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Ion Beam Analysis and ^{14}C Accelerator Mass Spectroscopy to Identify Ancient and Recent Art Forgeries §

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§ This article is dedicated to the memory of dear missing colleagues Claire Berthier (CEA), Thierry Borel and Joseph Salomon (C2RMF).

Abstract: Forgeries exist in many fields. Money, goods, and works of art have been imitated for centuries to deceive and make a profit. In the field of Cultural Heritage, nuclear techniques can be used to study art forgeries. Ion beam analysis (IBA), as well as ^{14}C accelerator mass spectrometry (AMS), are now established techniques, and the purpose of this paper is to report on their capacity to provide information on ancient, as well as modern, forgeries. Two case studies are presented: the production of silver counterfeit coins in the 16th century and the detection of recent forgeries of 20th century paintings. For the counterfeit coins, two silvering processes were identified by IBA: mercury silvering (also called amalgam silvering or fire silvering) and pure silver plating. The discovery of 14 mercury silvered coins is an important finding since there are very few known examples from before the 17th century. In the detection of recent forgeries, among the five paintings examined, ^{14}C dating showed that three of them are definitely fakes, one is most likely a fake, and one remains undetermined. These results were obtained by using the bomb peak calibration curve to date canvas and paint samples.

Keywords: ion beam analysis (IBA); ^{14}C accelerator mass spectroscopy (AMS); forgeries; silver coins; counterfeit coins; painting

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Citation: Beck, L. Ion Beam Analysis and ^{14}C Accelerator Mass Spectroscopy to Identify Ancient and Recent Art Forgeries. *Physics* **2022**, *4*, 462–472. https://doi.org/10.3390/physics4020031

Received: 24 February 2022
Accepted: 4 April 2022
Published: 26 April 2022

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1. Introduction

Counterfeiting is the fraudulent imitation of a valuable product with the intention to deceive. This illegal practice is found in many fields: forgery of currency or documents, imitation of items such as clothing, pharmaceuticals, automobile and aircraft parts, food, electronics, watches, and works of art. One of the main issues for experts is to identify or to date the material used in producing forgeries, as the appearance is generally close to that of the genuine product. Carried out together with other expertise tools, scientific investigations are, thus, conducted to clarify the authenticity of the product, based on composition and/or age determination.

When dealing with genuine or alleged cultural heritage objects, these investigations have to be as minimally invasive as possible. The most widespread methodology is mainly based on imaging techniques, such as multispectral imaging, X-ray radiography, or microscopy, and on chemical analysis to look for anachronisms or contentious materials [1–5]. In recent decades, the advent of nuclear techniques—derived from low-energy accelerators—for art and archaeological study has paved the way for new methodologies to analyze and authenticate cultural heritage objects [6–12].

Ion beam analysis (IBA), as well as ^{14}C accelerator mass spectrometry (AMS), are now established techniques [13,14], and the purpose of this paper is to report on their capacity to provide information on ancient and modern forgeries. Two case studies are presented: (1) the production of silver counterfeit coins in the 16th century AD and (2) the detection of modern painting forgeries.

The results obtained here contribute to our knowledge of ancient counterfeiting and modern forgery practices.

2. Materials and Methods

2.1. Counterfeit Coin Characterization by Ion Beam Analysis

It is relatively rare to find counterfeit coins for many reasons. First, counterfeit coins were not commonly kept as valuables by private individuals in the past and, when preserved, collectors have neglected them. Furthermore, the generally poor quality of the imitations, as well as corrosion, prevent good preservation.

However, the hoard of Preuschdorf, found in 2005, offered the opportunity to explore the production of counterfeit coins in the 16th century [15,16]. Among 7527 coins found, 38 counterfeit coins were identified (Table 1), imitating contemporaneous official issues (mainly Pfennige) produced in the Holy Roman Empire between 1535 and ~1620 (Figure 1). Non-destructive techniques were used to investigate their manufacture: X-ray radiography and Ion beam analysis (IBA) for the coins [16–18] and scanning electron microscopy (SEM) for fragments collected during restoration. Particle-induced X-ray emission (PIXE) and Rutherford backscattering spectrometry (RBS) were carried out simultaneously using the 3 MeV proton beam of the AGLAE accelerator at the Centre de Recherche et de Restauration des Musées de France (Louvre Palace, Paris, France) [19]. PIXE analysis provided elementary composition (mainly Ag, Cu, Au, Pb, Zn, Hg) and RBS—the depth profile of the major elements. The experiments were conducted with a setup combining two Si (Li) X-ray detectors for PIXE and one surface barrier detector for RBS [20]. PIXE spectra were fitted by GUPIX or GUPIXWIN [21], and RBS spectra were simulated with SIMNRA [22]. Altogether, 140 coins were ion beam analyzed; only results on the counterfeit coins and their official counterparts are reported in this paper.

