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DATING LATE PALEOLITHIC HARPOONS FROM LAKE LUBĀNS, LATVIA

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ABSTRACT. Over 3000 prehistoric bone and antler artifacts, collected in the late 1930s from the former lakebed of Lake Lubāns, are held by the National History Museum of Latvia. This collection is remarkable not only as one of the largest known assemblages of bone implements in northern Europe, but also in terms of diversity of forms. The most elaborately worked objects include harpoons, often with two rows of barbs and spade-shaped bases, which are believed to date to the Late Paleolithic, and to be among the oldest organic artifacts ever found in Latvia. Four broken specimens were sampled in 2011 for accelerator mass spectrometry (AMS) dating, stable isotope analysis, and taxonomic attribution by ZooMS. The results support the interpretation that these artifacts were made from large cervid bones, and date all four objects to the early Preboreal (mid-10th millennium cal BC). The Lake Lubāns harpoons therefore fall in the same period as similar harpoons from Denmark, northern Germany, and Poland, although only a handful of these have been dated directly.

INTRODUCTION

Lake Lubāns, situated in the lowlands of eastern Latvia, was formed following the deglaciation of the Lubāns ice lobe during the Linkuva glacial phase (probably after 13.5 ka cal BP: Zelčs et al. 2011). A minimum age of ~9800–9300 cal BC is given by the dates of reindeer antlers from the lakebed (Hela-607, 9980 ± 70 BP: Ukkonen et al. 2006; Poz-26786, 9990 ± 50 BP: Zagorska 2012: Table IV). In 1937–1940, after the lake level was artificially lowered to create new agricultural land, a unique collection of ~3500 bone and antler stray finds came to light. The artifacts were found by local residents on the former lakebed in the drained southwestern part of the lake and on an island, close to the bank, and lack archaeological contexts (Figure 1). This collection is remarkable not only as one of the largest known assemblages of bone implements in northern Europe, but also in terms of diversity of forms. It spans the period from the earliest human occupation of Latvia, at the end of the Paleolithic, to the end of the Bronze Age (Vankina 1999).

The periodization of the assemblage is based on comparisons with material excavated at stratified settlement sites, in Latvia and in neighboring countries (Zagorska 1983), but for many types, comparative material is absent or uncertain, which means that absolute ^{14}C dating is essential. This is particularly important for dating the earliest occupation of the region. The typologically more archaic forms of harpoons were therefore chosen for dating.

The harpoons are believed to date to the Late Paleolithic (~10,500–9000 cal BC), and to be among the oldest organic artifacts ever found in Latvia, although no absolute dating has ever been undertaken. Four broken specimens in the National History Museum in Riga were sampled in 2011 for accelerator mass spectrometry (AMS) dating, stable isotope analysis, and taxonomic attribution by collagen-peptide sequencing (ZooMS). Broken harpoons of types I and II were sampled. The only specimen of Type III is on display in the museum, and is inaccessible.

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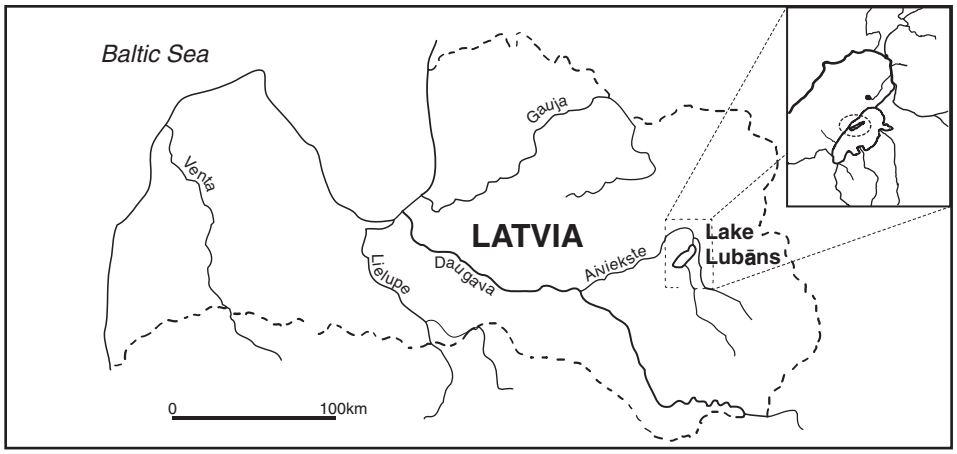

Figure 1 Map of Latvia, showing the location of Lake Lubāns and (inset, circled) the area where most of the bone and antler artifacts were collected.

