Invisible Technologies and the Container Revolution

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Abstract

This thesis evaluates the potential for fire-cracked rocks (FCRs) and organic cooking containers to be analysed and studied utilising methodologies previously used for archaeological ceramics. The development and spread of pottery technologies throughout the Holocene in the Old World represents a shift - both archaeologically and for the preservation potential of organic residue (OR) traces retained in the ceramic matrix. It has been possible to reveal the functions of these vessels by extracting and analysing lipids and small molecules. However, the cooking methods and food processing techniques that existed prior to the use of ceramics are less well characterised. This thesis aims to elucidate some of these techniques through a combined methodological approach of actualistic experimentation and organic residue analysis. Firstly these 'aceramic' cooking methods were systematised through reading the ethnographic and archaeological literature, then an experimental project undertaken to quantify some of the material differences between types of organic cooking vessels and heating methods. The previous OR analysis undertaken on stone was then compiled, and several experimental cooking systems were tested to examine whether crucial thermal biomarkers could be formed in stone. Finally six Mesolithic and Neolithic sites in northern Europe were sampled for FCRs, which were subject to lipid extraction and analysis. The results demonstrate that, not only is stone capable of retaining sufficient lipid quantities and producing anthropogenic biomarkers, but stone appears to differ in its thermal reaction to lipids. The results also suggest that organic cooking vessels made from animal materials possess different thermodynamic qualities to ceramics which favour sub-boiling temperatures. Finally the archaeological results from the northern European Mesolithic/Neolithic sites reveal several aceramic heating techniques (marine fat rendering/combustion, earth-oven technologies) and point to the multiplicity of cooking strategies among late foragers and early farmers, even where pottery vessels were available.

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List of Abbreviations

APAA: ω-(o-alkylphenyl)alkanoic acids

ACPAA: ω-(2-alkycyclopentyl) alkanoic acids

AQUASIM: aquatic scanning ion mode

BC: Before Christ

BCFA: branched chain fatty acid

BP: Before present, conventionally the year 1950

BPCA: benzene polycarboxylic acid

BSTFA: N,O-Bis(trimethylsilyl)trifluoroacetamide

cm: centimetre

DB5: type of gas chromatography column

DB23: type of gas chromatography column

DCM: dichloromethane

EBK: Ertebølle culture

eHRAF: Electronic human resource area files (Yale University database)

FA: fatty acid

FCR(s): Fire-cracked rock(s)

GC-FID: gas chromatography flame ionisation detection

GC-MS: gas chromatography-mass spectrometry

GC-C-IRMS: gas chromatography-combustion-isotopic ratio mass spectrometry

HCL: hydrochloric acid

L: litre

LCFA: long-chain fatty acid

LCK: long-chain ketone

LTLT: long-time, low-temperature

MCFA: medium chain fatty acid

m/z: mass to charge ratio

µg: microgram

mg: milligram

ml: mililitre

MS: mass spectrometry

ORA: Organic residue analysis

PCA: principal componant analysis

pH: the logarithmic scale for defining the acidity or alkalinity of a solution

PUFA: polyunsaturated fatty acids

SCFA: short chain fatty acid

SIM: selected ion monitoring

SRR: 3S,7R,11R,15-phytanic acid

TRB: Funnel Beaker culture

RRR: 3R,7R,11R,15-phytanic acid

TAG: Triacylglycerol

TMTD: 4,8,12-trimethyltridecanoic acid

UFA: unsaturated fatty acid

VLC: very long chain

Acknowledgments

In theory a PhD is meant to represent an individual piece of work, a solo journey. Yet my experience has been one of standing on the shoulders of all those who made it possible to even ask the questions that I have attempted to tackle. I am deeply grateful and humbled to have been a part of so many fascinating intersecting projects and lines of research.

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Author declaration

I declare that this thesis is a presentation of original work and I am the sole author. This work has not previously been presented for a degree or qualification at this, or any other, university or institution. All sources are acknowledged as references. The work carried out as part of this thesis has resulted in the following publication, presented here as chapter four:

Andy Langley, Andy Needham, Roland Kröger, Gabriel Cifuentes-Alcobendas, Mette Adegeest, Jess Cousen, Christopher Lance, Hannah Benton, Amy-Rose Mansbridge, Amanda Satchell, Lewis Tomlinson, Francesca Rockall-Birtles, Alexandre Lucquin, Aimée Little (2023). An experimental study of wet-cooking in organic vessels: implications for understanding the evolution of cooking technologies. Archaeological and Anthropological Sciences 15:142

The following papers are in the process of being prepared for submission, presented here as chapters six and seven respectively:

Andy Langley, Ivy Notterpek , Aija Macāne, Kerkko Nordqvist, Oliver E. Craig, Aimée Little, Alexandre Lucquin. Experimental evidence for heating biomarkers in archaeological fire-cracked rocks: potential for identifying aceramic cooking practices. *To submit to Archaeometry*

Andy Langley, Oliver E. Craig, Aimée Little, Heidi Mjelva Breivik, Harry Robson, Søren H. Andersen, Sönke Hartz, Alex Lucquin. Invisible technologies? Using organic residue analysis to reveal prehistoric aceramic cooking practices in Mesolithic and Neolithic Northern Europe. *To submit to Journal of Archaeological Science*

Chapter One: Introduction

1. Research Context: Before Pottery

It is widely acknowledged that the development of ceramic vessels was amongst the most significant technological innovations during human prehistory (Rice, 1999; Jordan and Zvelebil, 2016; Tsetlin, 2018). Not only are ceramics extremely useful as a *plastic* material which can form durable, reusable and portable cooking containers, but their longevity and resistance to degradation makes ceramic pots and sherds invaluable for archaeological research (Heron and Evershed, 1993; Pollard and Heron, 2008). However, this visibility has highlighted the relative invisibility of non-ceramic, or *aceramic* containers and vessels which were presumably commonplace throughout the Palaeolithic and likely utilised by other human species (Carbonell *et al.*, 1996). The act of *enclosing* foods within a vessel was one element in a technological revolution of manufacturing general containers - including baskets, nets, slings, water transportation and storage (Langley and Suddendorf, 2020, 2022). The ability to combine food with water - *wet-cooking* - made it possible to thermally modify and process all manner of small ingredients, many of which require heat treatment to render them edible (Crowther, 2012). Once foods could be contained within a vessel then many biological processes such as protein denaturation, starch gelatinisation, sterilisation, detoxification and fat rendering could be more easily conducted within a controlled microenvironment. Although very few examples of organic containers have survived in the archaeological record, there is an abundance of heated stone, or *fire-cracked rocks* (FCRs) across millennia of prehistoric contexts, which point to the potential existence of creative and diverse *aceramic cooking technologies* (Rapp, Balescu and Lamothe, 1999; Brink and Dawe, 2003; Thoms, 2008; Nakazawa et al., 2009).

These ‘invisible technologies’ extend beyond containers, to encompass all manner of food processing and cooking techniques. Drying, smoking, fermenting, soaking, brining, curing, boiling, simmering, roasting, baking, parching, frying, broiling and many other methods form the basis of food preparation, cooking and cuisine (Wandsnider, 1997; Morrison, 2012; Daviau, Hasan and Cowell, 2016; Fuller and Carretero, 2018; Graff, 2018) -

only some of which necessitate the use of FCRs or organic vessels. Identifying this wide range of activities in the archaeological record is difficult, and often requires studying the ethnoarchaeological record to provide pertinent examples and analogies (Nojima, 2008; Nelson, 2010). Even so, the material capacities and functionality of aceramic technologies remains poorly understood and experimentally under-investigated (Speth, 2015). FCRs themselves can be created through multiple pathways, many of which have little or nothing to do with cooking, as explained in Chapter Three. Despite this, the biomolecular evidence for aceramic food processing and organic container use continues to grow, offering a glimpse into pre-ceramic cuisines and technologies (Kabukcu *et al.*, 2023; Golovanova, Kostina and Doronichev, 2024). The question which continually returns in this context of pre-pottery cooking however, is *why invent ceramics at all?*

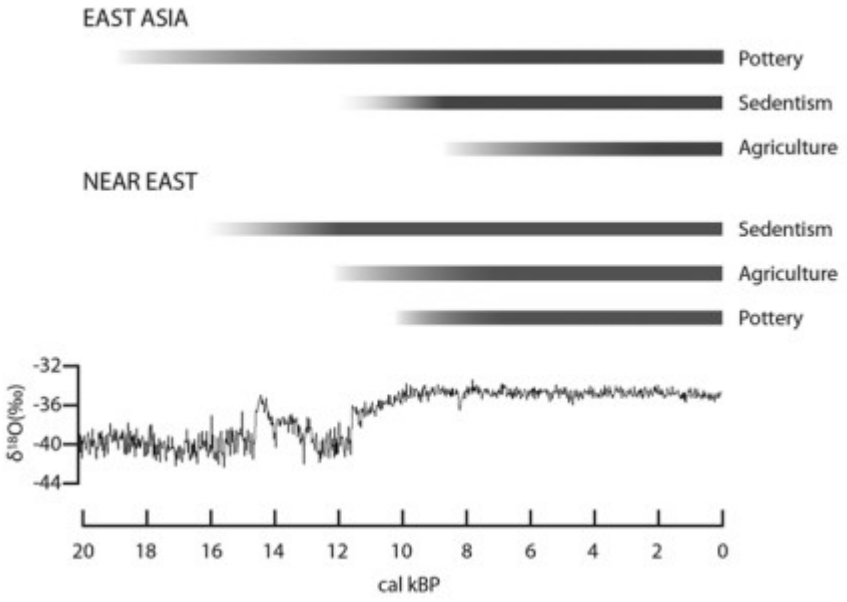

Fig 1.1 The approximate starting dates for pottery, sedentism and agriculture associated with two broad Neolithic transitions (East Asia and the Near East). Image after Gibbs, 2015.

So far there has been no clear consensus on identifying one key 'push-or-pull' mechanism which helped drive the invention and adoption of ceramics (Jordan and Zvelebil, 2016). In fact, each independent innovation in developing ceramics seems embedded within its own contingent factors, with no obvious unilinear evolutionary pattern emerging which could tie pottery to the invention of agriculture or the process of sedentarism (Fig.1.1). One puzzle in the question of ceramic invention is identifying any *advantage* that crude or 'proto-pottery' may have offered over established aceramic technologies and methods. Hayden

(1995) and Rice (1999) have both developed models for pottery development and adoption based on the desire of particular 'aggrandizing' individuals to acquire prestige and status, perhaps through competitive-feasting. They predict that this shift would involve the seasonal exploitation of particular foods (carbohydrates or high calorie lipid products) which necessitate more intensive exploitation of resources using specific forms of ceramic vessels. Jordan & Zvelebil (2016) have critiqued this model based on the complex *chaîne opératoire* of pottery production, that it would be a more general access prestige technology, rather than one controlled by a small number of people. One of the attractive points to this model though, is that it helps explain the difficult transition stage between crude pottery and its later refinement. As Brown (1989) has highlighted, if aceramic cooking vessels were previously available, and early ceramics were not as instantly efficient or labour-saving, why persist in their development at all? Removing performance from the equation and focusing on the prestige of a novel technology perhaps helps us explain this transition stage? What we lack though are good quantitative characterisations of the different aceramic technologies in order to respond to Brown's point.

As noted, the immediate advantages of early pottery are not obvious, and certainly the first generations of ceramic vessels would not have been capable of sustaining lengthy, high temperatures (Reid, 1984; Brown, 1989). Despite this many have argued that even simple pottery represents something of an improvement on aceramic methods. Reid (1984) summarises the debates concerning the transition from stone boiling (indirect heat transfer) to ceramics during the North American Early Woodland period, stating that pottery, even early pottery, possesses advantages in time, labour and resources, as well as the ability to process more difficult foods such as nuts, seeds and bones. Thus Jordan and Gibbs identify this possible *functional* advantage in the form of *direct boiling*, in which they see even the most basic pottery being more robust and utilitarian than any known aceramic boiling methods. Jordan & Zvelebil (2016) outline how this initial phase of Eurasian pottery development - the 'experimentation' phase - between 16,000-10,000 BC, involved a transition from simplistic and basic vessels towards lighter, plant fibre-tempered pots. This raises the possibility that ceramics arose directly from aceramic technologies through the simple association of clay with organic 'templates' such as nets or baskets - indeed there are early archaeological examples of fired clay fragments where the impressions of woven cordage or netting is clearly visible ((Zhushchikhovskaya, 1999; Hyland *et al.*, 2012), suggesting that a tradition of lining or coating plant-based containers with clay could have precipitated the exploration of firing and more efficiently manufacturing hybrid vessels, until

the ‘template’ became redundant. Pottery from the Incipient Jomon period also shows animal hair embedded within the clay, hinting that animal skins could have been the pre-ceramic vessel material (Iizuka, 2018). Morphologically the earliest forager pottery appears similar across the globe, a point which Piezonka (2021) argues could reflect either the preceding aceramic technologies previously mentioned, or the common constraints of mobility for ceramic hunter-gatherers. These Eurasian prehistoric findings are supported by archaeology from the American southwest, where researchers have long highlighted the evident transition from basketry to ceramics through the use of basket-moulds (Cushing, 1886; Amsden, 1949).

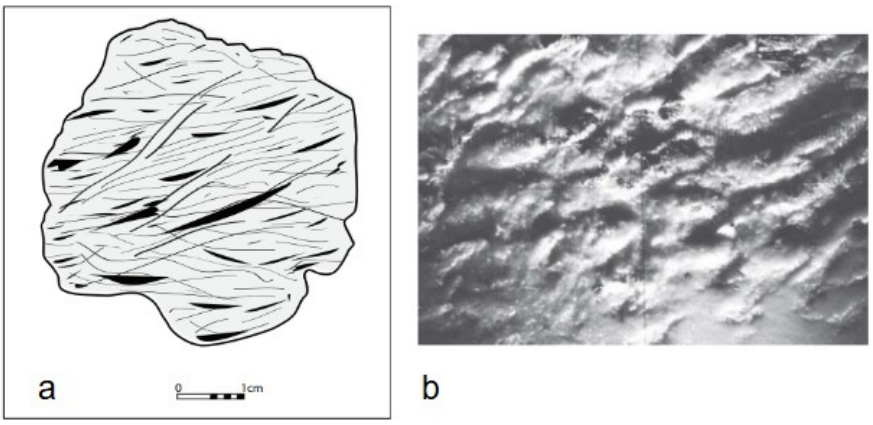

Fig 1.2. Early Russian pottery sherds with imprints of netting/cordage: (a) Chernigovka-1 (Primor’e Region) (from Zhushchikhovskaya, 2016) (b) Gromatukha. Image after Hyland *et al.*, 2012)

Organic residue analysis (ORA) has been able to contribute to this debate through the interpretation of recovered lipids and organic molecules from early pottery vessels. One previous hypothesis supported by ORA studies was the so-called ‘estuarine’ or ‘aquatic model’, where pottery was tied to the increasing sedentarism of Mesolithic Eurasian foragers around aquatic resources (Craig *et al.*, 2013; Lucquin *et al.*, 2016; Gibbs *et al.*, 2017; Hung *et al.*, 2017; Feng and Wang, 2022). In this model the development, refinement and specialisation of ceramics was part of a socio-technological period of *intensification,* focused on aquatic resources. Similarly the earliest proto-ceramics have also been theorised to be material components of resource intensification, linked to the possible drive for grease and fat rendering towards the end of the Pleistocene (Prendergast, Yuan and Bar-Yosef, 2009; Patania and Jaffe, 2021) and, in particular, the climatic downturn of the Younger Dryas (Elston, Guanghui and Dongju, 2011). However, the aquatic model has been challenged by

subsequent ORA results which suggest that pottery was adopted and *adapted* by new groups and communities as it diffused through Eurasia and into eastern Europe and the Baltic coastline (Courel *et al.*, 2020; Bondetti, 2021; Liu *et al.*, 2023) - its function and use tied to specific local needs and culinary practices. This culturally circumstantial aspect of pottery may help explain why it was never universally adopted. Understanding exactly how ceramic vessels came to be used, and whether they *replaced* existing aceramic techniques or complemented them, is a question that remains under study (Papakosta, Oras and Isaksson, 2019; Henderson *et al.*, 2022). The Mesolithic communities that lived around the Baltic and North Sea therefore represent an ideal broad archaeological context to study this transition from aceramic to ceramic use, as well as the transition from ceramic foragers to agriculturalists, in large part due to the extensive study of lipid residues left behind in these diverse pottery vessels (Philippsen and Meadows, 2014; Oras *et al.*, 2017; Robson *et al.*, 2019, 2021; Dolbunova *et al.*, 2022; Lucquin *et al.*, 2023). Many of these comparative ORA works support the wider Eurasian ceramic forager model that adopting pottery was a process of adapting it to the diet and cuisine of a particular community, rather than importing a new 'aquatic Neolithic' mode of subsistence.

Thus two identifiable 'moments' form the wider research context for this PhD: the *origin* of ceramics - what material, social and technological factors help explain the transition from aceramic to early ceramics? and the *adoption* of ceramics - what impact is made upon a forager community when they are introduced to, or adopt pottery from elsewhere? Is it possible to identify a shift in the aceramic foodways when ceramics arrive?

2. Aims & Objectives

2.1. Main objective

The main objective of this thesis is build on multiple lines of previous research, and bring together experimental archaeology and organic residue analysis in order *to evaluate the potential for aceramic cooking technologies to be analysed by adapting methodologies previously developed to understand ceramics.* Methodologically this incorporates techniques which have been used to analyse both the use and function of pottery vessels across different archaeological contexts, as well as assess and develop the smaller body of ORA work which has been conducted on FCRs. The main archaeological focus of the thesis is on the Mesolithic of northern Europe, but the methodology has the potential for a much wider

impact, since aceramic cooking technologies (including organic containers and FCRs) have been utilised around the world since perhaps the Middle Palaeolithic period onwards.

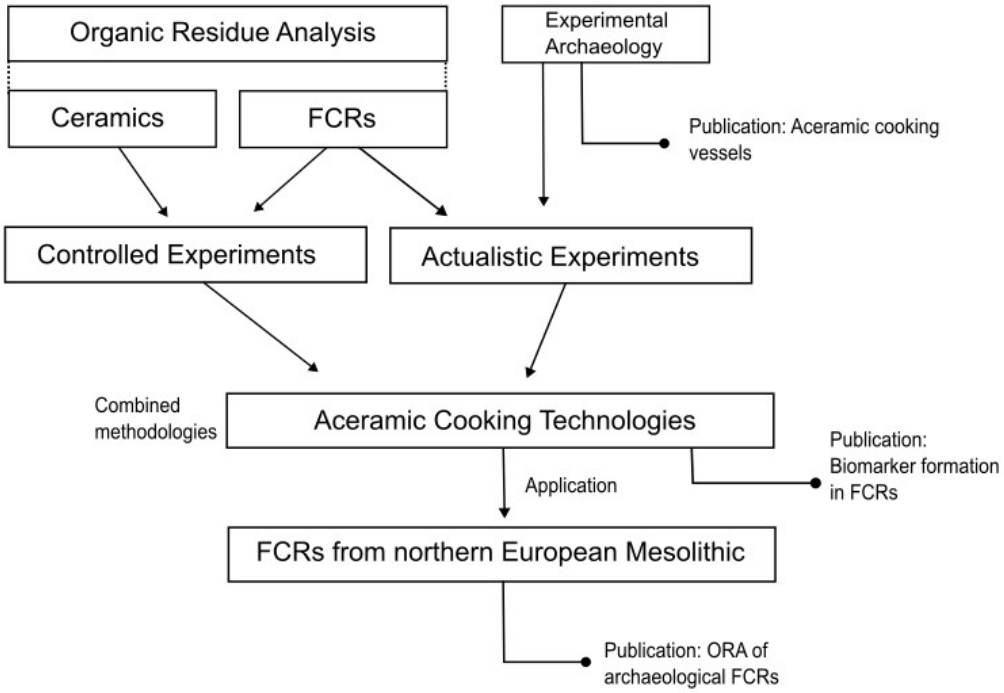

Fig 1.3. A visual depiction of the elements going into the thesis: the application of both experimental archaeology and organic residue analysis to a series of experiments aimed at characterising and evaluating aceramic cooking technologies, with an archaeological application examining the northern European Mesolithic.

2.2. Specific objectives

- Compile evidence for aceramic cooking technologies in the i) northern European Mesolithic and ii) recent forager records
- Examine aceramic cooking technology performance against ceramic analogues
- Investigate if aceramic technology use can be demonstrated by organic residue analysis and specific biomarker formation
- Analyse archaeological materials from northern European Mesolithic sites across the aceramic/ceramic transition using the approaches established above
- Informed by the above, evaluate the potential for future organic residue analysis on stone and explore the archaeological significance of any findings

3. Research Questions

- Is it possible to examine the performance of aceramic cooking vessels through experimental archaeology and comparisons to ceramic analogues?
- Does stone behave in a similar way to ceramics with regards to specific heating biomarker formation? If not, then what are the differences?
- Can the anthropogenic use of archaeological fire-cracked rocks be demonstrated through organic residue analysis?
- Can the results from the above help contribute to the wider questions of how aceramic technologies functioned and the development and adoption of ceramics by certain forager groups?

4. Thesis Structure

Given the wide scope of these research questions I have chosen to structure my thesis in such a way that it will help answer those core issues.

Chapter 2 - Ethnographic review of aceramic cooking technologies: Evidence for aceramic vessel and cooking practices are limited in the archaeological record due to the loss of organic materials through time. Therefore, in order to both contextualise and analyse the topic it is necessary to explore the ethnographic record for evidence of aceramic cooking methods. This was done using Yale University's *Human Relations Area Files* or eHRAF database. Keyword searches for a number of important topics are condensed and analysed, providing a simple but useful classificatory system for how stones were used in cooking and cuisine across a number of available geographical regions. Relevant conclusions regarding aceramic cooking practices drawn from the sample set are presented. The full table of ethnographic data drawn from eHRAF is presented in Appendix 1.

Chapter 3 - Review of Mesolithic aceramic cooking technologies: Archaeological evidence for aceramic cooking technologies in the northern European Mesolithic is often presented through the lens of diet and its components, rather than the methods employed to process and cook them. FCRs are often not retained and analysed during Mesolithic research, therefore few works are available to help characterise this technology. A review of

the available evidence for FCRs, aceramic cooking and processing technologies and any aceramic containers is presented for Britain, Ireland, Scandinavia, the Baltic nations and parts of northern and central Europe. Brief descriptions are given for each country discussing the evidence for cooking technologies. Appendix 2 is a table of additional fire-cracked rock data compiled from this research which both supports the arguments in the chapter and could serve as a library of artifacts for future Mesolithic organic residue work.

Chapter 4 - Experimental archaeology: *An experimental study of wet-cooking in organic vessels: implications for understanding the evolution of cooking technologies.* This paper was published in *Archaeological and Anthropological Sciences* in August 2023.

Techno-functional studies of ceramics have revealed a wealth of information regarding the comparative thermodynamics of different vessels and temper materials. However, we lack similar studies for aceramic vessels, which has led to a number of speculations and assumptions about their capacities and functionality. This chapter/publication presents results from actualistic cooking experiments where water was heated both directly (the vessel placed on the fire) and indirectly (the heat transferred through hot stones) in animal skin and paunch vessels. A calculated temperature curve for a deer skin container is compared to the experimental results, alongside a theoretical ceramic vessel, allowing for a direct comparison of their thermodynamic properties. Conclusions are drawn from these experiments and discussed alongside a cooking model known as '*long-time, low-temperature',* which proposes a re-evaluation of the utility of boiling and the supposed desirability of boiling foodstuffs for long periods of time.

Chapter 5 - Review of ORA: The development of ORA as an analytical method has largely focused on ceramic vessels rather than FCRs, leading to knowledge gaps in how stone reacts with lipids and other macromolecules when heated. To date there has been no collation of all the publications and results of ORA on stone, therefore a review of both ORA in general and specifically within stone is necessary to identify the limits of our current understanding. A table of all the ORA work on stone is presented, along with discussions of the methods and the evolution of FCR-based residue analysis.

Chapter 6 - Experimental biomarker formation: *Experimental evidence for heating biomarkers in archaeological fire-cracked rocks: potential for identifying aceramic cooking practices.* These results will soon be submitted to the journal *Archaeometry*.

In order to expand and develop our knowledge base of how FCRs function it is essential to explore their capacities for thermal biomarker formation, both by themselves and in contrast with ceramics. This publication-chapter builds on the work on Bondetti *et al.* (2021) and their pioneering research into APAA formation in ceramics. Three experiments are presented, two actualistic and one controlled. The results indicate that several thermal biomarkers readily form in stone under multiple heating conditions, although with some differences to ceramics. It also appears that lipids heated with stone result in deviations from previously established quantitative thresholds for lipid origins. These findings have implications for future ORA work on stone, and suggest that more research is needed to characterise how lipids react with hot-stones. Appendices 3 and 4 outline the sampling and organic residue methodologies, while appendices 5 and 6 list the physical descriptions of the FCRs and the biochemical data produced from the organic residue recovery and analysis protocols.

Chapter 7 - Mesolithic FCRs: *Invisible technologies? Using organic residue analysis to reveal prehistoric aceramic cooking practices in Mesolithic and Neolithic Northern Europe.* These results have been compiled for publication to the *Journal of Archaeological Sciences* and are currently with the co-authors.

The methodology established in Chapter 6 for extracting and analysing lipids from heated stone is next applied to northern Mesolithic and Neolithic FCR artefacts from six different sites:

Table 1.1. The FCR artefacts for the six sites analysed in this chapter, along with the details of their recovery contexts, morphology and time period (EBK: Ertebølle culture; TRB: Funnel Beaker culture).

Site	Period	Context	Type	Number
Neustadt (Ger)	late EBK, early TRB	Underwater/refuse	Slab	14
Rosenfelde (Ger)	late EBK	Hearth	Cobble	14
Havnø (Den)	late EBK, early TRB	Midden	Cobble	8
Visborg (Den)	late EBK, early TRB	Midden	Cobble	2
Ormen Lange (Nor)	early Mesolithic	Hearth	Cobble	5
Klakken (Nor)	early Mesolithic	Hearth	Cobble	3

The results assess both the bioarchaeological potential of these FCRs to yield high-quality anthropogenic lipid data, and the archaeological significance of the particular aceramic cooking technologies. The wider comparative context of aceramic and ceramic Mesolithic lifeways is explored, as well as the early transitional stage of agriculture in northern Germany/southern Denmark. Appendix 7 presents the radiocarbon dates for the archaeological sites. Appendices 8 and 9 list the stable carbon isotopes for both contemporary and archaeological references which were used to create figure 7.5. As with Chapter 6, the sampling and ORA methodologies and reported data from the FCRs are presented in appendices 3,4,5 and 6.

Chapter 8 - Conclusions & Discussions: The final chapter draws together all the results and conclusions from the previous chapters. The overall thesis aims and research questions are reassessed to demonstrate the success of the project, as well as any shortcomings and failures. Potential directions for future research are offered, both for the specific archaeological questions (the origin of ceramics, the adoption of ceramics during the northern European Mesolithic), and the broader methodological implications of the ORA results from heated stone.

4.1. Note on methodology

Three of the chapters in this thesis have been written with the intent that they will be published as peer-reviewed journal articles. This has necessarily meant that both contextual and methodological details have been repeated across multiple chapters. In place of one single lengthy introductory section there are instead several distributed sections across different chapters which serve to contextualise the different aspects of the thesis - aceramic cooking, Mesolithic ceramics, organic residue analysis and experimental archaeology.

The sample selection criteria for the ethnographic groups in Chapter Two was initially developed with the explicit aim of creating a direct analogy to the northern European Mesolithic, through the selection of solely northern hemisphere cultures. However, this was revisited towards the end of the thesis and the direct analogy approach was revised, moving instead towards the creation of a series of cooking techniques which could be incorporated into an archaeological framework without the need for environmental comparisons. As a result of the initial methodological approach the chapter lacks any southern hemisphere references. While this oversight does not invalidate the results, nor the use of the results in

later chapters, it does limit their overall robustness, completeness and perhaps future applicability in other archaeological contexts. Should this chapter be reworked for a publication, the inclusion of southern hemisphere cultures in the analysis would be highly desirable.

The experimental design in Chapter Four evolved through several iterations, beginning with an MA experimental archaeology workshop. The final version had passed through two rounds of peer review, once from a preliminary journal application process which was then retracted at the author's request. As a result the paper was refined and ultimately focused on generating a quantitative model for heating water in a red deer hide. The choice of materials was dictated by several criteria: availability of wild and domestic animal products in the United Kingdom; an estimation of reasonable proximity and analogy to ancient food sources and reviewer feedback.

The selection of sites for sampling archaeological fire-cracked rocks (Chapter Seven) was initially intended to have north Germany and the Estonian/Baltic coastline as its main focus, as per the initial project design. However, due to the disruption caused by COVID-19 and the response of both universities and national governments across Europe, it became very difficult to locate and acquire samples from these areas. Therefore the focus of the thesis had to shift to incorporate those sites that I could gain access to, primarily the Norwegian and Danish fire-cracked rock assemblages listed. It has been possible to construct a chronology and set of experimental questions by studying these sites together, however, it could be argued that a much closer range of samples in both time and space would potentially provide a much more focused set of results.

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Chapter Two : Review of Ethnographic Subsistence Technology, Aceramic Containers and Vessels

1.0. Introduction

The use of aceramic cooking technologies, particularly hot-stones, is increasingly limited today. Therefore, in order to explore the diversity of cooking techniques beyond the contemporary, this chapter will focus on ethnographic materials pertaining to northern hemisphere hunter-gatherers and pastoralists. A full appreciation of the material properties and capabilities of many aceramic cooking techniques is beyond most archaeologists, and since the wider topic of cuisine is so fundamentally culturally bounded (Speth and Eugène, 2022), it becomes necessary to explore outside of the modern, western paradigm (Sterelny, 2021; Killin and Pain, 2022). This chapter will first explain the rationale and search methods used with the Human Relations Area Files (eHRAF) database, and then explore the results, drawing out some key commonalities and constructing a basic set of cooking method categories which will be carried forward through the thesis. Although the main archaeological focus of the project is the northern European Mesolithic, this chapter will avoid any direct analogical comparisons between the ethnography and the archaeological record. Instead the aim is to explore the ethnographic record, in order to broaden the scope of knowledge which might underpin any archaeological hypotheses (Brady and Kearney, 2016; Hamon, 2016), and to establish a level of foundational data in order to generate meaningful technological categories, irrespective of time and place.

2.0. eHRAF rationale and search

2.1. Defining aceramic cooking technology search parameters

The electronic Human Relations Area Files (eHRAF) was used to search for examples and descriptions of aceramic cooking technologies amongst northern hemisphere

hunter-gatherers, horticulturalists and pastoralists. This database is a unique collection of ethnographic materials managed by Yale University who have organised their data to be searchable within each paragraph of ethnographic text. Their Outline of Cultural Materials thesaurus covers, among others: personal, social, environmental, botanical, zoological, religious and material categories. This allows users to search within and between cultures and target specific aspects of those cultures using a simple database search. Their archive contains information for over 300 unique cultures, which makes it ideal for larger scale research projects.

In order to explore the ethnographic literature for comparative cooking technologies, it is necessary to define the terminology used and provide definitions for search criteria. Aceramic is the term used to describe methods of heating, storing and preserving foodstuffs or other materials which do not rely on ceramic technology. The earliest use of the term in archaeology is potentially 1940; a description of ceramics from Indiana (Black, 1940). It became popular again during the 1960's excavations of Jericho, Ankara and sites in the Near East where it was used as a synonym for 'pre-pottery' Neolithic (Anati, 1962; Mellaart, 1962). More recently it has been used to describe techniques and methods for distilling birch-bark tar during the Middle and Upper Palaeolithic, as well as the Mesolithic (Groom, Schenck and Pedersen, 3/2015; Kozowyk *et al.*, 2017; Schenck and Groom, 2018). What these have in common is defining retrospectively the class of materials and methods that characterise the period *before* the development of ceramics. This effectively makes everything that is not explicitly ceramic - aceramic. This definition is one of exclusion and is broad in what it could encompass. Cooking is the thermal transformation of foodstuffs, but a wider consideration of all the methods that can be employed to render foodstuffs edible would also include: the consumption of raw foods; biochemical methods such as leaching, fermentation, and curing; and preservation techniques such as freezing, smoking and drying. Using heat to alter foods can also be further characterised through a description of how the heat is applied to the food (directly or indirectly) (Sumer and Oz, 2023) and if the food is enclosed or not (use of a container, vessel or enclosed within earth, sand or other materials) (Nelson, 2010).

Therefore, searching the ethnographic record for evidence and descriptions of aceramic cooking technologies requires decomposing these terms into smaller categories based on methods and techniques of processing and rendering foodstuffs edible. To do this a number of keywords were employed to help search the eHRAF database. These were:

“cook”; “roast”; “boil”; “broil”; “steam”; “fire”; “food”; “pottery”; “ceramic”; “container”; “vessel”; “pot”; “pit”; “oven”; “slab”; “hearth”; “smoke”; “dry” and “ferment”. Alongside this eHRAF provides its own search function, utilising numerical codes for ethnographic subjects such as ‘diet’ and ‘food processing’, which make it straightforward to parse a text. The search terms were not consistently straightforward to apply. Each set of texts held for each culture within the eHRAF database vary greatly, including diaries, memoirs, reports, academic publications, magazine articles, periodicals and books. The quality of each source was difficult to assess, and since this work is focused on the archaeological application of the ethnographic evidence, and furthermore that the author is not a trained archivist or anthropologist, each documented description of a cooking method was simply noted. There was no attempt made to confirm the accuracy of the sources, other than to avoid narrative descriptions such as mythological stories that involved cooking or processing food.

To gather the data, each culture was subject to a keyword search. Where a description existed within a source that noted, explained or observed a cooking practice, this was written down and the citation retained. A full table of results is available in Appendix 1.

2.2. Defining cultural search parameters

By definition archaeology, and in particular ethnoarchaeology, always involves a level of analogy, although how formal these should be and what purpose they should serve has been the subject of intense debate over the years (Gould, 1978; Cunningham, 2003; Kuzmanović, 2009; Lane, 2014; Gosselain, 2016; Sillar and Joffré, 2016). Mesolithic archaeology has not escaped these discussions, and increasingly the use of direct, formal analogies between the hunter-gatherer record and Mesolithic archaeological contexts has been questioned and critiqued (Warren, 2017; Elliott and Warren, 2022, 2023). The cultural search criteria used in this chapter were mostly confined to the northern hemisphere, that is to say, only cultures from the northern hemisphere that were available to query on eHRAF were selected (Fig 2.1). As detailed in Chapter One (section 4.1), the absence of southern hemisphere groups limits the universal applicability of this approach, and future research which includes other continents and peoples may produce additional or differing conclusions.. Temporally the scope of eHRAF spans the earliest available ethnographic literature until the present day - which results in a wide variation of materials available for study for each cultural group, as well as temporal discontinuities. For example, the spread of metal vessels into Alaska during the 19th century impacted upon traditional cooking

techniques and technologies (Frink and Harry, 2008). Likewise, many North American indigenous communities gained or adopted ceramic technologies through time (Quinn and Burton, 2009; Rocek, 2013). The presence of such metal or ceramic vessels were recorded alongside any documented aceramic cooking methods.

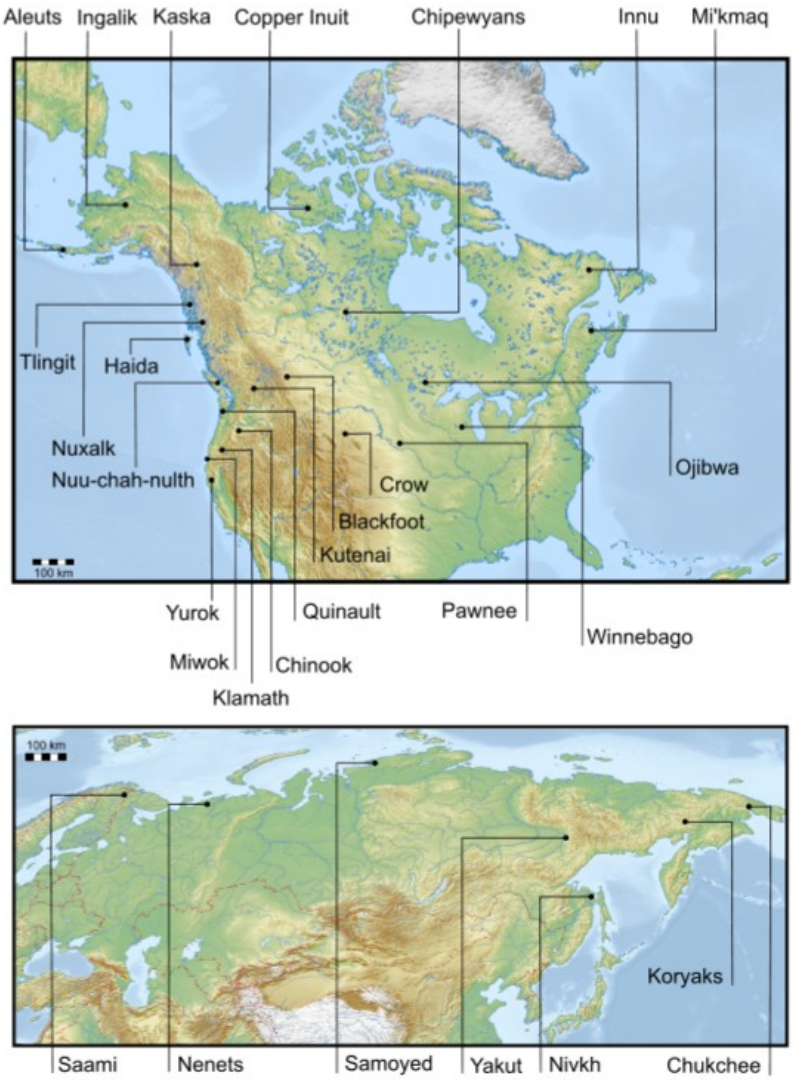

Fig 2.1. Map of the cultures sampled for this chapter. The majority came from North America, but included cultures from northern Europe and northern Asia. Image by author using the geographical coordinates provided on eHRAF to create the map.

Since the aim of this research was to explore the ethnoarchaeological record, rather than create formal analogical hypotheses between any one particular cultural group or region with northern European Mesolithic archaeology, the scope for inclusion was wider than ‘forager’ or ‘hunter-gatherer’. Leaving aside the problematic nature of simple

delineations between hunter-gatherers and other types of lifeways, it is difficult to parse between ‘horticulturalists’, ‘foragers’, ‘fisher-foragers’, ‘pastoralist-foragers’ and many other hybrid economic descriptors. Therefore there was no differentiation made between agriculturalists, horticulturalists, fisher-foragers, hunter-gatherers who used horses, pastoralists and ceramic foragers.

The 29 sampled cultures are presented in Table 2.1. These span across North America, Asia and Europe, with the majority based in the USA and Canada. This is partly a result of historical contingency and partly greater ethnographic documentation.

Table 2.1. A complete listing of ethnographic cultures with their countries. Cultural names are often disputed, therefore these were taken from the eHRAF database and not amended.

Culture	Country
Miwok	USA
Yurok	USA
Klamath	USA
Pawnee	USA
Winnebago	USA
Chinook	USA
Crow	USA
Mi'kmaq	Canada
Quinault	USA
Kutenai	USA / Canada
Blackfoot	USA / Canada
Nuu-chah-nulth	USA / Canada
Innu	Canada
Ojibwa	Canada
Nuxalk	Canada
Nivkh	Russia
Haida	Canada
Aleut	Canada
Tlingit	Canada
Copper Inuit	Canada

Kaska	Canada
Chipewyans	Canada
Ingalik	USA
Saami	Finland/Sweden/Russia
Nenets	Russia
Samoyed	Russia
Yakut	Russia
Koryaks	Russia
Chukchee	Russia

3.0. Results

3.1. Container types

The keyword searches for containers and vessels returned a number of results. These have been broken down by material type and by the cooking method associated with them in Table 2.2. The two cooking methods associated with aceramic vessels or containers were ‘stone boiling’ and ‘direct heat’. Neither term was common in the surveyed literature, but they capture the general approach. Stone boiling refers to the heating of stones or cobbles in a fireplace, whereupon they are placed into a container of water or foodstuffs using tongs or paddles before being removed to be reheated or discarded. Direct heat refers to the placing of the container onto the heat source, and the thermal energy transferred or conducted into the interior. Stone boiling was more commonly associated with plant-based vessels such as baskets or wooden containers, with the exception of bark. Animal-based containers were used for both stone boiling and direct heat.

Table 2.2. The different types of containers found in the ethnographic sample set listed alongside the different ways they were utilised.

Cooking container types			
Container type	**Cooking type**	**Culture**	**Reference**
Baskets	Stone boiling	Miwok	(Barret & Gifford 1933, 138)
		Kutenai	(Brunton 1998, 225)
		Chinook	(Beierle 2004, 4)
		Klamath	(Spier 1930, 162)
		Yurok	(Heizer 1952, 110)
		Haida	(Murdock 1961, 226)
		Tlingit	(De Laguna 1960, 102)
		Kaska	(Honigmann 1954, 43)
Wooden vessel	Stone boiling	Mi'kmaq	(Lockerby 2004, 414)
		Innu	(Lips 1947, 27)
		Ojibwa	(Rogers and Black 1976, 13)
		Kutenai	(Brunton 1998, 225)
		Chinook	(Beierle 2004, 4)
		Klamath	(Colson and Stern 1966, 8)
		Yurok	(Heizer 1952, 110)
		Quinault	(Olson 1936, 46)
		Nuu-chah-nulth	(Arima and Dewhirst 1990, 398)
		Haida	(Blackman 1990, 82)
		Nuxalk	(McIlwraith 1948, 215)
		Tlingit	(De Laguna 1960, 102)
Bark	Stone boiling	Innu	(Lane 1952, 10)
		Quinault	(Olson 1936, 46)
		Kaska	(Honigmann 1954, 43)
		Chipewyans	(Smith 1982, 18)
	Direct heat	Mi'kmaq	(Lockerby 2004, 415)
		Innu	(Lips 1947, 478)
		Ingalik	(Osgood 1970, 146)
Paunch/organs	Stone boiling	Blackfoot	(Wissler 1910, 26)
		Chipewyans	(Smith 1982, 18)
	Direct heat	Kaska	(Honigmann 1954, 43)
		Chipewyans	(Birket-Smith 1930, 32)
		Ojibwa	(Rogers and Black 1976, 17)
		Blackfoot	(Wissler 1910, 26)
Animal hide/skin/rawhide	Stone boiling	Crow	(Lowie 1922, 212)
		Blackfoot	(Wissler 1910, 26)
		Chipewyans	(Smith 1982, 18)
		Kaska	(Honigmann 1954, 43)
	Direct heat	Crow	(Lowie 1922, 212)
Stone vessel	Stone boiling	Mi'kmaq	(Lockerby 2004, 414)

		Copper Inuit	(Jenness 1946, 4)
	Direct heat	Miwok Mi'kmaq Copper Inuit	(Barret & Gifford 1933, 211) (Lockerby 2004, 415) (Jenness 1946, 4)
Ceramic	Direct heat	Winnebago Pawnee Aleut Ingalik Yakut	(Radin 1973, 70) (Smith 1852, 90) (Quimby 1945, 3) (Osgood 1970, 142) (Jochelson 1933, 158)
Metal	Direct heat	Saami Nenets Samoyed Yakut Koryaks	(Itkonen and Minn 1984, 98) (Islavin and Wise 1847, 57) (Popov and Ristinen 1966, 113) (Smith 1898, 76) (Bogoraz-Tan 1904, 56)

3.1.1. Plant-based containers

The details of the plant-based containers are expanded upon in Tables 2.3, 2.4 and 2.5 - drawing out more information about basketry, wooden vessels and bark based containers. In every instance the use of plant-based containers was confined to North America, with no examples from Europe or Asia. In general these containers were rarely placed directly onto the fire, and instead hot stones were utilised to provide a source of indirect heat to the foodstuffs. The types of food cooked in these containers varied, with acorn 'mush' most commonly encountered towards the south of the west coast, and fish/meat more common in the northerly cultures (Fig 2.2). Construction materials for the baskets included grass and spruce roots, and the wooden containers were often made from cedar. These wooden vessels came in different styles such as boxes, troughs and one culture made use of hollowed-out tree stumps. A typical description of the stone boiling process went thus:

"Hot stones were put into this thin gruel until it boiled violently and cooked thoroughly. As the cooking progressed more water or more gruel was added, to attain the desired consistency. When placed in the basket the cooking stones were at almost white heat. They were prevented from burning the basket by constant stirring with a paddle which was also used, among the Northern Miwok, to dip out the stones." (Barrett and Gifford 1933, 13)

Table 2.3. Documented uses of baskets for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of baskets
Chinook	Washington/Oregon	"Wove tight silk grass baskets in which they boiled salmon by dropping in hot stones" (Ruby and Brown, 1976) (Beierle 2004, 4)
Miwok	California	Stone boiling in baskets; parching with hot coals in baskets (Barret & Gifford 1933, 138)
Kutenai	British Columbia	Roots used to make boiling baskets (Brunton 1998, 225)
Klamath	Oregon	Cooking acorn mush with hot stones (Colson and Stern, 1966,356; Spier 1930, 162)
Yurok	California	Cooking acorn mush with hot stones (Waterman, 1920; Heizer 1952, 110)
Tlingit	Pacific NorthWest Coast	Stone boiling in baskets woven of spruce root (Jones, 1915); De Laguna 1960, 102)
Kaska	British Columbia	Spruce root baskets for stone boiling to cook meat (Honigmann, 1954)

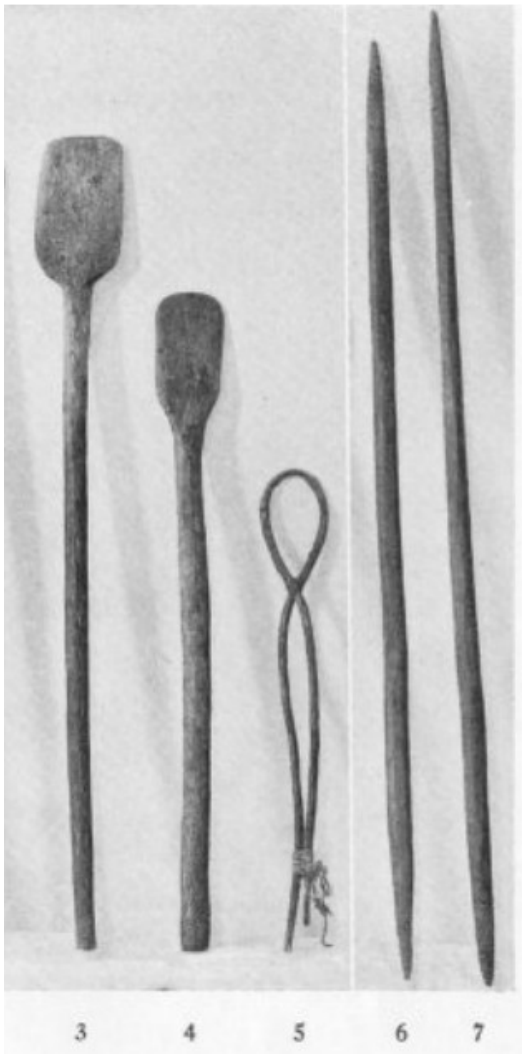

Fig 2.2. Miwok paddles, looped-stirrers and tongs for handling and manipulating hot stones whilst cooking acorn mush in baskets, after Barrett and Gifford 1933, 17

It is interesting to note in the above quotation that the hot stones were likely to cause damage to the vessel, if not stirred or agitated enough while heating. This points not only to a level of skill and familiarity with the process, but also active engagement, rather than letting the food cook passively. The birch-bark vessels were the only examples in the sample set of plant-based containers which were heated directly over the fire (Table 2.5). The methods included shallow pan-like vessels; tripod constructions for birch-bark pots and one mention of a technique in which birch-bark was laid around the edge of a pit and moulded into shape with boiling water, followed up by being sewed together to make a watertight vessel.

Table 2.4. Documented uses of wooden containers for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of wooden containers
Mi'kmaq	Nova Scotia	Stone boiling in hollowed tree stumps (Denys, 1908, 401-402); Lockerby 2004, 414)
Innu	Labrador/Quebec	Stone boiling in wooden troughs called *'ouragana'* (Lips 1947, 27)
Ojibwa	Great Lakes	Stone boiling in wooden troughs (Rogers and Black 1976, 13)
Kutenai	British Columbia	Stone boiling in wooden troughs (Brunton 1998, 225)
Chinook	Washington/Oregon	Stone boiling in wooden boxes (Beierle 2004, 4)
Klamath	Oregon	Stone boiling in wooden troughs/vessels (Colson and Stern 1966, 8)
Yurok	California	Cedar/red wood containers and boxes for cooking (Heizer 1952, 110)
Quinault	Washington	Wooden troughs and bowls used for cooking and rendering fat with hot stones (Olsen 1936, 44-46)
Nuu-chah-nulth	Pacific NorthWest Coast	Stone boiling in wooden boxes (Renker & Gunther 1990, 398; Colson 1953, 178)
Haida	Pacific NorthWest Coast	Stone boiling in cedar wood boxes (Murdock 1961, 226; Blackman 1990, 82)
Nuxalk	Pacific NorthWest Coast	Stone boiling in bentwood boxes (Mcllwraith 1948, 215)
Tlingit	Pacific NorthWest Coast	Stone boiling in bentwood boxes (De Laguna 1960, 102)

Many descriptions within the ethnographic sources included details of extensive material culture and food processing *chaîne opératoires* related to these containers, not limited to: paddles, tongs, loop-stirrers, types of baskets, hot stones, secondary vessels for cleaning the stones, anvils and grinding stones for preparing acorns, post-cooking

containers for storing cooked food or rendered fats and many other examples. The existence of such artefacts and the socially embedded roles of food preparation and cooking are of great importance to archaeologists interested in identifying and characterising aceramic cooking methods in the past.

Table 2.5. Documented uses of bark containers for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of bark containers
Mi'kmaq	Nova Scotia	Birch-bark vessels suspended for direct boiling (Denys, 1908); (Lockerby 2004, 415)
Innu	Labrador/Quebec	Boiling blood directly over the fire in birch-bark container; stone boiling in birch-bark container (Lips 1947, 478)
Kaska	British Columbia	Birch-bark vessels for stone boiling (Honigmann 1954, 43)
Quinault	Washington	Large stitched birch-bark container (Olson 1936, 46)
Chipewyans	Northwest Territories	Stone boiling in birch-bark container (Smith 1982, 18)
Ingalik	Alaska	Birch-bark container suspended over the fire and heated (Osgood 1970, 146)

3.1.2. Animal-based containers

The use of animal based containers in the sample set was more limited, but incorporated some different geographical regions such as the Great Plains. The animal containers were either natural vessels such as organ tissues, or the use of skin/hide to manufacture a waterproof container. Again, heated stones were often employed to transfer heat inside the vessel without the need to conduct it through the animal materials themselves, although several examples of direct heat cooking were identified. In some cases these direct methods exploited the edibility of the animal tissues by filling the organ with foodstuffs, cooking it all over the fire, and consuming everything along with the organ container itself. The size of caribou or bison hides was such that instances exist of pits being dug in the ground, lined with skins, and food cooked inside using hot stones. In contrast to

the plant containers, the animal containers seemed more *expedient* and *ad hoc*, with perhaps less labour utilised to manufacture each container. This may be in part due to the inevitable damage from the use of either stone boiling or direct heat.

Table 2.6. Documented uses of animal containers for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of animal containers
Blackfoot	Great Plains	Cow paunch used for stone boiling; rawhide and organ containers used for direct boiling (Wissler 1910, 26)
Chipewyans	Northwest Territories	Caribou body and stomach used for stone boiling; meat, fat and water added to caribou stomach and heated over the fire (Birket-Smith 1930, 32; Smith 1982, 18)
Kaska	British Columbia	Skin lined pit heated with hot stones; caribou stomachs heated directly over the fire (Honigmann 1954, 43)
Ojibwa	Great Lakes	Stuffed intestines heated over the fire (Rogers and Black 1976, 17)
Crow	Great Plains	Rawhide containers used for direct boiling and stone boiling (Lowie 1922, 212)

3.1.3. Earth ovens

Whilst not strictly a container, the prominence of earth ovens in the sample literature combined with the principle of *enclosing* foodstuffs means they should be discussed alongside the other types of vessels. Nearly half of all the surveyed cultures made use of earth ovens (Table 2.6), and in all instances cultures which used earth ovens also employed another form of container (metal pots, ceramics, stone vessels, plant or animal containers). Whilst the exact method varied from culture to culture, this description of a Quinault earth oven captures a seemingly typical example:

“A pit was then dug in the sand, partly filled with rocks, and a brisk fire built on top. A large supply of fresh fern leaves was gathered and when the rocks were thoroughly hot, the

fire was removed, the stones leveled, and a layer of fern leaves spread over them. On top of this bed the camas was spread (the size of the pit depending upon the amount of the roots). A thick layer of fern leaves was placed on top and covered with a three-inch layer of sand. The roots were allowed to cook overnight and a fire was kept burning on top of the covering sand during the baking. In the morning the roots were removed, mashed, and made into cakes about twice as large as a loaf of bread. These cakes were buried in the reheated pit between layers of fern leaves and baked for a day. They were now thoroughly cooked and would keep through the following winter." (Olson 1936, 54).

Many variations on this were found in the literature, including heaping mussels onto hot rocks and burying them in sand and leaves (Olson 1936); the baking of entire sea lions in rock lined ovens (Swanton 1905) and cooking grasshoppers in earth ovens with an additional fire lit over the top (Barrett and Gifford 1933, 17). One ubiquitous foodstuff that was mentioned in multiple sources was the plant root called 'camas' (*Camassia* spp). There is an extensive literature on the uses of camas within pre and post colonial North America (Turner and Kuhnlein, 1983; Thoms, 1989, 2008a; Wilson and DeLyria, 1999; Tomcek, 2009; Lyons and Ritchie, 2017; Carney *et al.*, 2021), and it is unsurprising to have found camas referred to regularly in the sources. The camas root is described as 'sticky' and 'sweet', almost a 'confection' and its use was widespread throughout the continent (Olson 1936). Archaeologically the use of camas root may date to around 6000 BC (Carney and Connolly, 2024), and the extensive use of earth-ovens to hydrolyse the abundant inulin within the root may have been part of an overall 'carbohydrate revolution' which resulted in significant land-use intensification (Thoms, 1989, 2008a). Another common observation within the source material was the deliberate use of hot stones in the pit to create steam, by using wet vegetation such as seaweed or leaves, or simply by adding water through cracks in the earth.

"The clolum [hard shell clam] is opened by being heaped on stones previously heated, then covered with sea weed and mats. The water contained in the clam runs down on the hot stones, causing steam which … soon cooks the whole pile, containing usually from ten to twenty bushels. From twenty minutes to three-quarters of an hour are generally occupied in performing the operation, and the coverings are then removed. The shells, now being opened, are easily separated, and the meat stuck on skewers … and dried in the smoke" ((Drucker and Ray, 1939, 54).

Of all the foods processed in earth ovens, plants and especially roots were the most frequently encountered.

Table 2.7. Documented uses of earth ovens for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of earth ovens
Winnebago	Nebraska	Hot rock oven used for cooking corn (Radin, 1973)
Chinook	Washington/Oregon	Cooking/steaming camas, edible thistle, lupine, bracken fern, horsetail, and cattail roots (Silverstein, 1990)
Nuu-Chah-Nulth	Pacific NorthWest Coast	Camas, and roots of the sand verbena, surf grass, and buttercup were steamed or baked in a pit oven (Renker and Gunther, 1990)
Haida	Pacific NorthWest Coast	Sea lions cooked in stone lined ovens (Murdock, 1961)
Kutenai	British Columbia	Camas baked in an earth oven with hot stones (Turney-High, 194133)
Miwok	California	Bulbs, greens, and grasshoppers cooked in an earth oven with hot stones, several varieties including with and without a fire above Barrett and Gifford 1933, 17)
Blackfoot	Great Plains	Camas baked in an earth oven with hot stones (Wissler, 1910,24-25)
Quinault	Washington	Camas baked in an earth oven with hot stones; mussels cooked in a pit of hot stones with sand (Olson, 1936, 54)
Tlingit	Pacific NorthWest Coast	Camas baked in an earth oven with hot stones (Jones, 1915, 48)
Nuxalk	Pacific NorthWest Coast	Use of earth ovens (McIlwraith, 1948, 215)
Saami	Fennoscandia/Russia	Use of earth ovens (Collinder, 1949, 82)

3.1.4. Stone vessels

The use of steatite/soapstone or other stone vessels was noted for only three cultures, presumably due to their proximity to a suitable source of raw material. In all three

cases the containers were placed directly in the fire, which may suggest that such direct heating was either more energy efficient or less labour intensive than stone boiling with a stone vessel. Only the Copper Inuit seemed to rely solely on stone vessels for cooking and burning blubber for heat and light (Fig 2.3), the Miwok and Mi'kmaq had access to plant materials such as baskets and birch-bark with which to cook and prepare foods in different ways.

Table 2.8. Documented uses of stone vessels for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of stone vessels
Miwok	California	Steatite vessels, directly in the fire (Barrett & Gifford 1933, 50)
Mi'kmaq	Nova Scotia	Soapstone/sandstone vessels for direct boiling (Lockerby 2004, 414)
Copper Inuit	Arctic	Soapstone vessels for direct boiling (Jenness 1946, 4)

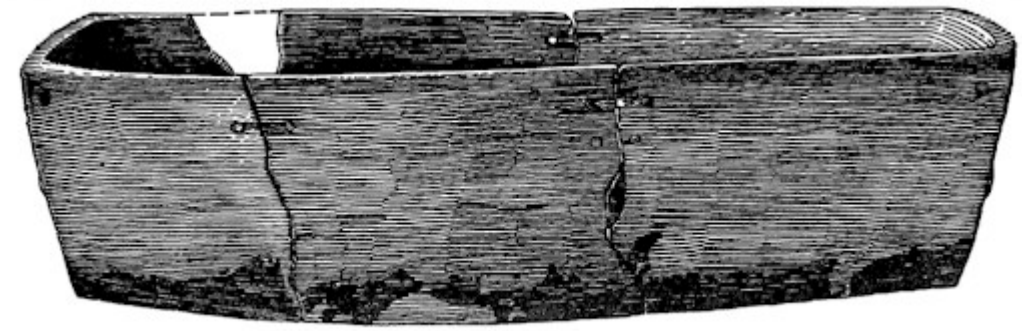

Figure 58. Soapstone pot repaired with copper bands. IV.D.1563. Approx. 1/3.

Fig 2.3. A repaired Copper Inuit soapstone vessel, after Jenness 1922

3.1.5. Heated clay

Several observations in the literature recorded instances where clay was heated as part of a vessel or cooking structure without reaching the level of ceramics. Amongst the

Blackfoot, one source documented an older, discarded method of making a proto-ceramic pot:

"They were fashioned of mud and sand. A bag of rawhide was filled with sand, greased on the outside and the pot shaped over it. The sand was then poured out and the bag withdrawn. The pot was filled with fat and hung over the fire to harden. When finished, it was tested by boiling water in it. Such pots grew gradually harder with use. They were supported by a rawhide cord passing around the rim. The cord had to be changed often." (Wissler, 1910)

A second example came from the Aleuts:

"The traditional method of making soup was to dig a fire pit and place over it a stone, flush with the ground. Then a very thin beach stone was placed on the fire stone and clay walls built on this base. The liquid was cooked in this. A bluish clay called *qudii u* was used for the walls of this vessel which turned white when heated. This kind of fire pit was called *unaalu*. The same vessel was used more than once. One way of preparing the cod soup was with seaweed and seal oil" (Shade, 1949).

Both of these intriguing anecdotes indicate that clay and related mineral soils were sometimes employed in unfamiliar ways, techniques that were not fully ceramic but nevertheless exploited the plastic and thermal properties of the materials.

3.2. Other direct heat methods

Aceramic cooking technologies are wider in scope than just vessels and containers, and other methods exist which utilise direct heat from a fire.

3.2.1. Stone griddles

For a number of the more northerly cultures, including those found in Siberia, the Russian Far East and northern Europe, the use of flat stones placed over the fire to cook food was observed in the literature. These typically involved long, linear stones with a flat surface that could act as a 'frying pan' to sear and cook meat or other foodstuffs with a fire lit underneath. It is noteworthy that not a single example was identified in North America

beyond the Aleutian Islands - however this might simply reflect the sample size and geographic coverage.

Table 2.9. Documented uses of griddle stones for cooking amongst the ethnographic sample set

Culture	Region	Documented uses of stone griddles
Aleuts	Aleutian Islands	Flat stone 'frying pans' used for cooking meat (Cook, 1784)
Saami	Fennoscandia	Flat stones for cooking food (Collinder, 1949)
Nenets	North Siberia	Flat cooking stones (Svoboda *et al.*, 2011)
Yakut	North Siberia	Flat cooking stones (Sieroszewski 1993, 617)
Koryaks	Russian Far East	Flat cooking stones (Kennan 1870, 224)
Chukchee	Russian Far East	Flat cooking stones (Bogoraz-Tan 1904, 193)

3.2.2. Directly on the fire

The majority of the cultures surveyed made use of the fire to cook food directly. This could take the form of deliberate structures to skewer or spit the meat next to, or above the flames (Murdock 1934, 268; Birket-Smith, 1930), or it could be observations of more informal methods:

"To roast the meat they cut it into fillets, split a stick, placed it therein, then stuck up the stick in front of the fire, each person having his own. When it was cooked on one side, and in proportion as it cooked, they ate it" (Mi'kmaq) (Denys 1908, 400).

The foods cooked in this way were almost without exception meat and/or fish. Ribs were mentioned on several occasions as a specific cut of meat which was roasted on the fire (Radin, 1973, 68; Henriksen, 2010).

3.3. Other aceramic methods

It is worth detailing several more specific food processing techniques which appeared frequently in the sampled literature: the rendering and preservation of fats; the use of smoke, air and sunlight to dry foodstuffs for another time, and the use of fermentation to both extend the viability of certain foods and to alter the taste and texture, in so doing making edible foods which previously would not have been. All three could be characterised as techniques of *preservation*, and they incorporate specific methods and technologies.

3.3.1. Fat rendering

Table 2.10 shows the large number of cultures sampled which practiced fat rendering. The majority of them used stone boiling in a container to heat the foodstuffs in order to separate the fat, which could then be skimmed from the surface and stored separately.

