Radiocarbon, Vol 61, Nr 5, 2019, p 1317–1325 DOI:10.1017/RDC.2019.37
Selected Papers from the 23rd International Radiocarbon Conference, Trondheim, Norway, 17–22 June, 2018

IDENTIFYING THE 993–994 CE MIYAKE EVENT IN THE OLDEST DATED LIVING TREE IN EUROPE

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ABSTRACT. Combined dendrochronology and accelerator mass spectrometry radiocarbon (AMS ^{14}C) dating analyses were used in order to date an old living tree named *Italus*, growing in the Pollino massif in Southern Italy. Wiggle match AMS ^{14}C dating analysis was performed on a 320-yr-long floating chronology obtained by cross-dating four wood cores extracted from the exposed roots of the tree. Following this approach, an age for the tree of ≈1230 yr was estimated. This age makes *Italus* the oldest living tree in Europe. High-resolution ^{14}C dating analyses performed on single rings extracted from the tree stem allowed us to identify the 993–994 CE large excursion in atmospheric ^{14}C concentration (Miyake event) revealing for the first time its presence in the Mediterranean basin.

KEYWORDS: 993–994 CE, old trees, Miyake events, tree longevity, wiggle matching dating.

INTRODUCTION

During a large-scale field survey carried out in the Pollino National park in Southern Italy, different Heldreich's pines (*Pinus heldreichii* H. Christ) were sampled in order to assess their ages and study their growth history trajectory. Indeed, previous studies carried out in the same area allowed the identification of trees with ages approaching one thousand years (Serre-Bachet 1985; Biondi 1992). In this paper we present the results of the analyses performed on one of these ancient trees, which was identified as the potentially oldest specimen. The tree was named *Italus* after the name of the legendary king of *Enotri*, the population ruling this region during the Bronze Age. The determination of the tree age was not possible in this case by simple ring counting and cross-dating because of the degradation of the innermost part of the trunk (hollow stem). It required the development of a novel approach based on wiggle match (WM) accelerator mass spectrometry radiocarbon (AMS ^{14}C) dating performed on tree-ring sequences extracted from the exposed roots. Through this approach, the relative dating information established between the different samples is used as *a priori* known information to constrain, in a Bayesian framework, the obtained ^{14}C ages, resulting in a significant refinement of the achievable chronological resolution. The second stage of the study exploits the data from *Italus* as a proxy record of the radiocarbon concentration in the past terrestrial atmosphere.

In this paper we concentrated on this second last aspect by measuring the ^{14}C concentration in single rings extracted from the tree trunk in the period 984–1003 CE. The aim of the study was the identification of the 993–994 CE rapid increase in the atmospheric radiocarbon concentration as linked with one of the so-called Miyake events. These events, so far identified in 774–775 CE (Miyake et al. 2012), 993–994 CE (Miyake et al. 2013, 2014, 2017), and 660 BCE (Park et al. 2017), are generally explained as due to very large SPEs (solar proton events) or a series of SPEs (Mekhaldi et al. 2015). The study of these events

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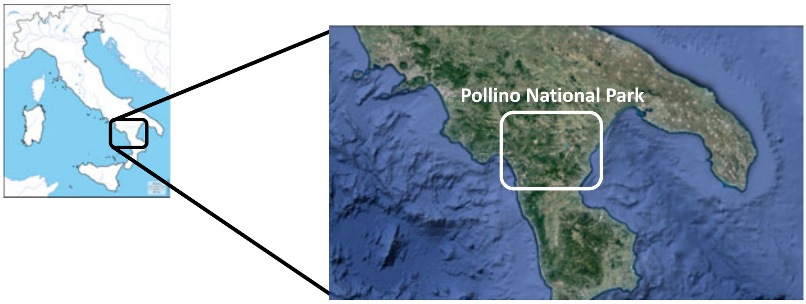

Figure 1 Location of the studied tree in the Pollino National Park, Southern Italy.

has attracted great interest in the scientific community in the last years and research efforts are aimed at clearly identifying the causal mechanism or the mechanisms, their exact timing (also with a sub-annual resolution) and event periodicity and magnitude as summarized by Dee and Pope (2016).

