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20 YEARS OF AMS ^{14}C DATING USING THE ARTEMIS FACILITY AT THE LMC14 NATIONAL LABORATORY: REVIEW OF SERVICE AND RESEARCH ACTIVITIES

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ABSTRACT. In 2001, five French public organizations (CNRS, CEA, IRD, IRSN, and the Ministère de la Culture) signed an agreement to purchase a new accelerator mass spectrometer to provide radiocarbon dating services at the national level. The Laboratoire de Mesure du Carbone 14 (LMC14) was set up in Saclay (France) around ARTEMIS, an AMS system based on a 3MV Pelletron from NEC and installed in early 2003. In 2015, the LMC14 joined the Laboratoire des Sciences du Climat et de l'Environnement, making it possible to develop research projects in addition to the service activity and since 2021, the LMC14 has been a member of the IAEA Collaborating Centre "Atoms for Heritage" at the Université Paris-Saclay. Since 2003, 70,000 samples have been measured. Two-thirds of the samples have been prepared on site and one-third in two associated laboratories in Paris and Lyon. Over the past years, the LMC14 has participated in several international inter-comparisons and has continuously improved its capabilities by developing new protocols for preparation and measurement. In this paper, the radiocarbon dating services of the last 20 years for research institutions, museums and environmental monitoring are reviewed and recent results from environmental and archaeological research programs are highlighted.

KEYWORDS: archaeometallurgy, ARTEMIS, environment, LMC14, radiocarbon AMS dating.

INTRODUCTION

Twenty-two years ago, five public organizations (Table 1) joined forces to create a new accelerator mass spectrometry (AMS) facility to meet the growing demand for ^{14}C dating in French research laboratories. In 2003, the Laboratoire de Mesure du Carbone 14 (LMC14) was set up in Saclay around ARTEMIS (French acronym for Accélérateur pour la Recherche en sciences de la Terre, Environnement, Muséologie Installé à Saclay), an AMS system based on a 3MV Pelletron purchased from National Electrostatics Corporation (NEC, Middleton, Wisconsin, USA). This new equipment replaced a multi-isotope AMS system that had been operating in Gif-sur-Yvette since the early 1980s (Arnold et al. 1987) and the LMC14 benefited from the great experience of the Centre des Faibles Radioactivités when setting up the new preparation laboratories. The official inauguration of the National laboratory took place on 8th of April 2004. The year 2003 was dedicated to the commissioning of the AMS and the preparation laboratories. After a testing period, the first experiment was registered in 2004 and routine operation began immediately. The first 99 measurements were dedicated to oxalic acid (OX1), blanks (IAEA-C1, coral, wood), and references for carbonates (FIRI C, IAEA-C2). The first unknown sample was composed of a group of foraminifera from the Atlantic Ocean (SacA101). In a second step, references of organic matters (FIRI-A, -B, -D, -E, -G, -H) were tested and the first unknown organic sample was a modern cereal (SacA309). Accuracy, reproducibility, and blanks obtained in these early years were reported by Cottereau et al. in 2007 and then by Moreau et al. in 2013 and 2020 (Table S1).

From 2004 to 2014, the missions of the LMC14 were restricted to service activities due to the administrative status of the laboratory. In order to fully develop research projects, the LMC14 joined the LSCE research laboratory in Saclay (Laboratoire des Sciences du Climat et de

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Table 1 LMC14 supervisory bodies and proportion of ^{14}C measurements dedicated to each of them.

Organization/institutions	Acronym	Status	Main area(s) of ^{14}C application	^{14}C measurements (%)
Centre national de la recherché scientifique *National Centre for Scientific Research*	CNRS	Research institution	Environmental sciences, Humanities and Social Sciences	40
Commissariat à l'énergie atomique et aux énergies alternatives *Alternative Energies and Atomic Energy Commission*	CEA	Research institution	Sciences of the climate and environment	25
Ministère de la Culture *Ministry of culture*	MC	Ministry	Museums, historical monuments, regional archaeology services	18
Institut de recherche pour le développement *Institute for Sustainable Development*	IRD	Research institution	Environmental sciences and archaeology (intertropical and Mediterranean zones)	8.5
Institut de radioprotection et sûreté nucléaire *Institute for Radiological Protection and Nuclear Safety*	IRSN	Institution expert in nuclear and radiation risks	Environmental monitoring	8.5