Figure 1. Examples of counterfeit coins from the hoard of Preuschdorf (**d**–**f**) compared to their official counterparts (**a**–**c**). Details of their provenance are given in Table 1. (Photos: Anaïs Vigneron, Archéologie-Alsace).

Table 1. Counterfeit coins identified in the hoard of Preuschdorf (Alsace, France) found in 2005. The design of the coin imitates that of the official coin (Pfennig) produced by entities having the right to mint coins, such as cities, bishoprics, counties, abbeys, and other small territories.

Imitation Type and Entity	Catalogue Number	Estimated Date	Number of Coins	Corresponding Figure
Pfalz-Veldenz, IAP–Johann August of Palatinate-Lützelstein–Pfalz-Veldenz county	104.F	1598 to 1611	12	Figure 1d
St Gallen City	195.F	2d. half 16th c	7	Figure 1f
Chur PEC–Peter II Rascher- Chur Bishopric	188.F	1581–1601	3	
Strasbourg City	151.F	16th–begin. 17th c.	2	Figure 1e
Marquard von Hattstein–Speyer bishopric	133.F	1560–1581	2	
Ludwig II of Stolberg-Königstein–County	135.F	1535–1574	1	
Wolfgang von Dalberg–Mainz bishopric	60.F	1582–1601	1	
Otto von Salm-Kirburg–Salm county	118.F	1548–1607	1	
Johann VII. von Schönenberg	160.F	1581–1590	1	
Other (Stolberg VLG, Zweibrücken)	146.F, 112.F		2	
Unidentified			6	

2.2. Painting Forgery Dating by ^{14}C AMS

^{14}C AMS was applied to the study of alleged 19th–20th century paintings. Impressionist, Pointillist, Expressionist, Abstract, and Contemporary paintings were selected. Four of them were seized by police after the discovery of a workshop, and another one came from a private collection. For confidentiality reasons, details on the alleged artists cannot be disclosed.

Different materials were sampled (Table 2): wood from the stretchers, fibers from the canvases, and paint (Figure 2). Fibers were previously identified as natural fibers under the microscope [12]. The paint sample was analyzed by X-ray diffraction (XRD), showing the presence of $BaSO_4$, ZnO, and $CaCO_3$-containing pigments in an organic binder.

Table 2. Paintings and materials selected for ^{14}C dating: wood from stretcher (W), fiber from canvas (F), paint (P).

Painting [1]	Estimated Date	Samples
Impressionist	Before 1945	W, F
Post-Impressionist	Before 1940	W, F
Expressionist	Before 1950	F
Abstract	~1970s	W, F, P
Contemporary	~1990s	W, F

[1] For confidentiality reasons, it is not possible to provide detailed information on the paintings.

Figure 2. (**Left**): samples of wood, canvas, and paint. (**Right**): details of the paint layer.

For radiocarbon dating, fiber and wood samples were pretreated with acid-base-acid washes, and the paint sample was only pretreated with acid due to its small size. Samples were dried under vacuum at 60 °C and then placed in quartz tubes with excess CuO and Ag. The quartz tubes were sealed under vacuum (5×10^{-6} mbar) and heated at 850 °C for 5 h. CO_2 gas was produced and separated from H_2O using a dry-ice/alcohol trap

(−78 °C) [23]. CO_2 samples were then reduced to graphite targets by hydrogen over an iron catalyst. Carbon isotopes were measured with the AMS LMC14/ARTEMIS facility (Saclay, France) [24]. The ^{14}C contents were converted into calendar years with the OxCal calibration program [25], using the Intcal20 atmospheric curve [26] for pre-bomb ages (i.e., before 1950) and the Bomb13 NH1 post-bomb atmospheric curve [27] for the most recent decades (i.e., after 1950).

