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THE IAEA FORENSICS PROGRAM: RESULTS OF THE AMS ^{14}C INTERCOMPARISON EXERCISE ON CONTEMPORARY WINES AND COFFEES

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ABSTRACT. In the frame of the IAEA-CRP (Coordinated Research Projects): *Enhancing Nuclear Analytical Techniques to Meet the Needs of Forensic Sciences*, an intercomparison exercise was organized between three AMS laboratories. Aim of the program is to promote the use of nuclear and accelerator-based techniques in routine forensics practice. In this view, one of the key points is the assessment of the precision and accuracy levels achievable on material of forensic interest. We review the general structure and status of the project, with emphasis on results obtained in the analysis of wines of different grape varieties and grounded coffee beans from different locations such as Brazil, Spain, and Italy. The three laboratories processed the samples according to different chemical protocols and performed the ^{14}C measurements using different systems: MICADAS in Zurich and Debrecen and a HVEE 4130HC 3 MV Tandetron in Lecce. Within the quoted uncertainty, the results showed good reproducibility, indicating that uncertainty level of the order of 0.3% are achieved by AMS on a single sample while multiple sample analyses results in precision down to 0.1–0.2%. The measured ^{14}C concentrations on coffee and wine samples resulted to be consistent with atmospheric ^{14}C levels in the growing years.

KEYWORDS: AMS, bomb peak, food, forensics, radiocarbon.

INTRODUCTION

Since 2017 the IAEA (International Atomic Energy Agency) has undertaken a Coordinated Research Project (CRP F11021) entitled "Enhancing Nuclear Analytical Techniques to meet the Needs of Forensics Sciences." The main scope of the program is to develop and utilize the capabilities of nuclear and accelerator-based analytical techniques towards recognized needs of forensic sciences that could not be efficiently addressed by other methods. Indeed, different techniques such as accelerator mass spectrometry (AMS) radiocarbon dating, ion beam analysis (IBA), nuclear reactors, and synchrotron-based analytical methods have important advantages which make them suitable for forensics applications such as non (or reduced) destructiveness and high analytical sensitivity. Nevertheless, their application in the forensics practice appears to be still limited by different reasons including primarily, the instrumental complexity, the high costs of instrumentation, and to a lesser extent, the scarce knowledge of their potential when compared with more "traditional" techniques. The aim of the CRP is then to try to overcome these limitations by building and supporting long-term collaborations and

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networking between experts in nuclear analytical techniques and forensic science stakeholders such as law enforcement agencies, police corps and international organizations. The program has fourteen participating countries and is structured in four work packages (WPs). The first three have been defined, mainly, on the base of the specific needs and priorities of the participating countries: forensic glass analysis (WP1), food authentication and provenance (WP2), nuclear techniques in heritage forensics (WP3). The fourth one, WP4 (*Radiocarbon for Forensics*) is the only one specifically dedicated to a single analytical technique and was intended, since the beginning, to be in strict connection with the other WPs, in particular WP2 and WP3.

The applications of radiocarbon dating in forensics is mainly based on the analysis of the excess of anthropogenic ^{14}C in the terrestrial atmosphere produced by aboveground nuclear detonation tests carried out after World War II, distributed among the different terrestrial reservoirs and uptaken by living organisms (Nydal 1968). The reconstruction of the curve (or "bomb peak") describing this excess as a function of time and its use as a calibration curve allows dating of samples younger than ~70 years and achieve the high chronological resolutions typically required in forensics (Levin and Hesshaimer 2000; Levin et al. 2010; Quarta et al. 2005; Hua et al. 2013; Hajdas et al. 2021). The use of "bomb peak dating" in forensics is surely not new and its diverse applications range from the analysis of human skeletal remains (Wild et al. 2000; Spalding et al. 2005; Cook et al. 2006; Calcagnile et al. 2013; Brock and Cook 2017; Handlos et al. 2018), to the identification of work of art forgeries (Caforio et al. 2014; Brock et al. 2019; Hendriks et al. 2019), the analysis of illicit drugs (Zoppi et al. 2004), the fight against the illegal trade of endangered animals (Wild et al. 2019; Quarta et al. 2019) and foodstuff (Varga et al. 2020). Nevertheless, one should note that, despite the wide scientific literature related to the potential of ^{14}C in forensics, its use in routine practice has been limited. Within WP4, in order to cope with the factors limiting the impact of AMS dating in forensics, a detailed research program was defined on the basis of discussions with forensic stakeholders and experts of other analytical techniques. The need to define common guidelines and quality assurance protocols for the application of ^{14}C was established, also considering possible ethical issues (Hajdas et al. 2019), interpretation of data and the assessment of the achievable chronological resolution on classes of samples of interest in forensics.

