Daniel Groß · Harald Lübke · John Meadows · Detlef Jantzen (eds.)

Working at the Sharp End: From Bone and Antler to Early Mesolithic Life in Northern Europe

10

Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein und im Ostseeraum

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Band 10

Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein und im Ostseeraum
aus dem Museum für Archäologie Schloss Gottorf und dem Zentrum für Baltische und Skandinavische Archäologie
in der Stiftung Schleswig-Holsteinische Landesmuseen Schloss Gottorf
Band 10

Begründet von
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Herausgegeben von
Sönke Hartz und Harald Lübke

Working at the Sharp End:
From Bone and Antler to Early Mesolithic Life in Northern Europe

Daniel Groß, Harald Lübke, John Meadows and Detlef Jantzen (eds.)

Wachholtz

MIX
Papier aus verantwortungsvollen Quellen
FSC® C107574

1. Auflage 2019

Redaktion: Gundula Lidke, SSHLM Schloss Gottorf, Schleswig
Satz: Daniel Groß, SSHLM Schloss Gottorf, Schleswig
Einbandgestaltung: Jürgen Schüller, SSHLM Schloss Gottorf, Schleswig; Foto: Markus Wild, SSHLM Schloss Gottorf, Schleswig

Bibliografische Informationen der Deutschen Nationalbibliothek: Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der deutschen Nationalbibliografie; detaillierte bibliografische Daten sind im Internet unter http://dnb.n-nb.de abrufbar.

ISBN 978-3-529-01861-9
ISSN 2510-313X
Druck und Vertrieb: Wachholtz Verlag, Kiel/Hamburg
Printed in Europe
Besuchen Sie uns im Internet:
www.wachholtz-verlag.de

Laserscan of the wooden sign that was attached to the excavation hut during the Hohen Viecheln excavations ('To the sharp harpoon'; Laserscan: J. Nowotny, ZBSA).

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Vorwort der Herausgeber

Die Schriftenreihe „Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein“ wurde von dem ursprünglichen Herausgeber Jürgen Hoika vor mittlerweile 25 Jahren im Jahre 1994 begründet, um am damaligen Archäologischen Landesmuseum Schleswig (ALM) und heutigem Museum für Archäologie Schloss Gottorf (MfA) ein Publikationsorgan für die Veröffentlichung von Forschungsergebnissen zur Steinzeit Schleswig-Holsteins zu schaffen. Dabei sollte es sich zum einen um Sammelwerke mit Beiträgen von vorzugsweise auf Schloss Gottorf veranstalteten Symposien, Workshops und Tagungen mit steinzeitlicher Thematik und zum anderen um zumeist in Dissertationen zusammengestellte ausführliche Materialvorlagen handeln. Entsprechend enthielt der 1994 vorgelegte erste Band der Reihe die Beiträge zum 1. Internationalen Trichterbechersymposium, welches, von Jürgen Hoika gemeinsam mit Jutta Meurers-Balke initiiert, 1984 am Archäologischen Landesmuseum in Schleswig stattgefunden hatte. In der Folge wurden dann aber beginnend mit den Arbeiten der beiden heutigen Herausgeber nunmehr acht überwiegend am Institut für Ur- und Frühgeschichte der Christian-Albrechts-Universität zu Kiel fertiggestellte Dissertationen veröffentlicht, die ganz wesentlich mit der wissenschaftlichen Vorlage und Auswertung von Forschungsgrabungen in Schleswig-Holstein und – seit der Beteiligung des Zentrums für Baltische und Skandinavische Archäologie an der Herausgeberschaft – aus dem gesamten Ostseeraum befasst sind.

Deshalb ist es eine besondere Freude für die Herausgeber, mit dem vorliegenden Band 10 „Working at the Sharp End: From Bone and Antler to Early Mesolithic Life in Northern Europe“ der Schriftenreihe „Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein und im Ostseeraum“ wiederum einen Sammelband mit den Beiträgen eines Workshops vorlegen zu können, der vom 14. bis 16. März 2016 auf Schloss Gottorf stattgefunden hat. Dabei handelt es sich um den Abschlussworkshop des von der Deutschen Forschungsgemeinschaft geförderten Projektes „Neubewertung von Chronologie und Stratigraphie des frühholozänen Fundplatzes Hohen Viecheln (Mecklenburg-Vorpommern) unter besonderer Berücksichtigung der diagnostischen Knochenartefakte“ (DFG-Projektnummer 271652103) unter Leitung von Daniel Groß, Harald Lübke, John Meadows (alle ZBSA) und Detlef Jantzen (Landesamt für Kultur und Denkmalpflege Mecklenburg-Vorpommern; Landesarchäologie). Entsprechend enthält dieser Band neben dem Abschlussbericht des Forschungsprojektes insgesamt 17 Beiträge der eingeladenen Workshop-Teilnehmer, die entweder ergänzende Studien zum Fundplatz Hohen Viecheln enthalten oder sich grundsätzlich mit verwandten Themen zur Erforschung des frühholozänen Mesolithikums im nördlichen Europa befassen.

Alle Beiträge wurden nach internationalem Standard von jeweils zwei anonymen Gutachtern in einem Peer-review-Verfahren bewertet und danach den Autoren zur erneuten Überarbeitung übergeben, bevor die abschließende redaktionelle Bearbeitung der Manuskripte erfolgte. Die Textredaktion für alle Beiträge wurde von Gundula Lidke durchgeführt, Jana Elisa Freigang und Jorna Titel leisteten dabei unterstützende Arbeiten. Das Layout übernahm Daniel Groß, Titelbild und Umschlag entwarf Jürgen Schüller. Die meisten Karten und Zeichnungen wurden von den Autoren selbst bereitgestellt. In einzelnen Fällen erfolgte eine Überarbeitung durch Daniel Groß. Allen sei dafür an dieser Stelle herzlich gedankt.

Neu im Rahmen der Schriftenreihe ist, dass die Beiträge unmittelbar nach Fertigstellung und Freigabe der Autoren in einem „online-first“-Verfahren auf der Homepage des Verlages im Open Access zum freien Download bereitgestellt wurden. Für die Umsetzung dieser Forderung der Herausgeber danken wir dem Wachholtz Verlag, insbesondere Herrn Henner Wachholtz, sehr.

Besonderer Dank gilt dem Vorstand des Zentrums für Baltische und Skandinavische Archäologie Schleswig, besonders dem Direktor, Claus von Carnap-Bornheim, und der Forschungsleiterin, Berit Valentin Eriksen, die die Veröffentlichung dieses Bandes durch die Bereitstellung der erforderlichen Mittel für den Druck der Arbeit maßgeblich unterstützten.

Sönke Hartz und Harald Lübke
Schleswig, im Oktober 2019

Editors' Preface

The series 'Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein' was founded by its first editor, Jürgen Hoika, in 1994, 25 years ago, in order to establish a possibilty to publish Stone Age research results from Schleswig-Holstein at the then Archaeological State Museum (Archäologisches Landesmuseum [ALM]), today's Museum for Archaeology (Museum für Archäologie, Schloss Gottorf [MfA]). Publications should, on the one hand, reflect proceedings of symposia, conferences and workshops with Stone Age topics primarily held at Gottorf Castle, on the other hand, dissertations presenting comprehensive material. According to that, the first volume, published in 1994, contained the contributions to the 1st International Funnelbeaker Symposium, which, initiated by Jürgen Hoika and Jutta Meurers-Balke, had taken place at the Archaeological State Museum in 1984. Following that, eight dissertations, mainly accomplished at the Institute for Pre- and early History at the Christian-Abrechts-University Kiel, were published, starting with those by today's editors. All these volumes contributed substantially to the scientific presentation and analysis of excavation materials from Schleswig-Holstein and – since 2012, when the Centre for Baltic and Scandinavian Archaeology (ZBSA) also became involved in editig the series – the whole of the Baltic Sea area.