Table 2.10. Documentation of fat rendering within the ethnographic sample set

Culture	Region	Fat rendered	Method used
Mi'kmaq	Nova Scotia	Seal	Stone boiling (Wallis 1955, 111)
Innu	Labrador/Quebec	Fish / Meat	Stone boiling (Lane, 1952, 9)
Ojibwa	Great Lakes	Animal marrow	Boiling (Hesketh, 1923)
Crow	Great Plains	Meat / Animal marrow	Stone boiling / direct boiling (Voget 2001, 698)
Blackfoot	Great Plains	Animal marrow	Stone boiling (Schaeffer, 1978)
Kutenai	British Columbia	Fish / Meat	Stone boiling (Chamberlain, 1893)
Chinook	Washington/Oregon	Marine mammals / fish	Stone boiling (Drucker and Ray, 1939)
Klamath	Oregon	Fish	Stone boiling (Colson and Stern, 1966, 11)
Yurok	California	Fish	Stone boiling (Waterman, 1920, 236)

Quinault	Washington	Marine mammals / animal marrow	Stone boiling (Willoughby, 1886, 46)
Nuu-chah-nulth	Pacific NorthWest Coast	Marine mammals / fish	Stone boiling (Renker and Gunther, 1990, 425)
Haida	Pacific NorthWest Coast	Marine mammals / fish	Stone boiling (Murdock, 1961, 225)
Nuxalk	Pacific NorthWest Coast	Marine mammals / fish	Stone boiling (Kennedy and Bouchard, 1990, 325)
Tlingit	Pacific NorthWest Coast	Marine mammals / fish	Stone boiling (Knapp and Dorr, 1896; Jones, 1915)
Aleut	Aleutian Islands	Marine mammals / fish	Pounding, stone vessels, ceramics (Hrdlička, 1944, 54)
Copper Inuit	Arctic	Marine mammals / fish	Pounding, stone vessels (Jenness, 1922)
Kaska	British Columbia	Marine mammals / fish	Stone boiling (Honigmann, 1954, 45)
Ingalik	Alaska	Fish	Pounding, stone vessels (Osgood, 1959, 46)
Samoyed	North Siberia	Fish / meat	Metal vessels (Popov and Ristinen, 1966, 110)

Several cultures around the Arctic circle pounded marine mammal fat in order to break it down from a solid to a liquid state, as well as using ceramic or stone vessels where available. The most common foods were marine mammals and fish, followed by terrestrial ruminant animals such as moose. On the Pacific Northwest coastline, some cultures rendered extremely large amounts of fat from the eulachon fish *(Thaleichthys pacificus)*, known as ‘ooligan oil’. Ooligan oil formed a culturally significant source of energy which can be readily stored and traded long distances (Patton *et al.*, 2019). Groups that produced ooligan oil were connected across the Pacific North-West through the so-called ‘grease trails’ which acted as trading routes between coastal and inland cultures (Brooks, 2002; Phinney, Wortman and Bibus, 2009). Ooligan oil was rendered at scale by different coastal communities, usually through the fermentation of the fish in large wooden boxes or canoes (Magdanz, 1988). The fermentation process, which lasted several days, produces a more nutritionally dense oil than unprocessed fish oil, particularly in the conversion of omega-3

fatty acids, which are ten times higher in ooligan oil than raw eulachon fish (Kuhnlein *et al.*, 1996). The containers are reported to have held up to 6300 kg of fish flesh (Kuhnlein, 1982). After a period of fermentation heated stones were added to the boxes or canoes in order to produce more oil and break down the fish (Suttles and Sturtevant, 1990). The final product, once the oil had been skimmed and pressed through fine basket weaves, was a thick golden grease (Jacobs, 1975). Aside from using it as a food, as discussed above, it was also distributed and consumed in large amounts at feasts. The Tlingit and Nuxalk were documented as drinking and ladling vast amounts at hosted potlatches for neighbouring guests and scarcity of the oil was considered a mark of poverty in the tribe (Kirk, 1986; Stewart, 2008).

Pemmican is a mixture of ground dried meat, rendered animal fat and dried fruits and berries. The exact composition differs according to what resources are available and the meat can be moose, elk, deer or bison, among others, similarly with the fat source; the berries are often chokeberries, blueberries, cherries or currents (Merriam, 1955; Colpitts, 2014). The mixture can survive for over 100 years and has often been used in long expeditions and marches as an emergency food, since it is stable and calorically dense (Quigg, 1997). Its use was considered so important in trade and military expeditions that conflicts have been fought to preserve the animals and ingredients needed for its preparation (Shore, 1994). The production of pemmican maximised the longevity of the three ingredients: fat, meat and fruit. The fat acts as a preservative for the meat and fruit, the fruit is a source of carbohydrate based energy, the fat a source of triglyceride-based energy and the meat a source of protein. The fruit and meat also make the fat more palatable. Combining them also made a stable mixture which could be easily stored in pouches or tins and could travel well (Scheiber, 2005; Colpitts, 2014). Traditionally it was produced on the Plains, where large amounts of buffalo were hunted and processed (Scheiber, 2005). The ability to render grease from animal bones represents an intensification in the production of food which may be necessary both for increased population requirements and for an increase in trade demands (Quigg, 1997; Oetelaar and Beaudoin, 2016; Bethke *et al.*, 2018).

3.3.2. Smoking and drying

Using smoke and warm air to dehydrate and preserve foodstuffs was a technique routinely mentioned and described in the ethnographic literature. Many cultures sampled smoked and dried fish (Olson 1936, 46; Knapp and Dorr 1896), sometimes using special smokehouses to preserve very large amounts of fish (Colson, 1953). Other examples included drying berries (Blackman, 1990, 82), meat (Birket-Smith, 1930, 31), cheese (Collinder, 1949) and wild rice (Vennum, 1988).

3.3.3. Fermentation

The final aceramic food preparation method encountered in the literature was fermentation. Fermented foods were often described at length, in part because the techniques and the final product were extremely unpalatable to the outside observer, a fact of cultural food diversity which has been discussed in the archaeological literature (Speth, 2019; Speth and Eugène, 2022). Alongside the unpleasant aromas, many descriptions focused on the texture and the habits of consuming the maggots and larvae which grew in the fermenting foods:

"In the fall of the year they casually cache their caribou without removing the stomach. The semi-digested vegetable contents ferment and taint all the flesh, but the Copper Eskimo relishes both the smell and the flavour, though his more sophisticated brother in the west pronounces them disgusting. I have seen a man take a bone from rotten caribou-meat cached more than a year before, crack it and eat the marrow with evident relish, although it swarmed with maggots… Dried fish that have become covered with mould are considered hardly inferior to freshly-dried. The grubs of the warble fly, which bore through the skins of the caribou in the spring, are picked out end eaten, either raw or boiled." (Jenness 1922)

Other examples included burying walruses (Bogoraz-Tan, 1904, 197), fermenting fish eggs in boxes (Willoughby, 1886), pickling fish in bark-lined pits (Sieroszewski, 1993, 551) and fermenting leaves and grasses to make a condiment (Bogoraz-Tan, 1904, 197).

3.4. Raw foods

Whilst not a preparation method, it is nonetheless worth mentioning that the consumption of raw animal foods was noted throughout the analysis of the ethnographic materials. Such examples include fish and blubber (Quimby, 1945), rabbit (Honigmann, 1954), marrow (Smith, 1982), meat and blood (Yakovleva, 1976), and sea mammals (Smith, 1898).

4.0. Using the ethnographic data

4.1. Geographical trends

Whilst the aim of this chapter was to explore the ethnographic literature, in order to better understand and broaden the scope of research into the material properties that underpin aceramic cooking methods - there were nevertheless some interesting trends. One in particular, visualised in Figure. 2.4, is the division between those cultures which utilised stone griddles, and those which utilised stone boiling. Not only were there no overlaps between the two groups, but there was an obvious geographical distinction as well. Aside from the Aleuts, all the instances of stone griddle use were found in Eurasia. As well as this observation was the prominence of griddle stones in cultures found in northern, circumpolar latitudes. Quite why this would be is beyond the scope and aims of this chapter. Nelson's (2010) work on quantifying environmental thresholds for particular cooking methods highlights a band between 58° and 41° latitude, above which stone boiling disappears as a technique. Nelson also mentions the apparent anomalies of the Aleuts and the Ingalik, the former of which would be expected to use stone boiling and the latter would not. This may simply be an anomalous result with no deeper significance, but the continuity and discontinuity of culinary technologies with reference to population and *population change* is an important theme throughout this thesis, in particular for Chapter 7 – therefore this apparent difference between stone griddling and boiling is worth considering.

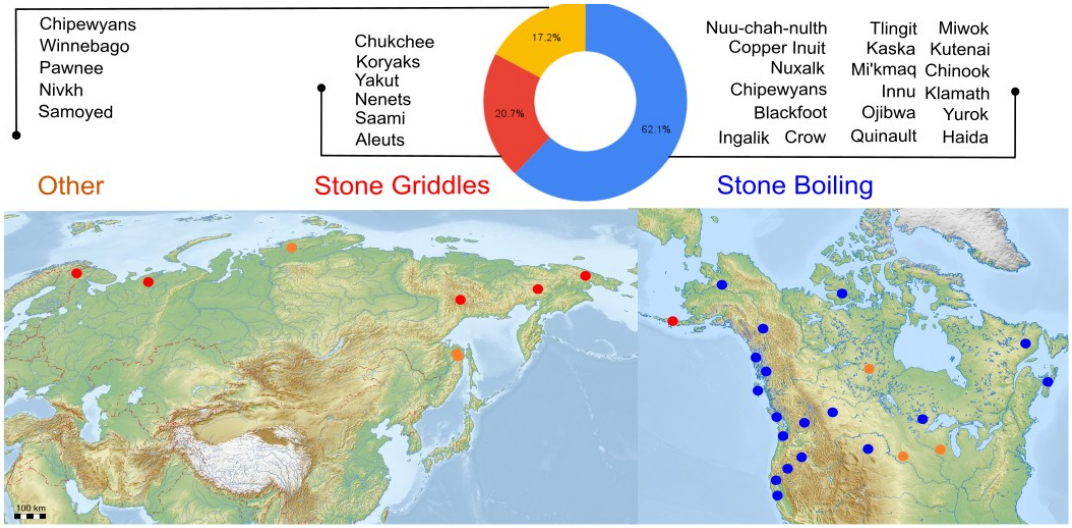

Fig 2.4. A visualisation of the ethnographic data for the use of stone griddles, stone boiling and other aceramic cooking methods collected for this chapter. Image by author.

4.2. Classifying aceramic cooking methods

Despite a sample size of only 29 cultures, the results demonstrate the existence of a wide, varied and complex set of aceramic cooking methods and technologies present in North America, the Russian Far East, northern Siberia and Fennoscandia within the Modern period. Even without any formal or direct analogies to the Mesolithic period, it is nonetheless possible to make use of the *general material properties* of these cooking methods in order to delineate some useful categories.

Table 2.11 shows the proposed categories which cover the main aceramic cooking techniques with relation to *heat*, containers and the thermal modification of food which may leave some archaeological markers (Fig 2.5). The use of more exotic heated clay structures and the specific processing techniques such as smoking and fermentation have been left out, since this thesis is mostly concerned with cooking, containers and the use of stone.

Table 2.11. The categories of aceramic cooking technologies, based on the ethnographic evidence presented.

Method	Category (heat type, enclosed)	Material	Description
Boiling stones	Indirect; container	Stone	The transfer of heated stones from a fire to a container. The stones will rapidly cool and need to be reheated. The container is not placed over or into the fire.
Directly heated containers	Direct; container	Animal products, plant products, stone vessels	The use of a vessel or container placed into or over a heat source in order to conduct the heat through the walls and into the foodstuff.
Direct heat without containers	Direct; no container, sometimes enclosed	Fire, wood, ash, coals	The suspending, placing or partial burying of food directly into a heat source in order to cook it. Can range from wooden skewers to covering in ash.
Earth ovens / cooking-pits	Indirect/direct; enclosed	Stones, earth, clay, plants	The creation of a pit-hearth, often lined with hot stones and wet plant material. Food is placed into the structure and buried for an amount of time. Sometimes a fire may be built above it or water poured into it to generate steam.
Griddling stones	Direct; no container	Stone	The heating of flat sections of stone and food being placed on one side. The stone is hot enough to cook the food.

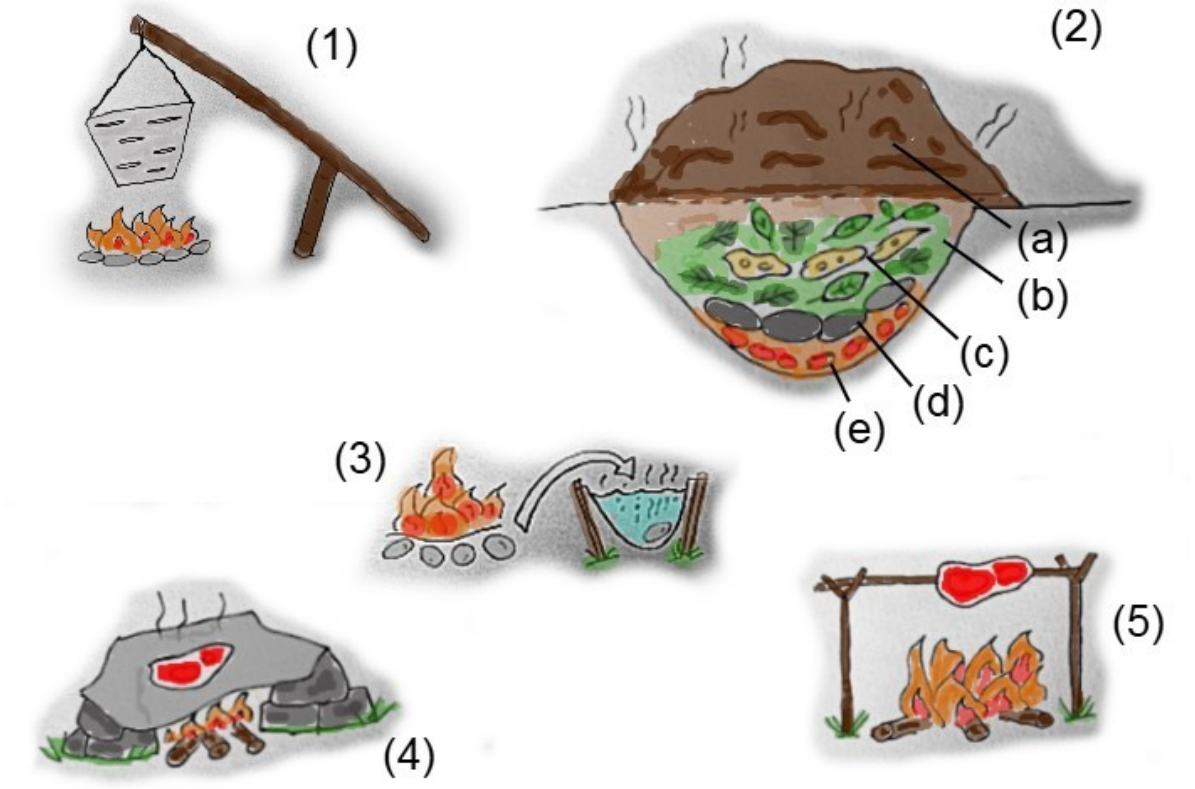

Fig 2.5. An illustration of each cooking method outlined in Table 2.11: (1) directly heated containers; (2) an earth oven with the following elements: (a) mounded earth/soil, (b) an envelope of plant materials surrounding the foodstuffs, (c) root vegetables or other plants foods, (d) a layer of heated stones, (e) a layer of hot ash and embers; (3) boiling stones; (4) griddling stones; (5) directly heated food without a container. Image by the author.

Each of these categories has also been studied archaeologically, in some cases extensively (Brink and Dawe, 2003; Thoms, 2008b; Gao *et al.*, 2014; Shantry, 2020; Speth, 2015; Admiraal and Knecht, 2018; Groß *et al.*, 2019; Wandsnider, 1997; Thoms, 2008b; Black and Thorns, 2014; Short, 2018; Thoms *et al.*, 2018; Wright, 2004; Jeanotte *et al.*, 2012; Donner *et al.*, 2019). Thus they are applicable to other archaeological contexts, being both supported by the archaeological, ethnoarchaeological and ethnographic literature. The categories are not exhaustive, and doubtless other cooking techniques and technologies existed which are not captured by this schema.

One aspect which has not been fully characterised in this investigation is the differences in *time* within and between these different methods in regards to the specific aims of the cooking technique. For example, *blanching* and *simmering* are two time-dependent methods which make use of boiling or near-boiling water (Adams, 1981; Sallau *et al.*, 2012; Reis, 2017), but would both be categorised under boiling stones or directly heated containers. Even without considering the physical method of cooking, time and temperature constitute two different dimensions of the cooking process, something explored experimentally in Chapter Four. The specific material properties of each component listed above need to be investigated in order to understand the nuanced effects of temperature, time, pH and other factors on the behaviour and outcome of each aceramic method.

4.3. Non-cooking uses of hot stones

An important caveat to be considered when categorising aceramic cooking methods from the available ethnographic evidence is the fact that heated stones were routinely utilised for non-cooking purposes. Table 2.12 shows a compilation of non-cooking uses for hot stones taken from the literature that was returned for each eHRAF cultural search.

Table 2.12. Non-cookery related functions of heated stones within the ethnographic literature for the sample set.

Culture	Region	Non-cooking uses for hot stones
Tlingit	Pacific Northwest	Sweat lodge for health, ceremony; sweat lodge as a courthouse; shamanistic performances (Oberg 1934, 154)
Crow	Great Plains	Sweat lodge for health, ceremony (Prando 1894, 483)
Ingalik	Alaska	To heat the interior of the home (Lantis 1938, 127
Ojibwa	Great Lakes	Manufacturing magical charms (Coleman 1937, 54), handling to display magical powers (Ray 1945, 100)

The use of heated stones to create steam in an enclosed space has long been known about and discussed in relation to Native American cultural practices of healing and

medicine (Egghart and Beach, 2006; Mehta 2007), and the 'sweat lodge' is one of many uses for heated stones. As per the table, these include the multiple functions of steam for health, ceremony and ritual, and even legal proceedings. Another practical example was the use of stones as indirect sources of heat inside the home. Finally there are observations of hot stones being used as part of ritual paraphernalia and for displaying supernatural powers by handling them without injury. Such diverse uses of stones in a non-cooking capacity should be considered when interpreting heated stone artefacts on archaeological sites. It must also be acknowledged that the production of fire-cracked rocks may be entirely incidental, as a result of placing stones around a hearth for delineation or by any other accidental means. Chapter Three will explore in more detail how archaeologists can attempt to identify the function of fire-cracked rocks, if indeed they had any at all.

5.0. Conclusions

Although imperfect, the ethnographic record offers archaeologists the opportunity to explore methods and techniques with which modern researchers are wholly unfamiliar. One outcome of this exercise in sampling the literature describing the 29 cultures listed above is a much wider appreciation of the complexity and sophistication which underpinned human subsistence without ceramics. As evidenced, different foodstuffs were prepared and cooked in numerous ways, making use of containers, earth ovens, fireplaces, heated stones, ash, sand, clay, plants materials and animal products to convert ingredients into final products. Parsing these different methods produces some straightforward categories which will be carried forward through the thesis to help differentiate use and method with regard to aceramic cooking in the past. Although cultures change, the material properties of stone, bark and skin do not.

It is clear from analysing the ethnographic literature that subsistence strategies tend to be diverse and mixed, even in challenging circumpolar environments. Containers of one form or another were always utilised, whether made from animal, plant or stone materials. The difficulties inherent to each method were managed differently, within a range of physical thresholds - for example, stones heated in the fire often shattered when placed directly in water and were typically dirty from the fireplace, cleaning or washing them *before* adding them to the container would have an impact on heat transfer and potentially longevity. It was unclear however that these aceramic methods were necessarily cruder or less effective than ceramics. The use of large wooden boxes and sometimes canoes by cultures of the Pacific Northwest such as the Haida, Tlingit and Nuxalk to render large quantities of fish oil would

seem more practical than attempts to use ceramic vessels might have been, simply in terms of scale. Such considerations are important for the wider archaeological question of 'why ceramics?' (Chapter 1).

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Chapter Three: Evidence for aceramic cooking and food-processing material culture in northern Mesolithic Europe

1.0. Introduction

The previous chapter outlined a set of categories to describe and define the general methods of aceramic cooking, a set aligned with both the ethnographic and archaeological literature (Chapter 2). In this chapter these categories will be applied to the archaeological literature of the northern European Mesolithic. More specifically, the evidence for aceramic cooking techniques and technologies across Ireland, Britain, Denmark, Sweden, Norway, the Baltic nations, the Netherlands, northern Germany and to a more limited degree, central Europe, will be presented and discussed. A table of documented Mesolithic-era FCRs from these countries is presented in Appendix 2. Historically the Mesolithic period has suffered from the same perceptions as affect hunter-gatherers more broadly, namely that their diets were expedient, poor and devoid of the richness which accompanies food *production* rather than *gathering*, as well as later concepts of higher *cuisine* (Lidén *et al.*, 2004; Warren, 2015). Early interpretations of Mesolithic cuisine were heavily influenced by ethnographic writings about shore-dwelling forager peoples in North America, Tasmania and Tierra del Fuego, importing a discourse of 'poverty' and 'savagery' (Morgan 1877; Lubbock 1865; Clark 1952; Wheeler 1954; Warren, 2022). In particular the Mesolithic was considered directly analogous to those contemporary cultures through the association of shell or kitchen middens, which began to be excavated in northern Europe during the 19th century (Gutiérrez-Zugasti *et al.*, 2011). The shift towards more bioarchaeological approaches to seasonality (Mellars, Wilkinson and Fieller, 1980; Rowley-Conwy, 1993; Ellis *et al.*, 2003; Dark, 2004), the identification of plant foods (Clarke, 1978; Zvelebil, 1994; Kubiak-Martens, 1996) and analysis of dietary isotopes (Tauber, 1981, 1985; Noe-Nygaard, 1988; Lubell *et al.*, 1994) helped move the focus away from the intensive exploitation of shellfish or terrestrial animals. More recently the confluence of organic residue analysis and the recognition of an 'indigenous' hunter-gatherer pottery tradition during the Late Mesolithic has yielded high-quality data relating to consumption patterns leading up to and beyond the transition to Neolithic agricultural economies (Boudin, Van Strydonck and Crombé, 2009; Philippsen and

Meadows, 2014; Papakosta, Oras and Isaksson, 2019; Robson *et al.*, 2021; Lucquin *et al.*, 2023).

Despite these advances there has been relatively little focus on the material and social elements of *cuisine*, that is to say - the processing and transformation of gathered or acquired foodstuffs into distinct cultural products. In her work outlining the development of thought and study in Britain towards Mesolithic food, diet and cooking, Milner (2009b) highlights how different methods of boiling, roasting, steaming and stewing foods are under-discussed, and that “hearths are often found on archaeological sites, but rarely is the relationship between hearths and cooking explored”. Recent publications do show an increasing interest in Mesolithic food processing and cuisine. The discovery of mustard seed phytoliths in western Baltic Mesolithic pottery demonstrates the use of non-essential foodstuffs to create flavour, a key component in cuisine (Saul *et al.*, 2013; Holst *et al.*, 2024)). Furthermore the identification of non-cereal grinding stones at the late Ertebølle/early Funnel Beaker site of Neustadt revealed a hitherto unknown aspect of non-thermal food processing material culture at the site (Holst *et al.*, 2024). But by and large the focus remains on consumption patterns and foodstuffs rather than on the methods and technologies related to cooking.

In Chapter 2 the categories for aceramic cooking methods were broken down into: boiling stones; direct heat (with a container); direct heat (without a container); earth ovens and griddling stones. Alongside these were descriptions of various processing and preservation techniques such as smoking/drying, fermentation and the rendering of fats. Finally there were also many observations of the consumption of raw foods. These methods and techniques will not be evenly identifiable within the archaeological record, in part due to the ephemeral nature of some and the high likelihood that objects made of organic materials such as animal skin or tree bark will degrade over time. One class of material artefact which does survive in the record however is stone, typically referred to as fire-cracked rock (FCR) after thermal modification and alteration. Identifying the *function* of FCRs is not straightforward and relies on use-wear analysis of fracture patterns, changes in porosity, colour and other geomorphological markers in order to interpret the life-history of the artefact (Lovick, 1983; Jackson, 1998; Rapp, Balescu and Lamothe, 1999; Graesch *et al.*, 2014; Custer, 2017; Neubauer, 2018, 2024). Attributing specific cooking functions to FCRs requires diagnostic trace evidence of that activity, for example - interpreting FCRs as boiling stones might depend on the presence of thermal ‘shock fractures’, or the rupturing of weaker

veins within the stone as the heated cobble is introduced to much colder water (Rapp, Balescu and Lamothe, 1999; Little, 2014; Gao *et al.*, 2014). Another issue raised in the previous chapter with regards to the interpretation of FCRs is the potential to *misinterpret* the function since heated stones may have been used for other non-cooking functions. This has been discussed before in the context of Bronze Age 'burnt mounds' - the phenomenon of large pits filled with heated, cracked rocks found across western Europe (Néill, 2009; Jeffery, 1991), known variously as *fulachtaí fia* in Ireland (Ó Néill, 2003; Hawkes, 2014), *four de terre (polynésien)* in French speaking countries (Ramseyer, 1991) and *skärvstenshögar* in Sweden (Larsson, 1990). It became apparent that distinguishing between burnt rock used for steaming food or generating steam for a sauna was very difficult (Barfield and Hodder, 1987; Drisceoil, 1988; Hawkes, 2015).

With these caveats in mind, this chapter will present the available evidence for aceramic cooking methods during the northern European Mesolithic, following the categories and descriptions previously mentioned. Each method will be briefly summarised and any archaeological evidence presented and explained.

2.0. Evidence for the use of boiling stones

As mentioned, the identification of boiling stones from the more generic FCRs often relies on careful interpretation of the stones and their surrounding context. The process of stone boiling requires (a) a fireplace to heat the stones, (b) selected stones usually of a particular geological type and size (Shantry, 2020), (c) a container to hold the foodstuffs/water, this could be an organic vessel, a stone or ceramic container or a pit-structure. The repeated heating, quenching, cooling, drying and reheating cycle will eventually shatter the stone, leaving behind a distinctive assemblage of angular, cracked rocks (Petraglia, 2002). The agitation of removing and replacing stones causes fractures to weaker stones, as well as concentrating fire-spalls at the base of the hearth and the retention of sharp colour changes across the stone as the rapid cooling prevents complete transformation (Thomas, 2010, 358).

Archaeologically the northern European Mesolithic offers several possible examples where boiling stones may have been used. In Ireland it is questionable whether any early

fulachtaí fia can be assigned to the Mesolithic period (Hawkes 2014), but the Late Mesolithic site of Clonava 1 in Co. Westmeath may present stronger evidence in the form of unworked sandstone/quartz FCRs. These artefacts displayed angular fractures, irregular cracks and fissures and charcoal staining, with some pieces re-fitting - all consistent with the quenching of heated stone (Fig 3.1) (Little 2014, 43). Despite the absence of clear ‘cooking-pits’, these heated stones may well have been added to organic containers such as baskets or animal hides. The Norwegian Mesolithic possesses a poor record of hearths, but several sites do show small patches of charcoal and collections of FCRs, sometimes unrelated to any pyrotechnological feature. These include: Kotedalen (Early Holocene), which has a shallow pit containing a large number of ‘scorched rocks’ (Damm 2022); concentrations of burnt cobbles on the surface at hunting camps around the Myrvatn and Fløyrlivatn lakes (Bang-Andersen, 2012); Unit G of Nyhamna 48 (Early Holocene) which possesses patches containing small to medium-sized stones, some of them fire-cracked (Bjerck, 2017; Breivik, 2020) and several more Mid-Holocene coastal sites described as possessing “well-built stone structures associated with pithouses, stone-filled cooking pits with or without charcoal, to small surface concentrations of fire-cracked stones without any charcoal” (Mansrud and Eymundsson, 2016). Many of these show one or more typical features of boiling stones, including the selection by size, irregular fractures and deposition away from hearth features. However, a recent review of Norwegian Mesolithic consumption and subsistence practices notes that the overall evidence for sustained pyrolithic technologies is relatively limited, and that the diet for Holocene foragers in Norway may have included more raw and fermented foods than previously believed (Damm, 2022).

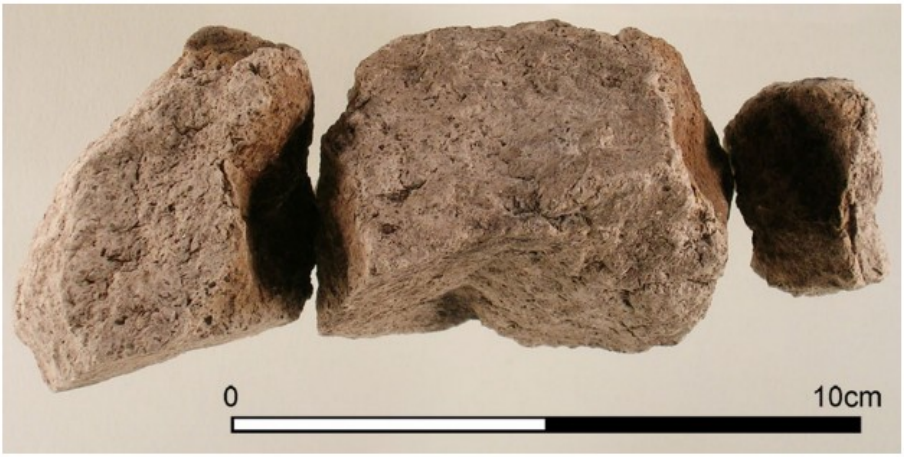

Fig 3.1. Refitted sandstone artefact from Clonava 1. Interpreted as a boiling stone due to macro-thermal changes such as angular fracturing patterns. Image after Little 2014

The Mesolithic pit-hearth phenomenon of the Netherlands and Belgium will be discussed in greater detail below, suffice to say that several pits have returned FCRs made of sandstone which have been interpreted as possible boiling stones (Beuker, 1989; Muller *et al.*, 2015). An analysis of a British Mesolithic hearth at Goldcliff Trench J identified "a cluster and diffuse scatter of heat-fractured quartzite, most likely heated to high temperatures and then rapidly cooled in water" (Bell, 2007; Mithen, 2019). However, the strongest evidence for the use of boiling stones comes from Bohemia, in Czechia. Not just scatters of fire-cracked rocks, but well-preserved pit and hearth features have been found in association with large amounts of stone, as well as some specialised and unusual hearth designs. Early Mesolithic sites at Kostelnírokle II, Smolný kámen and Dvě věže in northern Bohemia revealed a wealth of evidence for stone use in cookery - including hearth features with basalt pebbles and shallow depressions containing burnt sandstone blocks (Svoboda 2015). The rocky canyon sites of Okruhlik and Dolsky Mlyn present probably the clearest examples of a boiling stone system for the Mesolithic period, consisting of several very large hearths complete with sandstone and basalt cobbles, then multiple deep, narrow 'boiling pits' dotted around the hearths, and even clusters or piles of burnt and fractured basalt stones (Svoboda *et al.*, 2001). This combination of a hearth, FCRs *in situ* within the hearth, multiple exterior pits and discarded fractured FCRs shows almost every element of a pyrolithic boiling system, except for the type of container used to hold the foodstuffs within the cooking pits.

Chapter 2 showed that the complex of material culture supporting the use of boiling stones also included organic objects: paddles, tongs, loop-stirrers and organic containers into which hot stones were placed, such as wooden boxes, baskets and canoes. The evidence for organic containers will be presented below, but there are a number of wooden objects such as canoe paddles (Gabrielsen, 1953; Hartz and Lübke, 2000; Skriver, Borup and Astrup, 2017) and 'digging sticks' (Taylor *et al.*, 2018) which could be potentially interpreted as boiling stone paraphernalia. Indeed, the paddle from Tybrind Vig shows signs of heat damage along the shaft (Andersen, 2011), although more damage to the paddle face might be expected if handling heated stones. Beyond these there are many unidentifiable or ambiguous wooden artefacts found on Mesolithic sites where the preservation is ideal, such as Zamamostje 2 (Lozovskaya and Lozovski, 2016), some which could be items for manipulating or handling hot stones. Antler could also have been used to move hot stones or remove them from a container, and whilst speculative, such interpretations should be considered when analysing Mesolithic material artefacts.

3.0. Evidence for the use of direct heat, with a container

The evidence for the use of organic containers during the northern European Mesolithic is extremely limited. Since this type of artefact is so fragile, only a small number of examples exist, many from sites and regions not covered in this project. Nevertheless, listing them will be worthwhile. Firstly, examples of basketry and netting: plant fibre structures have been recovered from Tybrind Vig, Denmark (Jørgensen, 2013); impressed baked clay from Coves de Santa Maira, Spain, has revealed the existence of cordage or basketry, perhaps used as a container mould (Tortosa *et al.*, 2019), and several complete baskets were found in Cueva de los Murciélagos, Spain, the preservation of which is unparalleled for the European Mesolithic (Martínez-Sevilla *et al.*, 2023). Secondly, wooden objects which could be cups, bowls or troughs, two were discovered at Star Carr in Britain (Fletcher *et al.*, 2018), and one at Friesack IV in Germany (Gramsch and Kloss, 1990). Finally examples exist of containers made from birch bark, many of which have been discovered in Russia. These sites include: Vis 1; Szczepanki; Veretye I; Zamostje II as well as Friesack IV (Oshibkina, 1989; Gramsch, 1992; Burov, 1998; Gumiński, 2012; Fletcher *et al.*, 2018). The container at Veretye I contained lithics, and has been interpreted as a cache or storage device (Oshibkina, 2008). There is no indication from any of these suspected vessels that they were used to directly cook food. Despite this, birch bark should still be considered a likely material for organic and expedient cooking vessels, albeit one that is especially unlikely to be preserved if subject both to thermal and taphonomic degradation. As Chapter 2 demonstrated, the range of organic artefacts used for cooking included animal organs, hides and even canoes (although indirectly through the use of hot stones). Many examples of canoes or dugout vessels exist, particularly for the Ertebølle period (Christensen, 1990; Grøn and Skaarup, 1991; Andersen, 2011). At Møllegabet II the submerged canoe was found to have been badly burnt (Grøn and Skaarup, 1991, 47), although no definitive explanation could be offered for why. As seen in Chapter 2 and elsewhere in the ethnographic literature, particularly of the Pacific Northwest, canoes were regularly used as cooking vessels to help render large amounts of fish oil (Byram and Lewis, 2001). Future experimental work on the damage to wood through cooking with hot stones could help with similar interpretations. One final form of evidence for cooking with containers is the existence of tripod-style post holes over hearths, indicating that an object was being suspended over the fireplace. Examples of these include the Scottish Early Mesolithic site of East Barns (Engl *et al.*, 2021) and the Late Mesolithic site of Lisnasoo in Co.Antrim, Ireland (Nicol *et al.*, 2015).

4.0. Evidence for the use of direct heat, without a container

Directly heated foods are those which are placed, suspended or buried on top of, near to or inside a heat source such as a fireplace, burning ash/ember/charcoal or heated materials like stones or sand. These are in contrast to indirectly cooking methods where the heat is transferred from the fire through a medium such as heated stone or clay, away from the direct source of heat – which is the hearth – this would include the technique of adding hot stones to a vessel (Chapter 2). Identifying these activities within the archaeological record is possible where foodstuffs have been left *in situ* within a hearth (Troyer, 2014), or they have been discarded and show diagnostic markers of having been heated (Roberts *et al.*, 2002; Simões and Aldeias, 2022). There are several common foodstuffs that have been identified as being directly cooked within the northern European Mesolithic archaeological literature: hazelnuts, shellfish, plants and terrestrial animals. Each of these will be considered in turn.

4.1. Evidence for directly cooking hazelnuts

Hazel (*Corylus avellana*) kernels have become an archetypal Mesolithic food, and their shells are ubiquitous across northern European Holocene archaeological sites (Zvelebil, 1994; Kubiak-Martens, 1999; McComb and Simpson, 1999; Sørensen and Casati, 2009; Regnell, 2012; Bishop, Church and Rowley-Conwy, 2013; Ptáková, Šída and Kovačiková, 2021; Crombé *et al.*, 2023). The intensive exploitation has prompted questions about hazel woodland management (Caseldine and Hatton, 1993; Bishop, Church and Rowley-Conwy, 2015) and the development of a ‘hazelnut economy’ (Holst, 2010; Groß *et al.*, 2021). Cooking hazelnuts without burning them requires careful roasting procedures, preventing the flames from scorching and charring the exterior portion of the shell (Score and Mithen, 1988; López-Dóriga, 2015; Bishop, 2019). A number of methods have been proposed, in line with the archaeological evidence. The site of Staosnaig F24 (Scotland) revealed 30-40,000 intact hazelnuts, along with lesser celandine tubers, crab apples, seeds, charcoal and lithics (Bishop, Church and Rowley-Conwy, 2013). Exactly what this pit represented is debatable, with one possibility being the accidental charring of a prepared batch of nuts (Mithen, 2000). Larger sites such as Warren Fields (UK) (Murray, Murray and

Fraser, 2009) and Howick (UK) (Bayliss in Waddington, 2007) have revealed large pits, some with thousands of charred hazelnuts, along with FCRs, indicating that pit roasting was utilised for processing batches of nuts.

Palaeo-lake Duvensee in Schleswig-Holstein, northern Germany, possesses some of the best preserved early Holocene Mesolithic sites anywhere in Europe. Over twenty stone age sites have been identified, and 17 excavated in the last century (Groß *et al.*, 2019). Amongst the many organic finds have been several sites with evidence for high levels of hazelnut processing, conducted in a systematic and organised fashion. These include: WP1; WP5; WP6; WP8; WP11 and WP13 (Bokelmann, 1971, 1975, 1980, 1991; Lage, 2004, 2011; Sørensen, Lübke and Groß, 2018). The typical roasting facility uses sand, brought from elsewhere, heated in a shallow pit and used to cover hazelnuts to roast them without charring the exterior. The most thorough analysis of these roasting pits was conducted by Holst (2010), looking at WP6 & WP8. She highlights the construction and production of the roasting pits, along with pine planking at WP8, as well as the large pieces of sandstone used to grind the roasted nuts into a flour or paste afterwards. Based on the density of shells found and environmental data concerning the growth and management of hazel trees, Holst estimates that WP6 could have yielded between 966,600–19,33,200 kcal. Another possible cooking method based on the evidence from Duvensee is the use of a 'clay plate' to directly roast hazelnuts which have been covered in sand (Lage, 2011).

In Ireland a number of sites have revealed both charred and uncooked batches of hazelnuts, including at both Mount Sandel (Woodman, 1985; Mitchell and Mitchell, 1986; van Wijngaarden-Bakker, 1990) and Lough Boora (Ryan, 1980; McComb and Simpson, 1999). The unprocessed nuts could represent a form of caching or underground storage, alternatively given the small, circular dimensions of the pit, these were intended for roasting, perhaps employing an oxygen-free environment with a fire lit over the top of a shallow pit.

4.2. Evidence for directly cooking shellfish

As previously mentioned, early discursive writing about the Mesolithic focused on the abundance of shell or kitchen middens across northern Europe. These midden structures are typically dominated by oyster (*Ostrea sp.*), cockle (*Cerastoderma edulis*), mussel (*Mytilus edulis*), and periwinkle (*Littorina littorea*) shells (Andersen, 2000; Milner and

Woodman, 2007; Warren, 2015) in varying layers, which can also incorporate bones, flint, sand, pebbles, charcoal and FCRs (Andersen, 2000). Consuming these shellfish would have entailed cooking them at some point - despite the fact that shellfish can be consumed raw - and the best methods for cooking bivalves, crustaceans and marine molluscs is often steaming or short bursts of direct heat (Meehan, 1982; Milner, 2009b). The presence of fully intact shells (Hood and Melsæther, 2016), or tools to prise open cooked molluscs (Olsen, 1984), indicate that such cooking practices were commonplace. As Conneller notes about a hearth feature at Culverwell (UK), “Large quantities of burnt stones were common, indicative of a strong focus on food preparation and cooking. Many of the periwinkles and topshells were intact indicating use of boiling water to extract them” (Mannino and Thomas, 2001; Conneller, 2021, 329). However, identifying the precise cooking methods archaeologically is often impossible, since the act of retrieving the shellfish from the fireplace or ash mound destroys the stratigraphic integrity of the hearth feature (Aldeias *et al.*, 2016, 2019).

A review of the formation and use of Danish kitchen middens relates that three types of cooking structure are often discovered during excavations - a grey lens of burnt shell material; a stone-built structured hearth with FCRs and much larger pits “with successive layers of charcoal, burned shell, and clay” (Andersen, 2000). The latter have been interpreted as cooking pits (Klinge, 1931; Meehan, 1982; Andersen, 2000). One interpretation of these pits and hearths is as potential shellfish cooking structures, with some excavated beneath middens containing huge quantities of FCRs (Andersen, 1989; Milner, 2002). Milner also further describes these as possible evidence for large-scale feasting, perhaps for roasting or steaming large amounts of shellfish in rock-lined hearths or pits. The tradition of ‘stone-set hearths’ is discussed by Andersen (2018) as typified by finds from Vængesø III, where shallow hearth-pits were filled with FCRs, alongside deeper pits containing charcoal, some FCRs and many periwinkle shells (Fig 3.2). Many small hearths were found around the Cnoc Coig midden in Oronsay (Scotland), some containing burnt shells, which suggests that small amounts were being expediently cooked in batches (Mellars and Andrews, 1987; Pirie, Mellars and Mithen, 2015). Other Scottish midden sites have turned up the presence of crabs (*Brachyura* sp), razor clams (*Pharidae* sp), dog whelks (*Nucella lapillus)*, scallops (*Pectinidae* sp*)* and sea urchins (*Echinoidea* sp) (Mellars and Andrews, 1987; Russell, Bonsall and Sutherland, 1995; Richards and Mellars, 1998; Milner, 2002; Milner, 2009). Similarly, terrestrial land snails were known to have been consumed during the early Iberian Mesolithic (Lloveras *et al.*, 2011), but like all these mollusk species, their cooking time was likely so short that little evidence of the method has survived (Milner,

2009a). Recent bioarchaeological analysis of experimentally heated shellfish has demonstrated that mineralogical thermo-alterations occur within the shell structure, even when no macroscale evidence for cooking exists (Simões and Aldeias, 2022), but this technique has yet to be deployed at scale.

Fig 3.2. Stone-set hearths from Vængesø III 'Ishuset'. Image after Andersen 2018

4.3. Evidence for directly cooking plants

The evidence for cooking plant materials in earth ovens is more plentiful during the northern European Mesolithic than for direct cooking. However, there are examples of plant material remains in association with hearth features that raise the possibility of plants being cooked more directly. The deposition of many charred lesser celandine roots (*Ranunculus*

ficaria) and crab apple pips/endocarp (*Malus sylvestris*) at Staosnaig (Scotland), indicate that some plant foods may have been cooked on an open heat source (Mithen *et al.*, 2001; Mithen, 2019). Nearly 30 different species of edible plants were recovered from Tybrind Vig, including sea beets (*Beta vulgaris* ssp. *maritima*), acorns (*Quercus* sp), floating sweet grass (*Glyceria fluitans*) and soft fruits such as raspberries (*Rubus idaeus*) and rowan berries (*Sorbus aucuparia*) (Kubiak-Martens, 1999). Many of these were charred, which could reflect accidental burning or waste deposits into a hearth, equally it could indicate the use of rapid cooking techniques such as scorching, parching or covering plant foods with hot ash for short periods of time. Burnt and charred water-caltrop (*Trapa natans*) remains were uncovered around the edges of domestic hearths at Sarnate, Latvia (Vankina, 1970; Zvelebil, 1994). As with some of the other categories, it is not always straightforward to separate 'pit-cooking' from direct cooking on a hearth, therefore the remainder of the plant cooking evidence will be dealt with in the pits/earth oven section below.

4.4. Evidence for directly cooking terrestrial animals and fish

As evidenced in Chapter 2, the cooking of meat directly on or next to an open fire was a commonplace technique in the ethnographic record. This included joints or pieces of meat or fish being skewered over a flame, whole animals/fish being placed into hot coals and ashes or animal products such as blood and fat being stuffed into organs and cooked over a heat source. Most of these activities would leave little archaeological evidence, with the exception of bones.

Evidence for butchery in Ireland is limited, with the exception of breaking long bones and skulls to access marrow and the brain at Moynagh Lough (McCormick, 2004). One problem is that many recovered bones seem to have been discarded into hearths, such as at Mount Sandel, Kilnatierney, Lough Boora and Moynagh Lough, preventing more contextual evidence from being identified (Woodman, 1985; Warren, 2015). At the Kilnatierny midden, a small depression contained the remains of a simple hearth including burnt pig and fish bones, arguably cooked on hot stones or using a skewer (Murray *et al.*, 2011). In southern Scandinavian the presence of domestic 'lenses' have been noted, many of which are formed from shallow or deep pits containing a sandy-charcoal mixture, along with burnt stones, bones, flint waste, bark and soil (Grøn, 2003). Some examples from Sweden include: Hylteberga nr 9; Ageröd; Tobisborg 1, 2 & 3; Hagestad 6:2A 1, 2 3 & 4; Hagestad 44: 8A;

Bredasten and Tågerup 1, 2 & W (Larsson, 1973; Grøn and Skaarup, 1995; Grøn, 2003) (Fig 3.3). The sand is often interpreted as an external addition, intentionally included in the hearths, possibly as part of a heating and cooking set-up. However, it is not clear whether these are cooking features, or deposits of household waste. By contrast, in central and northern Sweden, Mesolithic settlements often contain sunken pits filled with fire-cracked rocks and bones, including: Dumpokjauratj (Bergman, 2008), Grafjell (Fretheim, 2003) and Sjovreten (Welinder, 1977). As described by Fretheim (2009) - these are generally around 50cm deep and no more than 2.5m in diameter, containing a bed of charcoal, then fire-cracked rocks and sometimes heated bones. These roasting pits are typically an outdoor feature, rather than an interior or domestic one, and have been argued to be a marker of regional identity, helping to form group bonds through collective cooking practices (Fretheim, 2009; Bergman, 2008). A similar example might be found in the so-called ‘funeral feast pits’ of Donkalnis and Spiginąs cemeteries in Lithuania, where pits containing animal and fish bones formed part of a mortuary context (Butrimas, 2016). Twelve fireplaces in northern Bohemia contained relatively large amounts of burnt amphibian bones, suggesting they were cooked directly over the fire, (Ptáková, Šída and Kovačiková, 2021) as also evidenced at Konejlova jeskyně (Czechia) where larger quantities of ‘meat-bearing’ frog bones were found in Mesolithic occupation layers (Kovačíková, Novák and Prostředník, 2012). Smaller scale finds, such as the mention of distinct patches of fish bones around hearths at Bjørnsholm, Norsminde and Ertebølle (Denmark) (Johansen, 2006), hint at simple cooking practices such as individuals skewering one or two fish for themselves around the fire. A possible instance of roasting an entire animal was found near Wawcott XXIII (UK), where the bones of a single pig were recovered from a pit infill containing burnt stones, charcoal and flint (Carter, 1975).

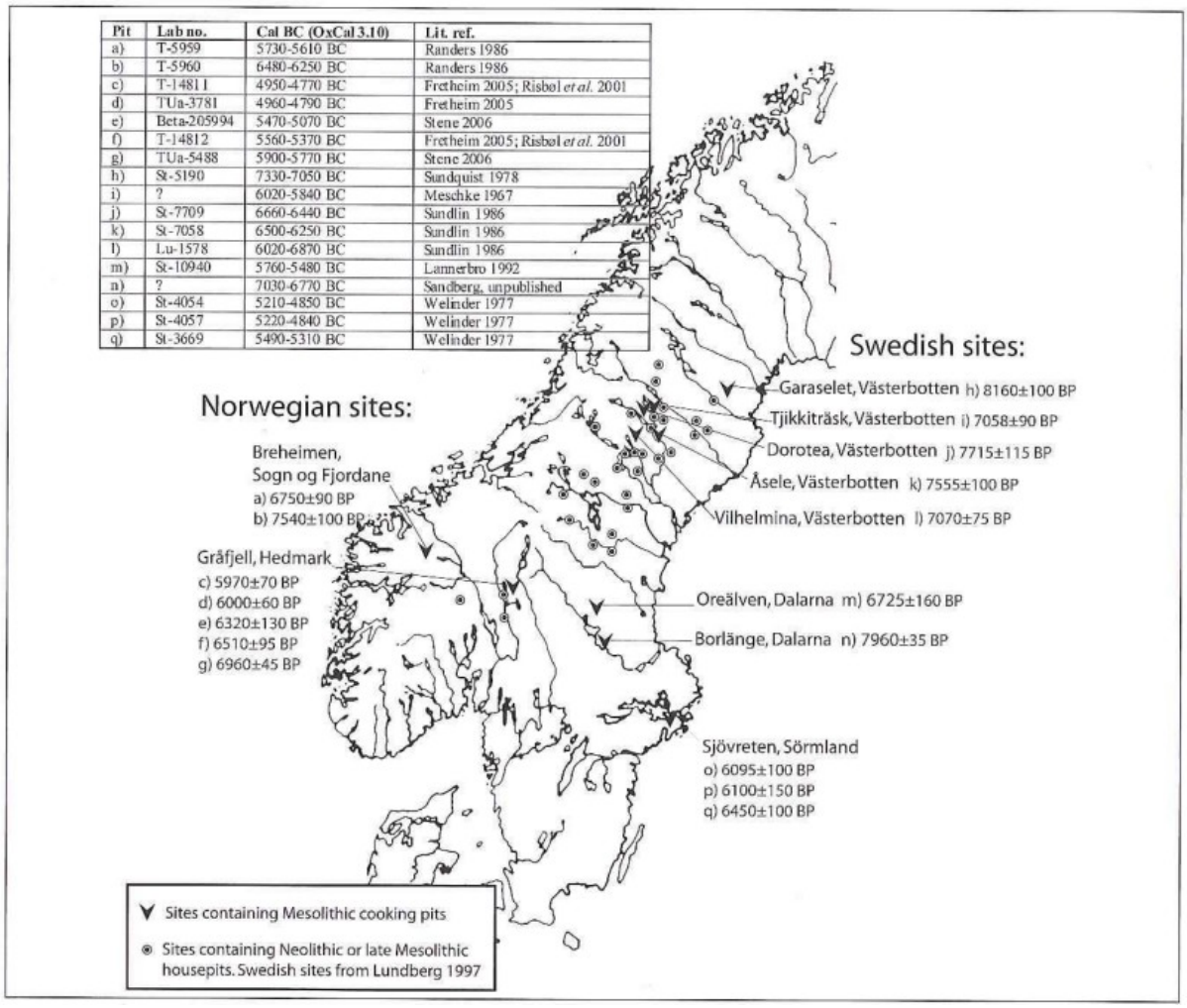

Fig 3.3. The distribution of Mesolithic cooking pits and Neolithic/Mesolithic housepit sites in Sweden and Norway. Image after Fretheim 2009

As Milner (2009) notes, the interpretative link between butchery, the burning of bone and how the food was *cooked* is not often explored. Consequently where burnt bone is documented, whether in association with a cookery feature or not, it is difficult to infer what thermal techniques were utilised, and whether they were as a result of intentional cooking or waste disposal. At the Russian site of Juhola 2, large amounts of burnt fish bones were uncovered from several refuse pits, indicating that they were likely cooked but using an unknown method (Seitsonen *et al.*, 2017). Cultural preferences for animal bone curation and disposal related to animistic rituals can also influence their final appearance and location (Jordan, 2001; Overton and Hamilakis, 2013; Seitsonen *et al.*, 2017), as well as the secondary use of bone for fuel (Vaneeckhout, Salmi and Junno, 2013; Lejay *et al.*, 2016).

5.0. Evidence for the use of earth ovens

Earth or pit ovens have been well documented in both the archaeological and ethnographic record (Wandsnider, 1997; Wilson and DeLyria, 1999; Black and Thorns, 2014; Thoms *et al.*, 2015), and their specific features have been identified in several northern European Mesolithic sites. Typically earth ovens are utilised for roasting/steaming plant foods, in particular rhizomes and roots to hydrolyse the starch and inulin content (Thoms, 1989; Carney *et al.*, 2021). At the site of Hallskov (Denmark) during the Ertebølle period, there is potential evidence for pit roasting different rhizomes, bulbs and tubers. Charred parenchymatous tissue of wild garlic (*Allium ursinum*) and pignut (*Conopodium majus*) were identified in association with four pits, containing ash, sand, charcoal, broken clay fragments, twigs and burnt stones (Kubiak-Martens, 2002). At Vaenget Nord (Denmark) many smaller cooking pits were found clustered with charcoal patches, post holes and refuse pits (Price and Petersen, 1987), indicating that aceramic cooking techniques could be scaled down for domestic locales. Other rhizomes and tubers have been suggested as common early-to-mid Holocene carbohydrate sources, based on modern environmental conditions and archaeological finds, including species of *Sagittaria* (Kubiak-Martens, 1996), ramsons (*Allium ursinum L*.) and common club-rush (*Schoenoplectus lacustris*) (Bishop, 2021). A potential tradition of earth oven cooking has been identified in the northern Pennines (UK). At Kingsdale Head two cooking pits matching the general description of an earth oven (charcoal, layer of FCRs) were uncovered, the earliest layer dating to 7025–6645 cal BC and a second recut dating to 6220 and 6070 BC (Melton, Russ and Johnson, 2014; Conneller, 2021, 288). At South Haw another earth oven was found at TP1, consisting of a pit feature with an arc of post holes surrounding one edge, perhaps indicating a windbreak structure (Chatterton in Conneller and Warren, 2006). This oven contained a single burnt bone, which might indicate that this cooking pit was used for processing animals rather than plants. Other possible examples of cooking animals in earth ovens have been found in Estonia, at the coastal sealing camps of Võhma, Ruhnu and Kõpu, where large cooking pits with FCRs at the base may have been used to process seals (Sikk, 2017) (Fig 3.4). It has been suggested that the use of earth ovens to cook meat is associated with northerly conditions, where carbohydrate sources are less readily available (Wandsnider, 1997).

Fig 3.4. A possible earth oven feature from Kopu IA. Image after Sikk 2016

A common feature of Mesolithic excavations in the Netherlands and Belgium are the presence of pit-hearths, often in closely-associated clusters. Thousands of such hearths have been discovered. In the Veenkoloniën region alone, site S51 turned up 40 hearths, site NP3 turned up 38 hearths and site S6 turned up 28 (Groenendijk, 2015). There has been extensive debate in the literature as to whether these features are anthropogenic in origin (Hamburg *et al.*, 2001; Groenendijk, 2015; Crombé, 2016; Huisman *et al.*, 2019; Woltinge, 2019; Crombé and Langohr, 2020; Huisman *et al.*, 2020), with some researchers arguing they are natural features or burnt ant-hills (Crombé, Langohr and Louwagie, 2015), but enough evidence has been presented to satisfy most that these hearths are man-made (Huisman *et al.*, 2020). The hearths themselves roughly conform to a standard shape and size, typically U-shaped and circular, usually full of an organic-rich heated material (charcoal, heated soils, plant matter, humus) (Huisman *et al.*, 2019). Sometimes the hearths can present with heated flint, wood tar, hazelnut shells, animal bones and cracked stones (Peeters and Niekus, 2017). The pits date from between 9,200 - 5,00 BC, and the tradition ends as the first Dutch Swifterbant pottery appears, during the period of Neolithisation (Niekus, 2006, 2022; Peeters and Niekus, 2017). Functional assessments of the pits have failed to point to one specific activity, with tar production, smoking foods or hides, cooking meat or hazelnuts, charcoal production and social functions all listed as possible uses.

Certainly close control of the temperature and visible flames and smoke has been interpreted from the addition of topsoil during the burning process (Huisman *et al.*, 2019). Interestingly several stone mace-heads were discovered in one pit (Drenth and Niekus, 2007), as well as pieces of fire-cracked sandstone, which have been suggested to be remnants of boiling stones (Beuker, 1989; Muller *et al.*, 2015).

Given the difficulty interpreting an earth oven from a more generic pit-hearth or waste infills containing bones and FCRs, it is perhaps not surprising that they have not featured more prominently in discussions of Mesolithic cooking. The North American archaeological literature has made more progress, linking recent ethnographic descriptions to archaeological contexts, and have delineated more detailed classifications of oven variability based on their capacity for steaming as well as roasting (Black and Thorns, 2014; Thoms *et al.*, 2018). At present it is difficult to assess from site descriptions and secondary literature exactly how Mesolithic earth ovens may have functioned and how they may have comparatively varied over time and place. The Miwok people, for example, used different words to describe an earth oven with a fire lit over the top and one without a fire over the top (Barrett and Gifford 1933). Conneller notes that one of the potential earth ovens at Kingsdale Head appeared to have a secondary fire lit over the earth packing of the pit (Conneller, 2021, 288). Clearly the Mesolithic period likely saw a similar richness of cooking techniques and methods, which are waiting to be discovered.

6.0. Evidence for the use of griddle stones

Identifying griddle stones from archaeological contexts currently has no clearly defined set of markers. Work on Aleutian griddle stones shows that they are often thin, cracked, and coated with an oily residue and burnt encrusted food (Admiraal *et al.*, 2019). The morphology of these stones was likely dictated by their natural source, rather than any deliberate manufacturing (Jeanotte *et al.*, 2012), and griddle stones from other parts of the world cannot therefore be expected to conform to this particular style. The term ‘griddle’ rarely appears in the archaeological literature of northern Mesolithic Europe, but the term ‘slab’ does appear more often. Slabs are not necessarily griddle stones however, and could be part of a structured hearth, oven or used for another unknown function. Such hearths have been identified, including at Rubha Port an t-Seilich (UK), where stone slabs were placed around the fireplace (Mithen *et al.*, 2015), and another at Kinloch (UK) where broken

and burnt stone slabs were found inside a fireplace (Wickham-Jones, 1990). Stone slabs are rare in Norway, with one example at Kvernbergmyra (Damm, 2022). Estonia may offer the clearest cases of griddle use. At Narva-Joaorg, “a limestone slab had been placed on smaller limestone pieces and fire had been made under it. About a 5 cm thick layer of coal was preserved under the stone slab” (Jaanits, 1960, 5; Sikk, 2017). Another was also identified at Siimussaare, but made of granite instead (Moora 1964; Sikk 2017). Limestone is an unusual choice, since it readily reacts with water upon heating (Vutukuri, 1974; Piazza, 1998). It may be that such slabs were used to heat the interior of domestic dwellings rather than for cooking, but the specific alkaline properties of heated limestone were likely utilised deliberately in cooking practices elsewhere (Ellwood *et al.*, 2013; Sikk, 2017). A final unusual feature was found in southern Bohemia, around the Schwarzenberg Lake. This was a unique hearth structure which appears to have been made from slabs of baked clay, layered with charcoal (Pokorny *et al.*, 2010). It is possible that this represents a form of griddling with hardened clay rather than stone, as was suggested for hazelnut roasting at Duvensee (Lage, 2011). The use of clay griddles has been documented archaeologically and ethnographically in the Americas, including in Alabama (Homsey and Sherwood, 2010), Puerto Rico (Jiménez 2006) and Nicaragua (Donner *et al.*, 2019).

7.0. Evidence for the use of preservation techniques

Preservation techniques such as smoking and drying, fermentation and the rendering of fats featured prominently in the ethnographic review of Chapter 2. However, only a few examples exist for the northern European Mesolithic period. The rendering and burning of marine oils from blubber and fish during the Mesolithic has been confirmed through organic residue analysis of ‘blubber lamps’ and bowls from the late Mesolithic Ertebølle and Narva cultures (Heron *et al.*, 2013; Robson *et al.*, 2022, 2018). These illumination devices were made from ceramics, but it seems likely that marine fuels were being used prior to the arrival of pottery technologies. One Early Holocene site on the eastern Swedish archipelago turned up “small black lumps of burnt organic matter containing marine fatty acids [...] as well as more than one kilogram of burnt seal bone” (Pettersson *et al.*, 2014; Damm, 2022,13). Some Norwegian Mesolithic hearths, such as Unit G of Ormen Lange, have been speculated to have burnt marine oils or blubber, given the oily residues covering the FCRs and soil below (Bjerck, 2017; Breivik, 2020). Given the later Norwegian Iron Age use of ‘slab-lined pits’ to render whale and marine mammal fats (Heron *et al.*, 2010; Nilsen, 2016), it is conceivable

that a similar aceramic pyrolithic heating system could have been used during the Early to Mid Holocene prior to the arrival of ceramics. Plants have been considerably underappreciated as a source of fats, but dogwood kernels (*Cornus sanguinea*) might have been used for rendering or pressing a kind of oil at Bökeberg (Sweden) (Regnell *et al.*, 1995; Regnell, 2012). This could have been achieved by simmering the fruit in water and skimming the fat from the surface, thus necessitating an aceramic container capable of withstanding those temperatures.

Evidence for the use of fermentation to preserve foodstuffs during the Mesolithic is scarce. The deposition of animals into bodies of water as a form of 'pre-digestion' has been suggested for the Palaeolithic (Speth, 2017), and there are several examples of terrestrial animals being butchered and placed in water for the Mesolithic - at Star Carr (UK) (Milner, Conneller and Taylor, 2018) and Rosenfelde (Germany) (Haartz et al. 2014), although in both instances alternative explanations are offered. The only definitive example of fermentation was found at the Early Mesolithic site Norje Sunnansund (Sweden) (Boethius, 2018; Boethius *et al.*, 2021). This unique find of a clay-lined gutter revealed a pit of fermented fish (cyprinids, mostly roach (*Rutilus rutilus*)) which would have required great experience at managing acidity levels over at least one or two years. Such 'delayed-return' food storage systems have been argued to be interlinked with the early production of pottery, granting semi-sedentary foragers more control over the fermentation process (Craig, 2021).

Finally, the evidence for smoking and drying foods for preservation and storage is equally sparse for this period. As discussed above, explanations for the function of the Dutch and Belgian pit-hearths have included their use as smokers, in particular where turf has been intentionally added back into the pit (Peeters and Niekus, 2017; Huisman *et al.*, 2019). At Criet Dubh on the Isle of Mull (UK), a possible 'smoke-house' structure was discovered between outcrops of rock (Mithen, Wicks and Anne Pirie, 2018). The authors also debated as to whether the structure could equally represent a 'sweat lodge'. A more compelling example was found at Strandvägen, Motala (Sweden). Here a full range of fish processing stages were on full view: hearths, cooking pits, storage pits, drying racks and even a possible limestone slab-lined fish smoker or oven (Fig 3.5) (Molin, Hagberg and Westermark, 2018).

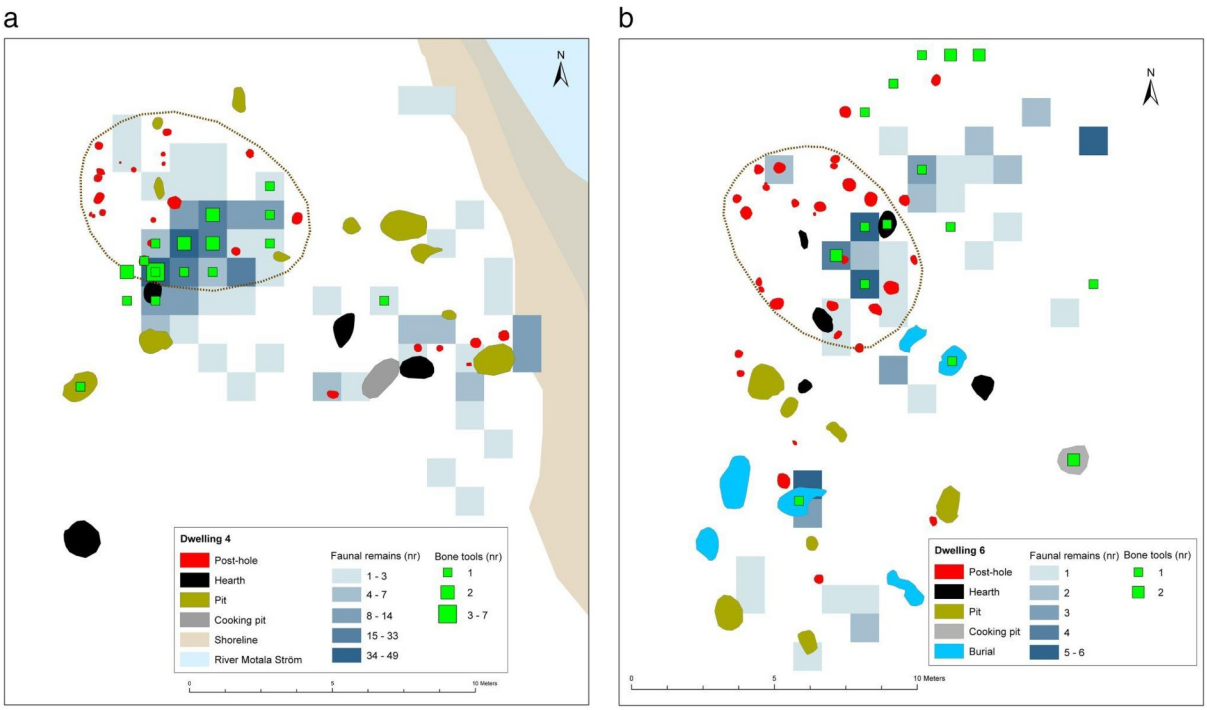

Fig 3.5. Dwelling 4 and 6 from the Late Mesolithic site of Motala in east-central Sweden, showing the domestic layout including hearths and cooking pits. Image after Molin, Hagberg and Westermark, 2018.

