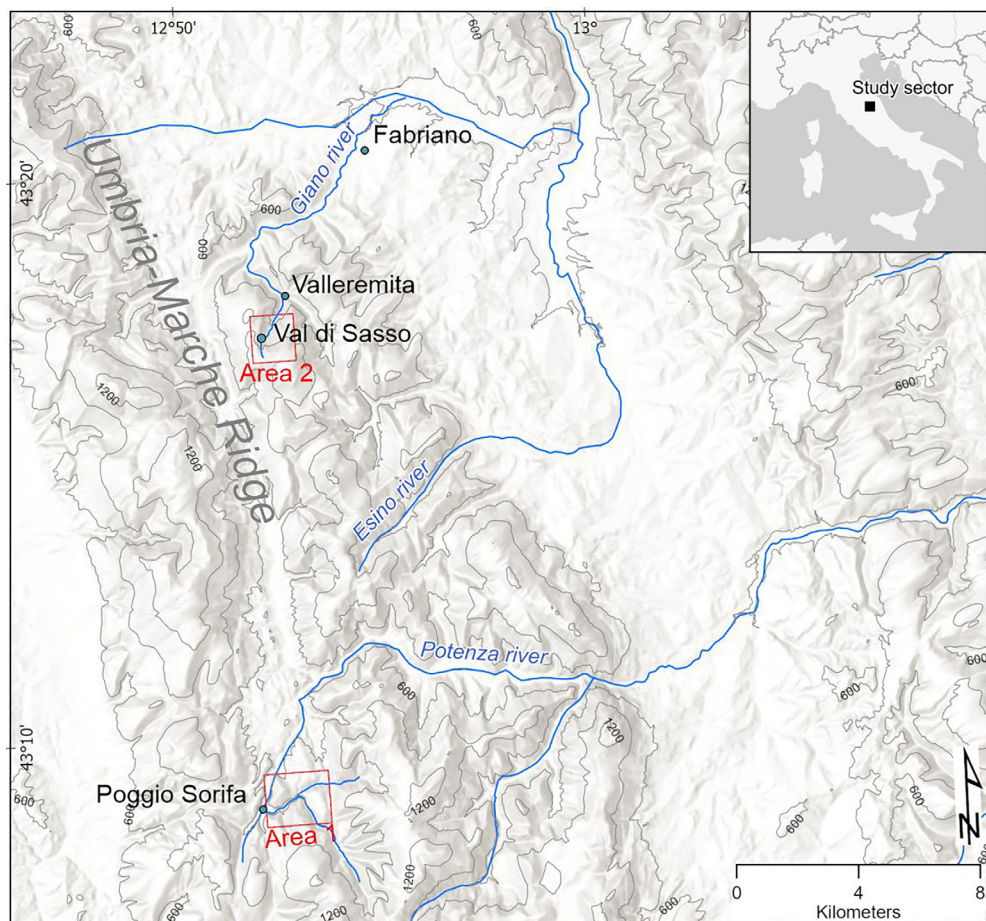


Figure 1 - Location map of the investigated tufa deposits.

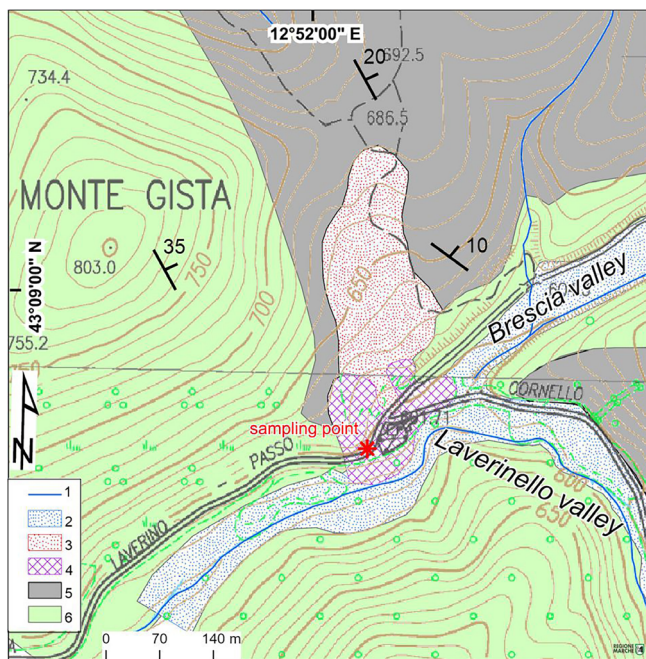


Figure 2 - Geological map of the area surrounding the Laverinello Valley tufa deposit (after Regione Marche, 2004): 1. Hydrographic network; 2. Fluvial deposits; 3. Slope deposits; 4. Tufa deposits; 5. *Scaglia Variegata e Cinerea*; 6. *Scaglia Bianca e Rosata*.

