








## Project Gallery

# Chronology of hominin activity at Vindija Cave, Croatia: new dates recorded via standard and ultrafiltration AMS

Ivor Karavanić<sup>1,\*</sup> , Ines Krajcar Bronić<sup>2</sup> , Andreja Sironić<sup>2</sup> , Siniša Radović<sup>3</sup> , Marko Banda<sup>1</sup>  & Fred H. Smith<sup>4</sup>

<sup>1</sup> Faculty of Humanities and Social Sciences, University of Zagreb, Croatia

<sup>2</sup> Ruđer Bošković Institute, Zagreb, Croatia

<sup>3</sup> Institute for Quaternary Paleontology and Geology, Croatian Academy of Sciences and Arts, Zagreb, Croatia

<sup>4</sup> Department of Sociology and Anthropology, Illinois State University, Normal, USA

\* Author for correspondence ✉ [ikaravanic@ffzg.hr](mailto:ikaravanic@ffzg.hr)

The project ‘Last Neanderthals at the Crossroads of Central Europe and the Mediterranean’ (NECEM) combines lithic analyses of previously excavated material with new sampling for dating and environmental DNA. New radiocarbon dates from Vindija, presented here, help clarify the chronology of late Neanderthal and early modern human occupations in South-eastern and Central Europe.

Keywords: South-eastern Europe, Neanderthals, early modern humans, radiocarbon dating, ultrafiltration, Mousterian, Aurignacian

## Introduction

Vindija cave, in northern Croatia, is one of the most significant Palaeolithic and Neanderthal fossil sites in Europe. It is one of nine Palaeolithic sites from the continental and Adriatic regions of Croatia included in the NECEM project (Figures 1 & 2), which began in 2020 and aims to combine multiple lines of research at several sites. Recent excavation and survey—including the measurement of sediment depth via earth resistance tomography and sampling for new dating (radiocarbon and Optically Stimulated Luminescence) and environmental DNA extraction—are complemented by re-analysis of previously excavated lithic material (technology, typology, raw material). The new analyses presented here enhance temporal accuracy for Middle/Upper Palaeolithic hominin activity at both Vindija and in the surrounding region and contribute to our understanding of Neanderthal/early modern human interactions in both regions of Croatia, Central Europe and beyond.

The project has already produced an analysis of Mousterian lithic materials from Vinica Cave, Malo polje-Krban, Radovin and Velika pećina in Kličevica and a preliminary lithic analysis of the Mousterian open-air site at Campanož (Vukosavljević *et al.* 2022; Banda *et al.* 2024). Excavation of part of a trench near the cave entrance at Velika pećina in Kličevica and an underwater survey of Kaštela bay have also been conducted (Karavanić *et al.* 2021a). Two sites from the continental region, Vindija Cave and Velika pećina (on the Ravna gora)—critical for understanding the demise of Neanderthals and the Middle/

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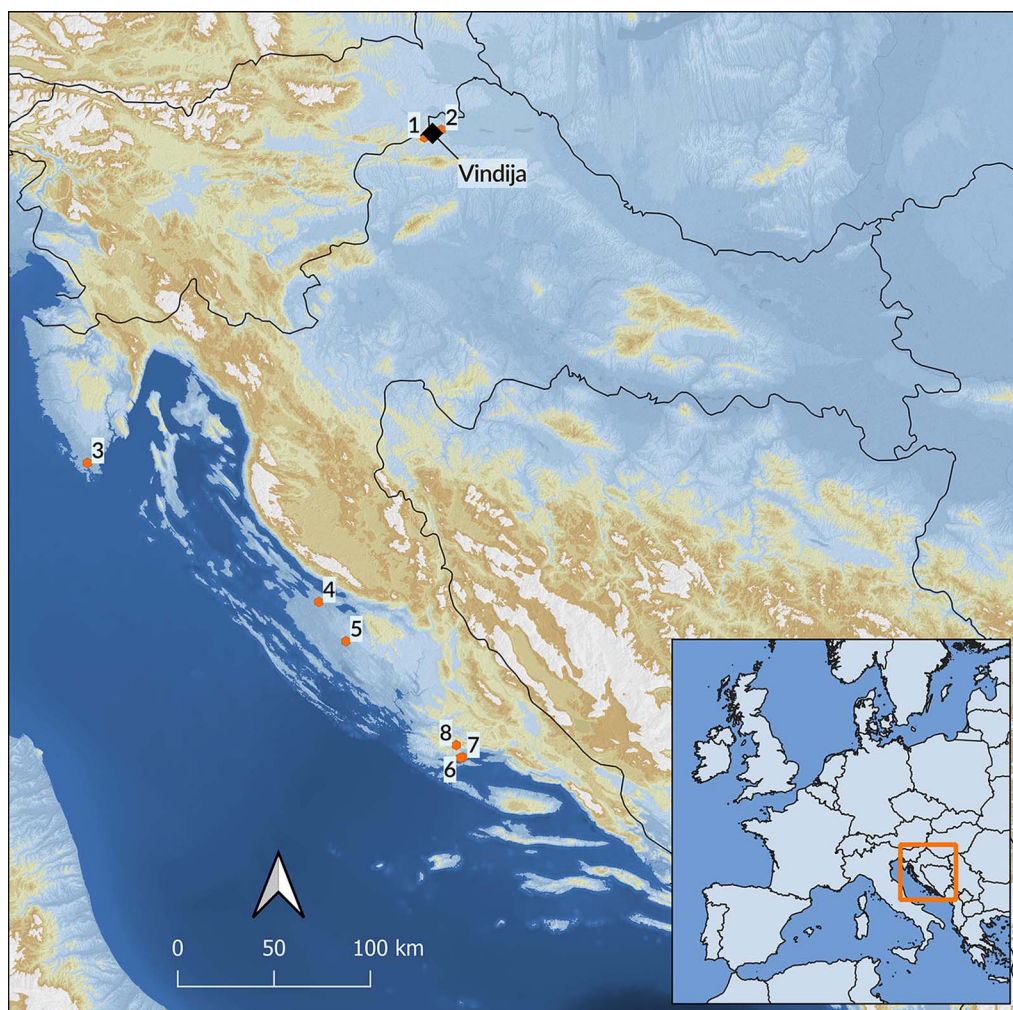