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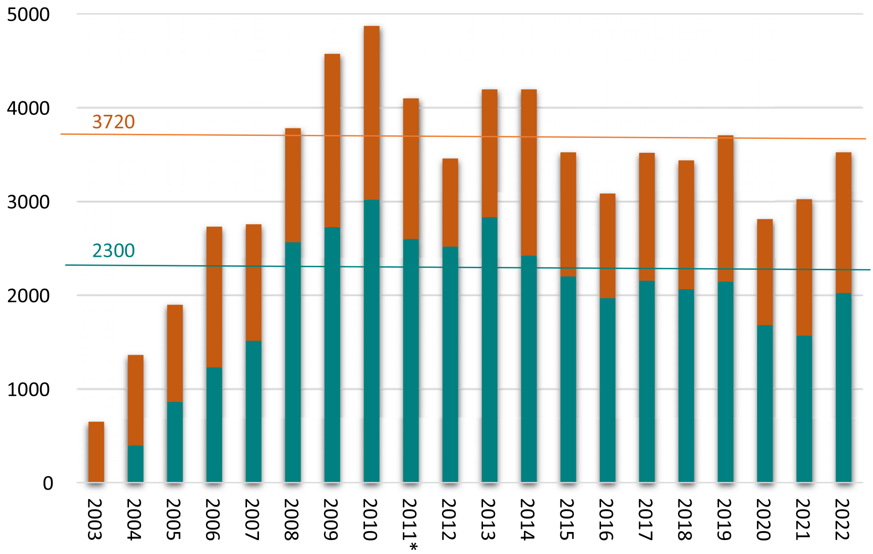

Figure 1 Number of samples ^{14}C measured per year at LMC14 (Saclay, France, AMS reference laboratory SacA): in blue, for service to the five supervisory bodies and, in orange, tests, accompanying samples (primary standards, blank samples, known-age samples) and R&D samples (experimental development and research projects). Lines represent mean values from 2008 to 2022. *The year 2011 is estimated.

l'Environnement). Since 2021, LMC14 has been a member of the IAEA Collaborating Center "Atoms for Heritage" of the University of Paris-Saclay.

Between 2004 and April 2023, the ^{14}C content in 70,000 samples was measured (Figure 1). Preparation and measurement capacities have increased over the years, with up to 4874 samples produced in 2010, and between 3090 and 4200 samples per year from 2011 to 2019. 2020 and 2021 were impacted by lockdowns and lack of samples due to the Covid-19 pandemic. The year 2022 returned to an optimal activity with 3500 samples analyzed.

In this paper, 20 years of radiocarbon dating services for research institutions, museums and environmental monitoring are briefly described and results from recent archaeological and environmental research programs are reviewed. Long-term collaborative projects on rock art studies, the chronology of Pharaonic Egypt, architectural use of iron, and lead white production are presented.

MATERIALS AND METHODS

Main Equipment and Procedures

In the last 20 years, the facilities of the LMC14 have evolved and diversified to provide a large range of services. Today, the preparation laboratories are equipped with the following devices:

- Two semi-automated in-house systems for collecting CO_2 from 7 organic matter samples (after combustion) and/or 5 carbonate samples (after acid hydrolysis) per day (Dumoulin et al. 2017a);

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- Three manual glass vacuum systems occasionally used for collecting CO_2 from 7 specific samples (lead white, etc.), to seal 5 quartz tubes or to extract DIC from water samples under helium flux, respectively;
- An Elemental Analyzer coupled to an automated in-house manifold for collecting CO_2 from modern samples (F14C>1). The CO_2 collection line can be turned into a sealed tube graphitization line;
- A laser ablation facility (laser 1064 nm and air-tight cell) for producing and collecting CO_2 from iron samples (in progress);
- Two in-house automatic systems to graphitize 24 CO_2 samples daily (12 reactors of 18 and 12 mL); and
- A specific system dedicated to small samples (between 10 and 150 µg), consisting of 3 reduction lines with a small inner volume of 6 mL (Delqué-Količ et al. 2013a, 2013b).

