

# New AMS Radiocarbon Dates from Kostenki 12

**Rupert A. Housley<sup>1</sup>, Thomas F.G. Higham<sup>2</sup>, and M. V. Anikovich<sup>3</sup>**

<sup>1</sup>Department of Archaeology, University of Glasgow, Glasgow, UK

<sup>2</sup>Oxford Radiocarbon Accelerator Unit, RLAHA, Oxford, UK

<sup>3</sup>Institute for the History of Material Culture of Russian Academy of Sciences, St. Petersburg, Russia

## Abstract

A new group of AMS <sup>14</sup>C age determinations have been obtained from the Oxford Radiocarbon Accelerator Unit for samples taken from lithological layers 12, 14 and 18 at the site of Kostenki 12. Dated as part of the EFCHED initiative, the new samples come from cultural horizons III, IV and V that underlie the volcanic Y5 Campanian Ignimbrite tephra horizon. Preliminary age correction of the AMS measurements for the effects of fluctuation in <sup>14</sup>C production suggests that the deposits were laid down prior to 40,000 calendar years ago. Comparison with the associated OSL dates of S. L. Forman (2006) shows that whilst there is broad general agreement between the respective dating methods, a significant age offset is still observable suggesting that further chronological investigation remains to be done.

## Introduction

This is a short report on a recent set of AMS radiocarbon age determinations from the lowermost and oldest horizons at Kostenki 12. The samples, all of which comprised of charcoal too small to be botanically characterised, were collected in the summer of 2004 by M V Anikovich and given to the AMS radiocarbon dating facility in Oxford University in 2005 by R A Housley as part of the UK's NERC funded EFCHED (Environmental Factors in the Chronology of Human Evolution and Dispersal) initiative.

The Palaeolithic site of Kostenki 12 is located where the second terrace along the main valley of the Don River merges with the second terrace running along the south side of Pokrovskii Ravine. A series of radiocarbon samples were collected for analysis, of which six have been dated. Two samples (EFD5C505, N1 and EFD5C512, N8) are associated with the *Lower Humic Bed* (LHB), a dark brown calcareous loam, at the base of which lies cultural horizon III (CL III) (Holliday *et al.*, 2006). The Y5 Campanian ignimbrite volcanic eruption horizon is believed to be present in a series of silts that lie between the *Lower Humic Bed* and the *Upper Humic Bed*. This horizon therefore provides a *terminus ante quem* for all the samples dated here. A single sample (EFD5C513, N9) is associated with Unit 1C that comprises of lenses of pale brown and lighter brown loam within which is located cultural horizon IV (CL IV). The remaining three samples (EFD5C514, 516 and 517; N10, N12 and N13 respectively) were collected from Unit 1A, a series of brown and light yellowish brown loam lenses that bear some similarity to a Humic Bed. These three samples are associated with cultural horizon V (CL V).

## Methodology and Results

The charcoal samples were chemically pretreated in Oxford with the routine acid-alkali-acid (AAA) pre-treatment. This is designed to mobilise the two major contaminants present in soils that may affect radiocarbon dating of these types of samples, humic acids and fulvic acids. Both are organic compounds derived from the decayed remains of plants in the surface layers of the soil. Their presence in archaeological charcoals may constitute error of unknown magnitude and is highly site specific. Humic substances within the soil have been classified according to the ease with which they can be removed from soils using alkaline solutions (Head, 1987). Humic acids may be defined as the fraction extracted by alkaline solution that becomes insoluble after acidification (Head, 1987: 144). Fulvic acids are soluble both in acid and alkaline solutions (Head, 1987). The residue soluble and insoluble in alkaline solutions is termed “humin” and is usually the fraction targeted for radiocarbon dating. In all cases, humin carbon was isolated for AMS dating at Oxford. The results are shown in Table X.

