Radiocarbon dating and ZooMS species identification of fragmentary bone at the Late Upper Palaeolithic and Mesolithic site of King Arthur's Cave

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Abstract:

King Arthur's Cave (Wye Valley) contains a late Pleistocene and Holocene sedimentary sequence, with evidence of Late Upper Palaeolithic, Mesolithic and later occupations. It currently provides the earliest dates for a human presence in the British Isles after the Last Glacial Maximum. Here we revisit the faunal material from the University of Bristol Speleological Society 1920s and 1950s excavations to further clarify the chronology of the stratigraphic sequence on the platform outside the cave mouth. The results of six new ultrafiltered radiocarbon dates confirm that fauna date to before the Last Glacial Maximum and to the Late Glacial, and that some post depositional stratigraphic mixing has occurred. We undertook peptide mass fingerprinting (ZooMS) of fragmentary bones from the platform archaeological levels to provide further insights into the fauna during the late Pleistocene and early Holocene. The ZooMS species identification indicate the fragmentary bone assemblage mirrors the species present in the morphologically identifiable bone assemblage. Although dominated by red deer, the presence of "mammoth steppe" fauna such as woolly rhino and spotted hyaena, alongside temperate species and domesticated animals (e.g. sheep) further confirm post depositional stratigraphic mixing. Amongst the fragments identified is a human bone which, based on its provenance, could be Late Glacial or early Holocene in age and relate to the Late Upper Palaeolithic or Mesolithic activity at the site. The specimen is currently being radiocarbon dated.

Key words: ZooMS, radiocarbon dating, Magdalenian, Federmesser, Late Glacial

Introduction

King Arthur's Cave is located on Great Doward Hill, 1km from Symmonds Yat in the Wye Valley, Herefordshire (Fig.1). The site contained an important late Pleistocene and Holocene sedimentary sequence, with evidence of Palaeolithic, Mesolithic and later occupations (ApSimon et al. 1992). Evidence for human activity at the site during the Late Upper Palaeolithic and Mesolithic comes from lithic artefacts and faunal remains bearing traces of anthropogenic activity. The lithics include bi-truncated trapezoidal backed blades ('Cheddar points'), in addition to a single curve-backed blade ('Federmesser') along with straight-backed blades and bladelets, and Mesolithic microliths (ApSimon et al. 1992; Jacobi and Higham, 2011). Faunal remains of horse and red deer bear evidence of cut marks and cultural fractures, indicating exploitation and processing by humans. Radiocarbon dating of these faunal remains demonstrate at least two separate episodes of Palaeolithic human activity, each exploiting a different prey (Jacobi and Higham, 2011). Fractured horse teeth date to 15,515 to 14,315 cal. BP (IntCal20, 95% confidence interval, n=4, OxA-19161 (UBSSM catalogue number W2.21/485), OxA-19166 (W2.21/484), OxA-X-2280-8 (W2.21/559), OxA-X-2280-9(W2.21/560), Table 1) and provide the earliest evidence of humans in the British Isles after the Last Glacial Maximum (LGM) (Jacobi and Higham, 2011). Cut-marked red deer bones date to 14,160 to 13800 cal. BP (IntCal20, 95% confidence interval, n=2, OxA-19159 (W2.20/123), OxA-19160 (W2.20/187), Table 1) (Jacobi and Higham, 2011). Together these dates span the onset and establishment of the Late Glacial Interstadial, a major global climate transition characterised by rapid warming (Jacobi and Higham, 2011). Ancient DNA and archaeological evidence from northern Europe indicate this period also witnessed a major human population turnover, alongside changes in mobility patterns, settlement structure, subsistence economy, technology and social organisation (Holzkämper et al. 2014; Miller, 2012; Pettitt and White, 2012; Maier, 2015; Naudinot et al. 2017; Fu et al. 2016; Posth et al. 2016; Charlton et al. submitted).

Understanding the ecological context of human activity at King Arthur's Cave is particularly important for understanding the subsistence strategies, mobility/settlement patterns, and landscape experiences of these early colonising populations. The site was first discovered and excavated by Symonds in the 1870's (Symonds 1871) and further examined by the University of Bristol Speleological Society between 1925 and 1929, and then again in 1952 (Hewer, 1925, 1926; Taylor, 1928; ApSimon et al. 1992). Most recently, excavations were undertaken in the 1990s by Barton (Barton, 1995; 1996; 1997). Some material from the early excavations, which lacked the detailed stratigraphic analysis of modern excavations (Flas, 2011; Hublin, 2015), has so far been largely unstudied; lack of stratigraphic context for this material means it has been assumed to provide limited insight into the archaeology at the site. However, this view is transforming as new biomolecular techniques are developed which can unlock information from old archaeological collections, such as those from King Arthur's Cave. In particular, ZooMS (Zooarchaeology by Mass Spectrometry) is a proteomics approach that can be used to identify morphologically unidentifiable bone fragments. Family/genus/species level information can be gained from protein amino acid sequence variation assessed through peptide mass fingerprinting. Establishing taxonomic identifications on bone fragments previously thought to be 'unidentifiable' provides a more complete picture of faunal assemblage composition, enabling any difference in the species representation between morphologically identifiable and unidentifiable bones to be considered in relation to human subsistence behaviour. In addition, ZooMS analyses may identify additional human remains at an archaeological site. This is of particular value in later Pleistocene and early Holocene contexts in the British Isles, where discoveries of such specimens have so far been extremely infrequent. Here we use ZooMS to explore the taxonomic identification of previously unidentified bone fragments from King Arthur's Cave and undertake further radiocarbon dating to clarify the chronology of the site stratigraphy. Through these analyses we aim to provide further insight into the faunal community and local ecology during the Late Upper Palaeolithic and Mesolithic human occupation of the cave.

Sample provenance

A total of 57 unidentified bone fragments were sampled for ZooMS for this study. Of these, 48 were recorded as being from the "1st Hearth and Humus" and 10 were recorded as being from the "Yellow Rubble and Mammoth Layer" from the platform from the University of Bristol Speleological Society excavations between 1925 and 1929. The platform supposedly consisted of six discrete Late Pleistocene/Holocene layers (Taylor, 1928). Radiocarbon dates from the platform are given in table 1. A summary of the archaeology and morphologically identified fauna from the stratigraphic levels of interest is given below.

The Humus (unit 1) consisted of a grey-black humic soil which rested on unit 2b (1st Hearth) at the mouth of the cave and further out on the Yellow Rubble (ApSimon et al. 1992). The Humus layer, which is Holocene in origin, was contaminated with Pleistocene material from an overlying old spoil heap from previous excavations. Due to the mixture of Holocene and Pleistocene fauna in this layer, material from this horizon was not included in the zooarchaeological report for the site (ApSimon et al. 1992).