5. between -11.90 and -17.30 m, the travertine assumes a massive aspect with a gradually lighter color towards the section base;
6. from -17.30 to -20 m, there are phytoclastic travertine sands with frequent decimetric banks of phytoclastic travertines and stromatolitic levels (fig. 5).

The sedimentological characteristics of the section indicate a deposition environment consisting of dam facies (massive phytothermal travertine from level 5 to 2), which advances growing over the back dam basin facies (stratified sandy travertine level 6) according to the evolutionary model of fig. 6.

The Brescia Valley, directly upstream of the tufa deposit, comprises high-discharge springs fed by the *Maiolica* and *Scaglia Bianca e Rosata* limestone aquifers in contact with the *Marne a Fucoidi* and *Scaglia Variegata e Cinerea* marly aquicludes, respectively (Dramis, 1969; Regione Marche, 2004). The waters coming from these springs most likely contributed to the formation of the tufa deposit. Still, in the past, a local spring might have also been present at the contact between the *Scaglia Bianca e Rosata* aquifer and the *Scaglia Variegata e Cinerea* aquiclude.

The Val di Sasso tufa deposit (fig. 7) is located at approximately 650 m a.s.l. in a small valley of the upper Giano River basin, upstream of the village of Valleremita. On the deposit stands the hermitage of Santa Maria di Val Sasso, an ancient building of considerable historical and cultural value built on the site of a Benedictine monastery where St. Francis of Assisi lived in 1210

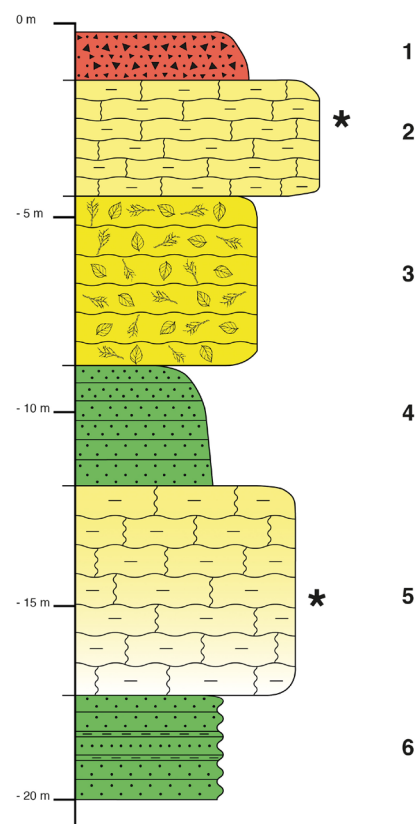


Figure 3 - Stratigraphic log of Laverinello Valley tufa deposits, the stars indicate the sampling points, the numbers correspond to the ones indicated in the text.

and 1215. The tufa deposit is located at the edge of a *Maiolica* limestone scarp. It has formed on a waterfall presently inactive and disconnected from the drainage network (fig. 8).

Upslope of the tufa deposit, water currently emerges from a spring located in the *Maiolica* limestone aquifer, here in contact with the underlying less permeable *Calcari Diasprini Umbro-Marchigiani* at the core of an NNW-NNE trending anticline fold (Servizio Geologico d'Italia, 1979; Regione Marche, 2004). However, the water flow responsible for freshwater travertine deposition might also have originated from other springs located upstream in Val di Sasso and probably related to the contact between the *Scaglia Bianca e Rosata* aquifer and the underlying *Marne a Fucoidi* aquiclude.

The deposit extends for about 30 m with a thickness ranging from 10 to 40 m. It is visibly hanging over the valley bottom where alluvial and tufa deposits dated to the Holocene are present (Calderoni *et al.*, 1996).

The travertine appears, at places, porous and fractured or crystalline and compact with phytothermal structures and imprints of trunks and branches (fig. 9). There are also abundant concretions of diatoms and bryophytic mosses in the form of casts or filaments.



Figure 4 - Massive phytothermal travertine in the Laverinello Valley deposit.



Figure 5 - Massive travertine overlying travertine sands at the base of the Laverinello Valley deposit.

