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A look at the region

# Time Travel with Radiocarbon Dating

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*ABSTRACT*

Radiocarbon dating is a critical tool in archaeology and Quaternary Earth science, providing absolute age estimates. Established in 2004, the Czech Radiocarbon Laboratory (CRL) has advanced from radiometric methods requiring large sample sizes to using Accelerator Mass Spectrometry, which allows for the dating of microsamples. Since 2018, CRL has undergone significant upgrades, including the acquisition of the MILEA AMS system, enhancing its capacity and precision. CRL's research spans a broad chronological range, providing insights into various periods from early human dispersal in Central Europe, through Neolithic subsistence, Early Iron Age to the Middle Ages. Additionally, CRL addresses modern challenges such as dating ivory to combat wildlife crime.

## 1. Introduction

Archaeology and Quaternary Earth sciences often wish to estimate the age of various objects, matrices or systems. Dating methods are generally absolute, providing a specific age, or relative, determining only age order in relation to context. One of the oldest relative dating methods used by archaeologists, palaeoecologists, and geologists is a stratigraphy: an analysis of sequence, the composition and relationship of stratified soils, sediments, and other material (Harris, 1989). Absolute dating methods fall into four categories – radioisotopic (with the most famous representative being radiocarbon), palaeomagnetic, chemical, and biological. Each method is characterised by its interest matrices, reach, and time estimation uncertainty (Figure 1). Radiocarbon dating stands out above the others in the variety of datable materials, as well as its range conveniently covering human history.

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## 2. Developing the Czech Radiocarbon Laboratory

The Czech Radiocarbon Laboratory was established in 2004 through the joint efforts of Ivo Světlík from the Nuclear Physics Institute and Dagmar Dreslerová from the Institute of Archaeology Prague, Czech Academy of Sciences, and was assigned the international radiocarbon laboratory code CRL in 2005 (Radiocarbon laboratories, 2023).

During its early years, radiocarbon analysis was based on benzene synthesis and its long-term measurement on a low-level liquid scintillation spectrometer QUANTULUS 1220. This radiometric method can provide robust results, though it does require large, gram quantities of samples – and even long measurement times of days do not lead to uncertainties better than 50 BP.

Since 2008, the laboratory team (at that time consisting of only a small number of people) have been developing a methodology for the preparation of microsamples to be measured by the superior accelerator mass spectrometry (AMS) method. AMS applied to radiocarbon dating allows