$^{14}C/^{12}C$ and $^{13}C/^{12}C$ ratios are determined by AMS using a 3MV NEC Pelletron equipped with two SNICS solid sources of 134 and 40 targets (Cottereau et al. 2007). One of the two sources is being modified to process gas samples. The $^{12}C^{3+}$ and $^{13}C^{3+}$ isotopes are measured in two offset Faraday cups at the exit of the 110° magnetic analyzer. The $^{14}C^{3+}$ are counted in an ionization chamber detector situated at the end of the beamline. The accelerator runs unattended after tuning and each sample is measured 8 to 12 times. The NEC's *abc* analysis code is used to analyze offline the data produced by the AMS system. This software calculates the average $^{14}C/^{12}C$ ratio, the average $^{13}C/^{12}C$ ratio, the $\delta^{13}C$ value for each sample and normalizes, after the removal of the anomalous measurements, the $^{14}C/^{12}C$ ratios of the unknown samples to the simultaneously measured standard sample ratio. These data are then transferred to an in-house database to determine the ^{14}C age and the associated error. Since 2012, the samples have been registered and tracked along with the process (pretreatment, CO_2 collection, graphitization, carbon isotope measurement, age in BP and $\delta^{13}C$ value) in an Access database developed in-house. Quality control procedures are described in Moreau et al. (2020).

Two associated laboratories—the Centre de recherche et restauration des musées de France (C2RMF) in Paris and the Centre de datation par le radiocarbon (CDRC) in Lyon—are associated to the LMC14 for the preparation of museum and archaeological samples (Billard 2008; Richardin et al. 2010). Two other laboratories—the National Museum of Natural History (MNHN) in Paris and Géosciences in Paris-Saclay (GEOPS)—occasionally provide CO_2 gases.

Samples

Since 2004, 69 850 samples have been handled in total (Figure 1). ~45,000 unknown samples have been analyzed for service purposes and research projects and ~25,000 samples have been measured as accompanying samples (primary standards, blank samples, known-age samples) or for tests.

For service purposes, the three laboratories (LMC14, C2RMF, CDRC) together prepare all the conventional materials (wood, charcoal, sediments, bones, ivory, hair, textile, cellulose, etc.) (Table 2). For research projects or for specific applications, the LMC14 has continuously improved its capabilities by implementing new devices and developing new protocols. Dedicated preparation protocols have been developed for water (Dumoulin et al.

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Table 2 Materials prepared for service (X) at the LMC14 and the associated preparation laboratories (C2RMF and CDRC) and for collaborative research projects only (*).

	Charcoal	Wood bulk	Cellulose	Biocarbonate /foram/ shell speleothem	Sediment/ soil/peat	Bone/tooth collagen	Water DIC	Hair/ keratin	Textile	Leather	Ivory	Lead white	Iron/ steel	Pigment	Aerosol	Mortar	Pollen	Charred residues on potsherds
C2RMF (Paris)	X	X	X			X		X	X	X	X							
CDRC (Lyon)	X	X	X	X	X	X		X	X	X	X					*	X	X
LMC14 (Saclay)	X	X	*	X	X	*	*		X	X		*	*	*	X	*	X	X

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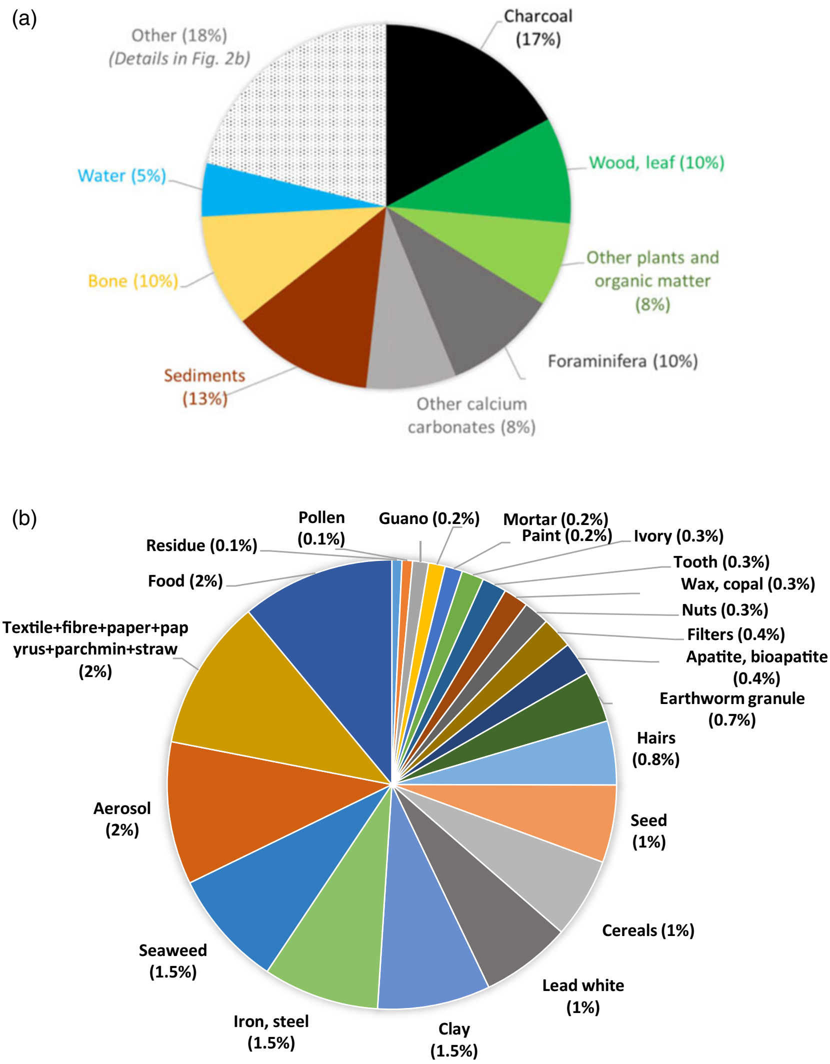