The 1st Hearth (unit 2b) consisted of a blackish soil with weathered limestone clasts and much ash (ApSimon et al. 1992). However, the age of the deposit is somewhat unclear. The lithics recovered from the 1st Hearth are reported to be typical of Mesolithic industries that include microliths. Numerous cut and chop marks on the bone show undoubted evidence of human activity. The 1st Hearth faunal assemblage primarily comprised red deer, aurochs and pig, with roe deer and horse also present, suggesting an early Holocene / Mesolithic age (ApSimon et al. 1992). The presence of some sheep in the faunal assemblage is suggestive of a Neolithic or later date (ApSimon et al. 1992). One brown bear carpal was also identified in this unit, which could be of Pleistocene or Holocene age (ApSimon et al. 1992). The dating of a horse tooth from this unit to 14895 - 14230 cal. BP (IntCal20, 95% confidence interval) (OxA-V-2797-

24C, 12,410 ± 50 BP, Reade et al. 2020), alongside undated reindeer and spotted hyaena bones demonstrate Pleistocene material of both pre- and post-LGM age are also present within unit 2b, although it is noted that the sample labels on both the reindeer and spotted hyaena specimens suggest they may have come instead from the old spoil heap (ApSimon et al. 1992). Horse teeth from this unit were found in its lower part and were different in condition compared to the other bones, suggesting they may well have been incorporated into unit 2b from the underlying Yellow Rubble (Unit 2c) (ApSimon et al. 1992).

The majority of the faunal remains from the Yellow Rubble (Unit 2c) were red deer, with some horse present. Domestic species present in overlying layers were absent in this unit, except for a single pig tooth which was probably intrusive. Reindeer, red fox, arctic or collared lemming, northern and tundra voles, steppe pika and arctic hare were also present (ApSimon et al. 1992). There are contradictions in reporting the contexts of part of the archaeological material which presently appear irresolvable (Jacobi and Higham, 2011), but it does appear that lithics typical of late Magdalenian (known locally as Creswellian) and Federmessergruppen industries were recovered from the Yellow Rubble, as well as the underlying 2nd Hearth (Unit 2d) and Mammoth Layer (Unit 3c). At the time of excavation human remains were reported to have been present in the Yellow Rubble but these have since been lost (ApSimon et al. 1992).

The 2nd Hearth (Unit 2d) sits below the Yellow rubble (2c) on the platform outside the cave. The a blackish sediment with weathered limestone clasts contained great quantities of ash but no identifiable charcoal, burnt bone or hearth structures (ApSimon et al. 1992). The unit was very rich in bones, which showed evidence of butchery but were more fragmented than those from the Yellow Rubble (ApSimon et al. 1992). The 2^{ndt} Hearth faunal assemblage comprised horses, red deer, and bovids. A single red deer tooth from this layer dates to 14840- 13790 cal. BP (IntCal20, 95% confidence interval) (OxA-1563, 12,210 ± 120 BP) (Hedges et al 1989).

The Mammoth Layer (Unit 3c), separated from the Yellow Rubble (Unit 2c) by the 2nd Hearth (Unit 2d), was so named due to the discovery of a juvenile mammoth tooth and was initially assumed to date to before LGM (Hedges et al. 1989). However, radiocarbon dating of faunal remains from the layer shows a mixed assemblage containing both pre- and post-LGM material (Hedges et al. 1989; Jacobi and Higham, 2011). The faunal assemblage from this layer comprised horse, spotted hyaena, brown bear, mammoth, woolly rhinoceros, red deer, and large bovid (*Bos/Bison*). Radiocarbon dates from the Yellow Rubble and Mammoth Layer show some mixing of faunal material between the layers (Table 1). Three groupings can be seen in the radiocarbon dates; horse teeth and red deer bones both date to the Late Glacial, forming two separate groupings, and mammoth dates to at least 36,000 cal. BP (Figs 2 and 3, Table 1). Heavy gnawing of some of the faunal remains along with a lack of evidence of butchery or known pre-LGM lithic technology suggest that the pre-LGM faunal material was accumulated by hyaenas rather than humans (ApSimon et al. 1992). It should be noted that the radiocarbon determinations on the mammoth specimens likely represent minimum ages due to the pre-treatment protocols and radiocarbon procedures used in the 1980s.

Radiocarbon methodology

Six new radiocarbon determinations were obtained, four from the Yellow Rubble and two from the Mammoth Layer. The collagen extraction and dating for three of the specimens was undertaken at Oxford Radiocarbon Accelerator Unit (ORAU) using their standard procedures (Brock et al. 2010). For three specimens, collagen was extracted at UCL following the same procedure and the sample was subsequently radiocarbon dated at ORAU. To denote the bone pretreatment at UCL rather than at ORAU, the measured date was given "OxA-V-wwww-pp" numbers, where "wwww" indicates the wheel number, and "pp" is the position of the sample on the wheel (Brock et al. 2010). A background correction was applied to these dates (OxA-V-2754-50C, OxA-V-2797-25C, OxA-V-3058-28C) to account for the collagen extraction being performed at UCL, following the method outlined by Wood et al. (2010). Corrected dates are denoted by adding a "C" to the end of the date code assigned by ORAU. Results are reported as uncalibrated radiocarbon dates (^{14}C BP) and discussed as calibrated dates BP (cal. BP, 95% confidence interval). Date calibration was performed using OxCal 4.4 (Bronk Ramsey, 2020) and the IntCal20 dataset (Reimer et al. 2020).

ZooMS methodology

Collagen peptide fingerprints were obtained following non-destructive collagen extraction methods (Buckley et al. 2009; van Doorn et al. 2011). Between 10 and 20mg of bone were soaked overnight in 100 µl of 50 mM Ammonium Bicarbonate (AmBic). The supernatant was discarded and samples were gelatinised in 100 µl 50 mM AmBic for 1h at 65°C. When this protocol failed to provide reliable fingerprints, collagen extraction was performed using an HCl pretreatment (Welker et al. 2016). Samples were demineralised in 0.6 M HCl at 4°C, rinsed with 50 mM AmBic, and incubated in 0.1 M NaOH for 5 min. After another rinse with 50 mM AmBic, gelatinisation was performed as previously described. Samples were then incubated overnight at 37°C with 0.4 µg of sequencing grade modified trypsin (Promega). Following trypsin digestion, samples were acidified with 0.5% trifluoroacetic acid (TFA) and purified using Pierce™ 100 µl C18 resin Tips (Thermo Scientific) using conditioning and eluting solutions composed of 50% acetonitrile and 0.1% TFA. Collagen was eluted in 50 µL.