Indeed, though simple in its basic principles, the proper and correct interpretation of bomb ^{14}C data can be quite complicated, related to carbon turnover and time of fixation in non-short living organisms or local deviations from the global average atmospheric curve as due to natural or anthropogenic causes, for example. Different intercomparison exercises were then organized and run among the three facilities: CEDAD (Italy), ETHZ (Switzerland), and Isotoptech-ATOMKI (Hungary) aimed at addressing these issues on sample materials relevant in forensics such as ivory, bones, paper and textiles (Quarta et al. 2019, 2021).

In this paper, we report on the intercomparison exercise organized in strict collaboration with WP2 and aimed at exploring the possible contribution of ^{14}C to the analysis of foodstuffs and in particular coffee and wines. Indeed, though intercomparison exercises are organized and run regularly within the ^{14}C dating community, these do not include typically foodstuff samples (Scott 2003; Scott et al. 2010).

Here, the use of ^{14}C can be twofold: supplying additional information about authenticity and identification of counterfeit or adultered products (Zoppi et al. 2004; Povinec et al. 2020). In

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the latter case radiocarbon is used as a tracer of the biogenic fraction and the large isotopic difference in term of ^{14}C content between fossil (completely depleted in ^{14}C) and biomass-derived materials (whose ^{14}C concentrations mirrors atmospheric levels) is used to quantify the relative proportion of the two fractions (Quarta et al. 2013; Haverly et al. 2019; Varga et al. 2018; Oinonen et al. 2010).

The results obtained in the analysis of wines of different grape varieties, provenances and vintages and ground coffee beans samples from different locations in the world such as Brazil, Spain and Italy are presented.

MATERIALS AND METHODS

The main goal of the present study is to assess the reproducibility of the measurements obtained on wines and grounded coffee by the three participating laboratories: CEDAD-Center for Applied Physics, Dating and Diagnostics at the University of Salento, Lecce, Italy; the Laboratory of Ion Beam Physics at the ETH in Zurich, Switzerland; and the HEKAL-Hertelendi Laboratory of Environmental Studies, joint lab of Isotoptech Ltd. and Institute for Nuclear Research, Debrecen, Hungary.

The laboratories used different procedures to chemically process the samples and are based on different AMS instruments. In particular, HEKAL and ETHZ are based on a MICADAS (Mini Carbon Dating System, IonPlus, Switzerland) AMS system operated at 200 kV (Synal et al. 2007; Molnar et al. 2013) while CEDAD is based on a 3 MV Tandetron™ type accelerator manufactured by High Voltage Engineering Europa, the Netherlands.

Sample Material

Overall, 8 samples were used for the study and submitted for AMS radiocarbon analyses: two ground coffee samples obtained from two different brands and six bottles of wine.