MATERIALS AND METHODS

Italus is an Heldreich's pine (*Pinus heldreichii* H. Christ) with a dead spike top and strip-bark trunk, typical attributes found in old conifers. The diameter at chest height is ca. 1.6 m and the tree is located at an altitude of about 1850 m along the steep rocky slopes of the Pollino Massif in the Pollino National park in Southern Italy (Figure 1).

The present study was performed in different steps and was intended to illustrate new aspects of the methods for absolutely dating the tree and then identifying the 993–994 Miyake event in the xylem ring sequence.

Determining the Age of Italus

The most straightforward method for the determination of the age of a living tree is, of course, ring counting on extracted cores. The tree-ring series should be dendrochronologically cross-dated to obtain an accurate age. This was not directly possible in the case of *Italus* because the innermost part of the stem was highly degraded and the corresponding tree-rings missing. Though the dating strategy has been detailed in previous papers (Piovesan et al. 2018a, 2018b), we report here, for reason of completeness, the employed strategy which was structured as follows:

1. Extraction of 5-mm-diameter cores by an increment borer at different positions around the stem, determination and cross-dating of the tree-ring width series, cross-dating the different series with the available master chronologies for the same species in the same area; determination of the age of the wood cores.
2. Extraction of four 5-mm-diameter cores from exposed roots (Figure 2) and as close as possible to tree germination pith, development of the ring-width series, cross-dating of the different series and comparison with available reference chronologies. Table 1 shows the number of rings of each of the four tree-ring series and the relative cross-dating. From each sequence two samples were selected to be submitted to AMS ^{14}C dating.

Table 1 Summary of the samples extracted from the four exposed roots, laboratory code, corresponding relative dating and conventional radiocarbon ages.

Root	Lab code	Relative dating (yr)	^{14}C age (BP)
Root 4	LTL17796A	256	1222 ± 45
	LTL17797A	209	1234 ± 45
Root 3	LTL17235A	165	1205 ± 45
	LTL17236A	149	1134 ± 45
Root 2	LTL17713A	98	1059 ± 45
	LTL17114A	48	1071 ± 45
Root 1	LTL17111A	52	1006 ± 45
	LTL17112A	0	943 ± 45

Figure 2 The *Italus* tree with the exposed roots shown in the white circle. Red dots indicate the sampling points where roots' cores were extracted. (Please see electronic version for color figures.)

Thanks to the floating root tree-ring chronology it was possible to "wiggle match" the ^{14}C data to the calibration curve by using the dendrochronology information as statistical constraints for the construction of a Bayesian model (wiggle match dating).

Selecting the Sequence for High-Resolution Analyses

Twelve single-year samples were identified, selected, and cut from one of the tree-ring sequences extracted from the stem and with ages corresponding to the period between 984–1003 CE thus bracketing the 993–994 CE Miyake event. The samples had masses between 11 and 16 mg and were submitted for AMS ^{14}C dating at CEDAD (Centre for Applied Physics, Dating and Diagnostics), Department of Mathematics and Physics "Ennio de Giorgi"-University of Salento.