For MALDI-TOF-MS, 0.5 µL of the trypsin-digested extract was spotted with 0.5 µL of α-cyano-hydroxycinnamic acid matrix solution (0.1% TFA in ACN/H2O 1:1 v/v) onto a 48 spot MALDI target plate, and air dried. MALDI-MS analyses were carried out in triplicate on a Shimadzu MALDI 8020 instrument, operating at up to 2000 laser shots per plate spot, over a m/z range of 900-4000. The mass spectra were calibrated against an adjacent MS standard spot containing eight calibrant peptides (TOFMix™) of 0.8 to 3.7 kiloDalton (kDa) range (Bradykinin 1-7, angiotensin II, angiotensin I, Glu1-fibrinopeptide B, N-acetyl Renin substrate, ACTH 1–17 clip, ACTH 18–39 clip and ACTH 7–38 clip) – of which seven were used (1.0 – 3.7 kDa range). The obtained collagen fingerprints were manually inspected for the presence of relevant peptide markers (α1 508 – α2 757; Brown et al. 2020) in mMass v. 5.5.0 (Strohalm et al. 2010), after filtering peaks with a signal-to-noise ratio (S/N) threshold of 3.5, and using previously published collagen peptide markers from reference spectra (Buckley et al. 2009, 2017; Welker et al. 2016).

Results and discussion

Four new radiocarbon determinations were obtained on bones from the Yellow Rubble. A red deer bone (OxA-21183, W2.20/127) was dated to 14845 to 14140 cal. BP, and a humanly fractured horse tooth (OxA-19165, W2.21/451) to 14975 to 14285 cal. BP. Both dates are consistent with previously published dates for this layer (Jacobi and Higham, 2011). Two further dates on horse bones, showing no evidencing of butchery or other anthropogenic modifications, were also obtained. These dated to 12,835 to 12,715 cal BP (OxA-V-2797-25C, W2.20/147) and 15,015 to 14,490 cal BP (OxA-V-3058-28C, W2.20/148). The former is significantly younger, while the latter is consistent with the range of other dated horse remains from the site. From the Mammoth Layer fauna, two radiocarbon determinations were obtained. A culturally fractured horse tooth (OxA-V-2754-50C, W2.21/726) was dated to 14,995 to 14,305 cal. BP and a red deer bone (OxA-21184, W2.20/354) to 14,175 to 13,805 cal. BP. The horse date is consistent with the other dated horse remains from both the Mammoth Layer and Yellow Rubble, and the red deer date is consistent with the other dated red deer remains, all of which are from the Yellow Rubble (Table 1). All horse and red deer post-date the LGM; this contrasts with all mammoth, which pre-date LGM. The six new radiocarbon determinations confirm the previous observed pattern of an age difference between horse and red deer at the site, related to at least two separate episodes of Late Upper Palaeolithic human activity (Jacobi and Higham, 2011). While we also find that one horse post-dates the red deer material, there is no evidence linking this particular bone to any human activity at the site. Despite some overall stratigraphic structure in the radiocarbon dates, it is clear that there has been a significant amount of mixing of material between stratigraphic units.

The results of the ZooMS analyses are presented in Table 3. For some fragments it was possible to use the collagen peptide fingerprints to identify the bone to a single genus/species, but for others it was only possible to restrict the identification to a range of genera. This information can, however, be considered in light of species' known biogeography and the morphologically identified species present at the site. Of the 57 fragments analysed for ZooMS, 27 from the Humus/1st Hearth Layer and 4 from the Yellow Rubble/Platform layer were identified to the genera *Alces, Cervus, Megaloceros,* or *Saiga*. While all these genera are possible based on their known biogeography, some of these identifications are more likely than others. The 1st Hearth morphologically identifiable faunal assemblage primarily comprised red deer (*Cervus elaphus)*, which were also present in the Yellow Rubble and the Mammoth Layer. Thus, while it is likely that the ZooMS identified specimens are *Cervus* rather than the other genera, none can be ruled out. Specimens of giant deer (*Megaloceros giganticus*) were found in the morphologically identifiable material, although they came from the Lower Cave Earth from the Symmond's excavations within the cave and are likely to be older that the Mammoth Layer and pre-LGM in age (ApSimon et al. 1992). However, as a small number of *Megaloceros* specimens recovered from Lancashire, Isle of Man, Scotland and Ireland have been dated to the Late Glacial, and from Devon, South Wales and Ireland to 47,000 cal. to 27,000 BP (Lister et al. 2019), we cannot rule out the presence of the species in the late glacial King Arthur's Cave assemblage. Elk (*Alces alces*) were not present in the morphologically identifiable faunal assemblage at King Arthur's Cave, but a handful of *Alces* specimens dated to the Late Glacial / early Holocene are known from Lancashire, Cumbria, Yorkshire and Berkshire (Healy et al. 1992; Kaagan, 2000; Hedges et al. 1987; Jacobi and Higham, 2009; Smith et al. 2013). Like *Alces, Saiga* was not found in the morphologically identifiable material, however a few specimens of Saiga (*Saiga tatarica*) dated to the Late Glacial have been identified in the Mendip Hills in Somerset (Currant and Jacobi, 2011; Gillespie et al. 1985; Hedges et al. 1989). A further 4 fragments from the Humus/1st Hearth Layer were identified via ZooMS to the above genera but could also be *Caprelous* (Roe deer), which was present in the morphologically identifiable fauna from the same contexts. Three fragments from the Humus/1st Hearth Layer were identified as *Bos/Bison*, and one as *Equus,* which is consistent with the presence of aurochs and horse in the morphologically identifiable fauna. One fragment from the Mammoth Layer was also identified as *Equus*, again consistent with the faunal assemblage from this context. Three fragments were identified via ZooMS as Suidae and one as Bovidae, either *Rupicapra* (chamois) or *Ovis* (domestic sheep). The Suidae could be wild boar which would likely be Mesolithic/early Holocene in age, or alternatively domestic pig and date to the Neolithic/middle Holocene. As *Rupicapra* are not known from Late Pleistocene or Holocene contexts in Britain it seems much more likely the latter fragment comes from a domestic sheep. One fragment from the Humus/1st Hearth Layer was identified as Rhinocerotidae and in the context of Late Pleistocene Britain, this would be a woolly rhino (*Coelodonta antiquitatis*). This woolly rhino likely dates to the pre-LGM period as this species is not known from any reliable stratigraphic context during or after the LGM (Stuart and Lister, 2012). One fragment from the Humus/1st Hearth Layer was identified as a carnivore, either *Crocuta* or *Panthera*; both are present in the morphologically identifiable faunal assemblage from the site (ApSimon et al. 1992). Finally, one fragment from the Humus/1st Hearth Layer was identified as human (see below for further discussion).