Figure 1. Map showing the location of Vindija Cave and other sites included in the project. Numbers for other sites: 1) Velika pećina (Ravna Gora); 2) Vinica; 3) Campanož; 4) Radovin – Debelo brdo; 5) Velika pećina (Kličevica); 6) Malo polje – Krban; 7) Kaštela bay; 8) Mujina pećina (figure by M. Banda).

Upper Palaeolithic transition in Europe—have been re-dated, as have two Mousterian sites from Dalmatia (Mujina pećina and Velika pećina in Kličevica).

In this article, we present eight new radiocarbon dates from four Vindija faunal samples combining two different techniques of radiocarbon dating—accelerator mass spectrometry (AMS) and accelerator mass spectrometry with collagen ultrafiltration (AMS UF). Two of the samples are from the Mousterian layer G3, which also contains Neanderthal remains, while the other two are from Aurignacian layer F/d, which is attributed to anatomically modern humans.

Dates from these layers enhance the general chronology of a critical period in South-eastern and Central European prehistory. They provide some clarification for the Middle/



Figure 2. Vindija cave (photograph by I. Karavanić).

Upper Palaeolithic interface in Vindija, specifically regarding the large discrepancies in direct dates of Neanderthal remains (see Devière *et al.* 2017 and references therein).

## Materials and methods

Four faunal bones—originally recovered during excavations in 1974–1986 directed by M. Malez and since curated at the Croatian Academy of Sciences and Arts in Zagreb (the Institute for Quaternary Palaeontology and Geology)—were selected for radiocarbon dating. The two bone fragments from layer G3 belong to ungulates and show traces of modification. The two bone pieces from the Aurignacian layer F/d belong to a canid and an ungulate. Two samples were taken from each bone for radiocarbon dating and two techniques, standard AMS and AMS with UF pre-treatment, were used in parallel to produce dates.

The standard AMS dating was performed at the Ruđer Bošković Institute (RBI). Graphite targets were produced from collagen derived from bone samples (Sironić *et al.* 2013). The  $^{14}\text{C}$  activity measurements on these were provided by the AMS facility of the Center for Applied Isotope Studies at the University of Georgia, USA. Carbon dioxide obtained from the combustion of the collagen was sent to the same institution for  $\delta^{13}\text{C}$  determination by isotope ratio mass spectrometry.

Table 1. Results of radiocarbon dating of bones from Vindija using standard AMS and AMS with UF pre-treatment.

Sample code (Zagreb/Oxford)	Layer	Zagreb			Oxford		
		$\delta^{13}\text{C}$	BP	Cal BP (68.3%)	$\delta^{13}\text{C}$	BP	Cal BP (68.3%)
Z-8149	F/d	-21.2	39740 ± 150	43040–42839	-21.0	44500 ± 2000	49583–45047
OxA-42773 UF							
Z-8151	F/d	-18.7	35460 ± 110	40830–40515	-18.1	40600 ± 1300	44640–42819
OxA-42772 UF							
Z-7493	G3	-21.7	40580 ± 130	43915–43325	-21.9	46400 ± 2600	52281–46641
OxA-41143 UF							
Z-7495	G3	-18.3	29410 ± 80	34176–33893	-20.6	42800 ± 1900	47931–43998
OxA-X-3114 34 UF							