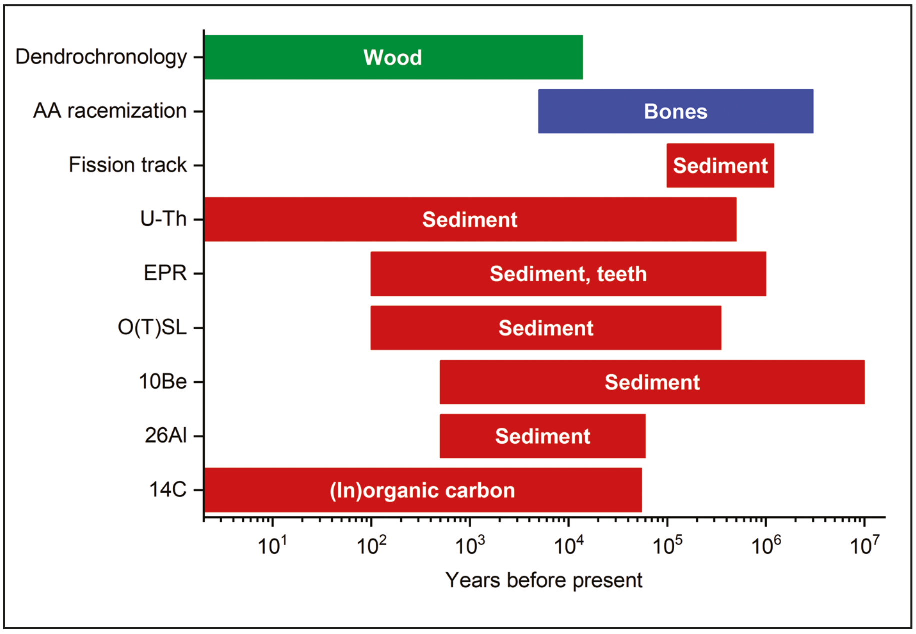


**Figure 1.** Working range of selected dating methods suitable for Quaternary events: several radioisotopic methods (red), amino acid (AA) racemisation as an example of a chemical dating method (blue), and dendrochronology as an example of a biological dating method (green). Datable materials are marked inside the ranges (Wenpeng *et al.*, 2021; Bada and Helfman, 1975).

a fundamental expansion in the "pallete" of datable samples: from bones and wood to plant residues including seeds, cremated bones, hair, fur, and many others, as the method is sufficient when using only tens of milligrams of a sample (Bíšková *et al.*, 2023).

From 2018 to 2023, the laboratory had undergone key upgrades of its equipment, thanks to its involvement in the OP RDE project RAMSES (Figure 2). Above all, the main booster has been the MILEA (Multi-isotope Low-Energy AMS) system, the first AMS in the Czech Republic (Kučera *et al.*, 2023), on which the team has already managed to measure more than three thousand $^{14}C$ samples. The performance of the MILEA system and CRL microsample preparation can be illustrated by the standard deviation of oxalic acid II, a radiocarbon standard measured regularly with samples (Figure 3). A graphitisation blank is determined with phthalic acid to 53 250 ± 2 429 BP (years before present, units expressing $^{14}C$ activity). Other key elements of the CRL equipment include the preparative gas chromatography-mass spectrometer system, the isotope-ratio mass spectrometer, and the Fourier-transformed infrared spectrometer.

From the very beginning, CRL has focused on the endless development of connected methodologies to cover the radiocarbon dating of new, atypical materials, and the establishment of laboratory practice with an emphasis on scientific potential, sustainability, and social usefulness. Additionally, a large team was assembled, comprising highly specialised, trained technicians, students and scientists, and which covers a uniquely wide range of skills and knowledge.

As a result, the latest CRL research focuses on several varied topics spanning the entire time range of radiocarbon dating. Selected examples are presented in the following chapters; arranged chronologically from the oldest period to the youngest, as an adventurous journey in time with the Czech Radiocarbon Laboratory.

**Figure 2.** Former CRL equipment for the benzene synthesis (left), current CRL after key upgrade (right).

## 3. Up to the reach

Our journey must naturally begin at the boundaries of radiocarbon dating itself, which extend approximately 55 thousand years back (Reimer *et al.*, 2020). This boundary coincides with the earliest indications of the appearance of a modern type of man, *i.e.* anatomically modern humans in Central Europe, and radiocarbon dating can offer detailed insights in how to link human dispersal with, for example, previous climatic changes.

The dispersal of anatomically modern humans during the Middle to Upper Palaeolithic overlapped with areas inhabited by Neanderthals (Hoffecker, 2011a; 2011b) and probably occurred during one of the warmer climatic oscillations. The most recent data from our territory come, for example, from the Pod Hradem Cave (Nejman *et al.,* 2017). Fine-grained excavation of the cave record revealed sedimentary units spanning approximately 20,000 years of the Early Upper Palaeolithic and Late Middle Palaeolithic periods, thus making it the first archaeological cave site in the Czech Republic with such a sedimentary and archaeological record. The excavations also confirmed infrequent human visitation, including during the Early Aurignacian, by people who brought with them piece of personal adornment (a bone tubular rod) that have no parallel in the Czech Republic. The raw material diversity of lithics suggests long-distance imports and ephemeral visits by highly mobile populations throughout the EUP period.

