



Short Paper

The influence of pretreatment chemistry on the radiocarbon dating of Campanian Ignimbrite-aged charcoal from Kostenki 14 (Russia)

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ABSTRACT

The presence of an independently dated marker in an archaeological site offers rare opportunities for assessing the reliability of radiocarbon dates, especially when these are close to the age limit of the technique. Two different pretreatment protocols (routine ABA and more rigorous ABOX-SC) were employed in the chemical preparation of the same charcoal sample from a layer closely associated to the Campanian Ignimbrite tephra at the Russian Palaeolithic site of Kostenki 14 (Markina Gora). The ABA-treated fraction gave an age of ~ 33 ^{14}C ka BP, comparable to a previous determination from the same layer, whereas the ABOX-SC produced an older age of ~ 35 ^{14}C ka BP. This is the first radiocarbon determination of an archaeological sample to provide an age consistent with the “calendar” age for the CI tephra marker.

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Introduction

A suite of publications in the last few years report the archaeological, chronological and geological sequences of the Kostenki-Borshchevo complex of Palaeolithic sites located on the west bank of the Don River in Russia (Sinitsyn, 2003; Sinitsyn and Hoffecker, 2006; Anikovitch et al., 2007; Holliday et al., 2007).

The case of Kostenki 14 (Markina Gora), specifically, has been used often to illustrate the alleged inability of radiocarbon (^{14}C) dating to produce reliable results in the period 32–42 ^{14}C ka BP, by comparison with the independent dates for the Campanian Ignimbrite (CI) tephra, an isochronous marker at the site (Fedele et al., 2008; Hoffecker et al., 2008). A recurring explanation over recent years is that this is due to severe variations in the production of atmospheric ^{14}C during this period. Although such fluctuations are undoubtedly manifest, owing to the Laschamp Geomagnetic Excursion (~ 40 – 42 cal ka BP), for instance, recent research at the ORAU (Higham et al., 2009) shows that probably the most important influence is inadequate sample cleaning of old (>30 ^{14}C ka BP) samples.

Background

The CI tephra

The CI tephra derives from a volcanic super-eruption in southern Italy, with the Phlegrean Fields thought to be the most likely source.

Over its wide dispersal area of at least 5,000,000 km², it is occasionally found in association with archaeological horizons, at cave and open-air sites (Fedele et al., 2003; Giaccio et al., 2006). In all cases, the ash layer occurs at a distinctive stratigraphic position, systematically sealing so-called “transitional” and early Upper Palaeolithic cultural deposits and marking a significant hiatus in the occupation of the sites (Giaccio et al., 2006). The CI has been independently and precisely dated by $^{40}\text{Ar}/^{39}\text{Ar}$ at 39.28 ± 0.11 ka (De Vivo et al., 2001), which adds to its importance as a key isochronous marker (Blockley et al., 2008). By contrast, the radiocarbon dating of archaeological material below and within the CI layer has been problematic and disclosed wide variations.

Radiocarbon dating around the time of the CI eruption

Over the past few decades, several radiocarbon laboratories have produced a considerable number of determinations from archaeological levels containing the CI, or sealed by it, which must date to around the time of the eruption, or be older. Most of these determinations are usually anomalously young and the Italian Palaeolithic, where the CI appears in many sites, offers a striking example (see Giaccio et al., 2006, Figs. 6 and 7).

This systematic inconsistency of ^{14}C dating led colleagues to suggest that the offset in archaeological dates must be related to geophysical factors that affect ^{14}C determinations around the time of the CI eruption on a global scale (Conard and Bolus, 2003; Giaccio et al., 2006; Pyle et al., 2006). On the basis of wide variations in the ^{14}C determinations obtained from German Palaeolithic cave sites, Conard and Bolus (2003, 2008) coined the phrases the “Middle Palaeolithic Dating Anomaly” and the “Coexistence effect.” They attributed the

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tendency towards younger and widely variable ^{14}C ages to major fluctuations in radioisotope production (mainly greater levels of ^{10}Be and ^{14}C) caused by the Laschamp Geomagnetic excursion. Svensson et al. (2006) locate the ^{10}Be peak around GIS 10 in the Greenland ice curve, several millennia before the CI tephra, which is expected to occur during the cold episode between GIS 8 and GIS 9.