8.0. Conclusions

Despite discussions of Mesolithic aceramic cooking practices often receiving scant attention (Milner, 2009b; Warren, 2015), the above evidence shows that northern Europe during the early to mid Holocene is rich with examples of diverse cooking and food processing practices. The categories outlined in Chapter 2 based on the ethnographic literature were sufficient to analyse the literature and begin to classify the specific features of Mesolithic cooking methods. Table 3.1 displays the main *evidentiary* features for the Mesolithic which link to the more general aceramic cooking methods previously described.

Table. 3.1. Evidentiary components of different aceramic cooking techniques found in the northern European Mesolithic.

Feature	Description	Example site	Period	Reference
Hearths with FCRs	Hearth feature incorporating FCRs - slabs, cobbles, waste. Ambiguous/mixed purpose	Vængesø III (Denmark)	Late Mesolithic (Terminal Ertebølle)	(Andersen, 2018)
Hearths without FCRs	Hearth features without FCRs	Glenbatrick Waterhole (UK)	Early Mesolithic	(Mercer, Bain and Forster, 1975)
Stone boiling FCRs	FCRs displaying quenching / re-use damage	Clonava 1 (Ireland)	Late Mesolithic	(Little, 2014)
Sand/charcoal lenses	Small surface, sub-surface feature hearths composed of heated sand/charcoal	Hylteberga nr 9 (Sweden)	Early Kongemose	(Grøn, 2003)
Cooking pits	Dug hearth feature incorporating FCRs, waste	Dumpokjauratj (Sweden)	Early Mesolithic	(Bergman, 2008)
Earth ovens	Cooking pit with evidence of earth oven features - layers of FCRs, charcoal, plants, earth, maybe fire over the top	Kopu IA (Estonia)	Late Mesolithic (Narva)	(Kriiska *et al.*, 2017)
Discarded FCRs	Piles or refuse areas of cracked, damaged FCRs away from hearth or fire features	Okrouhlík (Czechia)	Early Mesolithic	(Ptáková, Šída and Kovačiková, 2021)
Specialised (sand)	Heating system where sand is the dominant direct heating feature	Duvensee WP8 (Germany)	Early Mesolithic	(Holst, 2010)
Specialised (clay)	Heating system where clay or layers of clay are the dominant direct heating feature	Schwarzenberg Lake (Czechia)	Early Mesolithic	(Pokorny *et al.*, 2010)
Griddle stones	Flat stone slabs with signs of heating, cracking or burnt food	Narva-Joaorg (Estonia)	Late Mesolithic	(Sander and Kriiska, 2018)
Fermentation	Pit or gulley containing extensive animal/fish bones, biomolecular residues	Norje Sunnansund (Sweden)	Early Mesolithic	(Boethius, 2016)
Smoking/drying	Evidence for specialised structure or contextual based on site layout - post holes, hearth/pit for smoking, turf depositions	Kampen (Netherlands)	Late Mesolithic	(Huisman *et al.*, 2019)

Fat rendering/burning	Fatty soil residues, biomolecular residues of marine lipids, concentrations of plant kernels	Ormen Lange (Norway)	Early Mesolithic	(Breivik, 2020)
Aceramic vessels	Imprints of baskets or netting, survived container artefacts, tripod post holes	Tybrind Vig (Denmark)	Late Mesolithic (Ertebølle)	(Harris, 2014)

One major challenge in developing this systematisation of Mesolithic cooking techniques is the multivalency of the basic feature - the hearth. Despite the taphonomic value of charcoal, bone, phytoliths, burnt flint, FCRs and other materials extracted from hearths, establishing the *function* and *meaning* of fire and combustion is extremely difficult, a point recognised by Binford in his ethnoarchaeological work (Binford, 1967, 1980, 2012). Aside from cooking, pit-hearths and FCRs have numerous other practical functions, most obviously heat and light. Recent work on Upper Palaeolithic pyro- and luminescence technology has revealed the rich complexity of hearth structures which were likely designed to maximise heat and light (Braadbaart *et al.*, 2020; Hoare, 2020; Murphree and Aldeias, 2022). At the Neanderthal site of El Salt (Spain), pit-hearth H77 was estimated to reach between 500 - 600 C, which would have been more than sufficient to keep its inhabitants warm (Leierer *et al.*, 2020). As was discussed regarding the function of the Dutch and Belgian Mesolithic pits, other practical uses of hearths and FCRs include smoking (Skibo and Schiffer, 2008); distilling or processing adhesives (Koch and Schmidt, 2022); steam for medical or ritual purposes (Barfield and Hodder, 1987); hardening wood (Aranguren *et al.*, 2018) and other utilitarian activities. More broadly the uses of fire in the ethnographic record testify to the religious and animistic aspects of pyrotechnology, particularly for ceremony and the ‘cleansing’ ability of fire to burn things away (Mallol *et al.*, 2007; Spikins, Kelly and Manzi, 2010; Henry *et al.*, 2018).

Thus tackling the multivalency of hearths and FCRs is crucial to establishing the function of each pyrotechnological feature. Archaeological and experimental hearths have been studied using a broad range of approaches, including micromorphology (Huisman and Tebbens, 2021), petrography (De *et al.*, 2015), archaeomagnetism (Herrejón-Lagunilla *et al.*, 2024), optical stimulating luminescence (Polo-Díaz *et al.*, 2023), thermal luminescence (Sun *et al.*, 2018) and organic residue analysis (Jambrina-Enríquez *et al.*, 2019). The extraction of lipids and organic compounds from food crusts attached to Aleutian griddle stones

demonstrates the value of recovering retained fatty acids in establishing the use of the stones (Admiraal *et al.*, 2019). Chapter 5 will cover more comprehensively the work to date on organic residue analysis on stone directly, suffice to say this method holds great potential in helping to determine the functionality of Mesolithic hearths and FCRs.

9.0.References

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Chapter Four: An experimental study of wet-cooking in organic vessels: implications for understanding the evolution of cooking technologies

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1.0 Abstract

The ability to control and direct fire is a major evolutionary step in the human story. The development of aceramic cooking technologies is less well understood as they rarely survive in the archaeological record. However, inferential evidence such as fire-cracked rocks, earthen pits and heated bones suggest a variety of cooking methods were used prior to the invention of ceramics. Yet there is a paucity of experimental evidence testing the efficacy of perishable organic containers in tasks involving their use with heat. The study presents experimental results of organic containers and their use for heating water related to cooking. Containers were made from deer hide and pig stomach and water was heated using two different techniques: placing the container directly above a fire and placing hot stones into the container. The results suggest that different organic containers and heating types could attain and maintain a sub-boiling cooking temperature; however, not all could reach boiling point. It is argued that these sub-boiling methods may be as, or perhaps more, desirable than boiling, with potential implications for the development of vessels prior to the adoption of ceramics.

2.0 Introduction

The thermal alteration and processing of foodstuffs is widely considered to be a significant threshold in human evolution (Wrangham and Conklin-Brittain, 2003; Gorman, 2008). It has been argued that predation pressure acting on early hominins may have selected for increasing group size (Coward and Gamble 2008; Hart and Sussman 2005), demanding a larger brain geared towards negotiating a greater number of more complex social relationships, necessitating adaptations such as language (Aiello and Dunbar 1993; Dunbar 1993; Arsuaga and Martínez 1998; Gamble 2002; Gowlett 2006). The expensive tissue hypothesis, which claims that decreasing gut size energetically facilitated increasing brain size, is one explanation as to how the brain could so rapidly expand (Aiello and Wheeler 1995). Cooking may have reduced the caloric cost of digestion, providing the necessary free energy to help stimulate this cognitive advancement across generations (Aiello and Wheeler, 1995; Boback *et al.*, 2007). Yet, for all the attested importance of pre-ceramic cooking, the physical practices and methods of containment and heating technologies remain relatively under-researched and discussed (Wright, 2004; Wrangham, 2007).

Speth (2015) explored the mechanism of boiling water in perishable materials and found it to be accessible to Palaeolithic humans. Boiling has unique capabilities - such as fully eradicating pathogenic microbes from meat (Avens *et al.*, 2002) and degrading collagen strands to the point of gelatin formation (Lawrie and Ledward, 2006). However, foods that are boiled for long periods will eventually disintegrate. Additionally, the fuel required to raise water to boiling point and maintain it may be unavailable or a prohibitive investment of labour to collect. Perhaps more importantly, it may not be strictly necessary to achieve boiling to wet-cook food. When considering the use of organic containers in wet-cooking, it is therefore important to distinguish between the use of heated vs boiling water. For example, starches can be hydrolyzed at temperatures from 35˚C upwards (Shariffa *et al.*, 2009) and when cooked become significantly more bioavailable (Carmody and Wrangham, 2009). Likewise with meat, heat will denature and unravel complex proteins resulting in a larger number of proteolytic cleavage points and a more thermodynamically efficient digestion process (Carmody, Weintraub and Wrangham, 2011). Muscle meat specifically is comprised of a number of structural proteins, including actin, α-actinin and myosin, as well as sarcoplasmic and globular proteins, all of which begin to denature at temperatures between 40°C and 60˚C (Cheng and Parrish, 1979; Kemp, North and Leath, 2009; Yu *et al.*, 2017). The benefits of heating water are not limited to cooking. For example, in some plants this can extend to

increasing ease of peeling (Henry, 2017) and the extraction of potentially harmful compounds (Ressler *et al.*, 1997). However, an experimental assessment of the aceramic technology used in methods of cooking using a sub-boiling strategy has not been explored. Given that many of the advantages of cooking food can be achieved with sub-boiling temperatures, it is important in any experiment surrounding aceramic cooking technology to avoid focusing solely on boiling temperatures and the methods and materials that can be used to reach and sustain them. Thus, this study aimed to test via a programme of experimental archaeology the feasibility of heating water - both to boiling and sub-boiling temperatures using a range of organic containers, via both direct and indirect heating methods, to inform the question of the development and nature of wet-cooking prior to the adoption of ceramics.

2.1. Background

2.1.1. Cooking before the invention of ceramics

The earliest potential use of fire by hominins - the prerequisite for any type of cooking- has been suggested to originate from Africa as early as 1.5-1 mya. Evidence includes baked sediment and heat altered stones recovered from Koobi Fora (Kenya), dating to 1.4-1mya; concentrations of baked clay in association with tools and animal bones from Chesowanja (Kenya), dating to 1.4-1mya; burnt bone recovered from Member 3 of Swartkrans (South Africa), dating to 0.8-1mya; and evidence of routine burning of vegetation from layer 10 of Wonderwerk Cave (South Africa), dating to 1-0.8mya (Dunbar and Gowlett 2014; James et al. 1989; Gowlett 2015). The earliest substantial evidence for the controlled use of fire is found much later from sites including: Gesher Benot Ya'aqov (Israel) dating to c. 790kya, where ash, charcoal and burnt flint was recovered (Alperson-Afil and Goren-Inbar, 2010) Zhoukoudian Locality 1 (China) dating to c. 670-400kya, where burnt bone and chipped-stone artefacts were identified ; and Beeches Pit (Britain) dating to c. 400kya, where abundant burned stone tools were found (Gowlett 2006; James et al. 1989; Gowlett 2015). However, it is not until the transition from Lower to Middle Palaeolithic that evidence of fire control becomes a recurring feature of archaeological sites (Preece *et al.*, 2006; Roebroeks and Villa, 2011; Mallol *et al.*, 2013).

Perhaps as a result of the scarcity of available evidence, discussions of Palaeolithic cuisine have traditionally focused on the roasting of meat over open fires (Stratus, 1989; Germonpré and Lbova, 1996; Barkai *et al.*, 2017). 'Dry-cooking' techniques of this kind are presumed to dominate in large measure because 'wet-cooking' necessitates the surrounding of food in water within enclosed containers, a technology presumed to appear much later in the archaeological record (Gamble, 2009). Previous attempts to characterise aceramic cooking technologies have argued for a linear evolutionary model, placing different techniques into a chronological sequence (Benison, 1999; Thoms, 2009). Yet it remains difficult to identify a clear pattern in the prehistoric record given the limited available evidence. Indeed, Speth (2015) suggests that archaeologists may not have fully appreciated the potential for fragile organic materials to be used for boiling water directly over a fire, which amongst ethnographically documented societies includes containers made from bark, wood, shell, hide, animal organs and stone (Nelson, 2010). However, direct and indirect evidence for the use of non-ceramic or "aceramic" vessels" has been increasingly reported, which has advanced understanding.

Direct evidence includes the recovery of heated stones and cooking containers dating to the Middle Palaeolithic and Middle Stone Age (MSA) (Carbonell *et al.*, 1996; Oestmo, 2013; Bentsen and Wurz, 2017; Bentsen and Wurz, 2017, 2019; Carbonell *et al.*, 1996; Oestmo, 2013; Bentsen and Wurz, 2017); two wooden vessels from Abric Romani (Spain) dating to 45,000-49,000 BP found in association with a hearth and a possible tripod (Carbonell *et al.*, 1996); wooden troughs from the Mesolithic site of Friesack IV (Germany) dating to between 8170-6990 BP (Bonsall, 1989, p. 314); a wooden container and possible containers made of birch bark from the Early Mesolithic site of Star Carr (UK) dating to 9385-9260 cal BC – 8555-8380 cal BC (Fletcher *et al.*, 2018); possible containers made from birch bark Nizhny Veretye I (Russia) dating to 9000-8000 BP (Bonsall, 1989, p. 406) and from the Late Mesolithic site of Szczepanki (Poland) dating to ca. 7000-4500 cal BC (Gumiński, 2012). Diverse indirect traces have also been recognised – all of which would likely necessitate some type of organic container. This includes: the use of containers to boil bones for grease extraction (Krief *et al.*, 2015) hot stone cooking from Pavlov VI (Czech Republic) dating to 26,000 BP (Svoboda *et al.*, 2009) Shuidonggou, Locality 12 (China) dating to 11-12,000 BP (Gao and Dennell, 2014; Svoboda *et al.*, 2009); Magdalenian examples (Batchelor, 1979; Bolus, 1990; Lucquin, 2007; March and Lucquin, 2007; Nakazawa *et al.*, 2009); circular areas devoid of remains believed to represent the negative spaces left by perishable containers on the floor of the Magdalenian habitation number 1 of

Pincevent (France) (Leroi-Gourhan and Brézillon, 1966); pits believed to be waterproofed with hide to be used as containers at the Magdalenian site of Gonnersdorf (Bosinski, 1981); and hide containers used over direct heat (Speth 2015).

Indirect evidence from recovered food remains can also point to the probability of prehistoric wet-cooking. Charred food aggregates from Franchthi Cave in Greece, dating to between the Bølling-Allerød and early Holocene, revealed a starch-rich matrix made from different wild pulses (Kabukcu *et al.*, 2023). The authors point to the necessity of soaking, heating and possibly boiling the pulses to achieve the observed level of microscopic processing. Such an activity seemingly necessitates a container which could enclose the seeds and foodstuffs, whilst modifying them using heated water. Given the recovery of both direct and indirect evidence that might represent Palaeolithic and Mesolithic container use and the deep timeframe for hominin fire manipulation, this raises the possibility for an early origin to wet-cooking cuisine, with potential implications for human evolution.

The shift from aceramic to ceramic technology occurred much earlier than previously believed, with the oldest evidence discovered in Southern China, dated to approximately 18-20,000 BP (Kuzmin, 2017; Patania *et al.*, 2019; Patania and Jaffe, 2021; Kuzmin, 2017; Patania *et al.*, 2019), then Japan and the Russian Far East by 16-10,000 BP (Shoda *et al.*, 2020). A recent geoarchaeological assessment of the early pottery site of Yuchanyan (18,300 cal BP) revealed a sophisticated suite of pyrotechnologies, including clay lined hearths and ceramics, probably deployed for processing and rendering bone fats (Patania and Jaffe, 2021). This transition phase seems to have captured otherwise invisible information about aceramic organic containers, such as baskets or string nets, with these potentially serving as a template for producing pots. Cordage and netting impressions on ceramic sherds have been found on the earliest Russian pottery (Zhushchikhovskaya, 1999; Hyland *et al.*, 2012) and on fired clay fragments dated from Spanish Palaeolithic-Mesolithic transition (Tortosa *et al.*, 2019). Techniques employed to build Ertebolle pointed base vessels may also have derived from coiled basketry (Povlsen 2013). The contextual evidence and discussion about how and why ceramics emerged and dispersed across the world has been discussed in a number of works over recent decades (Murdock and Provost, 1973; White, Burton and Brudner, 1977; Reid, 1984; Brown, 1989; Hayden, Barnett and Hoopes, 1995; Sassaman, Barnett and Hoopes, 1995; Rice, 1999; Jordan and Zvelebil, 2016; Piezonka, 2021; Dolbunova *et al.*, 2022). These debates highlight that both ‘functional’ and ‘social’ driving forces contributed to the development and refinement of ceramics, with

both requiring consideration when trying to understand why different societies chose to prioritise pottery over pre-existing aceramic cooking methods. However, a number of presumptions still prevail in these discussions, epitomised here by Jordan and Zvelebil:

"With many organic technologies able to perform the roles played by pottery, what, other than direct boiling ability, might have made pottery more attractive?" (Jordan and Zvelebil, 2016, p. 57).

The two assumptions here - that aceramic technologies are incapable of direct boiling, and that boiling is in itself the most useful and productive cooking technique - deserve to be interrogated more fully. Despite advances based on the increasing available material evidence for early pottery and their uses, expansion of the discussion to the likely organic technologies that preceded it remains limited. Closer consideration of how and when humans began to use aceramic containers for wet-cooking is therefore required, with experimental archaeology being an important methodological tool for advancing understanding in this area. To address Speth's (2015) challenge to explore the possibilities and evidence for boiling and aceramic wet-cooking more generally in archaeology, an experimental programme was designed with the aim of testing how aceramic vessels perform and the particular material properties involved in their functionality.

3.0 Materials and Methods

3.1 Methods

In total six experiments were conducted: in five cases attempting to heat water to boiling and sub-boiling thresholds, and one experiment attempting to reach and sustain 'long-time low-temperature' cooking range of 45-70°C. Of the six experiments carried out, five used a 'Direct Heat' method (defined as the suspension of the container over a heat source to heat directly) and one used a 'Hot Stone' method (defined as an indirect heating technique where stones are heated and transported into the container which heats the contents). Two organic container materials were tested: red deer hide and pig stomach. Experiments consisted of:

1) direct heat via a fire using a suspended pig stomach container (Fig. 4.1);
2) direct heat via a fire using a suspended red deer hide container with decreasing distance to the fire (Fig. 4.1);
3) direct heat via a fire using a suspended red deer hide container with stable distance from the fire (Fig. 4.1);
4) direct heat via embers contained within a pit covered with a red deer hide container (Fig. 4.2);
5) indirect heating using hot stones added to a suspended pig stomach container (Fig. 4.3);
6) and direct heat via a fire using a suspended red deer hide container for over three hours (Fig. 4.1).

Each experiment consisted of a heating source (direct fire, embers, hot stones), a container and water, allowing for the following variables to be measured:
1) increase and decrease in water temperature over time;
2) the time taken for the water to reach boiling or sub-boiling point;
3) fuel consumed to achieve water heating;
4) whether cooking of the foodstuff was achieved; 5) the type and extent of damage or modification to the organic container.
This allowed for contrasts between both material types using the same heat source and for comparisons between the heating methods.

The experiments were conducted in two phases. Phase one was primarily qualitative and aimed to test aceramic boiling using a sample of materials and heating methods discussed by Speth (2015). Phase two expanded on the results of phase one, testing sustained sub-boiling. The temperature of a litre of water was raised to and held between 45-70˚C for three hours, thus subjecting the container to conditions consistent with 'long-time, low-temperature' cooking. An unprocessed red deer hide suspended directly over a fire was used, which allowed for the testing of Speth's (2015) challenge of using organic perishable containers placed directly into a fire, while also being compatible with the earliest technology available to produce 'simple containers', based on early dates for animal skinning (Verheijen et al. 2023).

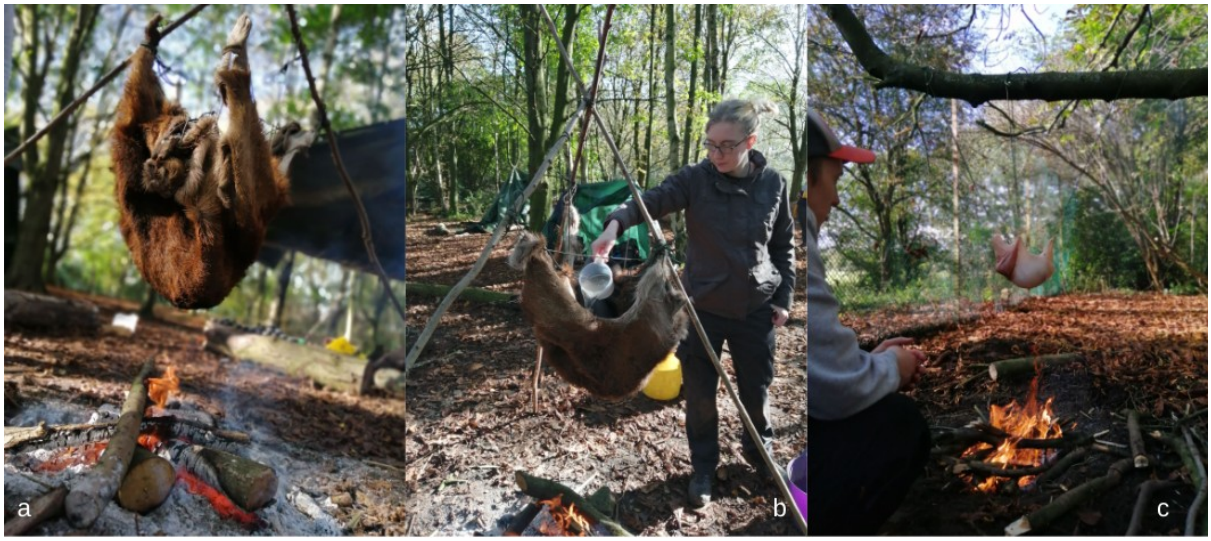


Figure 4.1. Photographs of the Direct Heat method experiments. (a) Vessel Three (V3) red deer hide with water over a fire. (b) Vessel Two (V2) red deer hide being filled with water. (c) pig stomach Vessel One (V1) suspended over a fire.

Figure 4.2. Pit cooking method photographs of Vessel Four (V4). (a) sticks being used to stop the hide slipping into the fire (b) steam visible as the water heats up.

Figure 4.3. Photographs of the Hot Stone method. (a) Vessel Five (V5) pig stomach vessel with water discolouration from the stones. (b) the stone rotation method, whereby stones were heated and then slowly dried out after use to avoid thermal shock.

3.1.1 Experiment 1: Direct Heat methods

In order to establish the efficiency of heating water with the heat source positioned directly beneath the vessel, two container types (pig stomach, red deer hide) and two suspension techniques (pit, tripod) were tested across four vessels: V1-V4. Vessel 1 (V1) was made from pig stomach (*Sus scrofa*) and was suspended from a tripod over a fire using paracord (Fig. 4.1). V1 was filled with c. 1L of water for the experiment. The pig stomach membranes proved ineffective in pre-experiment testing. Water penetrated between the inner and outer membranes, causing a bubble. Instead, the stomachs were used inside out, which prevented this problem during experimentation. The sphincters and mechanical damage were repaired using a leather sewing kit which produced a pouch, into which water could be poured. V2 and V3 used red deer hide containers (Fig. 4.1) each containing c. 2L of water and tied with paracord to a tripod built from hazel poles, suspended over a fire. The fur-bearing side of the hide was used as the outside of the container and the interior was filled with water. Hearths were centrally located beneath each tripod and a vessel suspended

from each using cord. The experiments differed only in the distance from the fire, which was kept the same in V3 and progressively lowered into the fire until the base of the hide was resting on the coals in V2. V4 used a deer hide container suspended over a c. 50x50x25cm pit filled with burning coals, embers and fresh wood. V4 was then placed over the pit with the central depression filled with c. 2L of water and the edges secured with branches. (Fig. 4.2).

3.1.2 Experiment 2: Hot Stone Methods

The Hot Stone method heats the contents of the container by placing heated stones directly inside the vessel. The aim of these experiments was to attempt to heat the water in the containers solely through the indirect transfer of heat via the stones. Pig stomach (V5) was selected for testing due to their ability to hold both water and heated stones. The pig stomach (V5) was prepared as in Experiment One and suspended from a branch with c. 1L of water added (Fig. 4.3). In order to raise the temperature, the hot stones needed to be placed into the water and quickly removed from the container before being added to the fire to reheat. To avoid thermal shock and for safety, rapid heating of stones was avoided by placing stones c. 30 cm away from the fire and moving them closer over time to allow for controlled heating to c. 250-500˚C. It was common throughout the experiments for multiple stones to be used simultaneously; typically two, in order to maximise the heating potential.

3.1.3 Experiment 3: Direct Heat ‘long-time, low temperature’

A single unprocessed red deer hide was modified to create a simple container by periodically piercing the outer edge with a sharp flint flake and threading string through the perforations. The hide was then attached to a simple wooden tripod - made from three pieces of wood tied together - using the string (Fig. 4.1). A litre of water was added to the hide and an assessment of any damage to the hide container - indicated by leakage - was made prior to commencement of the experiment. Hide container integrity was actively managed via periodic checking for tears or leakage, involving a reorientation of the hide when this occurred. This experiment placed greater emphasis on quantification to understand the dynamics involved in LTLT cooking. Firewood was measured before and after the experiment by measuring out an area in cubic feet, filling the space with the wood before use, and measuring the remainder after the experiment concluded, to quantify total

consumption. Thermocouples were used to record temperature data in both the container and the fire. Temperature data was recorded every five minutes on commencement of the experiment, which for the purpose of timing, was taken as the water in the hide container reached the desired temperature threshold. The experiment proceeded to attempt to maintain this temperature for 3 hours to simulate LTLT cooking conditions. Temperature was actively managed by adjusting the distance between heat source and container using the strings to raise up or lower down the container.

3.2 Materials

The experiments were conducted outdoors at the York Experimental Archaeology Research (YEAR) Centre, Department of Archaeology, University of York (UK) over two sessions, one in the autumn and the second in the spring. The vessels which required direct heat had their own separate hearths within the YEAR Centre. An infrared laser digital thermometer (temperature range: -50 - 750°C; accuracy: ±2°C) was used to make all temperature recordings for the first session, and a thermocouple set (TM-RS232 Thermometer) was used for the second. Other equipment - tripods, sewing kits, flint, etc. - were supplied by the YEAR Centre. For the first session, firewood was a mix of horse chestnut (*Aesculus hippocastanum*) and elder (*Sambucus nigra*) derived from the YEAR Centre itself, supplemented by kiln dried birch (*Betula pendula*), purchased from a commercial supplier. For the second session the firewood was mixed hardwood from a commercial supplier. Heating stone selection was balanced against the need for safety while maintaining archaeological and ethnographic fidelity. Coarse-grained, commercially shaped and rounded (8cm x 6cm x 2cm) basalt was selected for its resilience to thermal fracture (Wilson and DeLyria, 1999; Shantry, 2020). To comply with ethical guidelines set out by the university, unprocessed adult male red deer (*Cervus elaphus*) hides and pig stomachs (*Sus scrofa*) used in making containers were procured from commercial suppliers.

4.0 Results

The results from Experiment 1 and 2 are presented in Table 4.1 and Figure 4.5. Each experiment is discussed in turn, along with a summary comparison of Direct Heat results, Hot Stone results, with the two methods then compared.

Table 4.1. showing results of heating experiments V1-V5. The table shows the type of experiment configuration, the length of each experiment, the maximum water temperature achieved within this time and whether a cooking temperature was achieved, plus modifications to the vessel observed during the experiment.

Vessel	Material	Type	Distance to heat (cm)	Water vol. (ml)	Time (min)	Max water temp (°C)	Cooking temp. achieved	Vessel modification
V1	Pig stomach	Tripod, direct	30	1000	180	69.1	yes	Colour change, shrinkage (c. 70%)
V2	Red deer hide	Tripod, direct	50 initial, reducing to 0	2000	300	63	yes	Stiffening, charring of fur
V3	Red deer hide	Tripod, direct	50	2000	90	48.2	yes	Stiffening, charring of fur
V4	Red deer hide	Pit, direct	25	2000	90	88.6	yes	Stiffening, charring of fur, perforation
V5	Pig stomach	Tree branch, hot stone	0	1000	40	100	yes	Colour change, shrinkage (c. 50%)

4.1 Results: Direct Heat method

The results of experiment V1 - a pig stomach vessel containing 1L of water, suspended 50cm above an open fire - are presented in Table 4.1 and Figure 4.4. Figure 4.4 presents the change in temperature recorded through time for experiments V1-V5 and shows that water temperature inside the V1 container rose rapidly, reaching a maximum of 69.1˚C. However, this was not consistently maintained throughout the experiment (Fig. 4.4). While this experiment failed to achieve a temperature to boil water, a sub-boiling temperature suitable for cooking food or other non-cooking tasks was achieved and maintained. As the experiment continued, physical modifications to the vessel became apparent. The stomach shrank significantly, dropping its water capacity from an initial 1L to a final capacity of approximately 300ml, a loss of c. 70% (Fig. 4.5). Shrinkage of this magnitude may have implications for the types of uses or lengths of time to which a vessel of

this kind might be employed. Colour change in the container was also detected, likely as a result of the container beginning to cook after prolonged exposure to the heat source.

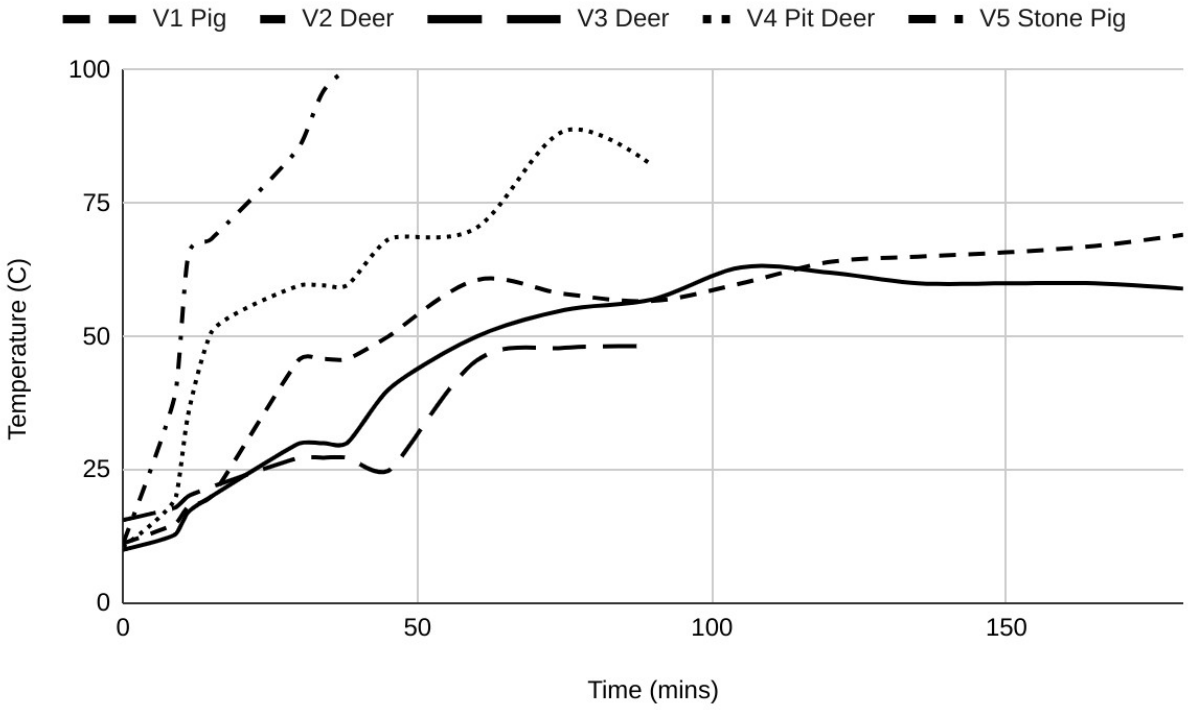


Figure 4.4. Graph showing temperature (°C) achieved through time (minutes) in experiments V1-V5.

Figure 4.5. Photographic illustrations of damage and wear to the organic vessels during cooking. (a) pig stomach (V1) showing signs of discolouration and shrinkage from the heat. (b) the fur burning and coming away from the red deer hide vessel (V6). (c) the blackening and scorching of the hide when placed over the fire (V6).

The results of experiment V2 - a red deer hide vessel containing 2L of water, suspended over and gradually lowered into a fire via a tripod - are presented in Table 1 and Figure 4.4. Recorded water temperature inside the V2 container initially rose rapidly, eventually reaching a maximum temperature of 63˚C, which was maintained for the remainder of the experiment (Table 4.1, Fig. 4.4). In this experiment, the distance between heat source and the base of the container was gradually reduced until the container was in direct contact with the fire and hot coals; nonetheless, the container did not achieve the greatest recorded temperature. The result suggests that both proximity to and structure of the fire are important for conserving and directing the heat into the vessel, rather than losing it into the surrounding air. As the experiment progressed, changes to the container became apparent. This included increasing rigidity of the hide and charring and blackening of the outer surface facing the heat source, but with no evidence of shrinkage (Fig. 4.5).

The results of experiment V3 - a red deer hide vessel containing 2L of water, suspended over a fire via a tripod - are presented in Table 1 and Figure 4. The heating pattern for the water in container V3 was markedly different to other experiments, with initial heating occurring gradually, followed by a rapid decline and equally rapid increase, before finally plateauing (Fig. 4.4). Fuelling of the fire may have contributed; the maximum temperature achieved was the lowest recorded: 48.2˚C across the direct heating experiments (Table 4.1, Fig. 4.4). However, the highest temperature recorded for the fire (877 °C) was associated with this experiment: evidencing a dramatic loss of heat in the air space between fire and container. It is probable that the thick deer hide acted as an efficient insulator, limiting heat transfer from fire to water. Observed changes to the vessel were in keeping with V2 (charring, blackening and stiffening), but less marked given the increased distance from the heat source.

The results of experiment V4 - a red deer hide vessel containing 2L of water, suspended over a pit with hot embers - are presented in Table 4.1 and Figure 4.7. The water within the V4 container was rapidly heated over a short period of time (Fig. 4.4). This is likely due to the small distance between heat source and container, but also because the pit walls and container base acted to trap and channel the heat. Because the heat source could not be replenished, a sharp temperature decline was observed prior to the termination of the experiment (Fig. 4.4). The greatest temperature of all direct heating experiments (88.6˚C) was achieved in this configuration, likely due to the short distance between container base and heat source and the channelling of heat. Modifications to the deer hide container were again similar to V2, but with no reduction in container capacity. A perforation in the container developed where the hide was not protected by water shortly after the termination of the experiment.

In summary, the results of the Direct Heat methods demonstrate the difficulty of raising water to boiling point within an organic container. Neither hide nor stomach are efficient conductors of heat and the fur on the exterior of the hide may have increased its insulating properties. This is in line with thermal conductivity studies of cow hides showing that hide has a similar efficiency level as air - an extremely poor conductor (Maia *et al.*, 2009). Nonetheless, sub-boiling temperatures were readily attained and maintained. While fuel consumption was not measured quantitatively, it was clear that the tripod method used significantly more firewood than the pit method. Characteristic modifications to the red deer

hide containers were observed across experiments, with extent varying with specific experimental parameters, especially proximity to heat source. In contrast to the pig stomach container, those made from deer hide displayed no visible signs of shrinkage.

4.2 Results: Hot Stone Method

The results of experiment V5 - a pig stomach vessel containing 1L of water into which hot stones were placed - are presented in Table 4.1 and Figure 4.4. Water temperature within container V5 rapidly and continuously heated throughout the experiment, exceeding the boiling point threshold of 100˚C prior to termination of the experiment (Fig. 4.4). To achieve this temperature, rapid rotation of heated stones was required to prevent the water cooling in between. A total of six stones was found to be sufficient: two were placed into the container while four heated in the fire. The stones themselves survived multiple rounds of heating and quenching with minimal cracking in the process. Repeated use of stones from the fire did, however, mean that the water began to be contaminated with ash, dirt and other particulates. This could have been mitigated by washing the stones before they were placed into the container after they had been heated and this should be taken into consideration when appraising the efficiency of heating the water. Physical modifications to the stomach were also noted. This included colour change, likely from cooking of the stomach in direct contact with hot stones, and shrinkage of around 50%. Despite this, it remained in a suitable condition to be reused for further heating. Overall the observations noted during this experiment closely matched many of those made by Ryder over 50 years ago, when he attempted to heat water in an animal stomach. The loss of volume through shrinkage, minor water displacement from the addition of hot stones and bubbling over of water at boiling point were observed during both experiments (Ryder 1969).

In summary, the two Hot Stone experiment results demonstrate that water can be heated rapidly and to a higher temperature when compared with Direct Heat methods. The experiments proved to be effective at boiling water (V5). However, there is a problem of contamination due to transfer of stones from a fire. Whilst indirect, the method is also fuel intensive as a fire must be maintained to heat the stones for the duration of the experiment.. It should always be acknowledged that actualistic experimentation carries many variables related to the skill and experience of the practitioners, and replication of these experiments by others would help make these results more rigorous. Nonetheless, given that the aim of

all experiments was to heat water, and that many forms of cooking do not require boiling water, both sets of results can be said to have achieved their initial goals.

4.3 Results: Direct Heat 'long-time, low-temperature'

The results of experiment V6 - a red deer hide vessel containing 1L of water suspended over a fire for three hours - are presented in Table 4.2, Figures 4.5, 4.6 and 4.7. Results demonstrate the viability of heating water to a consistent sub-boiling temperature for a protracted period without the vessel failing (Table 4.2; Fig. 4.6). Charring of the deer hide occurred and this was consistent with other direct heat experiments (Fig. 4.5). Active management requirements were minimal, with two interventions required: firstly to rearrange the hide to prevent the water pressure forcing a minor leak, and secondly to raise the vessel slightly to prevent the water becoming too hot. The volume of water remaining at the conclusion of the experiment was measured and totalled 650ml. When completely cooled, it became apparent that the substance had gelatinised, suggesting that the water was slowly breaking down the collagen from the interior membranes of the hide container. The fuel usage was approximately 65kg, which is high for a campfire (Pryor *et al.*, 2016), however this probably reflects the artificial nature of the experiment and the intentions of the practitioners to keep the fire as high as possible, knowing the temperature read-out from the thermocouple. Under 'realistic' circumstances, hunter-gatherer fires are often multipurpose and a cooking vessel placed over a fire would be just one function amongst many (insect repellant, warmth, social gathering, craftwork). Therefore it is difficult to directly compare the firewood usage for this single experiment with a lifelike scenario.

Table 4.2. The results for the Direct Heat 'long-time, low-temperature' experiment using vessel V6.

Vessel	Material	Type	Distance to heat (cm)	Water vol. (ml)	Time (min)	Max water temp (°C)	Cooking temp. achieved	Vessel modification
V6	Red deer hide	Tripod, direct	20, raised to 30 at 85 mins	1000	215	71.5	yes	Fur burnt, minor membrane damage

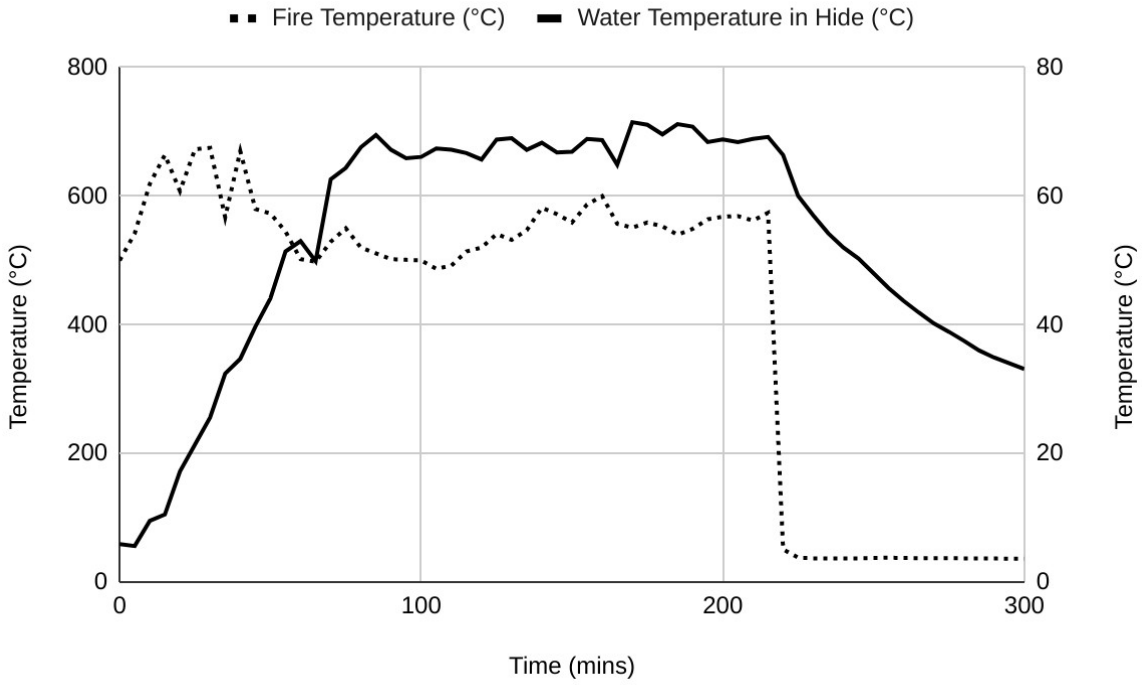


Figure 4.6. The temperature measurements for the Direct Heat 'long-time, low-temperature' experiment. Readings were taken every five minutes and the fire extinguished at 215 minutes in order to measure the cooling rate of the vessel.

The temperature data collected using thermocouples allows for characterisation of the thermodynamic system, expressed via a temperature against time curve T(t) model (Fig. 4.7). As the experiment was actualistic, a number of assumptions were made: 1kg of water was heated over an open fire using a red deer skin vessel; the skin was approximately 1mm thick; the fur, predominantly keratin, was assumed to be redundant following its rapid destruction by the fire; and the contact area (A) between the hide and fire was reasoned to be 800cm2. The T(t) curve was evaluated using Newton's heating law for the heating phase up to 215 minutes and after removal of the heat source Newton's cooling law was applied to fit the respective part of the curve.

The fit used is based on following consideration:
An instantaneous change in the transferred heat dQ is related to a small change of temperature dT by:

$$dQ = m_{water} \cdot c_{water} \cdot d\,T_{water}$$

where m_{water} is the mass of the heated water and c_{water} the specific heat of water. Hence the heating rate is

$$\frac{dQ}{dt} = m_{water} \cdot c_{water} \cdot \frac{dT_{water}}{dt}$$

Correspondingly, the heat transfer through the hide is proportional to the temperature gradient dT/dx and depends on its heat conductivity k_{skin} as well as the area A. The corresponding heating rate is then given in one dimension by

$$\frac{dQ}{dt} = -k_{skin} \cdot A \cdot \frac{T_{water} - T_{surface}}{d_{skin}}$$

Combining Eqs. 1 and 2 leads then to

$$m_{water} \cdot c_{water} \cdot \frac{dT_{water}}{dt} = -k_{skin} \cdot A \cdot \frac{T_{water} - T_{surface}}{d_{skin}}$$

Rearranging this we get

$$\frac{T_{water} - T_{surface}}{d_{skin}} = \frac{m_{water} \cdot c_{water}}{-k_{skin} \cdot A} \cdot \frac{dT_{water}}{dt}$$

This is a first order differential equation in space and time. Assuming now a linear gradient dT/dx = ΔT/d with d being the thickness of the skin this can be simplified to

$$\frac{dT_{water}}{dt} = \frac{-k_{skin} \cdot A}{m_{water} \cdot c_{water}} \cdot \frac{T_{water} - T_{surface}}{d_{skin}}$$

$$\int_{T_{water}(0)}^{T_{water}} \frac{dT'_{water}}{T'_{water} - T_{surface}} = -\int_{0}^{t} \frac{k_{skin} \cdot A}{m_{water} \cdot c_{water} \cdot d_{skin}} \cdot dt'$$

Integration with the boundary condition T_{water} either the initial temperature before heating or $T_{surface}$ the temperature before cooling. Distinguishing between the heating and cooling phases we find in the case of heating

$$T_{water}(t) = T_{surface} - \left(T_{surface} - T_{water}(0)\right) \cdot \exp^{\frac{-k_{skin} \cdot A}{m_{water} \cdot c_{water} \cdot d_{skin}} \cdot t}$$

(Eq.3)

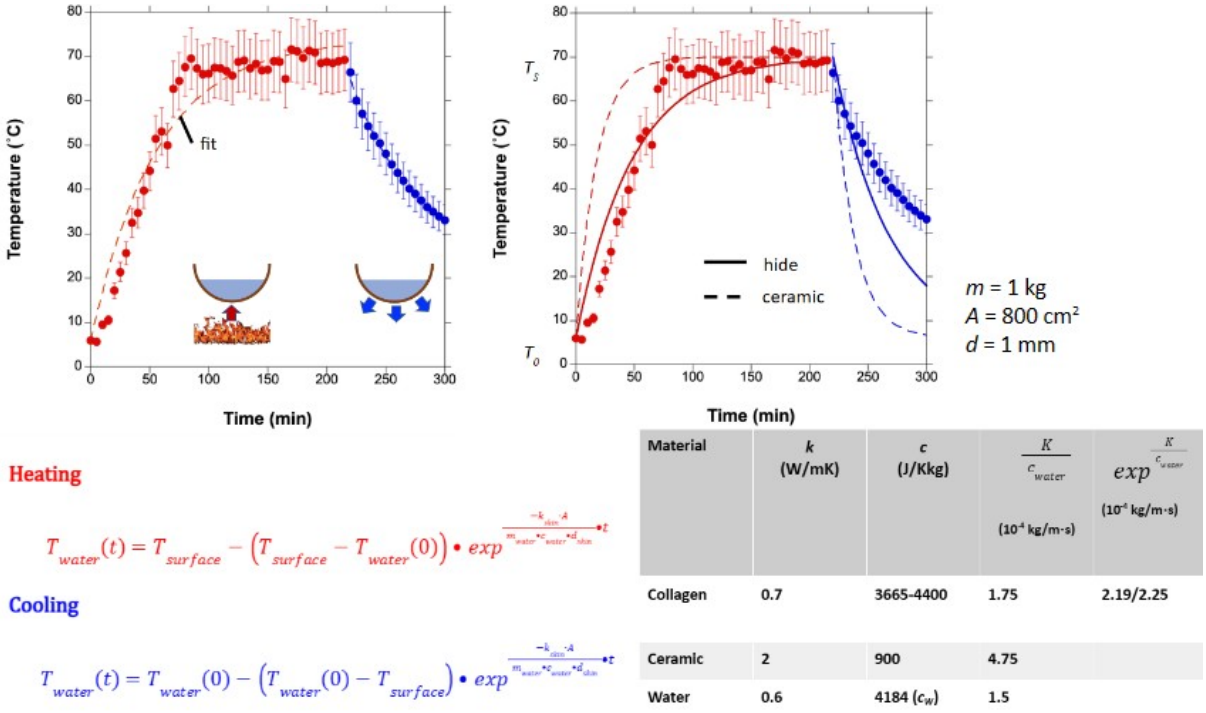
and in the case of cooling

$$T_{water}(t) = T_{water}(0) - \left(T_{water}(0) - T_{surface}\right) \cdot \exp^{\frac{-k_{skin} \cdot A}{m_{water} \cdot c_{water} \cdot d_{skin}} \cdot t}$$

(Eq.4)

Eqs 3 and 4, respectively, were used to fit the experimental T(t) data for heating and cooling with the parameters A = 800cm2, k_{skin} = 0.7 W/mK for collagen, c_{water}= 4184 J/kg for water, d = 1 mm, T_{water}= 6˚C (temperature of the environment) and $T_{surface}$= 70˚C (temperature of the heating zone above the fire).

The values for the k_{skin}/c_{water} ratios during the heating and cooling phase are 2.19 10-4 kg/ms and 2.25 10-4 kg/ms, respectively. Given the approximations made this is in remarkable proximity to the expected value of 1.75 10-4 kg/ms, corroborating the assumptions.



Material	k (W/mK)	c (J/Kkg)	$\frac{K}{c_{water}}$ (10^{-4} kg/m·s)	$exp^{\frac{K}{c_{water}}}$ (10^{-4} kg/m·s)
Collagen	0.7	3665-4400	1.75	2.19/2.25
Ceramic	2	900	4.75	
Water	0.6	4184 (c_w)	1.5	

Figure 4.7. Calculated temperature curves for the V6 Direct Heat 'long-time, low-temperature' experiment. The experimental data is plotted against a theoretical fit, as well as a comparative calculated ceramic curve. Equations 3 and 4 have been included, with the corresponding colours for 'heating' and 'cooling'.

Overall, the measured time dependence of temperature in- and decrease confirm that the key component of the hide acting as vessel for the heated water, is the collagenous deer skin. A comparison of the deer hide data with those theoretically expected for a ceramic vessel with a similar thickness revealed that the significantly higher thermal conductivity of ceramics led to attainment of the target temperature of 70˚C more quickly, while the cooling would also be faster. Hence, a deer hide based vessel is less favourable in terms of heating time to reach a targeted temperature but would keep the heated water hot for a longer period of time.

A number of useful observations can be drawn from these results. Firstly, the initial heating requirements are likely much greater to transfer the heat through the hide in comparison to ceramics, but the greater insulation properties suggests that either the fire could be reduced in intensity or even extinguished and the hide continues to cook the foodstuffs for longer. Secondly the outer layer of fur may contribute little to nothing to both the vessel's heating and cooling, presumably because the damage from the fire has undermined the molecular properties of the keratin fur matrix. Thirdly, that unprocessed

animal hide functions perfectly well as a vessel for LTLT cuisine - by eliminating the stipulation that cooking is synonymous with boiling, this type of direct heat food processing makes best use of the hide's thermal properties.

5.0 Discussion

Recent years have seen an increase in interest in hunter-gatherer aceramic container technologies and the keystone role they have played in our species survival and evolution, with ethnographic data used to inform a non-exhaustive range of uses, including: short and long-term storage and mobility of foodstuffs, water, raw materials, colourants, medicine, tools, equipment in addition to the carrying of infants and burial of corpses (Henrich 2015; Langley and Suddendorf 2020; Suddendorf et al. 2020). Not all, but many of these storage containers are made from materials that can be used for cooking and are at least semi-fire retardant (e.g., shells, plant leaves/fibres, animal skins/organs); however, the equation between containers and cooking is far from being a simplistic one. The *right* amount of heat, requiring use of the *right* type of vessel for cooking a particular foodstuff, has implications for how digestible the food is (e.g., detoxifying, tenderising and breaking down tubers and other fibrous plants; breaking down proteins found in meat), the digestive workload of our mouths, stomachs and colons, and ultimately, the degree of energetic benefits (Carmody and Wrangham 2009; Henrich 2015).

The experimental archaeological results demonstrate the effectiveness, but also key differences, between multiple methods of heating water in organic perishable containers. Experimental replication and use of hide containers with the Direct Heat method indicates that cooking at sub-boiling temperatures is achievable, while boiling may not be. Results from the stone cooking experiments confirm that this technique is more fuel efficient and effective at rapidly raising water temperature than placing the container directly over the heat. It is, however, interesting to consider that these results differ from other research investigating stone cooking. In Holman and Egan's (1985) experimental work studying the traditional methods of maple syrup production, they record that hot stone cooking was among the least efficient methods of reducing the liquid to a syrup. The time taken to make the syrup was measured at between five and six hours: an impractical amount of time to constantly rotate and manoeuvre the stones. This suggests that the efficiency of hot stone heating may be quickly undermined when the cooking procedure requires many hours of

heat. This is consistent with previously published work on stone boiling, which suggests the process can be rapid but increases labour costs (David and Massey 1957).

Across all experiments there was appreciable variation in the degree of monitoring and labour required to heat water. Hot stones were effective in raising the internal temperature of the vessel; however, they demanded a higher level of observation, planning, activity and monitoring. The use of animal skins directly over a fire may still require more active engagement than pottery, but for the user it is less involved than using heated stones. Equally, while both methods proved to be viable, the pit method was more fuel efficient than the tripod methods, which may have been a pertinent consideration when selecting the type of method to achieve a specific task. The container material also proved to be an important variable. The pig stomach container was more efficient at initial heat transfer when compared with the red deer hide containers, perhaps indicating that internal organ membranes might be more desirable as a vessel. It is, however, important to stress that containers were not tested to the point of destruction; in which case, its thinness, lower insularity and tendency towards shrinkage may prove less effective for slow cooking. When taken together, the results suggest that the material used in creating the vessel and the configuration used to heat its contents would be prominent variables that were actively considered in diverse tasks in different archaeological contexts. Viewing these results in the context of evolving and developing cooking strategies, it is likely that cooking foods using pottery would change the engagement for the user from active to potentially semi-active or passive. The social implications for users practising different methods of aceramic cooking are deserving of closer consideration in the future (see Wrangham 2010; Dunbar and Gowlett 2014).

Speth (2015) questioned the types of damage and alteration that might be seen in organic containers and the results provide insight. Red deer hides suspended above a fire using a tripod became increasingly hard and inflexible in the time it took to heat water. The fur on the underside of the hides became blackened and scorched but the heat failed to damage the skin tissues. The hide suspended above a pit was damaged soon after the experiment ended when the embers burnt through the sections which were unprotected by the heated water, demonstrating that a level of skill and attention is required to maintain even this method. The stomach containers used for hot stone and direct heating methods shrank considerably during the experiments, losing over half of their usable volume, likely due to the sensitivity of the tissue to heat. This suggests that contrary to Speth (2015), hot

stone cooking can be equally as damaging to the container as a direct method, depending on the material used. These findings further support the view that the material from which to make a container was taken into consideration when selecting the cooking method. For example, the capacity for damage from indirect hot stone cooking has been a key factor in identifying the function of different pottery styles (Sassaman, Barnett and Hoopes, 1995).

A related area of consideration is the extent to which inclusions from stone transfer or derived from leaching from the heated stones changes water composition, such as pH. While the experiments made use of basalt for safety reasons, globally it remains a lesser utilised stone and alternatives such as quartz and limestone may have their own effects on the cooking process. It is possible that specialised forms of plant processing relied on the deliberate selection of stones with particular properties. For example, work on a variety of plants has shown that alkaline solutions generated through limestone use can produce dramatic changes in plant and seed biochemistry (Abdel-Gawad, 1993; Ellwood *et al.*, 2013). Alongside changes in water composition is the more qualitative and sensory question of taste. Stone boiling has the potential to introduce foreign objects including ash, soil, grit and other particulate matter into the food. This can be avoided by cleaning the stones after they have left the fire, but before they are placed into the water, either through manually brushing or momentarily dipping them into another container of water. Fresh hide is not an instinctively appealing vessel material to contemporary western sensibilities, being a potential vector for zoonotic diseases, and potentially producing unpleasant odours and tastes if not used immediately from the animal. However, this should be balanced against knowledge from the ethnographic record that taste and smell are culturally contingent, and rotting, putrefying or decaying foods can be highly prized and savoured in different cuisines (Speth and Eugène, 2022). Therefore unprocessed animal skin can be considered viable when considering possible material choices throughout prehistory. Of course, other organic materials should also be trialled for their thermal properties in future research. Bark and wood are often noted in the ethnographic record as an alternative to animal-based containers, and an expansion of this type of actualistic experimentation into their working properties is essential to characterising aceramic cooking technologies as a whole.

During phase one of the experimental programme boiling of water using hot stones and direct heat was only achieved once. In this respect, reaching and maintaining boiling via aceramic methods can be seen to be challenging, but nonetheless, viable. Sub-boiling temperatures that would allow foodstuffs, such as starch or meat, to begin exhibiting

biochemical changes that would aid in their processing and digestion were consistently achieved. Phase two of experimentation demonstrated that such sub-boiling temperatures can also be sustained for protracted periods. Depending on the cuisine, maintaining consistent, low temperatures can be preferable to boiling. For example, cooking meat in water for long periods of time *below* 60 °C ('Long-Time, Low Temperature' (LTLT) cooking) results in significant collagen denaturation, while temperatures *above* this result in the meat being tougher (Paul, 1963; Bertola, Bevilacqua and Zaritzky, 1994; Latorre *et al.*, 2019). Research suggests that LTLT cooking produces a superior product both in flavour and tenderness (Tornberg, 2005; Christensen *et al.*, 2012; Dominguez-Hernandez, Salaseviciene and Ertbjerg, 2018), which is likely a prominent factor in why many cuisines employ long, slow cooking (Wandsnider, 1997). When these insights are applied to prehistoric contexts, it can be suggested that a cooking technology which is unable to attain boiling temperatures, but which can still heat water, may have even been desirable and advantageous. As Fig 4.8 demonstrates, the temperature spectrum of water possesses numerous useful sub-boiling thresholds, highlighting that the advantages of wet-cooking begin at lower temperatures than archaeologists have previously acknowledged. The experimental data indicates that Direct Heat cooking might be particularly well suited to harnessing the LTLT effect to process raw meat, and perhaps other types of food, into a bioavailable resource.

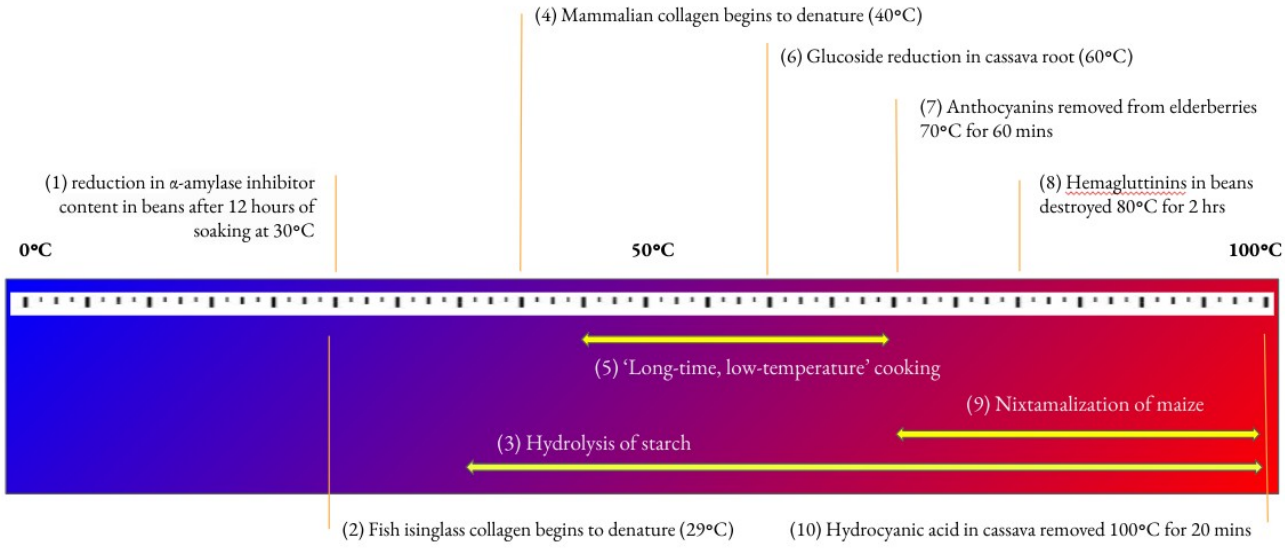


Figure 4.8. The utility of heated water does not begin at boiling point, important biochemical changes occur in different plant and animal products at sub-boiling temperatures for different periods of time. Many of these are crucial to either detoxifying or rendering carbohydrates

and proteins available for human digestion. (1) (Shi *et al.*, 2017); (2 & 4) (Hickman *et al.*, 2000); (3) (Shariffa *et al.*, 2009); (5) (Dominguez-Hernandez, Salaseviciene and Ertbjerg, 2018); (6) Oke 1983; (7) (Senica *et al.*, 2016); (8) (Thompson, Rea and Jenkins, 1983); (9) (Ellwood *et al.*, 2013); (10) (Modesto Junior, Chisté and Pena, 2019).

This conclusion is consistent with other experiments across diverse contexts. For example, a study by Hanson et al. (2019) of acorn processing tested both stone boiling and simmering in ceramic vessels, concluding that boiling the acorns had the effect of binding the bitter tannins to the nutmeat, while simmering methods instead leached them. Discussing the origins of ceramics during the North American Woodland Period, Skibo et al. (2009) observed that water boiling can bring with it limitations, including: overflow into the fire, unsuitability for extracting greases and oils, and the bubbling surface can inhibit skimming of fats. It should, however, be noted that boiling can have distinct advantages over heating in other contexts. For example, (Ellwood *et al.*, 2013)) demonstrate that using heated limestone to cook and nixtamalise maize relies on rapidly heating the water to between 70°C and 100°C, which provides the optimal environment for the kernels to absorb the alkaline water. Arnold's (1988) argument that the development of pottery was driven by the need to detoxify and make available the stored energy and nutrients in various plant foods remains important. Listing commonly used plants from around the world which require prolonged boiling to be rendered safe and edible also highlights a key advantage of pottery over aceramic cooking methods - the ability to withstand high temperatures for long periods of time. It has been proposed that the appearance of early pottery might be related to the exploitation of seasonal abundance of oily fish species, notably to the processing or storing of 'prized' aquatic oil with a highly symbolic value (Hayden 1995). Alternatively, pottery may have been used to cook previously stored commodities such as dried fish, as suggested by organic residue analysis of Incipient and Initial Jomon pottery (Lucquin et al. 2018). Lengthy simmering temperatures would have been easier to obtain using ceramic vessels in addition to providing a more secure and durable container within a context of increasing exploitation of aquatic ecotones (Wang and Sebillaud 2019).

Speth's (2015) original question of 'how did humans learn to boil?' can thus be usefully expanded to 'how did humans learn to cook?'. Since boiling may be both unnecessary and undesirable in various cooking recipes, separating 'rapid boiling' from 'LTLT' and recognising them as two specific and distinct methods of processing foods, each with potential advantages depending on the specific context, is important, with potential

implications for human evolution, the development of organic container technologies, and the transition to the use of ceramics. Thus, these two methods may have coexisted, targeting different commodities, fulfilling two different roles within a complex system involving diverse forms of food preparation: forming a cuisine.