Information about climatic oscillations is the best reflected in the palaeorecord, and great attention was and is still being paid to their study in our territory. Radiocarbon dating, in conjunction with other methods, has provided crucial insights into the palaeoenvironment which is well recorded in palaeosols. The introduction of the system of pedocomplexes has brought many insights into the palaeopedological research of our region; this intensified especially in the 1970s when Kukla suggested to the president of the United States the importance of studying palaeosols in the context of future climate change (*i.e.* global cooling versus global warming). In this regard, Kukla created the above-mentioned concept of pedocomplexes, which Smolíková elaborated in terms of soil micromorphology (Němeček *et al.*, 1990; Smolíková, 1971). The basic principles, on which pedocomplexes were determined, are described in great detail in her 1971 article. Since that time palaeopedological research has brought new insights to the fascinating history of sedimentary record (Antoine *et al.*, 2013; Hošek *et al.*, 2017, Lisá *et al.*, 2018; Adameková, Petřík, 2021; Adameková *et al.*, 2023). It is now clear that the sites linked to the arrival of anatomically modern humans and subjected to geoarchaeological analysis (Antoine *et al.*, 2013; Lisá *et al.,* 2014; Mlejnek *et al.*, 2016) not only hold important information about our history which may be revealed with radiocarbon dating, but also offer the significant promise of pushing its limits.

The interpretation of radiocarbon dating would not be possible without the calibration curve. Fortunately, the gradual expansion of calibration curve ranges (Reimer *et al.*, 2020) has offered substantial benefits in the analysis of the oldest radiocarbon data. Continual reassessments of the uncertainties associated with newly developed curves are vital across all datable periods with radiocarbon, ensuring that dating results align as closely as possible with reality.

## 4. First farmers in Neolithic

Now, we pass over to a pivotal moment when our ancestors shifted from a hunter-gatherer lifestyle to productive

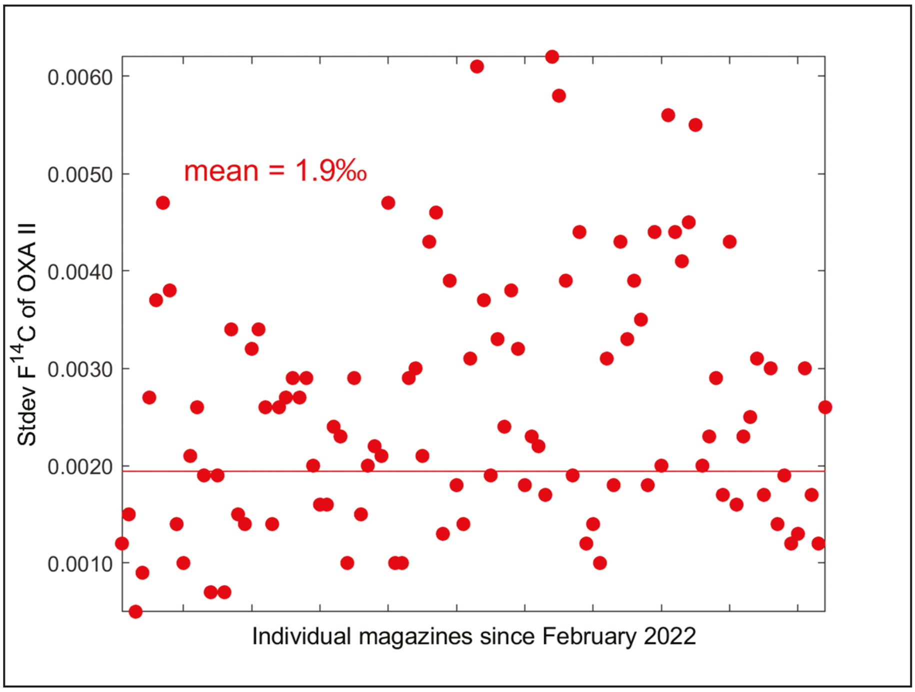


**Figure 3.** Standard deviations of radiocarbon standard oxalic acid II measured by MILEA AMS along with samples in the period February 2022 – February 2024.