3. Results

3.1. Counterfeit Coins

The compositions, in silver and copper, of the counterfeit coins and their official counterparts are presented in Figure 3. Two main groups are observed. The official coins contain between 20% and 42% of silver, whereas the counterfeit coins show a low silver content.

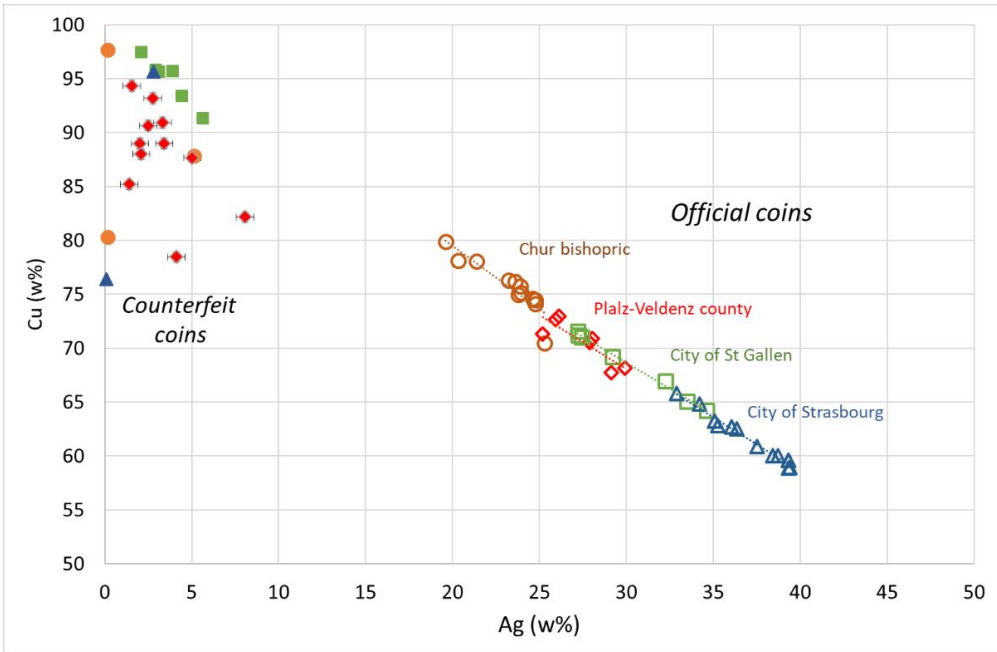


Figure 3. Silver vs. copper contents (in wt%) for selected coins of the Preuschdorf hoard coming from the cities of Strasbourg and St. Gallen, the county of Pfalz-Veldenz, and the bishopric of Chur. Open symbols correspond to official coins, and filled symbols correspond to counterfeit coins.

For the official coins, two major elements are present: silver and copper. The mean silver concentration for the coins issued by the city of Strasbourg is 37 ± 2%; this high content is in accordance with the official finesses of 375/1000 set by the Imperial minting ordinance (Reichsmünzordnung) of the Holy Roman Empire in 1559 [28]. In contrast, the coins issued by the bishopric of Chur in the Swiss Confederation have a low silver content (23 ± 3%), reflecting a production of poor quality and known to be struck for exportation [29]. The coins produced by the city of St. Gallen and the county of Palatinate (Pfalz-Veldenz) show intermediate silver contents of 30 ± 3% and 27.5 ± 1.8%, respectively.

The counterfeit coins are characterized by their low content in silver (less than 8%) and high content in copper (from 76 to 98 wt%). The values are scattered due to the variable presence of other elements such as mercury, zinc, and tin. Mercury is detected in the counterfeit coins imitating those of the county of Palatinate (Pfalz-Veldenz, (catalogue number 104.F; see Table 1)), in one coin copying that of the city of Strasbourg (151.F), and in another copying that of the bishopric of Chur (188.F). Mercury is clearly correlated to silver (Figure 4), suggesting the presence of an Ag-Hg alloy at the surface of the coin. The mean ratio between silver and mercury is about 1, corresponding to a layer composed of 50% Ag and 50% Hg. However, a large scatter is observed from one coin to another, with

ratios from 1.6 to 0.3, corresponding to compositions of ~62% Ag and 38% Hg to 25% Ag and 75% Hg.

