# New U-Th Dates from Vindija, Velika pećina (Kličevica) and Mujina pećina and Their Implications for Chronology of the Middle Paleolithic in Croatia

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# ABSTRACT

This paper reports new results obtained by Uranium-Thorium (U-Th) dating of animal bones, tooth and flowstone samples from three Croatian Middle Paleolithic sites. Dates were obtained on bones and teeth from Vindija (Hrvatsko zagorje) and flowstones from Velika pećina in Kličevica and Mujina pećina (both in Dalmatia). Obtained results support the previously established chronology of the Middle Paleolithic of Croatia by confirming that the oldest layers of Vindija belong to MIS 6 and that Velika pećina in Kličevica was visited by Neandertals after 40 ka BP.

Key words: U-Th dating, Middle Paleolithic, Vindija, Velika pećina in Kličevica, Mujina pećina, Croatia

# Introduction

Croatian Middle Paleolithic sites play an important role in numerous publications about the evolution, genetics and behavior of Neandertals<sup>1-6</sup>. These sites are situated in two main geographic regions of Croatia, continental and Adriatic. Two sites from continental northwestern Croatia (Hrvatsko zagorje) yielded fossil human remains associated with Mousterian artifacts (Krapina and Vindija), while human fossil remains associated with the Middle Paleolithic have not been found at any of the Adriatic sites. However, there are several sites with a Mousterian industry and associated fauna in both regions of Croatia that are important for the understanding of Neandertal adaptation, behavior and the chronology of their occupation in this portion of Europe. Although in recent years work in the Adriatic region has intensified, there is a lack of chronometric dates for these and for continental sites needed to establish a precise chronology of the Middle Paleolithic and Middle/Upper Paleolithic interface in both regions. Therefore, in this paper we present new results of U-Th dating, obtained on samples from three Middle Paleolithic sites, one of which is from the continental region (Vindija), while two (Velika pećina in Kličevica and Mujina

pećina) are from the Adriatic (Figure 1). Our aim is to contribute to the chronology of the Middle Paleolithic and shed some light on the timing of Neandertal disappearance in both continental (northwestern) and Adriatic (southern) Croatia.

### **Site Presentation**

# Vindija

Vindija cave is located 2 km west of the village of Donja Voća, and 20 km west of the town of Varaždin in the Hrvatsko zagorje region (Figure 1). It is more than 50 m deep, 28 m wide at maximum, and more than 10 m high. The first excavations at the site were by S. Vuković in 1928<sup>7</sup>. Systematic excavations were conducted by M. Malez between 1974 and 1986<sup>5,8,9</sup>. During these excavations most of the Paleolithic material, including all Neandertal fossil remains, was discovered. Additional actions (re-exposing the profiles and taking samples) were also conducted by M. Paunović and G. Rabeder from 1993 to 1994<sup>10</sup>.

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Fig. 1. Map of the sites dated by U/Th: 1. Vindija, 2. Velika pećina in Kličevica, 3. Mujina pećina. SRTM data from https:// earthdata.nasa.gov (accessed 20.11.2020.). Designed by M. Banda.

The stratigraphy of Vindija consists of over 9 m of sediment divided into 13 basic stratigraphic units which, according to Malez and Rukavina<sup>11</sup>, covered the period from the onset of the Riss glaciation (MIS6 or earlier) through the Holocene. Pleistocene layers contain finds from both the Middle and Upper Paleolithic<sup>12</sup>. Mousterian artifacts are made on local raw material, mainly guartz, chert and tuff<sup>13,14</sup>. Laver G3 contains a late Mousterian industry with notched and denticulated pieces, sidescrapers, retouched flakes and some Upper Paleolithic types, while the lithic assemblages from earlier layers have not been studied in detail<sup>15,16</sup>. The relatively meager lithic industry from layer G1 suggests continuation of the Mousterian technological tradition (with the lack of Levallois method, as in G3) although mixture of Middle and Upper Paleolithic typological characteristics is also present.

