[Supplementary material]

Recalibrating grave-good chronologies: new AMS radiocarbon dates from Late Bronze Age burials in Lika, Croatia

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AMS radiocarbon dating methods

Samples were analysed using standard procedures for collagen extraction at the Penn State University Human Palaeoecology and Isotope Geochemistry Laboratory. Approximately 800mg of dry bone were taken from each archaeological sample, with compact bone preferentially sampled to maximise collagen yield. Samples were crushed to increase the area of reactive surface, then washed in NanoPure water and demineralised in 0.5N HCl at 5°C for several days. Samples were then prepared for collagen extraction and purification by the modified Longin (1971) method with ultrafiltration (Brown et al. 1988) as outlined elsewhere (Kennett et al. 2017; Zavodny 2017). Poorly preserved collagen samples were processed using a modified Stafford method (Stafford et al. 1988, 1991) for XAD-purification (Lohse et al. 2014). Extracted collagen was gelatinised at 110°C in 1mL 0.02N HCl for 24 hours, then pipetted into a pre-cleaned 10ml disposable syringe with an attached pre-cleaned 0.45µm Millex Durapore PVDF filter and driven into a thick-walled culture tube. The sample gelatin was then hydrolysed in 1.5mL 6N HCl for 24 hours at 110°C before being driven through a SPE column with an additional 10ml 6N HCl and dried under UHP N2 gas while being heated at 50°C for 12 hours. Carbon and nitrogen concentrations and stable isotope ratios were analysed at the Yale Analytical and Stable Isotope Center (YASIC). Sample quality was evaluated with %C and %N. C:N ratios are between 3.3 and 3.4, denoting good preservation for radiocarbon dating (see Table S1; DeNiro 1985; van Klinken 1999).

Radiocarbon samples (~2.5mg) were combusted for three hours at 900°C in vacuum-sealed quartz tubes with CuO and Ag wire at the Penn State Human Paleoecology and Isotope Geochemistry Lab, then sent to the Keck Carbon Cycle AMS (KCCAMS) facility at UC Irvine for graphitisation and measurement following procedures outlined in McClure *et al.* (2014). Results were corrected for isotopic fractionation according to Stuiver and Polach (1977) and calibrated using Oxcal software and IntCal 13 (v. 4.2.3; Bronk Ramsey 2009, 2013; Reimer *et al.* 2013). Conventional radiocarbon ages and error are listed in Tables 1 and S1. Calibrated and modeled date ranges are reported at the 2σ level.

Humans consuming large amounts of organisms from depleted radiocarbon marine or freshwater systems can have artificially old dates (Stuiver *et al.* 1986; Lanting & van der Plicht 1998; Ascough *et al.* 2005; Culleton 2006; Keaveney and Reimer 2012). Quantifying this "reservoir effect"—the offset between real and calibrated radiocarbon ages—is essential for chronological studies such as ours. Recent studies have successfully measured regional "reservoir effects" by paired dating of human and terrestrial herbivore bones from the same contexts (e.g. Cook *et al.* 2002; Bonsall *et al.* 2004, 2007; Borić & Miracle 2004; Bronk Ramsey *et al.* 2014).

Unfortunately, the nature of the collections sampled for this study prohibits a similar approach in Lika at this time and so we cannot fully discount the possibility of a freshwater reservoir effect on our AMS radiocarbon dates. However, there is no archaeological or paleodietary evidence of significant human consumption of aquatic resources during the Bronze and Early Iron Ages in Lika (Zavodny *et al.* 2017) and samples from this study have stable carbon and nitrogen isotope values consistent with a strictly terrestrial diet (Table S1). Based on this evidence, we assume that any reservoir effect, if it exists, is minimal and does not significantly change our findings. Similar assumptions about the relationship between terrestrial stable isotope signatures and lack of a freshwater reservoir effect have been tested and confirmed in other areas of the world (e.g. Svyatko *et al.* 2017).