Giaccio et al. (2006) and Fedele et al. (2008) have reported that pronounced ^{14}C variability was also observed in a marine core from the Tyrrhenian Sea (core CT85-5). The ^{14}C determinations of foraminifera they obtained from across the identified CI layer in the core suggests a sharp increase in ^{14}C production, which renders the radiocarbon ages over that period anomalously young, ranging from 35 to 25 ^{14}C ka BP. By using previously published archaeological examples in which the ^{14}C chronology yielded ages at odds with the age determined for the CI (one being the site of Kostenki 14), the authors conclude that “these examples show how, at least for the moment, radiocarbon is of virtually no use for dating events across the cosmogenic nuclide peak and the Laschamp excursion” (Fedele et al., 2008).

With particular reference to the Kostenki-Borshchevo complex, where radiocarbon dates from relevant strata yielded significantly younger ages than expected, Hoffecker et al. (2008) concluded that, even when calibrated, the ^{14}C dates underestimate the age of the tephra layer and therefore must be affected by the variations in the ^{14}C flux.

Pyle et al. (2006) suggested that since the $^{40}\text{Ar}/^{39}\text{Ar}$ age of the CI does not agree with a much younger radiocarbon date from a charcoal sample within the ash layer in Kostenki 14 (GrA-18053; Haesaerts et al., 2004), the offset between uncalibrated and calibrated ^{14}C ages for the period must be ~ 7000 yr.

Leaving aside the issue of the discrepancies between available calibration curves, which can only be resolved by additional sets of data such as the recently published IntCal09 (Reimer et al., 2009), many workers have failed to consider other possible explanations for inconsistent ^{14}C determinations—especially in sites where isochronous markers are present. These issues might involve sample quality, certainty of sample association with the archaeological context, stratigraphic and taphonomic factors. Few have considered the importance of pretreatment chemistry prior to AMS dating (but see: Chappell et al., 1996; Turney et al., 2001; Santos et al., 2001; Jöris and Street, 2008; Roebroeks, 2008).

Pretreatment protocols as sources of (un)certainty in ^{14}C dating

The critical variable in the successful application of radiocarbon dating to old material is the effective removal of carbonaceous contaminants at a molecular level. Laboratory pretreatment protocols have been specifically designed to target these exogenous elements, but because of the difficulties associated with the accurate quantification of the removal of all carbon-bearing contaminants have largely been assumed to produce accurate ages.

For archaeological charcoal, the routine pretreatment protocol involves a combination of acid–base–acid (ABA) steps for the demineralization of intrusive carbonates and the removal of humic acids. A refined protocol, specifically targeting very old samples, was developed by Bird et al. (1999). This involves an acid–base treatment (AB), an additional oxidation step (Ox) and stepped combustions (SC) of the remaining elemental carbon. The method, known by the acronym ABOx-SC, has since been applied to a number of prehistoric sites resulting in older and seemingly more consistent dates (Bird et al., 1999, 2003; Santos et al., 2003; Higham et al., 2008, 2009). It is worth mentioning, however, that ABOx-SC seems to have a smaller effect on samples younger than 30 ^{14}C ka BP (Higham et al., 2009). For example, the re-dating of a charcoal sample from Layer 8ac of Ksar Akil (Lebanon) previously dated in Oxford at $29,300 \pm 800$ ^{14}C yr BP (OxA-1798; Mellars and Tixier, 1989) gave a statistically indistinguishable new date of $30,250 \pm 170$ ^{14}C yr BP (OxA-19194) after ABOx-SC pretreatment. This is because of the increased effect of even small

amounts of contaminating carbon on material $< \sim 2.5$ pMC, or older than $\sim 30,000$ ^{14}C yr BP.

