The early chronology of broomcorn millet (*Panicum miliaceum*) in Europe

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The majority of the early crops grown in Europe had their origins in south-west Asia, and were part of a package of domestic plants and animals that were introduced by the first farmers. Broomcorn millet, however, offers a very different narrative, being domesticated first in China, but present in Eastern Europe apparently as early as the sixth millennium BC. Might this be evidence of long-distance contact between east and west, long before there is any other evidence for such connections? Or is the existing chronology faulty in some way? To resolve that question, 10 grains of broomcorn millet were directly dated by AMS, taking advantage of the increasing ability to date smaller and smaller samples. These showed that the millet grains were significantly younger than the contexts in which they had been found, and that the hypothesis of an early transmission of the crop from east to west could not be sustained. The importance of direct dating of crop remains such as these is underlined.

Keywords: Europe, Neolithic, broomcorn millet, *Panicum miliaceum*, AMS dating, ceramic grain impressions, Eurasian connections

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Samples submitted for radiocarbon dating at the Oxford Radiocarbon Accelerator Unit (ORAU) were subjected to a less rigorous chemical pre-treatment than usual owing to their low starting weights. Specifically, this consisted of initial demineralisation with 1 M HCl for 1 hour, followed by 15 minutes ultrasonication in fresh 1 M HCl. After subsequent rinsing in ultrapure (MilliQ™) water four times, the samples were ultrasonicated for 5 minutes in fresh ultrapure water a further six times. The samples were re-acidified for 5 minutes in 1 M HCl, and finally rinsed twice more in ultrapure water. This gentler pre-treatment protocol is less rigorous than the acid-base-acid pre-treatment protocols more routinely applied to larger and more robust plant macrofossil samples (Brock *et al.* 2010). As noted in the main text, the fact that the broomcorn millet grains studied herein appeared to be in a good state of preservation, as well as being physically 'clean', gives us confidence that, despite the less rigorous chemical pre-treatment methodology applied, the ^{14}C measurements obtained remain reliable.

The samples were frozen and freeze-dried before being weighed into clean tin capsules. Samples were then combusted in an elemental analyser coupled to a gas source isotope ratio mass spectrometer (IRMS), and converted to CO_2. 1/50th of the gas produced was directed into the mass spectrometer for stable isotope ($\delta^{13}C$) measurement, with the remaining CO_2 transferred to a reactor rig and collected cryogenically. For routine samples submitted to ORAU, either ~1.8 mg C or ~0.8 mg C is collected for graphitisation (according to sample size) and run on separate 'large' or 'small' graphite AMS wheels, respectively; however, the millet samples described here produced smaller yields of between 0.311 and 0.592 mg C (apart from the larger sample OxA-26477). Hydrogen gas was then added to the rigs (in the ratio of ~2.2 H_2:CO_2), and the rigs heated at 560°C for 6 hours in the presence of 2.0–2.5 mg of iron powder, catalysing the conversion of CO_2 to pure C (graphite). A specific refinement to this routine ORAU graphitization protocol (presented by Dee & Bronk Ramsey 2000) is the addition of the desiccant magnesium perchlorate ($Mg(ClO_4)_2$) to the water trap of the reactor rigs. The presence of the desiccant helps to draw out H_2O, and therefore optimises the conversion of CO_2 to graphite (a more critical factor for these smaller sample sizes than for more

routine samples). The use of desiccants to improve the graphitisation of very small samples for radiocarbon dating has been applied elsewhere (Santos *et al.* 2007), but here we describe the application of such a methodology at ORAU for the first time.

The resulting graphite produced was pressed into aluminium targets for accelerator mass spectrometry (AMS) radiocarbon dating as for the routine, larger samples submitted to ORAU. Finally, the samples were dated on the ORAU 3 MV HVEE AMS system (Bronk Ramsey *et al.* 2004). Conventional radiocarbon ages before present (BP) were calculated relative to the oxalic acid (HOXII) standard and normalised for isotopic fractionation as per Stuiver and Polach (1977).

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Table S1: Archaeological contexts of the ten broomcorn millet samples, along with their ^{14}C (uncalibrated and calibrated) and $\delta^{13}C$ measurements. Sample weights are given prior to and following chemical pretreatment, as well as carbon yield following combustion and AMS target currents.

Site	Country	Expected culture and period	ORAU lab. code	Starting weight (mg)	Chemistry yield (mg)	Combustion yield (mg C)	$\delta^{13}C$ (‰)	Current (μA)	Conventional ^{14}C age BP (±1σ)	Calibrated age (cal. BC/AD, 95.4% hpd range)
Bruchenbrücken/ Friedberg	Germany	Bandkeramik (Neolithic 5500–4500 BC)	OxA-26700	1.36	0.77	0.429	–10.05	26.16	3163±33	1505–1386 BC
Fechenheim/ Frankfurt	Germany	Bandkeramik (Neolithic 5500–4500 BC)	OxA-26701	2.65	1.00	0.581	–10.63	27.01	2815±32	1055–851 BC
Goddelau/ Riedstadt	Germany	Bandkeramik (Neolithic 5500–4500 BC)	OxA-26702	0.92	0.71	0.464	–9.59	22.74	2484±34	772–417 BC
Fajsz 18	Hungary	Sopot (Late Neolithic 5500–4500 BC)	OxA-26703	1.03	0.60	0.361	–11.16	22.30	3075±36	1428–1262 BC
Fajsz 18	Hungary	Sopot (Late Neolithic 5500–4500 BC)	OxA-26704	1.21	0.71	0.413	–9.87	22.94	3214±36	1606–1414 BC

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Okoliste	Bosnia and Herzegovina	Butmir (5500–4800 BC)	OxA-X-2479-22	1.62	0.85	0.528	–11.04	1.29*	1740±130	AD 4–576
Yabalkovo	Bulgaria	Early Neolithic	OxA-26705	1.42	0.53	0.311	–9.57	17.30	1128±35	AD 781–991
Yabalkovo	Bulgaria	Early Neolithic	OxA-26477	2.09	1.61	1.106	–9.75	28.32	1176±28	AD 774–953
Măgura-Buduiasca	Romania	Dudesti (Neolithic 6th mill. BC)	OxA-26706	1.57	0.74	0.441	–9.66	23.87	3093±35	1434–1268 BC
Măgura-Buduiasca	Romania	Dudesti (Neolithic 6th mill. BC)	OxA-26707	1.23	0.92	0.592	–10.41	28.23	398±26	AD 1438–1620

Note: $\delta^{13}C$ (‰) data are relative to the Vienna Pee Dee Belemnite standard. The conventional 14C age BP (±1σ) data and a fractionation correction was calculated as per Stuiver & Polach 1977. With reference to the calibrated age, 'hpd' is highest probability density.

* The uncertainty on a radiocarbon measurement principally arises from the 'counting statistics' (achievable measurement precision from the AMS). With AMS targets that yield low target currents, the achievable measurement precision is impaired, resulting in radiocarbon dates with larger uncertainties. This was the case with sample OxA-X-2479-22, which yielded a negligible current of 1.2μA compared to normal (usually in the range 25–30μA). We therefore have reduced confidence in the reliability of such samples (and hence the target was given an 'OxA-X-' lab-code, rather than the 'OxA-' applied to 'good' samples.) Normally, target current relates to the sample size (combustion volume: i.e. more carbon in the target leads to a higher target current, which leads to greater measurement precision), although that is not the case with sample OxA-X-2479-22. The low target current with this sample was probably the result of a poor 'press' (i.e. the graphite was not properly pressed into the aluminium AMS target), which can itself be a problem with smaller-than-usual sample sizes.

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