Overall, the ZooMS results reinforce the picture of a mixed stratigraphy that includes both Pleistocene and early Holocene fauna. The extent to which this mixing represents more historic post-depositional processes versus more recent contamination from the old spoil heap of previous excavations is unclear. However, additional radiocarbon dating and stable isotope analysis of the assemblage may further resolve the age of individual animals and the ecological setting they inhabited (e.g. Stevens et al. 2021).

The identification of a human amongst the bone fragments from the Humus/1st Hearth Layer is of particular interest. It is possible that the human fragment could be early Holocene in age due to the presence of Mesolithic lithics in unit 2b (1st Hearth). Alternatively, the human bone could be Late Upper Palaeolithic in age as a horse recovered from the 1st hearth has been dated to 14,895 to 14,230 cal. BP (OxA-V-2797-24C). Furthermore, H. Taylor recorded 21 (possibly 22) fragments of human remains from the Yellow Rubble layer (unit 2C) (UBSS catalogue entries, found 23 Sept 1929). Taylor's 1952 faunal list notes them without suggesting disturbance, thus they could have been of Mesolithic or Palaeolithic age (Ap Simon et al. 1992). These specimens are now sadly lost (Ap Simon et al. 1992). This most likely occurred during the Second World War when a bomb landed on the UBSS Museum, and a large amount of the collections were destroyed. Given that Late Upper Palaeolithic human remains have only been recovered from three other sites in the British Isles (Gough's Cave, Sun Hole Cave and Kendrick's Cave), the discovery of a Late Upper Palaeolithic specimen would be an exciting find, particularly because ancient DNA studies at Gough's Cave and Kendrick's Cave have shown that two genetically distinct human populations were present in the British Isles around this time (Charlton et al. In review). However, more recent human remains, dating to 5,592 to 5,411 cal. BP (OxA-5863, 4,670± 60 ^{14}C BP, Hedges et al. 1997) have previously been recovered from King Arthur's Cave. Thus, there is the distinct possibility that this human bone fragment is Holocene in age. The specimen is currently being AMS radiocarbon dated and we eagerly await the results.

Erratum

After this paper was accepted for publication, the result of the AMS date for the human bone showed the specimen is Neolithic in age, dating to 3,830 to 3,590 cal. BP (IntCal20, 95% confidence interval) (OxA-V-3138-28C, 3440 ±20 ^{14}C BP).

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Figure 1: Location of King Arthur’s Cave.

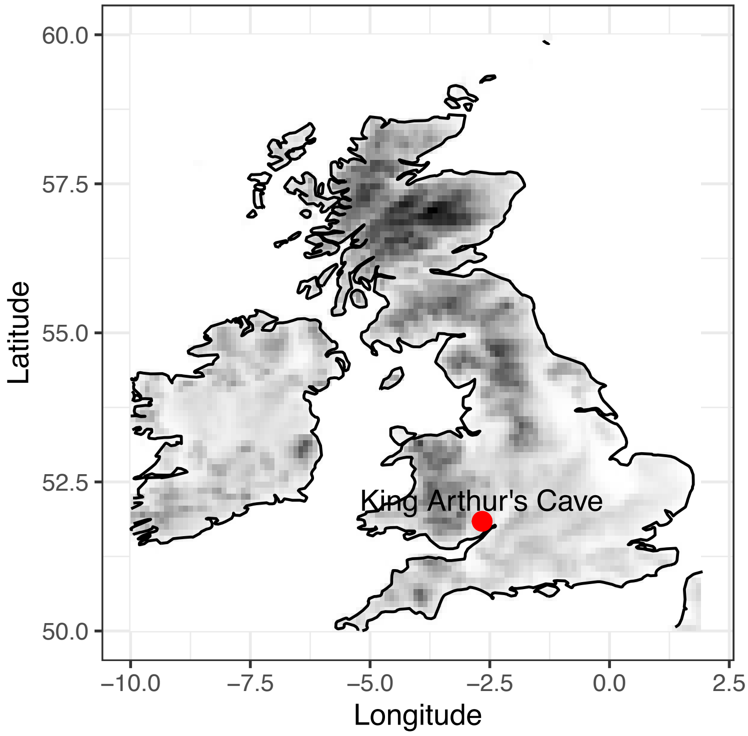

Figure 2: Calibrated post-Last Glacial Maximum (LGM) radiocarbon dates from the platform areas of King Arthur's Cave. Blue = *Equus ferus*, Grey = *Cervus elaphus.* The radiocarbon ages are compared against the NGRIP $\delta^{18}O$ ice core record.