Fossil remains from the site were dated by radiocarbon method<sup>17-25</sup> and U-Th<sup>10</sup> (Figure 2). In this paper we present new dates from Vindija obtained by U-Th on animal bones and teeth, and discuss the significance of these dates. Samples were selected by G. Rabeder from the material excavated by M. Malez (excavation seasons 1979, 1980 and 1983) and from the sampling conducted by G. Rabeder and M. Paunović (season 1994) (Table 1).



Fig. 2. Profile of Vindija with both old and new U/Th dates. Modified by M. Banda after Wild et al.<sup>10</sup>.

#### TABLE 1

# BONE AND TOOTH SAMPLES FROM VINDIJA SELECTED FOR U-TH DATING WITH CONTEXTUAL, STRATIGRAPHIC, TAXONOMIC AND ANATOMIC INFORMATION

Sample ID	Layer	Year	Labels on/from bag	Profile	Depth	Taxon	Element	Side	Material	Comment
VI-G3 bone	G3	1994	VI-G3 bone; 10cm bellow G1; Mt4 dex		10cm below VI-G1	Ursus ingressus	metatarsal 4	dex	bone	distal fragm.
VI-G3 tooth	G3	1994	VI-G3; 2.9.94.; excsite:5; I3S; -60cm	5	-60cm	Ursus ingressus	I3 (3rd upper incisor)		enamel+ dentine	completely preserved
VI-H tooth	Н	1994	VI-H tooth; Datum: 2.9.94.; Vi; Grabungsstelle: 7; Schicht: H; Tiefe: 41cm ueber I; Carninus	7	41cm above VI-I	Ursus ingressus	canine		enamel+ dentine	completely preserved
VI-J bone	J	1994	VI-J bone; Datum: 2.9.94; Vi66; Grabungsstelle: 3; Schicht: J; Tiefe: 184cm unter I		184cm below VI-I	Ursus sp.	metatarsal 4	dex	bone	prox. & dist. fragm.
VI-J tooth	J	1994	VI-J tooth; Datum: 1.9.94.; Vi 48; Grabungsstelle: 3; Schicht: J; Tiefe: 113cm unter I; Eckzahn (C)		113 cm below VI-I	Ursus sp.	canine		enamel+ dentine	completely preserved
VI-K bone	К	1994	VI-K bone; 35cm bellow Vi-J; costa-fragm.		30cm below VI-J	Ursus sp.	costa fragm.		bone	fragment
VI-K tooth	К	1994	VI-K tooth; Profil 6; 35cm bellow Vi J	6	35cm below VI-J	Ursus sp.	m2 inf. fragm.	sin	enamel+ dentine	germ
VI-L bone+tooth	L	1979	VI-L bone+tooth		-	Ursus sp.	mandible with m3	sin	bone+ tooth	fragment
VI-M bone	Μ	1983	VI-M bone		-	Ursus sp.	mandible fragm.	dex	bone	fragment
VI-M tooth	М	1980	VI-M tooth		-	Ursus sp.	m2 inf. fragm.		enamel+ dentine	germ, fragm.

# Velika pećina in Kličevica

Velika pećina lies in the canyon of the Kličevica brook near Benkovac in northern Dalmatia (Figure 1). The main channel of the cave is around 30 m long, 5 m wide on average and ca. 6 m high. At its end the channel turns left and forks after a dozen meters. M. Savić<sup>26</sup> collected several lithics from the cave and its surroundings. M. Malez also visited the site, collected several artifacts and did a smallscale excavation in the cave (M. Savić, personal communication). A test excavation was conducted by I. Karavanić and N. Čondić<sup>27,28</sup> in 2006, and systematic excavations were carried out from 2012 until 2015<sup>29,30</sup>. Five trenches were opened, one of which did not yield any finds. Mousterian artifacts with some animal bones and teeth were found in all other trenches. In the trench near the cave entrance several fragments of prehistoric ceramics (Bronze or Iron Age) were found in the upper layers.