Therefore the editors are especially happy to once more present conference proceedings with volume 10 of the series 'Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein und im Ostseeraum': 'Working at the Sharp End: From Bone and Antler to Early Mesolithic Life in Northern Europe' collects contributions to a workshop held at Gottorf Castle on 14th–16th March, 2016. This represented the closing workshop of the DFG-funded project 'Neubewertung von Chronologie und Stratigraphie des frühholozänen Fundplatzes Hohen Viecheln (Mecklenburg-Vorpommern) unter besonderer Berücksichtigung der diagnostischen Knochenartefakte' (DFG project no. 271652103), directed by Daniel Groß, Harald Lübke, John Meadows (all ZBSA) und Detlef Jantzen (Landesamt für Kultur und Denkmalpflege Mecklenburg-Vorpommern; Landesarchäologie). In addition to the project's final report the volume contains 17 papers by researchers invited to participate in the workshop, representing either additional studies on material from the site Hohen Viecheln or related topics in research of the early Holocene Mesolithic in northern Europe.

Each paper was, according to international standards, peer-reviewed by two anonymous reviewers and then returned to the author for reworking before final editorial work. Copy-editing was performed by Gundula Lide, supported by Jana Elisa Freigang and Jorna Titel. Daniel Groß realised the layout; cover and cover illustration were designed by Jürgen Schüller. Most maps and figures were provided by the authors themselves, some were reworked by Daniel Groß. We express our sincere thanks to all involved!

It is a novelty for the series to have papers published online first immediately after completion and authors' approval in open access for free download on the website of Wachholtz Publishers. We would like to thank Henner Wachholtz, Wachholtz Publishers, very much for making this possible!

Special thanks are due to the board of the Centre for Baltic and Scandinavian Archaeology (ZBSA) Schleswig, particularly to the director, Claus von Carnap-Bornheim, and the head-of-research, Berit Valentin Eriksen, who substantially supported this publication by providing financial means for its printing.

Sönke Hartz and Harald Lübke
Schleswig, October 2019

Grusswort des Landesarchäologen von Mecklenburg-Vorpommern

Mit seinen großflächigen, oft noch weitgehend unberührten Niederungen und Binnengewässern bietet Mecklenburg-Vorpommern beste Voraussetzungen, um die gewässeraffinen Kulturen des Mesolithikums zu erforschen. Die Überreste ihrer Wohn- und Jagdstationen sind im feuchten Milieu hervorragend erhalten geblieben. Störungen durch Torfabbau, Begradigung von Gewässern oder Meliorationsmaßnahmen blieben im Wesentlichen auf das 19. und 20. Jahrhundert beschränkt. Sie haben zwar einen gewissen Schaden angerichtet, aber, weil sie zumindest im 20. Jahrhundert oft von aufmerksamen ehrenamtlichen Bodendenkmalpflegern beobachtet wurden, überhaupt erst zur Entdeckung vieler Fundstellen geführt.

Welche Fundstellen eingehender erforscht werden und damit das Bild einer Epoche besonders prägen, unterliegt oft dem Zufall. Hohen Viecheln rückte in den Fokus der Forschung, weil die Entdeckung mehrerer Knochenharpunen zu Beginn der 1950er Jahre auf eine günstige Konstellation traf: 1953 war aus der Vorgeschichtlichen Abteilung des Staatlichen Museums das Museum für Ur- und Frühgeschichte Schwerin entstanden, das auch für die Bodendenkmalpflege in den drei Nordbezirken der DDR zuständig war. Der ehrgeizige Direktor des Museums, Ewald Schuldt, hatte sich durch Ausgrabungen auf der Burgwallinsel Teterow einen Namen gemacht und war nun auf der Suche nach einem geeigneten Fundplatz für ein eigenes Forschungsprojekt.

Wegen der sehr guten Erhaltungsbedingungen versprach Hohen Viecheln, zusätzlich zu dem bekannten Spektrum an Steinartefakten auch ein umfangreiches Geräteinventar aus organischen Materialien bergen zu können. Die ebenfalls ausgezeichnet erhaltenen Tierknochen sollten Aufschluss über das Jagdwild geben. Hinzu kam die Aussicht, aus der Stratigraphie neue Erkenntnisse zur Chronologie und zu den Veränderungen der naturräumlichen Verhältnisse zu gewinnen. Diese Erwartungen wurden nicht enttäuscht: Hohen Viecheln entwickelte sich zu einem der bedeutendsten Plätze mesolithischer Forschung, gleichrangig mit Duvensee, und inspirierte weitere Forschungen, u. a. in Friesack und Rothenklempenow.

Hohen Viecheln gehört nach wie vor zu den legendären archäologischen Fundstellen in Mecklenburg-Vorpommern, auch wenn es aus heutiger Sicht nicht mehr so einzigartig dasteht. Dank einer intensiv betriebenen ehrenamtlichen Bodendenkmalpflege ist die Zahl der bekannten mesolithischen Fundplätze im Land deutlich gestiegen, von denen vermutlich mehrere ein ähnliches Potenzial wie Hohen Viecheln aufweisen. Verändert haben sich aber nicht nur die Verbreitungskarten, sondern auch die Möglichkeiten archäologischer Forschung. Es drängte sich deshalb geradezu auf, Hohen Viecheln noch einmal unter die Lupe zu nehmen, bisherige Erkenntnisse kritisch zu prüfen und neue hinzuzufügen. Der DFG und allen Projektpartnern gebührt herzlicher Dank dafür, dass sie das ermöglicht haben.

So wird Hohen Viecheln auch weiterhin als exemplarischer Fundplatz für das Mesolithikum in der norddeutschen Tiefebene stehen – eine hochinteressante Umbruchszeit, in der Klimawandel, Anstieg des Meeresspiegels und andere Veränderungen eine ständige Anpassung der Menschen an ihre Umwelt erzwangen.

Detlef Jantzen
Schwerin, im September 2019

Welcome address by the State Archaeologist of Mecklenburg-Western Pomerania

Mecklenburg-Western Pomerania with its large, often unspoiled lowlands and inland waters offers outstanding possibilities for research into the water-oriented cultural groups of the Mesolithic. Remains of their settlement and hunting sites are often well preserved in wet conditions. Disturbances by peat extraction, straightening of watercourses or melioration measures mainly took place during the 19th and 20th centuries. They did some damage, but – as at least during the 20th century they were often supervised by vigilant amateur archaeologists – many sites were discovered this way in the first place.

But often it is left to chance which sites can be thoroughly investigated to largely characterise the picture of a whole timespan. Hohen Viecheln became the focal point of research interest under favourable circumstances: the discovery of several bone points there at the beginning of the 1950s fell together with the establishment of the Museum of Pre- and Early History in Schwerin (out of the former Department of Prehistory at the State Museum) which was also responsible for the preservation and care of field monuments in the three northern districts of the GDR.

The ambitious museum director, Ewald Schuldt, had already gained reputation through his excavations of the Slavic ring wall island near Teterow, and he was looking for a suitable site for another research project. Due to the very good preservation conditions at the site, Hohen Viecheln promised, in addition to the spectrum of artefacts known from other places, a substantial organic inventory. The well-preserved animal bones were expected to shed light on game species and hunting strategies. Furthermore, important results were expected concerning chronology and environmental changes. These hopes were not disappointed: Hohen Viecheln has become, alongside Duvensee, one of the most important sites for Mesolithic research, and research there has inspired further excavations, e.g. at Friesack or Rothenklempenow.

Hohen Viecheln is still one of the legendary archaeological sites in Mecklenburg-Western Pomerania, even if it no longer stands alone. Thanks to intensive voluntary archaeological surveys the number of Mesolithic sites has increased significantly; and several of these may have a potential similar to that of Hohen Viecheln. But not only distribution maps have changed during the last years, but also the possibilities of archaeological research. Therefore, the idea to have another look at Hohen Viecheln, to challenge old results and add new ones, suggested itself. I want to thank the German Research Foundation (DFG) and all project contributors for having made this possible. In this way, Hohen Viecheln will continue to be an exemplary North German Lowland site of the Mesolithic – a highly interesting time when climate change, sea-level rise and other changes enforced constant human adaptions to the environment.