Figure 2 Types of samples ^{14}C analyzed at the LMC14. (a) Major materials (>5%) represent 82% of the dated samples. (b) Many varieties of minor materials are also handled (<5%). All the types of materials are prepared at the LMC14 except bones and ivory prepared at C2RMF and CDRC.

2013), bone (Dumoulin et al. 2017b), iron (Leroy et al. 2015a), lead carbonates (Beck et al. 2019; Messager et al. 2020), oxalates (Dumoulin et al. 2020), and more recently mortars (Hayen et al. 2017; Moreau et al., submitted). The LMC14 has also participated in several international inter-comparisons (Cuzange et al. 2007; Quiles et al. 2014; Scott et al. 2017; Moreau et al. 2020) and more recently in the GIRI and MODIS2 campaigns (Scott et al. 2019; Dumoulin et al. accepted; Moreau et al. submitted).

Figure 2 presents the diversity of substances measured at the LMC14. Unsurprisingly, charcoals, wood and plants are the most frequently dated samples (35%), followed by calcium carbonate-based materials such as foraminifera, speleothems, shells (18%)

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(Figure 2a). Sediment, peat, and soils account for 13% but this proportion is increasing continuously as for water (5%), which has been prepared only since 2012. Bones prepared in the associated laboratories represent 10% of the measurements. Many minor types of samples are also handled in response to users' requests or for research purposes (Figure 2b).

ACHIEVEMENTS

Service Activity

Almost 40,000 samples have been measured for the service to the French research laboratories of the supervisory bodies (CNRS, CEA, IRD, IRSN, and MC), corresponding to 2000–3000 samples per year over the 12 years before Covid-19 (Figure 1).

Each institution organizes the access to ^{14}C measurements according to their own selection procedure. After acceptance of the proposal, the cost per sample for the user varies according to the institution, from free of charge to a reduced fee. About 50% of ^{14}C measurements are dedicated to environmental studies (past climate, carbon cycle, ocean studies, evolution of eco-hydrosystems and soils, sustainable development, etc.), 40% for cultural heritage applications (archaeological sites, museums, preventive and academic archaeological excavations) and 10% for radiological monitoring. Two-thirds of the dated samples are prepared on site using the facilities described above and one-third is prepared in the two associated laboratories, C2RMF and CDRC.

Results delivered to the users are the ^{14}C content in pMC and ^{14}C age in BP with their respective uncertainties and $\delta^{13}C$ values for information. For environmental monitoring, the results are expressed in Bq per kg of carbon. The users publish the results on their own. The publication must include the "SacA" AMS laboratory code and a mention of the "ARTEMIS programme" in the Acknowledgments section. However, when specific development is required, a collaborative project between LMC14 and the users can be set up, leading to a joint publication of the results.

Research Projects

Research projects are conducted internally or in collaboration with external partners. They are mainly in the field of archaeology and art history and cover the period from the Palaeolithic to contemporary times. Long-term research programmes have been dedicated to the chronology of Pharaonic Egypt (Bronk Ramsey et al. 2010; Quiles et al. 2013, 2021a, 2021b) and rock art studies.

