Europlanet TA Scientific Report

PROJECT LEADER

Project number: 20-EPN2-018
Name: Andrzej Zbigniew Rakowski
Home Institution: Silesian University of Technology, Poland
TA Facility visited: TA2.16. Isotoptech Carbon-14 dating Accelerator Mass Spectrometry Laboratory, Hungary.

Project Title: Structure of the radiocarbon calibration curve around Miyake effect in 660 BC, AD 775 and AD 994

Scientific Report Summary.
(plain text, no figures, maximum 250 words, to be included in database and published)

Evidence of a rapid increase in the radiocarbon concentration of the tree rings for the year 775 CE was initially presented by Miyake et al in 2012 (henceforth called M12). Since then, other events similar to the M12 have been confirmed for different periods. This project aims to provide new information about the increase in concentration of radiocarbon in the period of abrupt solar activity. For the study we have chosen the periods in XIth and XIIIth century CE and in VIIth century BCE, in which increase of radiocarbon concentration was noted. The samples have been collected from dendro-chronologically dated trees, and the annual rings has been extracted for measurement. During the EUROPLANET TA visit in the Isotoptech Zrt. AMS laboratory, all the samples were prepared to be measured using MICADAS AMS system. Each set of measurement was accompanying with standard samples (of known radiocarbon concentration) to control the quality of the measurement. To obtain high precision (<2 ‰) the measurement time was extended. The results show occurrence of Miyake events in analyzing periods. For the analyzing period in VIIth century we were able to determinate the occurrence during the year, by dividing the annual ring into three parts early-wood, early-late wood and late wood. During the TA visit we have possibility to learn about the procedures used in the laboratory to prepare samples (of different kinds) for radiocarbon measurement using AMS system. We had a fruitful discussion on possible future cooperation, including joint submission of a research project proposal.

Full Scientific Report on the outcome of your TNA visit

The first step, before starting the measurement, was to discuss the possible preparation strategies of the samples, the selection of control samples and the determination of the measurement time to obtain the best results. The samples of annual tree rings of sub-fossil oaks (*Quercus robur* L.) for the test were selected after absolute dating using the dendrochronological method. This activity was done during the Pre-analytical Preparation (3 days), using on-line meeting surface.

During the Main Analytical Phase of our TA visit (10 days) for the measurement of radiocarbon concentration in annual tree ring for all investigated periods, we decided to extract the α-cellulose from the wood. For the α-cellulose extraction we have applied the method routinely use in the Isotoptech Zrt. radiocarbon laboratory described in Molnar et al 2013. The obtained α-cellulose was then placed in the glass tube together with oxidizing agent (MnO_2), pumped out and sealed with a torch. Along with α-cellulose, control samples NBS Ox-II standard, IAEA-C3 (cellulose), and fossil wood background samples (IAEA-C9) were prepared (Janovics et al. 2018). After the sealed tube combustion in 550°C (12 h), the CO_2 gas was released and purify in vacuum line and sealed again in tube containing zinc, titanium hydride and iron. A typical amount of reagents and catalyst was used according to Rinyu et al. 2013. For sample mass between 0.5 to 1.5 mg, 10 mg of TiH_2, 60 mg of Zn and 4.5 mg of iron powder was used. CO_2 consists of 2 steps where the tube is placed in the furnace for 3 hours, initially at 500°C for hydrogen release and iron reduction, then the temperature is raised to 550°C to reduce CO_2. This second stage takes approximately 5 hours. The produced graphite is then pressed into the cathode and measured using EnvironMICADAS AMS system. The measurement time was extended to obtain more then 400.000 counts giving the overall uncertainties for modern samples better then ±2 ‰. All the samples from the project (>60 pcs) along with the control samples (>15 pcs) were prepared during our TA visit in the laboratory and measured in three batches with a correspondingly long measurement time to ensure the best precision.

All results obtained during our TA visit in the laboratory are presented on the figures 1, 2 and 3. The results confirm the presence of the rapid increase in radiocarbon concentration during five period with a magnitude in $\Delta^{14}C$ value of few permil. These results will be used to optimize our software, which allows you to search for rapid increases in radiocarbon concentration in data strings, such as the IntCal20 or SHcal20 calibration curve. This algorithm allows to search for periods in which rapid increases in radiocarbon concentration took place in the past. Detailed data analyses was done during Data Analysis (2 days), shortly after our TA visit, using on-line meeting surface.