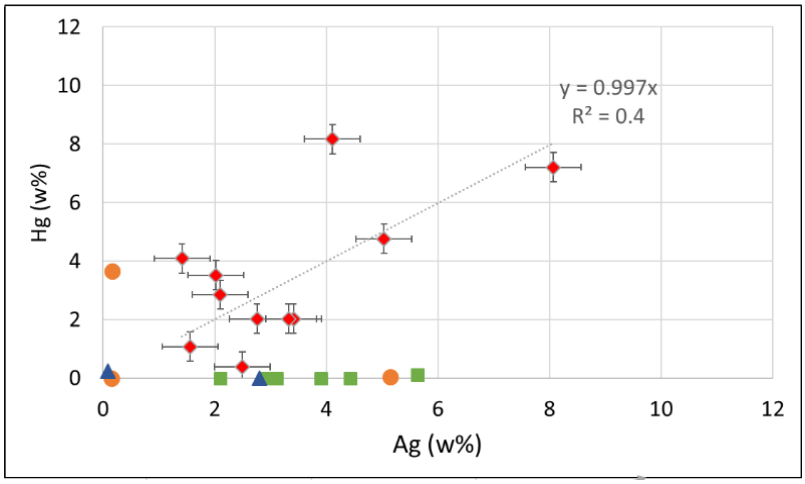


Figure 4. Silver vs. mercury contents (in wt%) for the counterfeit coins of the Preuschdorf hoard coming from the cities of Strasbourg (blue) and St. Gallen (green), the county of Pfalz-Veldenz (red), and the bishopric of Chur (orange).

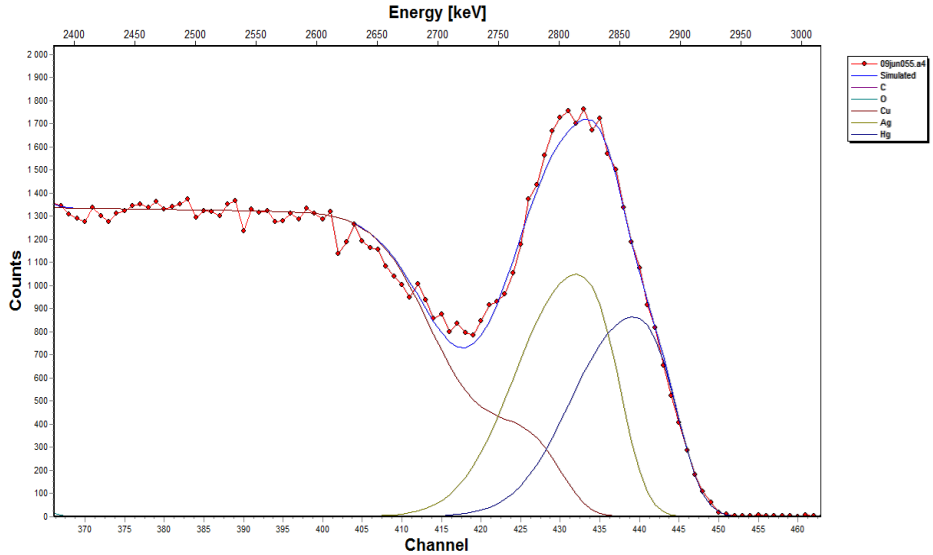
The RBS spectrum of a coin containing mercury is presented in Figure 5a. The SIMNRA simulation [22] indicates that silver and mercury are together at the surface of the coin, forming a 1–2 µm thick layer of 50 Hg wt% and 50 Ag wt%. A composition of 27 Hg wt% and 73 Ag wt% was also found for another coin [20]. Both compositions are in agreement with the PIXE results and confirm the presence of a silver-mercury layer at the surface of some counterfeit coins, mostly the imitations from the county of Palatinate (Pfalz-Veldenz, 104.F). For comparison, Figure 5b shows the RBS spectrum of an official coin made of a homogeneous silver-copper alloy.