Detlef Jantzen
Schwerin, September 2019

Acknowledgements

This volume of the series 'Untersuchungen und Materialien zur Steinzeit in Schleswig-Holstein und im Ostseeraum' represents the proceedings of a workshop held at the Centre for Baltic and Scandinavian Archaeology (ZBSA) in Schleswig in March 2016. It is a part of the editors' project 'Neubewertung von Chronologie und Stratigraphie des frühholozänen Fundplatzes Hohen Viecheln (Mecklenburg-Vorpommern) unter besonderer Berücksichtigung der diagnostischen Knochenartefakte', funded by the German Research Foundation (DFG) under the project number 271652103.

While the project was dealing with the re-evaluation of the site Hohen Viecheln 1 for chronological and stratigraphical aspects, this volume does not only cover its final publication but comprises additional modern studies about the site by different scholars. These are furthermore embedded into the international research landscape by adjacent studies covering an area from modern day Britain in the west to the Urals in the east.

All contributions are representing the authors' point of view and respective terminologies. Therefore differences in the vocabulary may appear to the careful reader. While a homogenisation of terms and data recording is relevant for comparative studies, it was beyond the scope and means of this project. As a consequence, terminologies may differ between the contributions, as exemplified by the terms 'uni-serial' and 'uni-lateral' bone points: both are characterised by barbs or notches on one lateral side. At the British site Star Carr those have ever since been named uni-serial, whereas uni-lateral is a more common term in other parts of Europe.

We, as editors, would like to thank all contributors for being part of this volume and their interesting and high-quality articles; also we are grateful for the voluntary support of all anonymous peer-reviewers and their help in improving the articles. Furthermore, we thank the German Research Foundation (DFG) for funding our research and the workshop as well as the Centre for Baltic and Scandinavian Archaeology represented by its director, Claus von Carnap-Bornheim, and the head-of-research, Berit Valentin Eriksen, for support of the project and its presentation in the current form. A tremendous help in the course of making this book was Gundula Lidke who was responsible for text editing, proof-reading, and correspondence with the authors and publishers. Thank you very much! Further editorial support was provided by Jana Elisa Freigang, Jorna Titel, Matthias Bolte, Isabel Sonnenschein and Jürgen Schüller. The latter is also responsible for the cover drawing. Much help and support was provided by Peter Teichert-Köster with respect to handling the finds and accessing them in the depot of the Landesamt für Kultur und Denkmalpflege Mecklenburg-Vorpommern; Landesarchäologie in Schwerin. Close collaboration with Mathieu Boudin of the Royal Institute for Cultural Heritage, Brussels, improved our radiocarbon measurements and the analysis of the consolidant.

We thank all people, mentioned and unmentioned here, who were involved in this book and the different research projects, who helped by further pushing the boundaries of our understanding of the cultural remains and chronologies of the past.

Daniel Groß, Harald Lübke, John Meadows, Detlef Jantzen
Schleswig, October 2019

Radiocarbon dating bone and antler artefacts from Mesolithic Hohen Viecheln (Mecklenburg-Western Pomerania, Germany)

John Meadows, Mathieu Boudin, Daniel Groß, Detlef Jantzen, Harald Lübke and Markus Wild

Abstract

Finds from Ewald Schuldt's 1952–54 excavations at Hohen Viecheln, on the shore of Lake Schwerin, form one of the most important assemblages of Mesolithic bone/antler tools in Germany, including over 300 projectile points. Re-evaluation of Schuldt's excavation records has created doubts about the published stratigraphic sequence. For reliable chronologies of different tool types, therefore, it is necessary to directly date diagnostic artefacts. However, artefacts were consolidated soon after the excavation, with unknown conservation agents. Our analyses suggest that two different compounds were used. Altogether, 35 finds were sampled, following a minimally invasive approach. Satisfactory dates were obtained for 28 artefacts. Collagen yields were highly variable, but all results from samples with >1 % collagen are plausible, and all extracts tested meet EA-IRMS acceptance criteria. FTIR was used throughout the process to monitor the removal of consolidants. Most of the dated samples were apparently consolidated with a compound based on cellulose nitrate. Tests suggest that this product would have been removed by the procedures followed to extract collagen, but its elemental and isotopic composition is such that we cannot exclude the possibility that enough consolidant remained in the dated collagen extracts to produce significant radiocarbon age offsets, particularly in low-yield samples.

1 Introduction

Hohen Viecheln is a village on the shore of Lake Schwerin, northeastern Germany. In September 1952, after local children discovered bone harpoons in a drainage ditch, Ewald Schuldt, the regional museum director, began the excavation of an Early Mesolithic site, eventually excavating c. 860 m^2 of the former lakeshore. No structural remains were found, but the peat and gyttja layers yielded large quantities of animal bone and antler, including almost 500 artefacts (Schuldt 1961). Soon after the excavation, the artefacts were consolidated with unknown conservation agents, because some of the finds were very fragile (Schuldt 1961, 104, 137). Palynology was used to confirm that the Mesolithic occupation coincided mainly with the late Boreal and early Atlantic periods, although the earliest finds were attributed to the Preboreal-Boreal transition (Schmitz 1961).

This interpretation would imply that the artefacts date mainly to the 8th–7th millennia cal. BC, but some finds may be as early as the mid-9th millennium. Prior to our research, the only ^{14}C dates from Hohen Viecheln were from two unworked animal bones recovered in Schuldt's excavations: a pond turtle

(*Emys orbicularis*; KIA-30246: 8955 ± 40 uncal. BP; Sommer et al. 2007) and a wild horse (*Equus ferus*; KIA-35740: 9180 ± 40 uncal. BP; Sommer et al. 2011). The turtle result (calibrated to 8280–7970 cal. BC) should be regarded as a maximum age, as a pond turtle's diet consists primarily of aquatic animals and plants (Ficetola/De Bernardi 2006), which in Lake Schwerin were probably subject to significant freshwater reservoir effects (Fernandes et al. 2013). While the horse bone date (8540–8290 cal. BC) is valid, its stratigraphic position is unknown.

These results do not contradict Schmitz and Schuldt's interpretation, but neither do they help to position Hohen Viecheln within the Early Mesolithic period overall, or to test another of Schuldt's ideas, that different types of bone/antler projectile points represented a chronological sequence. The 1950s excavation methods do not provide exact find-spots for these artefacts, so we cannot rely on stratigraphy to support Schuldt's suggestion that Duvensee-type points were gradually replaced by Pritzerbe-type points. Schuldt also did not consider whether simple bone/antler points (without diagnostic features of Duvensee or Pritzerbe types) might represent a third phase. To address these questions, it was necessary to date these artefacts directly. The opportunity to date a number of artefacts arose through a re-evaluation of Schuldt's excavation and publications (Gross et al., this volume). Permission to sample up to 40 artefacts in the excavation archive was granted by the Landesamt für Kultur und Denkmalpflege Mecklenburg-Vorpommern.

2 Methods

2.1 Sampling strategy

Prior to any invasive sampling, consolidant from the surface of an aurochs (*Bos primigenius*) phalanx (HV-3858) was sampled to test its identifiability and solubility.

Radiocarbon samples were then selected in two batches, the second round of sample selection being informed by the results of the first. In the first round, 17 artefacts with relatively precise provenance were selected, to see whether, contrary to expectations, provenance information provided a reliable stratigraphic sequence. In the second round, 17 more artefacts were sampled. Sampling focused on typologically diagnostic bone points, but some other unique artefacts were also selected. Two bone points found during a small (8 m^2) rescue excavation at Hohen Viecheln in 1995 (Schacht 1996), which had not been consolidated, were also sampled. Species determination was often impossible, due to extensive modification during manufacture, but all artefacts were probably made from of bones or antlers of large cervids (elk [*Alces alces*], red deer [*Cervus elaphus*]), aurochs, and possibly wild horse.

Separately, a red deer 'antler headdress' from Schuldt's excavation at Hohen Viecheln, and another such headdress, a 1950s stray find from Biesdorf, Berlin, were sampled (Wild et al. in prep; Wild, this volume). Their results are included here because they were processed with the other 34 samples from Hohen Viecheln, following the same approach.

This paper therefore considers 36 artefacts, of which 17 were dated at the Royal Institute for Cultural Heritage Radiocarbon Dating Laboratory (Brussels, RICH-), eleven at the Leibniz-Laboratory for AMS Radiocarbon Dating and Stable Isotope Research (Kiel, KIA-), and eight in both laboratories (Table 1).