For the samples around 660 BCE the annual tree ring were divided into early wood (EW) and late wood (LW) sections, which allows a more accurate (half-yearly) determination of the timing of the ^{14}C change. Based on this it was possible to estimate the period of the occurrence of the causing event seem to have been the late summer (July -2/+1month) of 664 BCE. This set of data will be also used to establish the absolute dendrochronological scale for floating chronology from Poland (Puścizna Wielka I Rucianka) covering the first millennia of BCE including the Hallstatt plateau. The method was described in Krąpiec et al 2020.

Because the Miyake event has a global character, and the absolute increase is constant, also the data from the XIIIth century will be used to find the time period for the

floating dendrochronological scale of algarobo tree (*Prosopis sp.*) from costal Peru. In this period three events of abrupt increase of radiocarbon occurs giving the additional structure to the calibration curve.

During our TA visit, TA host arranged time for us to present research carried out by our Team and the importance of the results of this project for the implementation of the goals assumed in the archaeological research conducted in Gliwice Radiocarbon Laboratory. We also discussed the possibility of preparing a joint project using the capabilities of each of the laboratory.

The results of this project will be presented during the next (24th) Radiocarbon conference organized in Zurich, Switzerland between 10th and 16th of September 2022. The titles of the poster are listed below. The project implemented under the Europlanet24 initiative can be considered a complete success, connecting people, science and the optimal use of the research potential of a research center.

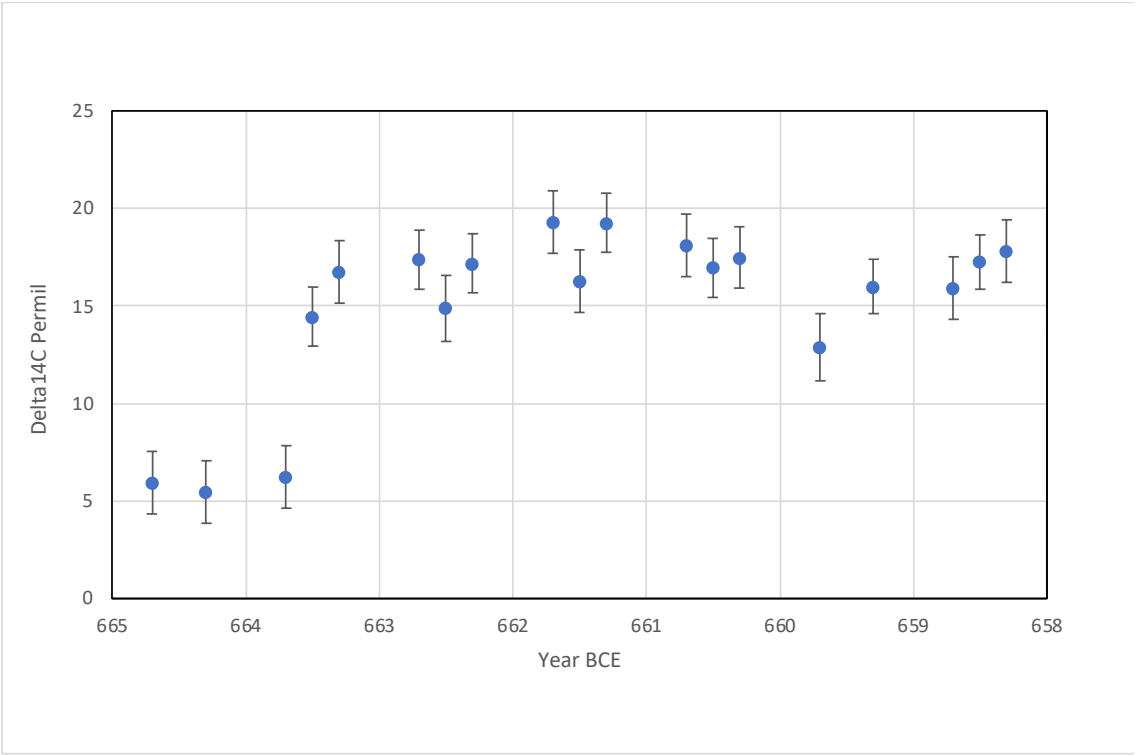

Figure 1. Radiocarbon concentration in $\Delta^{14}C$ in the period between 665 and 658 BCE including increase due to Miyake event in 664 BCE. The results suggest the time of increase during the summer months (July -2/+1month).