Mousterian tools were made from local chert (Z. Perhoč, personal communication). They are small and strongly resemble the Micromousterian. Among the tools diverse sidescrapers are present; and among them micro-lithic transverse scrapers are noteworthy<sup>31</sup>, strongly suggesting the Charentian type of Adriatic Mousterian. Ani-

mal bones and teeth from Middle Paleolithic layers are less common than artifacts. Although some finds (pottery) from later periods were found, there is no single find suggesting Upper Paleolithic affiliation.

Several radiocarbon dates were obtained on samples from the layers of Velika pećina<sup>5,28,29</sup> showing that the time range of the Middle Paleolithic occupations extended from >48 till about 38 ka cal BP. In this paper we present two dates on flowstone from this site, critical for confirmation of the validity of these radiocarbon dates.

#### Mujina pećina

Mujina pećina is situated in the hills north of Trogir and west of Split in southern Dalmatia (Figure 1). The cave is about 10 m deep and 8 m wide. Finds were initially collected in 1977 from the surface inside and outside the cave<sup>9</sup>, and the first test excavation took place in 1978<sup>32</sup>. Systematic excavations conducted by I. Karavanić and I. Kamenjarin took place from 1995 until 2003<sup>5,33,34</sup>. All occupation layers contain Mousterian industries. The tools were largely made from locally available cherts. They are generally small and strongly resemble the Micromousterian. In some layers sidescrapers are dominant, suggesting the Charentian type of Adriatic Mousterian, while in others denticulated and notched pieces as well as retouched flakes are frequent<sup>35</sup>. No Upper Paleolithic components were found in the stratigraphy of this site. The stratigraphic sequence was dated by both ESR and radiocarbon<sup>36,37</sup>. Radiocarbon dates from several layers suggests a period between about 49 and 39 cal ka BP<sup>37</sup> which is in agreement with ESR dates obtained on three samples of two stratigraphic units<sup>36</sup>. In this paper we present two dates on flowstone from this site.

# **Materials and Methods**

U-Th dating was conducted at the School of Earth Sciences, University of Melbourne (Australia). Isotopic measurements were performed using the multi-collector-inductively coupled plasma-mass spectrometer (MC-ICP-MS) on 20 animal bone and tooth (sub)samples from Vindija Cave and 4 flowstone samples from Mujina pećina and Velika pećina in Kličevica (2 for each). For the dating of bones and teeth from Vindija, in some cases two subsamples were analyzed from the same sample: one from the surface and one from the interior of the sample. Furthermore, three subsamples of a bone from layer K and four subsamples of a bone from layer M were dated. U-Th ages were calculated using <sup>230</sup>Th and <sup>234</sup>U decay constants by Cheng et al.<sup>38</sup>. All uncertainties are reported at 20 level. Final ages are reported relative to 1950 AD.

# Results

In Table 1 we present a listing of the bone and tooth samples collected from Vindija. The results of U-Th dating of these specimens are given in Table 2. We present all dates obtained on samples from this site. They show a time range approximately between 170 and 30 ka. Results on bones and teeth from Velika pećina in Kličevica and Mujina pećina are not listed since they gave unrealistic values, much younger in relation to the archaeological sequence and radiocarbon dating. More precisely, only 2 of the 26 obtained results on Mujina pećina samples show unequivocally Middle Paleolithic ages (one from layer E3A of  $43.80\pm1.90$  ka and another one from E3C of  $51.9\pm3.0$ ka) while others yielded Upper Paleolithic or later (Holocene) ages. This is inconsistent with the fact that all layers of Mujina pećina contain only the Mousterian industry and that the Middle Paleolithic age of these deposits has been confirmed by previous dating results<sup>36,37</sup>. From the same Middle Paleolithic stratigraphic unit (20) of Velika pećina in Kličevica, three Upper Paleolithic dates were obtained by U-Th, while one date is of Holocene age. On the other hand, dating of flowstones from these two sites gave valuable results that are presented in Table 3. Flowstone samples from Velika pećina in Kličevica were taken during excavations in 2006 from squares H-I 22 (Figure 3) and in 2014 from squares G 3-5 (Figure 4), while samples from Mujina pećina (cave wall, square H8) were taken by I. Karavanić in 2016 (Figure 5). The first sample

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was taken at the top of the flowstone, while the second one derives from the bottom. Almost the entire flowstone was covered by sediment before this portion of the cave was excavated. During the sampling there was no sediment left in this part of the cave.