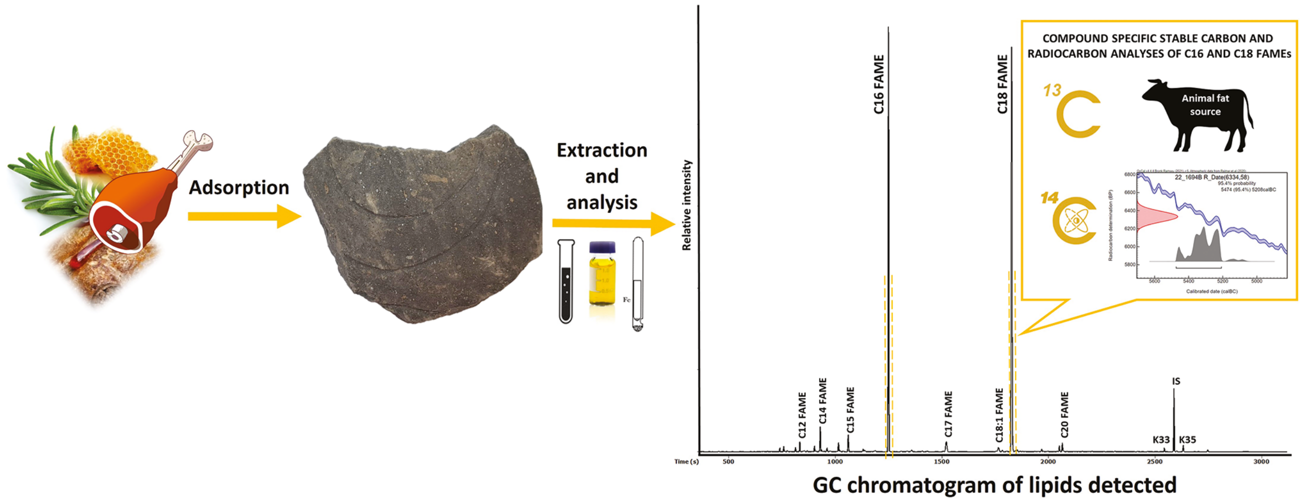


**Figure 4.** Diagram of biomolecular analysis of a Neolithic potsherd with possible outcomes: GC, compound-specific stable carbon and radiocarbon analyses. GC – gas chromatography, C*XY* – carbon chain length, FAME – fatty acid methyl ester, IS – internal standard, K –midchain ketone.

economy. The precise catalysts for this transformation remain subject to debate, but it likely arose from a confluence of revolutionary climate shifts marking the end of the last Ice Age and intellectual advancements that fostered sedentary settlements, introducing practices such as animal husbandry and plant cultivation (Pavlů and Zapotocká, 2007; Berthon *et al.*, 2018). We are in the Neolithic period, approximately 10 000 years ago.

Numerous methodologies exist for probing into the economic and subsistence strategies of our Neolithic forebears, among which is the biomolecular analysis of the pottery they crafted. Cooking within unglazed vessels liberated lipids, either through heat or mechanical means. These lipids infiltrated the porous ceramic structure, where, within the microscopic pores, they could remain preserved for millennia, awaiting their extraction to unveil the culinary secrets of ancient Neolithic kitchens. Thanks to a suite of modern analytical techniques, we can now trace various compounds preserved within pottery, discern their compositions (Evershed *et al.*, 1997), determine their original sources via stable isotope techniques (Dunne *et al.*, 2012), and even ascertain their ages through modern accelerator-based methods following various pretreatments (Casanova *et al.*, 2020). These applications collectively illuminate the survival strategies and lifestyles of our ancient ancestors (see Figure 4).