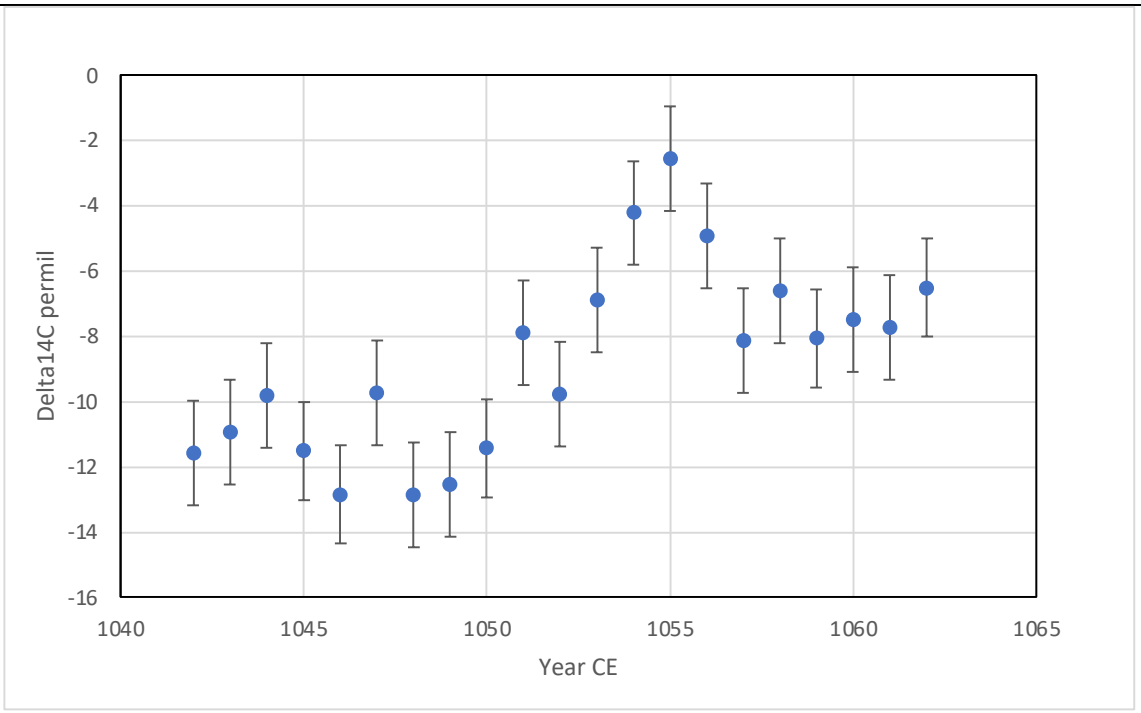

Figure 2. Radiocarbon concentration in $\Delta^{14}C$ in the period between 1040 and 1065 CE including increase due to Miyake event around 1055 CE.