# Discussion

Due to the disparities in ages from samples in the same layers and even two subsamples of the same specimen, it seems that at least some of the bones and teeth from Vindija exhibit open-system behavior of isotope uptake and loss. This means that some samples are unreliable for age determinations because the ratio of parent and daughter isotopes of the U-series is the result of both radioactive decay and the post-depositional exchange of uranium isotopes with the environment, producing either under- or overestimated ages for the deposition of bones and teeth<sup>39-42</sup>. Although a full comparison with the results reported in Wild et al.<sup>10</sup> should be performed after recalculating the ages according to up-to-date decay constants, we can make some general remarks. The new results indicate that layer M dates to MIS 6, probably around 160-170 ka BP, an age range that would have been previously reserved for layers J and/or K according to results reported in Wild et al.<sup>10</sup>. Going further, two subsamples from a tooth from layer H have yielded results of 102±6 and 88.7 +13.8/-12 ka BP, which is at least partially comparable to the average age of 88.2±2.3 ka for the sample from the border of layers H and I reported by Wild et al.<sup>10</sup>. However, the tooth used in the new dating was found ca. 40 cm above layer I, suggesting that even the lower part of layer H could broadly date to a period of ca. 90 ka. If ages for layers M and H are accepted, the dating of layers I to L is not reliable both in these results and those of Wild et al.<sup>10</sup>. The bone sample of the mandible from layer L failed, and the tooth from the same mandible produced incompatible ages (ca. 180 and 130 ka). For layer K one sample was dated only once, and one sample produced three incompatible ages (two of them infinite). Inconsistent dates were also obtained for two subsamples of bone from layer J. The reliability of the other date from a tooth from the same layer is also questionable because no other subsample from it was dated. Dates for these layers reported by Wild et al.10 are much older on average and likewise have inconsistent results. New dates obtained for layer G3 are 33.5+9.0/-8.1 and 38.6+7.6/-6.7 ka. They are in agreement with previously obtained U-Th date from this layer<sup>10</sup>.

Several radiocarbon dates were obtained from the same layer. Dates were calibrated using OxCal v. 4.2.2<sup>43</sup> and IntCal 2020 calibration curve<sup>44</sup>. Human remains from G3 were directly dated to over 42 ka uncal BP by AMS<sup>21</sup> and after that to about 38 ka uncal BP (44.5-41.0 ka (68,3%), 49.1-39.4 ka (95,4%) cal BP) by the same method<sup>22</sup>. An additional AMS radiocarbon date on a Neandertal bone from unit G (layer unknown) has yielded results of about 44 ka uncal BP (47.3-46.1 ka (68,3%), 48.1-45.7 ka (94,4%) cal BP)<sup>23</sup>. Another sample from G3, dated by AMS based on the extraction of the amino acid hydroxy-