## 5. Early Iron Age

Another CRL topic has been our participation in the archaeological revision excavation of the famous Early Iron Age site of the Býčí skála ("Bull Rock") Cave. This research, spanning from 2020 to 2021 for the field works, continues to progress with processing and evaluation of the excavated material extending to the present day. Originally explored by Dr. Heinrich Wankel in 1872, the cave exhibited intriguing characteristics from its earliest investigations due to its unique nature. The Býčí skála Cave stands out for its distinctive blend of natural formations and cultural significance. It boasts spacious, damp interiors that have remarkably preserved organic materials (Golec, 2017), making it an ideal site for dating analysis. Situated in the Moravian Karst region, the Býčí skála Cave served as a central sanctuary for elite members of the Hallstatt society in Moravia. Consequently, the cave's architecture incorporated various structural elements, including passages for ceremonial processions and chambers for burial rituals. The archaeological excavations have unearthed a plethora of artefacts, ranging from weaponry, tools, and ornamental jewellery crafted from gold, amber, glass, iron, and bronze, to bronze vessels and other valuable items. Moreover, the site has yielded a substantial amount of organic remains, such as food offerings, cereals, wood, flowers, and woven baskets (Golec and Mírová, 2020).

The unique characteristics of the site have facilitated the relatively good preservation of these objects, offering potential dating material. Moreover, the presence of distinctive elite artefacts, such as wagons, which can be dated to specific archaeological subphases (Golec Mírová *et al.*, in print), further supports the direct dating of materials from the cave. Since Heinrich Wankel's initial excavation in 1872, a substantial amount of material has been collected, with ongoing contributions from revision excavational efforts. Over time, the examination of museum material stored in the Natural History Museum in Vienna, some of which dates back almost 150 years, began to ascertain its suitability for dating. Fortunately, this material proved datable, with dates obtained through a combined methodology involving stratigraphy (to exclude recent or Pleistocene layers), detailed chrono-typology (to select precisely and narrowly dated objects), and radiocarbon dating in conjunction with other isotopic methods.

**Figure 5.** The samples obtained by H. Wankel in 1872, deposited in NHM Wien (left) and the samples obtained during the revision excavation in 2021 (right).

The chrono-typology has primarily focused on valuable, luxurious four-wheeled wagons, comparable to those found in the richest graves in Baden-Württemberg and eastern France, as well as select jewellery pieces and organic materials from within the cave structures, likely associated with the establishment of the burial site. Additionally, comparisons have been made with cave sinter, which encased some objects shortly after the archaeological context. Initial CRL radiocarbon dates obtained between 2022 and 2024 have yielded remarkable results, with ongoing dating efforts still underway. These results confirm the cave's use during the Reinecke's subphases Ha D1b–D3, corresponding to 575–450 BC. Furthermore, radiocarbon dating validates the efficacy of the fine archaeological method of relative chronology. While most dates fall within the so-called Hallstatt Plateau, the refinement of intervals through relative chronology, and further enhancement via other isotopic methods unaffected by the Hallstatt Plateau, hold promise. It is hoped that in the future we will transcend the limitations of the Hallstatt plateau, illuminating, at least in terms of dating, one of Europe's significant prehistoric periods.

## 6. Early Medieval bones contaminated by conservation

Archaeological finds have been of interest to people since ancient times, initially mainly metal, ceramic or stone artefacts, including jewellery, weapons, or utilitarian objects. Until the early 20$^{th}$ century, the collection of ecofacts or human and animal bones was not standard practice in

**Figure 6.** Evidence, description, and conservation of bones at the former State Institute of Archaeology in 1966. Archive of ARÚ Prague, photo number CFJ000002055.