## Discussion

The Y5 Campanian ignimbrite tephra has long been a key chronological marker for the early Upper Palaeolithic of the Kostenki-Borshchevo area (Velichko 1961). It is believed to have come from the Camp Flegrei eruptions of southern Italy (Melekestsev *et al.* 1984; Sinitsyn 1996: 279-280). In terms of age the eruption is believed to date to  $39,395 \pm 51$  calendar years BP (based on forty concordant  $^{40}\text{Ar}/^{39}\text{Ar}$  measurements from 20 single crystals of rock: Giaccio *et al.* 2006; see also Fedele *et al.*, 2003; Pyle *et al.*, in press). Giaccio *et al.* (2006) determine this to be equivalent to  $40012_{\text{GISP2}}$  yr BP when compared with that record based on the discovery of the CI signal within the Greenland ice cores. This should mark the time of the ash fall, although there has been some questioning of this age due to the fact that the tephra at Kostenki is not in a primary position, having undergone some reworking. As has been said, all the samples dated here underlie this key marker horizon.

To what extent do the new AMS determinations from Kostenki 12 fit in with the existing chronological framework (Sinitsyn *et al.* 1997; Anikovich 2005) for the site and the region? A previous  $^{14}\text{C}$  determination (GrA-5551:  $36280 \pm 360/350$  BP) from layer 12, CL III, appears to be in reasonable agreement with OxA-15482 ( $35820 \pm 230$  BP). The fact that OxA-X-2158-14 ( $31760 \pm 230$  BP) is younger and from the same context is not that surprising, given the technical difficulties encountered with this sample. Overall we believe the OxA-X results are less reliable than the OxA measurements and so less credence should be given to them in any assessment.

There are no existing  $^{14}\text{C}$  results from layer 14, CL IV, with which to compare to OxA-15555 ( $35540 \pm 260$  BP), however there are an extensive suite of IRSL measurements from the underlying layer 15 made in the Luminescence Dating Research Laboratory of the University of Illinois at Chicago by Steve Forman. These IRSL determinations range from c.  $43.5 \pm 3.6$  to  $50.1 \pm 3.6$  ka in age (Forman 2006). However, an important problem arises when attempting to compare the two datasets for radiocarbon ages are not equivalent to absolute dates produced by other dating methodologies. At present it is not possible to reliably calibrate radiocarbon ages greater than 26 ka cal BP (Reimer *et al.* 2004) because there is no agreement on

which of the various terrestrial and marine datasets that map the variation of  $^{14}\text{C}$  production over time is likely to be the most accurate. Comparing the IRSL measurements with the  $^{14}\text{C}$  chronology is clearly problematic in the absence of an accepted calibration dataset; however, some attempt at preliminary 'correction' must be made if we are to discuss the respective chronologies.

Matters are further complicated by recent reports by Giaccio *et al.* (2006) and Hajdas (personal communication to TFGH) which appear to show that much more substantial increases in the  $^{14}\text{C}$  concentration occurred during the time of the Laschamp geomagnetic excursion (c. 41-39 ka BP on the GISP2 timescale) than have hitherto been recorded (e.g. in the Cariaco Basin record of Hughen *et al.*, 2004). Since the Campanian ignimbrite is deposited at a time that is coeval with the Laschamp event it is clearly a crucial isochronous marker.

We have compared tentatively our radiocarbon determinations from Kostenki 12 with the Cariaco Basin record in a Bayesian model (using OxCal 4.0: Bronk Ramsey 1995; 2001) in which the radiocarbon and IRSL ages from different levels of the site are modelled within the overall dated sequence. The Y5 tephra is located above layer 12, suggesting that all determinations ought to be older than 40012<sub>GISP2</sub> yr BP (after Giaccio *et al.*, 2006). Further work is required but the initial results (figures X1 and X2) show that with the exception of the OxA-X- determinations, which produced much lower carbon yields than expected, the dates obtained from Kostenki are consistent with the age of Y5 tephra, which acts as a *terminus ante quem* in the model. The IRSL measurements appear to be slightly old, especially those in layer 14 (cultural horizon IV). The boundary distribution for the end of layer 12, based on the radiocarbon chronology only, yielded an age range of 41.0-39.5 ka BP at 95% probability on the GISP2 timescale with respect to the Cariaco record. It must be stressed that these results are preliminary and further modelling and dating work is needed to determine whether they are robust.