6.0 Conclusions

Results from this experimental programme demonstrate the complexity of engaging with the evolution of material technologies and the benefits of experimentally testing latent assumptions. The results demonstrate that organic, perishable containers placed over direct heat are capable of heating water sufficiently to process foods, bringing into question the presupposition that a cooking technology should be necessarily judged on its ability to heat more quickly and to a higher temperature than another. In some contexts, higher temperatures can be undesirable and instead a lower and more stable temperature may be favoured. The ability to heat water and to boil water should be seen as two separate methods of food processing. Combining the scientific research into ‘Long-Time, Low Temperature’ cooking with the experimental data of direct heat cooking in perishable containers, it is proposed that the need for lengthy simmering temperatures, as opposed to rapid boiling temperatures, may have been a driver in the creation of ceramic vessels during the Upper Palaeolithic and a factor in their increase in dominance thereafter. Future research can more fully test this hypothesis by characterising other common organic vessel materials, such as bark and wood. Finally, the results demonstrate how experimental archaeology is an important method for understanding the functionality of different material properties and container technologies, even for a topic as seemingly mundane as heating water.

7.0 Acknowledgments

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Chapter Five: Review of organic residue analysis in archaeology with a focus on fire-cracked rocks

1.0 Introduction

The development and evolution of chromatographic analytic technology has revolutionised the chemical and biochemical sector, both in industry and academia (Leclercq and Cramers, 1998; Amirav *et al.*, 2013). The combination of chromatography and mass spectrometry has allowed researchers to separate compounds to a resolution approaching the lowest end of the picomole spectrum (Koslow, Cattabeni and Costa, 1972; Hasegawa, Kunihara and Maruyama, 1982). This has provided bioarchaeologists with a series of powerful analytical methods to recover and assess archaeological materials, including lipids (Evershed, 2008b), proteins (Kaal, López-Costas and Martínez Cortizas, 2016) and small organic molecules (Rageot *et al.*, 2018). The field of organic residue analysis has been successful in exploiting the chemistry of lipid formation, degradation and preservation in order to interpret the origins and functions of ceramic cooking vessels (Lucquin *et al.*, 2016; Bondetti *et al.*, 2019; Cramp *et al.*, 2019), as well as other reservoirs of biomolecular information, including: mummified human remains (Hertzog *et al.*, 2023); the production and uses of prehistoric adhesives (Koch *et al.*, 2024); the manufacture of ornaments (Golovanova, Kostina and Doronichev, 2024) and the residues left behind in domestic hearths (Kedrowski *et al.*, 2009). One ubiquitous artefact type that has seen less interest within ORA is fire-cracked rocks (FCRs) and stone used for heating and cooking in general. This chapter aims to explore the literature on FCR-ORA, by outlining the work to date and the evolution of the methodologies employed, as well as some issues which face the study of archaeological FCRs. This review is embedded within a more general overview of ORA as a field, including sections on relevant thermal biomarkers.

2.0 Organic residue analysis in archaeology

2.1 The history of organic residue analysis

While the origins of organic residue analysis in archaeology date back to the 1930s (Pollard and Heron, 2015, p. 474), the use of GC-MS begins with the analysis of 'bog butter' by Thornton et al (1970). Since then the analytical power of ORA has been recognised by archaeologists who have applied it to areas as diverse as ancient Egyptian bodily fluids (Kuksis *et al.*, 1978), Roman military diets (Knights *et al.*, 1983) and mediaeval pine pitch (Evershed, Jerman and Eglinton, 1985). The application of GC-MS to pottery sherds began in the 1980s, looking at classical amphorae (Passi *et al.*, 1981) and later on more prehistoric samples from South Africa (Patrick, Koning and Smith, 1985). The 1990s saw an explosion of pottery analysis, particularly by Carl Heron and Richard Evershed (Evershed, Heron and Goad, 1991; Evershed *et al.*, 1992, 1997; Dudd, Evershed and Gibson, 1999). This pioneering work has led to a standardised and rigorous approach to lipid residue analysis in ceramics which has been described as a 'revolution' in bioarchaeology (Evershed, 2008b). Of particular significance has been the creation of the *Archaeological Biomarker Concept* - which has its origins in organic geochemistry and the study of bitumens (Evershed, 1993). The biomarker concept has allowed researchers to draw lines of inference between the recovered organic molecules and the food or materials that may have been held in the ceramic matrix.

2.2 Overview of Lipid Chemistry

2.2.1 Fatty Acids

The predominant organic molecules studied in ORA research are lipids. Lipids are a class of organic hydrocarbons, often characterised by their insolubility in water, but also classified by their structural and biological features (Low *et al.*, 2009). In living systems lipids play key roles in organising the lipid bilayer of cellular membranes (Dowhan and Bogdanov, 2002), acting as sources of energy (Zimmermann *et al.*, 2009), signalling molecules (Hannun and Obeid, 2008), forming specialised tissues such as myelin (Sedzik, Blaurock and Höchli, 1984), interacting with the microbiome (Wang *et al.*, 2016) and many other systemic functions across the phylogenetic kingdoms (Gross *et al.*, 2005; Escribá *et al.*, 2007; Muro,

Atilla-Gokcumen and Eggert, 2014; Kobayashi, Endo and Wada, 2016). The International Lipid Classification and Nomenclature Committee has condensed the types of lipids into eight fundamental classes: fatty acyls (acids), glycerolipids, glycerophospholipids, sphingolipids, sterol lipids, prenol lipids, saccharolipids and polyketides (Fahy *et al.*, 2009). Of these, fatty acids are the most common and therefore the most studied.

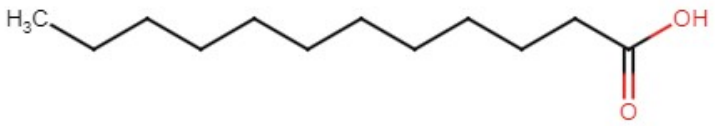

Fig 5.1. Dodecanoic acid ($C_{12:0}$), the sections in red highlight oxygen bonds, by author

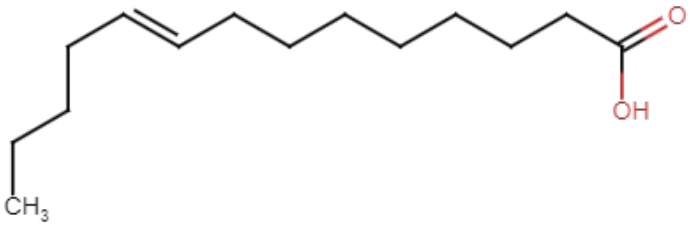

Fig 5.2. Myristoleic acid ($C_{14:1}$), the sections in red highlight oxygen bonds, by author

Fatty acids are constructed from linear sequences of carbon atoms and terminate with a carboxyl moiety (Fig. 5.1). The number of carbon atoms in the chain can vary from 2 to higher than 60 and alternative configurations include branches and one or more cyclic structures. Typically fatty acids are described as saturated, meaning that all the carbon atoms are joined by single bonds, but unsaturated fatty acids exist with one or more double bonds in the chain (Fig. 5.2). The double bond provides a degree of rotational flexibility which has consequences for the organism when the individual molecules are packed together (Eldho *et al.*, 2003). Branched chain fatty acids also provide an organism's structural functions; these are often generated by microorganisms for specific purposes and

the branch is classified as either *iso-* or *anteiso-* depending on which carbon atom the branch originated (Kaneda, 1991)(Fig 5.3). Straight chained fatty acids are referred to as *aliphatic.* Free fatty acids are rare in animal tissues and are typically found bound to other molecules, often having been *esterified (Hollenberg and Angel, 1963).* Three fatty acids bound to a glycerol backbone yields a triglyceride, a common form of energy storage in adipose cells (Gross, Snapp and Silver, 2010)(Fig 5.4).

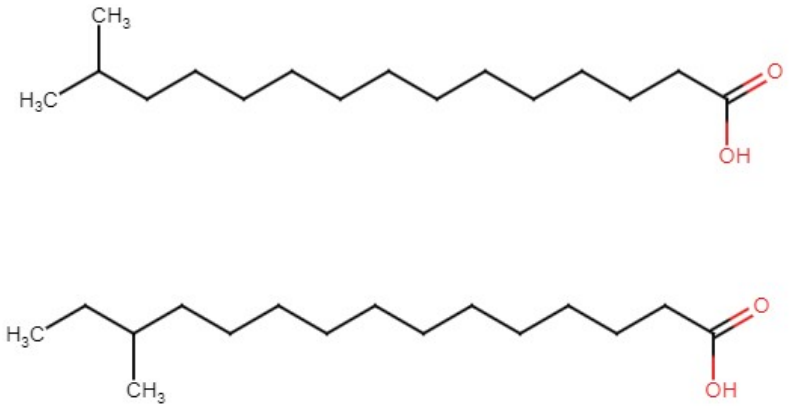

Fig 5.3. *Iso*-methyl (top) and *anteiso*-methyl (bottom) branched-chain fatty acids, the sections in red highlight oxygen bonds, by author

Saturated fatty acids are typically labelled by the number of carbons in the chain: hexanoic acid contains six carbon atoms; heptadecanoic acid contains 17 carbon atoms. These can be subdivided into short-chain fatty acids (less than five carbon atoms), medium-chain fatty acids (between 6 and 12), long-chain fatty acids (13-21) and very long-chain fatty acids (more than 22) (Fahy *et al.*, 2009). Most fatty acids found within the animal kingdom consist of less than 26 carbon atoms, although exceptions are found in products like waxes and wool (Kolattukudy, 1970; Körner, Höcker and Rivett, 1992). Unsaturated fatty acids are further designated by the terms *trans* and *cis,* which correspond to how the hydrogen and carbon atoms are fixed around the double bond (Oteng and Kersten, 2020). Unsaturated fatty acids are common across all plant, animal and aquatic life, but are more heavily concentrated in pigs, fish, marine mammals and nuts (Twining *et al.*, 2016). Branched chain fatty acids are typically the product of fermentation and bacterial activity (Vlaeminck *et al.*, 2006).

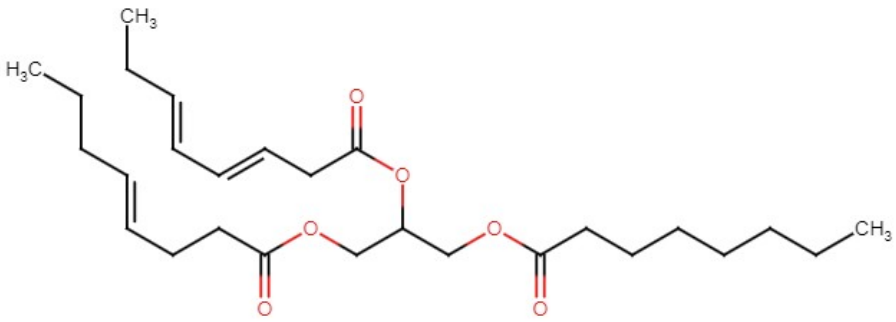

Fig 5.4. Triacylglycerol structure featuring different types of fatty acids, the sections in red highlight oxygen bonds, by author

2.2.2 Alkanes & alkanols

Linear aliphatic chains of carbon atoms with no functional groups - acyclic saturated hydrocarbons - are termed *n*-alkanes (Wentzel *et al.*, 2007)(Fig 5.5). Similar aliphatic chains with an additional hydroxyl group are termed *n*-alkanols (Ventolà *et al.*, 2002). Odd-number chain *n*-alkanes are known to be present in beeswax and plant waxes, such as those derived from leaves (Regert *et al.*, 2001; Jambrina-Enríquez, Herrera-Herrera and Mallol, 2018; Patalano, Zech and Roberts, 2020). Similarly *n*-alkanols are biomarkers of plant leaf degradation, since they are often derived from a parent *n*-alkane (Eglinton and Hamilton, 1967; Heron *et al.*, 2010).

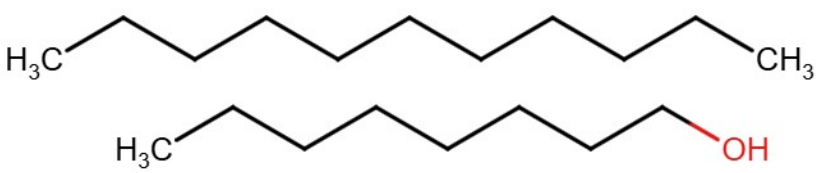

Fig 5.5. Top: linear alkane structure. Bottom: alkanol structure, the sections in red highlight oxygen bonds, by author.

2.2.3 Sterols

Sterols are a diverse group of molecules found both in plants (phytosterols) and animals (zoosterols), the latter of which are typically derivatives of cholesterol (Prost *et al.*, 2017; Rosiak, Kałużna-Czaplińska and Gątarek, 2020; Vallejo *et al.*, 2022). They consist of three six-membered rings and a four-membered ring, complete with a hydroxyl group (Fig 5.6). Numerous functional groups can be attached, and the functions of sterols reflect this diversity, including cell signalling (Sheng *et al.*, 2012; Zhang, Lin and Li, 2020), hormone precursors (Gimpl *et al.*, 2002) and cell membrane stability (Bloch, 1983). The presence of cholesterol derivatives such as coprostanol have been used to identify human and animal faecal presence on archaeological sites (Sistiaga *et al.*, 2014; Zocatelli *et al.*, 2017). However, the oxidation of cholesterol occurs rapidly under normal conditions, and therefore the presence of cholesterol, together with compounds such as squalene, likely indicates contamination from human handling (Evershed, 1993; Hammann *et al.*, 2018).

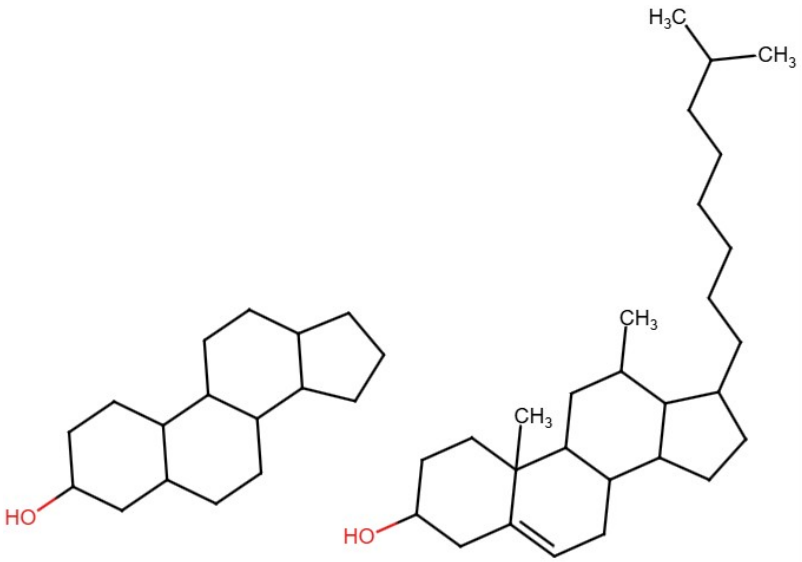

Fig 5.6. Left: depiction of a basic sterol molecule. Right: depiction of the structure of cholesterol, the sections in red highlight oxygen bonds, by author.

2.2.4 Terpenes

Terpenes are a large class of molecules derived from the polymerisation of units of isoprene (Ruzicka, 1953; Luong *et al.*, 2018). The resulting structures are highly diverse in

form, cyclisation, functional groups and reactivity (Evershed, 1993). In particular the diterpenoid and triterpenoid compounds that originate from ‘plant exudates’ have been extensively exploited by bioarchaeologists for their longevity and specificity of origins (Modugno, Ribechini and Colombini, 2006; Rageot *et al.*, 2016; Urem-Kotsou *et al.*, 2018; Courel, Adam and Schaeffer, 2019) (Fig 5.7).

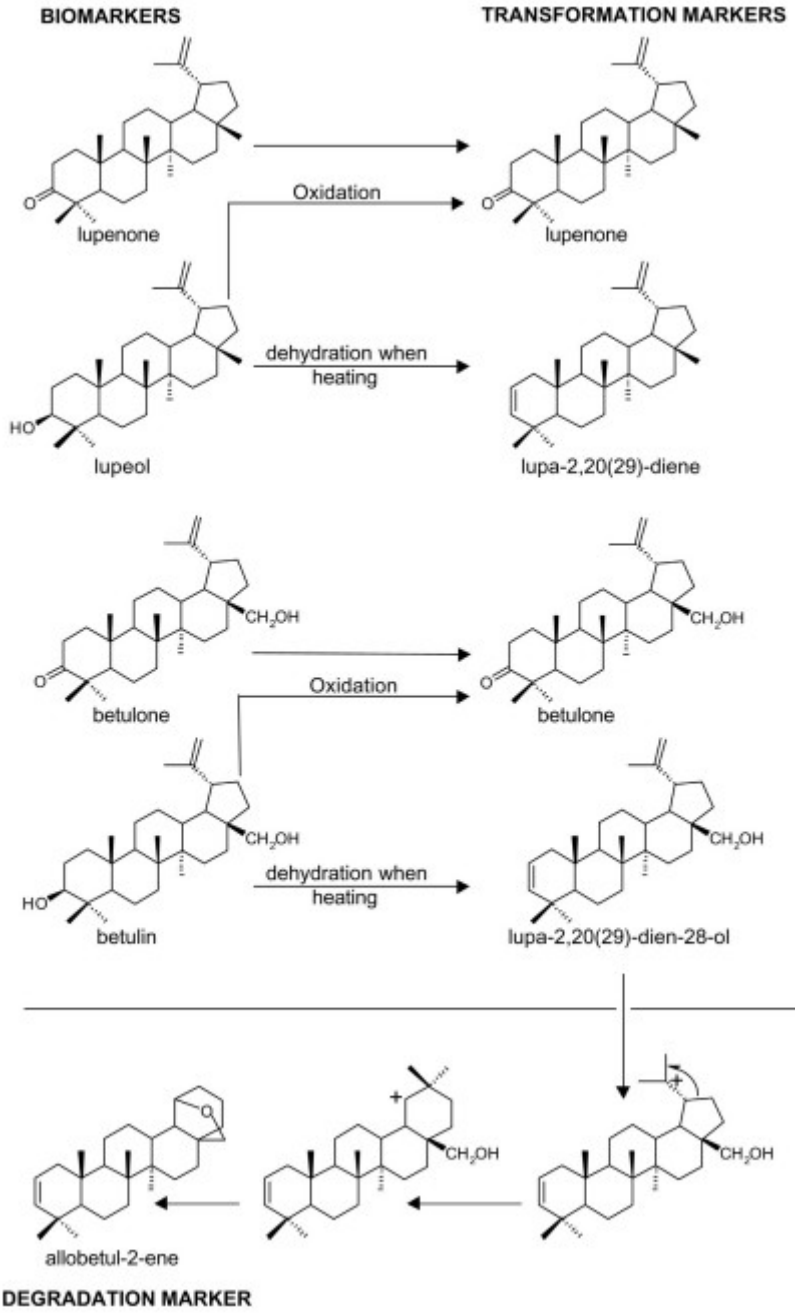

Fig 5.7. The transformation of various triterpenoid biomarkers through mechanisms such as oxidation and dehydration. Image after Regert 2004.

Some well-characterised examples include abietic acid, derived from conifer trees (Hjulström, Isaksson and Hennius, 2006), and birch-bark tar, derived from the pyrolysis of birch bark (*Betula pendula*) (Dudd and Evershed, 1999; Regert, 2004; Koch and Schmidt, 2021).

2.2.5 Dicarboxylic, oxo, hydroxy and dihydroxy acids

The incorporation of oxygen atoms into fatty acids can occur through a wide variety of functional groups and reaction mechanisms. Many of the end products are useful biomarkers of lipid degradation and oxidation, these include: α,ω-dicarboxylic acids; keto or oxo-acids; hydroxy acids and dihydroxy acids (Goossens *et al.*, 1986; Hansel and Evershed, 2009; Pozhidaev *et al.*, 2021; Koch *et al.*, 2022; Breu *et al.*, 2023(Fig 5.8).

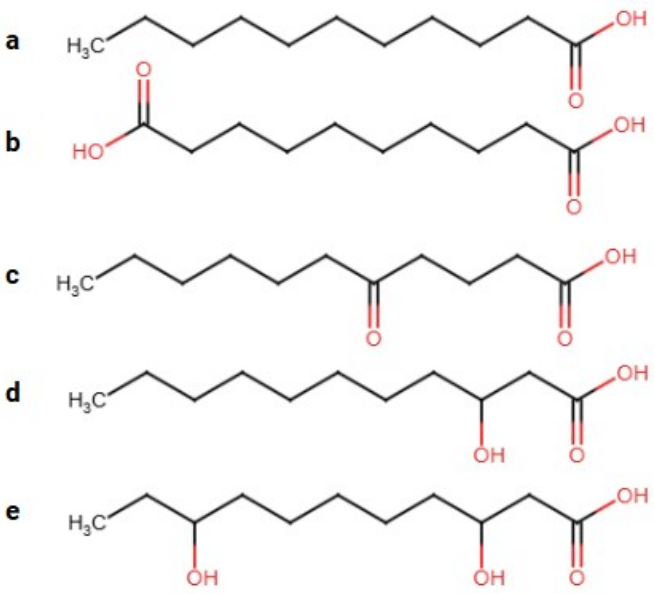

Fig 5.8. Structural depictions: (a) fatty acid (b) dicarboxylic acid (c) oxo acid (d) hydroxy acid (e) dihydroxy acid, the sections in red highlight oxygen bonds, by author

2.3 Lipid metabolism and function

Lipid metabolism varies widely between plants, mammals and fish, not to mention fungi and other biological phyla. As such the organic residue analyst must be familiar with the major mechanisms and functions of lipids in living organisms as specific metabolic

pathways may yield biochemical fingerprints related to topics as diverse as wine fermentation, fish oil rendering and plant resin processing (Baeten *et al.*, 2014; Lucquin *et al.*, 2016; Pecci *et al.*, 2020).

In general lipids perform a number of basic biological functions: energy storage, cell membrane formation, steroid and hormone synthesis, tissue structure, signalling molecules and heat preservation (Ridgway and McLeod, 2008; Bauman, Corl and Peterson, 2020). The typical form for saturated fatty acids is to be bound with glycerol into a triglyceride molecule. Triglycerides can be packed closely together into adipose cells and can be metabolised in a number of ways. The key enzyme involved in triglyceride metabolism is acetyl coenzyme A (acetyl-CoA) (Ohlrogge and Jaworski, 1997). Acetyl-CoA is converted to triglycerides during lipogenesis and is the final product during beta-oxidation of fatty acids (Shi and Tu, 2015). Free fatty acids are broken down through subsequent rounds of dehydrogenation, hydration, oxidation and thiolysis, until the chain has been fully broken into separate acetyl-CoA units (Russell and Martin, 2004).

Bacterial lipid synthesis is more unusual and results in odd-numbered branch-chained fatty acids, this is due to the bacterial use of β-ketoacyl-acyl carrier protein synthase III (FabH) and a combination of fatty acid synthase systems (Parsons and Rock, 2013) which yields a more diverse set of branch-chained lipids. Crucially, bacterial lipid metabolism is responsible for the conversion of dietary polyunsaturated fatty acids to saturated fatty acids within the rumen of animals such as cows (Polan, McNeill and Tove, 1964; Lourenço, Ramos-Morales and Wallace, 2010). A number of characteristic rumen microbiota are capable of PUFA *biohydrogenation*, which results in an increase in SFAs within the tissues of the animal (Conte *et al.*, 2022)(Fig 5.9). Ruminant animals are also capable of *de novo* fatty acid synthesis, as well as highly complex adaptations to different dietary PUFAs - involving lipolysis, isomerisation and the hydrogenation double-bonds (Noble, 1981; Jenkins, 1993; Buccioni *et al.*, 2012).

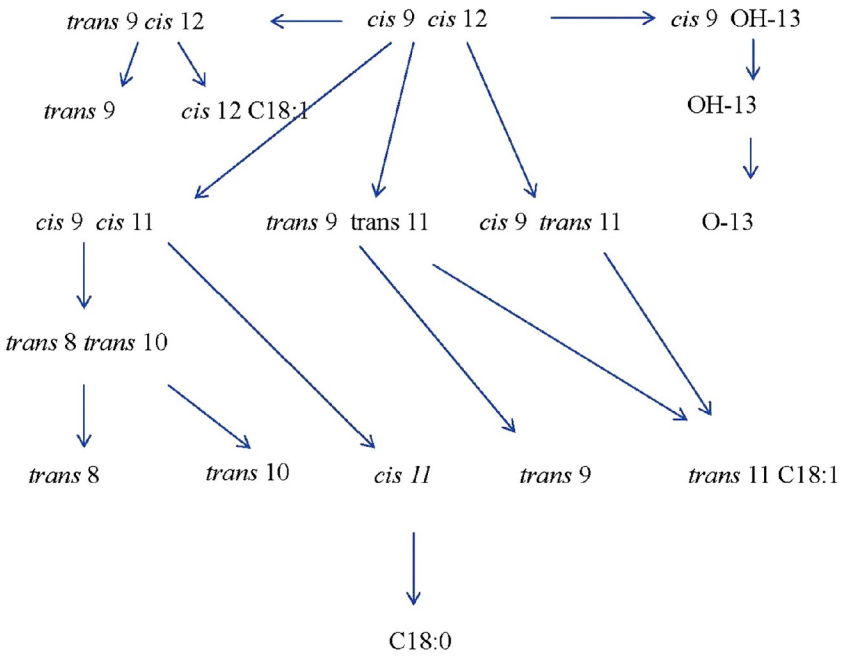

Fig 5.9. The biohydrogenation of linoleic acid. Image after Buccioni *et al*., 2012

Identifying the source of archaeologically recovered lipids also requires precise discrimination between the C^{12}/C^{13} ratio - the δC^{13}. Stable isotope analysis of the $C_{18:0}$ and $C_{16:0}$ fatty acids present in a sample can reveal the presence of ruminant or dairy fats due to the depletion of C^{13} in the animal's plant forage (Mottram *et al.*, 1999; Craig *et al.*, 2012). δC^{13} values of C_{18} fatty acids in particular can reveal dairy fats, since the mammary gland of the animal is unable to synthesis additional $C_{18:0}$ and therefore draws upon the pool of available fatty acids for desaturation and secretion, further lowering the δC^{13} values of preserved dairy fat residues (Evershed *et al.*, 2002; Bernard, Leroux and Chilliard, 2006, 2013; Glasser *et al.*, 2007).

Plant lipid metabolism is more complex and diverse than mammalian, and plants produce a wide variety of lipids for protection, structure and waterproofing, including suberin, cutin and various waxes (Ridgway and McLeod, 2008, p. 113). Plant waxes are a mixture of long-chain alkanes, aldehydes, ketones and esters, produced through specialised enzymes such as elongases (von Wettstein-Knowles, 1987). Plant energy storage lipids tend to be more unsaturated, although exceptions exist such as coconuts and palm oil. Triglyceride metabolism is therefore more complex and a variety of modification pathways exist. Rather than the more simple animal lipogenic method, plant triglycerides appear to pass through an inflection point where phosphatidylcholine is yielded which can then be desaturated or go on to form triglycerides (Shanklin and Cahoon, 1998; Ridgway and McLeod, 2008, p. 112).

Plant triglycerides are often further modified into a vast number of final forms. These include lauric acid (Ohlrogge and Jaworski, 1997), erucic acid (Ecke, Uzunova and Weißleder, 1995), vernolic acid (Liu, Hammond and Nikolau, 1998), ricinoleic acid (James, Hadaway and Webb, 1965), crepenynic acid (Haigh, Morris and James, 1968), sterculic acid (Castellucci and Griffin, 1960) and linolenic acid (Ohnishi and Yamada, 1980)).

2.4 Lipid degradation

Any lipid profile obtained from an archaeological source is unlikely to be fully representative of the original lipid constituents, since both microbial and chemical degradation/alteration are presumed to have occurred. Leaving aside the transformation of lipids *in situ*, such as when a cooking pot is re-heated (Raven *et al.*, 1997; Hansel *et al.*, 2004), the degradation of lipids diagenetically has been discussed extensively in the literature, in the hope of finding secondary biomarkers or understanding why certain products are missing ((Dudd, Regert and Evershed, 1998; Evershed, 2008a; Hansel, Bull and Evershed, 2011; Hammann *et al.*, 2018; Dunne *et al.*, 2019; Huber *et al.*, 2022). Burial conditions naturally affect the rate and type of degradation, with anoxic environments differing to oxic under laboratory conditions (Hammann and Cramp, 2018).

2.4.1 Lipid oxidation pathways

The oxidation of lipids can occur spontaneously by autooxidation, or through the specific mechanisms of photolysis or enzymatic degradation (Frankel, 1984; Schaich, 2005; Shahidi and Zhong, 2010). Increasing the temperature of a fatty acid will hugely increase the likelihood of a self-propagating free radical breakdown (Nawar, 1989) (Fig 5.10). This can affect both saturated and unsaturated lipids, but unsaturated are more prone to oxidation through a breaking of one or more double-bonds.

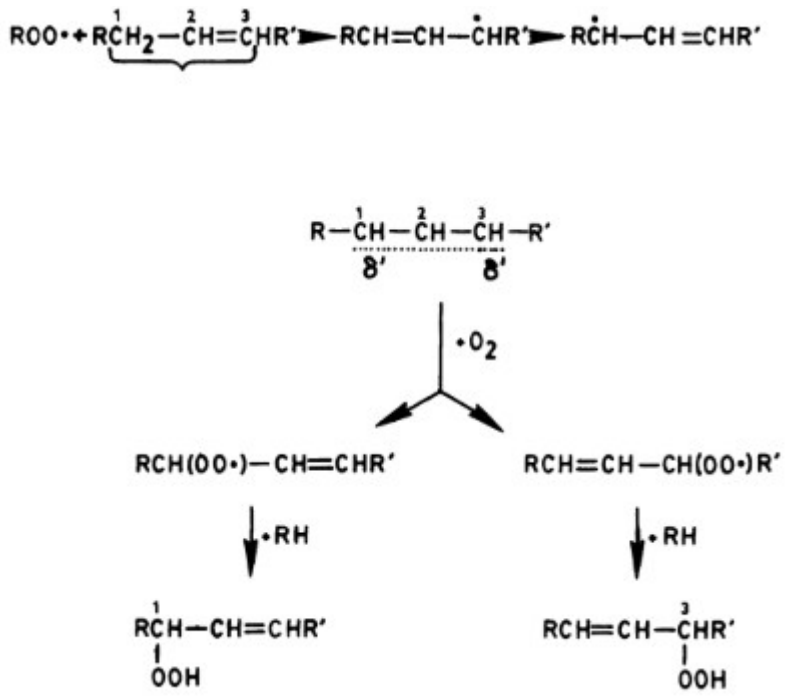

Fig 5.10. A generalised mechanism for the formation of hydroperoxides. Image after Paquette, Kupranycz and van de Voort, 1985

Under normal conditions atmospheric triplet oxygen would be unreactive in the presence of fatty acids. The formation of reactive oxygen species is required for the energetic barrier to be lowered enough for oxygen to abstract a hydrogen atom and generate an alkyl radical (Johnson and Decker, 2015). This in turn allows triplet oxygen to form a covalent bond and produce a peroxyl radical which prompts a chain reaction by abstracting hydrogen from a second lipid molecule to stabilise itself and produce a hydroperoxide. The second alkyl radical undergoes the same process until two radical species bond and bring the reaction to an end (Brodnitz, 1968). Hydroperoxides are the parent species to any secondary degradation products, which can include shorter-chain fatty acids or oxylipins such as keto acids and dicarboxylic acids as well as aldehydes, esters and alcohols (Gardner, 1989; Koch, Löwen and Schebb, 2024). Hydroperoxides are highly unstable and are very unlikely to survive long enough for detection. Epoxy species are also reactive due to the physical strain of the cyclic ether 3-atom ring, and often degrade further to diols under both acidic and basic conditions (Mubiru, 2018; Khor *et al.*, 2019; Koch *et al.*, 2023). The oxidation of saturated fats requires either metal catalysts or temperatures above 180C, since they are far more stable than unsaturated fats (Brodnitz, 1968; Nawar, 1989). Some studies indicate that thermal decomposition for saturated fatty acids primarily involves decarboxylation, yielding long-chain alkanes (Charuwat *et al.*, 2018), whilst others suggest

that dehydrogenation may occur, which would generate unsaturated fatty acids (Brodnitz, 1968).

Evershed et al (1992) identified hydrolysis and oxidation as two main pathways by which lipids could degrade. The hydrolysis of triacylglycerides accounts for why higher quantities of free fatty acids and di- and monoacylglycerols appear in residue studies compared to in nature. Inversely the proportion of unsaturated fatty acids is markedly lower under analysis, owing to the oxidation of the double bonds. Sterols such as cholesterol are also rarely found due to oxidation, a process which can be sped up from reactions within the ceramic matrix itself (Hammann *et al.*, 2018). The loss of unsaturated fatty acids is so great that secondary products had to be identified in order to infer their presence at all. These include ω-(o-alkylphenyl)alkanoic acids (Hansel *et al.*, 2004; Evershed *et al.*, 2008; Bondetti *et al.*, 2021), a stable molecule formed through the prolonged heating of poly- and monounsaturated fatty acids, and not typically found in nature. The oxidation of unsaturated fats can yield other byproducts, such as dihydroxy acids, compounds which prove the presence of monounsaturated alkanoic acids, only found in marine mammals (Hansel and Evershed, 2009); also α,ω-dicarboxylic acids and ω-hydroxycarboxylic acids, both of which are generated as oxidative products of unsaturated fats (Colombini, Modugno and Ribechini, 2005). These degradation/alteration products seem to be well preserved within the protective ceramic matrix, bound either through ionic/dipole interactions or ester linkages (Evershed *et al.*, 2002).

2.5 Key ORA biomarkers

2.5.1 Long-chain ketones

One molecule group of particular interest to residue analysts are long-chain ketones (> C29). These are thought to form under particular conditions, being a condensation reaction between two fatty acids, yielding a typically odd-chain saturated fat with the ketone group in the middle of the carbonyl chain. These long-chain products have been found in a wide variety of contexts, and are considered to be a reliable biomarker for thermal processing (Dudd, Evershed and Gibson, 1999; Copley *et al.*, 2005; Evershed *et al.*, 2008; Poulain *et al.*, 2016; Mayyas *et al.*, 2017). The formation of these ketones was investigated by Raven et

al (1997) who suggested that the pottery matrix and its metal salts were major catalysts, as well as temperatures upwards of 300 C. This has been further detailed by work looking at dicarboxylic acid involvement in long-chain ketone production (Breu *et al.*, 2023). It is generally accepted that the thermal degradation of free fatty acids and triacylglycerols in combination with the metal salt rich ceramic matrix prompts ketonic decarboxylation with three specific products being formed: 31K hexatriacontanone; 33K tritriacontanone and 35K pentatriacontanone. It is debatable whether their presence in a sample is reflective of the cooking environment and foodstuffs, or if they are retained artefacts from post-firing ceramic modifications where animal fats were used for their hydrophobic properties (Matlova *et al.*, 2017; Drieu, Lepère and Regert, 2020). More recently it has been shown that these long-chain ketones could potentially be formed through the heating action of a drill being used to sample archaeological potsherds, especially where the ceramic is rich in calcium carbonate (Longoni *et al.*, 2024).

2.5.2 APAAs

ω-(o-alkylphenyl)alkanoic acids, or APAAs, are a class of cyclic alkanoic acid formed through the heating of unsaturated fatty acids. Matikainen et al (1997, 2003) identified particular isomerisation and cyclisation reactions when C_{18} PUFA substrates were heated, including a 1,5 hydrogen shift. Continual heating at 260-270 C results in an intramolecular Diels-Alder cyclisation, forming six-membered rings (Fig 5.11). Further work by Hansel et al (2004) extended the knowledge of this process to include C_{16} and C_{20} PUFA homologues and a 1,7 hydrogen shift which would generate additional isomers for each species.

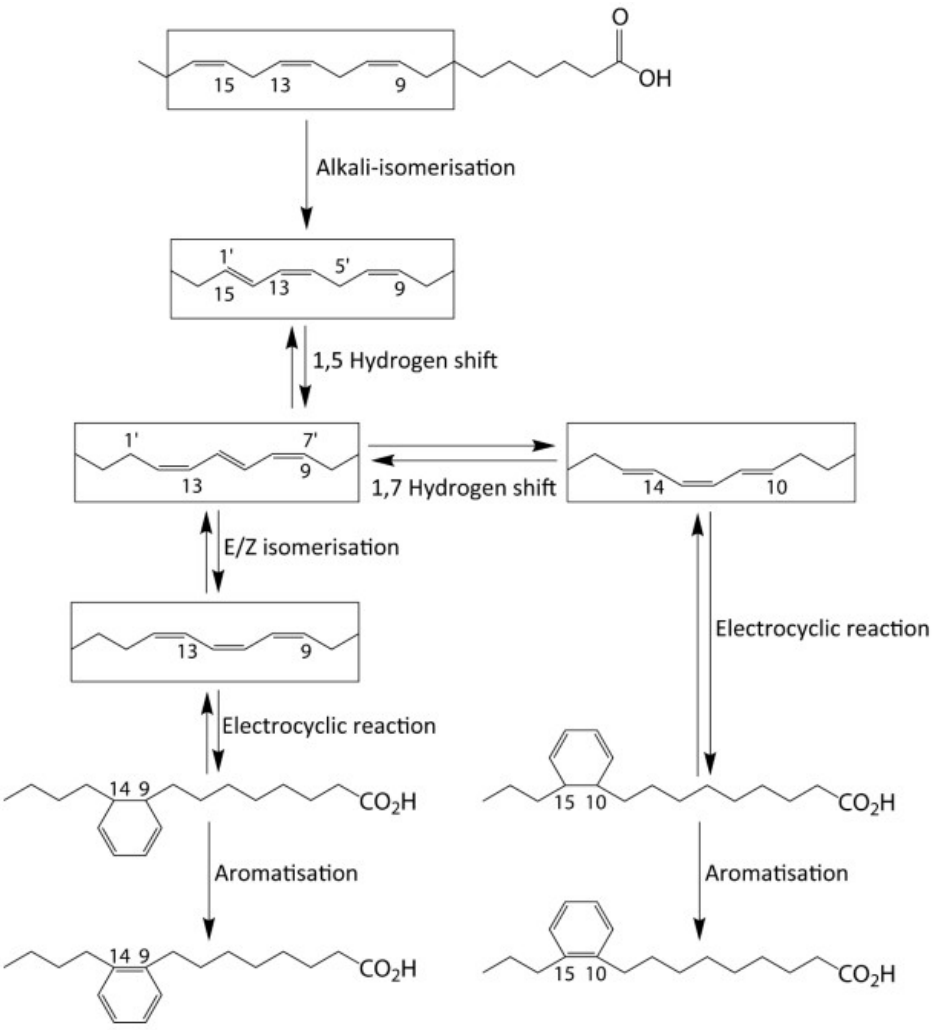

Fig 5.11. Schematic of APAA C_{18} formation from $C_{18:3}$ *cis* 9, *cis* 12, *cis* 18. Image after Hansel *et al*., 2004.

Furthermore, it has become possible to demonstrate a product-precursor relationship through the specific isomeric output from heating not only PUFAs, but mono and di-unsaturated FAs as well (Evershed *et al.*, 2008). Thus APAAs became synonymous with marine or aquatic product processing, by exploiting the identification of C_{18} and C_{20} APAA isomers, along with isoprenoids such as phytanic, pristanic and 4,8,12-trimethyltridecanoic acid (Cramp *et al.*, 2014; Admiraal *et al.*, 2019, 2020; Courel *et al.*, 2020; Dolbunova *et al.*, 2022). This has since been challenged, and the threshold of >0.06 from APAA-C_{20}/APAA-C_{18} present in a sample has been suggested to confirm the presence of aquatic products (Bondetti et al. 2021). More recently another class of cyclic alkanoic acids has been identified - ω-(2-alkylcyclopentyl)alkanoic acids (ACPAAs) which may form through a similar 1,5 and 1,6 hydrogen shift to form both cyclopentyl and cyclohexyl ring isomers (Breu *et al.*,

2023) (Fig 5.12). Little else is known about these alkanoic acids, and more research is needed to determine their origins and whether any similar isomeric distributions exist as for APAAs.

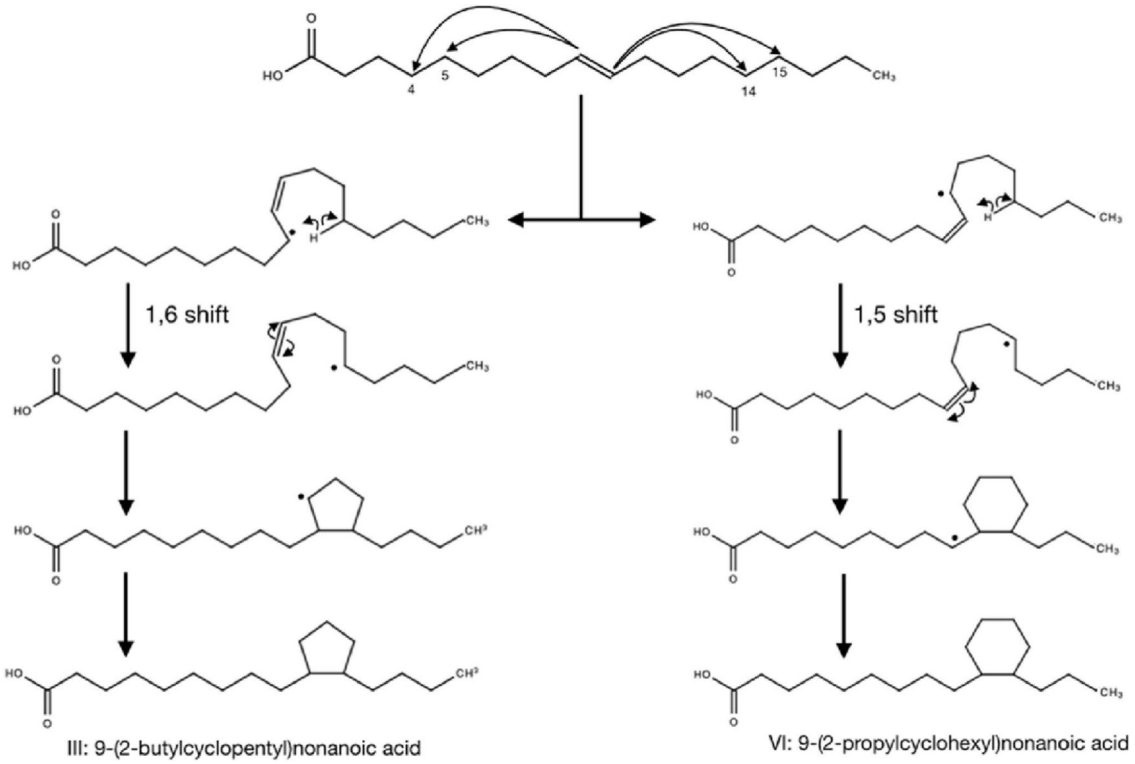

Fig 5.12. Possible mechanisms for the formation of ACPAAs from both *cis* and *trans* substrates. Image after Breu *et al*., 2023.

3.0 Organic residue analysis protocols and instrumentation

3.1 Lipid extraction

By their nature lipids are difficult to separate from the remainder of any biological tissue. Covalent modifications, as discussed above, can provide non-polar elements to the typically polar lipid species. Therefore high-quality extraction is the crucial first step in lipid analysis. The earliest protocol was developed in 1957, known as the Folch Method (Folch, Lees and Sloane Stanley, 1957). This uses a chloroform:methanol solution in a 2:1 ratio *v/v*, before washing out non-polar compounds with water, maintaining a strict 8:4:3 ratio for chloroform:methanol:water to avoid losing too many polar species (Iverson, Lang and Cooper, 2001). In 1959 a second modified variant was published, known as the Bligh and

Dyer Method (Bligh and Dyer, 1959). This follows a similar protocol but water is included from the start and the ratio is held at 2:2:1.8 (Smedes and Thomasen, 1996). Both methods made use of a solvent mixture with both polar and non-polar components, which is essential to extracting amphiphiles such as phosphatidylinositol. Choosing the correct solvent is crucial to the extraction process and a number of compounds have been tested over the years, including n-hexane (dos Santos *et al.*, 2015), butane (Xie *et al.*, 2017), acetone (Ren *et al.*, 2021), ethanol (González-Fernández *et al.*, 2020), isopropanol (Sarafian *et al.*, 2014) and 'green' variants such as ethyl acetate (Lin *et al.*, 2004) and cyclopentyl methyl ether (de Jesus *et al.*, 2019).

Several recent reviews into lipid extraction and analysis have highlighted the impressive longevity of both chloroform:methanol based methods - the Folch/Bligh & Dyer - and how most alternative extractive protocols are modified iterations on this basic step (Pati *et al.*, 2016; Saini *et al.*, 2021). In contrast to these however, the development of archaeological organic residue analysis has faced different challenges, the first of which is that conventional lipid extraction works with living, cellular based substrates. Prior to 2014 organic residue extraction often relied on the traditional chloroform:methanol protocol, but Correa-Ascencio & Evershed (2014) introduced a procedure called 'direct acidified methanol extraction'. The benefits of this method include: improved lipid recovery, simultaneous production of fatty acid methyl esters and a reduction in time spent on the process. A later comparative study of the Folch/Bligh & Dyer protocol against acidified methanol conclusively demonstrated that the acidified methanol protocol was superior at recovering bioarchaeologically important neutral molecules such as sterols, terpenes and alkanes (Reber, 2021).

The full range of lipid extraction methods in archaeological organic residue analysis has been reported and evaluated in several review papers (Whelton *et al.*, 2021; Irto *et al.*, 2022). The most common, aside from acidified methanol, include variations on a solvent extract, making use of chloroform or dichloromethane in 2:1 ratio with methanol (Evershed *et al.*, 1994; Evershed, Charters and Quye, 1995; Charters *et al.*, 1997; Regert *et al.*, 1998; Garnier *et al.*, 2002; Stott *et al.*, 2003; Copley *et al.*, 2005; Patalano, Zech and Roberts, 2020). Another variation includes the use of base alkaline treatments to disrupt the strong intermolecular interactions between non-polar functional groups found in some lipid species, including sodium hydroxide (Regert *et al.*, 1998) and potassium hydroxide (Pecci *et al.*, 2013). Finally some unusual methods have been trialled for further exploration such as

‘microwave-assisted extraction’ (Gregg and Slater, 2010; Blanco-Zubiaguirre *et al.*, 2018) and the use of non-destructive ‘supercritical fluids’, which could also increase the efficiency of lipid recovery (Devièse *et al.*, 2018).

3.2 Compound derivatisation

The range of functional groups within the possible lipids often extracted from ceramics is large, creating inconsistencies in polarity, volatility and stability (Halket, 1993; Schummer *et al.*, 2009). Therefore it is typical to derivatise the extract prior to chromatographic analysis. The utilisation of N,O-bis-(trimethylsilyl)trifluoroacetamide (BSTFA) + 1% trimethylchlorosilane (TMCS) to silylate the functional groups has been a standard technique since the 1970’s, both for amino acids (Gehrke and Leimer, no date1970) and lipids (Rosselló, Suñol and Gelpí, 1978), and has therefore been part of the typical organic residue protocol since its inception (Evershed, Heron and John Goad, 1990; Charters *et al.*, 1995; Dudd, Regert and Evershed, 1998). Silylation provides multiple benefits, including increasing volatility and reducing polar-polar interactions between alcohols, carboxylic acids and other molecule species (Irto *et al.*, 2022). Methylation, or the production of fatty acid methyl esters, is another common GC-MS derivatisation technique, aimed at increasing the volatility of free fatty acids and acylglycerides. Prior to the development of the acidified methanol protocol, organic residue analysts used a combination of saponification with a base and boron triflouride in methanol (Copley *et al.*, 2004; Romanus *et al.*, 2007). The advantage of Correa-Ascencio & Evershed’s method was to simplify this process, by simultaneously extracting and methylating any fatty acids, acylglycerols and wax esters present in the sample. Confirmation of the increased power provided by the direct acidified methanol method to recover lipids from even extremely small sample sizes (>0.1g) was demonstrated by Papakosta et al (2015).

3.3 Chromatography

Chromatography is based on the principle that chemical compounds can be separated from one another by passing a *mobile phase* through a *stationary phase* (Marriott and Shellie, 2002). In the case of gas chromatography, the mobile phase is an inert carrier gas and the stationary phase is a column, filled with different chemical species in order to

separate the compounds as they elute. These will then exit the column at different rates, a phenomenon called the *retention time* (Karasek and Clement, 2012). The analyte being studied will arrive at a partition equilibrium with the stationary phase species despite the fact that the two phases will also be in two different physical phases - gas and solid. It is crucial that the temperature of the column not exceed the boiling point of the analytes, but rather it allows the molecules to move through the column under their vapour pressure. Too much heat will prevent the analytes from interacting with the stationary phase. The retention of the analytes on the column can be measured using the *capacity factor* (K')

$$K' = \frac{(tr - t0)}{t0}$$

This equation shows the capacity factor to be equal to the *analyte retention time* subtracting the *void time* over the void time. The void time is the amount of time after the process is started before the elution of the analyte begins. Good peak separation or resolution requires that the retention time not be too minimal or too great. Related to this is the need for good efficiency, a measure of peak broadening. The peaks should correspond to different molecules, and when coupled with a mass spectrometer, can be readily identified.

3.4 Mass spectroscopy

A mass spectrometer ionises the target molecules and measures the output as a ratio of *mass* to *charge (m/z)*, which is presented as a spectrum. The y-axis uses *relative abundance* as the measurement of quantity or intensity, and the *m/z* for the x-axis. Neither correspond to any physical units: the y-axis is measured in arbitrary relative units and the x-axis is dimensionless, since the mass (daltons) divided by the charge (a single positive integer) yields nothing. Nevertheless, mass spectra are consistent in their output and present real information about the ions and parent molecules (Glish and Vachet, 2003; Awad, Khamis and El-Aneed, 2015).

What makes mass spectrometry a useful technique in the identification of unknown compounds is that molecules fragment in predictable, repeatable and consistent ways (Hufsky and Böcker, 2017; Qi and Volmer, 2017). As molecules are bombarded with electrons, bonds are broken in specific places, and with a sample size measuring into the

millions of individual molecules, the probability of a particular fragmentation pattern occurring can be mathematically calculated. A typical spectra shows the 'parent' or molecular ion at one end (Fig 5.13) and the characteristic fragmentation pattern displayed as peaks of abundance:

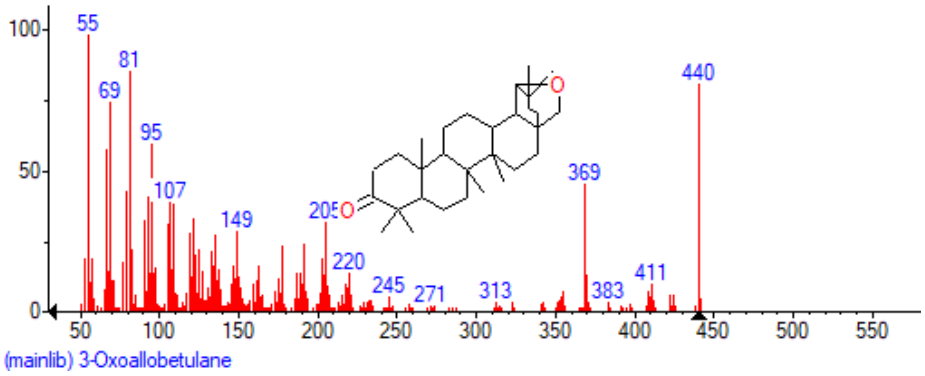

Fig 5.13. A mass spectra for 3-oxoallobetulane, by author

There are a number of specific mechanisms by which molecules are fragmented. It would be beyond the scope of this review to list them all, but one in particular is important for lipid analysis - the only named reaction: The McLafferty rearrangement, so called after Fred McLafferty who published the details of the reaction in 1959 (McLafferty, 1959). It involves the breaking of bonds between the alpha and beta carbon through a specific mechanism, namely that any carbonyl containing group will fragment with the gain of the gamma hydrogen. This is important for organic residue analysis because some crucial ion fragments are formed through this process, specifically *m/z* 74, which is the product of a McLafferty rearrangement on the methylated ester of a saturated fatty acid, such as stearic acid, resulting in the migration of the gamma-hydrogen to the carbo-methoxy group and cleavage between the alpha and beta carbons (Fig 5.14). The McLafferty ion is therefore a crucial diagnostic marker of saturated fats, and modifications to this ion can provide additional information about the target molecule.

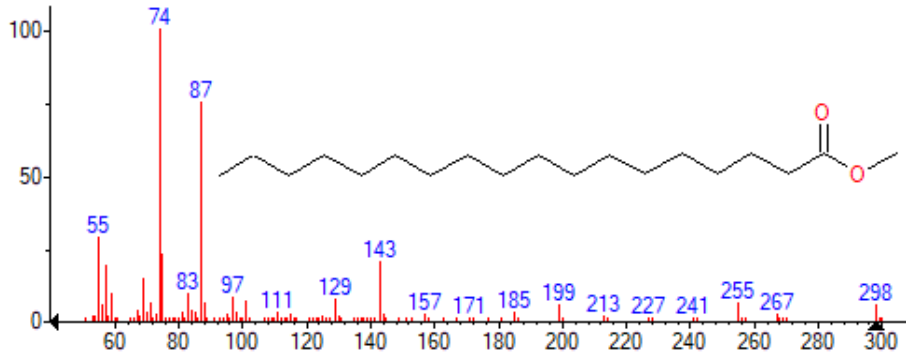

Fig 5.14. A mass spectra for methyl stearate showing the *m/z* 74 ion, image by author

4.0 Biochemistry of stone-cooking

Stone as a material is obviously of central importance to archaeologists, but stone as a material for cooking has too often been overlooked as a source of biochemical information. The analysis of FCRs can be broken down into: typological (Thoms, 2009; Gao *et al.*, 2014); functional (Tennis, Hunziker and Leach, 1997; Jackson, 1998; Brink and Dawe, 2003); use-alteration or wear-pattern analysis (Lovick, 1983; Neubauer, 2018) and biochemical (Quigg *et al.*, 2001; Buonasera, 2005; Short *et al.*, 2015; Short, 2018; Holst, 2023). Much research on FCRs has developed from a sustained focus on prehistoric pyrotechnologies and cuisine in several key locations, typically as a natural development of working on hearths, grindstones and food processing. By contrast, pottery function has become a more integrated field of study. As a result, trying to create a comprehensive overview of the ORA-FCR literature reveals several important locations where heated stones and grindstones are major topics of research, in particular the southeastern regions of the United States.

4.1 Previous archaeological work in organic residue analysis on stone

No publication or resource to date has collated all the research on organic residue analysis in stone. Therefore Table 5.1 has great value in summating all the English language literature on the topic. One clear outcome from this exercise is that organic residue analysis in stone has been attempted before, most often in the USA, as part of work to understand how prehistoric cultures made use of stones to grind and heat foodstuffs. The majority of the research tackles the subject of lipid preservation in groundstone artefacts and even static

rock features used as grinding stones. In particular the work of Malainey, Quigg and Buonasera shows a sustained interest in groundstone around California, New Mexico, Arizona and Texas. In the broader research topic of FCRs in cooking, the United States has also pioneered various techno-chronological models of 'hot-rock cookery', as well as use of actualistic experimentation and handheld Raman spectroscopy for non-destructive analysis (Jackson, 1998; Brink and Dawe, 2003; Thoms, 2008a, 2008b, 2009, 2023; Graesch *et al.*, 2014; Short *et al.*, 2015; Thoms and Boyd, 2015; Thoms *et al.*, 2015, 2018; Short, 2018). As concerns organic residue analysis of stone, several examples of sustained research exist.

In addition to the focus on the American south and southwest, research on the northeast coast has produced a large body of work into the origins and development of pottery, and how it provides advantages over traditional stone cooking methods. James Skibo has published extensively on the thermal properties of ceramics and included experimental work using boiling stones (Schiffer *et al.*, 1994; Skibo and Schiffer, 1995; Skibo and Blinman, 1999; Skibo, Malainey and Drake, 2009; Skibo, 2013, 2015; Skibo, Malainey and Kooiman, 2016; Hanson *et al.*, 2019). Since their focus was more on the technological comparisons of stone boiling versus ceramics, the chemistry of FCRs themselves was not analysed in any great detail, except to highlight monounsaturated fatty acids consistent with nut processing.

Pioneering work in organic residue analysis from hearth and some FCR samples has been conducted by the Rennes research group in France, focusing on combining experimental techniques with data from archaeological samples spanning the Middle Palaeolithic to the Iron Age (March, 1999; R. J. March and Soler Mayor, 1999; R. March and Soler Mayor, 1999; March and Lucquin, 2001; March, Largeau and Guenot, 2003; March *et al.*, 2006; A. Lucquin, 2007). Methodologically this involved utilising solvent extraction on large sample quantities (50-100g) and fractionating the fatty acids and neutral compounds.

Table 5.1. Publications presenting methods and results from ORA in stone.

Title	Year	Method	Instruments	Sample (g)	Summary	Citation
Einführung in die naturwissenschaftlichen Methoden in	1983	Modified Bligh & Dyer,	GC	2	ORA on an unknown number of limestone samples taken from a hearth context	Rottländer, 1983

der Archäologie						
Analysis of the Fatty Acid Compositions of Burned Rock Residues from Site 41ZP176, Zapata County, Texas	1999	/	/	/	/	Malainey 1999
Analysis of the Fatty Acid Composition of Burned Rocks and Groundstone Tool Residues from the Lino Site, 41 WB437, Webb County Texas	2000	/	/	/	/	Malainey 2000
No Bones About It: Using Lipid Analysis of Burned Rock and Groundstone Residues to Examine Late Archaic Subsistence Practices in South Texas	2001	Folch solvent extract, acid fraction (HCl), compositional ratios	Gas chromatography	20-40	ORA ~ 50 samples of FCRs, sandstone cobbles and five grinding stones	(Quigg *et al.*, 2001)
Unlocking the secrets of the stones: chemical methods to find tool usage in the Old World	2002	/	/	/	/	(Mclaren and Evans, 2002)
Gas chromatography /mass spectrometry analysis of organic residues in ceramic and ground stone	2003	/	/	/	/	(Burton, 2003)

artifacts from INY-1317 and INY- 1991.						
Fatty acid analysis of prehistoric burned rocks: a case study from central California	2005	Modified Bligh & Dyer, acid fraction (H2SO4), compositional ratios	Gas chromatography	20-50	ORA on 12 sandstone FCRs and several control samples	(Buonasera, 2005)
Les activités liées à l'utilisation du feu	2006	Modified Bligh & Dyer, acid & nuetral fraction	GC-MS	120	ORA on six hearth FCRs (sandstone, limestone, millstone)	(March et al, 2006)
Investigating the presence of ancient absorbed organic residues in groundstone using GC–MS and other analytical techniques: a residue study of several prehistoric milling tools from central California	2007	Modified Bligh & Dyer, acid fraction (H2SO4), compositional ratios, lipid concentration	GC-MS, UV spectroscopy	0.5-2	ORA/UV-Vis on eight grinding stones (sandstone, andesite, dacite)	(Buonasera, 2007)
Les activités réalisées en lien avec l'utilisation du feu. De la micro histoire à l'analyse générale des comportements	2007	Modified Bligh & Dyer, acid & nuetral fraction	GC-MS	120	ORA on 25 sandstone samples, including identified boiling and griddle stones	(March and Lucquin, 2007)
Étude physico-chimique des méthodes de cuisson pré et protohistoriques	2007	Folch solvent extract, McCarthy & Duthie 1962 fractionation	GC-MS	80-120g	ORA on FCRs from Magdalenian, Neolithic & Bronze Age contexts+experimental	(Lucquin, 2007)
"Stone	2009	Folch solvent	HT-GC, HT GC-MS	?	ORA on six potsherds and three	(Skibo, Malainey and

Boiling, Fire-Cracked Rock, and Nut Oil: Exploring the Origins of Pottery Making on Grand Island		extract, acid fraction (HCl), TMS fraction, compositional ratios			rocks - rhyolite, basalt and quartzite	Drake, 2009)
First results on thermally induced porosity in chlorite cooking vessels from Merv (Turkmenistan) and implications for the formation and preservation of archaeological lipid residues	2009	Folch solvent extract, lipid concentration, heating biomarkers	GC-MS	2-5	ORA and porosity on 18 soapstone vessels (LCK found)	(Namdar, Stacey and Simpson, 2009)
Extracting new information from old experiments: GC/MS analysis of organic residues in aged experimental grinding tools	2013	Microwave assisted extraction, acid fract (HCl), compositional ratios, lipid concentration, heating/degradation biomarkers	GC-MS	0.2-1.3	ORA on aged experimental grindstones (20+ years)	(Buonasera, 2013)
Lipid residues preserved in sheltered bedrock features at Gila Cliff Dwellings National Monument, New Mexico	2016	Modified Bligh & Dyer, acid fraction (HCl), lipid concentration, heating biomarkers, compositional ratios	GC-MS	0.1-2	ORA on bedrock used as a grinding surface	(Buonasera, 2016)
An experimental ethnoarchaeology and analytical approach to fire-related	2018	Folch solvent extract, McCarthy & Duthie 1962 fractionation, marine biomarkers/iso	GC-MS, GC-c-IRMS	?	ORA on soils and heated pebbles (isoprenoids found)	(García-Piquer *et al.*, 2018)

management strategies in a hunter–fisher–gatherer society from the southern tip of Tierra del Fuego (Argentina)		topes				
Residue analysis of grinding stones from Chalcolithic Gulpinar	2018	Solvent extract, compositional ratios	GC-MS	5-10	ORA on 28 grindstones (andesite, basalt, granite)	(Bamyaci, 2019)

4.2 Characterising previous work on ORA in stone

In order to highlight the evolution of methodology and interpretation within the ORA-FCR literature, three publications are presented below with more detailed analysis. The timeframe from 2001 to 2016 demonstrates the impact of ceramic-based ORA research and how those results have been utilised for FCRS.

4.2.1 Case study one: lipid analysis of burned rocks and groundstone residues from Late Archaic South Texas. Quigg et al., 2001.

One of the earliest comprehensive studies into the feasibility and use of ORA on FCRs was conducted by Quigg et al (2001), examining over 40 FCRs and five grindstones from a Late Archaic site in South Texas. The authors used between 20-40g of stone, cut from the parent FCR, and extracted any available lipids using the Folch method. A fraction was esterified with hydrochloric acid and analysed using gas-chromatography. The resulting FA peaks were identified using comparisons to external qualitative standards. It is unclear how these FAs were converted to percentages, presumably as a relative fraction of total peak area. Using identification criteria developed by Marchbanks (1989), Loy (1994) and Skibo (1992), the fatty acid ratios and relative concentrations were compared against a comprehensive lipid dataset of modern foodstuffs available in the American southwest, including mesquite beans and deer grease. One example of this is the Saturation Index,

which is calculated as SI = 1-[(C18:1 + C18:2)/C12:0+C14:0+C16:0+C18:0] (Loy, Ricklis and Collins, 1994). Experimental data quantifying FA changes through accelerated degradation were included, which provided an interpretative tool to understand the archaeological FA data. In their results they reported being able to discriminate between lipid profiles from large herbivores, from oily plants and potentially plants with very low lipid concentrations. Utilising percentages of MCFAs, C18:0 and C18:1 isomers, thresholds for *large herbivore; large herbivore with plant OR bone marrow; plant with large herbivore; beaver; fish OR corn; fish OR corn with plant* and *plant (except corn)* were established and samples assessed to fall within one of these ranges. What was missing from the analysis however, was a total lipid concentration, which makes it hard to assess the reliability of their results. The analytical framework is also lacking the concept of a 'biomarker', which in turns leaves the interpretation with only changes to fatty acid composition and ratio with which to make a judgement. The method of extraction and low quality resolution, using only GC rather than GC-MS, resulted in weaker overall conclusions.