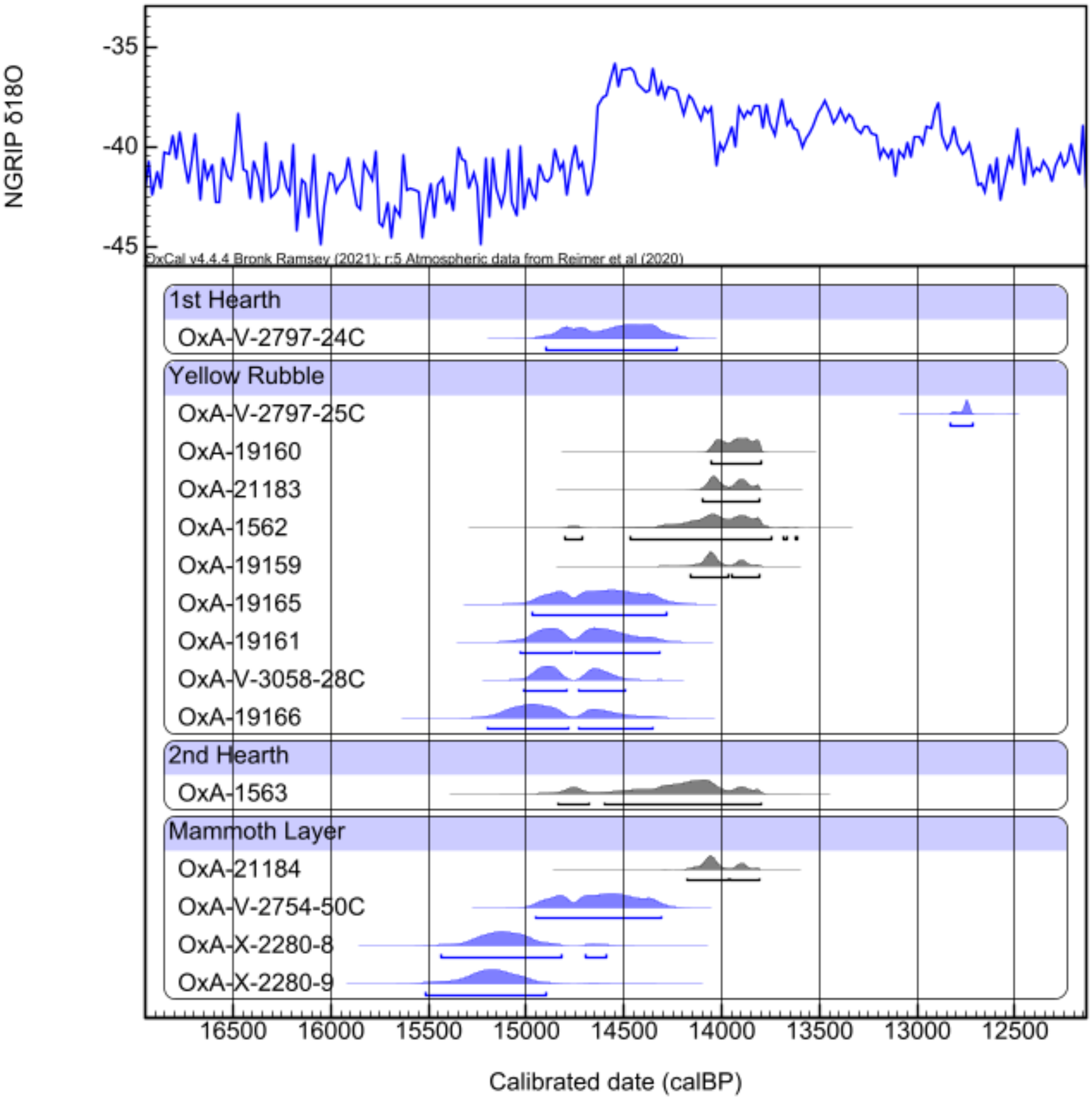

Figure 3: Calibrated pre- and post- Last Glacial Maximum (LGM) radiocarbon dates from the platform areas of King Arthur's Cave. Blue = *Equus ferus*, Red = *Mammuthus primigenius*, Grey = *Cervus elaphus.* The radiocarbon ages are compared against the NGRIP $\delta^{18}O$ ice core record.

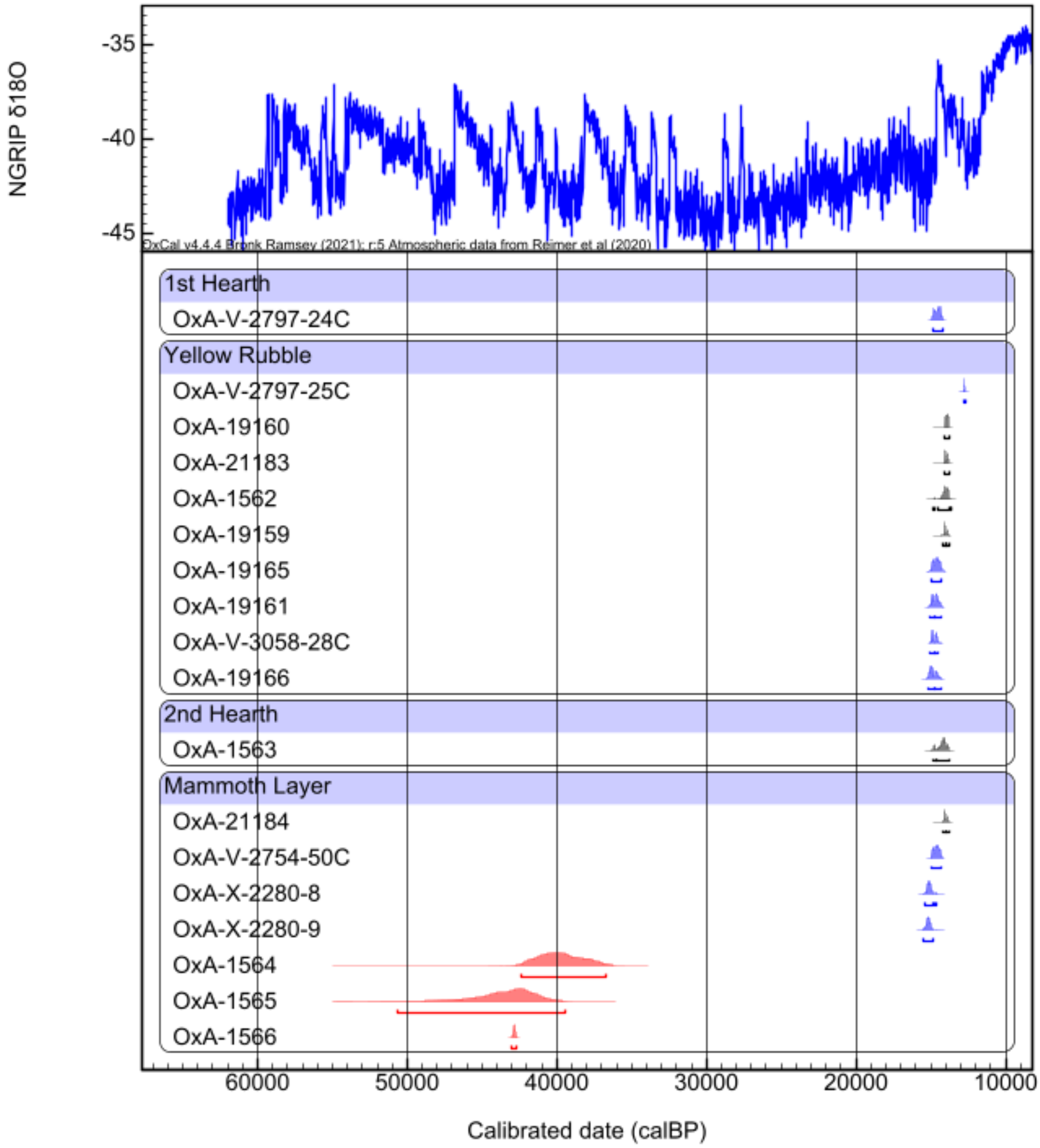

Figure 4: ZooMS identification of bone fragments. Grey = Humus / 1st Hearth, Black = Yellow Rubble / Mammoth Layer.