The three dates from layer 18, CL V, are quite divergent in age. In terms of result OxA-X-2158-15 (34710  $\pm$  330 BP) is not so different from the overlying sample in layer 14, OxA-15555, but it is at odds with the other results from this layer. In terms of pre-treatment though OxA-X-2158-15 is problematic and so is best regarded as no more than a minimum estimate of the true antiquity of the sample. OxA-15556 (41300  $\pm$  450 BP) and OxA-15902 (38410  $\pm$  300 BP) are of major significance since they are amongst the oldest radiocarbon dates associated with the early Upper Palaeolithic in the Kostenki-Borshchevo region. The implications are that the Upper Palaeolithic began significantly earlier on the Russian plains than have hitherto been suspected. There are directly comparable IRSL dates from this context that range from c. 44.2  $\pm$  3.8 ka to 45.3  $\pm$  3.3 ka BP (Forman 2006) and in general the Bayesian model suggests reasonable agreement for layer 18 between the datasets from the two dating methodologies.

In conclusion, the new AMS determinations provide valuable new information with regards to the timing of the earliest Upper Palaeolithic at Kostenki 12 and contribute to an emerging chronology in which different dating methodologies are beginning to be integrated.

## Acknowledgements

The authors would like to thank the Natural Environment Research Council who supported the research by providing grants as part of its wider EFCHED initiative.

## References

- Anikovich, M. V.**, 2005, The chronology of Paleolithic sites in the Kostenki-Borshchevo area. *Archaeology, Ethnology, and Anthropology of Eurasia*, 3 (23), 70-86.
- Bronk Ramsey, C.**, 1995, Radiocarbon calibration and analysis of stratigraphy. The OxCal Program, *Radiocarbon* 37 (2), 425-430.
- Bronk Ramsey, C.**, 2001, Development of the radiocarbon program OxCal, *Radiocarbon* 43 (2A), 355-363.
- Fedele, F. G., Giaccio, B., Isaia, R., & Orsi, G.**, 2003, The Campanian Ignimbrite eruption, Heinrich event 4 and Palaeolithic change in Europe. In: A. Robock and C. Oppenheimer (eds.) *Volcanism and the Earth's Atmosphere. Geophysical Monograph* 139, pp 301-328.
- Forman, S.**, 2006, OSL dating of Kostenki: method and results. In: M. V. Anikovich and N. I. Platonova (eds.), *The Early Paleolithic of Eurasia: General Trends, Local Developments* (International conference devoted to the 125<sup>th</sup> anniversary of the Paleolithic investigations in Kostenki, August 23-26<sup>th</sup>, 2004), pp 125-130, St. Petersburg: Nestor-History.
- Giaccio, B., Hajdas, I., Peresani, M., Fedele, F. G. and Isaia, R.** 2006, The Campanian Ignimbrite tephra and its relevance for the timing of the Middle to Upper Palaeolithic shift. In: N. J. Conard (ed.) *When Neanderthals and Modern Humans Met*. pp 343-375, Kerns Verlag: Tübingen.
- Head, M. J.** 1987, Categorisation of organic sediments from archaeological sites. In: W. R. Ambrose and J. M. J. Mummery (eds.) *Archaeometry: Further Australian Studies*. pp. 143-159, Department of Prehistory, Research School of Pacific Studies, ANU: Canberra.
- Holliday, V. T., Hoffecker, J. F., Anikovich, M. V., and Sinitsyn, A. A.**, 2006, Geoarchaeological studies at Kostenki-Borshchevo. In: M. V. Anikovich and N. I. Platonova (eds.), *The Early Paleolithic of Eurasia: General Trends, Local Developments* (International conference devoted to the 125<sup>th</sup> anniversary of the Paleolithic investigations in Kostenki, August 23-26<sup>th</sup>, 2004), pp 57-80, St. Petersburg: Nestor-History.
- Hughen, K., Lehman, S., Southon, J., Overpeck, J., Marchal, O., Herring, C. and Turnbull, J.** 2004. <sup>14</sup>C activity and global carbon cycle changes over the past 50,000 years, *Science* 303, 202-207
- Melekestsev, I. V., Kirianov, V., & Praslov, N. D.** 1984: Katastroficheskoe izverzhenie v raione Flegreiskikh polei (Italia) – vozmozhny istochnik vulkanicheskogo pepla v pozднеpleistotsenovykh otlozheniyakh Evropeiskoi chasti SSSR. *Vulkanologiya i seismologiya*, 3, 35-44.
- Reimer, P. J., Baillie, M. G. L., Bard, E., Bayliss, A., Beck, J. W., Bertrand, C. J. H., Blackwell, P. G., Buck, C. E., Burr, G., Cutler, K. B., Damon, P. E., Edwards, R. L., Fairbanks, R. G., Friedrich, M., Guilderson, T. P., Hogg, A. G., Hughen, K. A., Kromer, B., McCormac, F. G., Manning, S., Bronk Ramsey, C., Reimer, R. W., Remmele, S., Southon, J. R., Stuiver, M.,**