4.2.2 Case study two: a PhD thesis examining prehistoric cooking using bioarchaeological methods. Lucquin, 2007.

Lucquin's 2007 PhD thesis made use of both experimental and archaeological data in order to better identify the specific methods involved in prehistoric cooking, with stone cooking being one of several analysed. The sample preparation involved much greater quantities of ground stone than any other publication (80-120g). These samples underwent solvent lipid extraction and acidic/neutral fractionation. Multiple cooking methods were tested, including stone boiling, stone griddles and earth ovens, using both animal and plant foods.

Sample code	Cooking technique	Food	Location	Analysis	Experimental context
GBF1	Grilled	Beef	Meat	Rock	Laboratory
GBF2	Grilled	Beef	Meat	Rock	Laboratory
GRMO	Grilled	Beef	Marrow	Rock	Realistic
GSAN	Grilled	Boar	Meat	Rock	Realistic
GAR	Grilled	Lamb	Meat	Rock	Realistic
GAS	Grilled	Lamb	Meat	Sediment	Realistic
GSR	Grilled	Salmon	Meat	Rock	Realistic
GSS	Grilled	Salmon	Meat	Sediment	Realistic
SBEPS	Boiled	Spinach	Leaf	Rock	Laboratory
SBEPR	Boiled	Spinach	Leaf	Rock	Laboratory
SBCL	Boiled	Carrot	Root	Rock	Laboratory
SBCT	Boiled	Carrot	Root	Rock	Realistic
SBBF	Boiled	Beef	Meat	Rock	Laboratory
SBOS	Boiled	Beef	Bone	Rock	Laboratory
FECR	Earth oven	Carrot	Root	Rock	Laboratory
FECS	Earth oven	Carrot	Root	Sediment	Laboratory
FEPC	Earth oven	Pork	Whole	Rock	Realistic

Fig 5.15. Table of experimental cooking sample for analysis, image after Lucquin 2007 and translated for use.

The results for the stone griddle/earth oven revealed that the high temperature of the stone will generate a number of molecular by-products, including small methyl-ketones, mid-chain ketones and gamma lactones, which were interpreted as thermal transformation biomarkers. Stone boiling however generated no specific by-products, but small quantities of gamma lactones were formed. In general there was thermal degradation of the unsaturated fatty acids which presented in a manner similar to diagenesis. Lipid concentration was much higher in the griddle stone than for the other methods. Three archaeological case studies were then undertaken:

Case study 1 took place at the site of Pincevent, France (Magdalenian). Both sediments and FCRs were sampled for analysis. In 8 samples FAMEs, alkanes, sterols (cholesterol, betas sitosterol and other derivatives), small methyl-ketones, mid-chain ketones and gamma lactones were detected. The conclusion was that both stone boiling and griddle use had been undertaken. The presence of animal fat was interpreted as originating from reindeer according to the fatty acid profile. Study 2 involved the sampling of ceramics, sediments and stones from three hearths at Lillemer, France (middle Neolithic). A similar range of products were detected and one structure was interpreted as a roasting or griddling feature. Finally several Bronze Age pits were sampled for case study 3, which yielded more plant products than the previous sites, but the overall interpretation was less clear. Overall the project demonstrated the applicability of ORA on FCRs, in particular the successful identification of thermal biomarkers, likely helped by the much larger sample quantities involved. The identification of ketones, gamma-lactones and the breakdown of unsaturated fatty acids through thermal degradation provides some testable data for this project. The limitations of the analysis are a function of time, as extraction methods, instrumentation protocols and biomarker characterisation have advanced since 2007.

4.2.3 Case study three: organic residue analysis of sheltered bedrock features at Gila Cliff Dwellings, New Mexico. Buonasera, 2016.

One of the most recent ORA papers dealing with stones, looked specifically at rock surfaces within caves in the Gila Cliff Dwellings National Monument (Buonasera, 2016). Several of these surfaces presented with natural mortar depressions, anthropogenic grinding platforms and smaller cupola impressions containing blackened residues. Samples were taken using a drill and corer, and weighed between 0.1 and 1.4 grams. These were extracted using a modified Bligh and Dyer method and the neutral fraction esterified using hydrochloric acid and methanol. The samples were run on GC-MS and the peaks identified using commercial software. Unlike Quigg et al 2001, the author used an internal standard and was able to quantify the lipid concentrations, which ranged from 2 < μg g^{-1} to 278 μg g^{-1}. Alongside SFAs - diacids and 2-hydroxy acids were identified, as well as monocyclic aromatic compounds. Along with the absence of the maize biomarker - *n*-dotriacontanol - the author was able to argue for the use of *in situ* stone features as grinding surfaces for heated sunflower seeds, relying on Hansel & Evershed (2009) for confirmation that 9, 10-dihydroxy octadecanoic acid isomers are often produced through thermal degradation.

It is clear from these three examples that ORA in stone has advanced considerably over the past two decades, incorporating more rigorous methodologies such as mass spectrum instrumentation and the use of quantification and internal standards; improved recovery of a wide range of polar and neutral molecules, and reliance on a growing body of literature concerned with ORA in archaeological and experimental ceramics. What has not been tried yet is ORA in stone making use of the improved one-step acidified-methanol extraction technique, selective screening for heating biomarkers such as APAAs and a multi-instrumental approach of GC-FID, GC-MS and GC-c-IRMS.

4.2.4 Compositional Ratios

Much of the early ORA work in stone which came out of the United States relied on an interpretative method which dates back to the late 1980's. Namely that the fatty acids which composed an archaeological sample could be assessed through their relative abundance to one another and would fall into a particular category based on experimental lipid analysis of modern foodstuffs. The analytical justification for this was as follows: proxies for ancient foodstuffs could be sampled and their fatty acid content analysed, with enough samples and repetitions 'clusters' of food types could be identified through ratios and the absence/presence of particular fatty acids. Archaeological ceramic or stone samples could be compared against these clusters to provide a reasonable interpretation of their archaeological contents.

The origins of this method lie with Marchbanks (1989), who proposed the use of the following formula to identify uncooked foods in samples: %S = [C12:0 + C14:0] / [C12:0 + C14:0 + C18:2 + C18:3]. This was modified by Skibo (1992) who found that it failed to perform well when testing ceramic samples from the Philippines. It was noted that decomposition was a major factor in altering the initial fatty acid composition, and actualistic experiments were performed to assess oxidative degradation, in particular to unsaturated fatty acids. (Patrick, de Konig and Smith (1985). These findings led to Malainey, Przybylski and Sherri sampling hundreds of 'Native foods', including wild plants, berries, roots, fish, grouse and ruminant animals, in order to examine whether common patterns of fatty acid content would emerge (Malainey, Przybylski and Sherriff, 1999b). They also examined

modern foodstuffs such as bison meat and bone marrow after simulating depositional oxidation and degradation (Malainey, Przybylski and Sherriff, 1999a).

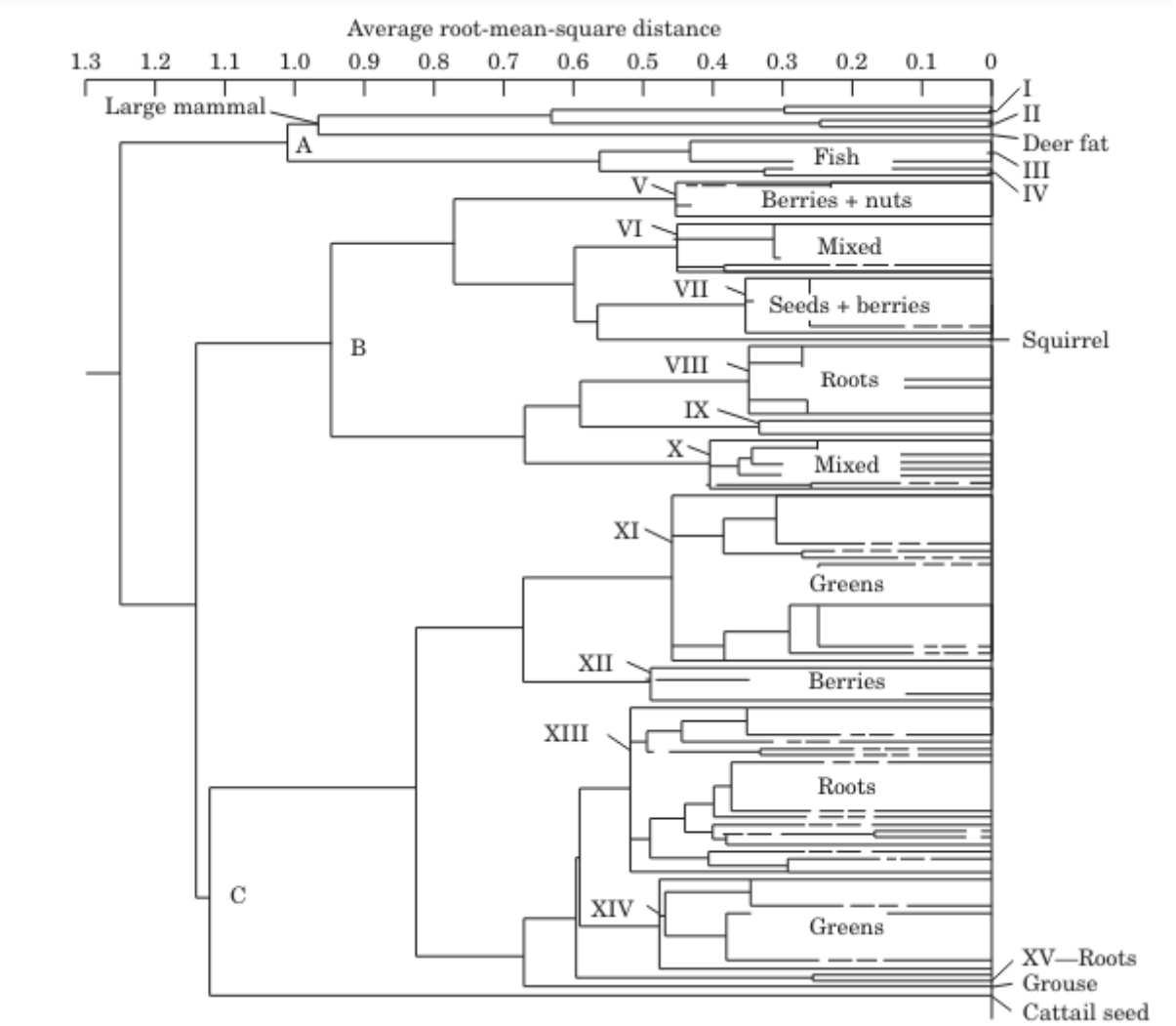

Table 1. Summary of average fatty acid compositions of food sample clusters

Cluster	A						B				C				
Subcluster	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	XIII	XIV	XV
Type	Mammal fat and marrow	Large herbivore meat	Fish	Fish	Berries and nuts	Mixed	Seeds and berries	Roots	Seeds	Mixed	Greens	Berries	Roots	Greens	Roots
C16:0	19·90	19·39	16·07	14·10	3·73	12·06	7·48	19·98	7·52	10·33	18·71	3·47	22·68	24·19	18·71
C18:0	7·06	20·35	3·87	2·78	1·73	2·36	2·58	2·59	3·55	2·43	2·48	1·34	3·15	3·66	5·94
C18:1	56·77	35·79	18·28	31·96	54·00	35·29	29·12	6·55	10·02	15·62	5·03	14·95	12·12	4·05	3·34
C18:2	7·01	8·93	2·91	4·04	37·85	35·83	54·69	48·74	64·14	39·24	18·82	29·08	26·24	16·15	15·61
C18:3	0·68	2·61	4·39	3·83	0·93	3·66	1·51	7·24	5·49	19·77	35·08	39·75	9·64	17·88	3·42
VLCS	0·16	0·32	0·23	0·15	0·71	4·46	2·98	8·50	5·19	3·73	6·77	9·10	15·32	18·68	43·36
VLCP	0·18	4·13	39·27	23·24	0·02	0·64	0·42	0·39	0·54	0·88	0·22	0·14	0·30	0·18	0·40

VLCS – Very Long Chain (C20, C22 and C24) Saturated Fatty Acids.
VLCP – Very Long Chain (C20, C22 and C24) Polyunsaturated Fatty Acids.

Fig 5.16. Hierarchical cluster analysis and the fatty acid composition of the identified food sample clusters. After Malainey *et al*., 1999.

These 'compositional ratios' were utilised in later ORA projects assessing FCRs, including the comprehensive study by Quigg et al (2001), where over 40 FCRs were sampled from a Late Archaic site in south Texas.

Criticisms of this approach came primarily from Buonasera (2005; 2007), where she highlighted the inability of the selection criteria to accurately account for food mixing, as well as providing demonstrably false positives. Buonasera noted that future ORA in rocks would need to account for background lipids already present in stone, and to make use of the more refined and targeted biomarker methods, drawing on European ORA researchers such as Evershed (1990) and Heron (1991). Buonasera later dropped the compositional ratio approach and made use of GC-MS instruments rather than just GC, in order to better quantify the total lipid content in stone and target specific compounds such as 9,10 dihydroxy octadecanoic acid.

4.4 Types of stone used in cooking

The selection of stone type used for cooking and heating may have an impact on the resulting organic residues and traces, and balances expediency with function from the point of view of the user. Ethnographic studies of the use and disposal of boiling stones reveals a

careful selection criteria based on porosity and visible discontinuities within the structure that might crack when subject to thermal shock (Batdorf, 1990; Jackson, 1998; Shantry, 2020). Archaeologically and experimentally the study of boiling stones confirms that particular characteristics were sought after in order to maximise the efficiency of the cooking process (Wilson and DeLyria, 1999; Strong and Croes, 2001; Neubauer, 2018). Shantry lists a number of boiling stone species used around Puget Sound, North America, including andesite, granite, basalt, dacite, gneiss, quartzite and schist (2020). Basalt was also favoured by Polynesian and Micronesian peoples for use in earth ovens (Piazza, 1998; Carson, 2002) and potentially selected for use as boiling stones at Göbekli Tepe (Dietrich *et al.*, 2020), Limestone is an interesting case where the chemical properties of the stone may have been intentionally exploited for its effects on the food - experimental work based on archaeological finds at Cedar Mesa, Utah, revealed that the alkaline environment produced by heating limestone in water made it possible to nixtamalize maize (Ellwood *et al.*, 2013). Analysis of the stone used at the Albertan site of 'Head-Smashed-In-Buffalo Jump' revealed that, despite the estimated weight of the total amount of FCR being in excess of 3 million kilograms, the majority of it was non-local, having been moved several kilometres (Brink and Dawe, 2003). The necessity of transporting large quantities of rock for cooking underlines the importance of rock 'type' to the users, with qualities relating to longevity and re-usability seemingly of high priority.

4.5 Background lipids in stone

Of the papers published concerning organic residue analysis on stone, only one directly addresses the question of background lipid presence in stone *prior* to its cultural use as a cooking aide (Buonasera, 2005). Here several control samples of sandstone contained enough lipids in type and quantity to plot as food, based on the compositional ratios outlined above. Similar concerns for background lipids in ceramic vessels have been addressed through experimentation (Reber *et al.*, 2019; Admiraal *et al.*, 2020), but little such work has been conducted for FCRs and stones. What is known about background lipids in stone comes from geological and biological subfields concerned with fossil fuel generation (Hunt, 1967; Utting and Wielens, 1992), microbial ecosystems (Blyth *et al.*, 2011) and even the damage to cultural stone artefacts such as statues (Scheerer, Ortega-Morales and Gaylarde, 2009; Mihajlovski *et al.*, 2015). Microbial communities which can live inside stones are called *endolithic*, and many studies have identified their presence even in the most extreme of

environments (Ziolkowski *et al.*, 2013; Wierzchos *et al.*, 2018). The lipid composition of stone objects such as stalagmites can be extensive, including *n*-alkanes from C14-C35, branched chain fatty acids, saturated, mono and diunsaturated fatty acids, sterols and hydroxy acids (Blyth *et al.*, 2011; Wang *et al.*, 2012). There is no precise method to estimate which kind of rock is likely to contain background lipids, and it is therefore essential to future organic residue projects involving FCRs to include control samples where possible, and potentially to construct databases of background lipid levels in different types of stone and locations. Without these control samples, there is a small but significant chance of producing false positives when analysing assemblages of FCRs and hearth-associated stones. Moreover, the presence of background lipids increases the noise-to-signal ratio, which could be detrimental when attempting to interpret the archaeological meaning of particular lipid or small molecule residues.

5.0 Conclusions

The advancement of GC-MS instrumentation and analysis has given archaeological researchers a powerful tool for interpreting past culinary methods and foodways. With the development of the biomarker concept and more efficient lipid extraction and derivitisation techniques the field of organic residue analysis has been able to offer great insight into the origins and diverse uses of archaeological pottery. However the application of residue analysis to heated stones and FCRs has been more limited. A fresh focus on organic residue extraction and analysis on FCRs would likely prove productive, utilising acidified-methanol and selective scans for crucial biomarkers to ensure that culturally modified stones can be distinguished from background lipid content.

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Chapter Six: Experimental evidence for heating biomarkers in archaeological fire-cracked rocks: potential for identifying aceramic cooking practices

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1.0 Abstract

Fire-cracked rocks (FCRs) are one of the most ubiquitous artefacts found on prehistoric archaeological sites. Their presumed mixed functionality has always included cooking and the preparation of foodstuffs, in keeping with the ethnographic record. However, with a few exceptions, the field of organic residue analysis has largely overlooked FCRs and remained focused on ceramic vessels. The result of this has been a revolution in our understanding about the origins, spread and the diverse uses of ancient pottery, but the limitations of which are apparent when assessing any pre or non-ceramic using cultures and time periods. Here we present results from three experiments aimed at characterising the formation of key heating biomarkers in stone, in order to incorporate FCRs into the increasingly sophisticated analytical methodologies of contemporary organic residue

analysis. Our results demonstrate that several robust biomarkers, such as *ω*-(*o*-alkylphenyl)alkanoic (APAAs) and benzene polycarboxylic acids, can form in stone, and that both quantitative and qualitative differences appear to exist between ceramics and stone. This has implications for any future organic residue work undertaken on archaeological FCRs, and for our understanding of the catalytic reactions which underpin the formation of APAAs, long-chain ketones and other key archaeological biomarkers.

2.0 Introduction

How humans learnt to cook, and how culinary methods and technologies evolved is a fundamental question in archaeology (Wrangham, 2017; Magargal, 2022). One approach to answering this has been to extract and analyse lipids and other biomolecules that have become trapped in the matrix of prehistoric pottery. Organic residue analysis has been instrumental to the study of these early ceramics, both in interpreting the function and diversification of pottery use (Cramp *et al.*, 2014; Shoda *et al.*, 2020; Bondetti, 2021; Hammann *et al.*, 2022), but also in providing empirical data to theoretical models explaining why hunter-gatherers adopted ceramics in the first place (Taché and Craig, 2015; Lucquin *et al.*, 2016; Robson *et al.*, 2019; Craig, 2021). The other side of this technological divide however, remains a mystery - how did people cook *before* ceramics? Despite experimental work aimed at characterising aceramic cooking methods (Ryder, 1966, 1969; Holman and Egan, 1985; Skibo, Malainey and Drake, 2009; Ellwood *et al.*, 2013; Graesch *et al.*, 2014; Hanson *et al.*, 2019; Lejay *et al.*, 2019; Langley *et al.*, 2023), there is a great gap in the biomolecular data available for study.

Functional interpretations of archaeological pottery have increasingly turned to organic residue analysis since the 1990's to extract and analyse lipids from both retained food crusts and the ceramic matrix (Evershed, Charters and Quye, 1995; Dudd, Evershed and Gibson, 1999; Stott *et al.*, 2003; Copley *et al.*, 2005; Boudin, Van Strydonck and Crombé, 2009; Steele, 2013; Philippsen and Meadows, 2014; Leclerc *et al.*, 2018; Liu *et al.*, 2023). With the development of the biomarker research framework (Evershed, 2008), advances in extraction methodology such as acidified-methanol (Correa-Ascencio and Evershed, 2014), single-ion monitoring (Admiraal *et al.*, 2019) and detailed studies of molecular species (Bondetti *et al.*, 2021; Breu *et al.*, 2023), it has been possible to track and

study major dietary changes in various parts of the world (Horiuchi *et al.*, 2015; Nishida, Lundy and Jordan, 2016; Chaile *et al.*, 2018; Leclerc *et al.*, 2018; Cramp *et al.*, 2019; Admiraal *et al.*, 2020; Taché *et al.*, 2021). However, this methodology is currently limited to areas and times where ceramics were available for cooking. As such, there exists significant gaps in our knowledge of diet and cooking both prior to the invention and adoption of ceramics, and in places where ceramics did not reach or were actively rejected (Elliott *et al.*, 2020; Piezonka, 2021). Even where ceramics do exist in the record, there are often discrepancies between the faunal assemblages, bone isotope results and biomolecular interpretations of preserved lipids in potsherds, suggesting that ceramics was simply one of many available cooking and processing strategies (Mukherjee, Gibson and Evershed, 2008; Dunne *et al.*, 2019; Robson *et al.*, 2019). Plentiful evidence exists for prehistoric structured hearths and FCRs, even deep into the Pleistocene (Movius, 1966; Karkanas *et al.*, 2004; Henry, 2017), but we are overlooking this ubiquitous reservoir of biomolecular data. Given that it is possible to extract lipids from archaeological FCRs, they offer great potential in answering many fundamental questions about the evolution of cooking, diet and cuisine, and human adaptations to climate change around the world.

2.1 Previous organic residue analysis on stone

Stone such as sandstone and granite are widely known in the ethnographic and archaeological literature as a source of indirect heat used as cooking implements (Driver and Massey, 1957; Thoms, 2008; Nelson, 2010), typically in one of three ways: (i) as griddles; (ii) as a hearth-oven; or (iii) as a source of indirect heat, such as heated stones added to another container. Some work has been done on building chronological and regional models for the development of stone-cooking methods, where simple hearths lead to more complex pit or oven structures, and the selective use of stones for boiling (Skibo, Malainey and Drake, 2009; Thoms, 2009; Shantry, 2020). FCRs interpreted as boiling stones appear more frequently towards the end of the Upper Palaeolithic, often associated with marrow extraction and grease production (Svoboda, 1990; Stiner, 2002; Nakazawa *et al.*, 2009; Gao *et al.*, 2014). Whilst there has been a steady advance in our understanding of how heat affects stone, and classifying the diagnostic markers of thermal use-wear (Lovick, 1983; Jackson, 1998; Brink and Dawe, 2003; March *et al.*, 2014; Neubauer, 2018; Needham *et al.*, 2022), there has been little equivalent development in the use of stone for organic residue analysis. Fourteen publications (Table 5.1 Chapter 5) over the past few decades have

detailed results based wholly or in part on organic residue analysis using FCRs, the majority from the southeastern United States (Quigg *et al.*, 2001; Buonasera, 2005, 2007, 2016). Methodologically the research makes use of the advances in ceramic-derived ORA, with a wide variation in sample sizes (0.1-120g of ground stone) and several key biomarkers identified, including long-chain ketones (Namdar, Stacey and Simpson, 2009) and cyclic alkanoic acids (García-Piquer *et al.*, 2018). However, despite this clear progression, stone is not a major material of ORA research interest and remains beholden to developments in ceramic lipid extraction. As such, we know relatively little about how stone compares and differs to ceramic in absorption, retention and transformation of lipids and other biomolecules.

2.2 Hot-rock cookery systems

An important factor to consider when assessing aceramic cooking methods is the overall system: how the heat is generated; how it is directed into the foodstuffs; how much moisture is included; how long it will take; and how high does the temperature need to be, to name but a few elements. Literature on stone-cooking technologies describes in detail how rock-cookery systems are manufactured to meet the needs of the particular foodstuffs; e.g., for the gelatinisation of starch (Perry and Michael Quigg, 2011); the detoxification of rhizomes (Wandsnider, 1997); the rendering of fats from nuts (Sassaman and Bartz, 2022) and bones (Brink and Dawe, 2003) - each with very different needs and requirements. Whilst awareness of this has led to the development of thermal use-wear analysis in FCRs, it has had comparatively little impact on ORA studies.

Following Speth (2015), Langley *et al*. (2023) demonstrated a clear distinction between *'boiling'* water and *'long time, low heat'* cooking in water (45 - 70 °C), which results in cultural choices being made to select one or the other for the desired outcome. For example, the leaching of tannins from acorn meat works far better at sub-boiling temperatures, whilst boiling causes the tannins to bind irreversibly to the nutmeat (Hanson *et al.*, 2019). Here we should extend these categorical distinctions to include *'short time, high direct heat'* for griddling and *'long time, low indirect heat'* for pit-cooking. Griddling is akin to frying in its action. The process of frying involves the interaction of hot oils with the water from the food, creating a thermal gradient from the edge to the interior (Hosseini *et al.*, 2016). Experimental work on different cooking methods has highlighted the greater loss of

fatty acids during frying compared to boiling or roasting, through the migration of fats from the food into the pan (Gere, 1982; Zhang *et al.*, 2013). With stone griddling we have two major interaction zones - the crust of the food where it comes into contact with the hot stone, and the matrix of the stone as the fatty acids migrate out of the food; in part pushed by the expanding water and air trapped inside (Dobarganes and Márquez-Ruiz, 2000). In contrast, pit-cooking creates a more sealed system. Hot stones and embers provide the heat at the bottom of the pit, followed by layers of plant matter, bark or skins above and below a layer of food, creating an envelope which keeps the food clean and moist - earth caps the pit, offering a very different physical environment to compare with the open-air stone griddle (Black and Thorns, 2014; Thoms *et al.*, 2015). The reduced ability for volatile compounds to escape, the reduced levels of oxygen and the circulation of water vapour along with fats and proteins from the food and surrounding plant species creates a semi-sealed steam oven somewhat akin to a *bain-marie* in a modern oven. Both of these hot-stone systems differ from typical ceramic cooking methods, since neither involve simmering or boiling in an enclosed container. With so much inherent variation it is important to evaluate whether FCRs from multiple hot rock cooking systems are capable of producing and retaining key biomarkers that can securely demonstrate the anthropogenic transformation of lipids (i.e., by-products of heat).

2.3 Key heating biomarkers - long-chain ketones and APAAs

The biomarker concept bridges the gap between nature and culture by linking a specific compound or ratio of compounds to an anthropogenic activity, like thermal alteration (Evershed, 2008; Baeten *et al.*, 2013). Both FCRs and ceramics often contain naturally occurring lipid products (Buonasera, 2005), although with the thermal transformation of clay to a stable ceramic matrix, much of these background molecules are removed (Reber *et al.*, 2019; M. Admiraal *et al.*, 2020). Unlike pottery, which is a plastic and synthetic product, stones used for cooking can simply be selected from the landscape with little to no modification. It is therefore crucial to ensure that any results from ORA on stone are not false positives. Some of the most robust biomarkers for human activity are those which are derived from thermal transformation, in particular those such as cyclic alkanoic acids, which do not occur naturally. These include APAAs (Hansel *et al.*, 2004; Evershed *et al.*, 2008; Admiraal *et al.*, 2019; Bondetti *et al.*, 2021), ACPAAs (Breu *et al.*, 2023), LCKs (Dudd, Evershed and Gibson, 1999; Copley *et al.*, 2005; Evershed *et al.*, 2008; Poulain *et al.*, 2016;

Mayyas *et al.*, 2017), VLC-oxo fatty acids (Breu *et al.*, 2023), BPCAs (Wiedemeier, Brodowski and Wiesenberg, 2015; Vaezzadeh, Zhong and Zhang, 2023) , and to a lesser extent gamma-lactones (Watanabe and Sato, 1970; Beeston *et al.*, 2006) and certain amides (Lejay *et al.*, 2019).

Long-chain ketones (> $C_{29:0}$) form through ketonic decarboxylation between two fatty acids. The formation of these ketones was investigated by Raven et al. (1997) who suggested that the pottery matrix and its metal salts were major catalysts, as well as temperatures upwards of 300 °C. Typically three specific products are formed within heated ceramics in a 1:2:1 ratio: 31K hexatriacontanone; 33K tritriacontanone and 35K pentatriacontanone. Very few studies have examined whether long-chain ketones can be formed within stone, or within different types of stone. Both Lucquin (2007) and Namdar et al. (2009) reported identifying LCKs within stone samples. In the first case this may be because the sample sizes were much larger than typically used for ceramics; in the latter possibly because the soapstone vessels were subject to multiple uses - given that FCR cooking may involve less cooking time than the continuous and repeated cooking involved with ceramics/stone vessels, LCKs may fail to form due to these improper conditions.

Alongside LCKs another recent long-chain ketonic biomarker has been identified - very long-chain oxo fatty acids (Breu *et al.*, 2023). These derive from saturated fatty acids and dicarboxylic acids, which can undergo a decarboxylation reaction producing an oxo-acid with a >$C_{22:0}$ chain length. Of particular note with this biomarker is the requirement for large amounts of dicarboxylic acids to be present prior to their thermal transformation, which the authors posit likely requires two rounds of heating - one to degrade unsaturated fatty acids, yielding the dicarboxylic acids, and the second to form the VLC-oxo fatty acids.

The presence of *ω*-(*o*-alkylphenyl)alkanoic acids (APAAs) in a sample provides confirmation that UFAs must have been present at some point during thermal treatment, since they are formed from the isomerisation and aromatisation of UFAs heated to at least 200 C for over an hour (Heron *et al.*, 2010; Craig *et al.*, 2013; Bondetti *et al.*, 2021). APAA precursor FAs sit within the range of C_{16}-C_{24} where one or more double bonds are present, which undergo several stages of isomerisation, hydrogen shifts and aromatisation, the final isomer depending on the quantity and position of the double bond (Hansel *et al.*, 2004; Evershed *et al.*, 2008). While it was argued that APAAs, and APAA-C_{20} in particular, are biomarkers related to the processing of marine and aquatic oils (Copley *et al.*, 2004; Hansel

et al., 2004; Craig *et al.*, 2007; Evershed *et al.*, 2008), it has since been demonstrated that the ratio of APAA C_{20}/C_{18} is a more reliable threshold, since other foodstuffs can generate APAA-C_{20} (Bondetti *et al.*, 2021; Sun *et al.*, 2023). Furthermore the precise isomeric distribution for each APAA species seems to be determined by the precursor UFA(Evershed *et al.*, 2008), which has allowed for greater interpretative discrimination between possible candidate foodstuffs through the ratio of isomers E and H (Bondetti *et al.*, 2021). However, the likely role of metal ions and steric effects coming from the ceramic matrix in determining APAA quantity and isomer spread is still not perfectly understood; it seems possible that stone, with its widely varying mineral composition, porosity and steric chemistry, will produce different results.

More recently other cyclic alkanoic acids have been identified as thermal processing biomarkers (Breu *et al.*, 2023). These *ω*-(2-alkycyclopentyl) alkanoic acids (ACPAAs) include *trans* 9-(2-butylcylocpentyl)-nonanoic acid, *cis* 4-(2-nonylcyclopentyl)-butanoic acid and an unknown compound with a characteristic 223 *m/z* ion. Since these either co-elute or appear very close to several $C_{18:1}$ isomer peaks, it is still unclear how many such acids exist or how their distribution might be affected by the parent substrate, heating times, temperatures or underlying ceramic chemistry.

BPCAs function as a molecular marker for condensed aromatic moieties (Vaezzadeh, Zhong and Zhang, 2023), specifically those produced during the combustion of organic matter to form pyrogenic carbon (PyC) or 'black' carbon (Glaser *et al.*, 1998; Schneider *et al.*, 2010; Wiedemeier *et al.*, 2015; Vaezzadeh, Zhong and Zhang, 2023). While the extraction of the full suite of BPCAs (containing up to 6 carboxylic acid substitutions) generally requires sample digestion with nitric acid at high temperature and pressure (Glaser *et al.*, 1998, 2021; Wiedemeier *et al.*, 2016), BPCAs with a fewer number of carboxylic acid substitutions have been recovered from archaeological ceramics using lipid extraction procedures (Sarret *et al.*, 2017; Admiraal *et al.*, 2020; Dolbunova *et al.*, 2022). A lack of benzene pentacarboxylic acid (B5CA) and mellitic acid (B6CA) unfortunately limits possible interpretations related to combustion temperature and/or fire intensity (e.g., (Schneider *et al.*, 2010; Wolf *et al.*, 2013; Wöstehoff *et al.*, 2022), yet the presence of BPCAs strongly supports the fact that the pyrolysis of organic matter has occurred and may shed light on the aromaticity and aromatic condensation of the charred material. Finally, both gamma-lactones and amide products like erucylamide and octadecanamide can help support an interpretation of thermal transformation (Lejay *et al.*, 2016, 2019).

Here we present results from three experiments which aim to evaluate if and how any of these biomarkers form in heated stone. Three different heating systems were employed:

- a closed, controlled laboratory system using rapeseed oil;
- an actualistic stone griddle;
- an actualistic pit-cooking set-up

Common foodstuffs were cooked with experimental stones to test whether heating biomarkers could be formed under these different conditions. Some of these heated stones were sampled for organic residues by hand using a small hammer and then ground in a pestle and mortar, while others were sampled using a bench-drill with a diamond coring attachment and reduced with a pestle and mortar. To ensure that any lipids sampled were only those which had come from the experimental foodstuffs themselves, the stone surfaces were lightly abraded and cleaned with distilled water and dichloromethane post-experiment.. Approximately 1-4g of ground stone was used for each sample, following the standard acidified-methanol protocol. Each sample was subject to GC-FID and GC-MS, including use of a DB5 column and DB23 for SIM mode (see Methods).

3.0 Results & Discussion

3.1 Are sandstone and granite a good matrix for heating-biomarker formation?

The laboratory experiment was designed as a development of the method published in Bondetti et al. (2021). Three materials - ceramic, sandstone and granite - were powdered and 4g added to a hach tube. Each material was then heated with 65 ml of rapeseed oil in an oven under four different conditions - for one hour at 200 °C; for one hour at 350 °C; for 5 hours at 200 °C and for 5 hours at 350 °C, resulting in 12 samples plus 3 controls (65 ml rapeseed oil in unheated stone or ceramic). The aim of this experiment was to evaluate heating biomarker formation within stone in a simple, closed experimental system. In total, three classes of biomarker were identified: APAAs, BPCAs and one sample with ACPAAs.

BPCAs containing at least two carboxylic acid substitutions were present in the majority of laboratory samples heated to at least 200 °C for more than 1 h, with the exception of granite heated at 200 °C for 5 hours. Their presence was positively correlated

with time and temperature such that ceramic at 350 °C yielded the greatest amount of benzene tricarboxylic (B3CA) and tetracarboxylic (B4CA) acids. In both ceramic samples heated to 350 °C (1h and 5h), the full range of B3CAs and two B4CAs (mellophanic and pyromellitic acid) were identified. The only other laboratory sample yielding a B4CA (pyromellitic acid) was sandstone heated to 350 °C for 5 hours. No BPCAs containing more than four carboxylic acid substitutions were identified.

All samples heated at 200 °C yielded APAA-C_{18s} and the granite sample set at 350°C (LBX-2C) yielded APAA-C_{18s} and a small amount of APAA-C_{20} (0.09). It was also the only sample to yield any ACPAAs (Appendix 6.5). In determining the relative quantities of APAA-C_{18} produced, it is clear that ceramic outperforms stone at 200 °C, (Table 6.1) however, granite yielded more APAAs, both in quantity and type, at 350 °C. This clear difference indicates that the ceramic matrix provides a more optimal and catalytic environment than stone within a particular temperature band.

Table 6.1. Relative quantities of APAA calculated using the 290 M+ and 87 M+ ions, along with the hexatriacontane ($C_{36:0}$) internal standard ($\mu g \cdot g^{-1}$). The final figure is dimensionless.

Sample	Conditions	Sample total lipid concentration (ug g^{-1})	Normalised APAA $C_{18:0}$ quantity
LBX-1A (ceramic)	1 hour, 200 C	257	19
LBX-1B (sandstone)	1 hour, 200 C	217	5
LBX-1C (granite)	1 hour, 200 C	113	0.41
LBX-2A (ceramic)	1 hour, 350 C	498	0.94
LBX-2B (sandstone)	1 hour, 350 C	276	1
LBX-2C (granite)	1 hour, 350 C	272	21
LBX-3A (ceramic)	5 hours, 200 C	250	100
LBX-3B (sandstone)	5 hours, 200 C	244	0.74
LBX-3C (granite)	5 hours, 200 C	293	3

Following Bondetti et al's (2021) method has enabled us to update some of the key observations they previously made, namely that APAAs were formed in as little as one hour at 200 °C. In fact, APAA-C_{18} was formed at both 200 and 350 °C in just one hour, and at 200 °C for five hours. The intensive heating of the fourth sample set (five hours at 350 °C) seems

to have degraded much of the APAAs. Curiously only sample LBX-2C (granite, 350 °C, 1 hour) yielded any APAA-C_{20} results; the C_{20}/C_{18} ratio was 0.09, which passes the threshold of 0.06 established by Bondetti et al. (2021) for identifying aquatic products. The APAA-C_{20} was unexpected, but rapeseed oil does contain $C_{20:1}$ *cis* 11 in a 0.04 ratio with $C_{18:1}$ *cis* 9. Most of the E/H ratios are between 1.4-3.9, however three outliers of 13.2, 17.8 and 25.1 came from several stone samples (Fig 6.1). In all these cases the skew was caused by very low levels of the H isomer, rather than larger amounts of the E isomer. Still, this result is significant, affecting 50% of all the stone samples, perhaps indicating an underlying mechanism yet to be elucidated. No LCKs were noted in any of the samples, including in the ceramics.

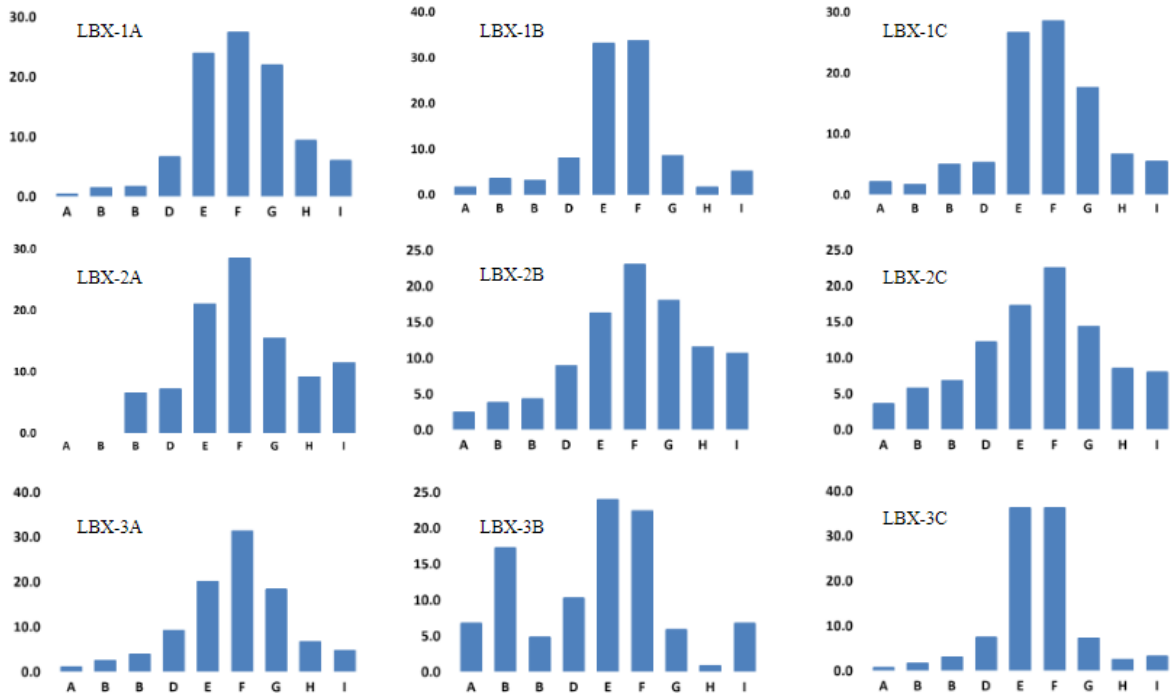

Fig 6.1. The relative quantity (%) and distribution of the APAA-C_{18} isomers (A-I) using the area of the *m/z* 290 ion.

3.2 Can heating biomarkers form in an actualistic cooking scenario - 'short periods of direct heat'?

The first set of samples were the product of an actualistic experiment involving the use of a piece of commercial sandstone tile to simulate a cooking griddle, onto which was

placed four different foodstuffs: beef rump steak (*Bos taurus*), rainbow trout (*Oncorhynchus mykiss*), hazelnut butter (*Corylus avellana*) and ground birch bark powder (*Betula pendula*). The stone was quickly heated, withfoods placed directly onto the surface and cooked until completed (Fig 6.2). Temperatures of both the stone and the foods were recorded.

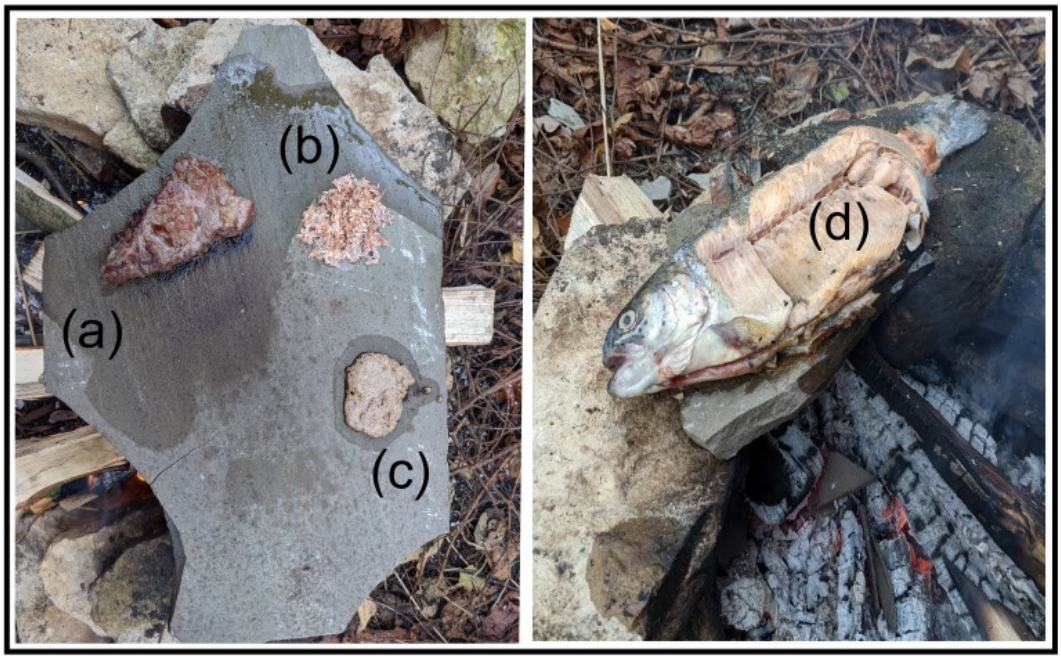

Fig 6.2. Images of the different foodstuffs being cooked on the sandstone griddle. Samples were taken from the stone directly beneath the food: a) X01 (steak), d) X02 (trout), c) X03 (hazelnut), b) X04 (birch bark) and X05 (control). The sampling methodology is outlined in section 1.2 (coring method) of Appendix 3.

Figure 6.2 shows the hot-rock cooking system for the griddle, with the heat transferring from the fire underneath to the foods. The temperatures and times were approximately 30 mins for each food, with a cooking temperature band between 130 - 270 °C. The lipid concentrations for each sample were as follows: beef steak, 84µg.g^{-1}; rainbow trout, 147µg.g^{-1}; ground hazelnut, 200µg.g^{-1}; birch bark, 4µg.g^{-1}; control, 1µg.g^{-1} (mean = 87.2µg.g^{-1}) reflecting the abundance of MUFAs in hazelnut at one end and the almost non-existent lipid presence in birch bark at the other.

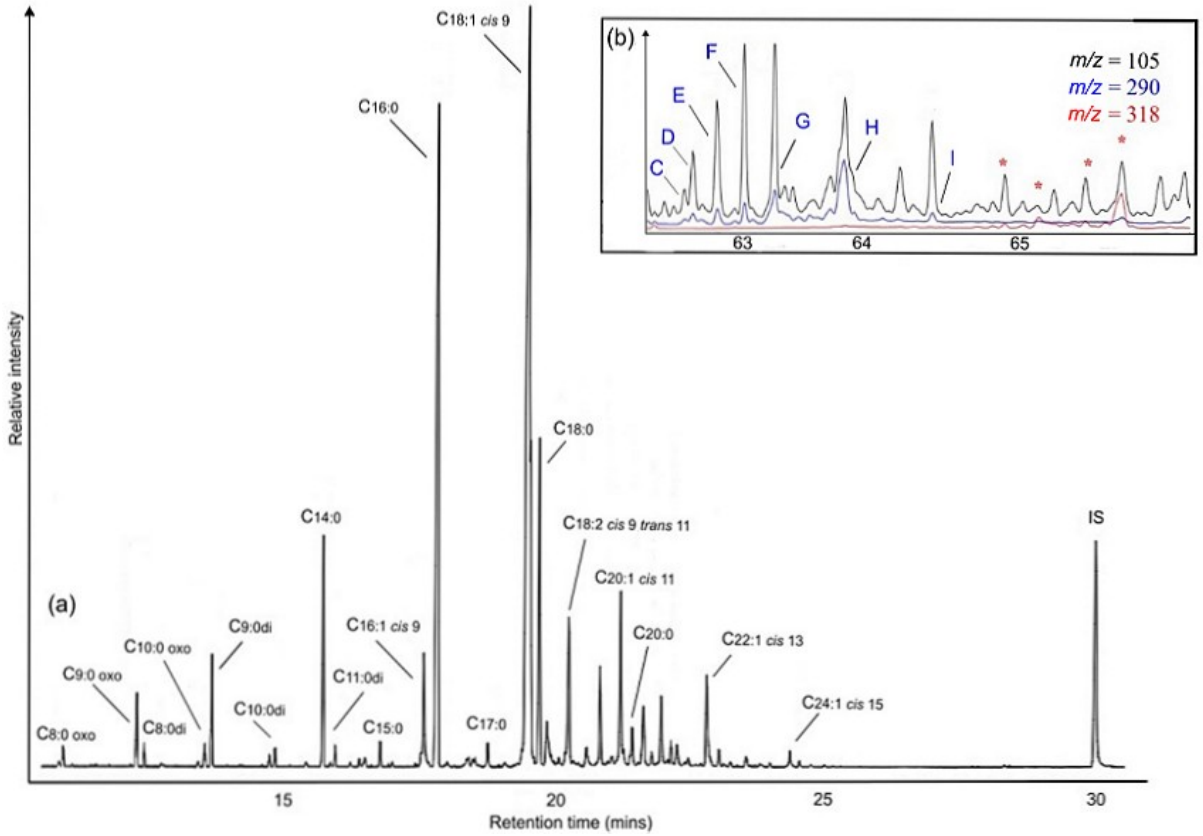

Fig 6.3. (a) Labelled chromatogram of an acidified-methanol sample extract: X02 'trout'. Identified APAA isomers from C_{18} and C_{20} originating from a separate SIM scan are incorporated **(b)**, displaying the weak production of APAAs in less than thirty minutes of cooking time.

A table of identified compounds was created (Appendix 6) from each sample, the composition of which reflected the foodstuffs they were derived from (Fig 6.3). These included saturated fatty acids ($C_{8:0}$ - $C_{24:0}$), monounsaturated fatty acids ($C_{8:1}$ - $C_{24:1}$) and polyunsaturated fatty acids ($C_{18:2}$). Products of spontaneous autooxidation/thermal degradation were identified, including α,ω-dicarboxylic acids (C_8 - C_{11}) and short-chain keto acids (C_8 - C_{10}). Consistent with the literature on frying unsaturated fatty acids, there was an increased number of oxylipin species in the X02 (trout) and X03 (hazelnut) sample. No long-chain ketones could be detected in any sample, despite efforts to isolate them, including the use of solvent and acidified methanol extraction, silylation, saponification and acidic/neutral fractionation. Small peaks corresponding to $C_{16:0}$ and $C_{18:0}$ gamma-lactones were identified in samples X01, X02 and X03, as well as the amide product erucylamide in X01 and X02.

Despite the short amount of time each foodstuff spent on the griddle (mean = 25 minutes), it was possible to detect small quantities of both APAA-C_{18} and APAA-C_{20} within the beef steak, trout and hazelnut samples. Small APAA-C_{18} peaks were detected in the birch bark sample, however, many of the isomers appeared to be co-eluting with other unknown products. These results further decrease the known length of time required to form APAAs to less than an hour. Interestingly the thresholds established by Bondetti et al (2021) were not applicable to these results, since the E/H isomer ratios fell outside the expected range for their origins (X01 = 0.48, X02 = 0.16, X03 = 1.25), and the C_{20}/C_{18} APAA ratio for the meat and hazelnut was much higher than expected (X01 = 0.06, X03 = 0.06). This could have been due to the very low quantities of APAAs generated, but also reflect underlying differences between ceramics and stone with regards to how reactions are catalysed, the steric hindrance of the matrix and how heat is absorbed and radiated.

3.3 Can heating biomarkers form in an actualistic cooking scenario - 'protracted heating, indirect heat'?

The second experiment (Appendix 4) aimed at replicating an underground heating pit, using wood and heated rocks. Experiments were conducted in two phases. The first involved cooking wild boar (*Sus scrofa*) and elk (*Alces alces*) meat wrapped in boar skins over heated stones covered in earth and plant materials (Fig 6.5). Stone samples were then taken and other stones were left in situ to test post-depositional changes to the lipid profiles. The experiment was repeated five months later using fish (roach*, Rutilus rutilus*, and crucian carp, *Carassius carassius)* and roe deer (*Capreolus capreolus*) wrapped in roe skins and ferns, after which further stone samples were removed for lipid extraction. Where possible each stone was sampled twice to increase coverage. All stone samples were extracted using acidified-methanol, with the exception of sample LTV-19, which was too alkaline in composition and thus inappropriate for acid-based protocols. In this case a solvent extract was performed. Soil samples were also gathered five months after the initial experiment to examine the comparability of the lipid compounds absorbed in the heated stones versus the soil matrix and to characterise the depth at which the lipid compounds generated by this experiment could be detected in the soil profile.

The lipid concentrations were lower than expected(range 10 - 32 µg g^{-1}, mean = 19), and sample LTV-03 was excluded due to very low lipid concentrations, but the remainder

were above the established threshold for ceramic vessel (5> µg g^{-1}) (Evershed *et al.*, 2008). Lipid identification revealed aliphatic saturated fatty acids ($C_{8:0}$ - $C_{28:0}$), monounsaturated fatty acids ($C_{16:1}$ - $C_{22:1}$) and some polyunsaturated fats ($C_{18:2}$). Searching directly for APAAs and isoprenoids revealed small amounts of APAA-C_{18} in samples LTV-18A and LTV-18B (Fig.3), confirming that APAAs can be formed in stone under very different cookery-systems. The E/H ratios fell outside the typical boundary for predominantly animal fats (LTV-18A = 7.4, LTV-18B = 7.5). Several B3CAs were identified in samples LTV-06 and LTV-18 (1,2,4-B3CA and 1,3,5-B3CA) (Fig 6.4). This demonstrates that BPCAs (and B3CAs in particular) can be formed under actualistic cooking conditions (combining both vegetal and animal products), were able to penetrate the stone mineral matrix during the course of the first experiment, and persisted over five months until their unearthing for the second phase of experimentation.

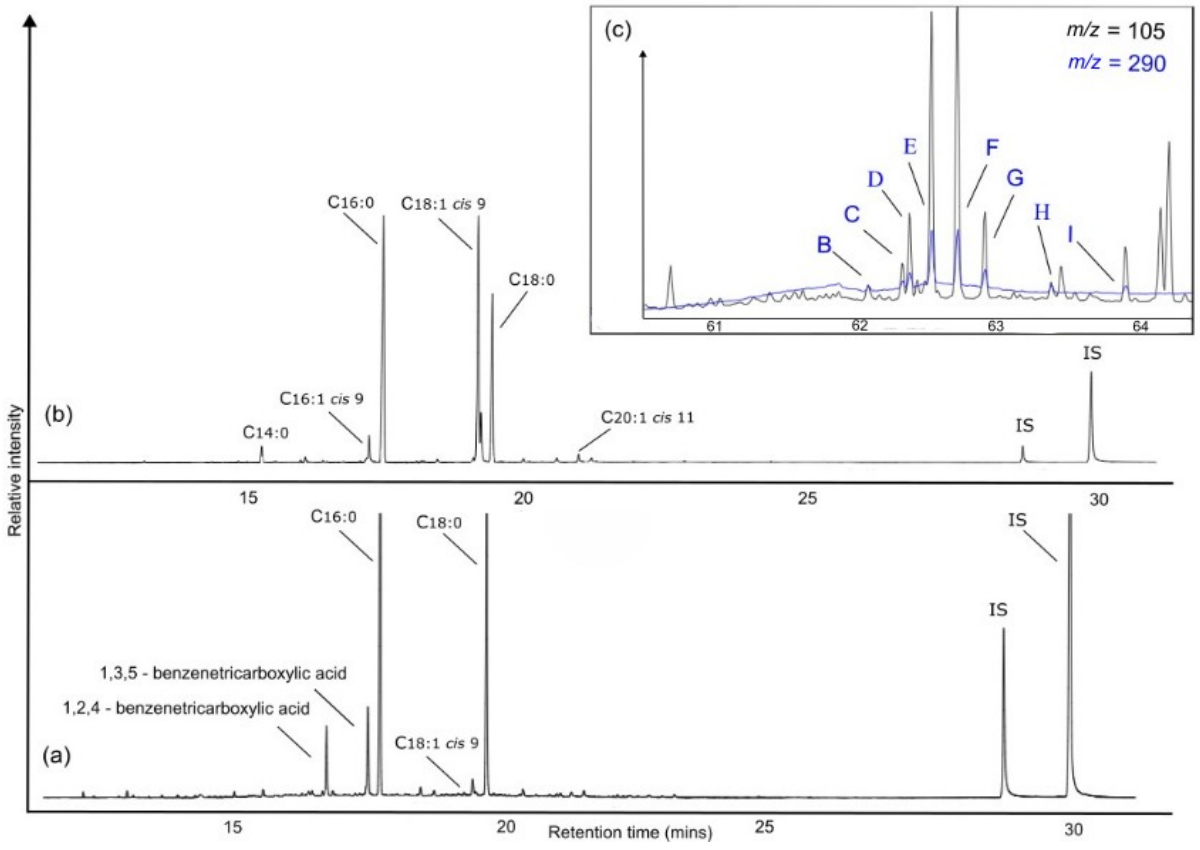

Fig 6.4. Labelled comparative chromatograms of samples LTV-06A **(a)** and LTV-18A **(b)** incorporating the APAA results taken from a separate SIM scan for LTV-18A **(c)**

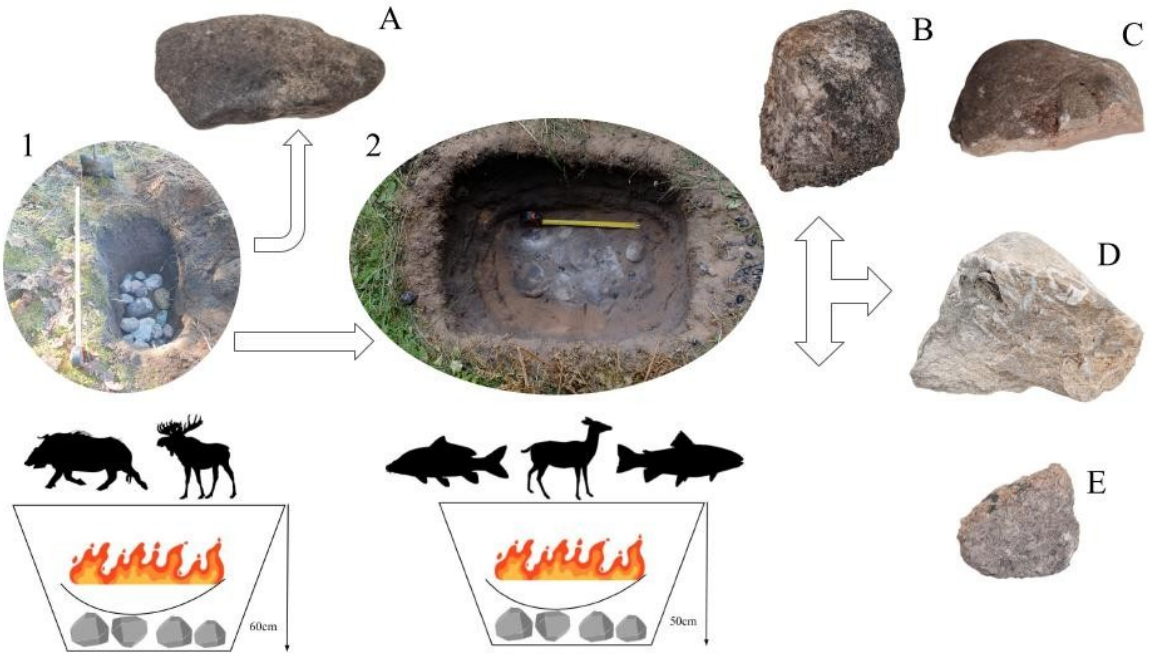

Fig 6.5. Depiction of pit-cooking experiment. 1. Experiment one, incorporating wild boar and elk. 2. Experiment two, incorporating fish and roe deer. Samples: A. LTV-03. B. LTV-06. C. LTV-18. D. LTV-19. E. LTV-20.

3.4 Evidence of short-term diagenesis?

Samples LTV-18, 19 and 20 were placed into the pit for the second experiment only, whilst samples LTV-06 participated in the first experiment and remained buried for five months. LTV-19 and 20 presented with a more reduced range of FAs and no diacids or short-chain ketones. By contrast LTV-06 and LTV-18 show very different profiles. LTV-06 shows higher levels of BCFAs, diacids, 2-hydroxy FAs and methyl dehydroabietate - the oxidised form of abietic acid. This is consistent with LTV-06 undergoing more oxidative degradation whilst buried between experiment one and experiment two: the breakdown of both PUFAs and MUFAs such as $C_{18:1}$ and $C_{16:1}$ often yields azelaic acid (C_9 dicarboxylic acid) (Evershed *et al.*, 2002; Pozhidaev *et al.*, 2021) and other products such as 9,10-octadecanoic acid (Colombini, Modugno and Ribechini, 2005), whilst previous experimental lipid decay results show an increase in branched-chain fatty acids from bacterial cell walls, particularly C_{15} (*iso* and *ante*) and C_{17} (*iso* and *ante*) (Marty *et al.*, 1996; Dudd, Regert and

Evershed, 1998). Despite LTV-06 being the only sample which was buried for five months, it also showed the highest levels of PUFAs ($C_{18:2}$ *cis* 9, *cis* 12, $C_{18:2}$ *cis* 9, *trans* 11), which likely originated from the wild boar meat and had yet to be fully degraded before being reheated in the second experiment.

LTV-18 shows higher levels of short-chain ketones, terpenes, alkanols, PAHs (pyrene, anthracene), cholesterol derivatives and a cardenolide sterol glycoside called gitoxigenin. Despite undergoing the same experimental protocols, we must conclude that minor variations and microenvironments within the pit-system itself, such as distance from the animal products, and variations in stone porosity, maximum temperature and bacterial activity in the soil played a major part in producing such different outcomes.

4.0 Conclusions

FCRs have the potential to yield valuable new sources of bioarchaeological data, particularly in places and time periods where ceramics are missing. Previous organic residue studies looking at FCRs have proven that, at the very least, lipids can bind to the internal matrix of stone, and be recovered using solvent extraction (March and Soler Mayor, 1999; Quigg *et al.*, 2001; Buonasera, 2005, 2016; Lucquin, 2007). Here we presented three experiments, two actualistic and one laboratory based, which produced 30 samples of lipids extracted from heated stone. The material differences between stone and ceramic were first explored through a controlled laboratory experiment, which generated a matrix of results based on material, time and temperature of heating. In trying to replicate and further develop Bondetti et al (2021)'s experimental work, we submit the following observations:

- APAAs, ACPAAs and BPCAs can be formed by heating lipids with stone
- In the majority of cases, ceramics outperforms stone in APAA-C_{18} quantity
- The E/H ratios, isomer distribution and C_{20}/C_{18} results indicate that stone may behave differently to ceramics, common threshold metrics may not apply
- Lipid react differently to varying types of stone matrix and composition under equal conditions

Two actualistic experiments confirmed that APAAs (C_{18}, C_{20}) can be generated within stone using two very different cooking-systems: '*short time, direct heat'* (stone griddle) and

'long time, indirect heat' (pit-cooking). APAAs were produced in less than thirty minutes on a stone griddle - this sets a new minimum time threshold. The relative scarcity or absence of other heating biomarkers (amides, lactones, LCKs, VLC-oxo acids, ACPAAs) indicates that stone may behave differently to ceramics, both in its essential chemistry, but also due to the mechanism of cooking utilised. Stone griddling yields a high lipid concentration, but the short cooking time may hinder the formation of particular products, conversely the pit-cooking produced more diagenic activity, but the low lipid concentrations and greater temperature gradient may also have prevented many heating biomarkers from forming.

However, the different cooking-systems appeared to alter the thermal decomposition products. Medium-chain saturated fatty acids were minimal to non-existent in all four griddle stone samples, suggesting that the degradation pathways for '*short-time, direct-heat'* were dominated by the β-scission route B, whereby the C-C bond closest to the double bond in the allylic hydroperoxide was broken, forming an initial aldehydic acid (Berdeaux *et al.*, 2012). The generation of *trans* isomer octadecanoic acid products, including double bonded variants such as $C_{18:2}$ *trans* 6 *trans* 9, is also likely a result of rapid thermally derived isomerisation (Wolff, 1993; Li *et al.*, 2013; He *et al.*, 2023). Oxylipins, those lipid compounds which have incorporated an oxygen atom such as keto, epoxy and hydroxy acids, appear frequently as secondary oxidation products from the thermal breakdown of unsaturated fatty acids (Khor *et al.*, 2019; Yang *et al.*, 2021; Cao *et al.*, 2022). Both the trout and hazelnut show multiple oxylipins, including oxo-acids and 9,10 dihydroxy octadecanoic acid. The lack of similar products in the steak sample conforms to the research on frying oils, whereby saturated fatty acids are more resistant to thermal hydrolysis and degradation (Choe and Min, 2007; Koch, Löwen and Schebb, 2024). Based on these results we submit that the presence of high levels of oxylipins in combination with low or absent levels of SCFA and MCFAs in a stone sample potentially indicates that it was used for dry-frying, although we must be cognizant of degradation and alteration to these lipid patterns in any possible archaeological FCR samples.

Pit-cooking on the other hand creates a very different environment, one where the stones may or may not be in direct contact with the foodstuffs, and where low levels of oxygen, higher levels of water and smoke and the presence of soil, sand, plant materials and secondary cooking products like pouches, skins or bark linings/containers may substantially alter the resulting lipid profile. From the thermal data available, higher temperatures appear to be achieved in pit-cooking than for frying; however, large gradients may exist from the

foodstuff to the mix of charcoal and stones underneath. Experimental pit-ovens using stones have recorded initial temperatures over 900 °C, which drop when both stones and food are added, but are still higher than the stone griddle (Graesch *et al.*, 2014; Thoms *et al.*, 2018). In keeping with this the pit-cooking stone samples were inconsistent, but at least one achieved the right temperature and conditions to form APAA-C_{18}.