2.2 Minimal impact sampling

Samples were taken at the Landesamt für Kultur- und Denkmalpflege Mecklenburg-Vorpommern, Schwerin, where the Schuldt archive is stored. A dental drill was used to abrade the surface of each artefact, before cutting or drilling out the 'dating samples'. Equipment was cleaned in acetone before sampling each object, and each object was photographed before and after sampling. The same procedure was followed when the Biesdorf antler headdress was sampled at the Landesmuseum für Kultur und Geschichte Berlins. Sampling locations were chosen to avoid damaging any traces left by the artefact's manufacture and use, to preserve diagnostic features and edges broken in antiquity (in case joining fragments are discovered in future), and to minimise visual impact. In most cases, this meant that samples were taken by drilling broad shallow holes into the internal unworked surface of long bone splinters, and collecting the bone powder. Samples of only 200–300 mg were taken, based on a calculation that at least 1 mg of carbon would be obtained if the collagen yield was >1 % of the starting weight (a threshold for satisfactory collagen preservation: Dobberstein et al. 2009). Where possible, double samples were taken, to allow replicate dating.

2.3 Laboratory methods

Enough consolidant had been applied to HV-3858, the aurochs foot, that it was possible to peel c. 15 mg of almost pure consolidant off the base of phalanx 3, for analysis in Kiel. Attenuated Total Reflectance-Fourier Transform Infrared (FTIR) spectroscopy revealed several prominent peaks in its spectrum that are not present in FTIR spectra of unconsolidated bone/antler or collagen (Fig. 34). The consolidant readily dissolved in acetone (<1 h at room temperature), but it appeared to be insoluble in 1 % HCl, 1 % NaOH, and demineralised water (2 weeks at 65 °C). After FTIR testing, a 2.2 mg aliquot of the insoluble consolidant was dried and sealed for AMS dating in Kiel; the remainder was sent for EA-IRMS analysis at the School of Archaeological Sciences, University of Bradford.

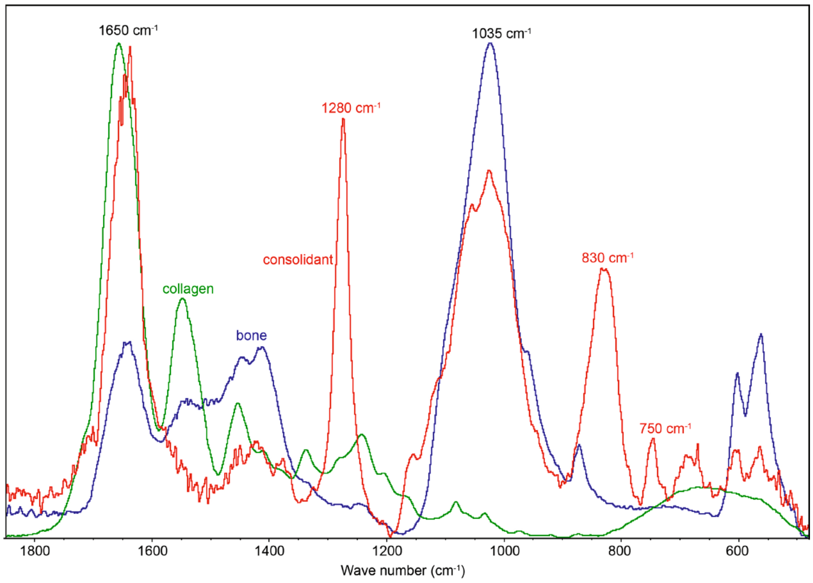

Fig. 1. ATR-FTIR spectrum of the HV-3858 consolidant, compared to spectra from an unconsolidated, well-preserved bone and pure collagen extracted from an unconsolidated bone. Absorbance peaks in the consolidant at c. 1280, c. 830 and c. 750 cm^{-1} are absent in both the bone and collagen spectra, whereas the peaks at c.1650 and c. 1035 cm^{-1} coincide with those in bone (the bone peak at c. 1650 reflects the high collagen content in this example), and are therefore not useful for detecting consolidant contamination of bone/antler samples.

Bone/antler powder from each dating sample, and from the abraded surface of each artefact, were tested by FTIR without pretreatment. Most artefacts appeared to have been consolidated, with compounds which often penetrated into the dating sample, although the surface layer was always more contaminated (Fig. 2). Two or more products were used, judging by FTIR spectra (Fig. 3), although all but one of the samples dated successfully had spectra consistent with the presence of varying amounts of the consolidant applied to HV-3858 (Type A in Table 1). Thus it was hoped that most, if not all, of any consolidant in the dating samples would be dissolved by pretreatment in solvents; that any undissolved consolidant would remain insoluble in the reagents used to extract collagen, and be trapped when the dissolved collagen was filtered; and that if these steps failed, FTIR of the extracted collagen would reveal that it was contaminated.

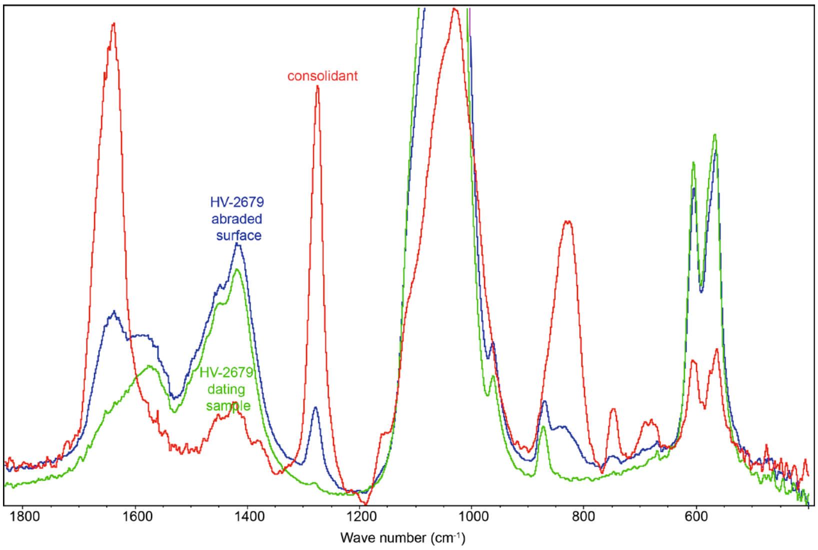

Fig. 2. ATR-FTIR spectra from abraded surface powder and dating sample from the same artefact, showing that peaks at c. 1650, c. 1280, c. 830 and c. 750 cm^{-1} in the surface powder spectrum coincide with those in the HV-3858 consolidant. Of these, only the c. 1280 cm^{-1} peak is visible (just) in the dating sample, suggesting that the consolidant was concentrated at the surface and was largely removed simply by light abrasion with a dental drill before sampling.

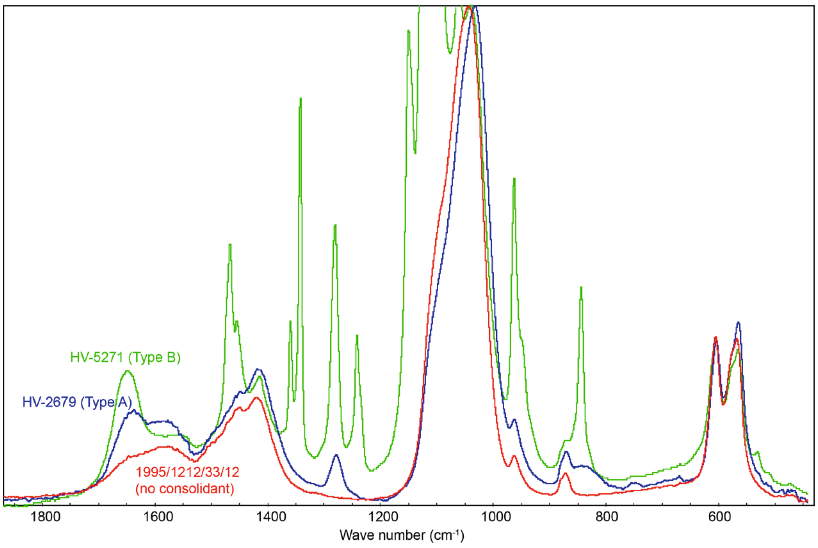

Fig. 3. Three ATR-FTIR spectra from untreated bone/antler surface powder scaled to the height of the phosphate peak (c. 1035 cm^{-1}). All three samples had negligible collagen contents; differences between the spectra of HV-2679, HV-5271 and the unconsolidated 1995 sample should therefore be due to contamination. Although the HV-2679 peaks at c. 1650, c. 1280 and c. 830 cm^{-1} are even more prominent in the HV-5271 spectrum, the latter includes several additional peaks suggesting that these artefacts were treated with different consolidants, not simply different amounts of the same consolidant.