							TABLE 2								
			U-TH D.	ATING R	ESULTS	ON BON	VE AND T(	IAS HTOC	MPLES F.	ROM VIN	DIJA				
Sample ID*	Lab number	$^{238}$ U	<sup>230</sup> Th.	/238U	823	4U	Age (uncor	rrected)	<sup>230</sup> Th/ <sup>232</sup> Th	Age (corre	cted)	$\delta^{234}$ U cor	rected		Age
			activity	$\pm 2\sigma$	activity	$\pm 2\sigma$	ka	$\pm 2\sigma$	activity	ka	$\pm 2\sigma$	initial	$\pm 2\sigma$	ka 1950	$\pm 2\sigma$
VI-G3 BONE I	UMD170706-X24	0.01	0.2853	0.0584	1.0695	0.0725	33.863	8.600	228.8	33.665	8.561	1.0761	0.0795	33.5	+9.0/-8.1
VI-G3 BONE O	UMD170706-X23	0.01	0.3430	0.0483	1.1275	0.0539	39.371	7.052	74.2	38.669	7.071	1.1419	0.0591	38.6	+7.6/-6.7
И-НИ ТООТН I	UMD170706-X06	0.06	0.7597	0.0248	1.2175	0.0172	102.552	5.948	242.0	102.142	5.997	1.2902	0.0213	102.0	6.0
VI-H TOOTH O	UMD170706-X05	0.01	0.6533	0.0605	1.1506	0.0235	89.543	12.907	175.7	88.976	13.014	1.1937	0.0294	88.7	+13.8/-12.0
VI-J BONE I	UMD170706-X10	0.00	0.8542	0.1051	1.2363	0.0727	122.551	30.566	210.9	121.942	30.615	1.3326	0.0926	121	+35/-25
VI-J BONE O	UMD170706-X09	0.02	0.8657	0.0480	1.1042	0.0197	160.626	21.051	107.2	159.478	21.233	1.1633	0.0288	159.1	+23.6 / -19.5
VI-J TOOTH O	UMD170706-X43	0.01	0.7830	0.0992	1.2012	0.0217	110.961	23.961	269.4	110.643	24.090	1.2750	0.0325	110	+27 / -22
VI-K BONE 3	UMD180703-X01	0.0046	1.2469	0.1558	1.1983	0.0601	373.893	270.080	183.5	371.542 2	67.459	1.6415	1.2177	430	+inf/-210
VI-K BONE 4	UMD180703-X02	0.0047	0.8996	0.1257	1.2270	0.0595	137.094	39.980	114.5	136.218	40.023	1.3326	0.0820	134	+47/-33
VI-K BONE O	UMD170706-X33	0.02	1.1202	0.0545	1.1168	0.0157	389.849	189.783	60.3	388.193 1	90.950	1.3657	0.4088	371	+inf/-93
VI-K TOOTH O	UMD170706-X31	0.01	0.8127	0.1124	1.2680	0.0464	107.116	25.331	20.7	102.165	25.730	1.3577	0.0607	101	+29/-23
VI-L BONE O	UMD170706-X35	0.36	1.2864	0.0379	1.1315	0.0116	n/a	n/a	10936.1	n/a	n/a	n/a	n/a	n/a	n/a
VI-L TOOTH I	UMD170706-X38	0.31	0.7536	0.0592	1.0777	0.0111	128.560	19.173	6774.8	128.487	19.125	1.1117	0.0162	128.0	+20.9/-17.5
VI-L TOOTH O	UMD170706-X37	0.26	0.9153	0.0160	1.1072	0.0111	180.946	9.461	8135.3	180.955	9.501	1.1786	0.0163	180.9	9.7
VI-M BONE 3	UMD180703-X13	0.5882	0.8854	0.0094	1.1246	0.0039	160.460	4.027	927.0	160.351	3.992	1.1959	0.0057	160.2	4.0
VI-M BONE 4	UMD180703-X14	0.4929	0.9137	0.0098	1.1345	0.0029	168.245	4.275	1540.6	168.162	4.258	1.2162	0.0047	168.1	4.3
VI-M TOOTH I	UMD170706-X18	0.04	0.8131	0.0353	1.1712	0.0309	123.930	11.791	3338.9	123.890	11.735	1.2426	0.0400	123.6	+12.5/-11.1

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VI-M TOOTH O UMD170706-X17 VI-M BONE I UMD170706-X12 0.0249