Although we have not explored the thermal properties of the different stones here, it is well known that granite, ceramic and sandstone differ in terms of heat capacity and thermal conductivity (Bronitsky, 1986; Miao, Li and Chen, 2014; Abdulagaov *et al.*, 2019; Miranda *et al.*, 2019). Such properties are likely to affect how lipids interact with the substrate matrix and perhaps in how reactions such as isomerisation and decarboxylation take place. Based on this study it appears that granite *behaves* as though it were at a lower temperature than the other two materials, yielding less BPCA molecules and forming a C_{20} APAA series at 350 °C, as well as several ACPAAs. Further research into the conductivity, porosity, heat capacity and composition of different stones will be important next steps to fully characterise how stone differs from ceramics under various heating and cooking conditions. Furthermore, since Bondetti et al. 2021 only used one variety of ceramic for their experiment, it will be necessary to replicate the method using different types of clay, in order to test how the different mineral and elemental compositions affect any APAA isomeric distribution. It may be that, by including other ceramic types into this dataset, the results from the granite and sandstone samples more closely match other compositional forms.

Overall, the conclusions of these experiments are positive and exciting, advancing the study of an understudied material type in organic residue analysis. The results confirm the utility of APAAs as a robust and key biomarker for anthropogenic heating, being present in all three experimental set-ups. Given that ceramics were a relatively late invention in the scope of archaeological analysis, the results of this study, and the methods developed herein, have significant implications for better understanding cooking systems, cuisine and diet in times and places where pottery was not used.

5.0 Methods

The extraction of ceramic is a well tested and understood protocol, however, no such experience exists for the best practice with stones and different types of stones. Therefore

the sampling and extraction methods are also under investigation here, and will hopefully provide some use to future researchers. Two methods were employed to sample the whole stones, dubbed 'kinetic' and 'coring'. Since the friability and ease of breaking an FCR differs according to context, some stones were simply broken by hand using a small hammer and then ground in a pestle and mortar, while others were sampled using a bench-drill with a diamond coring attachment and then reduced with a pestle and mortar. To ensure that any lipids sampled were only those which had come from the experimental foodstuffs themselves, the stone surfaces were post-experimentally lightly abraded and cleaned with distilled water and dichloromethane. Kinetically broken stones yielded less accurate sub-sections, but care was taken to select pieces for grinding which were within the main outer layer of the stone.

The aim was to employ the acid methanol protocol which has been successfully used to extract lipids from potsherds and ceramics (Craig *et al.*, 2013; Correa-Ascencio and Evershed, 2014). Approximately 1-4 g of each ground stone subsample was treated with excess methanol-sulfuric acid solution in a test tube. Differences in the composition of the stone meant some were treated with more solution than others until the pH was near to 3.5. The samples were heated in an ultrasonic water bath for 15 minutes before being heated for 4 hours at 70 ˚C in closed vials. The acid-stone suspension was then centrifuged and the supernatant removed. Each sample was mixed with 2 mL of *n*-hexane before being filtered through glass wool and potassium carbonate (washed with dichloromethane). The samples were dried down using N_2 at 37 ˚C. In preparation for GC-MS 10 µL of an internal standard (1.0 µg μL^{-1} hexatriacontane) was added to the samples in a new vial which were then resuspended in 100 µL of *n*-hexane and dried a final time before being transferred to their analysis vials. Some batches underwent silylation before analysis. This involved treating the samples with excess bis(trimethylsilyl)trifluoroacetamide (BSTFA) and hexane before heating for 1 hour at 70˚C. The solutions were then dried using N_2 for 15 minutes.

GC-FID was used to screen and quantify lipid compounds. GC-FID was carried out on acidified and solvent based lipid extracts using an Agilent 7890S gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK). A splitless injector was used to inject the sample (1 µL) at 300°C. The column was a polymide coated fused silica DB-1 (15 m x 320 µm x 0.1 µm; J&W Scientific, Folsom, CA, USA). The carrier gas was helium. The pressure was set at 3.3 psi with a flow rate of 2 mL min^{-1} and a velocity of 46.57 cm s^{-1}. The

temperature program was set at 100 °C for 2 minutes, which then rose by 20 °C min^{-1} until 325 °C, where it was held for 3 min.

GC-MS analysis was conducted using an Agilent 7890A series chromatograph with an Agilent 5975C Inert XL mass-selective detector and quadrupole mass analyser (Agilent technologies, Cheadle, Cheshire, UK). A splitless injector was used and maintained at 300 °C. The GC column was directly inserted into the ion source of the mass spectrometer. The carrier gas was helium and the inlet/column head-pressure was kept constant. The ionisation energy of the MS was 70 eV and spectra obtained by scanning between *m/z* 50 and 800. A DB-5MS (5%-phenyl)-methylpolysiloxane column (30 m x 0.250 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) was used for screening the samples in scan mode. The temperature was set at 50°C for 2 minutes, which rose by 10°C min^{-1} until 325°C was reached where it was held for 15 min.

To check the samples for heating biomarkers the samples were analysed using a GC-MS with a DB-23 (50%-Cyanopropyl)-methylpolysiloxane column (60 m × 0.250 mm × 0.25 µm; J&W Scientific, Folsom, CA, USA). The oven temperature was set at 50 °C for 2 minutes before increasing to 100 °C (10 °C min^{-1}). The temperature was then raised by 4 °C min^{-1} to 140 °C, then by 0.5 °C min^{-1} to 160 °C and, lastly, by 20 °C min^{-1} to 250 °C where it was maintained for 10 min. The carrier gas used was helium with a flow rate of 1.5 mL min^{-1}. The SIM (Selective Ion Monitoring) mode was utilised to target cooking biomarkers using the ions groups: *m/z* 74, 87, 213, 270 for 4,8,12-trimethyltridecanoic acid (TMTD), *m/z* 74, 88, 101, 312 for pristanic acid, *m/z* 74, 101, 171, 326 for phytanic acid and *m/z* 74, 105, 262, 290, 318, 346 for the detection of *ω*-(*o*-alkylphenyl)alkanoic acids of carbon lengths C_{16} to C_{22} ($APAA_{16\text{-}22}$).

All analytical work was conducted on available software. Chemstation and MassHunter for the GC-MS outputs.

6.0 Acknowledgements

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Chapter Seven: Invisible technologies? Using organic residue analysis to reveal prehistoric aceramic cooking practices in Mesolithic and Neolithic Northern Europe

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1.0 Introduction

Pottery has played a major role in advancing our knowledge of ancient cuisines and foodways, primarily because the matrix of the vessel is capable of retaining lipids (McGovern and Hall, 2016; Papakosta and Pesonen, 2019; Rao *et al.*, 2019; Courel *et al.*, 2021) and other identifiable small molecules (Pecci *et al.*, 2013); (Reber *et al.*, 2019). By linking these recovered compounds to the foodstuffs presumed to have been used at the time, organic residue analysis has proven to be a powerful analytical method for determining what broad food groups were being used and when (Skibo, Malainey and Kooiman, 2016; Gibbs *et al.*, 2017; Montalvo-Cabrera *et al.*, 2024). This has led to significant findings, such as the earliest evidence for dairy consumption (Chakraborty *et al.*, 2020) or revealing the different economic strategies between ceramic foragers and agriculturalists (Robson *et al.*, 2019). However, pottery was a relatively late invention, and its adoption globally was uneven and

never universal (Piezonka, 2012; Elliott *et al.*, 2020). This means we are missing crucial details about the food choices, cooking strategies and dietary adaptations made for the majority of human prehistory.

The introduction of ceramics did not wholly replace previous cooking technologies however, rather ceramics were employed for specific and culturally different purposes (Bondetti *et al.*, 2019; Papakosta, Oras and Isaksson, 2019). This has raised a longstanding question about what purposes ceramics fulfil and what ecological, social or economic forces move cultures towards adopting pottery (Skibo, Malainey and Drake, 2009; Jordan and Zvelebil, 2016a, 2016b). Our knowledge of the prior methods and materials available for enclosing, boiling, containing and fermenting (both food and drink) is largely dependent on the ethnographic record (Driver & Massey, 1957; Nelson, 2010). Still, there are many archaeological examples of aceramic vessels. These include plant containers such as baskets (Martínez-Sevilla *et al.*, 2023), bark and wood vessels (Oshibkina, 1989; Gramsch, 1992; Burov, 1998; Gumiński, 2012; Fletcher *et al.*, 2018); (Gramsch and Kloss, 1990) and inorganic containers such as steatite (Speth, 2015; Admiraal and Knecht, 2018). Organic materials rarely survive in the archaeological record; when they do they are often degraded and leave little clue as to their exact function (Larsson, 2004; Fletcher *et al.*, 2018)**.**

One material class that does survive is stone. Stones that have been used for cooking, such as boiling stones, earth ovens or direct griddling, will likely be permanently altered with specific diagnostic characteristics (Jackson, 1998; Thoms, 2008; Ozguven and Ozcelik, 2013; Gao *et al.*, 2014; Short, 2018). ‘Fire-cracked rocks’ (FCRs), in addition to being studied for use-wear markers (Lovick, 1983), can also preserve organic residues within the matrix of the stone (Quigg *et al.*, 2001; Buonasera, 2005, 2007, 2016; Lucquin, 2007; Skibo, Malainey and Kooiman, 2016). Whilst ceramic vessels are relatively well characterised in the organic residue literature, stone represents a different set of challenges and opportunities, including the chance to better understand prehistoric diets and cuisines before and during the transition to ceramic adoption.

The late Mesolithic and early Neolithic periods in northern Europe offer just such a chance, since both the movement of technologies and peoples are relatively well understood (Piezonka, 2012; Budja, 2016; Hommel, 2018; Dolbunova *et al.*, 2022; Allentoft, Sikora, Refoyo-Martínez, *et al.*, 2024). Some Holocene foragers living around the Baltic Sea received or adopted ceramics largely through cultural diffusion (Jordan *et al.*, 2016; 2020),

whilst later contact with southern agricultural communities introduced new foodstuffs such as dairy (Lucquin *et al.*, 2023). Although the dietary differences between agriculturalists and foragers has been explored through ceramic organic residue analysis (Papakosta, Oras and Isaksson, 2019; Robson *et al.*, 2019; Papakosta *et al.*, 2020), the transition from aceramic to ceramic use within the same community has been understudied using the same techniques.

To do this six Mesolithic/Neolithic sites were selected for their time periods and suspected pyrolithic methods or techniques (Fig 7.1), in order to capture potential data on not only the foodstuffs themselves, but the specific cooking or thermal processing approaches involved. For the aceramic Early Mesolithic, two sites from Norway (Ormen Lange, Klakken) were chosen with the aim of testing the hypothesis that marine oils or fats were being combusted using FCRs. For the ceramic Late Mesolithic, two midden sites (Havnø, Visborg) and one Ertebølle coastal site (Rosenfelde) were selected, with the possibility that FCRs in cooking pits and hearths were utilised to cook or process fish and shellfish. Finally, the transitional site of LA-Neustadt 156 was selected in order to analyse an assemblage of linear fire-cracked slabs, which may have been used as part of an earth-oven or griddle cooking system. Here we present FCR derived lipid and isotope data from these six sites, demonstrating the methodological applicability of organic residue analysis to archaeological stone, and providing new cultural and chronological insights into northern European Mesolithic cooking and food-processing technologies.

1.1 Early Holocene marine fat rendering/heating

The rendering and burning of marine oils from blubber and fish during the Mesolithic has been confirmed through organic residue analysis of ‘blubber lamps’ and bowls from the late Mesolithic Ertebølle and Narva cultures (Heron *et al.*, 2013; Robson *et al.*, 2022). These illumination devices were made from ceramics, but it seems likely that marine fuels were

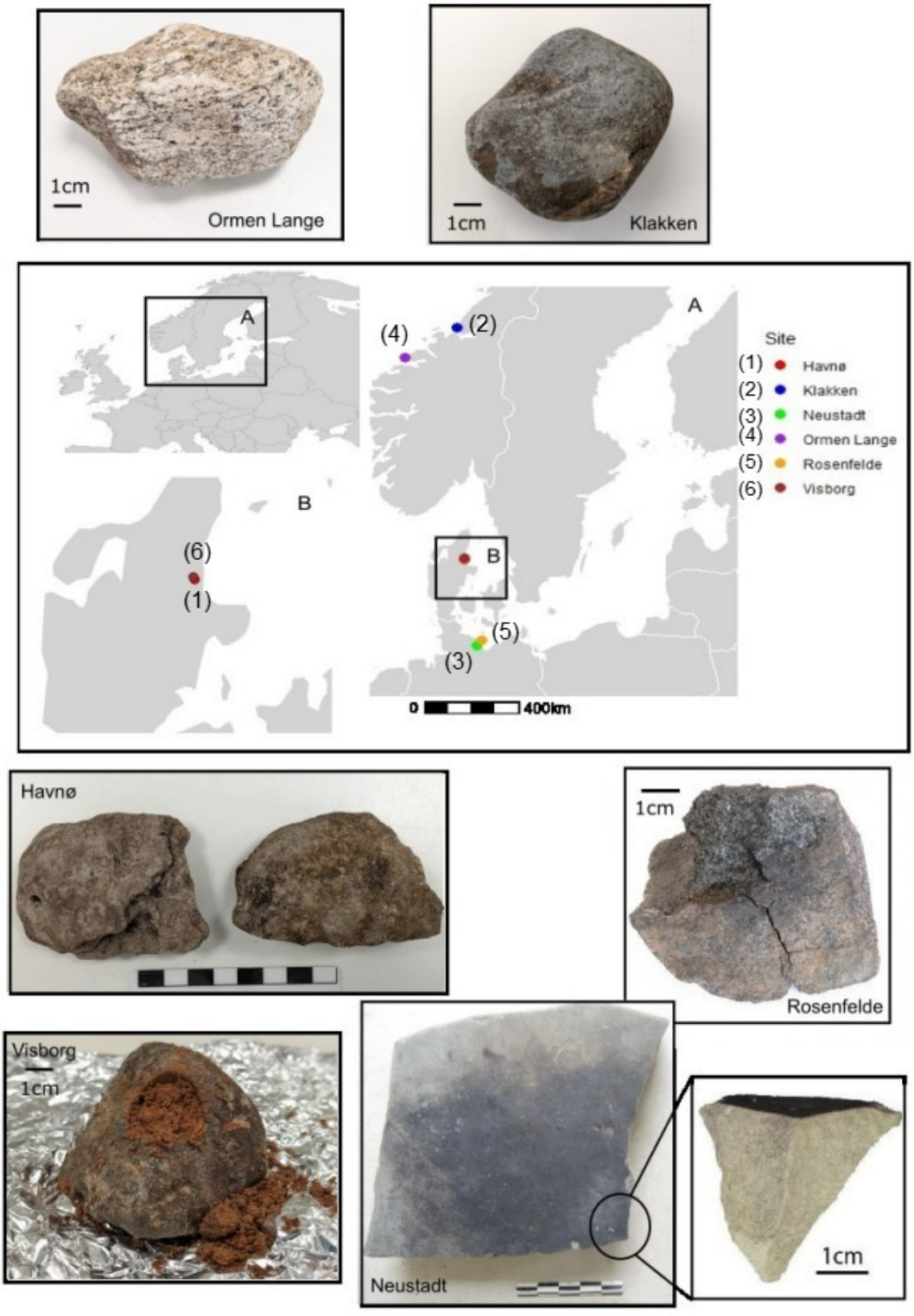

Fig 7.1. Site map and images of archaeological FCRs.

being used prior to the arrival of pottery technologies. One Early Holocene site on the eastern Swedish archipelago turned up “small black lumps of burnt organic matter containing

marine fatty acids [...] as well as more than one kilogram of burnt seal bone" (Pettersson *et al.*, 2014; Damm, 2022,13). Some Norwegian Mesolithic hearths have been speculated to have burnt marine oils or blubber, given the oily residues covering the FCRs and soil below (Bjerck, 2017; Breivik, 2020). Given the later Norwegian Iron Age use of 'slab-lined pits' to render whale and marine mammal fats (Heron *et al.*, 2010; Nilsen, 2016), it is conceivable that a similar aceramic pyrolithic heating system could have been used during the Early to Mid Holocene prior to the arrival of ceramics.

The excavated site of Ormen Lange (Isle of Gossen, Nyhamna, Møre and Romsdal County; Fig 7.1) was selected to test this hypothesis. Many hearths from localities dated to the Early Mesolithic (9410 ± 55 and 9515 ± 70 uncal. BP. (pers.comms)) present with large amounts of closely packed cobbles, pebbles and slabs, some of which were burnt and fire damaged. The soil directly underneath these was often sooty, and suspected to contain marine oils. (Bjerck 2017, 281). Previous organic residue analysis undertaken on soil samples taken from the fireplace in structure S1, Unit G, was inconclusive on the question of marine fuels (Heron, 2007). Five recovered stones from structure S1 were sampled for any retained lipids and isotope data. Alongside Ormen Lange, a second site was selected to sample FCRs. Klakken is a site on the Trøndelag coast (Fig 7.1), assumed to have been inhabited several times between the Early and Middle Mesolithic periods. Excavations over the past few decades (Petterson 1991; 2001), and one more recently (Gärtner and Valby, 2022), have identified the site as a shoreline encampment, evidenced mainly by lithic finds and the remains of hearths. The 2022 excavation took samples of a hearth, noting that the subsurface sediments had a fatty consistency. Given the absence of any organic finds or charcoal, the site has not been radiocarbon dated, but shoreline curve estimates place the most recent site in the Early Mesolithic period. Since this time period possesses no pottery nor many preserved organic vessels, data on their cuisine and pyrotechnology is limited. Three FCRs were recovered from the hearth for sampling.

Both sites represent Early Mesolithic coastal encampments or settlements, where ceramics were not yet available and marine resources likely constituted a core part of the diet.

1.2 Late Mesolithic cooking practices

The development and spread of ceramics has previously been understood as an adoption of a particular form of economic intensification focused on aquatic resources (Craig *et al.*, 2013; Lucquin *et al.*, 2016; Gibbs *et al.*, 2017; Hung *et al.*, 2017; Feng and Wang, 2022). Since this has been challenged, an important aspect of characterising Mesolithic subsistence is understanding how it may have changed locally and regionally as ceramics spread across the Baltic and southern Scandinavia - did ceramics replace previous methods and techniques of cooking and food processing? (Papakosta, Oras and Isaksson, 2019; Henderson *et al.*, 2022). Therefore, sampling FCRs from the ceramic Late Ertebølle period may be productive in yielding comparative organic residues for analysis.

Danish kitchen middens at the sites of Havnø and Visborg yielded a vast number of artefacts, faunal remains and data spanning the Late Mesolithic Ertebølle and Neolithic Funnel Beaker cultures (Robson *et al.*, 2013; Robson, 2015). The Late Ertebølle period saw the introduction of pottery from other related forager groups, as well as dietary changes associated with the trade and acquisition of new foodstuffs from the bordering agricultural populations (Papakosta, Oras and Isaksson, 2019; Dolbunova *et al.*, 2022; Lucquin *et al.*, 2023). The Havnø and Visborg middens (Fig 7.1.) were initially excavated in the late 19th century, and then again by Andersen between the 1990's and 2000's (Madsen, 1900; Andersen, 2000a, 2008), revealing a continuous deposition of material despite the now genetically characterised population turnover with the arrival of the Funnel Beaker communities (Allentoft, Sikora, Fischer, *et al.*, 2024; Allentoft, Sikora, Refoyo-Martínez, *et al.*, 2024). A review of the formation and use of Danish kitchen middens relates that three types of cooking structure are often discovered during excavations - a grey lens of burnt shell material; a stone-built structured hearth with FCRs and much larger pits "with successive layers of charcoal, burned shell, and clay" (Andersen, 2000b). The latter have been interpreted as cooking pits (Klinge 1931; (Meehan, 1982; Andersen, 2000b). One interpretation of these pits and hearths is as potential shellfish cooking structures, with some excavated beneath middens containing huge quantities of FCRs (Andersen, 1989; Milner, 2002). Milner also further describes these as possible evidence for large-scale feasting, perhaps for roasting or steaming large amounts of shellfish in rock-lined hearths or pits. Eight FCRs were recovered from the spoil heap during a recent excavation of Havnø,

estimated to be from the terminal Ertebølle period, although the lack of a secure context places them between 5250-1780 cal BC (Robson, pers comm). Two FCRs from Visborg were recovered during excavations of the midden, estimated to date from the terminal Ertebølle based on their context (Robson, pers comm).

Additional samples were also taken from an aceramic Ertebølle coastal site - Grube-Rosenfelde LA 83 (Fig 7.1). This site has been interpreted as a specialised fishing camp, based on some unusual features - the lack of a refuse area; wooden material culture related to fishing and several hearths associated with whitened fish bones, mostly *Anguilla anguilla* (Fehr, 2011). It has been suggested that the untypically high amount of aurochs bones recovered reflects the placement of meat into the water to attract nocturnal eels, since much of the faunal assemblage consisted of metapodials, tarsals and phalanges. The fish bones were heated and more fragmented than those found on similar Late Ertebølle coastal sites (Hartz *et al.*, 2014). One possibility is that the hearths were used for smoking or drying aquatic foods, predominantly eels. Fourteen cobble FCRs were taken from hearth feature 1, which was dated 4900 - 4500 cal BC to 5290 cal BC based on sampled charcoal (Hartz, 2005).

1.3 Terminal Mesolithic / early Neolithic transition

The transitional site of Neustadt (Schleswig-Holstein, northern Germany (Fig. 7.1)) offers the opportunity to sample FCRs during the period between the terminal Ertebølle culture (EBK) and the arrival of the Neolithic Funnel Beakers (TRB) (Hartz and Lübke, 2006; Glykou, 2011, 2016, 2017). At least two phases, an EBK and a TRB have been identified (Craig *et al.*, 2011; Glykou, 2011). Hartz (2005) dates the EBK to between 4500 and 4100 cal BC, while Craig et al (2011) dates the EBK to between 4600 and 4100 cal BC and the TRB to between 4100 and 3700 cal BC.The stratigraphy of Neustadt is not well characterised and the submarine nature of the site makes exact stratigraphic and specific contexts for artefacts difficult to identify with confidence. However, a considerable amount of Mesolithic and Neolithic ceramics, bones, flint, wood and antler have been recovered from the site (Glykou, 2011). Over 60 of these sherds have been analysed for lipid and other biomarkers, demonstrating a pattern of consistent marine foodstuff exploitation across the

EBK-TRB transition (Craig *et al.*, 2011; Robson *et al.*, 2021). Amongst the recovered artefacts were many flat, slab-like stones, deposited into the refuse area of the settlement. Many of these show clear patches of discolouration and sooting/burning. Holst (2023) has analysed a number of these using Fourier-transform infrared spectroscopy (FTIR), concluding that they were not used as griddle stones, but likely formed part of a 'slab-lined oven', based on their morphology and recorded heating intensity. Other unheated, unmodified sandstone slabs from the same context have also been assessed using FTIR and electron microscopy, revealing their mixed-utility for processing both plant and animal products on their surfaces (Holst *et al.*, 2024). Fourteen sub-samples were taken from the heated sandstone slab assemblage for organic residue analysis.

2.0 Materials and Methods

2.1 Archaeological Stones

The majority of the FCR samples showed signs of heat damage and alteration, including discolouration, fracturing, scarring and sometimes the disintegration of the stone where it had been strongly heated (Fig 7.1). Most were composed of quartzite and types of sandstone, with different inclusions and ranges of hardness (Appendix 5).

The stones were sampled either by using kinetic force to break the sample and select an appropriate section, or through more targeted means by use of diamond-tipped drill corers, either with a handheld or bench drill. The resulting fractured sections or core plugs were then ground to a powder using a stainless steel pestle-and-mortar. In order to ensure any recovered lipids came directly from within the stone matrix, all stones were cleaned before sampling, first by abrasively removing any exterior soot, soil or accretions, then by rinsing the surface with deionised water and dichloromethane (DCM). Some stones were sampled more than once, where possible, to provide a control, or to determine the variability of lipids within a single artefact. The Neustadt assemblage represents the only slab stones sampled; they were sampled in two locations - firstly where blackening/sooting was present, and secondly on the most 'natural' non-discoloured part.

2.2 Extraction methods

The aim was to employ the acidified methanol protocol which has been successfully used to extract lipids from potsherds and ceramics (Craig *et al.*, 2013; Correa-Ascencio and Evershed, 2014). Approximately 1-4g of each ground stone subsample was treated with excess methanol-sulfuric acid solution in a test tube. Differences in the composition of the stone meant some were treated with more solution than others until the pH was near to 3.5. Samples were heated in an ultrasonic water bath for 15 minutes before being heated for 4 hours at 70˚C in closed vials. The acid-stone suspension was then centrifuged and the supernatant removed. Each sample was mixed with 2 ml of *n*-hexane before being filtered through glass wool and potassium carbonate (washed with DCM). Samples were then dried down using N_2 at 37˚C. In preparation for GC-MS 10µl of an internal standard (1.0 µg $µl^{-1}$ hexatriacontane) was added to the extracts in a new vial which were then resuspended in 100µl of *n*-hexane and dried a final time before being transferred to their analysis vials. Some batches underwent silylation before analysis. This involved treating the samples with excess bis(trimethylsilyl)trifluoroacetamide (BSTFA) and hexane before heating for 60 minutes at 70˚C. The solutions were then dried using N_2 for 15 minutes.

2.3 Analytical methods

GC-FID was used to screen, quantify and identify lipid compounds. GC-FID was carried out on acidified and solvent based lipid extracts using an Agilent 7890S gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK). A splitless injector was used to inject the sample (1 µL) at 300 ºC. The column was a polymide coated fused silica DB-1 (15 m x 320 µm x 0.1 µm; J&W Scientific, Folsom, CA, USA). The carrier gas was helium. The pressure was set at 3.3 psi with a flow rate of 2 ml min^{-1} and a velocity of 46.57 cm s^{-1}. The temperature program was set at 100°C for 2 minutes, which then rose by 20°C/min until 325ºC, where it was held for 3 min.

GC-MS analysis was conducted using an Agilent 7890A series chromatograph with an Agilent 5975C Inert XL mass-selective detector and quadrupole mass analyser (Agilent technologies, Cheadle, Cheshire, UK). A splitless injector was used and maintained at 300°C. The GC column was directly inserted into the ion source of the mass spectrometer. The carrier gas was helium and the inlet/column head-pressure was kept constant. The ionisation energy of the MS was 70eV and spectra obtained by scanning between *m/z* 50

and 800. A DB-5MS (5%-phenyl)-methylpolysiloxane column (30 m x 0.250 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) was used for screening the samples in scan mode. The temperature was set at 50°C for 2 minutes, which rose by 10°C min^{-1} until 325°C was reached where it was held for 15 min.

To check the samples for heating biomarkers the samples were analysed using a GC-MS with a DB-23 (50%-Cyanopropyl)-methylpolysiloxane column (60 m × 0.250 mm × 0.25 µm; J&W Scientific, Folsom, CA, USA). The oven temperature was set at 50 °C for 2 minutes before increasing to 100 °C (10 °C/min). The temperature was then raised by 4 °C/min to 140 °C, then by 0.5 °C/min to 160 °C and, lastly, by 20 °C/min to 250 °C where it was maintained for 10 min. The carrier gas used was helium with a flow rate of 1.5 mL/min. The SIM (Selective Ion Monitoring) mode was utilised to target cooking biomarkers using the ions groups: *m/z* 74, 87, 213, 270 for 4,8,12-trimethyltridecanoic acid (TMTD), *m/z* 74, 88, 101, 312 for pristanic acid, *m/z* 74, 101, 171, 326 for phytanic acid and *m/z* 74, 105, 262, 290, 318, 346 for the detection of *ω*-(*o*-alkylphenyl)alkanoic acids of carbon lengths C_{16} to C_{22} ($APAA_{16\text{-}22}$).

Stable carbon isotope values of methyl palmitate ($C_{16:0}$) and methyl stearate ($C_{18:0}$) derived from the precursor fatty acids were assessed by GC-c-IRMS. An Isoprime 100 (Isoprime, Cheadle, UK) linked to a Hewlett Packard 7890B series GC (Agilent Technologies, Santa Clara, CA, USA) with an Isoprime GC5 interface (Isoprime Cheadle, UK) was used for the analysis. One ul of each sample was first injected into DB-5MS ultra-inert fused-silica column. The temperature was set at 50 °C for 0.5 min and raised by 0.5 °C per minute to 50 °C, then raised by 10 °C per minute to 300 °C where it was held for 10 min. The carrier gas used was ultra high purity grade helium with a flow rate of 3 mL per minute. The gas flows from the column were split. One was directed into an Agilent 5975C inert mass spectrometer detector (MSD), for sample identification and quantification, the other directed through the GC5 furnace kept at 850 °C to oxidise all the carbon species to CO_2. Clear resolution and a baseline separation of the analysed peaks were achieved.

Eluted products were ionized in the mass spectrometer by electron impact and ion intensities of m/z 44, 45 and 46 were recorded for automatic computing of the 13C/12C ratio of each peak in the extracts. Computation was made with IonVantage and IonOS softwares (Isoprime, Cheadle, UK) and was based on comparisons with standard reference gas (CO2)

of known isotopic composition that was repeatedly measured. The results of the analysis were expressed in per mill (‰) relative to an international standard, VPDB.

All analytical work was conducted on available software. Chemstation and MassHunter for the GC-MS outputs and IonOS for the GC-c-IRMS.

3.0 Results

3.1 Lipid concentration

The majority of samples yielded usable lipid concentrations (Fig 7.2). For ceramics, the concentration threshold is typically considered to be >5 μg g^{-1} (Evershed, 2008), and using that metric the majority of the samples from Rosenfelde would be considered too low in lipid concentration. However, determining a minimum threshold for stone is premature. The sample sizes ranged between 2 - 4 grams of ground stone and the mean lipid concentration has been reported both *per sample* and standardised *per gram*.

The GC-MS results from the Rosenfelde stones are reported in Appendix 6, but the total lipid concentration was so low that further discussion here was considered unnecessary.

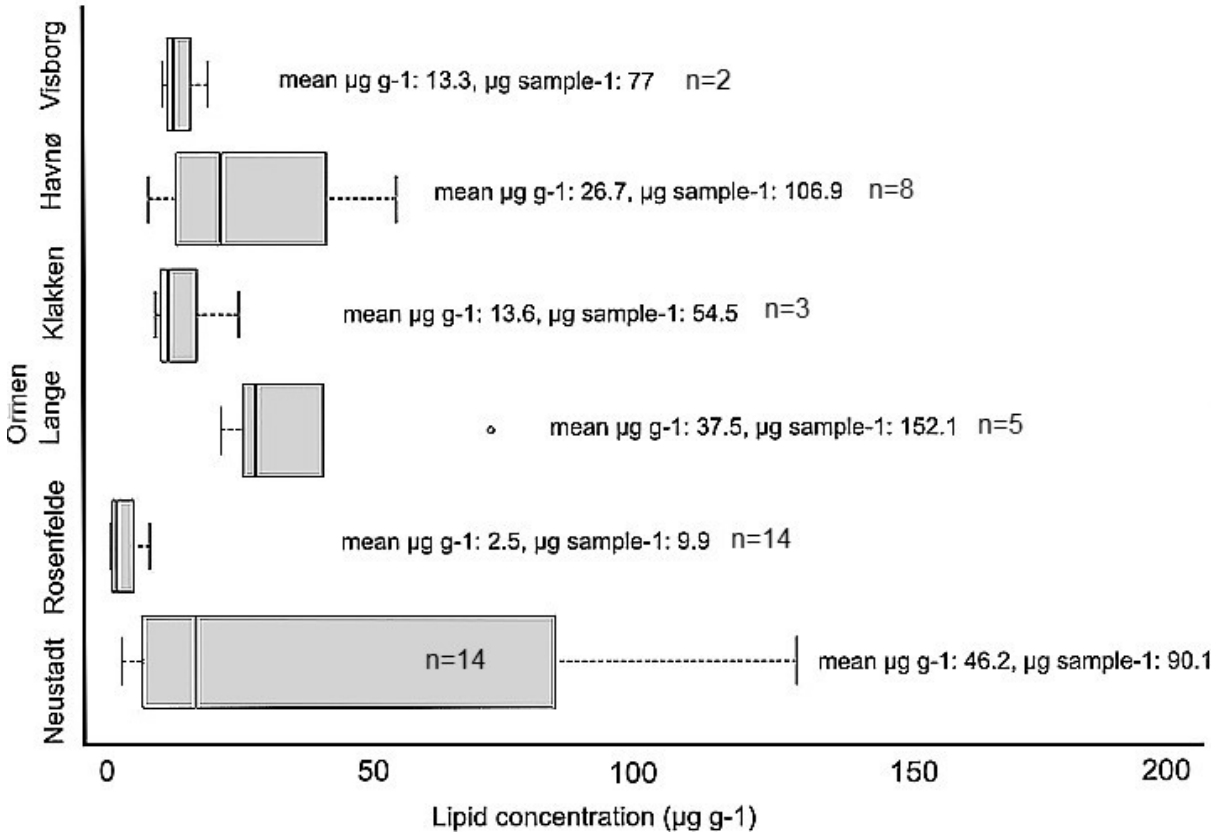

Fig 7.2. Lipid concentrations for each FCR assemblage. The boxplots represent the total number of samples measured as micrograms per gram. The mean lipid concentrations per gram for each site are depicted as well as per sample.

3.2 Ormen Lange and Klakken hearth stones

This assemblage was mostly dominated by $C_{16:0}$ and $C_{18:0}$, in an average 1:1 ratio. Chain length of the identified saturated fatty acids tended towards long-chain, with the shortest at $C_{10:0}$ and longest at $C_{28:0}$. Of the identified unsaturated fatty acids only monounsaturated acids seemed to be preserved. The abundance of long-chain fatty acids was particularly high, with more $C_{22:0}$ recorded than $C_{14:0}$, and relatively large amounts of $C_{20:0}$ - $C_{28:0}$. This, along with the presence of $C_{22:1}$ and $C_{18:1}$ indicates the lipids to be of marine mammal origins (Heron *et al.*, 2010). The presence of two dicarboxylic acids - $C_{9:0}$ and $C_{12:0}$ - further suggest the prior presence of UFAs. No ketones, triterpenoids or other markers could be identified in the samples (Fig 7.3).

Selective ion monitoring for aquatic and heating biomarkers were positive. Overall the assemblage displays multiple co-occurrences of different aquatic biomarkers, including TMTD, pristanic and phytanic acids, and APAA-$C_{18:0}/C_{20}$ (Hansel *et al.*, 2004; Evershed, 2008). 1/5th of the samples presented with the full suite of aquatic biomarkers, including a favourable SRR%. The remainder fell short of the SRR% threshold, but still possessed all three isoprenoids. Only sample NOR-4 shows all the necessary markers to conclude the assemblage was involved in heating marine oil (Table 7.1). Despite this, we propose that the assemblage was involved in processing aquatic resources.

Table 7.1. Results from the AQUASIM scan. Key: TMTD, 4,8,12-trimethyltridecanoic acid; SSR, 3S,7R,11R,15-phytanic acid; RRR, 3R,7R,11R,15-phytanic acid; SRR%, ((Area SSR/Area SSR+Area RRR)*100).

Sample	TMTD	Pristanic acid	Phytanic acid	APAAs
			SRR%	
NOR-1	present	present	28.3	absent
NOR-2	present	present	23.7	absent
NOR-3	present	present	/	absent
NOR-4	present	present	90.6	$C_{18:0}$ $C_{20:0}$ trace
NOR-5	present	present	43.2	absent

To further examine the origins of the lipids present in the assemblage, carbon stable isotope values were acquired using GC-I-IRMS. Two fatty acid methyl esters - $C_{18:0}$ and $C_{16:0}$ - were targeted for analysis, and the results are presented in Figure 7.5.

The Klakken stones had a very low lipid concentration, but nevertheless showed a range of SFAs from $C_{9:0}$ - $C_{30:0}$, dominated by $C_{16:0}$ and $C_{18:0}$ peaks (mean C_{16}/C_{18} = 1.2). Small amounts of UFAs were present, including $C_{16:1}$, $C_{18:1}$, $C_{20:1}$ and $C_{22:1}$ isomers. BCFAs were abundant, but the typical $C_{15:0}$ and $C_{17:0}$ iso and anteiso species were absent. Long-chain dicarboxylic acids were identified, including $C_{18:0}$, $C_{22:0}$ and $C_{30:0}$. Dicarboxylic BPCAs were present in all the samples, as were oxygenated compounds such as 2-hydroxy tetracosanoate and 3-hydroxy octadecanoic acid. The BPCAs suggest that the stones were subject to or in close proximity to a heat source, and the long-chain saturated fatty acids, abundant dicarboxylic acids and long-chain unsaturated fatty acids might indicate that

foodstuffs were being cooked or processed with the aid of the stones. However, without more definitive biomarkers this remains speculative.

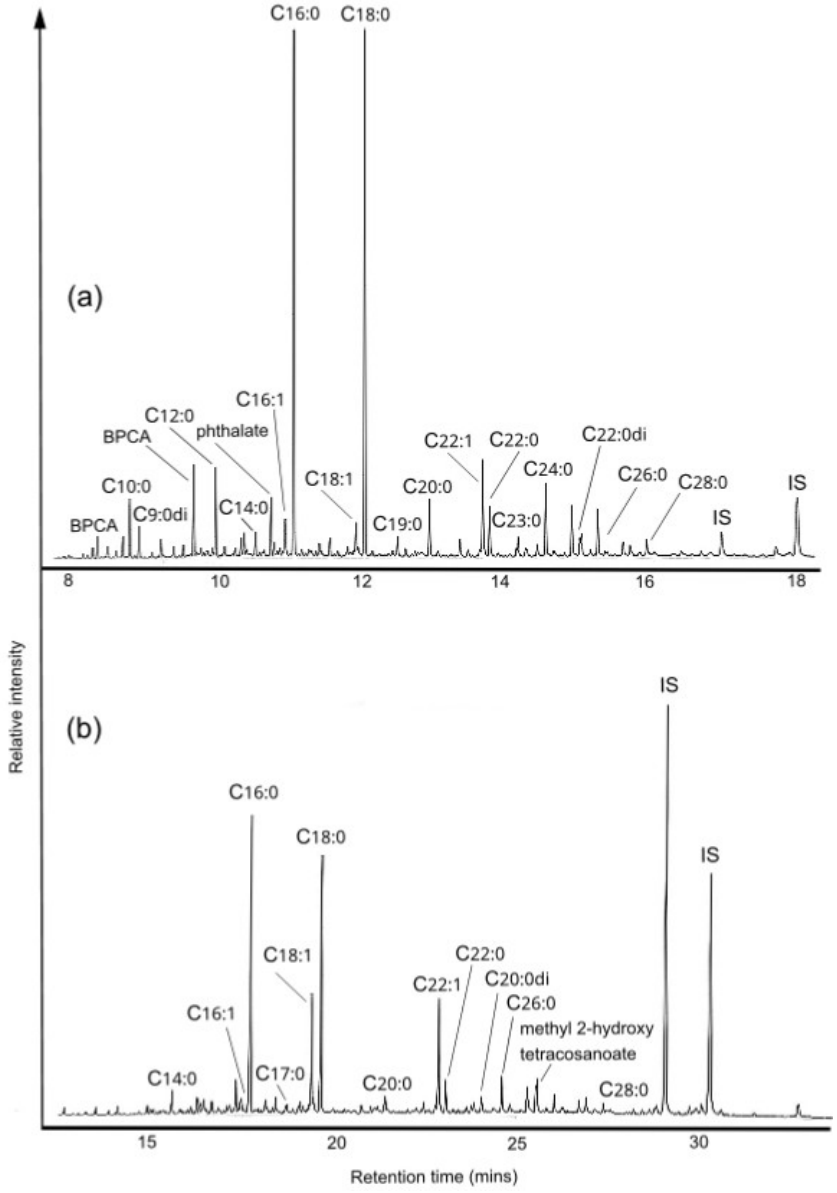

Fig 7.3. Labelled chromatograms for (a) sample NOR-5, and (b) sample KLK-3A

3.3 Neustadt heated slabs

The FCR assemblage from Neustadt presented with the greatest quantities of lipids, with significant variations of concentration across the stone surface. Many of the stones possessed dark, sometimes glossy and superficial, burnt patches or sections. The lipid concentrations between these darker sections and the lighter were significantly different (mean sample = 74.6 μg g^{-1}, mean control = 21.8 μg g^{-1}, paired t-test: t = 4.3186, *p* =

0.00099), suggesting that lipid absorption was not uniform, but more greatly concentrated in the stone matrix beneath the darkest patches.

GC-MS identification of the lipids showed the majority of the saturated fatty acids ranged between $C_{12:0}$ and $C_{24:0}$, dominated by $C_{16:0}$ and $C_{18:0}$. The mean C_{16}/C_{18} ratio was 1.2. Trace amounts of $C_{18:1}$ unsaturated fatty acids were detected, but otherwise very few UFA/BCFA species were identified. Other species present in the assemblage included methyl dehydroabietate (69% of samples) and dicarboxylic BPCAs (72%). One stone was identified as having a high amount of triterpenoid and polyaromatic hydrocarbon compounds. Sample R02 displayed peaks of 3-oxoallobetulane and other possible compounds such as lup-20(29)-en-3-one, betulin and betulinic acid (Fig.7.4) which are consistent with the thermal decomposition of birch bark and the production and subsequent degradation of birch-bark tar. However, since the sample was extracted with acidified-methanol the absolute identity of these compounds has been difficult to ascertain. Due to limited sample access, we were unable to perform a solvent extract on NST-R02, so instead conducted a further set of acidified-methanol extractions on three experimentally derived birch-bark tars to confirm that sample NST-R02 contained traces of birch-bark tar (Appendix 6.3).

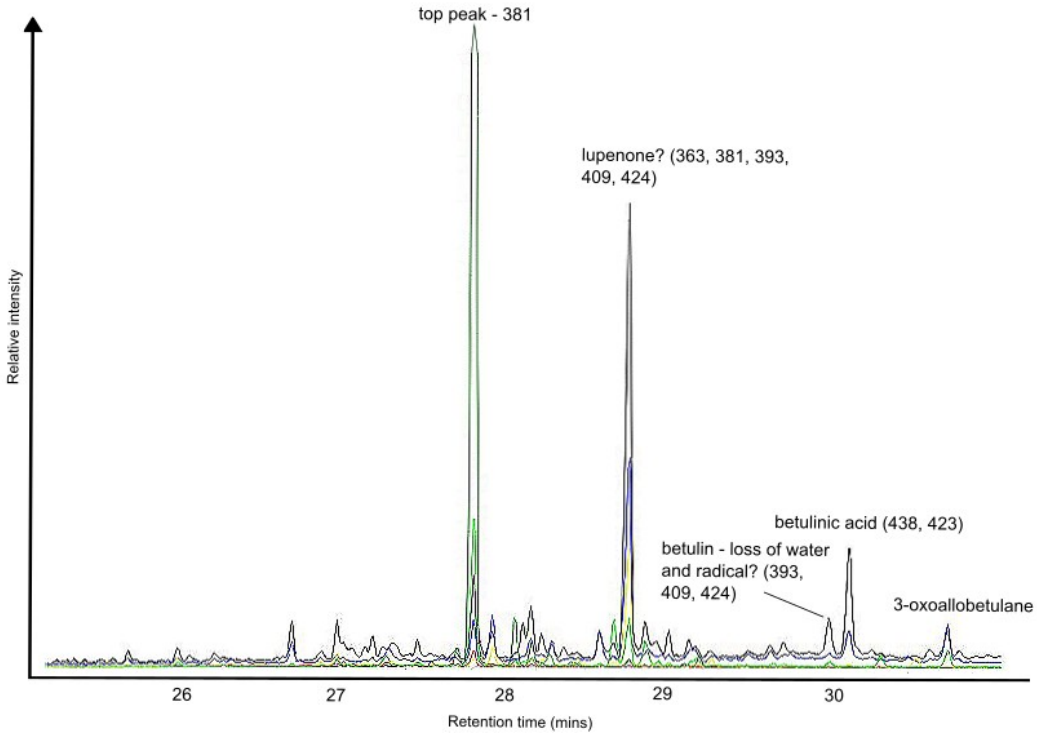

Fig 7.4. Partial chromatogram of the NST-R02A sample displaying ions 189 M^+, 203 M^+, 363 M^+, 381 M^+, 393 M^+ and 409 M^+. Identified and partially identified compounds have been labelled.

The acidification of the numerous terpenoid compounds has made many of them difficult to positively identify, however, the heating biomarker 3-oxoallobetulane has been labelled on the R02A sample, along with similar betulin, lupenone and amyrine related molecules. Along with numerous PAH species, there is a case for R02A being involved in producing birch-bark tar. However, it is difficult to infer whether this was intentional, or merely a by-product of heating birch wood and bark over the stone, as fuel or perhaps the accidental combustion of a birch bark cooking vessel.

The assemblage was also subject to a selective ion scanning, searching for APAAs and isoprenoids. Of the 29 samples tested, six were positive for APAA-$C_{18:0}$ isomers - of these only sample R02A possessed almost the full suite of isomers. The absence of TMTD, variable SRR% and small amounts of APAA-$C_{18:0}$ means that a definitive interpretation is difficult to offer.

Table 7.2. Results from the AQUASIM scan. Key: TMTD, 4,8, 12-trimethyltridecanoic acid; SSR, 3S,7R,11R,15-phytanic acid; RRR, 3R,7R,11R,15-phytanic acid; SRR%, ((Area SSR/Area SSR+Area RRR)*100).

Sample	TMTD	Pristanic acid	Phytanic acid	APAAs
			SRR%	
RO2A	absent	present	71.3	$C_{18:0}$
RO2B	absent	present	0	absent
RO3B	absent	present	0	absent
RO4A	absent	present	0	$C_{18:0}$
RO4B	absent	present	0	absent
RO5A	absent	present	0	absent
RO5B	absent	present	0	absent
RO6B	absent	present	0	absent
RO8B	absent	present	0	$C_{18:0}$
RO9B	absent	present	29.0	absent
R10A	absent	present	68.5	absent

R10B	absent	present	0	absent
R11B	absent	present	77.4	$C_{18:0}$
R12B	absent	present	/	$C_{18:0}$
R13B	absent	present	0	absent
R14A	absent	present	57.8	absent
R14B	absent	present	65.8	$C_{18:0}$
R14C	absent	present	0	absent

Carbon stable isotope ($\delta^{13}C$) values for $C_{16:0}$ and $C_{18:0}$ fatty acids were obtained from 17 FCR samples (n = 29 samples) plotted with reference ranges (95% CI) using previously reported values from modern animal fats (Dudd, 1999; Craig *et al.*, 2011, 2012; Cramp *et al.*, 2014; Lucquin *et al.*, 2016; Pääkkönen, Evershed and Asplund, 2017; Courel *et al.*, 2020). The $\delta^{13}C$ values revealed mixed-origins for the $C_{16:0}$ and $C_{18:0}$ fatty acids, with most plotting within freshwater fish fat ranges and some overlap with porcine/marine and ruminant fats. When plotted against previously reported $\delta^{13}C$ values for EBK and TRB pottery (Craig *et al.*, 2011; Courel *et al.*, 2020) it is clear that there is little to no overlap with the later TRB vessel values, and some overlap with the EBK, indicating the pots and the FCRs may have been used for processing different foodstuffs.

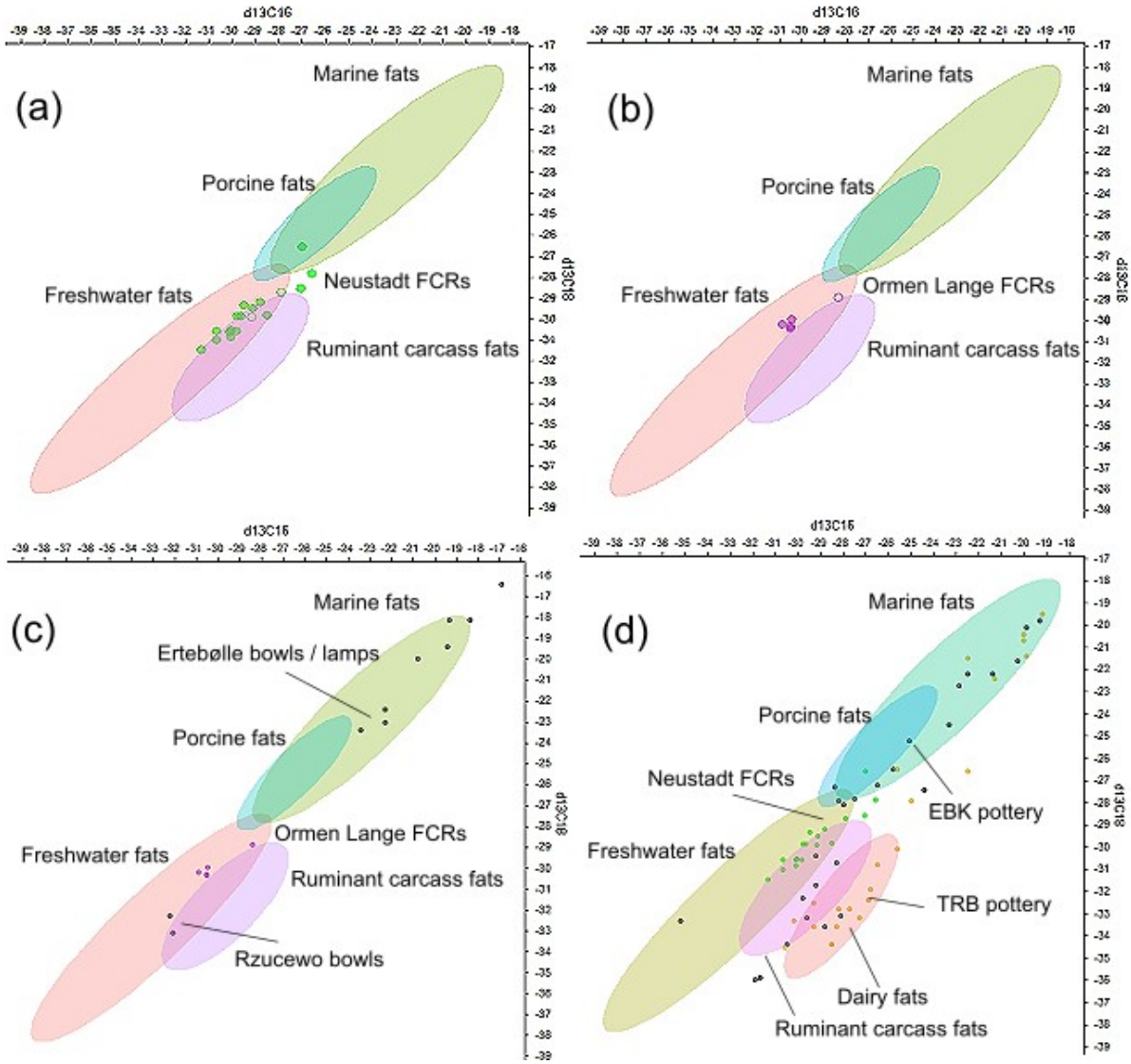

Fig 7.5. Archaeological FCR $\delta^{13}C$ values plotted alongside modern animal fat reference ranges (in text). (a) Neustadt samples - open circles indicating the presence of APAA-$C_{18:0}$. (b) Ormen Lange samples (n = 5) - open circles indicating the presence of APAA-$C_{18:0}$. (c) Ormen Lange samples plotted alongside previously reported $\delta^{13}C$ values for Mesolithic and Subneolithic lamps/bowls - (Heron *et al.*, 2015; Robson *et al.*, 2022). (d) Neustadt FCRs plotted alongside EBK and TRB pottery (in text). All reference values plotted with 95% confidence intervals.

3.4 Havnø and Visborg midden stones

The assemblage presented with a wide range of saturated fatty acids (Fig 7.6), from $C_{8:0}$-$C_{30:0}$, with dominant peaks at $C_{14:0}$, $C_{16:0}$, $C_{18:0}$ and $C_{20:0}$. The C_{16}/C_{18} ratio ranged from 1.2 to 4 (mean = 1.9), suggesting that terrestrial animal fats dominated over plant fats. A number

of monounsaturated fatty acids were also identified, including isomers at $C_{16:0}$, $C_{18:0}$, $C_{20:0}$ and $C_{22:0}$. Some more unusual species included $C_{16:1}$ *trans* 11, $C_{16:1}$ *trans* 7 and $C_{10:1}$ *trans* 2. Branched chain fatty acids were also found in every sample, most prominently at C_{15} and C_{17}. The range of *n*-alkanes spanned C_{11}-C_{28}, with no dominance of odd-chained alkanes. Most significant were the dicarboxylic acids - not only for their abundance, but also their length. $C_{18:0}$, $C_{22:0}$ and even $C_{30:0}$ dicarboxylic acids were identified in five out of the eight sample stones. Also present were various *n*-alkanols, 2-hydroxy and 9,10-dihydroxy fatty acids, terpenes such as sigmastanol and olean-13(18)ene, keto acids and methyl dehydroabietate (S.1).

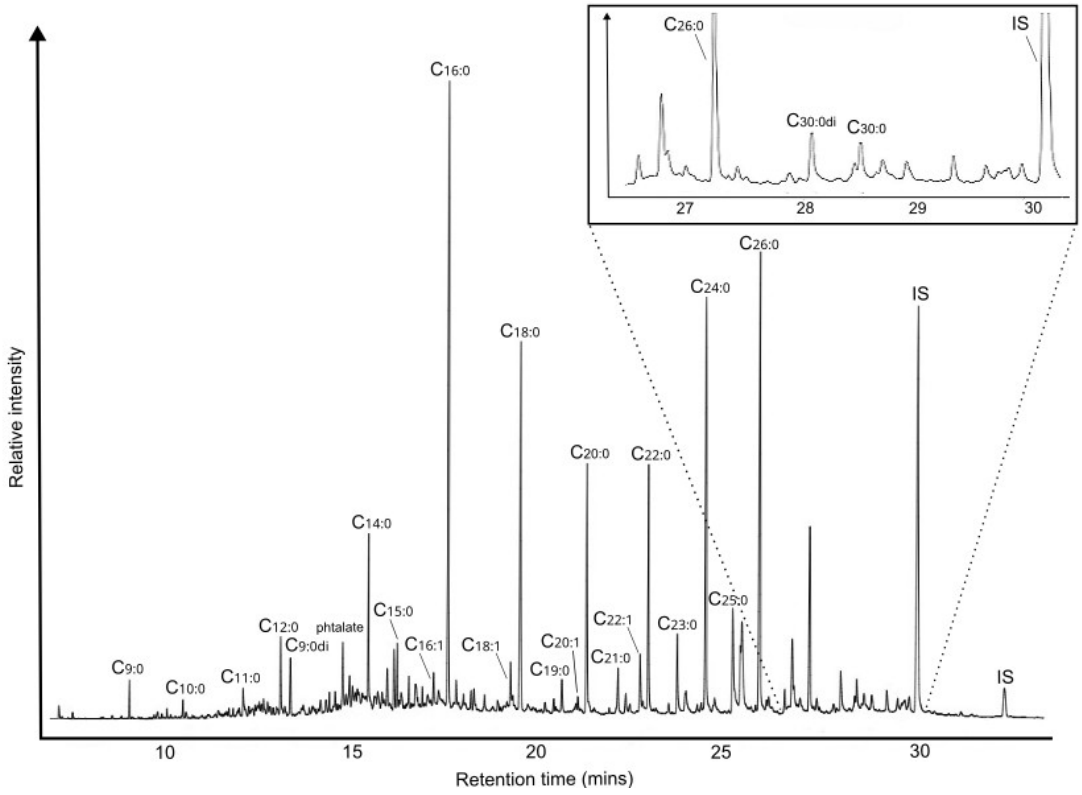

Fig 7.6. Chromatogram of sample HVN-2A including an enhanced insertion displaying the $C_{30:0di}$ peak.

No APAAs or other heating biomarkers such as gamma-lactones or long-chain ketones were detected in the stones, with the exception of small amounts of 1,2 BPCAs in samples HVN-1 and HVN-3. The alkanols present were either formed from $C_{12:0}$, $C_{16:0}$, $C_{18:0}$, $C_{20:0}$ or $C_{24:0}$ fatty acids, including some primary alcohols such as 1-hexadecanol and 1-eicosanol. Similarly a variety of hydroxy acids were identified, all deriving from $C_{14:0}$, $C_{16:0}$, $C_{18:0}$, $C_{22:0}$ and $C_{24:0}$ fatty acids. Interpretations of the Havnø assemblage are complicated by

the large number of oxylipin products in the samples, but without any corresponding thermal biomarkers. Three points in particular stand out: the relatively large amount of $C_{9:0}$ dicarboxylic acid; the mixture of $C_{14:0}$-$C_{30:0}$ dicarboxylic acids, hydroxy acids and alkanols; the presence of a $C_{30:0}$ or branched $C_{30:0}$ dicarboxylic acid. This could indicate both a high level of $C_{18:0}$ unsaturated fatty acid, as well as intrusions from plant-derived compounds such as suberin and cutin (Kolattukudy, 2001; Graça and Santos, 2007; Pollard *et al.*, 2008; Li-Beisson *et al.*, 2016). The presence of a $C_{30:0}$ dicarboxylic acid specifically could indicate the preservation of ancient *Equisetum* species, since their spores contain a number of $>C_{30:0}$ dicarboxylic acids (Řezanka, 1998). The null hypothesis would indicate that the assemblage has preserved naturally occurring plant lipids which have their origins in the long term infiltration and decomposition of organic midden materials.

The Visborg assemblage came with the opportunity to sample the surrounding soil and midden accretions which had become encrusted on the surface of the stones. This material was composed of soil, organic matter and broken sections of mussel or oyster shell. Two samples were taken, one with substantially more shell material, and the lipids extracted without use of acidified methanol. The combined results demonstrated that, although the stones contained relatively larger amounts of SFAs, the majority of the lipid types overlapped between the soil/shell and stone. There were some differences: only stone AHM-1 contained any dicarboxylic acids, only the stones had any fatty acids $< C_{14:0}$ or $C_{27:0}$ and the range of UFAs was greater in the stones. However, no samples showed any APAAs or other heating biomarkers and all contained a wide range of BCFAs, making any anthropogenic biomarker difficult to positively identify.

4.0 Discussion

The use of organic residue analysis to extract lipids from FCRs is still in its infancy. Although previous studies have been conducted (Quigg *et al.*, 2001; Buonasera, 2005, 2007, 2013, 2016; Lucquin, 2007; Namdar, Stacey and Simpson, 2009; García-Piquer *et al.*, 2018), there has been nothing like the sustained interest seen for ceramics. Consequently we cannot be so confident in our interpretations of FCR derived lipids and compounds, and must rely on robust biomarkers for anthropogenic activity in order to demonstrate that FCRs were involved in cooking or food processing. In this study of six Mesolithic-Neolithic sites, we have been able to show that APAA-$C_{18:0}$ has been formed through the heating of stone with

foodstuffs. We have also been able to show that birch bark was heated sufficiently to yield birch tar, and that BPCAs can be trapped within the matrix of heated stones - these three biomarkers are sufficient to demonstrate thermal activity and cooking/heating.

Of the six assemblages analysed, two add new insights to the archaeological discussion: Neustadt and Ormen Lange. The stone slabs found at Neustadt have been the subject of other studies, examining their use as tools involved in the processing of foodstuffs (Holst, 2023; Holst *et al.*, 2024). Alongside this we have a rich dataset related to the ceramic transformation, processing and storage of foods throughout the late Ertebølle/early Funnelbeaker transition period at Neustadt and the wider region, suggesting a continuity of aquatic and marine food exploitation, despite the rapid population turnover as displayed by the DNA evidence (Hartz and Lübke, 2006; Glykou, 2017; Courel *et al.*, 2020; Lucquin *et al.*, 2023; Allentoft, Sikora, Refoyo-Martínez, *et al.*, 2024). Our results are consistent with Holst's (2023) interpretation that the heated slabs at Neustadt were used to line some kind of oven structure, perhaps used on more than one occasion to cook different foodstuffs. The presence of PAHs, BPCAs and heated triterpenoid markers suggest that a fire was lit on top of the slabs, most likely as a way of heating them before they could indirectly transfer that heat to any kind of foodstuff. It is possible that the birch tar identified in sample RO2A was accidentally produced as part of this initial heating process – but it is also plausible that the stones were used to intentionally distil birch-bark tar, which would make this assemblage one of the first directly evidenced aceramic tar manufacturing systems for the late Mesolithic/early Neolithic. Aceramic birch bark tar production has received extensive attention in both the experimental and bioarchaeological literature , but the archaeological evidence for tar manufacturing methods across the northern European Mesolithic has been extremely limited (Osipowicz, 2005; Pawlik, 2011). Research focus on 'reverse engineering' birch tar production methods by cross-comparing specific lipid biomarkers between experimentally derived and archaeological tar samples has yielded some success in identifying particular distillation techniques (Rageot *et al*., 2019; Stacey *et al*., 2020; Kozowyk *et al*., 2023; Koch *et al*., 2024). However, this approach continues to be elusive as more tar samples have been analysed (Chasan *et al*., 2024). The Neustadt samples will need to be re-examined using a solvent based extraction technique in order to make a more rigorous comparison and to draw any conclusions on the specific conditions under which this tar was formed.

Single-compound isotope analysis of the $C_{16:0}$ and $C_{18:0}$ found in the Neustadt slabs does not point definitively to one specific origin for these fatty acids; when placed against isotope data from pottery from the same site, there is no clear overlap between them. Along with the absence of any marine/dairy signal, this suggests FCRs might have been utilised in a different way to the pottery, potentially pointing to multiple, separate, cooking and food processing methods during this transitional period. This demonstrates the wider applicability of organic residue analysis to non-ceramic artefacts whilst also providing evidence for the continuation of aceramic cooking and food processing technologies during periods when pottery was available. However, only 58% of the samples possessed sufficient quantities of $C_{16:0}$ and $C_{18:0}$ fatty acids to be analysed isotopically, with no clear correlation between total lipid quantity and single compound quantity. This suggests that many Neustadt samples contained high levels of background lipids – via contamination from the marine environment and combustion by-products – which may have affected the overall result.

Results from Ormen Lange similarly demonstrate how organic residue analysis can be used, even where no pottery exists. Previous speculations about the functions of the cobble-lined hearths on the site have tended to point to blubber as the likely fuel, a hypothesis which we can lend weight to with our results. Although only one sample was definitive, the presence of APAA-$C_{18:0}$ isomers, the long-chain fatty acids, marine-derived isoprenoids and the phytanic acid diastereomer ratio all indicate that the stones were in contact with thermally altered marine oils. Interestingly though, the $C_{16:0}$ and $C_{18:0}$ isotope values for the Ormen Lange stones do not fall within the established ranges for seal or marine foods. The shift towards $\delta^{13}C$ enrichment by the sample with APAAs suggests that more samples from different areas of the hearth may be needed to better characterise the lipid patterns. That said, the $\delta^{13}C$ depletion across the whole assemblage is unexpected. The combination of marine oil biomarkers and $\delta^{13}C$ depletion points towards a marine animal with a greater than typical amount of freshwater fish in the diet. Such a suggestion has been made for the oval bowl/lamp artefacts found across a number of Mesolithic/sub Neolithic sites (Heron *et al.*, 2015; Robson *et al.*, 2022). It is possible that small communities of seals feeding in the bays and inlets around the fjords were seasonally consuming quantities of freshwater prey, which would account for the ^{13}C results seen in figure 7.5. Further sampling and comparisons between bulk and single-compound ^{13}C isotope analysis would help confirm the role of marine oils at Ormen Lange and other Norwegian coastal sites during the Mesolithic.