At Kiel, dating samples were pretreated in a Soxhlet-type apparatus, using a sequence of boiling solvents: tetrahydrofurane, chloroform, petroleum-ether, acetone and methanol (3 x each; each solvent should remove its predecessor), and rinsed 4 x with demineralised water (Bruhn et al. 2001).

After drying, they were tested again by FTIR, before collagen extraction at room temperature following a modified Longin protocol, consisting of full demineralization in c. 1 % HCl, followed by 1 % NaOH (1 h) to remove possible humic substances and then 1 % HCl again (1 h) to remove any CO_2 absorbed during the NaOH step, with multiple rinses between each reagent. Collagen was gelatinized in a covered test tube in a pH=2.7 solution (85 °C, 17 h), and insoluble particles were removed by filtration through a pre-baked 0.45 µm pore silver filter. The freeze-dried collagen was tested again by FTIR. An aliquot was sealed for AMS dating (after Nadeau et al. 1998), and where possible a second aliquot was sent to Bradford or the Department of Physics and Astronomy, Aarhus University, for EA-IRMS analysis.

In Brussels, FTIR analysis before pretreatment of some samples confirmed the results obtained in Kiel. Samples were pretreated by ultrasonication in warm acetone (40 °C, 30 min, 2 x). After repeated rinsing, collagen was extracted following a modified Longin protocol, by demineralization in 8 % HCl (15 min), alkaline washing with 1 % NaOH (15 min), and a final treatment with 1 % HCl. After each treatment the sample was thoroughly rinsed using MilliQ water. The extract was gelatinized in a closed test tube filled with a pH=3 solution (90 °C, 10 h). The gelatin was filtered through a 0.7 µm pore hydrophilic glass fibre filter (Merck-Milipore) and then freeze-dried for 10–15 h. All collagen extracts were tested by FTIR. Collagen was combusted and graphitised using an AGE3 automated system and graphite targets were dated in a MICADAS AMS system (Boudin et al. 2015). Where possible, an aliquot of collagen was sent for EA-IRMS analysis at the Department of Earth and Environmental Sciences, KU Leuven.

3 Results

3.1 Identification of consolidants

The FTIR spectrum of the 'Type A' consolidant scraped off HV-3858 has a prominent peak at c. 1280 cm^{-1} and minor peaks at c. 830 and c. 750 cm^{-1} which are not seen in unconsolidated bone, as well as strong absorption at c. 1650 and c. 1025 cm^{-1}, which would coincide with peaks in spectra of well-preserved bone. Mitchell et al. (2013) compared ATR-FTIR spectra from several early polymers, and recorded prominent peaks at 1647, 1274 and 830 cm-1 in cellulose nitrate, with minor peaks at 1721 and 749 cm^{-1} and a broader peak centered on 1065 cm^{-1}. Cellulose nitrate was used by conservators in East Germany to consolidate bone and antler artefacts (Ines Quitsch, pers. comm.). Elemental analysis of the HV-3858 consolidant (22 % C, 6 % N) suggests mixing with other products, but does not exclude that cellulose nitrate ($C_{18}H_{21}N_{11}O_{38}$) was a major constituent. The consolidant's $\delta^{13}C$ (-25.5 ‰) merely suggests an organic origin, but its depleted $\delta^{15}N$ value (-18.4 ‰) implies that the nitrogen did not come from collagen. Its ^{14}C concentration of 71.34 ± 0.27 pMC (KIA-50663, 2710 ± 30 BP) implies that carbon in the Type A consolidant came from both recent and fossil sources[1]. Unless the proportions of recent and fossil carbon varied significantly between artefacts, however, any contamination from Type A consolidant would lead to results which are too young, not too old.

The Type B consolidant could not be tested directly, but HV-3828, which had an unacceptably low collagen content and an FTIR spectrum showing the Type B consolidant, was dated. The ^{14}C result (RICH-22171) is implausibly early for the species, location and artefact type, which suggests that the dated extract was contaminated with fossil carbon, probably from the Type B consolidant, as the collagen FTIR spectrum included a peak not associated with uncontaminated collagen or cellulose nitrate. However, HV-3843, a well-preserved sample (10 % collagen yield) with an FTIR spectrum showing the Type B consolidant, was dated in Kiel, giving a plausible ^{14}C result (KIA-51093). Its collagen FTIR spectrum was identical to that of reference uncontaminated collagen.

3.2 Removal of consolidants

Cellulose nitrate should dissolve in acetone, yet FTIR spectra of even Soxhlet-treated dating samples (before demineralization) showed minor peaks at c. 1280 cm^{-1}. Thus solvent extraction appears not to have been 100 % effective[2]. FTIR spectra of collagen extracts did not show a peak at 1280 cm^{-1}, however, and were indistinguishable from spectra of uncontaminated collagen (Fig. 4). Either the consolidant

1 Crann/Grant (2019) reported a ^{14}C concentration of 63.21 ± 0.23 pMC (UOC-2017, 3685 ± 29 BP) in cellulose nitrate produced in 2005; the higher ^{14}C concentration in the Hohen Viecheln consolidant probably reflects the fact that it would have been produced in the 1960s, when atmospheric ^{14}C levels were much higher than in 2005.

2 Brock et al. (2018) also reported that cellulose-nitrate-based consolidants were not easily removed with normal solvent extraction methods, but they tested recently produced commercial products that may have contained plasticisers and other additives, which were not used in the 1960s, when problems with the long-term stability of cellulose-nitrate-based consolidants were not recognised.

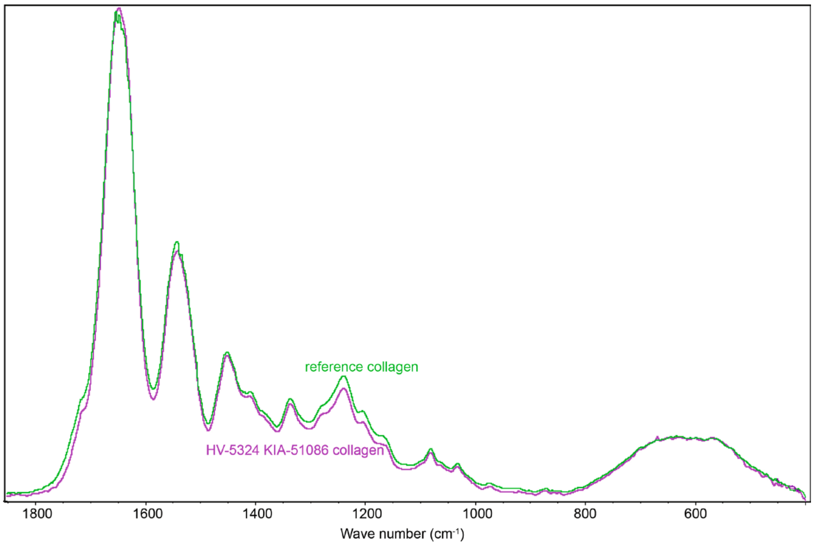

Fig. 4. ATR-FTIR spectra from collagen extracted after Soxhlet pretreatment. The reference collagen is from an unconsolidated bone used as a dating standard (VIRI F). HV-5324 appeared to be heavily contaminated by Type A consolidant, but FTIR is unable to detect any contamination in the collagen extract.

remained insoluble, and was filtered out,[3] or the chemical bond responsible for the 1280 cm^{-1} peak was broken during collagen gelatinization (which seems unlikely, as the heights and positions of the 1650, 1280, 830 and 750 cm^{-1} peaks did not change when the HV-3858 consolidant was left in 65 °C water for two weeks). In an artificial ageing experiment, Shashoua et al. (1992) were able to greatly simplify the FTIR spectrum of cellulose nitrate, but the 1280 cm^{-1} peak was still prominent after extended exposure to moderate heat (60–100 °C, up to 80 days). It is difficult to check the ATR-FTIR detection limit for the consolidant dissolved in collagen, but gelatinization does not appear to explain the absence of the 1280 cm^{-1} peak in our collagen extracts.