The coffee samples (sample#1 and sample#2) were obtained from two 250-g packages available on the market in Brazil. They were opened, carefully mixed, in order to obtain three homogeneous subsamples, and they were then submitted to the three laboratories for AMS determinations. Sample#1 was obtained from a popular Brazilian roasted ground coffee from Melitta brand (*Tradicional*) packed in 2017. For this coffee, no certification of origin was available since several farms across Brazil typically supply coffee beans for the production of packed roasted ground coffee. The second sample (sample#2) was obtained from the same Melitta brand labeled *Sul de Minas*, where the name indicates the region where the respective crops were cultivated and harvested. Therefore, this coffee was produced in a defined region and its origin was controlled for superior quality, with a known production year of 2017. The same samples were also submitted in the frame of the same IAEA CRP to compositional analyses by different techniques such as PIXE (particle induced X-ray emission) and FTIR (Fourier transform infrared analysis) whose results are being published elsewhere.

The list of the six analyzed wines is given in Table 2 summarizing vintage, provenance, and production year as indicated on the label. All the samples were red wines except for sample #5, which was a white wine. The wines were produced from 2008 to 2019 in different regions in Italy (South Tyrol, Sardinia), Spain, Portugal, and Brazil. Wine bottles were all

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Table 1 List of the coffee samples and measured radiocarbon concentrations.

Laboratory	Chemical processing	$F^{14}C$-sample#1	$F^{14}C$-sample#2
CEDAD	None	1.0285 ± 0.0030	1.0223 ± 0.0032
	AAA	1.0296 ± 0.0030	1.0230 ± 0.0032
HEKAL	None	—	1.0265 ± 0.0029
	AAA	1.0232 ± 0.0026	1.0264 ± 0.0029
ETHZ	None	—	1.0247 ± 0.001 (n=3)
	AAA	1.0278 ± 0.0012	1.0245 ± 0.002 (n=2)

Table 2 List of the analyzed wine samples.

Sample	Wine	Provenance	Label year
#1	Cave Merlot	Serra Gaucha, Brazil	2008
#2	Cave Merlot	Serra Gaucha, Brazil	2015
#3	Reserva	Cintruénigo, Navarra, Spain	2014
#4	Montessu	Sardinia (Sulcis), Italy	2011
#5	Moscato	Lisbon, Portugal	2019
#6	Cabernet	Nalles Sud Tirol-Italy	2017

opened at ETH in Zurich where subsamples were obtained and sent in sealed and labeled ampoules to the other two laboratories.

Sample Processing

CEDAD Sample Processing

For each of the two coffee samples (~5 mg), two different processing procedures were used. The sample were either combusted without any previous chemical processing or processed following the AAA protocol and consisting of alternate acid (HCl)-alkali (NaOH)-acid (HCl) washings.

Sample material was then combusted to carbon dioxide at 900°C with copper oxide for 4 hr in sealed quartz tubes. Released CO_2 was cryogenically purified and then reduced to graphite at 600°C by using H_2 as reducing agent and 2 mg Fe powder as catalyst (D'Elia et al. 2004). All the samples yielded an optimal quantity of ~1 mg of graphite which was then pressed in the aluminum cathodes of the AMS system (3 MV Tandetron™ Mod. HVEE 4130HC) for the measurement of the isotopic ratios (Calcagnile et al. 2019). Measured $^{14}C/^{12}C$ ratios were then corrected for mass fractionation by using the $\delta^{13}C$ measured online with the AMS system, and for machine and chemical processing background. Uncertainty in measured isotopic ratios was calculated by considering both the scattering of ten repeat determinations performed on each sample and the radioisotope counting statistics (Calcagnile et al. 2005).

For wines, about 50 µL of liquid were sampled, heated at 60°C in quartz tubes to remove alcohol and volatile components. The tubes were then vacuum sealed and kept at 900°C for 4 hr to produce carbon dioxide which was then recovered, purified, converted to graphite and measured as described above for coffee samples.

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HEKAL Sample Processing

Coffee samples (~5 mg) were combusted either without any chemical processing or after being processed according to the AAA protocol.

About 50 µL of each wine sample was dried in a combustion tube at 70°C overnight. In this way, all the alcohol component was removed, and the solid residue was used for the following ^{14}C measurement.