Sample Selection

Eleven archaic bone and antler harpoons—hunting or fishing weapons, designed for throwing and detachable from the shaft—are recorded in the Lake Lubāns collection (Zagorska 1999: Figure 3; Zagorska 2006). Typologically, these harpoons have traditionally been divided into three groups:

I. Biserial harpoons, with asymmetrically arranged, widely spaced, angular or rounded barbs, and a spade-shaped base.

II. Biserial harpoons, with slanting, symmetrically or asymmetrically arranged barbs, and spade-shaped base.

III. A uniserial harpoon, with widely spaced and strongly curved beak-like barbs, and an irregularly formed base.

Six harpoons are attributed to Type I. Two of them are intact, ~200 mm long, with barbs widely spaced, angular or slightly rounded. There are four to six barbs on each side, arranged alternately. Both objects have spade-shaped bases, with a basal inverse barb on one or both sides. In cross-section, the artifacts are triangular, rounded, or plano-convex. Three of the broken Type I harpoons were sampled (Figure 2):

A10519:1488 – the point of a harpoon, of which the lower part is missing. It has two widely spaced small angular barbs on one side and three on the other, asymmetrically arranged. The bone is dark brown. Dimensions: L 132 mm, W 15 mm, T 5 mm.

A11928:489 – a broken harpoon, of which the base is missing. It has three widely spaced small angular barbs on one side, and four on the other side, arranged asymmetrically. The bone is dark brown. Dimensions: L 173 mm, W 12 mm, T 6 mm.

A11928:495 – the point of a harpoon, of which the lower part is missing. It has two widely spaced, angular (but slightly slanting) barbs on each side, arranged alternately. The bone is dark brown. Dimensions: L 113 mm, W 18 mm, T 8 mm.

Four harpoons are attributed to Type II. All four are incomplete; three are points, the fourth a spade-shaped basal section. Typologically, they are very close to Type I harpoons, but the barbs are more

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Figure 2 Sampled harpoons. Left to right: A10519:1488, A11928:495, A10519:1480, A11928:489. Dimensions given in the text.

oblique. One of the Lake Lubāns Type II harpoons was sampled (Figure 2):

A10519:1480 – upper part of a harpoon, with six slanting barbs along one side and five along the other. The upper barbs are arranged symmetrically, the lower ones asymmetrically. Unlike the Type I samples, which appear to be of bone, this specimen is probably made from antler (perhaps reindeer), with a finer structure and light brown color. Dimensions: L 165 mm, W 24 mm, T 7 mm.

Because all the harpoon surfaces are heavily worked, we cannot determine, by traditional archaeozoological analysis, the species or skeletal element from which the harpoon was made. Similar artifacts found in northern Germany and Denmark have been attributed to antlers of reindeer (*Rangifer tarandus*) and elk (*Alces alces*) or long bone from an unspecified deer (Mathiassen 1938; Taute 1968; Bokelmann 1988; Andersen and Petersen 2009). It is believed that the Latvian harpoons too are most likely to have been made from elk or reindeer bones or antlers.

The unbroken harpoons on display in the National History Museum cannot be analyzed by destructive methods. Moreover, they were probably conserved, using consolidants that may be impossible to remove completely, whereas there is no record of the incomplete artifacts in the National History Museum archives having been consolidated.

Radiocarbon Dating

Small slices, weighing ~0.5 g, were cut from the broken ends of the harpoons at the National History Museum, using an electric rotary cutting tool. Whether the harpoons were made from bone or antler, collagen is regarded as the most reliable component for radiocarbon dating (Longin 1971). At the Leibniz Labor, Kiel, a small fragment of each sample (~2–3 mg) was crushed to fine powder for

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Fourier transform infrared (FTIR) spectroscopy, which is used to estimate the approximate collagen content, and thus the amount of bone or antler required to yield enough collagen for accelerator mass spectrometry (AMS) dating. These tests indicated good to excellent collagen preservation, and it was therefore possible to subsample each fragment.