Cultural layer in volcanic ash, Kostenki 14 (Markina Gora)

The open-air Palaeolithic site of Kostenki 14 (Markina Gora) contains the CI tephra deposit directly associated with an Upper Palaeolithic occupational layer (Sinitsyn, 2003). Discrete lenses of cultural debris are separated from each other by lenses of high-purity volcanic glass. The excavators suggested rapid burial of the cultural material shortly after the eruption and liken the sudden destruction and abandonment of the site to that of Roman Pompeii. This archaeological horizon, “Layer in Volcanic Ash” (hereafter LVA), features typical Aurignacian lithic elements.

The CI has been located in a number of sites of the Kostenki-Borshchevo complex (Giaccio et al., 2008). Current evidence suggests that the tephra has been reworked at most locations. In Kostenki 1 and 12 the tephra has been heavily reworked. In Kostenki 14, the CI has been identified throughout the site, with the exception of the NW part of the excavated area, and is considered redeposited and stretched possibly by solifluction, although *in situ* deposits have also been located (P. Haesaerts, personal communication 2009). The occurrence of a first set of ash lenses underneath the LVA and around 20 cm above cultural layer IVa (Haesaerts et al., 2004, Fig. 2) may be the original position of the ash. Only at Borshchevo 5 does the tephra appear laterally *in situ* and relatively undisturbed by erosion or other slope processes.

The LVA in Kostenki 14 is associated with one radiocarbon date (GrA-8053: $32,420 \pm 440/420$ ^{14}C yr BP) made on charcoal. This sample was collected and submitted for dating by one of us (AS), and consisted of several smaller charcoal pieces originating from the Aurignacian layer interbedded with the volcanic ash.

Calibration of this determination however, shows a lack of correspondence with the accepted CI age.

Materials and methods

To investigate the possibility of inadequate decontamination of the Kostenki 14 charcoal, two different pretreatment protocols (routine ABA and improved ABOx-SC) were applied to the same charcoal sample coming from a hearth within the LVA, which appeared to be in a good state of preservation and free from visible impurities.

For the ABA protocol, 30 mg of charcoal was pretreated with 1 M HCl, pursued by 0.2 M NaOH and final rinsing with 1 M HCl. All steps were interspersed by three rinses with Milli-Q water.

For the ABOx-SC method, we used 180 mg of the same charcoal sample, which underwent acid–base chemistry, wet-oxidation and stepped combustion, prior to graphitization and AMS measurement, as described in Higham et al. (2008).

Results

The date obtained for the ABA-treated fraction was $33,220 \pm 220$ ^{14}C yr BP (OxA-19787), roughly concordant with the previous available date for LVA (GrA-18053) but still young in comparison to the CI.

The ABOx-SC fraction produced a date of $35,080 \pm 240$ ^{14}C yr BP (OxA-19021), older by almost 2000 ^{14}C yr when compared to the ABA determination of the same sample, and in excellent agreement with its chronostratigraphic position.

A Bayesian model (Fig. 1) was built using OxCal 4.1.3 (Bronk Ramsey, 2001, 2009) with the following assumptions:

1. The accepted CI age ($39,280 \pm 110$ yr BP) as published by De Vivo et al. (2001) is the closest approximation to the date of eruption.
2. All dates above the CI should postdate the CI eruption.

