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Radiocarbon dating of cultural heritage objects – case studies of regional importance

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Radiocarbon (^{14}C) dating is a convenient and accurate method of absolute dating of organic materials. Radiocarbon is a cosmogenic isotope formed in the upper atmosphere and after oxidation to CO_2 it becomes involved in the carbon cycle together with other carbon isotopes (^{12}C, ^{13}C). Equilibrium between radioactive ^{14}C decay and its replenishment is established in all living organisms. After death the replenishment stops while the radioactive decay continues. By measuring the ^{14}C remained in the organic material we can determine the time elapsed since the death of the organism. Various organic materials can be ^{14}C dated, such as wood, charcoal, bone, seeds and grains, paper, parchment, hair, etc. Here we present details of two recent studies of the Croatian culture heritage dated in the Zagreb Radiocarbon Laboratory. Dating of several other cultural heritage objects from the neighboring countries will be also briefly described. All the results were obtained by AMS (accelerator mass spectrometry) measurement technique [1] that enables analysis of very small samples containing a few milligrams of carbon, and is therefore applicable to various objects of cultural heritage. The conventional radiocarbon age expressed in years Before Present (BP) were calibrated by OxCal software using the IntCal13 calibration curve [2]. The calibrated ages and age spans are expressed as "cal AD".

The Cathedral of St. Domnius in Split received a massive gilded wooden door on the Feast of St. George in 1214. The doors (Figure 1), made by the local craftsman Master Andrija Buvina, are 530 cm high and the two wings are 360 cm wide. The doors contain 28 scenes from the life of Jesus Christ. Five wood samples (oak and walnut) from the Buvina's door were taken, as well as 2 samples from the choir benches from the Cathedral [3]. At least two wooden pieces belong to the original doors from AD 1214 (Figure 2) with the calibrated age spans cal AD 1155 – 1210 (Z-5720) and cal AD 1054 – 1191 (Z-5721). Two samples (Z-5719, Z-6037) are somewhat younger (cal AD 1245 – 1272 and cal AD 1225 – 1266) and one sample is considerably younger (cal AD 1481 – 1650) indicating that they could not have been built into the original doors. The sample Z-6605 from the choir benches was dated to the second half of the 13th century (cal AD 1266 – 1281, Figure 2), as expected, while the other was probably placed into the benches during the repair in the second half of the 20th century.

The Old olive tree (*Olea europaea L.*) on the Veli Brijun Island, National Park Brijuni, Istria, Croatia, was expected to be 1600 years old (Figure 3). However, the inner and oldest part of the trunk in olive trees usually rots, making the radiocarbon analysis of material from the first years of life of the tree impossible. The inside of the Old olive tree was hollow so that the central, older wood was missing. Five samples were taken from the inner side of the trunk. Four of them resulted in conventional radiocarbon ages of up to 150 BP. After calibration it was found that the wood was not older than 300 years. This is in agreement with most other radiocarbon dates of internal wood from living olive trees, rarely older than 300 years [4]. The last sample clearly indicated a younger branch dated to the period cal AD 1979 – 1981, showing thus the complexity of the olive trunk structure.

Figure 1. Master Buvina's wooden door of the Cathedral in Split, Croatia.

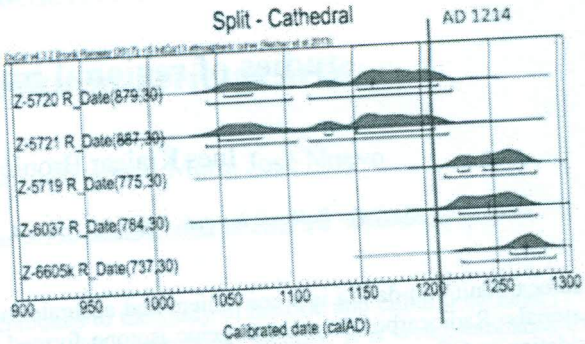

Figure 2. Calibrated age spans of four samples from the Master Buvina's door (Z-5720, Z-5721, Z-5719 and Z-6037) and sample Z-6605 from the choir benches.