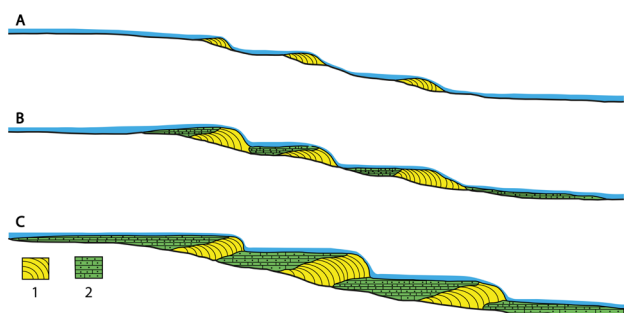


Figure 6 - Evolutionary model of tufa dams. 1. Phytothermal travertine; 2. Phytoclastic travertine. A. The phytoclastic travertine begins to form on the profile breaks of a watercourse. B. With the growth of the dams, small lakes with phytoclastic travertine deposits form on their backs. C. The growing dams prograde on the lake deposits.

DATING

The calcareous tufas under investigation were dated using the uranium-series disequilibrium method, as described by Ivanovich and Harmon (1992) and Goldstein and Stirling (2003). This method relies on measuring authigenic ^{230}Th , which develops from the radioactive decay of ^{234}U . The analysis was carried out using alpha spectrometry at the Radiometric Geochronology Laboratory within the Department of Sciences at Roma Tre University. The equipment used included silicon surface barrier detectors, electronic signal amplifiers, a discriminator, a pulse analyzer, and a computer for data management.

From the Laverinello Valley tufa outcrop, two samples, labeled LV_1 and LV_2 , were collected from areas where the travertine appeared compact, unaltered, and non-recrystallized. The LV_1 sample was taken from the upper section of the outcrop (level 2), where a compact,

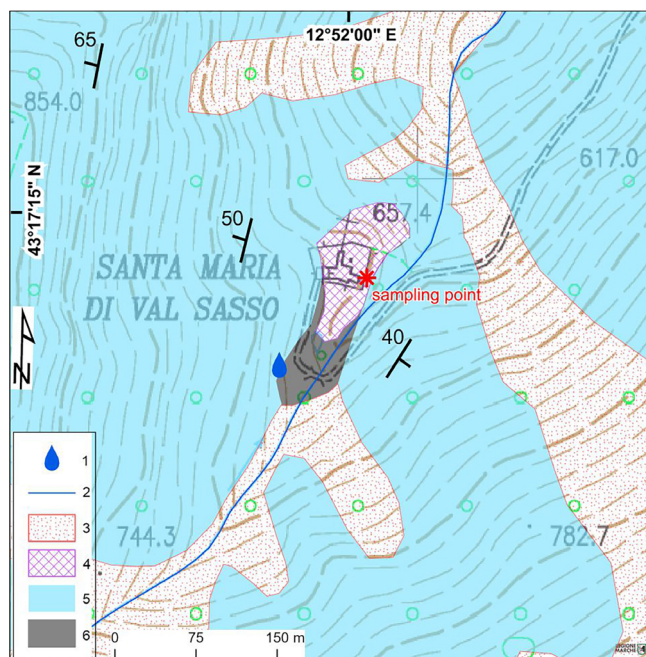


Figure 7 - Geological map of the area surrounding the Val di Sasso tufa deposit (Upper Esino River basin) (after Regione Marche, 2004): 1. Spring; 2. Hydrographic network; 3. Slope deposits; 4. Tufa; 5. *Maiolica*; 6. *Calcari Diasprini Umbro-Marchigiani*.

light-colored area of travertine remained undisturbed by atmospheric agents. The LV_2 sample was extracted from a lower section (level 5), where an unaltered travertine vein was visible.

Two samples, labeled VS_1 and VS_2 , were also taken from the Val di Sasso tufa deposit, representing the two main areas of the waterfall: the front (VS_1) and the back-side (VS_2). These samples were chosen from points where the travertine was as compact as possible and without any signs of vegetation or recrystallization.



Figure 8 - The Val di Sasso tufa deposit overlying the *Maiolica* limestone.



Figure 9 - Imprints of trunks and branches in a tufa outcrop.

Following the laboratory routine, the samples were cut with a diamond saw to remove the altered parts and then crushed and ultrasonically washed in deionized water. Fragments were also examined with a stereoscopic microscope to discard any recrystallized portions and chemically processed to separate the uranium and thorium isotopic complexes. The samples (ca. 50 g each) were dissolved in nitric acid and filtered to separate the leachates from the insoluble residue. Once the sample was filtered, an artificial tracer (spike) with a known isotope ratio of $^{228}\text{Th}/^{232}\text{U}$ was added to the solution, which was then treated with hydrogen peroxide and heated to 100 °C to destroy organic matter. Isotopic complexes of uranium and thorium were extracted, according to the procedure described in Edwards *et al.* (1986), and then alpha-counted using high-resolution ion-implanted Ortec silicon-surface barrier detectors. The age of the sample was calculated by means of Isoplot/Ex (version 3.0), a plotting and regression program designed by Ludwig (2003) for radiogenic-isotope data (Tab. 1).