The selected sample fractions (coffee and wine after chemical preparation) were then oxidized at 550°C for 12 hr in sealed glass tubes by using MnO_2 as catalyst. The produced CO_2 was then purified from any other by-product gases and quantified using a dedicated vacuum line (Janovics et al. 2018). A carbon yield between 1 to 2 m/m% was measured for all the samples. For ^{14}C dating by AMS, graphite targets from the purified CO_2 samples were prepared using a customized sealed tube graphitization method (Rinyu et al. 2013). The ^{14}C measurements were performed using the EnvironMICADAS AMS instrument at HEKAL (Synal et al. 2007; Molnár et al. 2013). The overall measurement uncertainty for modern samples was < 3.0‰, including normalization, background subtraction and counting statistics. The ^{14}C determinations were evaluated by the "Bats" software package (version 3.66; Wacker et al. 2010; Stuiver and Polach 1977).

ETHZ Sample Processing

One coffee sample (#1) was weighed into the aluminum cups for combustion without treatment. Sample#2 was analyzed as untreated and after the standard ABA 60°C treatment. Ca. 3 mg of coffee was combusted in Elemental Analyzer and transferred to AGE system for graphitization (Wacker et al. 2010). Wine samples (ca. 2 mg) were placed in small Q-vials and placed in preheated 9 mm diameter Vycor tubes containing CuO wires. Tubes were subsequently placed in LN bath and after frozen evacuated and closed, combusted at 950°C and transferred to 6 mm diameter tubes for cracker interface of AGE3. All the samples were pressed into Al cathodes and measured together with blank and standards using MICADAS (Synal et al. 2007). The ^{14}C determinations were evaluated by the "Bats" software package (version 3.66; Stuiver and Polach 1977; Wacker et al. 2010).

RESULTS AND DISCUSSION

The radiocarbon concentrations measured for the two coffees samples are given in Table 1 as $F^{14}C$, the normalized fraction of ^{4}C calculated as the ratio A_{SN}/A_{ON} between the $^{14}C/^{12}C$ ratio in the samples (A_{SN}) and in the standard (A_{ON}) both corrected for mass fractionation (Reimer et al. 2004). Results are summarized in Figure 1 with a quoted uncertainty of one standard deviation for each of the three laboratories.

We can observe that the ^{14}C concentrations measured on samples processed by the AAA protocol and unprocessed ones give results within one standard deviation for both samples and for the three laboratories. This is likely the result of the absence of contaminating substances containing exogenous sources of (radio)carbon. It also suggests that for the analysis of these types of samples an intense and time-consuming chemical processing should not be needed.

For both the samples, all the measured values from the three laboratories overlap within one standard deviation and average values of 1.0269 ± 0.0033 and 1.0246 ± 0.0024 can be calculated for AAA processed sample#1 and sample#2, respectively. A similar picture is