Aceramic cooking practices would have been the norm prior to the invention and adoption of ceramic cooking vessels, but so far our knowledge of these methods during the European Mesolithic remains limited and speculative. Our study demonstrates that organic residue analysis, a method which has been largely focused on pottery, can be extended to FCRs. This presents an expansion in our ability to interpret and analyse biomolecular data from prehistoric sites, as FCRs can be included along with soils from hearths, food crusts, pottery sherds and other reservoirs of trapped or retained archaeological compounds.

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Chapter Eight: Conclusions

1.0 Introduction

The final conclusions from the previous chapters will be presented in this chapter. The original aims and research questions (Chapter 1) will be reassessed in order to provide structure. Summaries from the conclusions of each chapter will be outlined and broadened to demonstrate how the overall aim of the thesis has been fulfilled, what challenges were faced and what future research is needed.

The scope of the thesis in its entirety is as follows: **to evaluate the potential for aceramic cooking technologies to be analysed by adapting methodologies previously developed to understand ceramics, with an archaeological focus on the northern European Mesolithic.**

To tackle this the thesis made use of a combined methodological approach, employing ethnoarchaeology (Chapter 2), experimental archaeology (Chapter 4) and organic residue analysis, both experimentally (Chapter 6) and with archaeological artefacts (Chapter 7). The results fulfil the final research questions of the thesis:

- Is it possible to examine the performance of aceramic cooking vessels through experimental archaeology and comparisons to ceramic analogues?
- Does stone behave in a similar way to ceramics with regards to specific heating biomarker formation? If not, then what are the differences?
- Can the anthropogenic use of archaeological fire-cracked rocks be demonstrated through organic residue analysis?
- Can the results from the above help contribute to the wider questions of how aceramic technologies functioned and the development and adoption of ceramics by certain forager groups?

1.1 Is it possible to examine the performance of aceramic cooking vessels through experimental archaeology and comparisons to ceramic analogues?

The introduction to Chapter 4 highlighted the need to investigate and analyse the development and adoption of ceramics by hunter-gatherer communities, crucially by focusing on the performance and functionality of the preceding *aceramic* cooking technologies. As highlighted through a quotation from Jordan and Zvelebil (2016, 57) - "With many organic technologies able to perform the roles played by pottery, what, other than direct boiling ability, might have made pottery more attractive?" - the prevailing presumptions that aceramic technologies were incapable of sustained boiling, and that boiling is necessarily useful and desirable, were challenged through a series of actualistic cooking experiments. The conclusions reached included a) some aceramic methods were capable of raising the temperature of enclosed water to boiling point; b) that reaching and maintaining boiling point is unnecessary and undesirable for processing and cooking many foodstuffs.

By calculating the theoretical and experimental temperature curves for heating water in an unprocessed red deer hide, it was possible to quantify the expected vs actual differences for temperature gain and loss of an aceramic and ceramic vessel. This revealed that the aceramic deer hide container performed more poorly than the ceramic for conductivity, but possessed greater insulative properties. The crucial physical characteristic here is the thermal conductivity of collagen versus ceramic (κ_{skin}=0.7 $W \cdot mK^{-1}$ for collagen, κ_{skin}=2 $W \cdot mK^{-1}$ for ceramics) which required larger amounts of energy to heat the water inside the skin, but the heat loss was slower during the cooling period. The outer layer of keratin was burnt away, so was not included in the calculation. The data from all six experimental vessels confirmed that the *indirect* heating method of adding hot stones to an aceramic container of water resulted in a rapid temperature rise, while the *direct* heating method of placing an aceramic container over a heat source was not an efficient way of transferring heat quickly into the vessel, nor of raising the internal temperature to boiling or near-boiling point.

However, as discussed in the conclusions to Chapter 4 - boiling is only one physical threshold of many that would have been useful for processing foodstuffs. Rather than a linear model of temperature, the incorporation of time, through the '*long-time, low-temperature'* (LTLT) cooking method (Latorre et al. 2019; Paul 1963; Bertola et al. 1994), opened up the possibility that sub-boiling temperatures were desirable and, in some cases, necessary. Therefore, in contrast to some previous assumptions, it may have been precisely this need to utilise lengthy sub-boiling cooking methods which helped drive the invention and subsequent adoption of ceramics during the Upper Palaeolithic. Skibo et al (2009) had already discussed this possibility, but in a more limited chronological framework, noting that the transition from using boiling stones to pottery during the Archaic and Woodland periods could have been motivated by the need to *reduce* the temperature from boiling to "simmering", in order to more efficiently render fat from nuts and bones. Skibo and Schiffer's work on quantifying and characterising ceramic technologies (Schiffer and Skibo, 1987; Skibo and Schiffer, 1987; Skibo, Schiffer and Reid, 1989; Skibo, 2013, 2015) provides an ideal foundation to develop a similar set of experiments to better understand *aceramic cooking technologies*. Indeed, the ceramic analogue comparison used in Chapter 4 was drawn from such available literature, where quantitative data on the conductivity and heat capacity of different ceramics and their tempers has been published, and can be used to test theoretical heat models against experimental data.

This conclusion demonstrates the need for more engagement with aceramic cooking technologies, to challenge latent assumptions about their capacities and performance. There were limitations with this study however, primarily that the materials chosen to be tested were all derived from animals. As Chapter 2 shows, the ethnographic record is replete with examples of plant-based aceramic cooking containers, including wooden boxes, troughs, birch-bark containers, canoes and baskets. Testing their functionality and performance through actualistic experimentation may reveal significant differences to the animal materials, both through direct and indirect heating methods. The overall results from this chapter satisfy the original research aims and question which was to evaluate the possibility of studying aceramic cooking technology through experimental archaeology and by so doing, compare the results to ceramic analogues. Through this series of experiments it has been possible to present new quantitative and qualitative evidence for the functionality and performance of animal-derived cooking vessels, and make an original contribution to the literature on prehistoric cooking and experimental archaeology.

1.2 Does stone behave in a similar way to ceramics with regards to specific heating biomarker formation? If not, then what are the differences?

An overview of the literature examining organic residue analysis in stone makes up the second half of Chapter 5, and a series of experiments assessing thermal biomarker formation in heated stones are presented in Chapter 6. Many of the early studies examining the preservation and origins of lipids within FCRs came from archaeological sites around the American southwest (Quigg *et al.*, 2001; Buonasera, 2005, 2007, 2016), these typically employed compositional lipid ratio analysis and experimental food datasets to interpret any recovered fatty acids. The identification of specific biomarkers associated with anthropogenic heating has only occurred in a few studies: long-chain ketones in cooking stones (Lucquin 2007); long-chain ketones in stone cooking vessels (Namdar, Stacey and Simpson, 2009) and isoprenoids in FCRs (García-Piquer *et al.*, 2018). The specific questions of how particular heating biomarkers form in stone, and how that formation might be affected by the type of stone and the heating conditions, have therefore been underexplored compared to the same questions in ceramics.

The experiments undertaken in Chapter 6, and their results, have aimed at the initial characterisation and exploration of heating biomarker formation in stone. Previous work has varied greatly in the specific methods employed for stone sampling, lipid extraction and molecular analysis - the methodology utilised for Chapter 6 was borrowed from organic residue analysis in ceramics, in particular the use of acidified methanol to recover lipids and organic molecules, GC-FID and GC-MS to quantify and identify any lipids and other compounds and selective ion monitoring to identify isoprenoids and cyclic alkanoic acids. The three experiments undertaken using this approach - two actualistic cooking experiments and one closed-system laboratory experiment - demonstrated that stones, even when heated for short periods of time, were capable of forming three heating biomarkers: APAAs, ACPAAs and BPCAs. Of these, the isomers of APAA-C_{18} appeared the most reliable and robust.

Current understandings of APAA formation in ceramics has developed in part through the inference that tri-polyunsaturated fatty acids form APAAs through protracted heating, which would point to marine oils as the most likely archaeological source (Copley *et*

al., 2004; Hansel *et al.*, 2004). This interpretation has since been refined (Evershed *et al.*, 2008; Admiraal *et al.*, 2019) and current quantitative thresholds are now employed which can differentiate between food sources (E/H ratio, APAA C_{20}/C_{18}) (Bondetti et al. 2021). The results from Chapter 6 however are not entirely consistent with these thresholds, and the isomeric distribution of APAA-C_{18} from both heated sandstone and granite does not align with those of ceramics (Fig 8.1).

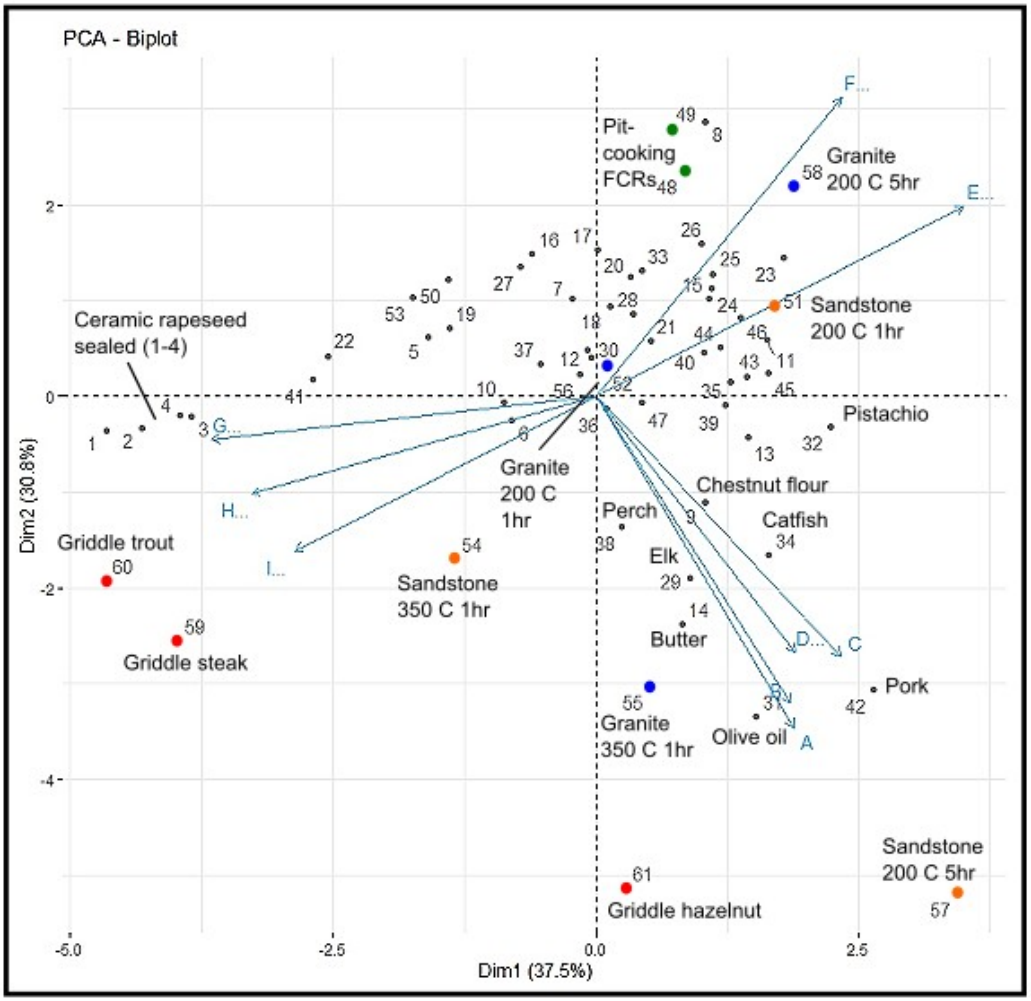

Fig 8.1. PCA plot of the APAA-C_{18} isomer distribution incorporating all three experiments from Chapter 6 as well as those from Bondetti et al 2021. *1-4. Bondetti rapeseed ceramic sealed; 5-8. Bondetti rapeseed ceramic unsealed; 10. Cod liver oil ceramic 270 C unsealed 5hrs; 11. Deer fat ceramic 270 C unsealed 5hrs; 12. Hazelnut oil ceramic 270 C unsealed 5hrs; 13. Hemp oil ceramic 270 C unsealed 5hrs; 15. Rice bran oil ceramic 270 C unsealed 5hrs; 16-17. Salmon ceramic 270 C unsealed 5hrs; 18. Walnut oil ceramic 270 C unsealed 5hrs; 19-20. Leek leaf ceramic 270 C unsealed 5hrs; 21. Onion ceramic 270 C unsealed 5hrs; 22. Cabbage ceramic 270 C unsealed 5hrs; 23. Almond ceramic 270 C unsealed 5hrs;*

24. Walnut ceramic 270 C unsealed 5hrs; 25. Wheat ceramic 270 C unsealed 5hrs; 26. Barley ceramic 270 C unsealed 5hrs; 27-28. Carrot ceramic 270 C unsealed 5hrs; 30. Duck ceramic 270 C unsealed 5hrs; 33. Red deer ceramic 270 C unsealed 5hrs; 35. Sturgeon ceramic 270 C unsealed 5hrs; 36-37. Shellfish ceramic 270 C unsealed 5hrs; 39. Viburnum ceramic 270 C unsealed 5hrs; 40. Beaver ceramic 270 C unsealed 5hrs; 41. Spinach ceramic 270 C unsealed 5hrs; 43. Millet ceramic 270 C unsealed 5hrs; 44. Quinoa ceramic 270 C unsealed 5hrs; 45. Rice ceramic 270 C unsealed 5hrs; 46. Sesame seed ceramic 270 C unsealed 5hrs; 47. Acorn ceramic 270 C unsealed 5hrs; 52. Langley rapeseed granite 200 C 1 hr

Despite the methodology used for the laboratory experiment closely following that of Bondotti et al (2021), and the sandstone used for the laboratory experiment and the griddle experiment coming from the same source - the APAA-C_{18} isomer distributions do not follow a straightforward pattern. In part this may be due to the unexpectedly low levels of the H-isomer found in two sandstone and one granite sample (LBX-1B, LBX-3B, LBX-3C). Without more repetitions of the experiment it is difficult to know why this occurred, but neither Bondotti et al (2021) nor Evershed et al (2008) reported similarly skewed E/H ratios despite heating rapeseed oil under similar conditions. Another factor to consider is the difference between types of ceramics. The composition of the clay, temper and any slip or post-firing modifications may alter the formation of particular biomarkers, in particular where metal salts are likely to be catalysts to compound formation (long-chain ketones) (Raven *et al.*, 1997). Since this study carefully replicated Bondetti et al (2021) where possible, the ceramics employed were identical, and future research may need to test biomarker formation in different types of ceramics to prove whether the stones are outliers, or whether they fall within the natural variation of pottery vessels.

A final way in which FCRs appear to behave differently to ceramics is their differentiation by *cooking method*. In this thesis several examples of actualistic cooking practices have been presented, including aceramic vessels, stone griddles and earth-ovens/pit cooking. In the APAA-C_{18} isomer distribution PCR figure above, the griddle and pit-cooking stones are in distinct quadrants, which may simply reflect the different foods used during the experiment, or it may point to the method impacting upon the formation of the isomers. Despite identical sandstone being used for the controlled experiment and the griddle, there were very low levels of medium-chain fatty acids and an increase in *trans* isomeric PUFA products in the griddle stones. Griddling has its closest analogue in frying,

but the literature on lipid degradation and cooking by-products is concerned with contemporary deep-fat frying and the use of cooking oils in metal pans - ‘dry-frying’ on stone (or clay) has rarely been analysed either by archaeologists or food scientists. The different methods of cooking with stone (boiling, griddling, steaming) are often distinct from pottery, with the exception of steatite/chlorite-talc stone bowls and containers. Future research into any distinctive lipid ‘fingerprints’ for the various methods may reveal identifiable patterns to help distinguish possible archaeological FCR cooking methods.

In attempting to investigate the thesis aim of biomarker formation in stone, one anomaly persisted throughout the entire project - the absence of long-chain ketones. Only two ORA publications have reported identifying LCKs in FCRs or stone, and these were Lucquin (2007) and Namdar et al (2009) - these differed from the current methodological approach in two key ways: Lucquin’s sampling approach involved hundreds of grams of ground stone from FCRs, rather than the 1-4g used in this thesis, and other FCR ORA publications; Namdar et al were analysing recovered lipids from stone vessels, rather than FCRs. Specifically the LCKs found in that study came from the neck/rim of the vessel, an observation which has been repeated in ceramic vessels (Cramp, Evershed and Eckardt, 2012; Breu *et al.*, 2023). This may be due to increased temperatures in the upper portions of the vessel, where the liquid is not cooling the walls, and therefore reaches the 300 - 350 C needed to form the LCKs -the neck/rim typically also receives the highest amount of lipids due to the boiling/evaporation line leaving immiscible fats and organic compounds at the surface (Reber, 2022, 10, 40). It may therefore be the case that LCKs are not forming because of the composition of the stone, or because of the necessity for high sustained temperatures. Alternatively they are forming, but the sample size used in this thesis was too small to detect them. All efforts were made to recover them, including silylation, methylation, saponification, the use of both acidic-methanol and solvent extracts, and the use of acid/neutral fractionation. The extremely low levels of γ-lactone-C_{16} identified in the griddle stones suggested again that either the sample sizes were too small, that the cooking conditions were not optimal, or that the composition of particular stones are not favourable to their formation.

Overall the experimentation projects undertaken for the thesis have addressed and partially answered the original research question. The formation of typical biomarkers found in ceramics *can* occur in stone under particular conditions - however - there may be some

significant differences in the lipid profiles produced due to (a) the composition of the heated-stone, and (b) the exact cooking technique or method employed.

1.3 Can the anthropogenic use of archaeological fire-cracked rocks be demonstrated through organic residue analysis?

Unlike pottery, FCRs present a more ambiguous case for researchers to determine the degree to which they were utilised for cooking and food preparation. The association of stone with fire or heat does not *necessarily* indicate that it was involved in cooking. Therefore an additional burden exists when analysing FCRs to prove that any recovered lipids or organic molecules were directly associated with food preparation. False positives could include: lipids already present in the stone before selection for use; anthropogenic lipids that have *indirectly* entered the stone matrix; lipids that have entered the stone matrix after deposition and lipids or molecules that are a result of researcher contamination. Many of these problems are shared with ceramics, but FCRs have multiple other functions aside from cooking and food processing - heating adhesives; heating or steaming wood, antler, hide and other materials; saunas and steaming structures and indirectly heating domestic spaces to name but a few (Chapter 2 and 3). They may also have complex biographies, such as prior use as a grinding stone before being used for heat conduction. For some of these activities, such as processing and preparing resinous adhesives, well established biomarkers make it possible to confidently identify them in the archaeological record.

The results of Chapters 6 and 7 present several instances where clear anthropogenic activity could *not* be definitively shown (Rosenfelde, Havnø, Visborg, Klakken and several samples from the pit-cooking experiment). One reason is the low or near-absent lipid concentrations recovered from some samples. Organic residue analysis in ceramics has established a quantitative threshold of >5 micrograms of lipid per gram of ceramic in order to be confident that the recovered organic compounds are not mostly noise from contaminants (Evershed 2008). However, it is not possible at this stage to determine whether this threshold applies to FCRs. Table 8.1 Shows the mean lipid concentration from other FCR ORA studies, including from Chapters 6 and 7.

Table 8.1. Mean lipid concentrations and sample weights from several publications and experiments in this thesis.

Publication	Site	Source	Mean lipid concentration ($\mu g g^{-1}$)	Sample weight (g)	Extraction method	Analytical method
(Buonasera, 2007)	Northern California	Grindstones	24	0.5-2	Modified Bligh & Dyer, H_2SO_4 acid fraction	GC-MS
(Namdar, Stacey and Simpson, 2009)	Merv, Turkmenistan	Talc-chlorite schist vessels	83	2-5	Folch solvent extract	GC-MS
(Buonasera, 2016)	Gila Cliff Dwellings, New Mexico	*In-situ* grindstones	53	0.1-2	Modified Bligh & Dyer, HCl acid fraction	GC-MS
Thesis (Chapter 6)	Pit-cooking, actualistic experiment	FCR earth oven	19	4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Neustadt	FCR slabs	46	1-4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Rosenfelde	FCR hearth	2	1-4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Ormen Lange	FCR hearth	37	4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Klakken	FCR hearth	13	4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Havno	FCR midden	26	4	Acidified - methanol	GC-FID, GC-MS
Thesis (Chapter 7)	Visborg	FCR midden	13	4	Acidified - methanol, solvent extract	GC-FID, GC-MS

Evershed (2008) notes that, using a typical sample weight of 2 grams, the mean total lipid concentration for the average potsherd is 100 $\mu g g^{-1}$, which is higher than any of the mean totals presented in Table 8.1. Given that the starting sample weight from the FCRs is not substantially different for these results from the usual potsherd sample weight - it is reasonable to conclude that FCRs yield a lower average lipid concentration than archaeological ceramics. This may be because the porosity and internal matrix structure of pottery is more conducive to retaining lipids and small organic molecules, or it may be due to the differences in how FCRs are used. Ceramics have the capacity to enclose a hot liquid or semi-liquid for many hours, repeatedly, whilst FCR cooking techniques vary and may only be utilised once. One other factor to test is the lipid concentration differences between the two main sampling methods: "kinetic" and "coring". The sample set from Havnø contained 8 FCRs, some of which were sampled using a bench drill and some broken apart on a laboratory bench. However, there was no significant difference in lipid concentration between the two groups (two-sample t-test, $t = 1.6567$, $p = > 0.05$), suggesting that natural variation is the more important determining factor than the sampling method.

Interpreting lipid and organic molecular content from FCR samples <5 $\mu g g^{-1}$ total concentration may depend on the starting weight, and on whether any heating or specific compound biomarkers can be identified. Sample NST-4A (Nesustadt, Chapter 7) yielded a total lipid concentration of only 4 $\mu g\ g^{-1}$, yet also showed small amounts of three APAA-C_{18} isomers (E,F,G) (Appendix 6.4). Since the standard threshold of >5 $\mu g\ g^{-1}$ has been developed in the context of ceramics, it may be that ORA interpretations for FCRs need to be more flexible, and accept lower or more trace amounts of particular biomarkers. One problem to consider for future studies is that lower total lipid concentrations results in lower $C_{16:0}$ and $C_{18:0}$ levels available for stable isotope analysis. As was the case with this project, once levels of these two dominant SFAs are too low, confidence in the instrumentation to accurately measure $\delta^{13}C$ is reduced and the results cannot be relied upon for any reasonable interpretation of lipid origins within the samples. The minimum carbon quantity for $^{12}C/^{13}C$ sensitivity using a GC-C-IRMS instrument is around 0.1-5 nmol (Sessions 2006). Only 58% of the Neustadt samples yielded carbon quantities equal to or above this limit. Furthermore the standard deviations for both $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ ranged from 0.05% to 0.35%, suggesting that the assemblage was highly heterogeneous in $C_{16:0}$ and $C_{18:0}$ quantity and perhaps origins. The background lipid content and quantity did not necessarily correlate with the ability to subject the samples to compound specific stable isotope analysis - for example sample NST-4A contained 5 micrograms of lipid per gram of stone, but more than

the minimum amount for isotope analysis, yet sample NST-11A contained 104 $\mu g g^{-1}$ and could not be used for GC-C-IRMS. Future studies of FCRs should aim to use soil sediment controls where possible to quantify these background lipids in order to determine what should be the minimum interpretable lipid quantity per gram, and in order to compare bulk and compound-specific isotopes.

However, with the exception of Rosenfelde, the majority of the samples from the remaining sites yielded sufficient total lipid concentration to reasonably interpret their contents. A total of 7 samples (15% of the total sample set) showed some or all of the APAA-C_{18} isomers. One sample (NST-R02A) contained biomarkers consistent with heated birch-bark tar (3-oxoallobetulane, lup-20(29)-en-3-one, betulin and betulinic acid) and several more contained 1,2 and 1,3-BPCAs. Alongside these thermal biomarkers were a wide variety of degradative biomarkers, including α,ω-dicarboxylic acids, *n*-alkanols, hydroxy and dihydroxy acids and oxo acids. Making sense of these, as well as the range of saturated and unsaturated fatty acids identified in the FCRS, has been one of the challenges of this project. The wide variety of archaeological contexts (submarine refuse area, prehistoric midden, domestic hearths) precluded straightforward comparisons, and in the absence of identifiable anthropogenic biomarkers it has been difficult to infer about the origins of specific lipids. The lack of corresponding soil samples for most FCR contexts also prevented clarification as to whether degradative and other compounds (terpenes, long-chain diacids, branched-chain fatty acids) originated with human activity or with diagenetic processes.

In the final analysis one of the main aims of the PhD - to evaluate the potential for FCRs to be analysed by ORA - has been sufficiently addressed, even if the artefact assemblages did not all yield positive results.

1.4 Can the results from the above help contribute to the wider questions of how aceramic technologies functioned and the development and adoption of ceramics by certain forager groups?

This thesis has addressed the question of aceramic cooking technology functionality through several approaches, including testing an *experimental* vessel (see above) as well as utilising ORA to examine specific biomarker formation and stable isotope analysis within experimental and archaeological FCR artefacts. The use of established biomarkers in

particular draws on a form of bioarchaeological theory which exploits the link between chemical transformation and human activity (Evershed, 2008; Hansel and Evershed, 2009). In doing so it is possible to demonstrate to a high level of probability that the identification of a specific biomarker *necessarily* indicates that a particular activity occurred at the time of usage. For example, the discovery of APAA-C_{20} isomers in a pottery sherd indicates that a C_{20} unsaturated fatty acid was heated, causing a type of cyclic isomerisation that does not occur naturally. Other examples include abietic acid (Hertzog *et al.*, 2023), tartaric acid (Pecci *et al.*, 2013), betulin compounds (Koch *et al.*, 2024) and LCKs (Baeten *et al.*, 2013).

The step from proving an artefact contains lipids derived from anthropogenic activity to proving a more precise function or usage requires an integration of archaeological context, macro use-wear traces, lipid and organic molecule concentration and specific compound identification. In the case of both Neustadt and Ormen Lange, such an integration was successfully applied, and in both instances a functional interpretation was offered (Chapter 7). Conveniently the two cases represented markedly different functions, and in two very different archaeological contexts. For Neustadt the ORA results supported thermal FTIR analysis conducted by Holst (2023), which provided a range of estimated temperatures for some of slabs analysed in this thesis. The identification of APAA-C_{18} isomers and several betulin-derived compounds confirmed that both unsaturated fatty acids and birch bark (*Betula pendula*) had been sufficiently heated on top of the slabs to form these distinct biomarkers. For Ormen Lange, the identification of APAA isomers similarly confirmed that at least one cobble had been heated with unsaturated fatty acids, but the high chain-length of many of the fatty acids, combined with the presence of TMTD and phytanic acid diastereomers supported the hypothesis that the stone-lined hearths had been used by aceramic Mesolithic foragers to render/heat marine fats (Bjerck, 2017; Breivik, 2020). Thus for at least two archaeological sites a range of aceramic pyro-technological functions could be suggested which would be commensurate with the ORA, including: an earth-oven for processing plant/animal foods; aceramic adhesive production; marine oil rendering for food or burning; marine oil combustion to produce light and/or heat.

Archaeologically these results can be placed into the broader context of Mesolithic hunter-gatherer technology and the transition from EBK ceramic forager to TRB agricultural lifeways. The introduction of ceramics into the EBK occurred around 4,800 - 4,500 BC (Povlsen, 2013;), which saw centuries of potentially close interactions with southerly TRB communities, including the exchange or procurement of dairy products (Lucquin *et al.*,

2023). Circa 4,000 BC saw the beginning of the replacement of the EBK with the TRB, although the complex nature of the transition means that sites such as Neustadt are difficult to assign to one culture or the other (Glykou, 2011, 2016). Pottery vessels associated with both the EBK and TRB demonstrate the emerging mixed economy which incorporated marine foods, wild plants, ruminant animals and dairy (Craig *et al.*, 2011; Saul *et al.*, 2013). Stable isotope analysis of $C_{16:0}$ and $C_{18:0}$ fatty acids was undertaken for a number of Neustadt FCR samples, which revealed no distinct $\delta^{13}C$ enrichment patterns, but nor did they show any clear overlap with either EBK or TRB vessels (Chapter 7). The linear orientation of the slabs made them more likely to be used as griddles or as lining-stones for an oven or structured-hearth, rather than as boiling stones. Taken together, the contextual detail for the transitional site of Neustadt shows that a number of mixed cooking strategies existed alongside one another, a result supported by the recent ethnographic forager record (Chapter 2).

A likely possibility is that the slabs ultimately derived from an earth-oven or structured-hearth, perhaps reused on several occasions as a different but complementary cooking or food processing technique. Thus the introduction of pottery to the EBK represented an adoption and adaptation to the new technology, which did not necessarily replace all previous aceramic cooking techniques. The possibility that the slabs could have been used to distil birch bark tar is also intriguing, since aceramic tar distillation methods during the Mesolithic are not well understood (Osipowicz, 2005; Koch and Schmidt, 2021), but the use of ceramics to manufacture birch tar in the following Neolithic period is well established (Lucquin, March and Cassen, 2007; Rageot *et al.*, 2018; Urem-Kotsou *et al.*, 2018). If the slabs *were* used to manufacture birch tar during the late EBK/early TRB period, then it demonstrates the multimodal nature of ceramics but also cultural decisions not to replace older aceramic traditions with pottery. It could also indicate a lack of knowledge about the technological know-how to use ceramics for tar distillation.

It is clear then that ORA using FCRs can generate results which contribute to the wider questions of aceramic functionality and ceramic adoption by hunter-gatherers, in particular those along the Baltic coastline and southern Scandinavia during the mid-Holocene. Aceramic technologies, whether they are containers or heated stones, possess a wide range of possible functionalities, and their performance needs to be better understood within their specific archaeological contexts in order to properly characterise the transition

from aceramic to ceramic. ORA and experimental archaeology are well placed to test and explore these functionalities.

2.0 Methodological considerations

Several different methodologies were employed for this thesis, arising from distinct but overlapping subdisciplines and fields within archaeology. In the cases of ORA and experimental archaeology the aim was to expand and develop previous work to assess aceramic cooking technologies, and different techniques, ideas and methods were tested and refined over the course of the project. These of course need to be subject to criticism and review in order to assess their efficacy and highlight areas of weakness and opportunities for future improvement.

How best to take a sample of stone from a parent FCR slab or cobble was a major methodological challenge of the thesis, and several approaches were employed. Ceramic-based ORA has evolved over the decades and has seen the use of scalpels (Evershed, Heron and John Goad, 1990), modelling drills (Evershed *et al.*, 2002; Craig *et al.*, 2005), diamond-tipped drills (Longoni *et al.*, 2024) and other physical sampling techniques to extract a sample of pottery from a sherd, or remove a portion of burnt food crust. At the outset of the thesis it was decided that stone would present an additional challenge in that many FCR artefacts would either be too hard, or too friable to use a small handheld drill to extract a powdered sample. Additionally FCRs present with a variety of macro and micro morphologies - i.e, a linear slab shape or an oval/circular cobble, and internal crystalline grain structures and porosity differences which could not be anticipated.

Therefore two distinct sampling methods were pursued, dubbed ‘kinetic’ and ‘coring’ (Table 8.2). In addition, two grinding methods were used - the first was to reduce the sample to small enough pieces to grind using simply a pestle-and-mortar (successful, but broke several mortars), and the second was to use a bespoke combination of a metal pipe cap, metal screws and a hammer to reduce the sample to appropriately small enough pieces to grind in a mortar (only used once).

Table 8.2. The key characteristics of the different FCR sampling techniques used over the course of the project. More details can be found in Appendix 3.

Method	Tool(s)	Accuracy	Depth	Post treatment	Example sample sets
Kinetic	Hammer, pestle	Low	Shallow, irregular	Ground	Rosenfelde
Coring (hand)	Cordless drill, vice, water-bath	High	Deep	Linear sections, ground	Griddle
Coring (saw)	Diamond saw blade, vice, water-bath	Medium	Deep	Linear sections, secondary kinetic, ground	Neustadt
Coring (bench)	Modified bench-drill	High	Deep	Ground	Visborg

Ultimately the workflow involved several key parameters which determined the most appropriate method (Fig 8.2).

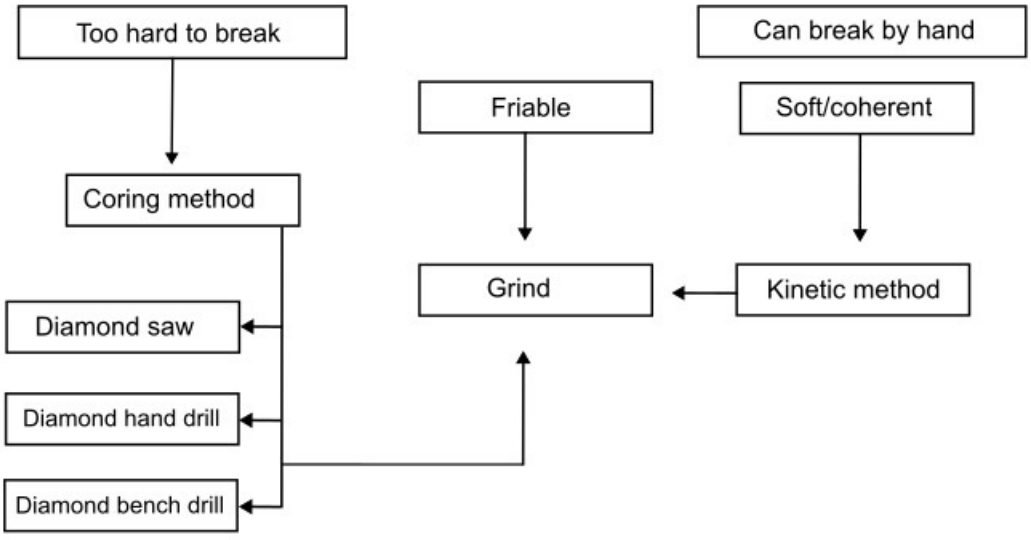

Fig 8.2. The workflow for sampling FCRs during this project

As discovered with repeat sampling, the total lipid concentration varied across many FCRs - in particular in the Neustadt samples, since they were linear slabs rather than

cobbles. Even so, where repeat sampling was performed, it would be difficult to point to the sampling method rather than inherent variation as the key factor driving differential lipid concentrations. As seen above, the Havnø samples were sampled using both kinetic and coring methods, and no statistical difference between the two was found with regards to lipid concentration.

However, it would be fruitful to better understand the depth of lipid penetration into FCRs for different cooking techniques, by taking sub-samples of core 'plugs'. This project also did not utilise surface grinding, rather just washing and cleaning. It would be worth testing if the top few millimetres of each FCR contains the most lipid content, or if soil and/or plasticiser contaminants could be reduced through removing the most superficial outer layer of stone..

Finally it is worth mentioning that there exists, to the best of my knowledge, no commercial or other drill rig which can stabilise and orient each sample towards the drill bit in order to remove a sample core. For this project a bespoke set-up was designed and made, but there is no standardised product which can be purchased by different institutions.

2.1 Actualistic experimentation

Experimental archaeology has a long history within the wider discipline, and has become increasingly more sophisticated with regards to experimental design, variable control and issues of experimenter skill (Coles, 1966; Callahan, 1999; Reynolds, 1999; Eren *et al.*, 2016; Lin, Rezek and Dibble, 2018; Eren and Bebber, 2019). For ORA, experimental archaeology has become a valuable tool to testing inferences, observing chemical reactions such as degradation, and simulating cooking scenarios in both controlled and actualistic settings (Charters *et al.*, 1997; Evershed *et al.*, 2008; Oras *et al.*, 2017; Huber *et al.*, 2022). However, for both ORA and for testing the functionality of aceramic cooking vessels, it is important to recognise the limitations of experimental archaeology. Equifinality is the reality that the final 'point' of an experiment could be reached by multiple different ways (Eren and Meltzer, 2024), and that experimental results may not be able to definitively answer the question. For example, when testing aceramic cooking materials, there is a temptation to *replicate* an ethnographic example and infer from the final results an illegitimate universal interpretation. This was largely avoided in this thesis by focusing on the material properties

of the vessels, and investigating variables related to their thermodynamic capabilities, rather than attempting to replicate any particular vessel design or set-up. However - the experimenters involved in the design and execution of the experiments were *unskilled*, in the sense that they did not have much prior experience of cooking with animal hides or hot stones beforehand. Therefore, a more skilled individual may have yielded different results based on their *familiarity* and *know-how* with the materials and practice (Larsson, 2016; Currie, 2022). The problem circles back though, with the issue of *conceit* from those practitioners considered more skilled in a particular task (Thomas, 1986; Eren and Meltzer, 2024), projecting their interpretation.

Ultimately the actualistic experiments undertaken in this thesis were not aimed at *realism,* strictly speaking, but rather at heuristically testing *specific* material properties under conditions where not every variable could, or should, be controlled (griddle cooking, earth ovens, aceramic vessels). Actualism is a key experimental design feature where particular actions - use of hearth-fires to heat stones for example - produces a more *valid* inferential result than a tightly controlled experiment, in the sense that it more closely matches the, albeit epistemologically unknowable, archaeological conditions under study. Controlled laboratory experiments are the ideal complement to these studies, where the looser experimental conditions are tightened, allowing for a better signal-to-noise ratio. To this end, the combination of both methods helped better answer the original thesis aims.

Some drawbacks of the different actualistic experiments undertaken include: low replications; difficulty sourcing precise types of stone and difficulties with recording precise temperatures. Cooking on or with stones involves a huge number of variables - type of geology, heat of the fire, decisions whether to cool boiling stones prior to adding them to the water and many others - each of these could result in alterations to the lipid residues recovered. In the case of the griddle stones, the lack of degradation meant that each sample contained *very high levels* of lipid content, which resulted in the samples being saponified to *reduce* lipid levels. This replicability gap between non-degraded experimental samples and degraded archaeological samples can be partially resolved through specific degradation experiments, for example burying samples for a certain length of time, but in this case focusing on the key biomarkers of thermal processing avoids this issue.

2.2. Organic residue analysis

The absence of long-chain ketones has been discussed above, but from a methodological point of view there are two possibilities to explore. The first is to increase the FCR sample size, most likely to around 40-50 grams rather than 4 grams. The second is to replicate the closed laboratory experiment but make use of other lipid and stone substrates, in order to thoroughly test what precise compositional element is helping to catalyse LCK formation.

Similarly the production of APAAs in stone needs more validation. Increasing the length of time lipids are heated on a griddle; increasing the replication of the controlled laboratory samples and reproducing the same conditions with different foodstuffs, as in Bondetti et al (2021) will help elucidate why the H isomer levels were so low in some instances, and whether the isomer patterns seen are reproducible in other stones and with other foods.

Identifying several ACPAAs was a surprise. Very little is known about these cyclic alkanoic acids, indeed one of them identified in Breu et al (2023) was left unnamed. Investigating under what conditions these form, and whether their formation differs compared to ceramics could result in another robust biomarker similar to APAAs. Designing a separate selective ion mode scanning procedure for ACPAAs might be a productive avenue of future research.

In comparison to ceramics it would appear that FCRs preserve lower concentrations of lipids, a result confirmed through collating all the residue analysis conducted on stone to date. The inability to use stable carbon isotope analysis on four of the six FCR assemblages studied for this thesis again further reinforces this limitation. It may be that the *nature* of aceramic cooking methods necessarily results in less time and lower exposure of the stones to the foodstuffs, or that actions like moving stones from the food back into the fire result in the destruction of any retained fatty acids or small organic compounds. However, alternative extraction methods could improve the total lipid quantity, including supercritical fluid extraction - which has been shown to increase lipid yield from archaeological samples (Devièse et al. 2018), or microwave assisted extraction (Blanco-Zubiaguirre et al. 2018).

3.0 Final conclusion

The broad focus of this thesis, aimed at evaluating aceramic cooking practices through a number of methodologies, necessarily resulted in conclusions which cover biochemical and archaeological interpretations. Drawing these together into a coherent final analysis is not straightforward. Overall the thesis has presented: a short ethnographic survey of aceramic cooking techniques; a literature review of aceramic cooking techniques in the northern European Mesolithic; an actualistic experimental investigation into the function and performance of aceramic cooking vessels; a review of ORA studies conducted on FCRs; a series of experiments aimed at characterising thermal biomarker formation in FCRs and the application of ORA to archaeological FCR artefacts from the northern European Mesolithic and Neolithic. What can be summarised from these is that aceramic cooking tools, including FCRs, are valuable archaeological subjects of study, capable of yielding high-quality physical and biochemical data. Despite their relative invisibility in the prehistoric record, these containers and stone artefacts *can* be analysed and the results can contribute both towards broad questions of ceramic evolution, and specific lifeways amongst ceramic and aceramic Mesolithic foragers. Through analysing the ethnographic and archaeological record, it is possible to infer what materials were being used prior to the invention and adoption of ceramics, and therefore test their performance and functional capabilities against pottery vessels. It is also possible to apply contemporary ORA methods and knowledge to FCRs, and in the process widen the field both for archaeology and bioarchaeology, bringing stone more prominently into the class of materials commonly analysed when recovering organic residues. The potential of FCRs in particular is both broad and deep - since the methodology is applicable to all times and places, including in the deep Palaeolithic past, where organic preservation is extremely rare.

4.0 References

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Appendices

1.0 Appendix One: Ethnographic data (Chapter 2)

The citations within the table can be found in the reference list of Chapter Two.

Culture	Country	Latitude	Pottery	Stone Boiling	Direct Heat (Container)	Direct Heat (No Container)	Earth Oven	Stone Grilling	Fat Rendering	Drying/ Smoking	Fermenting	Sources
Miwok	USA	37	No	Baskets pg 138 (1933)	Steatite pg 211 (1933)	Ash, coals, spits pg 138-140 (1933)	Yes pg 138 (1933)	Unknown	Unknown	Yes pg 139 (1933)	Unknown	(Barret & Gifford 1933)
Mi'kmaq	Canada	46.99	No	Steatite/ sandstone, wooden troughs pg 414 (2004) pg 6 (1968)	Steatite/ sandstone, birch-bark (suspended) pg 415 (2004)	Ash, coals, spits pg 408 (2004) pg 400 (1908)	Unknown	Unknown	Seal pg 410 (2004) pg 111 (1955)	Meat pg 411 (2004)	Fish pg 417 (2004)	(Lockerby 2004) (Denys 1908); (Wallis 1955); (Lepage 1968)
Innu	Canada	50	No	Bark, wooden troughs pg 10 (1952) pg 27 (1947)	Birch bark pg 478 (1947)	Ash, coals, spits pg 9 (1952) pg 23 (2010) pg 98 (1977)	Unknown	Unknown	Fish/Meat pg 9 (1952)	Yes pg 9 (1952)	Unknown	(Lane 1952) (Speck 1977); (Henriksen 2010); (Lips 1947); (Lips 1947)
Winnebago	USA	43.5	Yes	Unknown	Ceramics pg 70 (1973)	Ash, coals, spits pg 68 (1973)	Yes, stones pg 70 (1973)	Unknown	Unknown	Yes pg 69 (1973)	Unknown	(Radin 1973) (Radin 1923)
Ojibwa	Canada	52	No	Bark, wooden troughs pg 13 (1976)	Intestine pg 17 (1976)	Ash, coals, spits pg 17 (1976)	Unknown	Unknown	Fish/meat (1923)	Fish/Meat (1923)	Unknown	(Rogers and Black 1976) (Vennum 1988) (Hesketh 1923)
Pawnee	USA	42	Yes	Unknown	Ceramics pg 90 (1852)	Unknown	Unknown	Unknown	Unknown	Meat pg 90 (1852)	Corn pg 90 (1852)	(Smith 1852)
Crow	USA	45.37	No	Hide pg 212 (1922) rawhide pg 698 (2001)	Rawhide pg 212 (1922) pg 268 (1934)	Ash, coals, spits pg 268 (1934)	Unknown	Unknown	Meat pg 698 (2001)	Meat / Fruit pg 698 (2001)	Unknown	(Voget 2001) (Murdock 1934) (Lowie 1922)
Blackfoot	USA / Canada	49.34	No	Hide, paunch, whole animal pg 26 (1910)	Intestines pg 26 (1910)	Ash, coals, spits pg 170 (1930)	Yes, stones, steaming pg 24-25 (1910)	Unknown	Meat pg 248 (1978)	Meat / Fruit pg 21 (1910)	Unknown	(Wissler 1910) (Schultz and Donaldson 1930) (Schaeffer 1978)
Kutenai	USA / Canada	49	No	Baskets, wooden containers pg225 (1998)	Unknown	Unknown	Yes, stones pg 33 (1941)	Unknown	Fish/Meat pg 9 (1952) pg565 (1893)	Meat / Fruit pg 2 (1967) pg 34 (1941)	Unknown	(Brunton 1998) (Turney-High 1941) (Tro 1967) (Tro 1968) (Chamberlain 1893)
Chinook	USA	45.17	No	Baskets, wooden containers pg 4 (2004)	Unknown	Ash, coals, spits pg 142 (1939)	Yes, stones, steaming pg119 (1939) pg 537 (1990)	Unknown	Seal, whale, sealion, fish (1939)	Meat/fish/ fruit pg 121 (1939)	Unknown	(Ruby and Brown 1976) (Drucker and Ray 1939) (Silverstein 1990) (Beierle 2004)
Klamath	USA	42	No	Baskets, wooden containers pg 162 (1930) pg 8 (1966)	Unknown	Unknown	Unknown	Unknown	Fish pg 11 (1966)	Fish/fruit pg 165 (1030)	Fish pg 167 (1930)	(Spier 1930) (Colson and Stern 1966)
Yurok	USA	41.5	No	Baskets, wooden containers pg 110 (1952)	Unknown	Unknown	Unknown	Unknown	Fish pg 236 (1920)	Nuts/plants pg 145 (1952)	Fish pg 236 (1920)	(Heizer 1952) (Waterman 1920)
Quinault	USA	47.12	No	Wooden troughs, bowls, bark containers pg 44, 46 (1936)	Unknown	Ash, coals, spits pg 269 (1886)	Yes, stones, steaming pg 54 (1936)	Unknown	Fish, whale pg 46 (1895)	Fish/Meat pg 46 (1936)	Fish / Roe pg 40 (1936) pg 268 (1886)	(Olson 1936) (Willoughby 1886)
Nuu-chah-nulth	USA / Canada	49.67	No	Wooden 'boxes' pg 398 (1990) pg 178 (1953)	Unknown	Ash, coals, spits pg 398 (1990)	Yes, stones, steaming	Unknown	Fish, whale pg 425 (1900)	Fish/fruit pg 398 (1990) pg 425	Roe pg 64 (1951)	(Jacobs et al. 1957) (Arima and Dewhirst 1990) (Colson 1953) (Renker and Gunther 1990) (Drucker

							pg 425 (1900)			(1900)		1951)
Haida	Canada	54	No	Baskets, wooden containers pg 226 (1961) pg 82 (1990)	Unknown	Ash, coals, spits pg 226 (1961)	Yes, stones, steaming pg 226 (1961)	Unknown	Fish, whale. seal pg 225 (1961)	Fish/meat/ fruit pg 223, 225 (1961) pg 82 (1990)	Fish pg 225 (1961)	(Swanton 1905) (Murdock 1961) (Blackman 1990)
Nuxalk	Canada	52.33	No	Wooden containers (bentwood boxes) pg 215 (1948)	Unknown	Ash, coals, spits pg 215 (1948)	Yes, stones, steaming pg 215 (1948)	Unknown	Fish, sea lion pg 325 (1990)	Fish/meat/ fruit pg 325 (1990)	Unkown	(McIlwraith 1948 (Kennedy and Bouchard 1990)
Tlingit	Canada	57	No	Wooden containers (bentwood boxes), baskets pg 102 (1960) pg 48 (1915)	Unknown	Ash, coals, spits (1960)	yes, stones, steaming pg 48 (1915)	Unknown	Fish, seal pg 105 (1915) pg 88 (1896)	fish/meat/ fruit pg 110 (1915)	fish pg 110 (1915)	(De Laguna 1960) (Jones 1915) (Knapp and Dorr 1896)
Aleut	Canada	55	Yes	Unknown	Ceramics pg 3 (1945)	Ash, coals, spits pg 61 (1953) pg 46 (1975)	Yes pg 172 (1975)	Yes (1748)	Fish, seal, whale pg 54 (1975)	Fish/meat pg 28 (1944) pg 20 (1952)	Fish, seal pg 26 (1944)	(Jochelson 1928) (Quimby 1945) (Cook 1784) (Hrdlička 1944) (Birket-Smith 1953)
Nivkh	Russia	53	No	Unknown	Unknown	Ash, coals, spits (1973)	Unknown	Unknown	Fish, seal, dolphin (1973)	Unknown	Fish /fruit (1882)	(Seeland and Schütze 1882) (Black 1973)
Copper Inuit	Canada	68.72	No	Stone containers pg 4 (1946)	Stone containers pg 4 (1946)	Unknown	Unknown	Unknown	Fish, seal pg 100 (1922)	Meat, fish pg 99 (1922)	meat, fish pg 106 (1922)	(Jenness 1922; Jenness 1946)
Kaska	Canada	60	No	Baskets, bark containers pg 43 (1954)	Paunches pg 43 (1954)	Ash, coals, spits pg 43 (1954)	Unknown	Unknown	Fish, seal, bear pg 45 (1954)	Meat/fish/ fruit pg 43 (1954)	Unknown	(Honigmann 1954)
Chipewyans	Canada	58.3	No	Paunches, bark, whole animal pg 18 (1982)	Paunches pg 32 (1930)	Ash, coals, spits pg 31 (1930)	Unknown	Unknown	Unknown	Fish/meat/ fruit pg 31 (1930)	Unknown	(Birket-Smith 1930) (Smith 1982)
Ingalik	USA	62.5	Yes	Ceramics pg 230 (1957)	Pottery, bark containers pg 142 (1970) pg 146 (1970)	Ash, coals, spits 176 (1970)	Unknown	Unknown	Fish pg 40 (1958)	Fish/meat/ fruit 176 (1970)	Fish pg 46 (1959)	(Osgood 1970; Osgood 1958; Osgood 1959) (Driver & Massey 1957)
Saami	Finland/ Sweden/ Russia	68.7	No	Unknown	Metal pots pg 98 (1984)	Ash, coals, spits pg 171 (1949)	Yes pg 82 (1949)	Yes pg 82 (1949)	Unknown	Unknown	Milk, meat pg 459 (1984)	(Itkonen and Minn 1984; Collinder 1949; Itkonen 1962)
Nenets	Russia	68	No	Unknown	Metal pots pg 57 (1847)	Unknown	Unknown	Yes pg 33 (2011)	Unknown	Unknown	Unknown	(Islavin and Wise, 1847)(Svoboda *et al.*, 2011)
Samoyed	Russia	73.14	No	Unknown	Metal pots pg 113 (1966)	Ash, coals, spits (pg 112 (1966)	Unknown	Unknown	Fish pg 112 (1966)	meat pg 110 (1966)	Unknown	(Popov and Ristinen 1966)
Yakut	Russia	61.7	Yes	Unknown	Metal pots, ceramics pg 158 (1933)	Ash, coals, spits (1993)	Unknown	Yes pg 617 (1993)	Unknown	Unknown	Meat, dairy, fish pg 551 (1993)	(Sieroszewski 1993) (Wrangel 1842) (Jochelson 1933)
Koryaks	Russia	63.9	Yes	Unknown	Metal pots pg 76 (1898)	Unknown	Unknown	Yes pg 224 (1870)	Unknown	Unknown	Fish pg 857 (1964)	(Kennan 1870) (Dunn 1964) (Smith 1898)
Chukchee	Russia	66.5	No	Unknown	Metal pots pg 56 (1904)	Ash, coals, spits pg 194 (1904)	Unknown	Yes 193 (1904)	Unknown	Fish/meat pg 134 (1904)	Meat, vegetables, leaves, grasses pg 197 (1904)	Bogoraz-Tan (1904)

2.0 Appendix Two: Additional Mesolithic FCR table (Chapter 3)

Country	Date	Site	Archaeological Culture	Description (if given)	Reference
Ireland	4225-3965 BC	Ferriter's Cove	Irish Mesolithic	The small spreads of burnt stone were interpreted as the waste product from possible roasting activities where various food produce was cooked.	Hawkes 2014
Ireland		Fanore More	Irish Mesolithic	burnt stone and shell midden deposit on the shores of Fanore More,	Hawkes 2014
Ireland		Lake Derravaragh	Irish Mesolithic		Hawkes 2014
Ireland	4690 - 4040 cal BC	Clonova 1	Irish Mesolithic	a small sample of nonworked stones that exhibit signs of being heated then submersed in boiling water. It is proposed, on the basis of macro-thermal alterations and geology, that these stones are remnants of a cooking feature	Little 2014
UK		Caisteal nan Gillean II	British Mesolithic	3 concentrations of heavily burnt & fragmented shells, localised close to the centre of the midden. These lack hearthstones but have fire cracked rock nearby. Dispersed charcoal is present, this most likely having been spread by trampling & wind erosion	MIthen 2019
UK		Priory Midden	British Mesolithic	Burning throughout the sequence with 2 clearly defined hearths in direct superposition & closely associated with at least 1 tight cluster of fire-cracked pebbles. A large fragment of burnt & worked whale bone was found immediately below the lower of the two hearths	MIthen 2019
UK	5935+55 and 5040+55 BC	Staosnaig	British Mesolithic	4.5 m diameter circular pit, 0.3–0.4 m deep, containing a circular cut 1.8 m diameter on its eastern edge (Feature F24). It contained large quantities of charred hazelnut shells & other plant material, burnt flint, & bone fragments, but with limited quantities of fire cracked rock & wood charcoal. The burnt flint was concentrated within the centre of the feature	MIthen et al 2001
UK	4360–4000 cal BC	Dunford Bridge Site A	British Mesolithic	Concentration of fire-cracked 'finds' which are interpreted as 'undoubtedly' representing a hearth	MIthen 2019
UK	4940–4710 cal BC	Goldcliff Trench J	British Mesolithic	A cluster & diffuse scatter of heat-fractured quartzite, most likely heated to high temperatures & then rapidly cooled in water	MIthen 2019
UK	8300–7900 BC	Oakhanger	British Mesolithic	Within Site V, 5 hearths were identified on the basis of clusters of burnt flint & fire-cracked stone. These were amidst an extensive scatter of chipped stone artefacts	MIthen 2019
UK		Culverwell	British Mesolithic	4 hearths indicated by well-defined areas of a very black appearance & areas of red well-fired clay & containing large quantities of burnt stone. An unusually large number of heavy choppers/chopping tools, pounders, & picks were found concentrated round the hearths. The hearths were found in a straight row adjacent to the eastern edge of what was interpreted as a living floor.	MIthen 2019
UK	7600 cal BC	Kinloch	British Mesolithic	Numerous pits & hollows that contained burnt flint & charred hazelnut shell. Feature BA1 also contained several fragments of stone slabs, some of which could be rejoined & were burnt, & were interpreted as 'hearth slabs'	MIthen 2019
UK		South Haw	British Mesolithic	2 circular hearths in shallow pits containing burnt stones & charcoal. Possibly hearths consisting of burnt stone seemingly positioned in a row & on their sides	MIthen 2019
UK		Thatcham I	British Mesolithic	Several hearths, scraped out of the ground &, in 1 instance, surrounded & lined with large pieces of sarsen stone, associated with burnt flints, bone & antler, & charcoal	MIthen 2019
UK	8640–8260 cal BC	Thatcham II	British Mesolithic		MIthen 2019

UK		Criet Dubh	British Mesolithic	Hearth stones positioned in centre of shallow pit, likely to have been recut on several occasions, over which there was a heavily burned, organic rich sediment with charred hazelnut shell & burnt flint	MIthen 2019
UK		Rubha Port an t-Seilich	British Mesolithic	Accumulation of burnt stones originally positioned in crevice between 2 outcropping slabs of bedrock, with horizontally laid slabs around uppermost surface of the fireplace, with charred hazelnut shell, charcoal, burnt flint, & calcined fragments of bone	MIthen 2019
UK	4680–4370 cal BC	Cnoc Coig	British Mesolithic	Clearly defined hearths in at least 50 locations, defined by welldefined patches of heavily burned shells, usually roughly circular in shape & typically more compact than the surrounding midden. Most were small, 60–80 cm in diameter, with a few up to 1.5 m. Also, area of diffuse burnt material that appears to be material cleaned out of hearths. Some hearths had stone settings, as described in text. Many of the hearths have clear evidence for several episodes of use marked by distinct horizons of burnt shells, separated by 1 or more deposits of intervening unburnt shell	MIthen 2019
UK		Lussa Wood	British Mesolithic	A scoop made into gravel within which 3 adjacent stone rings were constructed, each c. 1 m in diameter, with flat stone bases. Charcoal was present, but in limited quantity, with a number of minute bone fragments. The excavator suggests this structure might have been used for roasting meat	MIthen 2019
UK		Morton	British Mesolithic	Within Site A, a slightly scooped area delimited by burnt hearth stones, dating to the earliest occupation. This was sealed by wind-blown sand & then succeeded by 2 hearth settings from occupation II. Artefacts tend to cluster around the hearths. Occupation III had a ‘well-built’ hearth, to the west of what was interpreted as a living floor with possible sleeping places. Within Site B there were several settings of stones which form either small hearths 0.2 × 0.3 m across the central area with some charcoal, or supports for stakes & posts	MIthen 2019
UK		Redkirk Point	British Mesolithic	A hollow/pit on the shore of the Solway Firth, 2.0 × 3.0 × 0.15 m deep, containing burnt sandstone pebbles & charcoal, set within discoloured sand. A setting of stones was situated towards the bottom of the hollow/pit. The charcoal was mainly from oak & elm, with some birch twigs	MIthen 2019
UK	8500–8200 cal BC	Deepcar	British Mesolithic	3 ‘relatively hard’ areas within a purported structure were interpreted as hearths. 1 has a ring of stones surrounding a concentration of burnt artefacts & another was bounded by stones on one side	MIthen 2019
UK		Dunford Bridge Site B	British Mesolithic	A small hearth, 0.23 m across & 0.2 m deep, flat bottomed & full of sand, charcoal, & burnt flints, with a compact stone surround. The flint distribution is focussed on this hearth	MIthen 2019
UK	4190 and 3970 cal BC	March Hill	British Mesolithic	4 hearths, with evidence for re-use on several occasions. Hearths 1 & 2 were surrounded by stone settings; hearth 3 was set into a small depression; hearth 4 was the largest, with an elongated shape. A fifth hearth was located 200 m away from this cluster	MIthen 2019
UK		Wawcott IV	British Mesolithic	The excavator describes ‘a carefully constructed hearth, approximately one meter is diameter, made from closely packed sarsen stones’	MIthen 2019

UK		Hawkcombe Head	British Mesolithic	A shallow pit with charcoal inside a circle of stones, with loose stone inside it. Predominately charcoal from oak, no flint knapping debris & located away from the clay floor of a probable structure	MIthen 2019
UK	9130–8250 cal BC	Marsh Benham	British Mesolithic	patch of charcoal, burnt hazelnuts and burnt stones around 45 cm in diameter was interpreted as a hearth	Conneller 2022
UK	7970 - 7610 cal BC	Warren Fields	British Mesolithic	deposit in pit 19 consisted of charcoal, burnt stones, burnt animal bone and a bunt seed of fat hen. The chemical signature of several of the charcoal fills suggested the presence of ground-up minerals	Conneller 2022
UK	7200–6700 cal BC	Chest of Dee	British Mesolithic	A large cooking pit, measuring nearly a metre across which dates to this period was located in a nearby trench. This contained pine charcoal and burnt stones and seems to have been used at least twice	Conneller 2022
UK		Stainton West	British Mesolithic	pit with burnt stones that may represent a cooking pit and an external hearth	Conneller 2022
UK	6650–6385 cal BC	Brenig	British Mesolithic	a series of large, intercutting pits (F19), which seemed to re-present a succession of hearths and earth ovens, date to the Mesolithic. Within these pits, layers of stones and burnt stones interspersed with layers of earth were recorded	Conneller 2022
		Brenig 53	British Mesolithic	a series of small firepits, earth ovens, pits and lines of stakeholes were discovered in association with 1600 pieces of lithic material, which was formed several distinct clusters.	Conneller 2022
UK	7570–7045 cal BC	Tolpits Lane	British Mesolithic	he upper layer includes tips of midden material, including bands of burnt stone, clusters of lithic debitage and bands of charcoal, burnt seeds and hazelnut shells	Conneller 2022
UK		Avington VI	British Mesolithic	small pit, 40 cm in diameter that contained charcoal and burnt stones.	Conneller 2022
UK	6100–6000 cal BC	Bouldnor Cliff BC-II	British Mesolithic	a pit, filled with charcoal, burnt stone and burnt flint was found in the eroding cliff section	Conneller 2022
UK	6000 cal BC	Culverwell	British Mesolithic	Large quantities of burnt stones were common, indicative of a strong focus on food preparation and cooking. Many of the periwinkles and topshells were intact indicating use of boiling water to extract them	Conneller 2022
UK	6000 cal BC	Culverwell	British Mesolithic	an artificial scoop about 1.5 m across, cut through the lower layers of the midden and the clay below; the clay itself has been baked. It is associated with burnt stones and small bone fragments, some of which could be identified as pig.	Conneller 2022
UK	5630 - 4745 cal BC	Little Dartmouth Farm	British Mesolithic	clusters of burnt stone and clear charcoal lenses, seem to represent deliberate episodes of infilling	Conneller 2022
UK	5990–5790 cal BC	Nab Head	British Mesolithic	Burnt microdebitage and some burnt stones may indicate a hearth was present.	Conneller 2022
UK		Risga	British Mesolithic	The midden was 30 cm thick, and in the centre, a layer of burnt stones and several hearths was recorded	Conneller 2022
UK		Cnoc Coig	British Mesolithic	A major hearth, one of several at the lowest levels in this area, was associated with clusters of burnt stones and a number of ‘stone holes’, perhaps in-dicating large-scale cooking activities	Conneller 2022
UK		March Hill	British Mesolithic	two were stone-built hearths, and one a cooking pit or earth oven, measuring 50 cm wide and 20 cm deep (Spikins 2002). This was filled with charcoal and burnt stones.	Conneller 2022
UK	4330–3950 cal BC	Nant Hall	British Mesolithic	smaller heaps of mussels and other shells (periwinkles, cockles and oyster), a few animal bones (red deer) and occasional lithics and burnt stone.	Conneller 2022
UK	5345–4725 cal BC	Wawcott III	British Mesolithic	a tree throw, albeit one that may have seen human use, as its base contained concentrations of burnt stone, charcoal and charred hazelnut shells.	Conneller 2022
UK	5295–4730 cal BC	Wawcott XXII	British Mesolithic	spreads of charcoal and burnt stones were relatively common at the site. A single pit, 1 m in diameter, was located; this was infilled with silt containing flint, burnt stone and charcoal. Some faunal remains were present,	Conneller 2022
UK	4940–4710 cal BC	Goldcliff Island	British Mesolithic	Scatter A is also associated with animal processing and cooking activities, indicated by faunal remains and clusters of burnt stones.	Conneller 2022
UK	9130–8250 cal BC	Marsh Benham	British Mesolithic	a patch of charcoal, burnt hazelnuts and burnt stones around 45 cm in diameter was interpreted as a hearth.	Conneller 2022
UK	6600–6440 cal BC	Kingsdale Head	British Mesolithic	second smaller pit, filled with charcoal at its base and sealed with a layer of stones was found close to the cooking pit	Conneller 2022
UK		South Haw	British Mesolithic	The presence of burnt bone in an earth oven from South Haw	Conneller 2022
UK	5980–5645 cal BC	Gwernvale	British Mesolithic	an earth oven, a pit with charcoal at the base, overlain with stones.	Conneller 2022
UK		South Haw	British Mesolithic	an earth oven was found surrounded by an arc of sta-keholes enclosing an area measuring around 2 m in diameter	Conneller 2022
UK	7500 BC	Kinloch	British Mesolithic	fill of flint, burnt flint and hazelnuts, but one contained a series of refitting burnt slabs, which seem to represent a deposited hearth setting	Conneller 2022

UK	7800–7700 cal BC	Howick	British Mesolithic	area still remained a focus with a couple of hearths being set and two hazelnut roasting pits (one containing more than 7000 charred hazelnuts)	Conneller 2022
UK	8235 - 7586 cal BC	Coupland	British Mesolithic	may have been a hearth or roasting pit within a larger structure	Conneller 2022
Norway		Unit G of Nyhamna 48	Norwegian Mesolithic	possesses patches of 1 × 1 m to 1 × 2 m areas containing small to medium-sized stones, some of them fire-cracked	Damm 2022
Norway		Kotedalen	Norwegian Mesolithic	shallow pit containing a large number of heated rocks	Damm 2022
Norway		Knubba	Norwegian Mesolithic	substantial number of heated stones in a shallow pit	Damm 2022
Norway		Kvernbergmyra	Norwegian Mesolithic	flat stone slabs	Damm 2022
Sweden	ca. 6600 BC	Dumpokjauratj	northern early Mesolithic	two pits - FCRs - 125kg 88kg	Bergman 2008, in Fretheim 2009
Sweden	7330-7050 cal BC	Garaselet	northern early Mesolithic	cooking pit	Sundquist 1978, in Fretheim 2009
Sweden	6020-5840 cal BC	Tjikkitrask	northern early Mesolithic	cooking pit	Meschke 1967, in Fretheim 2009
Sweden	6660-6440 cal BC	Dorosea	northern early Mesolithic	cooking pit	Sundlin 1986, in Fretheim 2009
Sweden	6500-6250 cal BC	Asele	northern early Mesolithic	cooking pit	Sundlin 1986, in Fretheim 2009
Sweden	6870-6020 cal BC	Vilhelmina	northern early Mesolithic	cooking pit	Sundlin 1986, in Fretheim 2009
Sweden	5760-5480 cal BC	Orealven	northern early Mesolithic	cooking pit	Lannerbro 1992, in Fretheim 2009
Sweden	7030-6770 cal BC	Borlange	northern early Mesolithic	cooking pit	Sandberg, unpublished, in Fretheim 2009
Sweden	5210-4850 cal BC	Sjovreten	northern early Mesolithic	cooking pit	Welinder 1977, in Fretheim 2009
Sweden	5220-4840 cal BC	Sjovreten	northern early Mesolithic	cooking pit	Welinder 1977, in Fretheim 2009
Sweden	5490-5310 cal BC	Sjovreten	northern early Mesolithic	cooking pit	Welinder 1977, in Fretheim 2009
Sweden	Late Mesolithic	Grafjell	northern early Mesolithic	cooking pit	Fretheim 2003, Risbol et al 2001
Sweden	Late Mesolithic	Grafjell	northern early Mesolithic	cooking pit	Fretheim 2003, Risbol et al 2001
Sweden	5900-4800 BC	Grafjell	northern early Mesolithic	cooking pit	Fretheim 2005, Stene 2006
Sweden	5900-4800 BC	Grafjell	northern early Mesolithic	cooking pit	Fretheim 2005, Stene 2006
Sweden	5900-4800 BC	Grafjell	northern early Mesolithic	cooking pit	Fretheim 2005, Stene 2006
Sweden	7800 BC.	Linnebjär	early Mesolithic	two hearths with soot and brittle burnt stone	
Sweden	8100–6700 BC	Högby		two concentrations of post-holes. In this area two areas with fi re-cracked stones, that were most likely originally connected, were excavated. Th e structures contain a thin layer of fi re-cracked stone with dark grey, sooty sand	
Sweden	7000– 6550 BC	Storlyckan		Domestic hearth: The hearth pit was oval, measuring 1.05 × 0.70 m. A large quantity of fi re-cracked stone was found in the hearth pit	
Denmark	5970–5570 cal BC	Argus		area below a sand layer of an intact stone-lined hearth, about 0.5 m in diameter, with charred branches, ash, burnt flint and charred food remains including hazelnuts, pips of raspberry and blackberry and fish bones	Bailey et al 2020
Denmark		Ronæs Skov		stone-lined hearths, small branches of wood used for the fire and a tinder fungus	Bailey et al 2020

Denmark		Gamborg Fjord		several stone-lined hearths, three thick vertical wooden poles and a single charred human bone	Bailey et al 2020
Denmark	4496 - 4335 cal BC	Vængesø III	Ertebolle	stone-set hearths, midden waste sites with FCRs, pits containing periwinkle shells and cooking stones, small pits with FCRs	Andersen 2018
Germany	6400 and 6000 cal BC	Jäckelberg-Huk	Kongemose	remains of two hearths indicated by fire-cracked stones.	Bailey et al 2020
Germany		Hesel		serveral pits filled with charcoal and partly burned cobblestones (cooking stones) have been discovered	Mahlstedt et al 2018

3.0 Appendix Three: FCR sampling (Chapters 6 and 7)

3.1. Kinetic method

Before sampling all samples were lightly cleaned using distilled water and dichloromethane, to remove any soil or obvious contaminants. The kinetic sampling approach was to reduce the sample to small enough pieces to grind in a pestle-and-mortar using physical force. The majority of these were as follows: the sample was wrapped in foil and placed on a board, force was applied with a small hammer until the sample broke, sections were selected and if necessary broken again. The second method was used only once: the sample was placed into a metal pipe cap and metal screws were struck into the sample with a hammer.