The statistical error associated with the dating procedure is $\pm 10\%$. As a result, measurements obtained from alpha spectrometry are not as reliable as those from mass spectrometry, whose error may reach $\pm 2-3\%$ (Goldstein and Stirling, 2003). Nevertheless, despite the lower precision of alpha spectrometry, the results can still be considered reasonably probable.

DISCUSSION AND CONCLUSION

The dates of the two pairs of samples taken from the Laverinello Valley and Val di Sasso tufa deposits, respectively, are stratigraphically consistent, considering that those extracted from the upper levels of the deposits follow those obtained from their lower parts. They place the formation of the investigated calcareous tufas in the Upper Pleistocene, within a time interval already recognized for other tufa deposits in the Apennines (Carrara *et al.*, 1995; Carrara, 1998; Carrara *et al.*, 2006).

More specifically, despite the limitations of the dating procedure performed and the possible chronological overlaps, the most probable dates obtained from the examined samples, when compared with the Pleistocene temperature curve based on the analysis of foraminifera shells from the ocean floor (Rasmussen *et al.*, 2016; Li and Born, 2019), seem to place the Laverinello Valley and the Val di Sasso tufas in the MIS 3 (60-27 ka BP) and MIS 4 (71-57 ka BP), respectively, an overall period of very cold temperatures at times interrupted by minor warmer peaks characterized by abrupt rises of sea surface temperature by more than 3 °C after long cold intervals (Ganopolski and Rahmstorf, 2001).

Following the hypothesis (to be confirmed with more precise dating) that the chronological probability peaks correspond to reality, the Laverinello Valley tufa dates would

Table 1 - Results of the laboratory analysis and resulting sample ages calculated by means of the Isoplot/Ex (version 3.0).

Sample	U (ppm)	$^{230}\text{Th}/^{232}\text{Th}$	$^{230}\text{Th}/^{234}\text{U}$	$^{230}\text{Th}/^{234}\text{U}$ corrected	$^{234}\text{U}/^{238}\text{U}$	Age (ka)
LV ₁	0.057 ± 0.004	175.520 ± 98.079	0.420 ± 0.035		1.296 ± 0.093	58 ± 6
LV ₂	0.047 ± 0.003	55.014 ± 4.050	0.401 ± 0.032		1.289 ± 0.104	55 ± 6
VS ₁	0.040 ± 0.002	13.731 ± 1.472	0.474 ± 0.022*	0.458 ± 0.043	1.429 ± 0.043	65 ± 8
VS ₂	0.085 ± 0.003	48.806 ± 4.723	0.473 ± 0.028		1.466 ± 0.051	67 ± 5

* Given that the value of $^{230}\text{Th}/^{232}\text{Th}$ activity ratio of the VS₁ sample, equal to 13.731 ± 1.472 , is considered too low because it is less than 20, indicating slight contamination of detrital thorium, it was necessary to correct $^{230}\text{Th}/^{234}\text{U}$ activity ratio by assuming the $^{230}\text{Th} / ^{232}\text{Th}$ activity ratio of the detrital thorium equal to 0.85 ± 0.36 (Wedepohl, 1995).

be placed in MIS 3 immediately before warm peaks: 54.5 ka BP (corresponding to the Dansgaard-Oeschger event 15) and 58 ka BP (Dansgaard *et al.*, 1993; Fubelli *et al.*, 2021), respectively, and the Val di Sasso tufa dates would be placed just before the Warm Peak of 64.5 ka BP, characterized by an abrupt rise in sea surface temperature by more than 4 °C after a long cold interval (Rasmussen *et al.*, 2016).

In conclusion, the above data seem to confirm, as has happened in other cases (Dramis *et al.*, 2003; Fubelli *et al.*, 2013; Dramis *et al.*, 2014; Dramis and Fubelli, 2015; Fubelli *et al.*, 2021; Fubelli and Dramis, 2023), the close relationship between climatic conditions and the deposition of freshwater travertine and, in particular, the influence of reversed thermal gradients in the limestone aquifer in response to climate shifting to warming conditions.

AUTHOR CONTRIBUTION

Giuseppe Cilla, field survey, text; Francesco Dramis, general framework, organization, text; Giandomenico Fubelli, field survey, organization, text; Deborah Maceroni, field survey, laboratory; Stefano Malocco, figures preparation, text; Marco Materazzi, figures, text, field survey; Massimiliano Remigio, field survey, laboratory; Michele Soligo, laboratory.

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(Ms. received 8 May 2025, accepted 20 October 2025)