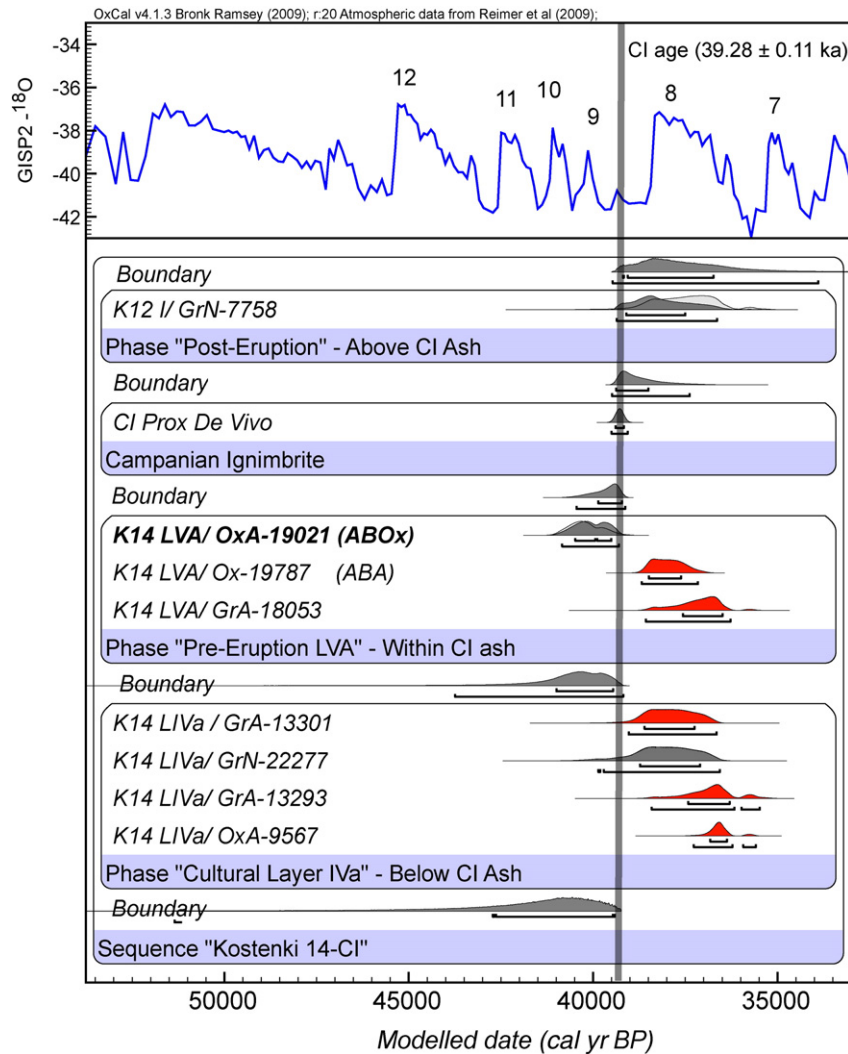


Figure 1. Bayesian model built with OxCal 4.1.3 (Bronk Ramsey, 2009), following the framework constructed by Blockley et al. (2008). The radiocarbon dates from Kostenki-Borshchevo sites discussed in the paper are calibrated using the IntCal09 (Reimer et al., 2009). Samples GrN-7758 from layers just above the ash horizons in Kostenki 12 represents *terminus ante quem*. The ABA dates from samples within the volcanic ash (LVA) and underneath it (IVa) show very low agreement with the overall model and are classified as outliers (in red). Note that GrN-22277 marginally overlaps with the CI at 95.4% probability and was not “failed,” despite the very low agreement index and its stratigraphic position (IVa), clearly below the CI horizon. The ABOx-SC date (OxA-19021) is the only determination which convincingly predates the CI eruption (gray line) given here as the ⁴⁰Ar/³⁹Ar age of De Vivo et al. (2001), also recommended by Pyle et al. (2006), at 39.28 ± 0.11 ka. The ages are compared with the GISP2 δ¹⁸O record, and the Greenland interstadials are numbered where relevant.

Table 1

Radiocarbon ages from Kostenki 12 and 14 relevant to the text, and calibrated ranges obtained using IntCal09 (Reimer et al., 2009). All Groningen samples (with the exception of GrA-18053) were physically cleaned by F. Dambon in order to eliminate exogenous material (Dambon et al., 1996). Compared ages are rounded to the nearest 10.