None of the other 24 counterfeit coins contain a significant amount of mercury. These are the imitations of the city of St. Gallen (195.F), the bishopric of Speyer (133.F), the bishopric of Mainz (60.F), the county of Stolberg (135.F), the county of Salm (118.F), the two other specimens of the bishopric of Chur (188.F), the other specimen of the city of Strasbourg (151.F), as well as the other identified (160.F, 146.F, 112.F) and unidentified coins. These coins were not well preserved, and observation under the microscope was necessary to interpret the low content in silver (0.2 to 5.6 %). This result is due to a thin layer of pure silver coated at the surface of the coin, and the apparent percentage of silver determined by PIXE is related to the thickness of the silver layer. The cores of these counterfeit coins are composed of copper or brass (copper and zinc).

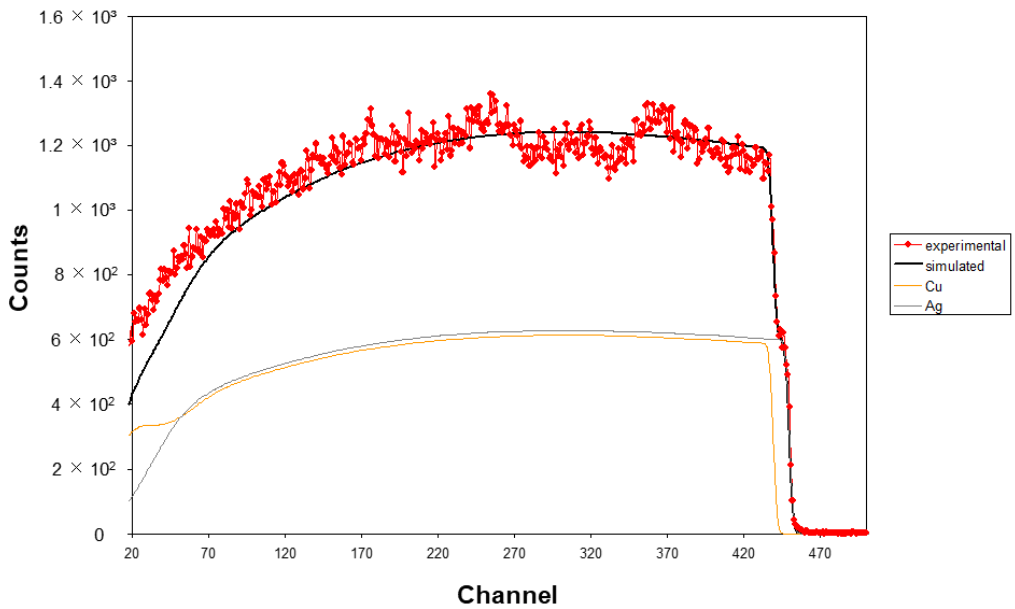
In summary, two counterfeiting processes were identified by IBA: (a) mercury silvering (also called amalgam silvering or fire silvering) of a copper core for 14 coins, 12 of which come from the county of Palatinate (Pfalz-Veldenz, 104.F), as well as (b) a thin layer of pure silver coated on a brass, bronze, or copper core for 24 coins. The application of pure silver can be achieved by coating a thin silver foil or by electrochemical replacement. The latter process is a plating technique that uses the electro-differential between the solution and the metal to be plated; copper blanks are immersed in a solution containing silver flakes, salt (NaCl), and wine lees (potassium bitartrate, $KC_4H_5O_6$) [30]. After a few hours, a thin layer of silver is deposited on the substrate [14], and the coins are ready to be struck.

The discovery of 14 mercury silvered coins is an important finding since there are very few known examples from before the 17th century [31,32]. The earliest examples are the forged Iranian dirhem dating from the 9th to 10th centuries [33], four pennies of the 13th century [34], and one coin of the 15th century [35]. The imitation (135.F) of Ludwig II of Stolberg-Königstein (1535–1574) was wrongly attributed to the group of mercury-silvered coins in a previous publication [18]. As a result, the identification

of one counterfeit coin (188.F) of Peter II Rascher (1581–1601), the bishop of Chur, and 12 counterfeit coins (104.F) of Johann August of Palatinate-Lützelstein (1598 to 1611), the count of Pfalz-Veldenz, constitutes a significant contribution to our knowledge of ancient counterfeiting practices [36] and the amalgam silvering process.