3.3 EA-IRMS

Where possible, collagen was split for EA-IRMS analysis as well as AMS dating. Collagen yields were highly variable: a fifth of the objects yielded <1 % collagen by weight, and almost as many gave >10 % collagen. Elemental analysis (% C, % N) allows the atomic C:N ratio to be calculated; a value in the range of 2.9–3.6 is usually regarded as evidence of acceptable collagen quality (DeNiro 1985). Whilst all the measured extracts gave C:N ratios in this range, the five dates from low-yielding (1–4 % collagen) samples with no EA-IRMS data should naturally be viewed with caution. The stable isotope data ($\delta^{13}C$ and $\delta^{15}N$) are tightly clustered (Fig. 5) and are consistent with expectations for early Holocene herbivores in northern Europe. The relatively low $\delta^{15}N$ values of +4 ‰ are normal for elk (e.g. δIANA Dietary Isotopic Baseline for the Ancient North, http://www.oasisnorth.org/), and somewhat higher $\delta^{15}N$ values for aurochs (i.e. HoVi-609) are also unsurprising (e.g. Meadows et al. 2016). Thus there is no indication in the EA-IRMS data that the ^{14}C results might be compromised.

3 Unfortunately filters were not retained, so it was not possible to check the FTIR spectra of insoluble residues.

3.4 ^{14}C

The overall range of ^{14}C ages is broad, but, when converted to calendar dates using OxCal v4.2.4 (Bronk Ramsey 2009) and the IntCal13 calibration curve (Reimer et al. 2013), consistent with expectations that Hohen Viecheln was mainly occupied from the mid-9th millennium to the early 7th millennium cal. BC (Table 1; Fig. 6). Two Hohen Viecheln samples (the antler headdress HV-5863, and HV-5314, a worked metapodial) appear to date to the early 9th millennium, as does the Biesdorf sample. The antler headdress dates are plausible for this artefact type (Wild et al. in prep.). Such a wide date range may mask significant ^{14}C offsets, which cannot be detected from contextual data or typological attributions. Inter-laboratory replication should provide some quality control; as the laboratories used different decontamination methods, consistent replicate results would imply that both methods removed all (or nearly all) contamination. Of the eight replicated samples, however, only three had acceptable collagen contents (Table 1: HV-5610, HoVi-609, Biesdorf). The slight discrepancy (150 ± 62, 2.44σ) between the HV-5610 results may be unrelated to contamination, as FTIR suggested that little if any consolidant had penetrated the HV-5610 dating sample. Differences between results from the other replicated samples were insignificant (HoVi-609, 13 ± 60, 0.22σ; Biesdorf, 64 ± 67, 0.95σ). There is no apparent relationship between dating laboratory, collagen content, or FTIR evidence of consolidants in the dating sample and calibrated ^{14}C dates (Fig. 6).

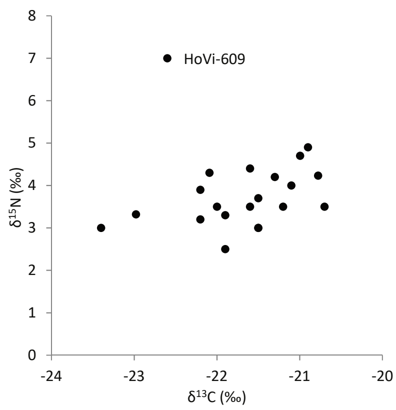

Fig. 5. Stable isotope results, collagen extracts from Hohen Viecheln bone/antler artefacts. HoVi-609 was identified as an aurochs bone, possibly the only one sampled, which may account for its higher $\delta^{15}N$ value. The relatively low $\delta^{15}N$ values of other samples are typical of elk, and may also reflect the date range of the material.

4 Discussion

This paper is not concerned with the archaeological implications of ^{14}C ages from the Hohen Viecheln artefacts, only with the reliability of these results. There is no obvious reason to doubt the ^{14}C ages, but equally we cannot prove that all traces of consolidant were removed during collagen extraction. More rigorous procedures for dealing with potential organic contamination (e.g. isolation of hydroxyproline: Fiedel et al. 2013) are not available routinely, and would require larger samples. Such approaches may be necessary when dating important isolated finds, but at Hohen Viecheln we were interested in dating a representative number of objects from a large assemblage.

To gauge the risk that seriously misleading ^{14}C ages could be obtained from collagen with undetectable contamination, elemental concentrations and isotopic values in the Type A consolidant (Table 1, KIA-50663) were used to calculate C:N, $\delta^{13}C$, $\delta^{15}N$ and ^{14}C values in hypothetical mixtures of uncontaminated collagen (typically 42 % C, 15 % N, $\delta^{13}C$ -22 ‰, $\delta^{15}N$ +4‰, and 30 pMC) and different amounts of consolidant. If 10 % of the mass of an extract were Type A consolidant, its ^{14}C age would be c. 500 years too young, but its C:N value would be only c. 3.4, well within the normally accepted range for pure collagen. The same contamination would lower the extract $\delta^{13}C$ by 0.3 ‰ and its $\delta^{15}N$ by 1 ‰, not enough for the sample to be rejected.

The amount of consolidant in an extract depends not only on how much was removed during pretreatment, but also on how much was originally applied and penetrated below the surface, which may have been inversely proportional to collagen content (assuming low-collagen bones were more