Radiocarbon ages BP			Unmodelled calibrated dates (IntCal09)				References
Site and layer	Laboratory ID	¹⁴ C ± 1σ	68.2%		95.4%		
			From	To	From	To	
<i>Phase "Post-eruption" – above CI ash</i>							
K12 I	GrN-7758	32,700 ± 700	38,390	36,630	39,220	35,540	Praslov and Rogachev (1982)
<i>Phase "Pre-eruption" – within CI ash</i>							
K14 LVA	OxA-19021	35,080 ± 240	40,790	39,730	40,990	39,340	This paper
K14 LVA	OxA-19787	33,220 ± 220	38,490	37,610	38,690	37,150	This paper
K14 LVA	GrA-18053	32,420 ± 440/420	37,570	36,490	38,570	36,270	Haesaerts et al. (2004)
<i>Phase "Cultural layer IVa" – below CI ash</i>							
K14 IVa	GrN-22277	33,280 ± 650/600	38,610	37,250	39,040	36,650	Haesaerts et al. (2004)
K14 IVa	GrA-13301	33,200 ± 510/480	38,740	37,130	39,900	36,560	Haesaerts et al. (2004)
K14 IVa	GrA-13293	32,180 ± 450/420	37,420	36,290	38,410	35,470	Haesaerts et al. (2004)
K14 IVa	OxA-9567	32,060 ± 260	36,830	36,370	37,270	35,590	Sinitsyn (1996)

3. All dates from the LVA should be contemporaneous or predate the CI.
4. All dates from layers below the CI should be progressively older.

The radiocarbon determinations were calibrated using the recently published calibration curve, IntCal09 (Reimer et al., 2009), which is the first internationally agreed curve spanning from 0 to 50,000 yr BP. The conventional radiocarbon ages and the unmodelled calibrated ones are given in Table 1.

A charcoal date obtained from a sample just above the CI ash, in Cultural layer I in Kostenki 12 (GrN-7758: $32,700 \pm 700$ ^{14}C yr BP), is included in the model as *terminus ante quem* for the deposition of tephra in Kostenki-Borshchevo region.

The LVA phase in the model contains the available radiocarbon ABA dates (GrA-18053; OxA-19787) and the new ABOx-SC date (OxA-19021). The latter fits perfectly the calibrated “pre-eruption phase” whereas the much younger ABA determinations show very low agreement with the overall model and are classified as outliers.

The dates from the LVA phase are additionally compared with these obtained on ABA-treated charcoal for the cultural unit IVa, below the volcanic ash at Kostenki 14 (Haesaerts et al., 2004; Fig. 2). The available dates are: GrA-13301: $33,200 \pm 510/480$ ^{14}C yr BP; GrN-22277: $33,280 \pm 650/600$ ^{14}C yr BP; GrA-13293: $32,180 \pm 450/420$ ^{14}C yr BP; OxA-9567: $32,060 \pm 260$ ^{14}C yr BP.

When calibrated these dates appear younger than the CI by several millennia (with the marginal exception of GrN-22277), and given the deeper stratigraphic position of IVa beneath the LVA, they must be regarded as aberrant.

Discussion

Similar results to those reported in the present study have been obtained recently for the Italian Palaeolithic site of Grotta di Fumane (Higham et al., 2009). The new determinations (both ABOx-Sc and ABA dates) from Fumane cover the period between about 29 and 42 ^{14}C ka BP and show a consistent difference between the two preparative methods, with the ABOx-SC determinations being always older.

In the case of Kostenki 14, the new ABOx-SC date is the first radiometric determination that so far agrees with the CI tephrostratigraphy and supports the archaeological evidence for the contemporaneity of the cultural debris with the CI distal tephra.

Again, this comes as a result of the effective removal of contamination from the dated sample, and we suggest that the ABA dates from the LVA in the range of $\sim 32/33$ ^{14}C ka BP should be considered underestimates of the true age. Likewise, the majority of the existing charcoal dates from below the tephra, which also seem to cluster at around 32 ^{14}C ka BP, are much too young due to insufficient chemical pretreatment.

The new data in this paper is not extensive and more results are required to add confidence. However, the new ABOx-SC date from the LVA at Kostenki 14 emphasizes once more that adequate sample pretreatment is essential in the accurate ^{14}C dating of Palaeolithic-aged samples. Further work on Kostenki-Borshchevo sites and a number of southern Italian sites containing the CI is currently underway at the ORAU.

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