Although the FTIR spectra did not suggest any contamination due to handling or unrecorded conservation treatment, as a precaution the samples were cleaned before pretreatment using a Soxhlet-type solvent extraction, designed to remove fatty and waxy organic contaminants (Bruhn et al. 2001). In sequence, samples were treated three times each with boiling tetrahydrofurane, chloroform, petroleum-ether, acetone, and methanol, and then rinsed with deionized water (each solvent in the sequence removes its predecessor, and methanol is water-soluble). Collagen was then extracted, following a variant of the method described by Longin (1971): the sample was broken into 0.5–2 mm fragments, which were completely demineralized in HCl (~1%). The demineralized bone material was treated with 1% NaOH (20°C, 1 hr) to remove humic acids, and then again with 1% HCl (20°C, 1 hr). The remaining material was dissolved in deionized water overnight at 85°C and pH = 2.7, producing a gelatin solution, which was passed through a 0.45-µm pore silver filter to remove insoluble constituents. The gelatin was then freeze-dried and weighed. Collagen yields (Table 1) were 9–16% of the starting weights, usually indicative of good to excellent preservation (e.g. Dobberstein et al. 2009). This is reflected in the atomic C:N ratios (Table 1), which are all within the expected range for well-preserved collagen (DeNiro 1985; cf. Harbeck and Grupe 2009).

The extracts were combusted, graphitized, and measured by AMS at the Leibniz-Labor in Kiel, following Nadeau et al. (1998). Enough collagen was extracted from KIA-46261 (A11928:489) and KIA-46262 (A11928:495) for the extracts to be split, so that two graphite targets from separate combustions of the same extraction could be dated. Table 1 gives the results of all six targets. The AMS-measured $\delta^{13}C$ values (not shown) are within the expected range for terrestrial herbivores and similar to isotope ratio mass spectrometer (IRMS)-measured values for the same samples. The two ^{14}C measurements from KIA-46261 (A11928:489) and KIA-46262 (A11928:495) are statistically consistent, allowing a weighted mean ^{14}C age to be calculated for each harpoon (Table 1; Ward and Wilson 1978).

Stable Isotope Measurement

An aliquot of each extract was sent to the School of Life Sciences, University of Bradford, England, for measurement of carbon and nitrogen concentrations, and isotopic $^{13}C/^{12}C$ and $^{15}N/^{14}N$ ratios ($\delta^{13}C$ and $\delta^{15}N$) using a coupled elemental analyzer/isotope ratio mass spectrometer (EA-IRMS). The samples were run on a Thermo Flash 1112 Elemental Analyzer coupled to a Thermo Delta plus XL mass spectrometer. Typical measurement errors of ±0.2‰ are quoted for both $\delta^{13}C$ and $\delta^{15}N$ in both samples and standards. Laboratory standards and international standards were analyzed in the sample runs and acceptable results were obtained. Duplicate results were obtained where possible (Table 1).

The stable isotope values fall within a narrow range consistent with expectations: wild herbivores in Europe, whose diets are based on plants that use the C_3 photosynthetic pathway, generally give collagen $\delta^{13}C$ values between –22 and –20‰. A10519:1480 (KIA-46259) gave a slightly enriched $\delta^{13}C$ value compared to the other three samples. Given the ^{14}C age of A10519:1480, we could interpret this enrichment as reflecting a more open landscape at the beginning of the Preboreal, and subsequent forest development producing a "canopy effect" on herbivore $\delta^{13}C$ values (Drucker et al. 2008). Equally, it may mean that harpoon A10519:1480 was from a reindeer and the other three were from elk. Reindeer often give more enriched $\delta^{13}C$ values due to their ability to consume large

Table 1 Radiocarbon and stable isotope results, KIA-46259–62.