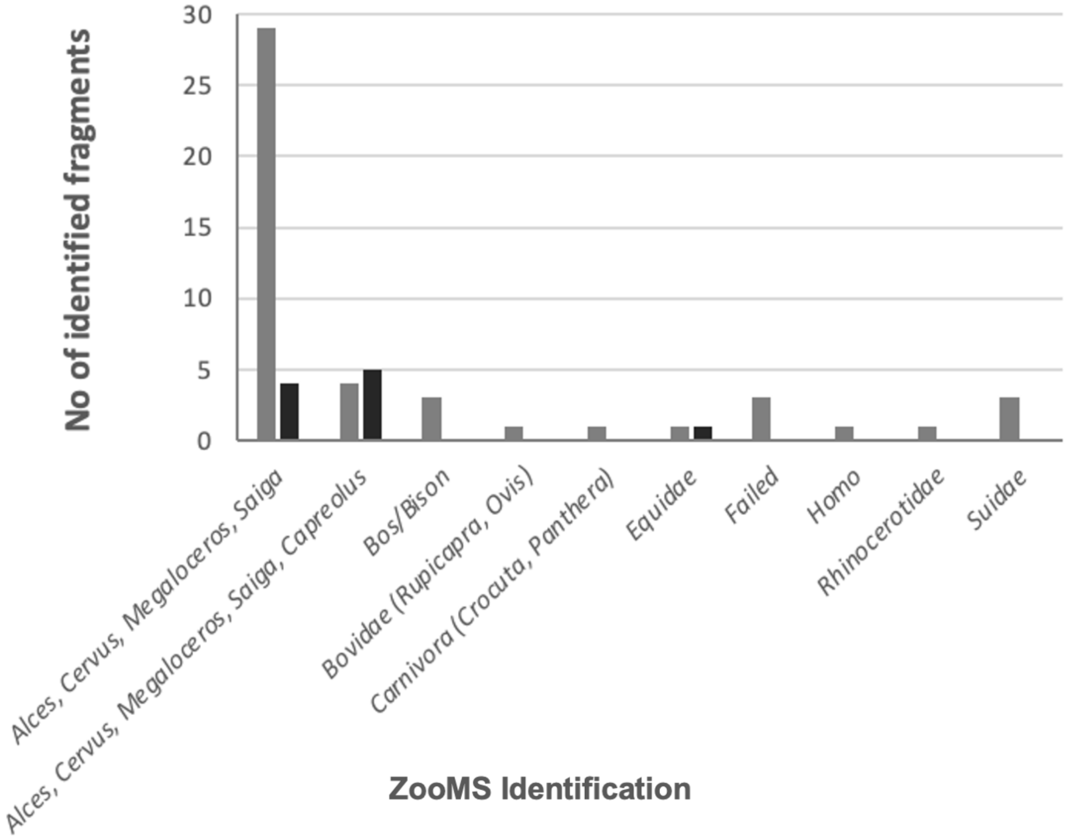

Table 1. Radiocarbon dates from the platform area of King Arthur's Cave. Further radiocarbon dates have been obtained from other areas of the site. Details of pre-treatment codes and protocols can be found in Brock et al 2010 and Jacobi et al 2006.

Species	**Element**	**Sample information**	**Stratigraphic unit**	**Lab code**	**Date**	**Uncertainty**	**Radiocarbon pre-treatment type**	**Date reference**
Equus ferus	?Left upper M3	W2.21/285	1st Hearth	OxA-V-2797-24C	12410	50	AF	Reade et al. 2020
Equus ferus	Sesamoid	W2.20/147	Yellow Rubble	OxA-V-2797-25C	10810	50	AF	This paper
Cervus elaphus	Innominate, right, cut	W2.20/187	Yellow Rubble	OxA-19160	12055	55	AF	Jacobi & Higham, 2011
Cervus elaphus	Bone	KAC 10 W2.20/127.	Yellow Rubble	OxA-21183	12110	55	AF	This paper
Cervus elaphus	Tooth	W.2.21/468	Yellow Rubble	OxA-1562	12120	120	AC	Hedges et al. 1989
Cervus elaphus	Dentary, partial right, cut	W2.20/123	Yellow Rubble	OxA-19159	12140	50	AF	Jacobi & Higham, 2011
Equus ferus	M3, left upper, fractured	W2.21/451	Yellow Rubble	OxA-19165	12450	60	AF	This paper
Equus ferus	P2, right lower, fractured	W2.21/485	Yellow Rubble	OxA-19161	12490	60	AF	Jacobi & Higham, 2011
Equus ferus	oesamoid	W2.20/148	Yellow Rubble	OxA-V-3058-28C	12507	31	AF	This paper
Equus ferus	M1/M2, right lower, fractured	W2.21/484	Yellow Rubble	OxA-19166	12565	80	AF	Jacobi & Higham, 2011
Cervus elaphus	Tooth	W.2.21/115	2nd Hearth	OxA-1563	12,210	120	AC	Hedges et al. 1989
Cervus elaphus	Bone	KAC 11 W2.20/354.	Mammoth Layer	OxA-21184	12145	55	AF	This paper
Equus ferus	Lower left P3/P4, fractured	W2.21/726	Mammoth Layer	OxA-V-2754-50C	12450	50	AF	This paper
Equus ferus	Cheek tooth, left lower, fractured	W2.21/559	Mammoth Layer	OxA-X-2280-8	12680	90	AF	Jacobi & Higham, 2011
Equus ferus	M1/M2, right lower, fractured	W2.21/560	Mammoth Layer	OxA-X-2280-9	12720	90	AF	Jacobi & Higham, 2011
Mammuthus primigenius	Tooth	W.2.21/169	Mammoth Layer	OxA-1564	34850	1500	AC	Hedges et al. 1989
Mammuthus primigenius	Tooth	W.2.21/1185	Mammoth Layer	OxA-1565	38500	2300	AC	Hedges et al. 1989
Mammuthus primigenius	Tooth	W.2.21/954	Mammoth Layer	OxA-1566	>39500		AC	Hedges et al. 1989

Table 2: Results of ZooMS identification of bone fragments from King Arthur's Cave platform area.