Concerning the latter topic, the LMC14 has long experience in dating rock art. Drawings and paintings of many decorated Paleolithic caves located in France (Chauvet, Cosquer, Grottes aux points, les deux ouvertures, Villars), in Spain (Nerja) and more recently of rock shelters in Italy and in Africa have been investigated (Genty et al. 2011; Beck et al. 2013; Valladas et al. 2013, 2017; Quiles et al. 2016; Dumoulin et al. 2020; Palmerini et al. 2021; Heimlich et al. 2022; Pons-Branchu et al. 2022). For the Chauvet cave (France), more than 250 AMS-radiocarbon dates have been performed by different laboratories since its discovery in 1995 (Quiles et al. 2016). Among them, 111 were obtained using the ARTEMIS AMS facility after sample preparation at the LMC14 or in sister laboratories (LSCE [Gif-sur-Yvette], MNHN [Paris], CDRC [Lyon]). ^{14}C measurements were carried out on 19 drawings and 11 charcoal marks and on 17 humic fractions. To document human occupation, more than 160 charcoals from the cave floor were analyzed for dating and intercomparison (Cuzange et al. 2007; Quiles et al. 2014), of which 64 were measured using the ARTEMIS facility. Based on the radiocarbon dates, a high-precision chronological model was developed showing that there were two distinct periods of human activity in the cave, one from 37 to 33,500 yr ago, and the other from 31 to 28,000 yr ago (Quiles et al. 2016).

For the Cosquer cave, 41 samples have been dated since its discovery including 25 ^{14}C analyses by ARTEMIS. Charcoal samples taken from animal representations, hand stencils and signs were dated. The results showed that the Cosquer Cave was visited by prehistoric people over a long period, between 33,000 and 20,000 cal BP and that the oldest decoration period falls in the same time range as the Chauvet Cave's latest occupation (Valladas et al. 2017). In the Villars cave, five radiocarbon dates were obtained from torch marks, sampled after analyzing 22 representations in situ by X-ray fluorescence (Beck et al. 2013). The dates range from 22.1 to 17.1 cal ka BP. These three sites present varied examples of decorated caves, showing the longevity over at least 20 centuries and the spatial distribution of rock art in France.

Since the last decade, one of the objectives of the LMC14 has also been to explore the possibility of extending the range of datable materials by handling non-conventional materials such as iron and lead carbonates. These approaches are detailed in the following sections.

Iron

Dating archaeological ferrous objects is essential to investigate the history of iron and steel. Until modern times, ore was reduced to metal in furnaces using charcoal. During the reduction process, part of the charcoal carbon diffused into the metal and was incorporated into the structure of the ferrous alloy in the form of iron carbide Fe_3C (cementite). By extracting carbon from the metal matrix, it is possible to date the tree from which the charcoal was obtained and thus estimate the age of the metal itself. However, due to the complex and heterogeneous structure of ferrous alloys, it is necessary to fully characterize the samples prior to ^{14}C dating. For that purpose a close collaboration was established 10 years ago between the LMC14 and the Laboratoire Archéomatériaux et Prévision de l'Altération (LAPA-IRAMAT, NIMBE, CEA, CNRS, Université Paris-Saclay). A rigorous analytical framework was defined not only to avoid sources of contamination due to the presence of corrosion zones and recycled steel, but also to determine the most carburized sectors and thus the most relevant ones for the analysis by carbon 14 (Leroy et al. 2015a).

The use of this protocol in the study of monuments and archaeological sites is already contributing to extending our knowledge on the use of iron in the construction of Gothic cathedrals (Leroy et al. 2015b; L'Héritier et al. 2023), the diffusion of ferrous alloys in the Iron Age and Antiquity (Delqué-Kolic et al. 2017; Berranger et al. 2021; Berthaut-Clarac et al. 2022) and the role of iron in the development of the Khmer Empire (Leroy et al. 2015b, 2018, 2020). A novel approach is being developed for in situ sampling of iron armatures of bronze Khmer statues (Leroy et al. 2021). The protocol is described in Dumoulin et al. (accepted).