Sample, lab nr	Yield (%)	Atomic C/N	$\delta^{15}N$ (‰)	$\delta^{13}C$ (‰)	Corrected ^{14}C concentration ($F^{14}C$)	Conventional ^{14}C age (BP)	Calibrated date (95% confidence)
A10519:1480 KIA-46259	16.1	3.35	1.85	–20.18	0.2882 ± 0.0016	9993 ± 44	9760–9310 cal BC
A10519:1488 KIA-46260	11.0	3.24	2.14	–22.26	0.2960 ± 0.0014	9780 ± 38	9310–9210 cal BC
A11928:489 KIA-46261	11.4	3.21	2.12	–22.15	0.2909 ± 0.0027	9918 ± 73	
			2.06	–22.22	0.2875 ± 0.0017	10,014 ± 49	
			2.19	*–22.19*	*weighted mean†*	*9985 ± 41†*	*9750–9310 cal BC*
					(T' = 1.2, T'(5%) = 3.8, ν = 1)		
A11928:495 KIA-46262	8.9	3.16	1.76	–22.21	0.2922 ± 0.0016	9884 ± 43	
			1.52	–22.15	0.2909 ± 0.0017	9920 ± 48	
			1.64	*–22.18*	*weighted mean†*	*9900 ± 33†*	*9450–9280 cal BC*
					(T' = 0.3, T'(5%) = 3.8, ν = 1)		

Note: C/N, $\delta^{15}N$ and $\delta^{13}C$ were measured by EA/IRMS at the University of Bradford, England; $F^{14}C$ was measured by AMS at the Leibniz Labor für Altersbestimmung und Isotopenforschung, Kiel, Germany. Results in plain type are mean data from two aliquots of the same extract; results in italics are mean data from two aliquots of the same extract; results in plain type are measurements of a single aliquot.
†Calculated following Ward and Wilson (1978).

Table 2 ZooMS collagen peptide sequencing results, KIA-46259–62.

Sample, lab nr	Peptides commonly observed in ruminant collagen	Peptides commonly observed in marine mammal collagen	Mascot identification
A10519:1480/KIA-46259	Present	Not found	Ruminant *
A10519:1488/KIA-46260	Present	Not found	Ruminant *
A11928:489/KIA-46261	Present	Not found	Ruminant: Cervidae
A11928:495/KIA-46262	Present	Not found	Ruminant *

*Not enough unique peptide markers in spectra to further identify the sample.

amounts of lichen. Reindeer antlers from Latvia have produced $\delta^{13}C$ values between –20.2 and –17.9‰ in this period (Ukkonen et al. 2006). The $\delta^{15}N$ values of all four samples are lower than those in mid-late Holocene herbivores (e.g. Noe-Nygaard et al. 2005), but typical of Preboreal herbivores, reflecting the gradual rise in soil $\delta^{15}N$ in the early Holocene (Drucker et al. 2011).

Species Identification by Collagen-Peptide Mass Fingerprinting

The working of bone material into artifacts such as harpoons often removes morphological features that would once have allowed species identification. Mindful of the possibility of ^{14}C reservoir effects, we hoped to identify the species concerned, but normal archaeozoological determination of the Lake Lubāns harpoons was not possible. One method of separating marine and terrestrial mammalian collagen is by detecting differences in their collagen amino acid sequences.

Samples of untreated bone/antler material were taken for ZooMS collagen-peptide sequencing at the Centre of Excellence in Mass Spectrometry, University of York. The samples, of material left over from the samples prepared for ^{14}C dating, consisted of small fragments or coarse powder (~10 mg) where possible, and the fine powder (~2–3 mg) previously used for FTIR analysis of all four artifacts. Each sample was transferred to a separate plastic microfuge tube (Figure 3). In order to clean the samples and reduce the amount of unwanted organic contaminants, such as humic acids, a small amount of buffer solution was added, and the samples were left to soak overnight. The buffer solution was removed and the collagen extracted by resuspending in buffer and warming for 1 hr at 65°C (following Buckley et al. 2009). The extracted collagen was then cleaved into shorter peptides (approximately 10 amino acids in length) using the proteolytic enzyme trypsin. All of the samples were prepared in duplicate, and each analyzed in triplicate.