(**a**)



(**b**)

Figure 5. The 3 MeV proton Rutherford backscattering spectrometry (RBS) spectrum of a counterfeit coin (**a**) and that of an official coin (**b**). For the counterfeit coin, RBS shows a silver-mercury layer on a copper substrate, whereas for the official coin, RBS shows a homogeneous silver-copper alloy.

3.2. Art Forgery Dating

Radiocarbon dating results, obtained for the wooden stretcher of four paintings, are presented in Table 3 and Figure 6.

Table 3. ^{14}C accelerator mass spectrometry (AMS) dating results of the wood samples taken from the painting stretchers. Uncalibrated radiocarbon ages are reported in years Before Present (BP), i.e., in years before 1950. Calibrated dates are obtained by calibrating the radiocarbon ages using the Intcal20 atmospheric curve [26] (Figure 7).

Painting	Radiocarbon Age (BP)	Calibrated Dates (95.4%)	AMS Laboratory Number
Impressionist	30 ± 23	1697–1724 (29.6%) 1812–1836 (28.4%) 1880–1911 (37.5%)	SacA57262
Pointillist	135 ± 23	1675–1744 (26.5%) 1750–1765 (4.2%) 1798–1942 (64.7%)	SacA57264
Expressionist	No sample	-	-
Abstract	220 ± 21	1644–1681 (42.1%) 1739–1753 (5.4%) 1762–1800 (45.5%) 1939– . . . (2.6%)	SacA57267
Contemporary	120 ± 21	1683–1735 (24.9%) 1803–1938 (70.6%)	SacA57269

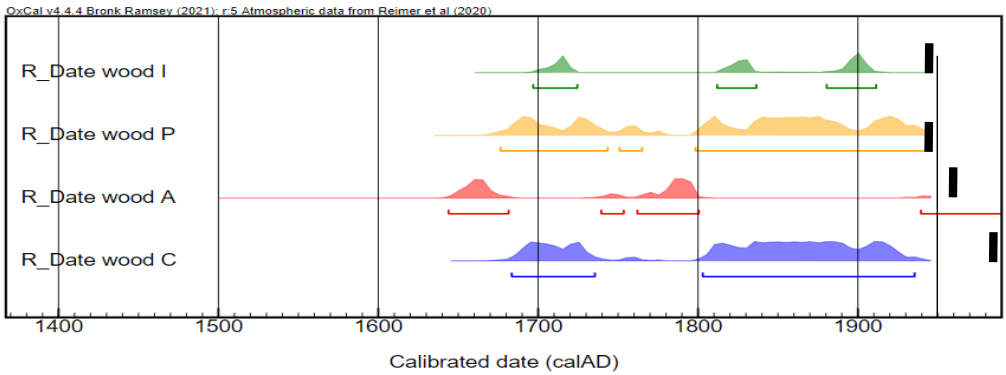


Figure 6. Radiocarbon calibrated dates (shown in calAD) of the wood samples, taken from the stretchers of the Impressionist (I), Pointillist (P), Abstract (A), and Contemporary (C) paintings. The black bars indicate the death of the alleged artists. The radiocarbon calibrated dates were obtained using the atmospheric calibration curve Intcal20 [26].

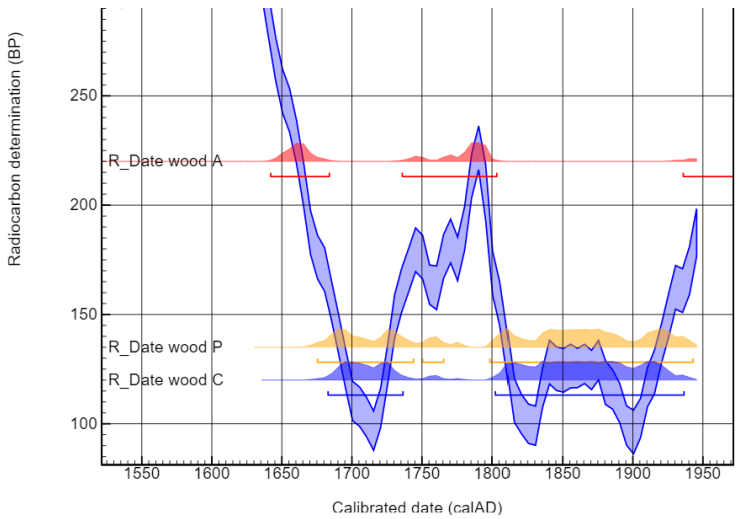


Figure 7. Calibration of the wood samples taken from the painting stretchers. The blue curve is the atmospheric calibration curve (Intcal20) used to calibrate ^{14}C dates before 1950 [26].