ZooMS Identification	No. of identified fragments: Humus / 1st Hearth	No. of identified fragments: Yellow Rubble / Mammoth Layer
Alces, Cervus, Megaloceros, Saiga	29	4
Alces, Cervus, Megaloceros, Saiga, Capreolus	4	5
Bos/Bison	3	
Bovidae *(Rupicapra, Ovis)*	1	
Carnivora *(Crocuta, Panthera)*	1	
Equidae	1	1
Failed	3	
Homo	1	
Rhinocerotidae	1	
Suidae	3	
Total	**47**	**10**

Table 3: ZooMS results. Columns P1 to G’ indicate identified peaks in the mass spectra. ZooMS identification is based on these peaks

		P1	A	A'	B	C	P2	D	E	F	F'	G	G'	
Sample ID	**Context**	**α1 508**	**α2 978**	**α2 978 (+16)**	**α2 484**	**α2 502**	**α2 292**	**α2 793**	**α2 454**	**α1 586**	**α1 586 (+16)**	**α2 757**	**α2 757 (+16)**	**ZooMS ID**
Z01	Humus / 1st Hearth	1105.6	1182.7		1427.7	1550.8		2145.1		2882.7 - *shifted by 1 amu*		2998.8 - *shifted by 1 amu*		Equidae
Z02	Humus / 1st Hearth	1105.7	1180.7	1196.7	1427.7	1550.7	1648.7	2131.0		2883.1	2899.1		3033.2	*Alces, Cervus, Megaloceros, Saiga*
Z03	Humus / 1st Hearth	1105.6	1182.6		1453.7	1550.6		2145.7					2999.9	Rhinocerotidae
Z04	Humus / 1st Hearth	1105.5	1180.4	1196.6	1427.7	1550.8	1648.9	2131.1		2882.7		3017.4		*Alces, Cervus, Megaloceros, Saiga*
Z05	Humus / 1st Hearth	1105.5	1192.6	1208.6	1427.6	1580.7	1648.7	2131.1		2853.4		3017.8		*Bos/Bison*
Z06	Humus / 1st Hearth	1105.6	1192.6	1208.6	1427.7	1580.8	1648.8	2131.1	2853.6	2869.6		3017.4		*Bos/Bison*
Z07	Humus / 1st Hearth	1105.5	1180.5	1196.6	1427.7	1550.8	1648.9	2131.1	2792.5	2882.5 - *shifted by 1 amu*	2898.4 - *shifted by 1 amu*		3032.6 - *shifted by 1 amu*	*Alces, Cervus, Megaloceros, Saiga*
Z08	Humus / 1st Hearth	1105.5	1180.6	1196.6	1427.7	1550.7	1648.8	2131.0		2882.4 - *shifted by 1 amu*	2898.5 - *shifted by 1 amu*	3016.4 - *shifted by 1 amu*	3032.5 - *shifted by 1 amu*	*Alces, Cervus, Megaloceros, Saiga*
Z09	Humus / 1st Hearth	1105.6		1196.6	1427.7	1550.8	1648.8	2131.1	2792.3	2883.5	2899.5	3017.5	3033.6	*Alces, Cervus, Megaloceros, Saiga*
Z10	Humus / 1st Hearth	1105.2			1427.4	1550.5	1648.6							*Failed*
Z11	Humus / 1st Hearth	1105.5		1196.6	1427.7	1550.7	1648.8	2131.0		2882.3 - *shifted by 1 amu*	2898.4 - *shifted by 1 amu*	3016.7 - *shifted by 1 amu*	3032.4 - *shifted by 1 amu*	*Alces, Cervus, Megaloceros, Saiga*
Z12	Humus / 1st Hearth	1105.6		1196.6	1427.8	1550.8	1648.9	2130.7						*Alces, Cervus, Megaloceros, Saiga*
Z13	Humus / 1st Hearth	1105.6		1196.6	1427.6	1550.5	1648.5	2130.5		2881.7 - *shifted by 1 amu*		3016.2 - *shifted by 1 amu*	3032.2 - *shifted by 1 amu*	*Alces, Cervus, Megaloceros, Saiga*

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		P1	A	A'	B	C	P2	D	E	F	F'	G	G'	
Sample ID	**Context**	**α1 508**	**α2 978**	**α2 978 (+16)**	**α2 484**	**α2 502**	**α2 292**	**α2 793**	**α2 454**	**α1 586**	**α1 586 (+16)**	**α2 757**	**α2 757 (+16)**	**ZooMS ID**
Z14	Humus / 1st Hearth	1105.9	1180.9	1196.9	1428.0	1551.0	1649.0	2131.1						*Alces, Cervus, Megaloceros, Saiga*
Z15	Humus / 1st Hearth	-	-	-	-	-	-	-	-	-	-	-	-	*Failed - No collagen*
Z16	Humus / 1st Hearth	1105.5			1427.7	1550.7	1648.8	2131.0		2883.4	2899.4	3017.4		*Alces, Cervus, Megaloceros, Saiga*
Z17	Humus / 1st Hearth	1105.7	1207.7		1453.8	1566.8		2146.9		2853.5				Carnivora (*Crocuta, Panthera*)
Z18	Humus / 1st Hearth	1105.6		1208.7	1427.7	1580.7	1648.7	2130.9		2853.1				*Bos/Bison*
Z19	Humus / 1st Hearth	1105.5		1196.6	1427.7	1550.8	1648.9	2131.1		2882.3 - *shifted by 1 amu*	2898.3 - *shifted by 1 amu*	3017.4		*Alces, Cervus, Megaloceros, Saiga*
Z20	Humus / 1st Hearth	1105.4	1150.4	1166.5	1427.5	1580.5	1648.6	2130.6			2899.0	3017.9		*Failed - mixed signal (A-A': Rangifer / F'-G: Rupicapra, Ovibos)*
Z21	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.7	1550.7	1648.8	2131.2	2792.3	2883.4	2899.4	3017.4	3033.4	*Alces, Cervus, Megaloceros, Saiga*
Z22	Humus / 1st Hearth	1105.4	1180.5	1196.6	1427.7	1550.8	1648.9	2131.2		2882.9	2898.9	3017.7		*Alces, Cervus, Megaloceros, Saiga*
Z23	Humus / 1st Hearth	1105.5	1180.6	1196.6	1427.7	1550.8	1648.9	2131.1		2882.6		3017.5		*Alces, Cervus, Megaloceros, Saiga*
Z24	Humus / 1st Hearth	1105.5		1196.5	1427.7	1550.7	1648.8	2130.9				3017.4		*Alces, Cervus, Megaloceros, Saiga*
Z25	Humus / 1st Hearth	1105.8	1180.9	1196.9	1428.0	1551.0	1649.1	2131.3						*Alces, Cervus, Megaloceros, Saiga*
Z26	Humus / 1st Hearth	1105.5		1196.5	1427.7	1550.7	1648.8	2131.0		2882.7				*Alces, Cervus, Megaloceros, Saiga*