The collagen digests were purified on ZipTips and the resulting peptide mixtures were analyzed using matrix-assisted laser desorption/ionization time of flight mass spectrometry (MALDI-TOF MS). This is a rapid analytical technique in which a UV laser is used to desorb and ionize the peptides; these peptide ions are then accelerated and their mass/charge (m/z) ratios determined from their times-of-flight. Peptides with smaller masses travel a fixed distance faster, and thus

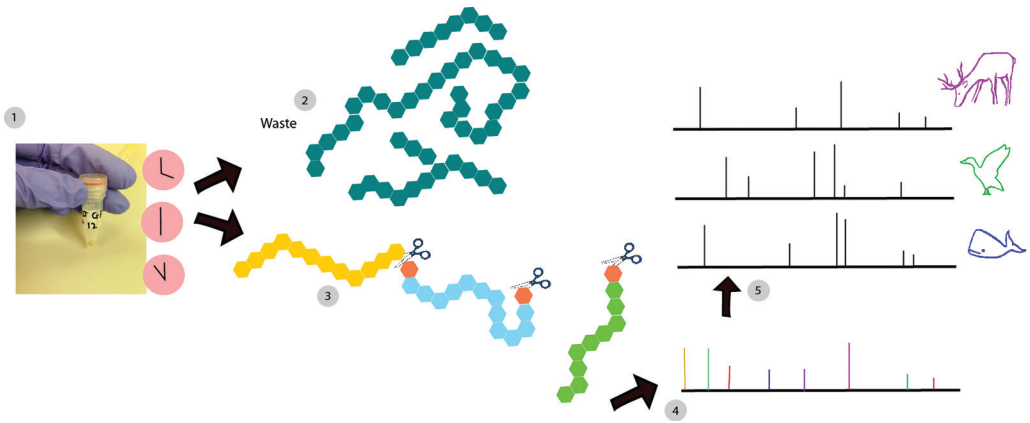

Figure 3 ZooMS screening of bone: (1) The sample is soaked overnight in buffer solution (50 mM NH_4HCO_3, pH 8.0) at room temperature, to reduce organic contaminants, such as humic acids; this solution is discarded. (2) Collagen is extracted in buffer solution for 1 hr at 65°C. (3) Using trypsin, the extract is cleaved into shorter peptides (~10 amino acids long) and the digests purified on a ZipTip, before MALDI-TOF analysis. (4) All samples were prepared in duplicate, and then analyzed in triplicate on the MALDI plate.

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reach the detector before heavier peptides. The resulting spectrum is characterized by a series of signals, one for each peptide. The *m*/*z* ratios of the signals depend on the amino acid sequence of the collagen from which the peptides were derived, and so this is referred to as a "peptide mass fingerprint."

The primary structure of type I collagen consists of a series of amino acids that join together to form the alpha-1 and alpha-2 polypeptide chains. Two alpha-1 chains and one alpha-2 polypeptide chain wind together to form the right-handed triple helical structure characteristic of collagen. Although the amino acid sequences that make up all Type I collagens are similar, there are variations between species. These variations are detected as different tryptic peptide mass fingerprints. The observed peptide mass fingerprints can be used for species identification (ZooMS) if the amino acid sequence of collagen from the particular species is known, or if an authentic sample of collagen from that species is available for comparison. No peptide signals were observed in the peptide mass fingerprints of the harpoon samples that correspond with signals commonly observed in marine mammal collagen digests. All of the spectra obtained from the samples contained *m*/*z* signals consistent with peptides found in ruminant collagen. The results support the interpretation that the harpoons were made from bones or antlers of large herbivores, which means that their ^{14}C ages should provide reliable dates.

Chronological Interpretation

Table 1 gives the calibration of the weighted mean ^{14}C age of each sample, expressed as a 95% confidence interval. Calibrations were performed using OxCal v 4.2 (Bronk Ramsey 2009) and the IntCal13 calibration data (Reimer et al. 2013). Figure 4 shows the calibration of the ^{14}C ages by the probability method (Stuiver and Reimer 1993).