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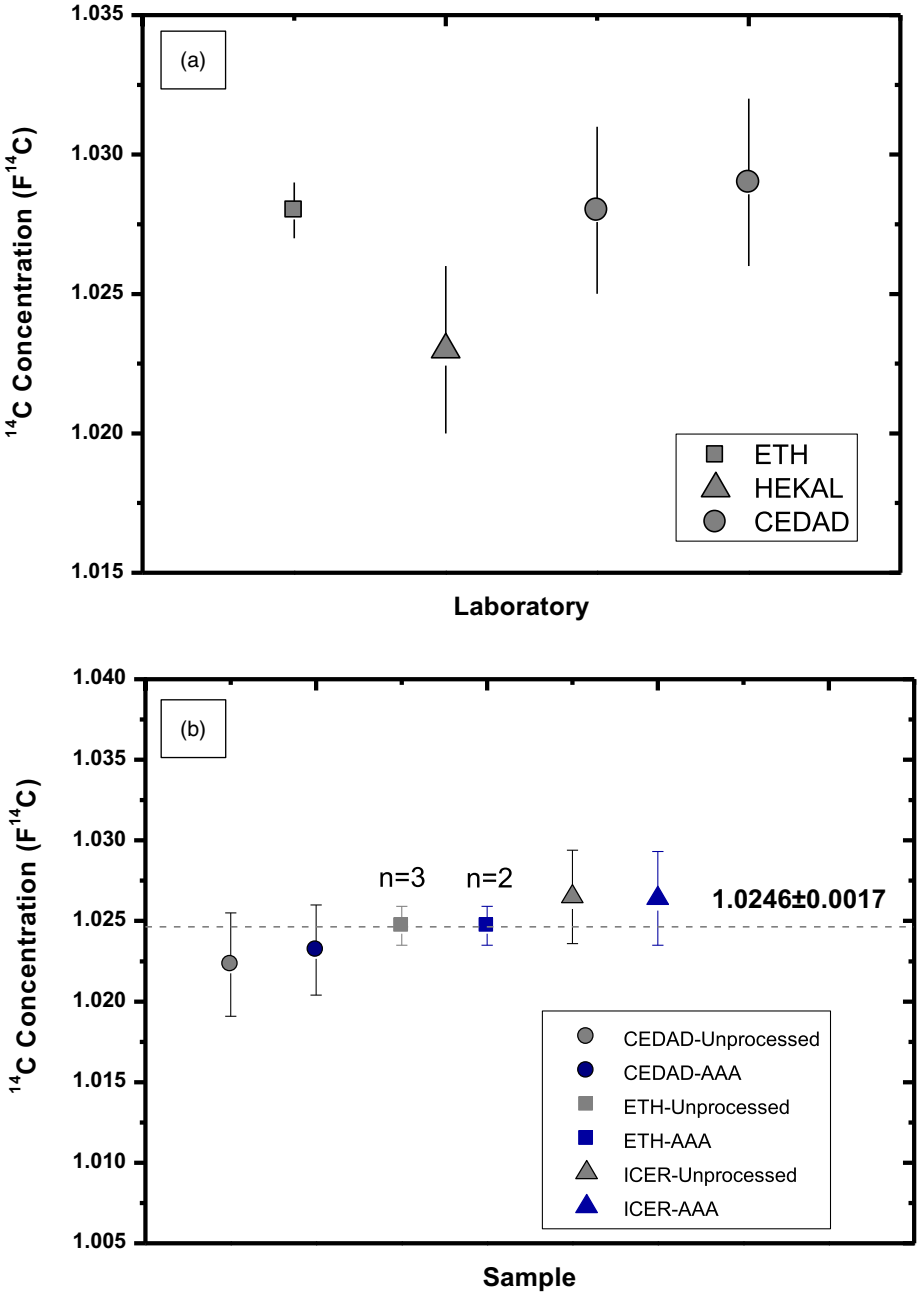

Figure 1 Radiocarbon concentrations measured for coffee sample#1 (a) and sample#2 (b) in the three laboratories.

also obtained for sample#2 when comparing the results found when no chemical processing is applied. In this case the average value measured for the three laboratories is 1.0245 ± 0.0021. When the results obtained on processed and unprocessed sample are averaged, a ^{14}C concentration of 1.0246 ± 0.0017 can be calculated for sample#2.