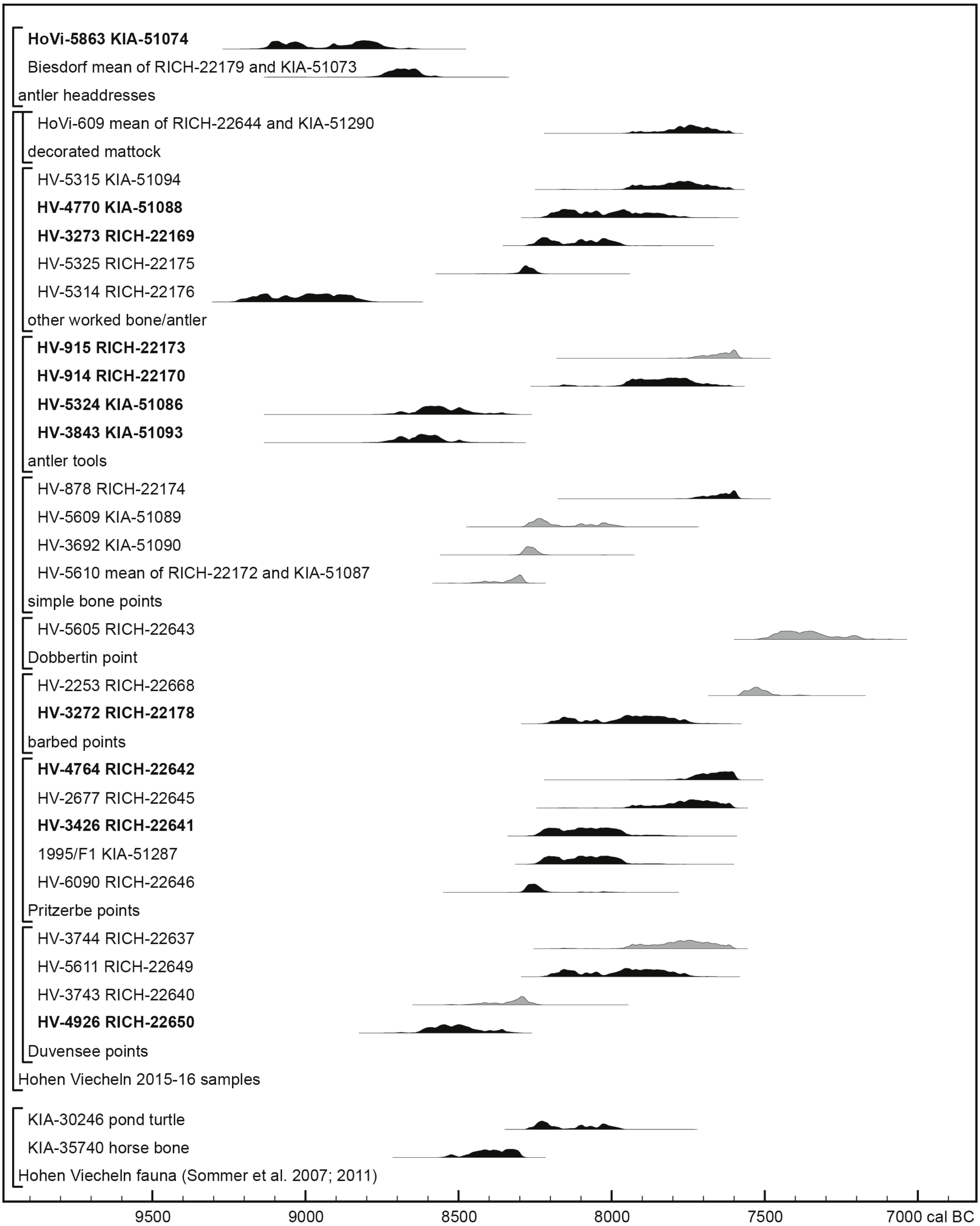

Fig. 6. Calibration of ^{14}C results to calendar dates using OxCal v4.2.4 (Bronk Ramsey 2009) and the IntCal13 calibration curve (Reimer et al. 2013). The calibrated date for KIA-30246 (pond turtle) should be regarded as a maximum age for this bone as a significant freshwater reservoir effect is likely. Grey: results from samples with 1–5 % collagen yields. Bold type: FTIR spectrum of dating sample before pretreatment had a peak at 1280 cm^{-1}. Replicate results were combined before calibration (OxCal function R_Combine).

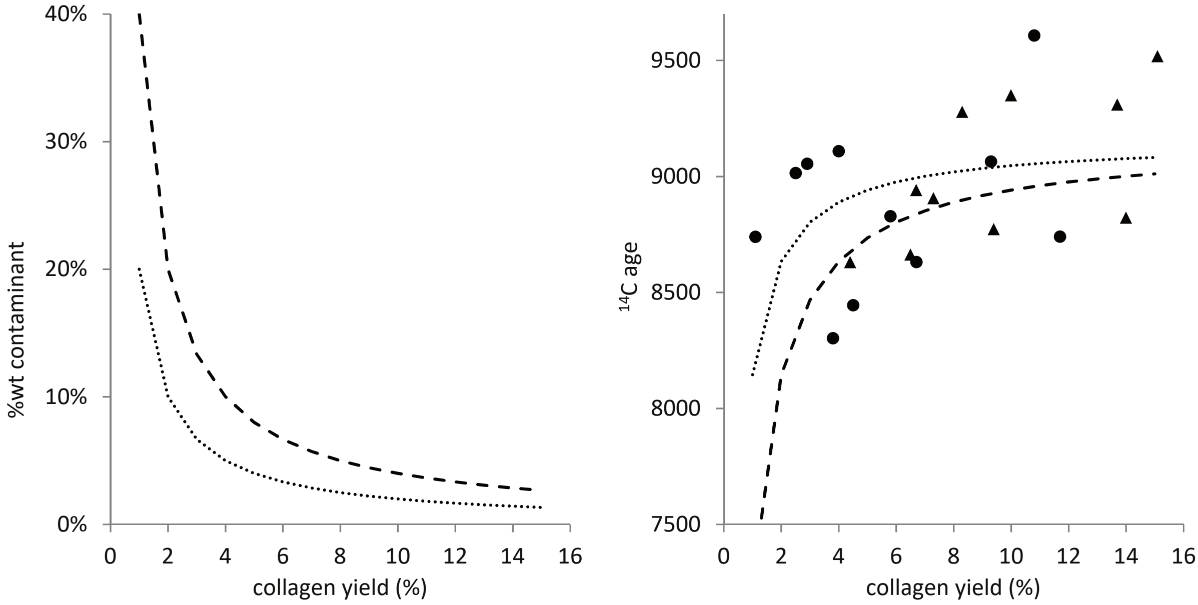

Fig. 7. Left: hypothetical contamination as a % of the total weight of the extract from a 250 mg bone/antler sample, as a function of the collagen yield (dashed line: 1 mg of consolidant in extract; dotted line: 100 µg of consolidant in extract). Right: Hohen Viecheln artefact bone/antler ^{14}C ages plotted against collagen yields (triangles: 1280 cm^{-1} peak visible in FTIR spectrum of untreated dating sample; dots: no 1280 cm^{-1} peak visible in dating sample). Lines represent ^{14}C ages of hypothetical extracts from a 250 mg bone/antler sample with a true ^{14}C age of 9150 BP, if 1 mg (dashed line) or 100 µg (dotted line) of Type A consolidant remains in the dated extract.

fragile and porous). Even if a constant amount of consolidant was left in the extract, the effective level of contamination would increase exponentially as the collagen yield decreased (Fig. 7 left). If ^{14}C ages are plotted against collagen yields, and lower yields appear to be associated with younger ages, it may therefore be suspected that much of the variation in ^{14}C ages is due to differences in contamination by Type A consolidant (Fig. 7 right), particularly if the rejected ^{14}C result for HV-2137 is considered. However, it is not unlikely that collagen preservation was better in deeper layers (a pattern observed at other wetland sites in the region), and at Hohen Viecheln there is no relationship between collagen yields or ^{14}C ages and the presence of a 1280 cm^{-1} peak in FTIR spectra of untreated dating samples (Fig. 7 right). Moreover, if the Type A consolidant was responsible for younger ^{14}C ages in low-collagen samples, it would also lower their $\delta^{15}N$ values, but low-collagen samples tend to have higher $\delta^{15}N$ (Table 1). Thus although only small amounts of consolidant would produce significant ^{14}C age offsets in low-collagen samples, the Hohen Viecheln results do not indicate systematic contamination problems.

5 Conclusion

The Hohen Viecheln dating program has shown that it is possible to apply several quality-control tests while dating bone/antler samples (both powder and fragments) of <300 mg, using normal laboratory techniques and equipment. The use of minimally invasive sampling and routine laboratory procedures was essential to the feasibility and objectives of the dating program.

Variability in collagen preservation at Hohen Viecheln, while often frustrating, provided an opportunity to assess the effectiveness of the dating program, as we could model the dependence of various parameters on collagen yield, and compare hypothetical data to actual results. One sample of relatively pure consolidant was analysed, and if the results are representative, it is clear that normal acceptance criteria (collagen yield, C:N, stable isotopes) are insufficient to validate the bone/antler ^{14}C ages. Inter-laboratory replication was attempted, with some success, but it is the overall pattern of results, particularly the lack of correlation between FTIR evidence of contamination before pretreatment and any of the data from the collagen extracts, which supports our interpretation that the ^{14}C ages are essentially reliable. Nevertheless, in any chronological interpretation, it would be advisable to give more weight to the dates of samples with higher collagen yields, and to treat with considerable caution results from samples without EA-IRMS data.