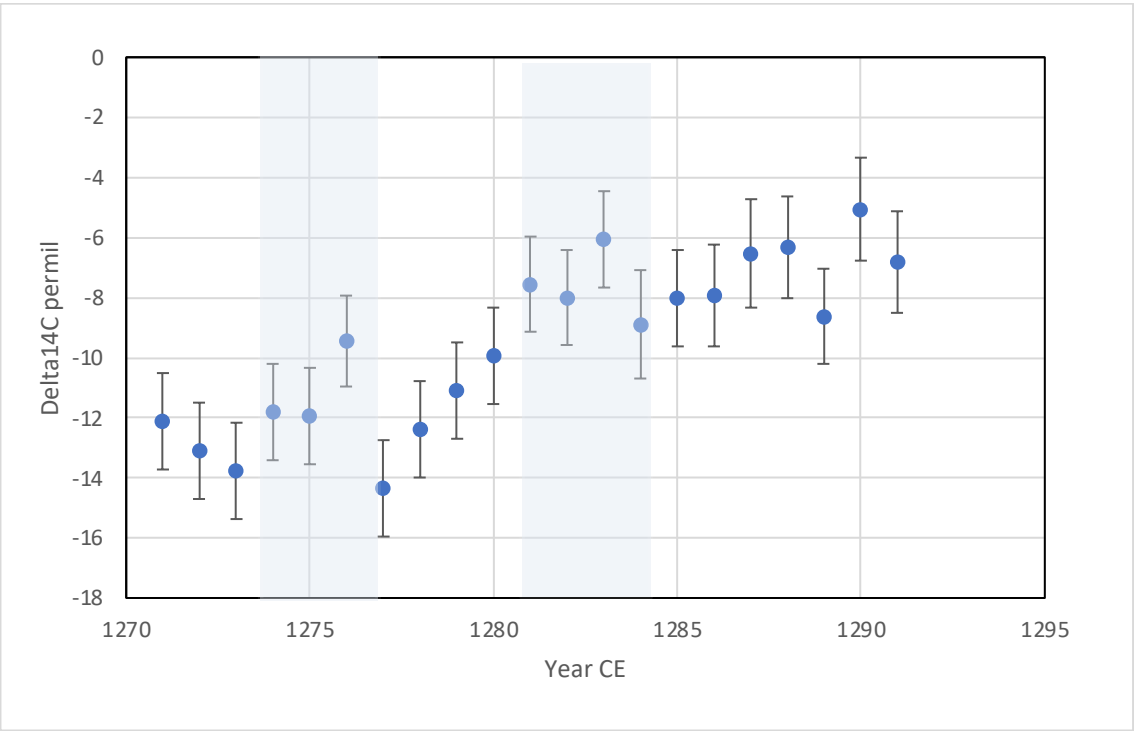

Figure 3. Radiocarbon concentration in $\Delta^{14}C$ in the period between 1270 and 1295 CE including increase due to Miyake event around 1275 CE and 1284 CE.

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Give details of any publications arising/planned (include conference abstracts etc)

24th Radiocarbon and 10th 14C & Archaeology, Zurich, Switzerland – 10-16.09.2022

1. Variations in the radiocarbon calibration curves around known and suspected $\Delta^{14}C$ excursions – poster presentation
2. Implementation of Fe/Zn graphitization method in Dendrochronological laboratory at AGH University of Science and Technology, Krakow, Poland – poster presentation
3. The potential for using $\Delta^{14}C$ excursions to accurately date floating pine chronologies from the Hallstatt period – poster presentation

All of the presented study will be published in Radiocarbon journal.

- Host confirmation

Please can hosts fill in/check this table confirming the breakdown of time for this TA project:

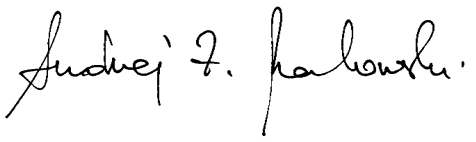
Dates for travel to accommodation for TA visit (if physical visit by applicant)	Start Date of TA project at facility	Number of lab/field days spent on TA Visit pre-analytical preparation	Number of days in lab/field site for TA Visit	Number of days spent in lab for TA Visit data analysis	End Date of TA project at facility	Dates for travel home (if physical visit by applicant)
Departed: 10-07-22 Arrived: 10-07-22	06-07-22	3	10	2	26-07-22	Departed: 22-07-22 Arrived: 22-07-22

The host is required to approve the report agreeing it is an accurate account of the research performed.

Host Name	**Dr. Mihály Molnár**
Host Signature	
Date	**15-08-2022**

- Project Leader confirmation

Do you give permission for the full version of this TA Scientific Report (in addition to the 250 word summary) to be published by Europlanet 2024 RI on its website and/or public reports? YES

Project Leader Name	**Andrzej Zbigniew Rakowski**
Project Leader Signature	
Date	**15-08-2022**