References

ASCOUGH, P., G. COOK & A. DUGMORE. 2005. Methodological approaches to determining the

marine radiocarbon reservoir effect. *Progress in Physical Geography* 29: 532–47. https://doi.org/10.1191/0309133305pp461ra

BONSALL, C., G. COOK, R. HEDGES, T. HIGHAM, C. PICKARD & I. RADOVANOVIĆ. 2004. Radiocarbon and stable isotope evidence of dietary change from the Mesolithic to the Middle Ages in the Iron Gates: new results from Lepenski Vir. *Radiocarbon* 46: 293–300. https://doi.org/10.1017/S0033822200039606

BONSALL, C., K. MCSWEENEY, M. MASSON, T. HIGHAM, C. PICKARD & G. COOK. 2007.

Chronological and dietary aspects of the human burials from Ajdovska Cave, Slovenia.

Radiocarbon 49: 727-40. https://doi.org/10.1017/S0033822200042612

BORIĆ, D. & P. MIRACLE. 2004. Mesolithic and Neolithic (dis)continuities in the Danube Gorges: new AMS dates from Padina and Hajdučka Vodenica (Serbia). *Oxford Journal of Archaeology* 23: 341–71. https://doi.org/10.1111/j.1468-0092.2004.00215.x

BROWN, T., D. NELSON, J. VOGEL & J. SOUTHON. 1988. Improved collagen extraction by modified Longin method. *Radiocarbon* 30: 171–77. https://doi.org/10.1017/S0033822200044118 BRONK RAMSEY, C. 2009. Bayesian analysis of radiocarbon dates. *Radiocarbon* 51: 337–60. https://doi.org/10.1017/S0033822200033865

– 2013. OxCal v.4.2.3. Released 2013 Sept 23. Oxford Radiocarbon Accelerator Unit (ORAU). Available at: https://c14.arch.ox.ac.uk, accessed November 26 2018.

BRONK RAMSEY, C., R. SCHULTING, O. GORIUNOVA, V. BAZALIISKII & A. WEBER. 2014.

Analyzing radiocarbon reservoir offsets through stable nitrogen isotopes and Bayesian modeling: a case study using paired human and faunal remains from the Cis-Baikal region, Siberia.

Radiocarbon 56: 789–99. https://doi.org/10.2458/56.17160

COOK, G., C. BONSALL, R. HEDGES, K. MCSWEENEY, V. BOREONEANT, L. BARTOSIEWICZ & P.

PETTITT. 2002. Problems of dating human bones from the Iron Gates. *Antiquity* 76: 77–85. https://doi.org/10.1017/S0003598X00089821

CULLETON, B. 2006. Implications of a freshwater radiocarbon reservoir correction for the timing of late Holocene settlement of the Elk Hills, Kern County, California. *Journal of Archaeological Science* 33: 1331–39. https://doi.org/10.1016/j.jas.2006.01.013

DENIRO, M.J. 1985. Postmortem preservation and alteration of in vivo bone collagen isotope ratios in relation to palaeodietary reconstruction. *Nature* 317: 806–809.

https://doi.org/10.1038/317806a0

KEAVENEY, E. & P. REIMER. Understanding the variability in freshwater radiocarbon reservoir offsets: a cautionary tale. *Journal of Archaeological Science* 39: 1306–16.

https://doi.org/10.1016/j.jas.2011.12.025

KENNETT, D., S. PLOG, R. GEORGE, B. CULLETON, A. WATSON, P. SKOGLUND, N. ROHLAND, S.

MALLICK, K. STEWARDSON, L. KISTLER, S. LEBLANC, P. WHITELEY, D. REICH & G. PERRY. 2017. Archaeogenomic evidence reveals prehistoric matrilineal dynasty. *Nature Communications* 8: 14115. https://doi.org/10.1038/ncomms14115

LANTING, J. & J. VAN DER PLICHT. 1998. Reservoir effects and apparent ¹⁴C ages. *Journal of Irish Archaeology* 9: 151–65.

LOHSE, J., D. MADSEN, B. CULLETON & D. KENNETT. 2014. Isotope paleoecology of episodic Mid to Late Holocene bison population expansions in the Southern Plains, USA. *Quaternary Science Reviews* 102: 14–26. https://doi.org/10.1016/j.quascirev.2014.07.021

LONGIN, R. 1971. New method of collagen extraction for radiocarbon dating. *Nature* 230: 241–42. https://doi.org/10.1038/230241a0

REIMER, P. *et al.* 2013. IntCal13 and Marine13 radiocarbon age calibration curves 0–50 000 years cal BP. *Radiocarbon* 55: 1869–87. https://doi.org/10.2458/azu_js_rc.55.16947