All the dates are between the 17th century and 1950, with variable distributions. The ranges are very large due to the shape of the calibration curve for these times (Figure 7). These dates, obtained for the wood, correspond to the time when the trees were still standing and incorporated ^{14}C. To know the time when the trees were cut down, it is necessary to have samples containing sapwood, which is not the case here. As a result, the dated piece of wood can be older than the date of the tree felling (known as the "old wood effect"), making it difficult to estimate the stretcher manufacture. The most recent year recorded by ^{14}C for the stretcher wood is 1911, 1942, 1950, and 1938 for the Impressionist, Pointillist, Abstract, and Contemporary paintings, respectively. These dates are coherent with the lifetime of the alleged artists. However, due to the wide distribution, as well as the old wood effect, which also includes the unknown duration between the felling of the trees and the stretcher manufacture, it is not possible to conclude the date at which the paintings were executed.

Radiocarbon dating results obtained for the canvas of four paintings and for the paint of one painting are presented in Table 4 and Figure 8.

Table 4. ^{14}C Fraction Modern ($F^{14}C$) results for the fibers taken from the painting canvases and for a sample of paint taken from the white paint of the Abstract painting. $F^{14}C$ is the unit used for post-bomb samples. Radiocarbon dates were calibrated using the Bomb13 NH1 post-bomb atmospheric curve [27] (Figure 8). See text for details.

Painting	$F^{14}C$	Calibrated Dates (95.4%)	AMS Lab Number
Impressionist	1.0859 ± 0.0027	1957 & 2000–2003	SacA57263
Pointillist	1.0560 ± 0.0027	1956–1957 & 2004–2010	SacA57265
Expressionist	1.1646 ± 0.0027	1957–1959 &1987–1990	SacA64025
Abstract, canvas	1.0301 ± 0.0027	1955–1956 & 2012–2015	SacA57268
Abstract, paint	1.0094 ± 0.0027	1954–1955 & 2017– . . .	SacA57275
Contemporary	Failed	-	-

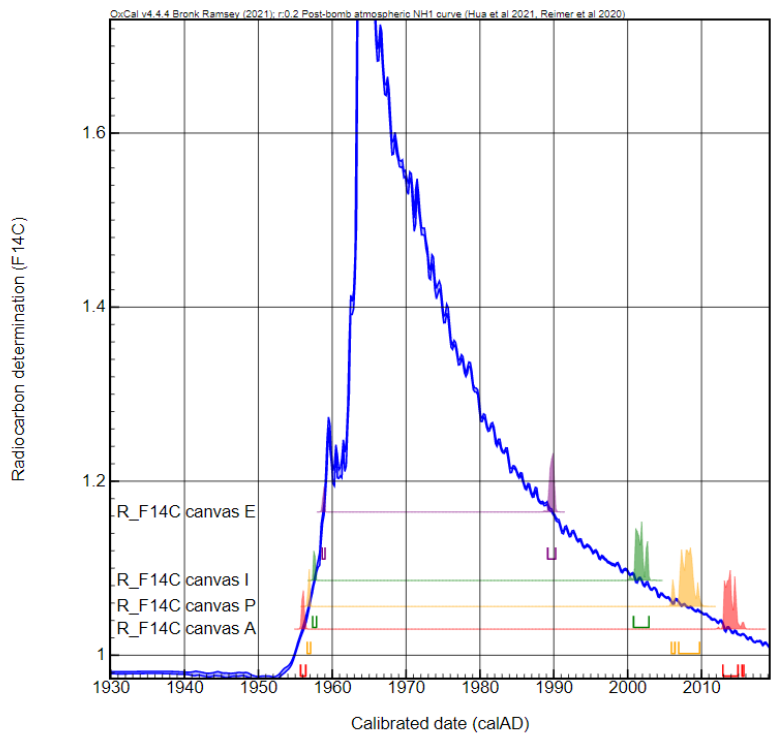


Figure 8. Calibration of the fiber samples taken from the canvas of the Impressionist, Pointillist, Abstract, and Expressionist paintings. The blue curve is the post-bomb atmospheric calibration curve (Bomb13 NH1) used to calibrate ^{14}C dates after 1950 [25–27]. See text for details.