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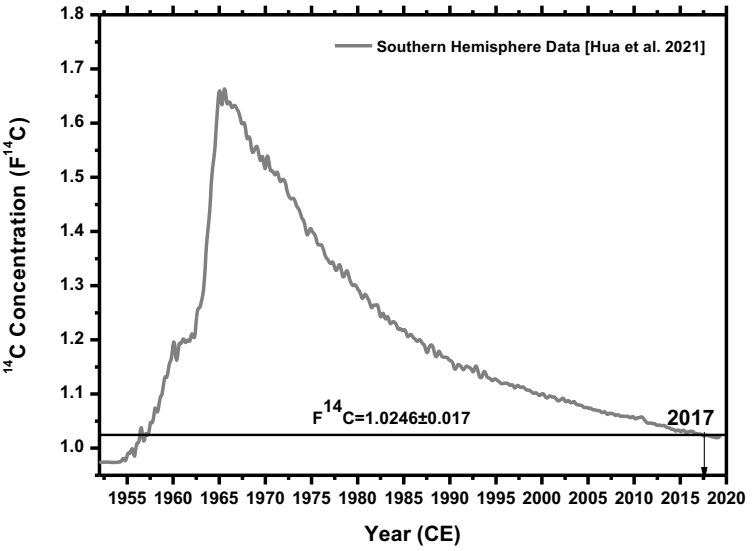

Figure 2 Calibration of average ^{14}C content measured for the coffee sample#2.

In order to compare the measured radiocarbon concentration with the expected age of the coffee (2017), we used the compilation of atmospheric data valid for the Southern Hemisphere (Hua et al. 2021). The result of the calibration procedure is shown in Figure 2. The first intercept with the curve falls in the late 1950s and can be discarded in the present case while the second one indicates a date in 2017 with an uncertainty of one year which is consistent with the packing date indicated on the label.

The results obtained for the wine sample are given in Table 3 and summarized in Figure 3 for the six samples, together with the average value and the corresponding uncertainty calculated from the scatter of the data. It can be noted that for all the samples (except sample#1 and #2) the values measured by the three laboratories agree within one standard deviation. For sample#2 the larger difference is observed between the Hekal and CEDAD results but it is still of the order of 1.3 standard deviations. For sample#1 the statistical agreement is worse though still within two standard deviations and a more detailed discussion about the results for this wine will be given later.

The measured radiocarbon concentrations are plotted versus the labeled production year in Figure 4, where the atmospheric data for the Northern Hemisphere are also shown (Hammer and Levin, 2017). Also, in this case we can observe that all the wines (again except sample#1 dated to 2008) gave radiocarbon concentrations corresponding well to a declining trend coherent with the expected one. It is also clear that the curve is flattening and that it is now approaching pre-bomb levels.

For sample#1, the obtained results are somehow troublesome since the data measured by the three laboratories show more scatter than expected and the average value is significantly lower than the global atmospheric level in 2008. At the moment, we do not have a satisfactory explanation for this. Possible causes for the measured ^{14}C level include a vintage younger than declared, a large contribution of fossil sources of carbon in the grape growing area or the use of additives with low ^{14}C levels. Further analyses are planned to determine the

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Table 3 Measured radiocarbon concentrations for the six wine samples. Quoted uncertainties refer to one standard deviation confidence level.

Lab code	Sample#1	Sample#2	Sample#3	Sample#4	Sample#5	Sample#6
ETH	1.0200 (0.0035)	1.0260 (0.0035)	1.0180 (0.0030)	1.0450 (0.0030)	1.0150 (0.0029)	1.0070 (0.0030)
CEDAD	1.0373 (0.0040)	1.0305 (0.0035)	1.0224 (0.0040)	1.0445 (0.0035)	1.0115 (0.0035)	1.0145 (0.0040)
HEKAL	1.0283 (0.0039)	1.0211 (0.0039)	1.0168 (0.0040)	1.0475 (0.0038)	1.0181 (0.0034)	1.0085 (0.0038)
Average	*1.0285 (0.0087)*	*1.0259 (0.0047)*	*1.0191 (0.0029)*	*1.0457 (0.0016)*	*1.0149 (0.0033)*	*1.0100 (0.0040)*