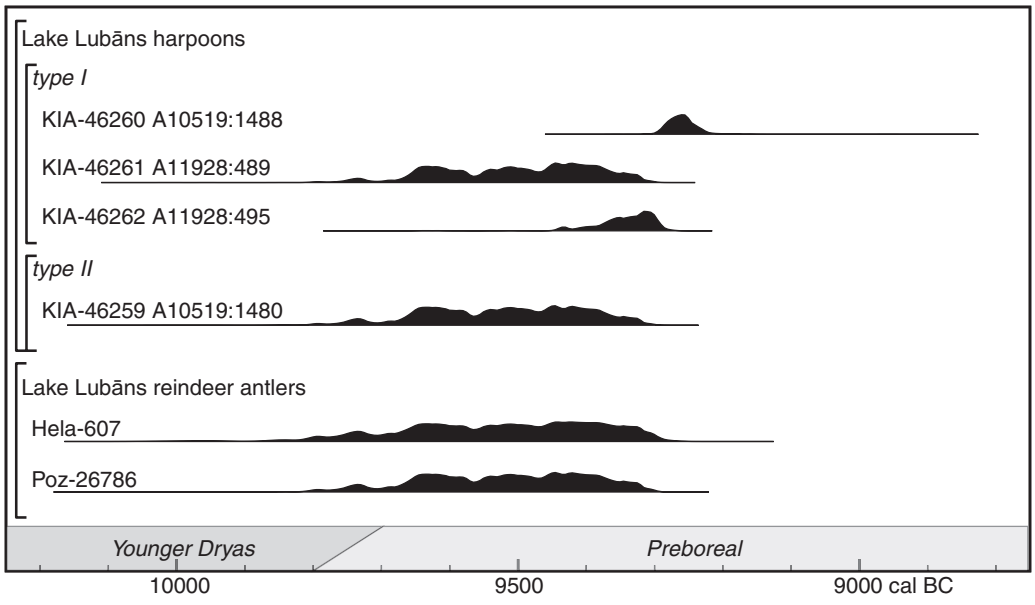

Figure 4 Calibration of the ^{14}C results, using OxCal v 4.2.1 (Bronk Ramsey 2009) and the IntCal13 calibration data (Reimer et al. 2013).

All four harpoons date to the mid-10th millennium cal BC, in the early Preboreal period, and are broadly contemporary with the reindeer antlers from Lake Lubāns dated by Ukkonen et al. (2006)

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and Zagorska (2012) (Figure 4). The calibrated dates relate to the lifetime of the animal concerned, not to when the artifacts were actually made, but there is no reason to assume that the harpoons were made using old bones (cf. Gramsch et al. 2013 for a counter-example). The calibrated dates are only *termini post quos* for the loss or disposal of the harpoons, but it is difficult to imagine that a harpoon remained in use for more than a few years.

A10519:1480 (Type II) could be the earliest of the dated harpoons. A fairly similar harpoon from Vallensgårds Mose on Bornholm (Denmark) was dated to 9200–8780 cal BC (9585 ± 55 BP, AAR-9404; Petersen 2009: 52 n.2). Another Danish harpoon from Lille Åmose was recently dated slightly earlier, but still within the Preboreal (P V Petersen, personal communication, 2013).

Our Type I harpoons (A10519:1488, A11928:489, A11928:495) correspond to biserial harpoons with spade-shaped bases found throughout Denmark, Germany, and Poland (Bokelmann 1988; Cziesla 2004; Taute 1968). At Stellmoor, near Hamburg, these harpoons were dated to the final Paleolithic Ahrensburgian culture (end of the Younger Dryas/beginning of Preboreal; Fischer and Tauber 1986). Two such harpoons, stray finds from Bützsee, north of Berlin, are dated to 10,680–10,150 and 9820–9310 cal BC (10,480 ± 75 BP, OxA-8742; 10,020 ± 60 BP, OxA-8841; Cziesla and Pettitt 2003; Cziesla 2004). The Latvian harpoons are a little younger and could represent the last appearance of this type.

CONCLUSION

Archaic bone/antler harpoons are rarely, if ever, found in well-stratified archaeological contexts, and the handful of stray finds that have previously been dated directly were found over 1000 km to the southwest of Lake Lubāns. Their chronology in the eastern Baltic was unknown, and our results are therefore quite significant, even if only a few artifacts have been dated so far. Moreover, we have shown that the collagen preservation in the Lake Lubāns collection of bone and antler artifacts is excellent, which opens the prospect of dating even smaller samples (from undamaged artifacts) or extracts from different pretreatment protocols (where contamination with consolidants is suspected). The first application of mass spectrometric peptide mass fingerprinting, or ZooMS, to Late Paleolithic finds has been shown to be informative and suggests that this technique, together with stable isotope analysis, can be employed routinely as part of the dating of bone artifacts. A wider program of research into the chronology of biserial harpoons, incorporating stray finds from around the Baltic region, can now be developed, and research on the chronology of other artifact types at Lake Lubāns should be considered.

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