The samples, as well as the previous ones extracted from the roots, were processed by following the base-acid-base-acid-bleaching (BABAB) procedure as described in Nemec et al. (2010). The BABAB procedure is a modified version of the widely used alkali-base-alkali (ABA) protocol with two important modifications. The first one consists of the first alkali step at the beginning of the protocol (4% NaOH at 75°C for 8 hr). The second modification is the addition of a bleaching step after the ABA and consisting in the addition of 5 mL of 10% NaClO2, 5 mL of deionized water, and 4% HCl to the wood suspension followed by 15 min of ultrasonication. The yield of this chemical process, established as the ratio between the extracted purified material and the original sample mass, ranged from 20 to 30%. The amount of purified material available for the following steps ranged between 2 and 4 mg. The purified material was then sealed under vacuum in a quartz tube together with CuO and silver wool and then combusted to CO_2 at 900°C. The carbon dioxide was then cryogenically purified and transferred to the graphitization lines where it was reduced at 600°C to graphite by using H_2 as reductant on iron powder as the catalyst (D'Elia et al. 2004). Extracted graphite was then pressed in the aluminium cathodes of the ion source of the AMS ^{14}C beam line in operation at CEDAD where $^{14}C/^{12}C$ ratios were measured (Calcagnile et al. 2004, 2005). Isotopic ratios were then corrected for mass fractionation by using the $\partial^{13}C$ term measured on line with the accelerator and machine and processing blanks. ^{14}C ages and $\Delta^{14}C$ values were then calculated according to Stuiver and Polach (1977).

RESULTS AND DISCUSSION

The cross-dating of the different stem tree-ring series also with available reference or master chronology for the same species in the same area, allowed us to date the last preserved ring of the core to 955 CE. Considering the presence of the hole in the middle of the trunk, with a radius of 23.8 cm, this has to be considered as a minimum age of the tree.

As detailed elsewhere (Piovesan et al. 2018a), a first estimation of the age of the tree was then obtained by extrapolating the number of missing rings (assuming a circular shape) from the radius of the central hole and the measured average value of the last 20 or 50 rings: 1.05 and 1.16 mm, respectively. By adding the estimated number of missing rings (205–227) to the cross-dated age of the oldest dated ring (955 CE), the final age of the tree could be estimated to be between 727–749 CE. We underline that this is a rough estimation, which is likely to produce an overestimated number of missing rings considering typical juvenile ring patterns for conifers, but it does provide a first approximation of the tree lifespan.

A much more robust estimation of the longevity of *Italus* was obtained by analyzing the results of the ^{14}C analyses performed on the roots samples. The analysis of the ring-width patterns obtained from the cores from the roots showed little statistical correlation with the stem growth patterns and prevented the direct cross-dating of the root sequences. However, the four sequences obtained from the four root cores showed good correlation between

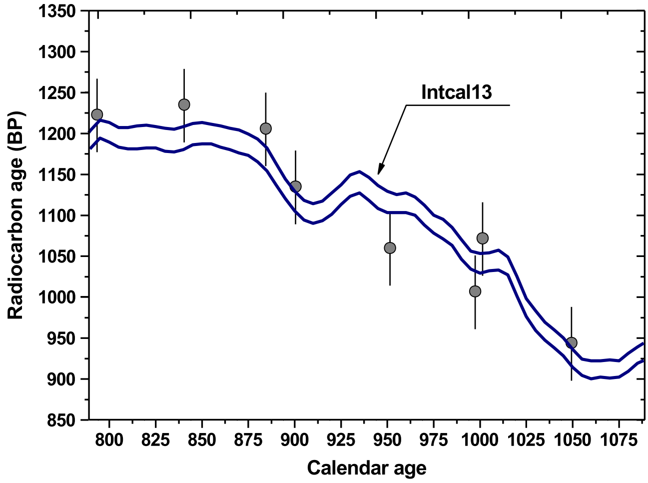

Figure 3 Visual match between the ^{14}C ages obtained on the tree-ring samples extracted from the stem and the IntCal13 calibration curve (errors bars refer to 1σ confidence level).