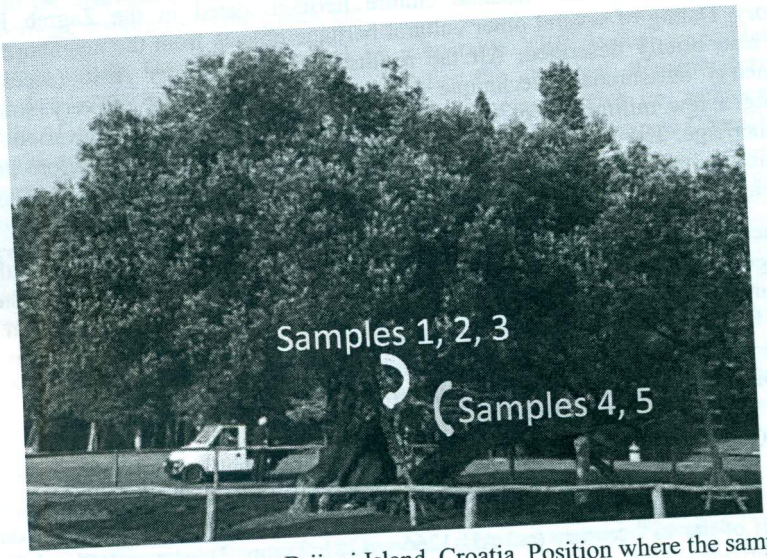

Figure 3. The Old olive tree, Brijuni Island, Croatia. Position where the samples for ^{14}C dating were taken.

The museum collection of the Franciscan Monastery of the Holy Spirit in Fojnica, Bosnia and Herzegovina, houses numerous objects important for the nation's cultural heritage, among which is the 15th century document *Ahdname from Fojnica* [5]. Two different types of paper were identified and sampled for ^{14}C dating. The calibrated age span of the "lower piece" of the document (Z-5126), cal AD 1438–1451 (68.2 %), corresponded with the expected historical age of the Ahdname (AD 1463) proving that this part was the original document. Here we have to keep in mind that ^{14}C dating gives the age of a material and not the date of the document issuing. The other piece (Z-5124) was younger indicating restoration after some

damaging. Although there is no written evidence, the opinion of monks in the Franciscan Monastery in Fojnica is that the Ahdname was renewed in the 17^{th} – 18^{th} century, in accordance with the obtained dates.

The museum collection of the same monastery also houses the Book of Coats of Arms, the so-called *Fojnica Armorial*. Two types of paper were identified and both were ^{14}C dated. The "thick" paper (Z-5700) showed the calibrated age span cal AD 1635 – 1662 (with the probability of 60.5 %), corroborating the most likely assumption of the age of the book. The "thin" paper (Z-5701) was younger suggesting that the Armorial was compiled over different times [5].

Several parchment sheets were discovered in an unmarked box in the Archive of Vojvodina (Novi Sad, Serbia) in 2015 [6]. The sheets contained fragments of manuscripts written in Latin, but did not contain chronological qualifiers and were used as binders for code books from 16^{th} century. The K3 fragment (Z-5806) was ^{14}C dated to 14^{th} century, in accordance with the archaeographical analysis [6]. The K4 fragment (Z-5807) contained text written in Carolingian minuscule calligraphy that was used in 12^{th} century [6]. Radiocarbon dating placed this fragment to 13^{th} century. Since there were no other indications of the age, the K4 fragment was officially attributed to 12^{th} - 13^{th} century. It was concluded that the K4 parchment fragment is the oldest document in the Archive of Vojvodina and one of the oldest documents in all the archives of Serbia, and therefore, it represents a unique example of overall manuscript heritage in the Republic of Serbia.

Acknowledgments. The staff of the Zagreb Radiocarbon Laboratory is greatly acknowledged for sample preparation. Ž. Matulić Bilač (Croatian Conservation Institute, Split) performed a careful restoration of the Master Buvina's door and submitted the samples. National Park Brijuni enabled sampling and dating of The Old olive tree.