The ^{14}C Fraction Modern ($F^{14}C$) indicates the proportion of radiocarbon atoms in a sample as compared to samples that were modern in 1950. In the late 1950s and 1960s the ^{14}C concentration in the atmosphere almost doubled due to the atmospheric nuclear weapon tests. Thus, a $F^{14}C$ value higher than 1 indicates "post-bomb" samples. After the atmospheric nuclear test ban treaty in 1963, the ^{14}C content decreased due to its dilution in the atmosphere [37]. This event, including artificial ^{14}C production and decrease, referred to as the "bomb peak", is used as the calibration curve for radiocarbon dating of recent samples.

The ^{14}C content ($F^{14}C$) of all the fibers is higher than 1, showing, unambiguously, that the canvas fabrics originate from plants that grew at the time when ^{14}C was in excess in the atmosphere. After calibration, two solutions are determined for each radiocarbon result due to the shape of the calibration curve (Figure 8). The dates, corresponding to the plant harvesting, are the following: 1957 or 2000–2003, 1956–1957 or 2004–2010, 1957–1959 or 1987–1990, and 1955–1956 or 2012–2015 for the Impressionist, Pointillist, Expressionist, and Abstract paintings, respectively. For the first three paintings, both solutions are after the death of the alleged artists in the 1940s-early 1950s. These results demonstrate that these paintings are forgeries performed in the years 1956–1957 or 2000–2010, for the Impressionist and Pointillist specimens, and in the years 1957–1959 or 1987–1990 for the Expressionist one. For the Abstract painting, the interpretation of the results—1955–1956 and 2012–2015—is less straightforward since the alleged artist died in the 70s. The second solution is after the death of the artist, but the first one is 15–20 years earlier. It cannot be excluded that an artist may keep untouched canvases in his/her workshop for one or two decades, even if the typical storage duration is between two and five years [38]. A similar result was obtained for the paint sample (Table 4); however, it is less common to preserve a tube of paint for such a long time. The latter result suggests that the Abstract painting is also a fake. For the Contemporary painting, the canvas dating failed due to the massive presence of glue still embedded in the fibers even after a strong cleaning treatment.

Among the five paintings examined, ^{14}C dating shows that three of them are definitely fake, one is most likely fake, and one remains undetermined.

4. Conclusions

Ion beam analysis (IBA) and ^{14}C accelerator mass spectrometry (AMS) are methods of choice to analyze ancient artefacts and works of art since they are minimally invasive. Both techniques can also contribute to the study of counterfeiting and art forgery [39]. In this paper, the metallurgical processes used to produce silver counterfeit coins in the 16th century were studied in detail, revealing the presence of 14 coins silvered with mercury. This discovery is an important finding since there are very few known examples before the 17th century. Five paintings were radiocarbon dated. Using the bomb peak calibration curve, it was unambiguously demonstrated that three paintings, alleged to be of the beginning of the 20th century, are forgeries made after 1956. It is suggested that another painting alleged to be painted in the 1970s could be a more recent forgery.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to restricted access to databases. The IBA archived dataset is managed by the AGLAE group at the Centre de Recherche et de Restauration des Musées de France (Paris, France). The ^{14}C AMS archived dataset is managed by the LMC14 Laboratory (Saclay, France).

Acknowledgments: The author wishes to thank Dominique Robcis (C2RMF), Elise Alloin and Anaïs Vigneron (Archéologie Alsace), Ulrich Klein (numismatist) and the AGLAE group (C2RMF, Paris, France) for their invaluable contribution to the study of the Preuschdorf hoard and her current colleagues of the LMC14 laboratory for the painting sample preparation, ^{14}C measurements and fruitful discussion. Many thanks also to Estelle Itié and Ilenia Cassan for their confidence in the study of one of the paintings presented here. This is LSCE contribution number 7901.

Conflicts of Interest: The authors declare no conflict of interest.

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