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U-TH DATI	NG RESU	LTS O	NF.	LOW	STONE	E SAM	PLES F	ROM I	MUJIN	A PE	CINA A	NDVE	ELIKA	PECIN	A IN K	LICEVI	CA
Sample ID	Lab number	Mass	23	<sup>18</sup> U	<sup>230</sup> Th	/238U δ23		4U	Ag (uncorr	ge rected)	<sup>230</sup> Th/ <sup>232</sup> Th	Ag (corre	ge ected)	$\delta^{234}$ U co	rrected	Age	e
		g	ppb	±2σ	activity	±2σ	activity	±2σ	ka	±2σ	activity	ka	$\pm 2\sigma$	initial	±2σ	Ka 1950	±2σ
MP-flowstone lower	UMD 160418-23	0.1336	95	7	0.5426	0.0033	0.9941	0.0043	86.154	1.020	4.1	61.115	27.676	0.9930	0.0052	62	+25 / -31
MP-flowstone upper	UMD 160418-24	0.1429	213	16	0.3722	0.0026	1.0057	0.0049	50.377	0.559	12.9	45.558	4.910	1.0065	0.0056	45.5	4.9
VPK-flowstone 2006 trench	UMD 160418-21	0.1367	238	18	0.3106	0.0022	0.9804	0.0041	41.603	0.423	28.5	39.750	1.905	0.9781	0.0046	39.69	1.89
VPK-flowstone entrance trench	UMD 160418-20	0.1235	436	33	0.2802	0.0016	1.0610	0.0046	33.336	0.283	81.5	32.809	0.593	1.0669	0.0050	32.74	0.61

TABLE 3



Fig. 3. Stratigraphic profile of Velika pećina in Kličevica. Photo by I. Karavanić. Designed by M. Banda.



Fig. 4. Stratigraphic profile of Velika pećina in Kličevice (near entrance). Arrows point to the interface between layers 17 and 18. Modified by M. Banda after Karavanić and Vukosavljević<sup>31</sup>.

proline also gave result of about 44 ka uncal BP<sup>25</sup>. We are aware that comparing the results obtained by different methods should be approached with great caution. As calibrated, these radiocarbon dates are older, but still many of them overlap with new U-Th dates. Directly dated Neandertal remains from layer G1 vielded uncalibrated AMS radiocarbon results of around 32-33 ka BP24, but new AMS dates on these specimens, based on the extraction of the amino acid hydroxyproline, yielded results of about 43 and 44 uncalibrated ka BP. More precisely Vi-207 was dated to 43.9±2 ka uncal BP, while the date obtained on Vi-208 is 42.7±1.6 ka uncal BP25. Calibrated ages for these dates are: 48.91-44.57 (68.3%), 54.61-43.42 (95.4%) ka cal BP for Vi-207 and 47.26-44.12 ka (68.3%), 49.98-42.91 (95.4%) ka cal BP for Vi-208. These dates, together with new dates obtained on fauna<sup>24</sup>, show that Neandertals from G1 are in fact older than previously thought and that previous dates (both radiocarbon and U-Th) from this layer<sup>10,20,24,45</sup> are too recent. However, comparison of recent dates obtained on human and faunal remains from G125 with old and new U-Th dates of layer G3 might suggest that there are no significant chronological differences between G3 and G1. This assumption is supported by the previously mentioned date of ~ 44 ka uncal BP for a sample from G3 (Vi-33.19), obtained by AMS based on amino acid hydroxyproline extraction, although this date may also be explained by post-depositional mixing<sup>25</sup>.



Fig. 5. Flowstone in Mujina pećina. Photo by I. Karavanić.

If the most recent dates by Deviése et al.<sup>25</sup> are correct, they do suggest that some mixing between layers occurred at Vindija. The presence of mixing between layers, particularly between G1 and Fd/d, has been considered possible by us for many years<sup>16,46</sup>. However, while archaeological evidence for mixing involving relatively small lithic pieces was demonstrated<sup>47</sup>, we considered it less likely that larger pieces were mixed. A specific example is the split-based osseous point (Vi 3437) found in association with a Neandertal mandible in layer G1. When excavated, this specimen exhibited the characteristic G1 sediment in crevices (J. Radovčić, personal communication) which strongly suggested its origin from G1. The red sediment is no longer in this specimen, but the situation is similar to the massive-based (so-called Mladeč) osseous point (Vi 3439) that was also found in layer G1 (Figure 6). But the new dates for G1 are too old for such a characteristic Aurignacian specimen (Vi 3437 and possible also for 3439) which indicates that, despite the other evidence, Vi 3437 was admixed into G1 from an upper level. The specimen likely derived from the definitively Upper Paleolithic level Fd/d. However, there is no evidence that admixture