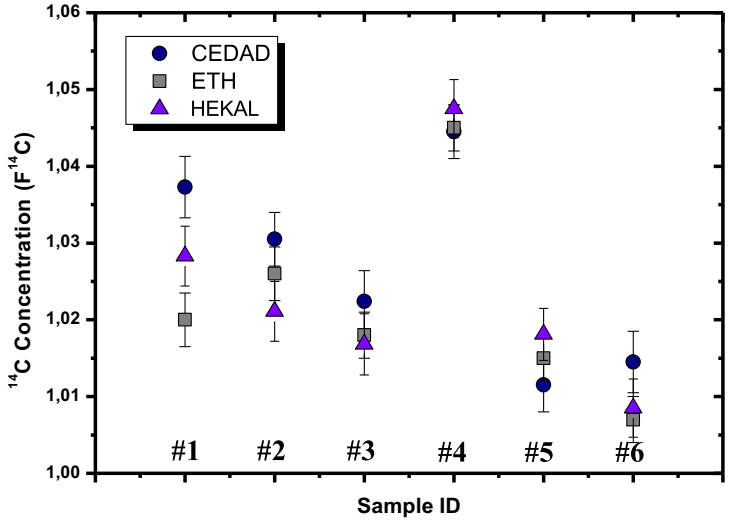

Figure 3 Measured radiocarbon concentrations for the six wine samples.

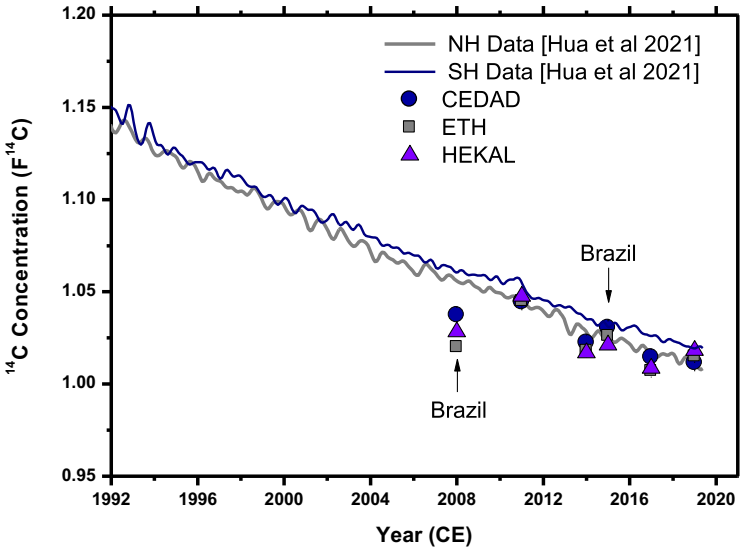

Figure 4 Comparison between radiocarbon concentrations measured for the wine samples and the Northern and Southern Hemisphere bomb spike curves.

origin of these differences, by analyzing other wine components such as ethanol allowing a more comprehensive picture (Povinec et al. 2020).

CONCLUSIONS

In the frame of the IAEA (International Atomic Energy Agency) Coordinated Research Project (CRP F11021) entitled "Enhancing Nuclear Analytical Techniques to meet the

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Needs of Forensics Sciences" an intercomparison exercise was performed on roasted and grounded coffee beans and wine samples among three AMS laboratories. The aim of the study was to assess the achievable precision levels and demonstrate the potential uses of ^{14}C in food authentication, which is now a major concern at the global level. The results indicate that uncertainty level of the order of 0.3% are routinely achieved by AMS on a single sample while multiple sample analyses results in precision down to 0.1–0.2%. A remarkable reproducibility of the results between the different laboratories has been also assessed on both wine and coffee samples. The measured radiocarbon concentrations for the analyzed samples resulted to be consistent with atmospheric ^{14}C levels in the growing years within the uncertainty of the analyses. We also observe that the flattening of the bomb curve results in reduced chronological resolution which is of the order of 1.5/2 years with a confidence level of one standard deviation, for the analyzed samples and time ranges (2008–2019). The apparently inconsistent results obtained on one of the analyzed wine samples also demonstrates the potential of ^{14}C analysis to highlight the possible presence of unwanted or undeclared substances or, at least, to identify sample deserving further attention and investigations.

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