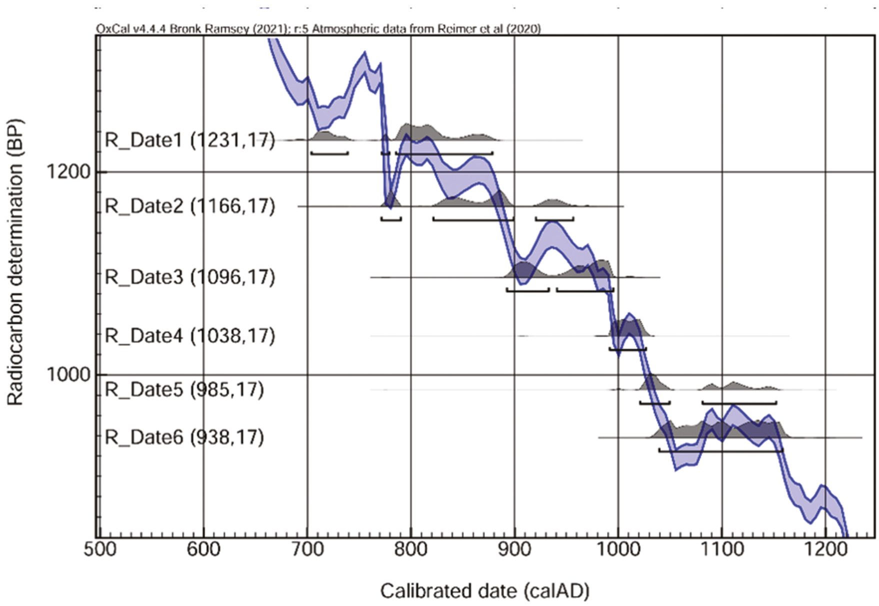


**Figure 7.** The effect of the calibration curve on dating results in the early Middle Ages (Bronk Ramsey, 2009).

archaeological excavations. Such items were typically only retrieved from the most significant sites, such as the Upper Palaeolithic site Předmostí u Přerova in Moravia, Czech Republic. Even then, scholars recognised that these ancient finds were prone to rapid degradation post-excavation (Čermák, 1895–1896, pp.23–24). Consequently, efforts were made to conserve these collections, initially on a sporadic basis and later on a more widespread scale (Novotný, 1894; Čermák, 1895–1896; Stocký, 1927; Losos, 1959).

In January 1950, the Department of Anthropology was established within the former State Institute of Archaeology, marking the first specialised workplace focused on the study of prehistoric and historic human inhumation and cremation remains from Czech archaeological sites. The department amassed a large collection of human bones. All of them were conserved until early 1990s and they preserved up to now (Blajerová and Šaldová, 1986; Likovský *et al.*, 2005).

In the early days of conservation, the primary goal was to protect collection objects from degradation, with little consideration given to the potential removal of preservatives. A variety of preservatives were initially utilised, including natural substances such as oils, dammar gum, shellac, and animal glue, which gradually gave way to synthetic compounds in the post-war era (Zelinger *et al.*, 1982, p.9). The selection of preservatives evolved over time, influenced by factors such as availability, cost, and practical experience (Unger, 1990). Some substances proved unstable, exhibiting issues such as flaking over time or significant discolouration of bones (Chochol, 1964, pp.9–10). Consequently, preservatives such as nitrocellulose lacquers, methyl methacrylate, or polyvinyl acetate, were successively employed. Unfortunately, records detailing conservation procedures were lost during floods in 2002, making it challenging to ascertain the timeline of preservative use or when individual sets of remains were treated.

The radiocarbon dating method is highly sensitive to contamination, which can profoundly impact dating results. Therefore, it is imperative to effectively remove preservatives during the pre-treatment process. While some preservatives are relatively straightforward to eliminate, others pose significant challenges or are nearly impossible to remove entirely (for example, animal glue containing similar proteins to collagen found in bone samples). Furthermore, the ageing or degradation of preservatives, as well as changes in their composition, can adversely affect results (Bíšková *et al.*, 2023, p.43; Brock *et al.*, 2018; Bruhn *et al.*, 2001; Yuan *et al.*, 2007).