Fig. 6. Massive base (Mladeč type) osseous point found in Layer G1 with imbedded distinctive reddish sediment typical of layer G1. Photo by: I. Karavanić. Figure designed by M. Vuković.

involved any of the significant Neandertal fossils from G1. The only possibility for mixing of Neandertal skeletal remains involves three isolated anterior teeth (Vi 286, 288, and 289) found in layer Fd but described as Neandertallike<sup>48</sup>. However, other Aurignacian-associated anterior teeth exhibit similar Neandertal-like features<sup>1</sup>.

Despite the new G1 radiocarbon dates, it is possible that the Neandertals from both layers (G3 and G1) of Vindija might overlap in time with earliest arrival of modern humans in Southeastern Europe. Specifically, the recent AMS radiocarbon dates from the Bacho Kiro cave in central Bulgaria indicate an age range for the earliest definitive evidence of early modern Europeans between 45.82-43.65 kacal BP (95.4% probability), probably beginning from 46.94 kacal BP (95.4% probability)<sup>49</sup>. It is important to note that these ranges are encompassed by the lower portion of the ranges for Vindija G1/G3. Thus, the documented pattern of "transitional" morphology in the Vindija Neandertals<sup>50,51</sup> could still be the result of biological admixture. If the appearance of the Bohunician in Central Europe during ca. 48–46 ka and certainly by 44 ka<sup>52</sup>, is associated with the arrival of modern humans, the Vindija Neandertals could have been contemporaries of those modern human populations also. Furthermore, the recent claim that modern humans may have reached Greece (southern Balkans) by ~210 ka53 could mean that there are other early incursions of modern humans into this region that have yet to be detected. While this last possibility requires verification, it is certainly not impossible.

As mentioned earlier, U-Th dates from two flowstone samples were obtained from Velika pećina in Kličevica. Dated flowstone (sample ID: VPK-flowstone entrance trench) from the trench near the cave entrance (squares F-G 3-5) (Figure 4) vielded an age of  $32.74\pm0.61$  ka BP. This age is in stratigraphic agreement with a radiocarbon result of >47.7 ka uncal BP (OxA-33732) for a sample coming from layer 20 from the same trench<sup>5</sup>, since the flowstone was deposited between layers 17 and 18 (Figure 4). A bone sample from layer 17 resulted in an age of about 3.4 ka cal BP (OxA-32207), while charcoal sample from layer 19 that is below the flowstone produced the age of 4.3 ka cal BP (OxA-33808)<sup>5</sup>. The age of the dated charcoal sample OxA-33808 is not consistent with the U-Th date on the superimposed flowstone. The very recent age is probably the result of recent intrusion into layer 19. In this trench near the cave entrance, it seems that the flowstone represents a dividing line between Mousterian and Bronze Age layers. While stone artifacts are present in layer 18 which is deposited below the flowstone, they were not found in layer 17 that is deposited above the flowstone. Given the fact that between the dated flowstone and laver 20 there is a ca. one-meter-thick layer in which some Mousterian lithic finds were found, we can conclude that these remains of material culture were deposited before 32.74 ka BP while Mousterian artifacts from layer 20 and lower layers are older than 47.7 ka uncal BP.

The second dated flowstone sample from Velika pećina in Kličevica (sample ID: VPK-flowstone 2006 trench) comes from the interface of layers C and D (Figure 3). The

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age of the flowstone is 39.69±1.89 ka BP. Earlier radiocarbon dates from layers C and D correspond well with the U-Th age of the flowstone. From layer D, an AMS-dated bone of a mid-size ungulate gave a result of about 43 ka cal BP (Beta-228733)<sup>28</sup>. Another animal bone from layer D (cut in two pieces) yielded two results of ca. 40.9-39.6 ka cal BP (Beta-372935) and 37.4-36.3 ka cal BP (Beta-372934). The latter subsample was prepared by ultrafiltration<sup>29</sup>, and its age seems unrealistic if available dates are considered together with the stratigraphic position of layer D. AMS dating of a Cervus elaphus bone from layer C (square H 22) yielded an age of 36.2±0.75 ka uncal BP (OxA-33654), 42.2–39.9 ka cal BP (95.4%)<sup>5</sup>. A Rupicapra bone sample from layer D gave an unexpectedly recent AMS age of ca. 0.5 ka cal BP (OxA-34162). This could be a result of recent intrusion from the top layer or cave surface or result of bioturbations caused by badger burrows that are documented in the neighboring squares.