		P1	A	A'	B	C	P2	D	E	F	F'	G	G'	
Sample ID	**Context**	**α1 508**	**α2 978**	**α2 978 (+16)**	**α2 484**	**α2 502**	**α2 292**	**α2 793**	**α2 454**	**α1 586**	**α1 586 (+16)**	**α2 757**	**α2 757 (+16)**	**ZooMS ID**
Z27	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.7	1550.7	1648.8	2131.1				3017.3		*Alces, Cervus, Megaloceros, Saiga*
Z28	Humus / 1st Hearth	1105.5	1180.5	1196.5	1427.7	1550.8	1648.8	2131.1				3017.5		*Alces, Cervus, Megaloceros, Saiga*
Z29	Humus / 1st Hearth	1105.5			1427.6	1550.6	1648.7	2131.2	2792.2	2883.2	2899.2	3017.0	3033.2	*Alces, Cervus, Megaloceros, Saiga*
Z30	Humus / 1st Hearth	1105.6			1427.8	1550.9	1648.9	2131.1		2883.3				*Alces, Cervus, Megaloceros, Saiga, Capreolus*
Z31	Humus / 1st Hearth	1105.7	1180.6	1196.6	1427.8	1550.8	1649.0	2130.8					3033.8	*Alces, Cervus, Megaloceros, Saiga*
Z32	Humus / 1st Hearth	1105.8	1180.8	1196.8	1427.9	1550.9	1649.0	2131.3		2883.4			3033.4	*Alces, Cervus, Megaloceros, Saiga*
Z33	Humus / 1st Hearth	1105.5	1180.5	1196.6	1427.5	1550.5	1648.7	2130.8						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
Z34	Humus / 1st Hearth	1105.7	1180.9	1196.8	1428.0	1551.0	1649.0	2131.3		2883.6	2899.6	3017.6	3033.5	*Alces, Cervus, Megaloceros, Saiga*
Z35	Humus / 1st Hearth	1105.5	1180.6	1196.6	1453.7	1550.8	1647.8	2131.1	2820.3	2883.4	2899.4	3017.6	3033.5	Suidae
Z36	Humus / 1st Hearth	1105.5	1180.7	1196.6	1427.7	1550.8	1648.9	2131.2		2883.3			3033.4	*Alces, Cervus, Megaloceros, Saiga*
Z37	Humus / 1st Hearth	1105.3	1180.3	1196.3	1427.2	1550.2	1648.3	2130.0 - *shifted by 1 amu*						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
Z38	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.5	1550.5	1648.6	2130.4 - *shifted by 1 amu*						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
Z39	Humus / 1st Hearth	1105.5	1180.6	1196.6	1427.7	1550.7	1648.8	2131.1		2883.4	2899.5	3017.6	3033.7	*Alces, Cervus, Megaloceros, Saiga*
Z40	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.7	1550.7	1648.8	2131.0	2791.9			3017.8		*Alces, Cervus, Megaloceros, Saiga*

		P1	A	A'	B	C	P2	D	E	F	F'	G	G'	
Sample ID	**Context**	**α1 508**	**α2 978**	**α2 978 (+16)**	**α2 484**	**α2 502**	**α2 292**	**α2 793**	**α2 454**	**α1 586**	**α1 586 (+16)**	**α2 757**	**α2 757 (+16)**	**ZooMS ID**
Z41	Humus / 1st Hearth	1105.6	1180.6	1196.6	1453.6	1550.7		2130.9						Suidae
Z42	Humus / 1st Hearth	1105.6		1235.7	1477.8	1580.9		2114.8	2832.0	2869.2	2885.2	2957.3		Homo
Z43	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.8	1550.8	1648.9	2130.9						*Alces, Cervus, Megaloceros, Saiga*
Z44	Humus / 1st Hearth	1105.5	1180.6	1196.6	1427.7	1550.8	1648.8	2131.1				3018.4 - *shifted by 1 amu*		*Alces, Cervus, Megaloceros, Saiga*
Z45	Humus / 1st Hearth	1105.5	1180.6	1196.6	1427.7	1550.8	1648.8	2131.1		2883.4	3017.5	3033.5		*Alces, Cervus, Megaloceros, Saiga*
Z46	Humus / 1st Hearth	1105.6	1180.7	1196.7	1453.7	1550.8		2131.2	2820.1	2883.2	2899.2	3017.1	3033.1	Suidae
Z47	Humus / 1st Hearth	1105.6	1180.6	1196.6	1427.7	1580.7	1648.6	2131.1	2792.0	2883.1		3016.9	3033.1	Bovidae *(Rupicapra, Ovis)*
ZT-01	Yellow Rubble/ Mammoth Layer	1105.6	1180.6	1196.6	1427.8	1550.8	1648.9	2131.1		2883.6	2899.6	3017.9	3033.7	*Alces, Cervus, Megaloceros, Saiga*
ZT-02	Yellow Rubble/ Mammoth Layer	1105.5	1180.6	1196.6	1427.7	1550.8	1648.8	2131.1		2883.3		3017.2	3033.3	*Alces, Cervus, Megaloceros, Saiga*
ZT-03	Yellow Rubble/ Mammoth Layer	1105.6	1180.6	1196.6	1427.5	1550.5	1648.6	2131.1						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
ZT-04	Yellow Rubble/ Mammoth Layer	1105.6	1180.7	1196.7	1427.8	1550.8	1649.0	2131.2						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
ZT-05	Yellow Rubble/ Mammoth Layer	1105.5	1180.6	1196.6	1427.6	1550.6	1648.6	2130.9		2883.1			3033.3	*Alces, Cervus, Megaloceros, Saiga*
ZT-06	Yellow Rubble/ Mammoth Layer	1105.6		1196.6	1427.7	1550.7	1648.8	2131.1		2882.9		3017.9		*Alces, Cervus, Megaloceros, Saiga*
ZT-07	Yellow Rubble/ Mammoth Layer	1105.4		1196.5	1427.4	1550.5	1648.7	2130.5						*Alces, Cervus, Megaloceros, Saiga, Capreolus*

		P1	A	A'	B	C	P2	D	E	F	F'	G	G'	
Sample ID	**Context**	**α1 508**	**α2 978**	**α2 978 (+16)**	**α2 484**	**α2 502**	**α2 292**	**α2 793**	**α2 454**	**α1 586**	**α1 586 (+16)**	**α2 757**	**α2 757 (+16)**	**ZooMS ID**
ZT-08	Yellow Rubble/ Mammoth Layer	1105.3	1180.4	1196.4	1427.3	1550.3	1648.4	2130.4 - *shifted by 1 amu*						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
ZT-09	Yellow Rubble/ Mammoth Layer	1105.7		1196.7	1427.9	1550.9	1648.7	2131.2						*Alces, Cervus, Megaloceros, Saiga, Capreolus*
ZT-10	Yellow Rubble/ Mammoth Layer	1105.7	1182.7	1198.7	1427.9	1550.9		2145.3		2883.1				Equidae