- Talamo, S., Taylor, F. W., van der Plicht, J., and Weyhenmeyer, C. E., 2004**  
INTCAL04 terrestrial radiocarbon age calibration, 0-26 kyr BP. *Radiocarbon*  
46(3): 1029-1058.
- Pyle, D. M., Ricketts, G. D., Margari, V., van Andel, T. H., Sinitsyn, A. A.,  
Praslov, N. D., & Lisitsyn, S., 2006,** Wide dispersal and deposition of distal  
tephra during the Pleistocene ‘Campanian Ignimbrite/Y5’ eruption, Italy,  
*Quaternary Science Reviews* 25 (21-22), 2713-2728.
- Sinitsyn, A. A., Praslov, N. D., Svezhentsev, Yu. S., & Sulerzhitskii, L. D. 1997:**  
Radiouglerodnaya khronologiya verkhnego paleolita Vostochnoi Evropy. In A. A.  
Sinitsyn & N. D. Praslov (eds.), *Radiouglerodnaya khronologiya paleolita  
Vostochnoi Evropy i Severnoi Azii. Problemy i perspektivny*, pp. 21-66. St.  
Petersburg: Russian Academy of Sciences.
- Velichko, A. A., 1961,** *Geologicheskii vozrast verkhnego paleolita tsentral’nykh  
raionov Russkoi ravniny*. Moscow: USSR Academy of Sciences.

Table X: conventional radiocarbon ages (BP) for the dated samples from Kostenki 12. OxA-X- prefixes are given where the pre-treatment chemistry is non-routine, or where there are potential problems with the measurement. The OxA-X- numbers in column 2 represent the wheel number (the four digit number) and position number (2 digit suffix). Samples N1 and N10 were lower than expected in their percentage carbon on combustion. The expected values ought to be c. 50-65%, but these were much lower and indicate degraded or poorly preserved material, therefore an OxA-X- number was given. The resulting ages are best viewed as being potentially problematic.

Sample reference	Layer & cultural level	Lab No.	<sup>14</sup> C age years BP	Error	δ <sup>13</sup> C (‰)	pyield	%C yield
		OxA-X-					
EFD5C505, N1	12, CL III	2158-14	31760	230	-22.4	49.7	23.6
EFD5C512, N8	12, CL III	15482	35820	230	-24.0	71.0	73.7
EFD5C513, N9	14, CL IV	15555	35540	260	-24.9	21.0	60.5
		OxA-X-					
EFD5C514, N10	18, CL V	2158-15	34710	330	-21.7	43.8	16.3
EFD5C516, N12	18, CL V	15556	41300	450	-23.1	90.2	50.7
EFD5C517, N13	18, CL V	15902	38410	300	-21.1	46.5	58.5

Figure X1. A Bayesian model of the radiocarbon chronology of layers 12, 14 and 18 of Kostenki 12.

Figure X2. The end boundary for layer 12 (cultural horizon III) at Kostenki 12 based on the  $^{14}\text{C}$  measurements