Obtained dates suggest that Mousterian humans visited the cave for the first time around or before 50 ka BP and were present there until slightly later than 40 ka BP. Such a late survival of Neandertals in the eastern Adriatic is also documented in Bioče rockshelter in Montenegro<sup>54,55</sup>. Therefore, this region joins other examples of post-40 ka Neandertal presence such as southern Iberia<sup>56</sup> and Spy<sup>57</sup>.

Two samples of flowstone from Mujina pećina were also dated. The upper part is dated to approximately 45 ka (the real age falls in between about 50 and 40 ka) which according to radiocarbon and ESR dates corresponds with human occupations of the cave<sup>36,37</sup>. Before this part of the cave (square H8) was excavated, a lower part of the flowstone was covered by layer D1. Unfortunately, the result of dating of this lower part of flowstone is not reliable due to very large uncertainties, which are caused by large detrital Th contamination (<sup>230</sup>Th/<sup>232</sup>Th ratio is 4.1).

#### Conclusion

New results of U-Th dating based on bones and teeth (Vindija) and flowstones (Velika pećina in Kličevica and Mujina pećina) presented in this paper update the chronology of the Middle Paleolithic of Croatia. The application of U-Th dating for these sites is important for two reasons. Firstly, it enabled dating of human occupational episodes whose age is beyond the limits of radiocarbon methods; and secondly, it was used to check the validity (consistency) of previously obtained radiocarbon dates. New U-Th dates confirm that the oldest layers of Vindija were deposited during MIS 6 and, compared to other dates, suggest no significant chronological difference between layers G3 (U-Th and AMS radiocarbon dates) and G1 (AMS radiocarbon dates based on amino acid hydroxyproline extraction). This indicates that Neandertals from both layers might overlap in time with arrival of early modern humans in Southeastern Europe<sup>49</sup>.

Furthermore, results obtained on flowstone samples from Velika pećina in Kličevica confirm the possibility that Neandertals visited the site after 40 ka cal BP. This also strengthens the possibility that Neandertals lived in Dalmatia at the time when modern humans appeared in the eastern part of southeastern Europe<sup>49</sup> and on the Apennine Peninsula<sup>37,58</sup>. According to available radiometric dates, the Eastern Adriatic represents a region with very late survival of Neandertals in Europe.

Further chronometric dating of Croatian Paleolithic sites by various methods is needed to obtain a more precise chronology of the Middle/Upper Paleolithic interface, a period crucial for understanding temporal

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# NOVI REZULTATI DATIRANJA VINDIJE, VELIKE PEĆINE (KLIČEVICA) I MUJINE PEĆINE U-TH METODOM I NJIHOVA IMPLIKACIJA ZA KRONOLOGIJU SREDNJEG PALEOLITIKA U HRVATSKOJ

# SAŽETAK

Ovaj rad donosi nove rezultate datiranja uran-torij (U-Th) metodom uzoraka kostiju, zuba i siga s tri hrvatska nalazišta srednjeg paleolitika. Rezultati dobiveni na kostima i zubima potječu iz Vindije (Hrvatsko zagorje), a na sigama s nalazišta Velika pećina u Kličevici i Mujina pećina (oba u Dalmaciji). Dobiveni rezultati podržavaju ranije uspostavljenu kronologiju srednjeg paleolitika Hrvatske jer potvrđuju da najstariji sloj iz Vindije pripada periodu MIS 6 te da su neandertalci posjećivali Veliku pećinu u Kličevici i nakon 40 tisuća godina prije sadašnjosti.