Lead Carbonate and Lead White

Lead carbonates were employed as cosmetics, eye remedies and white pigment (known as lead white) from the 4th century BC to the beginning of the 20th century. Recently, it has been

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demonstrated that organic carbon was incorporated during their manufacturing, making ^{14}C dating possible (Beck et al. 2018; Hendriks et al. 2019; Messager et al. 2022). Protocols based on the thermal decomposition of lead carbonates have been developed at LMC14 in order to avoid contamination by dead carbon from calcite or other carbonate compounds potentially mixed with them (Beck et al. 2019; Messager et al. 2020; Dumoulin et al. accepted).

^{14}C analysis of Egyptian and Greek cosmetics showed that phosgenite ($Pb_2(CO_3)Cl_2$) was synthesised as early as the middle of the 2nd millennium BC and cerussite ($PbCO_3$) from the 4th century BC (Beck et al. 2018). ^{14}C dates were also obtained from lead carbonate compounds used as eye collyria during the Roman period in the 2nd century AD (Messager et al. 2021). Medieval mural paintings were also investigated. Dating results attested the use of lead white for the decoration of private homes such as the castle of Germolles (France) at the end of the 13th century or larger public buildings such as churches, in Angers at the beginning of the 12th century (Duchene in prep.) or later in Fribourg in the 15th century (Beck et al. 2020). Including papers published by ETH, Zurich, Switzerland (Hendrick et al. 2020; Sa et al. 2021), these studies provide direct evidence of the capacity of the radiocarbon method to directly date lead carbonates and extend the range of inorganic materials that are datable by this method.

More recent collaborative research projects have focused on societal issues such as the protection of cultural heritage (Hajdas et al. 2019). An illicit production of fake paintings was uncovered by radiocarbon dating in the context of a police investigation conducted by the French Central Office for the Fight against Illicit Trafficking in Cultural Property (OCBC) (Beck 2022; Beck et al. 2022b). Concerning museum objects, a controversy of more than a century about the attribution of the Flora bust to Leonardo da Vinci has also been definitively resolved after dating the object to the 19th century (Reiche et al. 2021; Beck et al. 2022a). Finally, the disaster of the fire of Notre-Dame de Paris on April 15, 2019, led to the creation of a vast scientific project in parallel with the restoration work. The unfortunate destruction miraculously spared part of the frame and made the wooden pieces and metallic structure accessible to the scientific community. In the context of a programmatic action, the LMC14 is involved in two projects. The first one investigates the role of iron in the construction of this monument (L'Héritier et al. 2023), following the studies carried out on numerous Gothic churches (see above). The second aims at implementing the ^{14}C recorded in the wooden beams at a yearly resolution in the next ^{14}C calibration curve and documenting past changes in ^{14}C production resulting either from changes in solar activity or supernovae (Daux et al. 2022).

In addition to cultural heritage applications, the LMC14 has been involved in various programs on environmental studies (Haddam et al. 2018; Pons-Branchu et al. 2018; Rapuc et al. 2019; Waelbroeck et al. 2019). In other cases, ^{14}C was used as a tracer to better understand the carbon cycle in water ecosystems such as lakes (Messager 2020; Thouret et al. 2021), rivers (Dumoulin et al. 2018; Pozzato et al. 2018), or oceans (Dumoulin et al. 2022). The isotopic analyses ($\delta^{13}C$, $\Delta^{14}C$) of the pore waters trapped in this sediment and of the sediment itself give valuable information to follow the fate of the organic matters of terrestrial or marine origin.

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CONCLUSION

The Laboratoire de Mesure du Carbone 14 has just celebrated its 22 years of existence and its 20 years of carbon 14 measurements for the French scientific community. After two years impacted by the Covid pandemic, the LMC14 has recovered its full production capacity with more than 3500 samples per year. The data produced contribute to research efforts in the fields of environmental studies—paleoclimatology, carbon cycle, ocean studies, evolution of eco-hydrosystems and soils, sustainable development—cultural heritage—for museums and archaeological services—as well as for environmental monitoring. Since 2015, the laboratory has been attached to the Laboratoire des Sciences du Climat et de l'Environnement, which enables it to carry out research projects in parallel with the service activities. Long-term research programs have been developed, among others, on the chronology of Pharaonic Egypt, prehistoric rock art, the use of iron in the medieval buildings, the manufacture of the lead white pigment, the protection of cultural heritage and the carbon cycle in water ecosystems. Since 2021, the LMC14 has been a member of the IAEA Collaborating Center "Atoms for Heritage" of the University of Paris-Saclay.

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SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit https://doi.org/10.1017/RDC.2023.23

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