Keywords: ^{14}C dating, The Old olive tree, Master Buvina's door, Brijuni, Split, Croatia

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Limitation and comparison of two methods for determination of biogenic fraction in liquid fuels by ^{14}C

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European Union's promotion of the use of sustainable and renewable resources requires use of at least 10 % of synthesized biodiesel in liquid fuels by the year 2020. This legislation has stimulated various types of petrodiesel and bio-based component blends production, and development of methods for exact, effective and reliable quantification of biodiesel content. The method for determination of the biogenic fraction in liquid fuels by direct measurement of the ^{14}C activity concentration via liquid scintillation counting (LSC) technique was developed in few laboratories worldwide. It is based on different ^{14}C signatures of the two components: the biogenic component reflects the modern atmospheric ^{14}C activity, and no ^{14}C is present in fossil fuels. The quantity of ^{14}C in the fuel is the criterion for bio-fuel presence. This method is regarded as fast, simple, accurate and sensitive determination procedure for the mass assessment of biogenic fraction in biofuels.

Laboratory for low radioactivity at the Department of Physics, University of Novi Sad (UNS), Serbia, and Laboratory for low-level radioactivities of the Ruđer Bošković Institute (RBI) in Zagreb, Croatia have developed two different methods for biogenic fraction determination in liquid fuels [1,2]. Both laboratories tested the stability of prepared samples by measuring the same samples after some period of time and used the same set of mixtures with the known fractions of the biogenic component, i.e. FAME produced from either sunflower oil or lard fat. The comparison revealed in general good agreement of the results and also indicated some constraints of the methods used. The RBI evaluation technique is limited by the SQP(E) values lower than 600 where the count rates of the biogenic and the fossil samples become indistinguishable. The lower the SQP(E), the larger the relative differences between the measured and the expected biogenic fraction for $600 < SQP(E) < 700$. The biogenic fraction can be successfully determined at $SQP(E) > 700$. The two-step calibration procedure that UNS used implements quench correction and therefore offers more reliable ^{14}C determination in fuels in comparison to one-step calibration method [1]. The application of these calibration curves is limited to samples with chemically identical bio and fossil components. It can be used for precise biogenic fraction determination in examined fuel samples if the components of the fuel mixture are well known in advance. The results of participation of both laboratories in the international proficiency test – interlaboratory comparison in 2018 will be also presented.

Keywords: ^{14}C, liquid fuel, biogenic fraction, intercomparison

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Screening LSC method for monitoring ^{14}C activity in water samples

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This paper explores the possibility of a direct measurement of ^{14}C activity concentration in water samples without chemical pretreatment of samples. A method via Liquid Scintillation Counting (LSC) technique has been implemented and optimized. The method assumes mixing of water sample with a scintillation cocktail. Permafluor E+ and Ultima Gold AB cocktails were tested. Polyethylene vials were selected since they induce lower background count rate than glass vials. Measurements were performed on Ultra Low Level Liquid Scintillation Spectrometer Wallac 1220 Quantulus. Optimization of the method included optimal water:cocktail volume ratio and optimal counting window selection as well as PAC (Pulse Amplitude Comparator) parameter adjustment, based on the highest FOM (Figure Of Merit) value achieved. Quench level parameter has been monitored for all samples while counting. Detection efficiency and detection limit have been determined as the main parameters for method's evaluation. Results with Permafluor E+ samples were the following: obtained detection efficiency was (78.79±0.23)% with the detection limit 1.05 Bq l^{-1} for 300 min of measurement for samples prepared in 6:14 sample:cocktail volume ratio, and (71.05±0.16)% with the detection limit 0.68 Bq l^{-1} for 300 min of measurement for samples with 10:10 ratio. Results with Ultima Gold AB samples included the detection efficiency (61.96±0.03)% with the detection limit 0.88 Bq l^{-1} for 300 min of measurement for samples prepared in optimal 9:11 sample:cocktail volume ratio. Method's constraints, its accuracy and precision, have been considered from the obtained results of measurements of nuclear power plant waste water samples in which ^{14}C activity concentration has been determined independently. The presented method is ultrafast, simple and inexpensive test for the efficient screening of water samples which could be used for ^{14}C monitoring purposes in the case of nuclear emergencies. Its application is limited only to ^{14}C screening of samples with much higher ^{14}C activities than those of the environmental (surface and ground) waters.

Keywords: ^{14}C in water, Liquid Scintillation Counting (LSC), optimization