themselves and allowed the production of a 320-yr-long floating chronology (Wrońska-Wałach et al. 2016; Piovesan et al. 2018b). A detailed analysis of the roots cross-dating, including the statistical synchronization between the floating ring-width series from the external roots, is given in the supplemental material of Piovesan et al. (2018a). In this way, each of the eight samples taken from the four roots could be relatively dated and the age gap between each sample established (Table 1). The relative dating information and the measured ^{14}C ages where then used to match the obtained data with the IntCal13 atmospheric dataset (Reimer et al. 2013; Figure 3). The refined analysis was performed by building a Bayesian model in OxCal v.4.3 using the *D_Sequence* function (Galimberti et al. 2004; Bronk Ramsey 2009), which allowed dating of the oldest ^{14}C dated rings (LTL17796A) to 781–804 CE with a probability of 68.2% and 770–816 with a probability of 95.4% (Figure 4). It is worth noting that also this age has to be considered as a minimum age of the tree as it seems to be confirmed by comparing the ^{14}C-determined age with the age extrapolated from the stem (727–749 CE), especially when, as mentioned before, it is likely that this is an overestimated age of the tree.

Our results show that *Italus* is older than *Adonis*, a tree of the same species growing in Northern Greece which, with a dendrochronologically determined age of 1076 yr, was so far considered the oldest scientifically dated living tree in Europe (Konter et al. 2017).

The ^{14}C concentrations measured for the single-ring samples extracted from the stem and expressed as $\Delta^{14}C$ are shown in Figure 5 as a function of the growth year determined by dendrochronological methods. Our results show a sharp increase in the ^{14}C concentration in 993 CE corresponding to 11.3 ± 3.8‰ in the $\Delta^{14}C$ scale. This change-value compares well with those published by others for this Miyake-event, for instance for Denmark (10.5 ± 3.4‰) and Japan (11.3 ± 3.5‰) (Miyake et al. 2014; Fogtmann et al. 2017). Also the shape of the observed increase is consistent with what is generally observed by others, namely a slight decline before 993 CE, a sharp increase, and a slow decline over the following years probably because of the exchanges with the terrestrial carbon pools. Our

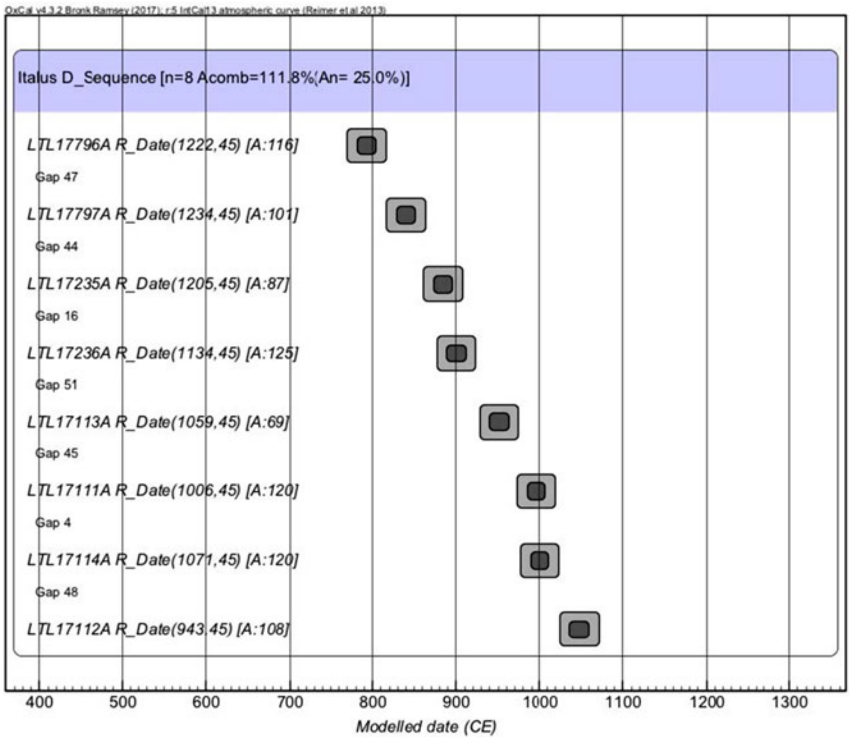

Figure 4 Wiggle match Bayesian model obtained for the analyzed samples.