Utilising bones from current archaeological excavations that have not undergone conservation is the safest approach. However, it would be unfortunate to overlook key sites in Bohemia that could provide valuable information, including radiocarbon dating, simply because they were excavated in the past. To address this, a selection of human bone samples from various sites in Bohemia dating to the Early Middle Ages, treated with different preservatives, was chosen. This period was selected due to the presence of chronologically sensitive artefacts in skeletal burials and the relatively favourable calibration curve during this time. Radiocarbon dates from the 9$^{th}$ to the 11$^{th}$ century generally fall into three periods. The first spans almost the entire 9$^{th}$ century, overlapping with the earlier period (referred to as the Great Moravian Plateau), while the second group covers the 10$^{th}$ century. A steep section on the calibration curve between 880 and 910 AD and around 1000 AD facilitates a significant separation of dates from the 10$^{th}$ and 11$^{th}$ centuries, sometimes narrowing data to a few decades around 1000 AD (Figure 7).

During this period, radiocarbon dates can also be cross-referenced with relatively precise archaeological dates derived from the typology of specific artefacts, particularly jewellery, militaria, or coins found within graves. Adopting a rigorous critical approach and revisiting old excavations is crucial (*e.g.* Koštová and Košta, 2023). Thanks to Fourier-transform infrared spectroscopy (FTIR), it is now feasible to directly identify the type of conservation utilised in the CRL laboratory, enabling the selection of an appropriate treatment method. This process allows us to assess the potential impact of preservatives on samples and apply the insights gained when analysing samples from other periods.

## 7. Ivory from the Anthropocene

Perhaps somewhat surprisingly, our laboratory also handles samples from very recent sources, primarily those obtained from protected animals to aid in combatting wildlife crime. Wildlife crime, a subset of environmental crime, takes different forms, such as poaching, illegal methods of killing with snares, leg-hold traps, poisons or explosives, animal abuse, smuggling, and, also, illegal trade. Proving these criminal acts is challenging due to their often international nature and the lack of well-developed forensic methods tailored to the specificities of animal tissues. Despite some improvements, wildlife crime remains a low priority in many countries. For instance, in the Czech Republic environmental offences are often handled by enforcement bodies also tasked with addressing tax frauds, bankruptcy, money laundering, copyright protection, and other matters. Consequently, the rights of poached animals must compete fiercely for attention in this demanding landscape.

Legal protection for species typically involves regulations on the trade of their body parts and derivatives. Except for the most endangered species, for which trade is entirely prohibited, there is usually a cutoff date that distinguishes older, tradable individuals from younger, non-tradable ones. In the European Union, for instance, many protected species are subject to a cutoff date of 1947. However, determining the age of such tissue is no simple matter. Ivory, for example, is an exceptionally durable material, only showing noticeable signs of advanced age such as darker colouration or cracks (Figure 8). Czech law allows for the proof of ivory originating before 1947 through solid documentation, expert opinion, or radiocarbon dating. If the first two are unavailable or raise doubts, our laboratory steps in. Unfortunately, our long-term research indicates that much of the ivory offered on the Central European market originates from poached elephants (Kufnerová *et al.*, 2021).

## 8. And what lies ahead?

In the future, our focus remains on advancing instrumentation and refining analytical methods in line with the latest technologies and knowledge. We are committed to deepening our understanding of sample properties and potential interference effects. An ambitious goal we have set is to implement control checks at every stage of sample processing and measurement. This comprehensive approach will not only enhance the accuracy and reliability of radiocarbon dating but also allow us to assess the success rate and establish realistic limitations.

We recognise the significance of fostering open communication with users of radiocarbon dating services. Their suggestions, comments, and perspectives provide valuable independent insights into our laboratory's quality control system. It is equally essential to keep users informed about the current state of the method, ensuring

**Figure 8.** Old ivory of a mammoth (left) and young ivory of an African elephant poached around 2005 (right).

that they are aware of the available options and can have realistic expectations. This transparency fosters trust and collaboration, ultimately contributing to the advancement of radiocarbon dating research.

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