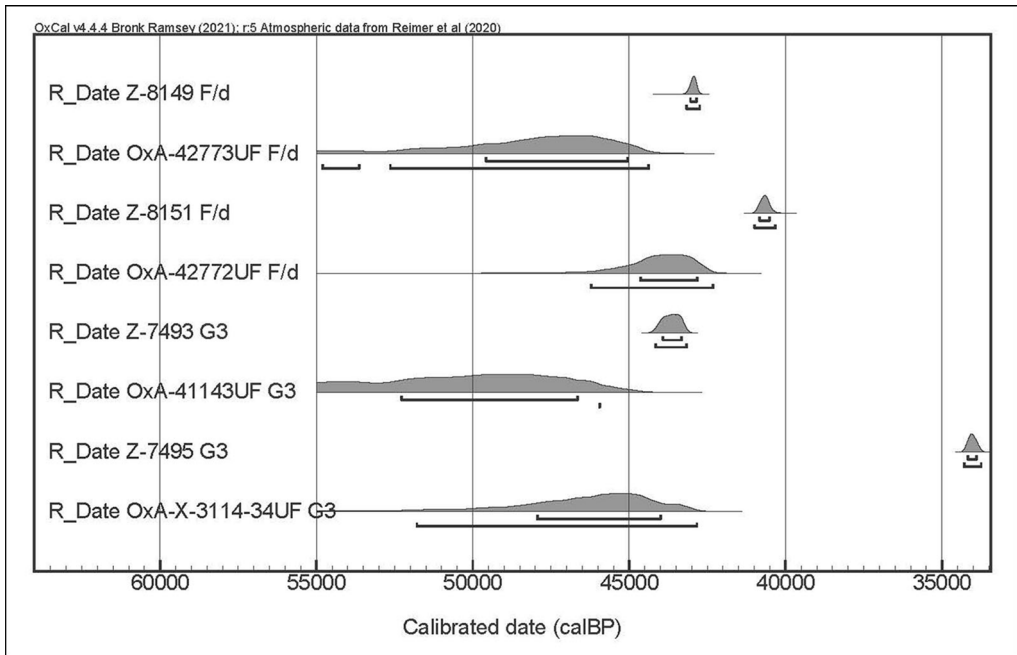


Figure 3. Comparison of calibrated radiocarbon results from Vindija using standard AMS and AMS with UF pre-treatment (1 and 2 sigma error) (figure by I. Krajcar Bronić).

The University of Oxford Radiocarbon Accelerator Unit provided AMS UF dating following standard procedures (Bronk Ramsey *et al.* 2004; Brock *et al.* 2010). Calibration was performed on radiocarbon dates utilising the OxCal v.4.4.4 (Bronk Ramsey 2021) with the IntCal20 calibration curve (Reimer *et al.* 2020).

## Results

The range of  $\delta^{13}\text{C}$  results from both laboratories falls between  $-18.1\text{‰}$  and  $-21.9\text{‰}$ , values typical for bone collagen (Table 1). Figure 3 shows the calibrated AMS and AMS UF ages for each bone. AMS UF consistently returns older dates and there is no overlap with standard AMS dates. Z-7495 gave an exceptionally young date.

## Conclusions

The NECEM project aims to enhance understanding of the temporal aspects of the Middle/Upper Palaeolithic interface in Croatia. Within this project, faunal finds from the late Middle Palaeolithic and early Upper Palaeolithic layers of Vindija cave were dated by standard AMS radiocarbon and UF pre-treatment techniques.

All dates obtained using UF pre-treatment are several thousand years older than standard AMS ages from the same specimens. Results, especially for layer G3, are close to the upper limit of the radiocarbon method. Therefore, we consider the older results (UF pre-treatment) relevant while standard AMS results might only represent minimum ages.



Based on obtained results (UF pre-treatment), the age of the Middle Palaeolithic layer G3 falls between 52 000 and 44 000 cal BP, while the age of early Upper Palaeolithic layer F/d is between 50 000 and 43 000 cal BP (68.3% probability). Results from layer G3 also overlap with direct radiocarbon dates on Neanderthals from G1 (older than approximately 44 000 cal BP), based on amino acid hydroxyproline extraction (Devièse *et al.* 2017). This apparent chronological overlap between layers G3, G1 and F/d strongly suggests that Neanderthals from Vindija are contemporaries of the earliest modern humans in South-eastern and Central Europe. This is consistent with evidence for the presence of Neanderthals at a potentially even later date in the eastern Adriatic (Karavanić *et al.* 2021b).

Therefore, new dates obtained from faunal bones from Vindija cave update our understanding of the Middle/Upper Palaeolithic interface at the site and help clarify the chronology of late Neanderthal/early modern human occupations in South-eastern and Central Europe.

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### Data availability statement

All data supporting the conclusions presented are available within the article.

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