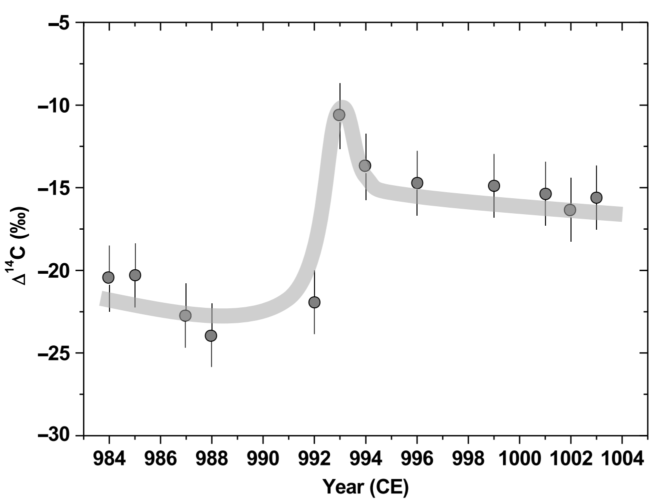

Figure 5 Measured radiocarbon concentrations, decay corrected and expressed as $\Delta^{14}C$ as a function of the dendrochronologically determined age for the single year samples extracted from the stem (errors bars refer to 1σ confidence level).

results then confirm that the 993–994 CE event was a planet-scale phenomenon, not depending on the hemisphere and whose magnitude does not depend on latitude, at least within the experimental uncertainties associated with the measurements (Büntgen et al. 2018). This observation further supports the hypothesis of a solar origin of the measured increase as point out by Güttler et al. (2015) for the 774–775 CE event.

We also note that our results seem to place the event in 993 CE although we do not have sub-annual information because no distinction was made between early- and late-wood. We also note that Hayakawa et al. (2017) have found evidence, in ancient written sources, of Aurora observations in the Korean Peninsula, Saxon cities (nowadays Germany) and Ireland clustering in the period between the late 992 and early 993 CE (Hayakawa et al. 2017). On the basis of these observations, Hayakawa et al. conclude that, if the recorded auroras were associated with the 993–994 Miyake event, the ^{14}C spike should have been in 993 CE. Though further investigations are surely needed, our data obtained on *Italus* seems to support this hypothesis.

CONCLUSIONS

The combined application of ^{14}C AMS dating and dendrochronology allowed us to absolutely date with a high level of confidence *Italus*, a Heldreich's pine growing in the Pollino National Park in Southern Italy. The obtained results allow us to determine a minimum age of the tree ranging between 1201 and 1247 yr in 2017 (with a probability of 95.4%). The comparison of the obtained age with data in the literature data shows that this is currently the oldest scientifically dated tree living in Europe.

Italus can then be considered a proxy record of the climatic and environmental conditions in an area located in the middle of the Mediterranean Basin. As a first application we were able to identify the 993–994 CE increase in the ^{14}C atmospheric concentration (Miyake event). It is worth noting that this high-resolution ^{14}C date is also a direct confirmation of the cross-dated age obtained for the stem through dendrochronological methods. Our results confirm the shape and the amplitude of the increase as observed by others in different locations in both hemispheres. Concerning the timing of the event, our current data seem to indicate that the event occurred in 993 CE though further investigations are needed and already planned.

Concerning future projects, efforts will be devoted to identifying the 993–994 CE Miyake event in the root-derived tree-ring sequences which will also result in a significant improvement of the achieved chronological resolution. Considering the average width of the tree-rings and so the mass of the available material, this will require the use of the gas-accepting ion source in operation at CEDAD (Braione et al. 2015; Maruccio et al. 2017). Future sampling campaigns will be also carried out in order to identify other roots extending further back in time and, hopefully, including also the other 774–775 CE Miyake event.

SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit https://doi.org/10.1017/RDC.2019.37.

ACKNOWLEDGMENTS

This research project was funded by PollinoNational Park (MATTM Biodiversity 2014). F. Biondi was supported, in part, by a grant from the Office of Research and Innovation at the University of Nevada, Reno (USA).

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