STAFFORD, T., K. BRENDEL & R. DUHAMEL. 1988. Radiocarbon, ¹³C and ¹⁵N analysis of fossil bone: removal of humates with XAD-2 resin. *Geochimica et Cosmochimica Acta* 52: 2257–67. https://doi.org/10.1016/0016-7037(88)90128-7

STAFFORD, T., P. HARE, L. CURRIE, A. JULL & D. DONAHUE. 1991. Accelerator radiocarbon dating at the molecular level. *Journal of Archaeological Science* 18: 35–72.

https://doi.org/10.1016/0305-4403(91)90078-4

STUIVER, M., & H. POLACH. 1977. Discussion: reporting of ¹⁴C data. *Radiocarbon* 19: 355–63. https://doi.org/10.1017/S0033822200003672

STUIVER, M., G. PEARSON & T. BRAZIUNAS. 1986. Radiocarbon age calibration of marine samples back to 9000 cal yr BP. *Radiocarbon* 28(2B): 980–1021.

https://doi.org/10.1017/S0033822200060264

SVYATKO, S., R. SCHULTING, A. POLIAKOV, N. OGLE & P. REIMER. 2017. A lack of freshwater reservoir effects in human radiocarbon dates in the Eneolithic to Iron Age in the Minusinsk Basin. *Archaeological and Anthropological Sciences* 9: 1379–88. https://doi.org/10.1007/s12520-016-0383-3 VAN KLINKEN, G. 1999. Bone collagen quality indicators for palaeodietary and radiocarbon measurements. *Journal of Archaeological Science* 26: 687–95.

https://doi.org/10.1006/jasc.1998.0385

ZAVODNY, E. 2017. New approaches to cultural change and continuity at the Bronze–Iron Age transition in Lika, Croatia. Unpublished PhD dissertation, The Pennsylvania State University. ZAVODNY, E., B. CULLETON, S. MCCLURE, D. KENNETT & J. BALEN. 2017. Minimizing risk on the margins: insights on Iron Age agriculture from stable isotope analyses in central Croatia. *Journal of Anthropological Archaeology* 48: 250–61. https://doi.org/10.1016/j.jaa.2017.08.004

Site	Psu Lab	Burial	Age &	Element	Pre-	δ ¹³ C	%C	δ1 ⁵ N	%N	C:N	UCIAMS	¹⁴ C BP
	ID	#	Sex		treat ¹	(‰)		(‰)			Lab #	
Kompolje	KO-22	22	Adult	Cranial	UF	-17.3	42.2	8.7	15.2	3.3	169825	2590±20
	KO-119	119	Adult	Tibia shaft	UF	-16.9	46.5	8.4	16.4	3.3	169826	2735±15
	KO-197	197	Adult	Cranial	XAD	-17.3	30.6	8.8	11.1	3.3	172393	2505±20
Konjsko Brdo	KB-01	n/a	Adult F	Rib shaft	UF	-18.0	41.1	8.1	14.3	3.4	158544	2480±20
Plešina Glavica	GKP-06	6	<18	Tibia shaft	XAD	-18.0	20.4	8.6	7.4	3.2	179815	2705±15
	GKP-07	7	Adult	Tibia shaft	XAD	-17.9	21.8	8.5	7.9	3.2	181718	2710±15
Prozor	PR-06	6	Adult	Cranial	UF	-17.6	44.0	10.5	15.8	3.3	169827	2460±15
Smiljan	SM-01	4	Juvenile	Cranial	UF	-18.5	39.9	9.3	14.3	3.3	169831	2560±15
	SM-2A	2	Adult	Long bone shaft	UF	-17.4	43.0	9.1	15.3	3.3	173184	2560±20
	SM-2B	2	Juvenile	Mandible	XAD	-17.8	25.0	8.9	9.0	3.2	172394	2505±20
	SM-17A	17	Adult F	Mandible	UF	-17.1	40.0	8.9	14.5	3.2	169836	2535±15
	SM-17B	17	Juvenile	Tibia shaft	UF	-17.1	44.7	8.1	16.2	3.2	174939	2545±20

 Table S1. Contextual and archaeometric information for study samples.

¹ UF: >30kDa ultrafiltered gelatin; XAD: XAD-purified amino acids