Acknowledgements

Most analyses reported here were funded by German Research Foundation (Deutsche Forschungsgemeinschaft) grant ME 4557/1-1, within the DFG-funded project 'A reassessment of the chronology and stratigraphy of the early Holocene site Hohen Viecheln [Mecklenburg-Western Pomerania] with special focus on diagnostic bone artefacts', directed by Daniel Groß, Harald Lübke, John Meadows and Detlef Jantzen. The study is also part of the CRC 1266 'Scales of Transformation' (DFG Projektnummer 2901391021 – SFB 1266). Analyses of antler headdresses were funded by the Centre for Baltic and Scandinavian Archaeology (ZBSA), Schloss Gottorf, under its research theme 'Man and Artefact', for Markus Wild, and University of Cologne, Institute for Prehistory, DFG-CRC 806 Project D4, for Birgit Gehlen. We are grateful to Peter Teichert-Köster (Landesamt für Kultur- und Denkmalpflege Mecklenburg-Vorpommern) and Martina Weinland and Ines Quitsch (Stiftung Stadtmuseum Berlin, Landesmuseum für Kultur und Geschichte Berlins) for facilitating sampling; to technical staff of the Royal Institute for Cultural Heritage Radiocarbon Dating Laboratory (Brussels) and Leibniz-Laboratory for AMS Dating and Isotope Research (Christian-Albrechts-University, Kiel) for extracting and dating the samples; and to Steven Bouillon (Department of Earth and Environmental Sciences, KU Leuven), Julia Beaumont (School of Archaeological Sciences, University of Bradford) and Marie Kanstrup (Department of Physics and Astronomy, Aarhus University) for EA-IRMS analyses. Robert Sommer (Rostock University) is thanked for providing literature.

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Table 1. (next two pages) Analytical results. ‡see text and Figure 3 for explanation of consolidant types, which are based on FTIR spectra of untreated material. Bold type indicates that the consolidant was also detected in the untreated dating sample, not only in powder abraded from the surface. § calculated from CO_2 pressure; + insufficient collagen for EA-IRMS. A significant fraction of the bone powder from KIA-51088, 51089, and 51094 was lost during the Soxhlet process, and, as the solvent-extracted powder recovered was not weighed before demineralisation, collagen weights from these samples under-estimate their true collagen contents.

Table 1. See previous page for legend.

Object	Sample	Laboratory code	Consolidant‡
HV-3858 articulating perforated aurochs phalanges 2 and 3	consolidant 15 mg	KIA-50663	A
HV-4926 bone point Duvensee	fragment 222 mg	RICH-22650	**A**
HV-3743 bone point Duvensee	powder 244 mg	RICH-22640	A
HV-5611 bone point Duvensee	fragment 248 mg	RICH-22649	A
HV-3744 bone point Duvensee	powder 261 mg	RICH-22637	A
1995 1212/33/12 bone point Duvensee	fragments c. 0.4 g	KIA-51286	-
HV-6090 bone point Pritzerbe	powder 296 mg	RICH-22646	A
1995 F1 bone point Pritzerbe	powder 277 mg	KIA-51287	-
HV-3426 bone point Pritzerbe	powder 296 mg	RICH-22641	**A**
HV-2677 bone point Pritzerbe	powder 240 mg	RICH-22645	A
HV-4764 bone point Pritzerbe	powder 253 mg	RICH-22642	**A**
HV-3870 bone point Pritzerbe	powder 270 mg	KIA-51288	A
HV-2679 bone point Pritzerbe	powder 296 mg	RICH-22647	A
HV-2135 bone point Pritzerbe	powder 296 mg	RICH-22178	A
HV-3272 bone point barbed	fragment 192 mg	RICH-22178	**A**
HV-2253 bone point barbed	fragment 210 mg	RICH-22668	A
HV-5605 bone point Dobbertin	powder 288 mg	RICH-22643	none?
HV-5610 bone point simple	fragment 647 mg	RICH-22172 KIA-51087	A
HV-3692 bone point simple	fragment 233 mg	KIA-51090	A
HV-5609 bone point simple	powder 230 mg	KIA-51089	A
HV-878 bone point simple	powder 282 mg	RICH-22174	A
HV-3843 antler axe	powder 267 mg	KIA-51093	**B**
HV-5324 antler axe	powder 367 mg	KIA-51086	**A**
HV-914 antler socket	powder 280 mg	RICH-22170	**A**
HV-915 antler socket	powder 263 mg	RICH-22173	**A**
HV-5271 antler axe	powder 291 mg	KIA-51093	B
HV-5314 metapodial production waste	powder c. 0.3 g	RICH-22176	A
HV-5325 antler tine	powder 296 mg	RICH-22175	A
HV-3273 bone splinter	powder 303 mg	RICH-22169	A
HV-4770 perforated wild boar(?) phalanx	powder 291 mg	KIA-51088	**A**
HV-5315 metapodial production waste	powder 253 mg	KIA-51094	**A**
HV-3828 perforated elk(?) antler	fragments 881 mg	RICH-22171 KIA-51085	B
HoVi-609 decorated mattock, aurochs radius	powder 559 mg	RICH-22644 KIA-51290	none?
HoVi-610 decorated bone bâton de commandement	fragment 495 mg	RICH-22648 KIA-51289	none?
HV-2137 decorated antler	powder 693 mg	RICH-22177 KIA-51289	A+?
HV-5863 red deer 'antler headdress'; skull bone	powder 367 mg	KIA-51074	**A**
Biesdorf red deer 'antler headdress'; antler	powder c. 0.5 g	RICH-22179 KIA-51073	A

Table 1. continued.

Yield (%)	% C	% N	C:N	δ13 (‰)	δ15 (‰)	14C age BP	Calibrated date, cal. BC (95 % probability)
-	21.9	5.9	4.3	-25.3	-18.4	2712 ± 30	(modern)
8.3	34.2	12.2	3.3	-21.9	2.5	9278 ± 44	8630–8340
4	41.3	15	3.2	-20.9	4.9	9109 ± 49	8460–8240
5.8	30.6	10.9	3.3	-21.6	3.5	8829 ± 44	8210–7750
1.1	+	+	+	+	+	8740 ± 44	7950–7610
0							
2.5	38.9	14.1	3.2	-21.1	4.0	9015 ± 43	8310–7990
4.6	36.9	13.6	3.2	-20.8	4.2	8908 ± 42	8250–7950
7.3	32.8	11.8	3.3	-22.2	3.2	8906 ± 49	8260–7840
3.3	+	+	+	+	+	8728 ± 42	7940–7600
6.5	41.1	14.7	3.3	-21.3	4.2	8663 ± 44	7790–7580
0							
0							
0							
14.0	33.7	11.6	3.4	-21.5	3.0	8822 ± 45	8210–7740
4.5	28.6	9.3	3.1	-23.4	3.0	8445 ± 43	7590–7380
3.8	39.5	14.0	3.3	-20.7	3.5	8303 ± 47	7500–7180
2.9	33.2	11.6	3.3	-21.6	4.4	9055 ± 44	8330–8210
8.6	39.6	14.4	3.2	-21.1	4.5	9205 ± 43	8550–8300
3.9	42§	+	+	+	+	9043 ± 42	8310–8210
>3.2	40§	+	+	+	+	8973 ± 46	8290–7970
6.7	45.9	16.2	3.3	-22.0	3.5	8631 ± 41	7740–7580
10.0	38.6	13.8	3.3	-23.0	3.3	9349 ± 45	8750–8470
13.7	44.4	16.2	3.2	-22.1	4.3	9309 ± 51	8720–8340
9.4	37.6	13.0	3.4	-21.5	3.0	8772 ± 43	8170–7610
4.4	26.8	8.7	3.6	-22.2	3.9	8630 ± 42	7740–7580
0							
10.8	30.7	10.9	3.3	-21.2	3.5	9608 ± 44	9220–8820
9.3	31.3	10.9	3.4	-21.5	3.7	9064 ± 43	8340–8220
6.7	33.3	11.4	3.4	-21.9	3.3	8941 ± 44	8270–7960
>3.2	39§	+	+	+	+	8850 ± 41	8210–7790
>7.3	39.7	14.5	3.2	-21.0	4.7	8748 ± 39	7950–7610
<1	+	+	+	+	+	(10427 ± 52)	rejected
0							
11.7	41.1	15.1	3.2	-22.6	7.0	8741 ± 43	7940–7600
3.9	40.2	14.7	3.2	-22.6	7.4	8728 ± 42	
0							
0							
<1	+	+	+	+	+	(7516 ± 41)	rejected
0							
15.1	46§					9518 ± 46	9140–8710
8.7	37.8	13.1	3.4	-22.0	3.3	9425 ± 45	8770–8570
	43.6	15.8	3.2	-21.1	4.7	9361 ± 50	

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