3.2. Coring method

Three forms of coring method were used. Firstly a hand-held cordless drill with diamond-tipped tile corers was used to drill out a plug of stone from the sample. The corers were coated in a metallic paint and the DCM solvent initially caused the paint to contaminate the process. DCM was changed for methanol and acetone. Secondly a thin-bladed diamond bone saw was used on several samples, but this broke quickly and was abandoned. Thirdly a bench drill was purchased and a rig-system for keeping the stones immobile was constructed. This again used diamond-tipped corers.

Photograph of sample HVN-5 after the bench drill had removed a core for sampling.

Again the cores were reduced to a powder in a pestle-and-mortar (stainless steel).

4.0 Appendix Four: Organic residue analysis (Chapters 6 and 7)

4.1. Lipid extraction

4.1.1. Acidified-methanol extraction

The use of acidified methanol has been successfully employed to extract lipids from potsherds and ceramics (Craig *et al.*, 2013; Correa-Ascencio and Evershed, 2014). Approximately 1-4 g of each ground stone subsample was treated with excess methanol-

sulfuric acid solution in a test tube. Differences in the composition of the stone meant some were treated with more solution than others until the pH was near to 3.5. The samples were heated in an ultrasonic water bath for 15 minutes before being heated for 4 hours at 70 ˚C in closed vials. The acid-stone suspension was then centrifuged and the supernatant removed. Each sample was mixed with 2 mL of *n*-hexane before being filtered through glass wool and potassium carbonate (washed with DCM). The samples were dried down using N_2 at 37 ˚C. In preparation for GC-MS 10 µL of an internal standard (1.0 µg μL^{-1} hexatriacontane) was added to the samples in a new vial which were then resuspended in 100 µL of *n*-hexane and dried a final time before being transferred to their analysis vials.

4.1.2. Solvent extraction and trimethylsilyl (TMS) derivatisation

Owing to the alkalinity of some FCR samples, a number were subjected to solvent extraction following previous protocols (Charters *et al.*, 1993; Regert *et al.*, 1998; Colonese *et al.*, 2017). The ground samples were weighed (1-4g) and combined with a mixture of DCM-methanol (4 mL; 2:1 v/v). Samples were ultrasonicated (3 × 15 min) and centrifuged (3000 rpm, 10 min). The resulting total lipid extract was derivatised with BSTFA (N, O-bis (trimethylsilyl) trifluorocetamide) (8 drops), for 1 hour at 70 ° C, evaporated and rediluted in *n*-hexane prior to GC-MS analysis.

4.1.3. Trimethylsilyl (TMS) derivatisation for acidified extracts

In order to better identify possible products such as alcohols, carboxylic acids and phenols, trimethylsilyl derivatisation was employed with a number of samples. Approximately 8 drops of *N*,*O*-Bis(trimethylsilyl)trifluoroacetamide (BSTFA) was added to a sub-sample of the total lipid extract. These were heated for 1 hour at 70 ° C, evaporated and rediluted in *n*-hexane prior to GC-MS analysis.

4.2. Instrumentation setting

4.2.1 Gas chromatography - Flame ionisation detector (GC-FID)

GC-FID was used to screen and quantify lipid compounds. GC-FID was carried out on acidified and solvent based lipid extracts using an Agilent 7890S gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK). A splitless injector was used to inject the sample (1 µL) at 300°C. The column was a polymide coated fused silica DB-1 (15 m x 320 µm x 0.1 µm; J&W Scientific, Folsom, CA, USA). The carrier gas was helium. The pressure was set at 3.3 psi with a flow rate of 2 mL min^{-1} and a velocity of 46.57 cm s^{-1}. The temperature program was set at 100 °C for 2 minutes, which then rose by 20 °C min^{-1} until 325 °C, where it was held for 3 min.

4.2.2 Gas chromatography - Mass Spectrometry (DB5)

GC-MS analysis was conducted using an Agilent 7890A series chromatograph with an Agilent 5975C Inert XL mass-selective detector and quadrupole mass analyser (Agilent technologies, Cheadle, Cheshire, UK). A splitless injector was used and maintained at 300 °C. The GC column was directly inserted into the ion source of the mass spectrometer. The carrier gas was helium and the inlet/column head-pressure was kept constant. The ionisation energy of the MS was 70 eV and spectra obtained by scanning between *m/z* 50 and 800. A DB-5MS (5%-phenyl)-methylpolysiloxane column (30 m x 0.250 mm x 0.25 µm; J&W Scientific, Folsom, CA, USA) was used for screening the samples in scan mode. The temperature was set at 50°C for 2 minutes, which rose by 10°C min^{-1} until 325°C was reached where it was held for 15 min.

4.2.3.Gas chromatography - Mass Spectrometry (DB23)

To check the samples for heating biomarkers the samples were analysed using a GC-MS with a DB-23 (50%-Cyanopropyl)-methylpolysiloxane column (60 m × 0.250 mm × 0.25 µm; J&W Scientific, Folsom, CA, USA). The oven temperature was set at 50 °C for 2 minutes before increasing to 100 °C (10 °C min^{-1}). The temperature was then raised by 4 °C min^{-1} to 140 °C, then by 0.5 °C min^{-1} to 160 °C and, lastly, by 20 °C min^{-1} to 250 °C where it was maintained for 10 min. The carrier gas used was helium with a flow rate of 1.5 mL min^{-1}. The SIM (Selective Ion Monitoring) mode was utilised to target cooking biomarkers using the ions

groups: *m/z* 74, 87, 213, 270 for 4,8,12-trimethyltridecanoic acid (TMTD), *m/z* 74, 88, 101, 312 for pristanic acid, *m/z* 74, 101, 171, 326 for phytanic acid and *m/z* 74, 105, 262, 290, 318, 346 for the detection of *ω*-(*o*-alkylphenyl)alkanoic acids of carbon lengths C_{16} to C_{22} ($APAA_{16-22}$).

4.2.4.Gas Chromatography-combustion-Isotopic Ratio Mass Spectrometry (GC-c-IRMS)

Stable carbon isotope values of methyl palmitate (C16:0) and methyl stearate (C18:0) derived from the precursor fatty acids were assessed by GC-c-IRMS. An Isoprime 100 (Isoprime, Cheadle, UK) linked to a Hewlett Packard 7890B series GC (Agilent Technologies, Santa Clara, CA, USA) with an Isoprime GC5 interface (Isoprime Cheadle, UK) was used for the analysis. One ul of each sample was first injected into DB-5MS ultra-inert fused-silica column. The temperature was set at 50 °C for 0.5 min and raised by 0.5 °C per minute to 50 °C, then raised by 10 °C per minute to 300 °C where it was held for 10 min. The carrier gas used was ultra high purity grade helium with a flow rate of 3 mL per minute. The gas flows from the column were split. One was directed into an Agilent 5975C inert mass spectrometer detector (MSD), for sample identification and quantification, the other directed through the GC5 furnace kept at 850 °C to oxidise all the carbon species to CO_2. Clear resolution and a baseline separation of the analysed peaks were achieved.

Eluted products were ionized in the mass spectrometer by electron impact and ion intensities of m/z 44, 45 and 46 were recorded for automatic computing of the 13C/12C ratio of each peak in the extracts. Computation was made with IonVantage and IonOS softwares (Isoprime, Cheadle, UK) and was based on comparisons with standard reference gas (CO2) of known isotopic composition that was repeatedly measured. The results of the analysis were expressed in per mill (‰) relative to an international standard, VPDB.

4.2.5 Sample analysis

GC-MS analysis was conducted using MSD Chemstation software. Compounds were identified based on their retention time, mass spectrum and best fit with the National Institute

of Standards and Technology (NIST) library. Peak integration was conducted using Agilent MassHunter Quantitative Analysis software.

4.3. Actualistic experimental methods

4.3.1. Methods: Experiment One - Lab oven

The laboratory experiment was designed as a development of the method laid out in Bondetti et al. (2021), where the formation of ω-(o-alkylphenyl)alkanoic acids (APAAs) was characterised in ceramics through testing different heating times and temperatures. Therefore much of the methodology is adapted from this publication. The aim was to test stone rather than ceramics, and so three mediums were selected for experimentation - ceramics, sandstone and granite. The ceramic was the same as Bondetti et al's (2021) experiment, replica, wheel-thrown pots (Mr Graham Taylor, Experimental Archaeologist, Ancient Pottery Technology Specialist, Rothbury, UK) which contain a high amount of metal ions (Al_2O_3, 22.78; Fe_2O_3, 7.37; CaO, 0.57; MgO, 0.86; K_2O, 1.6; and Na_2O, 0.1). This 'Standard Red Clay' ceramic was ground into a powder with a pestle and mortar. The sandstone was the same as used for the griddle experiment, and the granite was a commercially sourced granite block. Both were heated in an oven to drive off any endogenous lipids or other contaminants. Four grams of each material were added to a clean hach tube, and approximately 65 mL of rapeseed oil was added (Commercial Organic, cold pressed, extra-virgin rapeseed oil, UK). Each material was then heated in four different ways - for one hour at 200 °C; for one hour at 350 °C; for 5 hours at 200 °C and for 5 hours at 350 °C. These times were chosen based on Bondetti et al's (2021) work and that of Raven et al. (1997), as well as expediency, in order to capture data concerned with two main classes of molecule - APAAs and long-chain ketones. Differences between stone and ceramic, and between types of stone were examined, providing insight into the chemistry of ketone and APAA formation and their relationship with the stone/ceramic matrix, metal ions and time/temperature.

Material	Ceramic	Sandstone	Granite
Conditions (Heat in °C/Time)			
1 hour			
200	LBX-1A	LBX-1B	LBX-1C
350	LBX- 2A	LBX- 2B	LBX-2C
5 hours			
200	LBX-3A	LBX-3B	LBX-3C
350	LBX-4A	LBX-4B	LBX-4C
Control (unheated)	LBX-5A	LBX-5B	LBX-5C

4.3.2. Methods: Experiment Two - Stone Griddle

Experiment two consisted of an actualistic experiment conducted outside at the YEAR Centre (University of York) on 20/11/2020. This involved the use of a piece of commercial sandstone tile to simulate a cooking griddle, onto which was placed four different foodstuffs - beef rump steak (*Bos taurus*), rainbow trout (*Oncorhynchus mykiss*), ground hazelnut (*Corylus avellana*) and ground birch bark powder (*Betula pendula*). These were chosen as proxies for common food groups available during prehistory and reflect some of the widest differences in lipid profiles - a ruminant animal, a freshwater fish, nuts and ground plant material. Birch was included not only as a possible candidate for early flour, but also to test the possibility of yielding birch bark tar, a function which could easily overlap with using stones for cooking. The stone was quickly heated and foods placed directly onto the surface and cooked until completed (Table.1.). The temperature of the stone and the foods were recorded. Afterwards the stones were cooled, cleaned of all surface residues and lightly abraded. The outermost surface was briefly rinsed with deionised water and dichloromethane before sample cores were taken with a diamond-edged cordless drill corer. The cores were then reduced to a powder using a pestle and mortar and weighed for extraction.

Time (mins)	Stone main (C)	Steak (C)	Hazel butter (C)	Birch bread (C)	Trout (C)
0	1	0	0	0	0
5	128	0	0	0	0
10	212	165	0	0	0

15	241	171	0	0	0
20	246	148	246	176	0
25	251	151	249	168	0
30	255	156	252	170	0
35	267	165	263	175	136
40	254	0	0	0	249
45	233	0	0	0	274
50	195	0	0	0	262
55	211	0	0	0	265
60	186	0	0	0	224

4.3.3. Methods: Experiment Three - Pit-Cooking

The second set of experimental stones came from a multi-participatory experiment conducted in Īdeņa, Latvia, over several months in 2022. The experiment was aimed at replicating an underground heating pit, using wood and heated rocks, to test the hypothesis that teeth from different animals could be removed more easily after cooking. Both soil and heated stone samples were collected for lipidomic analysis. The experiments were conducted in two phases. The first took place on the 26/27th of February 2022, when the pit was dug (85x50x60cm). The pit sequence was as follows: larger stones > smaller stones > birch wood fire (3hrs) > boar skin > animal parts > boar skin > sand > second fire > sealed with sand and turf. This was left for 17.5 hours before excavating. Soil samples and a single stone sample (LTV-03) were taken at this point. The pit was then excavated five months later on July 29th 2022, the stones removed and replaced with the addition of several new stones. The sequence for the second phase of the experiment went as follows: new sand > new and old stones > fire (mixed wood, majority alder, 4 hours) > roe deer and fish > roe deer skin > ferns > soil > second fire (3 hours) > turf. The pit was left for over 24 hours and re-opened for sampling. Four stone samples were taken, one old stone (LTV-06) and three new (LTV-18/19/20). Approximately 4g of ground stone was used for extraction, with the exception of sample LTV-20, which was used in its entirety. Samples LTV-03/06/18/19 were duplicated, to extend analytical coverage, since the assemblage was small. This was done by taking samples from a different part of the stone.

5.0 Appendix Five: FCR physical descriptions (Chapters 6 and 7)

Sample	Site	Date (cal BC)	Context	Weight (g)	Species	Type
NST-R01	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	90	Quartz arenite sandstone	Linear slab
NST-R02	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	15	Quartz arenite sandstone	Linear slab
NST-R03	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	131	Quartz arenite sandstone	Linear slab
NST-R04	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	24	Quartz arenite sandstone	Linear slab
NST-R05	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	75	Quartz arenite sandstone	Linear slab
NST-R06	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	64	Quartz arenite sandstone + limestone	Linear slab
NST-R07	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	145	Quartz arenite sandstone	Linear slab
NST-R08	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	151	Quartz arenite sandstone	Linear slab
NST-R09	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	454	Quartz arenite sandstone	Linear slab
NST-R10	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	104	Quartz arenite sandstone	Linear slab
NST-R11	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	183	Quartz arenite sandstone	Linear slab
NST-R12	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	106	Quartz arenite sandstone	Linear slab
NST-R13	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	54	Quartz arenite sandstone	Linear slab

NST-R14	Neustadt (Ger)	4,600 - 3,700	EBK-TRB refuse area	236	Quartz arenite sandstone	Linear slab
RSF-01	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	45	Quartz arenite sandstone	Cobble
RSF-02	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	169	Quartz arenite sandstone	Cobble
RSF-03	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	258	Quartz arenite sandstone	Cobble
RSF-04	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	39	Quartz arenite sandstone	Cobble
RSF-05	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	128	Quartz arenite sandstone	Cobble
RSF-06	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	64	Quartz arenite sandstone	Cobble
RSF-07	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	46	Quartz arenite sandstone	Cobble
RSF-08	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	65	Quartz arenite sandstone	Cobble
RSF-09	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	69	Quartz arenite sandstone	Cobble
RSF-10	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	30	Quartz arenite sandstone	Cobble
RSF-11	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	56	Quartz arenite sandstone	Cobble
RSF-12	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	361	Quartz arenite sandstone	Cobble
RSF-13	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	185	Quartz arenite sandstone	Cobble
RSF-14	Rosenfelde (Ger)	5260 - 5320	EBK fishing site	90	Quartz arenite sandstone	Cobble
NOR-1	Ormen Lange (Nor)	7410 ± 55 and 7515 ± 70 uncal. BC	Domestic hearth	76	Quartzite sandstone	Cobble

NOR-2	Ormen Lange (Nor)	7410 ± 55 and 7515 ± 70 uncal. BC	Domestic hearth	136	Quartzite sandstone	Cobble
NOR-3	Ormen Lange (Nor)	7410 ± 55 and 7515 ± 70 uncal. BC	Domestic hearth	105	Quartzite sandstone	Cobble
NOR-4	Ormen Lange (Nor)	7410 ± 55 and 7515 ± 70 uncal. BC	Domestic hearth	164	Quartzite sandstone	Cobble
NOR-5	Ormen Lange (Nor)	7410 ± 55 and 7515 ± 70 uncal. BC	Domestic hearth	224	Quartzite sandstone	Cobble
KLK-1	Klakken (Nor)	Early Mesolithic	Domestic hearth	383	Granite-like with quartz veins	Cobble
KLK-2	Klakken (Nor)	Early Mesolithic	Domestic hearth	266	Hard sandstone	Cobble
KLK-3	Klakken (Nor)	Early Mesolithic	Domestic hearth	129	Hard sandstone	Cobble
HVN-1	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	137	Quartzite sandstone	Cobble
HVN-2	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	54	Quartzite sandstone	Cobble
HVN-3	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	75	Quartzite sandstone	Cobble
HVN-4	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	87	Quartzite sandstone	Cobble
HVN-5	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	177	Quartzite sandstone	Cobble
HVN-6	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen	156	Quartzite sandstone	Cobble

			midden			
HVN-7	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	163	Quartzite sandstone	Cobble
HVN-8	Havnø (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	298	Quartzite sandstone	Cobble
AHM-1	Visborg (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	575	Quartzite sandstone	Cobble
AHM-2	Visborg (Dmk)	5400 - 4000	EBK-TRB Kitchen midden	172	'Old Red' sandstone	Cobble

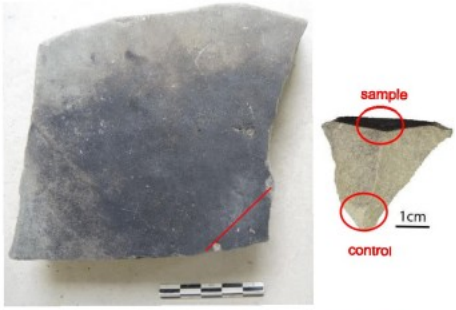
The below table outlines the physical description and contexts for the Latvian pit-cooking experiment (Chapter 6)

Sample	**Weight (g)**	**Shape**	**Description**	**Identity**	**Context**	**Max temp (˚C)**
LTV-03	900	Large cobble	intact, blackening, reddening	sandstone	Immediate post phase 1	Unknown
LTV-06	600	Large cobble	crumbling on the edges, reddening	quartz	Post phase 2(old)	Unknown
LTV-18	80	Small/fractured cobble	heat cracks visible, reddening	sandstone	Post phase 2 (new)	560
LTV-19	183	Small/fractured cobble	heat cracks visible, blackening and reddening	sandstone	Post phase 2 (new)	560
LTV-20	3	chip	grey heat discolouration	granite	Post phase 2 (new)	560

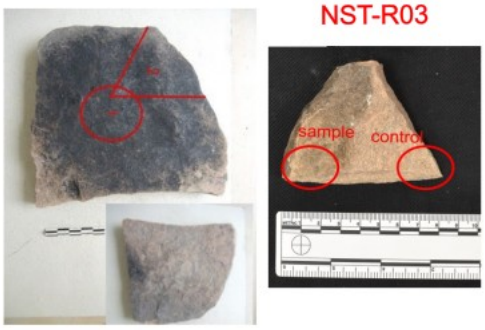
5.1. Photographs of FCRs for Neustadt, Rosenfelde, Ormen Lange, Klakken, Visborg and Havnø

NST-R01

NST-R02



NST-R03



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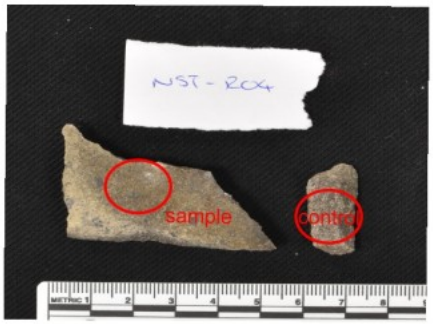

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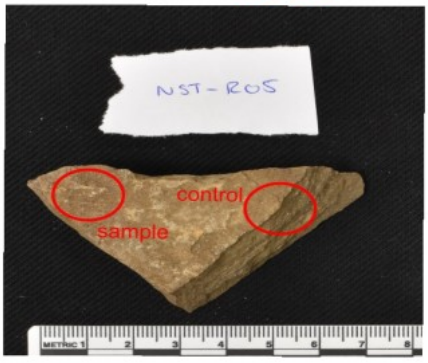

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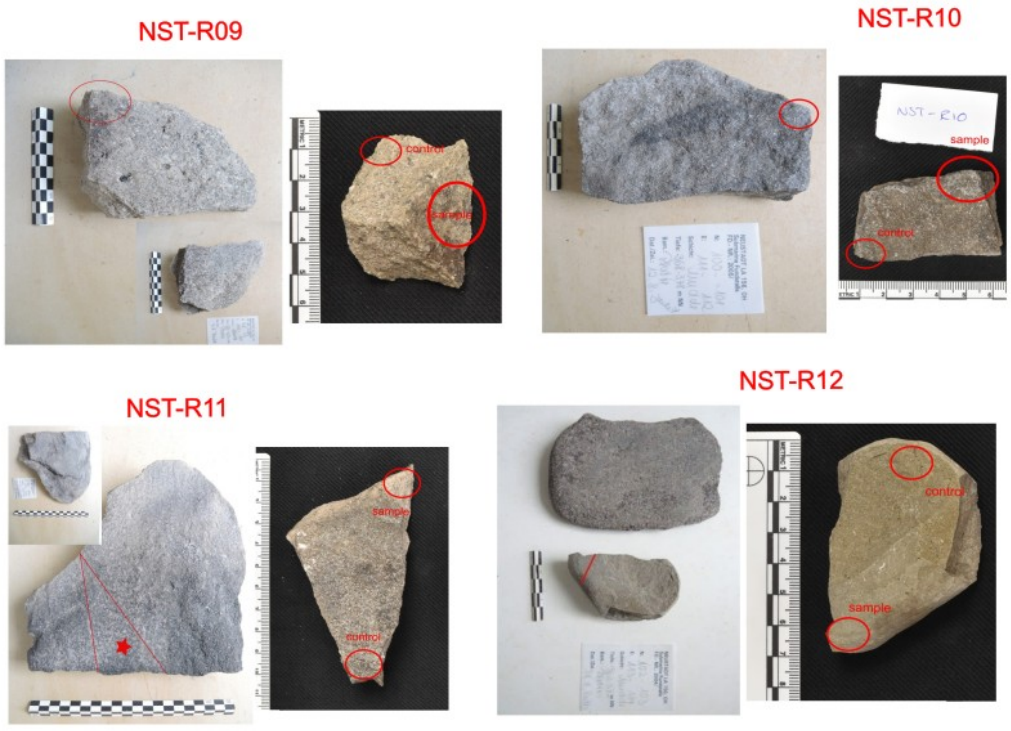
NST-R07

NST-R08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NST-R10
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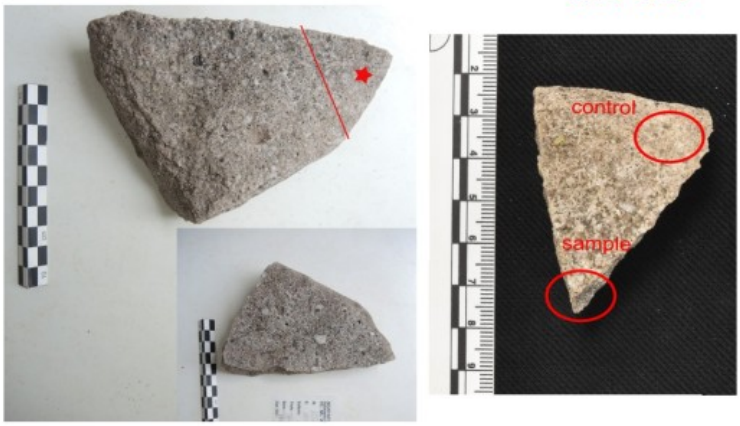
control
NST-R11
sample
control
NST-R12
control
sample

NST-R13



NST-R14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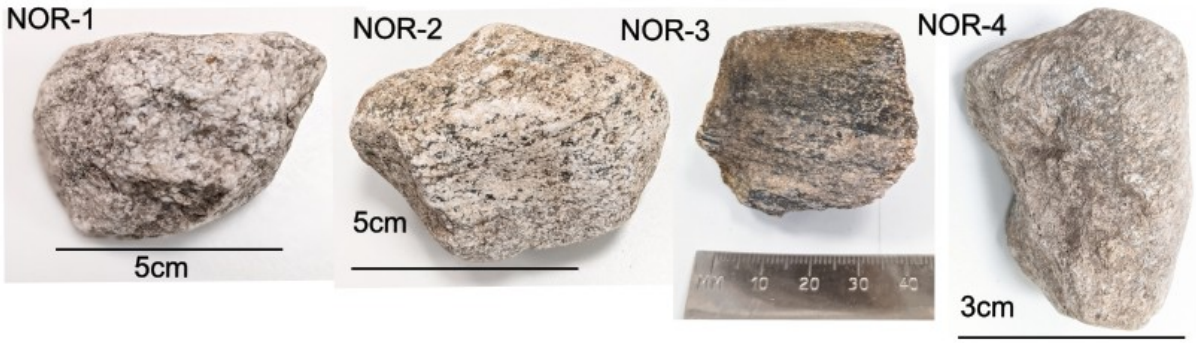
RSF-R02
RSF-R03
RSF-R04
RSF-R05
RSF-R06


NOR-1
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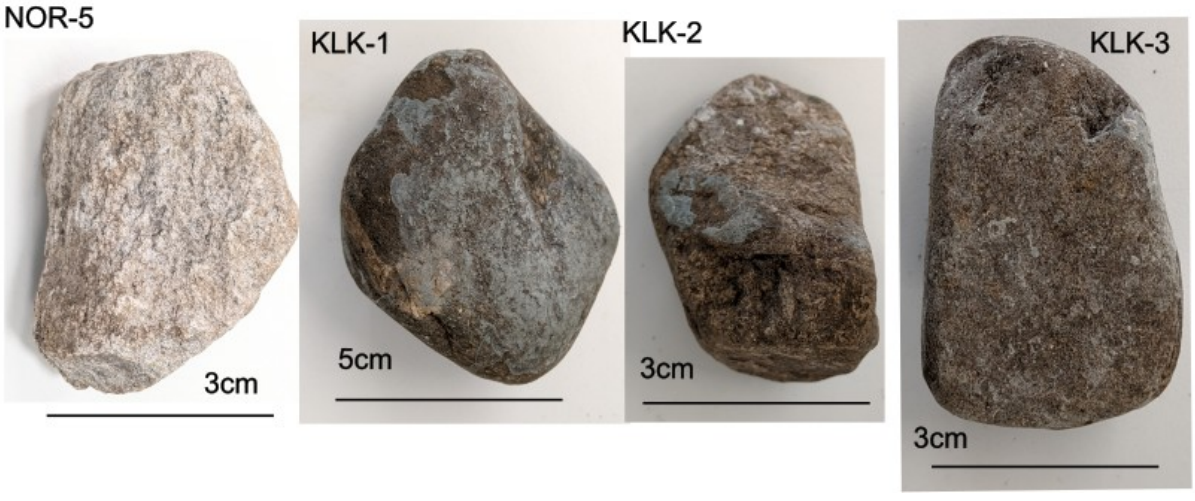
NOR-2
5cm
NOR-3
NOR-4
3cm


NOR-5
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KLK-1
5cm
KLK-2
3cm
KLK-3
3cm

6.0 Appendix Six: Reporting GC-MS identified lipids and compounds from all samples (Chapters 6 and 7)

6.1. Archaeological FCR ORA

6.1.1 Neustadt LA 156

Sample	Sub sample	Lipid concentration gg^{-1}	SFA Range	SFA Main	UFA	BCFA
NST - RO1	RO1A	211	C14-18	C16-18	0	0

	RO1B	105	C16-24	C16-18	0	C19i
NST - RO2	RO2A	131	C12-22	C16-18	0	C17a
	RO2B	20	C10-18	C16-18	0	0
NST - RO3	RO3A	85	C14-18	C16-18	16:1 cis 9, 18:1 trans 11, 18:1 cis 9	C19i
	RO3B	33	C14-24	C16-18	18:1 trans 11	C17i, C17a, C19i
NST - RO4	RO4A	5	C16-20	C16-18	0	0
	RO4B	2	C14-18	C16-18	0	0
NST - RO5	RO5A	6	C14-18	C16-18	0	0
	RO5B	13	C16-18	C16-18	0	0
NST - RO6	RO6A	16	C16	C16	0	0
	RO6B	26	C12-18	C16-18	0	C15a
NST - RO7	RO7A	92	0	0	0	0
	RO7B	9	C14-18	C16-18	0	0
NST - RO8	RO8A	121	C18	C18	0	0
	RO8B	6	C16-20	C16-18	0	0
NST - RO9	RO9A	123	C16-18	C16-18	0	0
	RO9B	21	C14-24	C16-18	0	0
NST - RO10	RO10A	3	C16-20	C16-18	0	0
	RO10B	3	C16-18	C16-18	0	0
NST - RO11	R11A	104	C16	C16	0	0
	R11B	6	C14-24	C16-18	0	0
NST - RO12	R12A	4	C14-18	C16-18	0	0
	R12B	14	C14-18	C16-18	0	0
NST - RO13	R13A	72	0	0	0	0
	R13B	25	C16-18	C16-18	0	0
NST - RO14	R14A	11	C12-18	C16-18	C18:1	0
	R14B	14	C12-18	C16-18	C18:1	0
	R14C	12	C12-18	C16-18	0	C15a

6.1.2 Grube-Rosenfelde LA 83

Sample	Lipid concentration ugg^{-1}	SFA range	SFA mains	UFA	BCFA	Other

RSF-R01	0.6	C12-28	C16-18		Ci19:0	
RSF-R02	1.0	C12-28	C16-18		Ca15:0	
RSF-R03	4.6	C14-28	C16-18		Ci19:0	Olean-12-ene
RSF-R04	0.3	C14-24	C16-18		Ca15:0	
RSF-R05	0.9	C14-28	C16-18		Ci19:0	
RSF-R06	1.5	C14-C24	C16-18			
RSF-R07	1.4	C12-C18	C16-18		Ca15:0, Ca17:0	
RSF-R08	7.7	C12-C26	C14-18	C18:1 *cis* 9, C22:1 *cis* 13	Ci15:0, Ca15:0, Ci17:0	Olean-12-ene Methyl 2 hydroxy tetracosanoate
RSF-R09	1.2	C12-28	C14-18	C16:1 cis 9, C22:1w9	Ca15:0, Ci15:0, Ca17:0, Ci17:0, Ci19:0	
RSF-R10	0.2	C14-28	C16-18		Ci15:0, Ca15:0, Ci17:0	
RSF-R11	7.7	C12-C28	C12-18	C22:1w9	Ci17:0, Ci19:0	Olean-12-ene
RSF-R12	2.5	C12-C24	C16-18			
RSF-R13	0.4	C14-C24	C16-18		Ca15:0, Ci15:0, Ca17:0, Ci17:0	
RSF-R14	4.6	C12-C28	C16-18	C22:1w9	Ca15:0, Ci15:0, Ca17:0, Ci17:0, Ci19:0	

6.1.3 Havnø

Sample	Lipid concentration ugg^{-1}	SFA range	SFA main	UFA	BCFA	DA	Other
HVN-1A	7.6	C14 - C28	C14 C16 C18 C20	C16:1 *cis* 9 C18:1 *cis*9 C20:1 *cis* 13 C22:1 *cis* 13	C15a C17i + a	C9 C12 C13 C22	*n*-C12 *n*-C16 *n*-C17 *n*-C19

						C30	*n*-C24 *n*-C28
HVN-1B	30.2	C08- C30	C12-C18	C10:1 trans 2; C16:1 cis 9, C18:1 trans 10; C18:1 cis 9, C20:1 cis 13; C22:1 cis 13; C24 cis 15	C14a, C14i; C17i + a; C20(14);	C9; C13; C20; C22; C30	
HVN-1C	16.3	C08- C30	C12-C18	C10:1 trans 2; C16:1 cis 9; C18:1 cis 9; C22:1 cis 13; C24 cis 15		C7; C8; C9; C13; C20; C22; C30	
HVN-2A	16.6	C8 - C30	C14 - C26	C16:1 *cis* 9 C18:1 *cis*9 C20:1 *cis* 11 C22:1 *cis* 13	C14(9) C15a	C9 C13 C16 C18 C22 C30	*n*-C11 *n*-C19 *n*-C24 *n*-C28
HVN-2B	51.4	C8 - C30	C16-C18, C22	C10:1 trans 2; C16:1 cis 9, C18:1 trans 10; C18:1 cis9; C20:1 cis 13; C22:1 cis 13; C24 cis 15	C15a + i; C17a + i	C8; C9; C13; C16; C20; C22; C30	
HVN-2C	53.0	C7 - C30	C16-C18, C22	C10:1 trans 2; C16:1 cis 9, C18:1 trans 16; C18:1 cis9; C20:1 cis 13; C22:1 cis 13; C24:1 cis 15	C14a; C15a + i; C17a + i	C8; C9; C13; C16; C18; C22; C30	
HVN-3A	10.9	C9 - C30	C16	C16:1 *trans* 11 C18:1 *cis* 9 C22:1 *cis* 13	C12i C15i C17i + a	C9 C18 C22	*n*-C17 *n*-C24 *n*-C28
HVN-3B	23.2	C8 - C30	C16-C18, C22	C16:1 trans 11, C18:1 cis 9, C22:1 cis 13; C24:1 cis 15	C14a; C17i + a	C9; C13; C20; C22; C30	
HVN-3C	43.9	C7 - C30	C16-C18, C22	C18:1 cis 9; C22:1 cis 9; C24:1 cis 15	C14a; C17i + a	C9; C13; C20; C22; C30	
HVN-4A	19.2	C8 - C30	C12 C14 C16	C10:1 *trans* 2 C16:1 *cis* 9 C18:1 *cis* 9 C20:1 *cis* 11	C14i C17i + a	C9 C16 C18 C22	*n*-C11 *n*-C17 *n*-C24

			C18	C22:1 *cis* 13			
HVN-4B	38.7	C8 - C30	C16-C18, C22	C10:1 trans 2; C16:1 cis 9, C18:1 cis 9; C22:1 cis 13; C24:1 cis 15	C14a; C15a + i; C17a + i	C9; C11; C13; C16; C20; C22; C30	
HVN-4C	54.6	C8 - C30	C16-C18, C22	C10:1 trans 2; C11:1 (10); C16:1 cis 9, C18:1 cis 9; C22:1 cis 13; C24:1 cis 15		C8; C9; C13; C20; C22; C30	
HVN-5	10.8	C9-C30	C16-C18	C16:1 *cis* 9 C18:1 *cis* 9 C22:1 *cis* 13	C10i C14a & C14i C17a & C17i C20(14)	C9 C13	
HVN-6	14.5	C12-C30	C16-C18	C16:1 *cis* 9 C18:1 *cis* 9 C20:1 *cis* 11 C22:1 *cis* 13	C14a & C14i C17a & C17i C20(14)	C8 C18 C20 C22 C30	
HVN-7	26.2	C11-C30	C16-C18	C16:1 *trans* 7 C16:1 *cis* 9 C18:1 *cis* 9 C22:1 *cis* 13	C14a & C14i C17a & C17i C20(14)	C9 C13	
HVN-8	10.6	C12-C30	C16-C18	C16:1 *cis* 9 C18:1 *cis* 9 C22:1 *cis* 13	C14a & C14i C20(14)	C9 C13	

6.1.4 Ormen Lange site 48

Sample	Lipid concentration ugg^{-1}	SFA range	SFA main	UFA	BCFA	DA	Other
NOR-1	72.5	C12-C26	C16-C18	C16:1 *cis* 7, C22:1 *cis* 13	C10:0a	C9	1,2 BDCA
NOR-2	25.5	C11-C26	C16-C18	C18:1 *cis* 9, C22:1 *cis 13*	C10:0a, C12:0i		1,2 BDCA
NOR-3	21.1	C10-C26	C16-C18	C14:1 *cis* 9, C18:1 *cis* 9, C22:1 *cis* 13	C14:0i		2-hydroxydodecanoic acid; 3-hydroxy octadecanoic acid; 3-

							hydroxy hexadecanoic acid; 2-hydroxy hexadecanoic acid; 1,2 BDCA
NOR-4	27.9	C12-C26	C16-C18	C18:1 *cis* 9	C12:0i, C14:0i		3-hydroxy octadecanoic acid; 1,2 BDCA
NOR-5	40.6	C12-C28	C16-C18	C18:1 *cis* 9, C22:1 *cis* 13	C14:0i	C9 C10	1,2 BDCA

6.1.5 Klakken

Sample	Lipid concentration ugg^{-1}	SFA range	SFA mains	UFA	BCFA	DA	Other
KLK-1A	9.8	C12-C28	C16-C18	C16:1 *cis 9;* C18:1 *cis* 9; C18:1 *trans* 10; C20 *cis* 11	Ca12:0; Ci14:0; Ca14:0; C16i:0; Ca16:0; C17(10); C20(14); Ci22:0	C9 C13 C16 C22	1,4; 1,2 BPCA
KLK-1B	8.7	C9-C28	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 trans 10; C20 cis 11	Ca12:0; Ci14:0; Ca14:0; C16i:0; Ca16:0; C17(10); C20(14); Ci22:0	C9 C13 C22	1,4; 1,2 BPCA
KLK-2A	10.1	C12-C28	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 trans 10; C20 cis 11	Ca12:0; Ci14:0; Ca14:0; C17(9); C20(14);	C9 C30	methyl dihydroabietate,1,4; 1,2 BPCA
KLK-2B	16.4	C9-C30	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 trans 10; C20 cis 11; C22 *cis* 13	Ca12:0; Ci14:0; Ca14:0; Ca16:0; Ci17:0; C17(10); C20(14);	C8 C16 C18 C30	1,3; 1,2 BPCA

KLK-3A	24.4	C9-C30	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 trans 10; C20 cis 11	Ca12:0; Ci14:0; Ca14:0; Ci16:0; Ca16:0; C17(10); C20(14);	C8 C9 C12 C16 C18 C22	1,3; 1,2 BPCA
KLK-3B	12	C10-C30	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 trans 10; C18:2 trans 9 trans 12; C22 cis 13	Ci14:0; Ca14:0; Ci16:0; Ca16:0; C17(10) C20(14)	C8 C9 C22	methyl dihydroabietate, 1,2 BPCA

6.1.6 Visborg

Sample	Lipid concentration ugg^{-1}	SFA range	SFA mains	UFA	BCFA	DA	Other
AHM-1A	12.2	C16-C18	C16-C18	C16:1 *cis* 9; C16:1 *cis* 11; C17:1 *cis* 10; C18:1 *cis* 9; C18:1 *cis* 11; C20:1 *cis* 13; C22 *cis* 13;	Ci13:0; C15i:0; Ca15:0; C16(10); Ci17:1(9); C19(10); Ci19i; C20(10); Ci21; C22(14); Ci24:0	C9 C10 C11 C16 C18 C20 C22 C30	Methyl 22-hydroxydocosanoate; Methyl 2-hydroxy-tetracosanoate; Docosanoic acid, 2-hydroxy
AHM-1B	11.9	C16-C22	C16-C22	C16:1 cis 9; C16:1 cis 11; C18:1 cis 9; C18:1 cis 11; C22 cis 13	Ci14 & Ca14:0; C15i:0; C17(10); C18(10);	C11 C13 C16 C18 C20	
AHM-1C (soil)	44.5	C16-C18	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 cis 11; C22 cis 13	Ci11:0; Ci13:0; Ci15:0; Ca15:0; Ci17:0;		tetramethyl 10, 14, 18, 22, tricosanoic acid

					Ca17:0		
AHM-1D (soil)	17.9	C16-C18	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 cis 11; C22 cis 13	Ci11:0; Ci13:0; Ci15:0; Ca15:0; Ci17:0; Ca17:0		
AHM-2A	10.1	C16-C18	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 cis 11	Ci13:0; Ci15:0; Ca15:0; Ci17:0; Ca17:0; C18(11)		tetramethyl 10, 14, 18, 22, tricosanoic acid
AHM-2B	18.6	C16-C18	C16-C18	C16:1 cis 9; C18:1 cis 9; C18:1 cis 11	Ca13:0; Ci14:0; Ci15:0; Ca15:0; Ci17:0; Ca17:0; C18(11);		

6.2. Actualistic FCR ORA

6.2.1. Griddle-cooking

Stone Sample	Lipid concentration ugg^{-1}	SFA range	SFA mains	UFA	BCFAs	DA	Other
X01 (steak)	84	C12-C20	C14-C18	C12:1 cis 9, C17:1 cis 10, C18:1 trans 9 C18:2 cis cis 9, 12, C18:2 cis 9 trans 11, C19:1 cis 10, C20:1 cis 11,	Ca15:0, Ci16:0, Ca17:0, Ci19:0		tetracosane
X02 (trout)	147	C9-C24	C14-C18	C16:1 cis 9, C18:1 trans 9, C18:2 cis 9	Ca15:0, Ci16:0, Ci17:0,	C8 C9 C10	4,8,12 trimethyl tridecanoic acid cholesta-3,5-diene

				trans 11, C20:1 cis 11, C22:1 cis 9, 24:1 cis 15	Ca17:0, Ci19:0	C11	8-oxooctanoic acid, 9-oxononanoic acid, 10-oxodecanoic acid
X03 (hazelnut)	200	C8-C24	C16-C18	C8:1 trans 2, C10:1 trans 2, C18:1 trans 9, C18:2 cis 9 trans 11, C18:2 trans 10 cis 12	Ci19:0	C7 C8 C9 C10 C11	8-oxooctanoic acid, 10-oxodecanoic acid10-oxooctadecanoic acid, decanal
X04 (birch bark)	4	C14-C20	C16-C18	C18:1 trans 9	Ca17:0		octadecane, eicosane
X05 (blank)	1	C14-C24	C16-C18	C18:1 trans 9			
X06 (blank)	0	C14-C18	C16-C18	C18:1 trans 9			
X07 (blank)	0	C16-C18	C16-C18				
X08 (blank)	0	C16-C18	C16-C18				

6.2.2. Latvia pit-cooking

Stone Sample	**Lipid concentration ugg^{-1}**	**SFA range**	**SFA mains**	**UFA**	**BCFA**	**DA**	**Other**
LTV-03	14.1						
LTV-06A	22.3	C14 - C26	C16 - C18	C16:1 *cis* 9 C18:1 *cis* 6 C18:1 *cis* 9 C19:1 *cis* 10 C20:1 *cis* 11 C22:1 *cis* 13 C18:2 (9,12) C18:2 (7,10) C18:2 (13,16)	Ci13:0 Ci15:0 Ca15:0 Ci17:0 Ca17:0 C21(14)	C9 C11	
LTV-06B	31.6	C12 - C28	C16 - C18	C16:1 *cis* 7 C16:1 *cis* 9 C17:1 *cis* 10 C18:1 *cis* 6 C18:1 *cis* 9 C18:1 *cis* 13 C20:1 *cis* 11	Ca13:0 Ci13:0 Ci15:0 Ca15:0 Ca16:0 C16 (10) C19 (10)	C8 C9 C11 C12 C16 C22	Decanoic acid, 10-chloro-10-oxo; 9-oxo noanoic acid, 11-oxo-9 undecanoate;

				C22:1 *cis* 13 C18:2 *cis cis* 9,12 C18:2 *cis* 9, *trans* 11	C21(14)		
LTV-18A	13.5	C14 - C24	C16 - C18	C18:1 *cis* 6 C18:1 *cis* 9 C20:1 *cis* 11	Ca13:0 Ca20:0	C9	Cholesta-5,7,9(11)-trien-3-ol acetate; 9-oxo noanoic acid
LTV-18B	16.1	C14 - C24	C16 - C18	C18:1 *cis* 6 C18:1 *cis* 9		C9	Cholesta-5,7,9(11)-trien-3-ol acetate; 9-oxo noanoic acid
LTV-19	28.2	C14-C18	C16 - C18	16:1 cis 7, 18:1 cis 9	Ci11:0, Ca14:0		
LTV-20	10.4	C8-C18	C16 - C18	18:1 cis 9	Ca14:0		

6.2.3. Laboratory controlled experiment

Stone Sample	**Lipid concentration ugg^{-1}/10**	**SFA range**	**SFA mains**	**UFA**	**DA**	**Other**
LBX-1A	257	C8-C22	C16-C18	C8:1 *trans* 2, C9:1 *trans* 8, C9:1 *trans* 2, C10:1 *trans* 4, C10:1 *trans* 2, C11:1 *trans* 10, C14:1 *cis* 11, C16:1 *cis* 9, C18:2 7,8, C18:1 *cis 9*, C18:2 *cis cis* 9,12,	C4 C5 C7 C8 C9 C10 C11 C12 C13 C14	Octadecanoic acid, 9,10-dihydroxy
LBX-1B	217	C8-C22	C16-C18	C9:1 8, C10:1 trans 4, C10:1 trans 2, C11:1 trans 10, C14:1 cis 11, C16:1 cis 9, C18:1 cis 9	C5 C7 C8 C9 C10 C11 C12 C13	Octadecanoic acid, 9,10-dihydroxy
LBX-1C	113	C8-C22	C16-C18	C8:1 cis 3, C8:1 trans 2, C9:1 trans 8, C10:1 trans 4, C10:1 trans 2, C11:1 trans	C4 C5	Octadecanoic acid, 9,10-

				10, C18:2 7,8, C16:1 cis 9, C18:1 cis 9, C18:2 cis 9, trans 11, C20:1 cis 13,	C7 C8 C9 C10 C11 C12 C13	dihydroxy
LBX-2A	499	C16-C20	C16-C18	C18:1 *cis* 9; C18:1 *trans* 10	C9 C10 C11 C12 C13	1,4; 1,3-1,2-Benzenedicarboxylic acid; 1,2,3; 1,2,4; 1,3,5-Benzenetricarboxylic acid; 1,2,4,5-Benzenetetracarboxylic acid;
LBX-2B	277	C14-C18	C16-C18	C18:1 *cis* 9; C18:1 *trans* 10	C9 C10 C11 C12 C13 C14 C18	1,4; 1,3; 1,2-Benzenedicarboxylic acid; 1,2,3; 1,2,4; 1,3,5-Benzenetricarboxylic acid
LBX-2C	273	C10-	C16-C18	C18:1 *cis* 9; C18:1 *trans* 10	C8 C9 C10 C11 C12	1,4; 1,3; 1,2-Benzenedicarboxylic acid; 1,2,4; 1,3,5-Benzenetricarboxylic acid
LBX-3A	250	C8-C22	C16-C18	C8:1 trans 2, C9:1 trans 8, C9:1 trans 2, C10:1 trans 2, C16:1 cis 9, C18:1 cis 9	C4 C5 C6 C7 C8 C9 C10 C11 C12 C13 C14	1,4; 1,3-Benzenedicarboxylic acid Octadecanoic acid, 9,10-dihydroxy
LBX-3B	244	C8-C22	C16-C18	C10:1 trans 2, C11:1 *trans* 10, C16:1 cis 9, C18:1 cis 9, C20:1 cis 13, C14:1 cis 11,	C4 C5 C7 C8 C9 C10 C11 C12	1,4; 1,3-Benzenedicarboxylic acid

					C13 C14 C18	
LBX-3C	293	C8-C22	C16-C18	C8:1 cis 3, C8:1 trans 2, C9:1 trans 8, C10:1 trans 4, C10:1 trans 2, C10:1 10, C16:1 cis 9, C17:1 cis 10, C18:1 cis 9, C20:1 cis 13, C22:1 *trans* 13,	C4 C5 C7 C8 C9 C10 C11 C12 C13	Octadecanoic acid, 9,10-dihydroxy
LBX-4A	356	C16-C20	C16-C18	C18:1 cis 9	C9	1,4; 1,3; 1,2-Benzenedicarboxylic acid; 1,2,3; 1,2,4; 1,3,5-Benzenetricarboxylic acid; 1,2,3,5; 1,2,4,5-benzentetracarboxylic acid
LBX-4B	73		C16-C18			1,2,4; 1,3,5-Benzenetricarboxylic acid; 1,2,4,5-benzentetracarboxylic acid
LBX-4C	127		C16-C18		C9 C10 C11	1,4; 1,3-Benzenedicarboxylic acid; 1,2,4; 1,3,5-Benzenetricarboxylic acid
LBX-5A	657	C8-C24	C16-C18	C16:1 cis 9, C17:1 cis 10, C20:1 cis 1, C22:1 cis 13,	C9	
LBX-5B	319	C8-C24	C16-C18	C16:1 cis 9, C17:1 cis 10, C20:1 cis 1, C22:1 cis 13, C24:1 cis 15		
LBX-5C	237	C8-C24	C16-C18	C16:1 cis 9, C17:1 cis 10, C20:1 cis 1, C22:1 cis 13	C9	

Name	(*m/z*)	MW	Class	Possible origin	1A	1B	1C	2A	2B	2C	3A	3B	3C	4A	4B	4C	LTV-18A	LTV-18B	X01	X02	X03
APAA C18	105 290	290	alkylphenyl alkanoic acid	Unsaturated fatty acids	X	X	X	X	X	X	X	X	X				X	X	X	X	X
APAA C20	105 318	318	alkylphenyl alkanoic acid	Unsaturated fatty acids						X									X	X	X
APCAA	125 130 134 148 189 203 211 223 246 256 296	296	cyclopentyl alkanoic acid	Unsaturated fatty acids						X											
LCK	239 255 67 283		Ketones	Unsaturated fatty acids																	
oxoVLCFAs	171 186 239 254 351	385 396 410 410 424 424 438 452	Ketones	Unsaturated fatty acids													X	X			
B2CA	31 162	162	PAH					X	X	X	X	X		X		X					
B3CA	31 162 221, 252	252	PAH					X	X	X				X	X	X	X	X			
B4CA	31 162 279, 310	310	PAH					X						X	X						
g-lactone C16	85 236 192 97	254	Lactone	Unsaturated fatty acids															X	X	X
g-lactone C18	85 97 220 264	282	Lactone	Unsaturated fatty acids															X		X
y-Dodecalactone	85 41 55 69	198	Lactone	Thermal alteration animal fat																	
Erucylamide	59 72 83 126 240 294 337	337	Amide	Thermal alteration animal fat															X	X	
Octadecanamide	59 72	283	Amide	Thermal alteration animal																	

	55 238 239 283			fat																	
Hexadecanamide	59 72 212 255	255	Amide	Thermal alteration animal fat																	
9-Octadecenamide, (Z)-	59 72 55 238 281	281	Amide	Thermal alteration animal fat																	

6.3. Experimental birch-bark tar

As part of interpreting sample NST-R02 three experimental birch bark tar samples were analysed using GC-MS. The results are below.

Stone Sample	SFA range	SFA mains	UFA	BCFA	DA	Other
BBT-1	C9 - C24	C18 - C22	C17:1 cis 10 C18:1 trans 10 C18:1 trans 3 C22:1 trans 13	Ca12:0	C9 C18 C22	Methanophenanthren-9β-ol Cucurbitacin b 25-desacetoxy-, phorbol 13,27-Cycloursan-3-one betulin
BBT-2	C8 - C24	C16-C22	C16:1 trans 7, C18:1 trans 10, C20:1 trans 11, C22:1 trans 13		C8 C9 C11 C16 C18 C22	1(2H)-Naphthalenone
BBT-3	C9-C22	C16-C20	C18:1 trans 16, C22:1 trans 13			Anthracene Anthracene 2-methyl Fluoranthene Cyclopentanecarbonitrile Cyclopenta[cd]pyrene Olean-12-ene phorbol

Peak time (mins)	BBT-1 *m/z* values	Peak time (mins)	BBT-2 *m/z* values	Peak time (mins)	BBT-3 *m/z* values
24.325	427, 59	24.322	427, 59	24.321	549, 59
24.664	394, 379, 312, 297, 229, 193, 145, 119, 105	27.172	452, 422, 407, 203, 187	24.847	413, 252, 125, 113
25.415	98, 351, 394, 503	27.34	452, 424, 409, 381, 161, 121	25.825	452, 394, 379, 349, 241, 229, 189, 119
25.827	563, 394, 379, 241, 229	27.461	452, 424, 393, 353, 203, 189, 119, 109, 95	26.816	452, 424, 409, 203
26.82	409, 424, 452	27.635	453, 379, 159	27.343	436, 424, 409, 381, 121
27.004	452, 408, 229, 205, 189, 149, 135, 119, 109, 95, 81	27.717	450, 424, 381, 205, 189, 119	27.401	452, 424, 381, 205, 189, 119, 105, 95
27.161	452, 424, 406, 391, 363, 229, 203, 189, 173, 159, 119, 105, 95, 81	27.83	452, 424, 409, 393, 327, 203, 189, 95	27.462	436, 424, 363, 353, 203, 189, 119, 95
27.344	452, 424, 409, 381	27.983	484, 424, 205, 189, 161, 119, 105, 95	27.731	454, 424, 381, 189, 119
27.404	452, 424, 381, 203, 189	28.296	484, 424, 393, 353, 205, 189, 134, 121, 107, 95	27.83	452, 424, 409, 393, 218, 203, 189, 119, 95
27.458	452, 424, 406, 391, 218, 203, 189, 145, 119, 105, 95, 81	28.697	484, 424, 393, 353, 205, 189, 134, 121, 107, 95	27.986	452, 424, 381, 205, 189, 161, 119, 105, 95
27.723	454, 424, 409, 381, 205, 189, 159, 119, 105, 95	28.909	478, 97, 85, 71, 57	28.294	448, 424, 363, 353, 205, 189, 134, 119, 107, 95, 81
27.831	452, 424, 409, 393, 203, 189, 119, 95, 81	29.08	438, 423, 395, 205, 189, 159, 119, 105	28.447	452, 438, 424, 409, 393, 353, 205, 189, 177, 119, 95
27.99	448, 438, 424, 409, 381, 205, 189, 161, 149, 119, 105, 95, 81	29.655	448, 438, 423, 203, 189, 173, 121, 107, 93	28.696	438, 423, 395, 121
28.295	452, 424, 393, 353, 205, 189, 134, 119, 107, 95, 81	30.201	452, 440, 369, 205, 149, 95, 81	29.082	438, 423, 395, 205, 189, 159, 119, 105, 91
28.447	452, 438, 424, 409, 393, 353, 218, 205, 189, 173, 95	30.273	514, 424, 385, 247, 189, 95	29.654	475, 438, 423, 204, 189, 173, 119, 107, 93
28.703	452, 438, 423, 395, 121	30.412	442, 424, 371, 207, 189, 95, 81	30.198	440, 422, 369, 205, 191, 177, 149, 95, 81
28.909	478, 424, 205, 85, 71, 57				
29.083	438, 423, 395, 205, 189, 159, 119, 105				
29.657	468, 438, 423, 204, 189, 173, 119, 107, 93				
30.201	440, 422, 411, 369, 205, 191, 149, 95, 81				

6.4. APAA results

6.4.1. Neustadt LA 156

Sample	APAA C18	APAA C18 A	APAA C18 B	APAA C18 C	APAA C18 D	APAA C18 E	APAA C18 F	APAA C18 G	APAA C18 H	APAA C18 I	ratio E/H
NST-R01A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R01B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R02A	x	0	865	1742	1450	3418	2916	1516	2447	3516	1.40
NST-R02B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R03A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R03B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R04A	x	0	0	0	0	4254	3991	1050	0	0	0.00
NST-R04B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R05A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R05B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R06	0	0	0	0	0	0	0	0	0	0	0.00
NST-R06A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R07a	0	0	0	0	0	0	0	0	0	0	0.00
NST-R07B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R08A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R08B	x	0	0	0	0	763	742	0	0	0	0.00
NST-R09A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R09B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R10A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R10B	0	0	0	0	0	0	0	0	0	0	0.00
NST-R11A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R11B	x	0	0	0	0	896	782	0	0	0	0.00
NST-R12A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R12B	x	0	0	0	0	0	0	709	0	0	0.00
NST-R13A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R13B	0	0	0	0	0	0	0	0	0	0	0.00

NST-R14A	0	0	0	0	0	0	0	0	0	0	0.00
NST-R14B	x	0	0	0	0	832	721	0	0	0	0.00
NST-R14C	0	0	0	0	0	0	0	0	0	0	0.00

Sample	TMTD	Pristanic acid	Phytanic acid			APAAs
			SRR	RRR	SRR%	
RO1A	absent	absent	0	0	0	absent
RO1B	absent	absent	0	0	0	absent
RO2A	absent	present	9669	3892	71.3	C18
RO2B	absent	present	0	0	0	absent
RO3A	absent	absent	0	0	0	absent
RO3B	absent	present	0	0	0	absent
RO4A	absent	present	0	0	0	C18 trace
RO4B	absent	present	0	0	0	absent
RO5A	absent	present	0	0	0	absent
RO5B	absent	present	0	0	0	absent
RO6A	absent	absent	0	0	0	absent
RO6B	absent	present	0	0	0	absent
RO7A	absent	absent	0	0	0	absent
RO7B	absent	absent	0	0	0	absent
RO8A	absent	absent	0	0	0	absent
RO8B	absent	present	0	0	0	C18 trace
RO9A	absent	absent	0	0	0	absent
RO9B	absent	present	1663	4075	29.0	absent
R10A	absent	present	7926	3647	68.5	absent
R10B	absent	present	0	0	0	absent

R11A	absent	absent	0	0	0	absent
R11B	absent	present	5297	1550	77.4	C18 trace
R12A	absent	absent	0	0	0	absent
R12B	absent	present	3062	0	/	C18 trace
R13A	absent	absent	0	0	0	absent
R13B	absent	present	0	0	0	absent
R14A	absent	present	2408	1760	57.8	absent
R14B	absent	present	4637	2408	65.8	C18 trace
R14C	absent	present	0	0	0	absent

6.4.2. Ormen Lange site 48

Samples code	APAA C18	APAA C20	APAA C18 B	APAA C18 C	APAA C18 D	APAA C18 E	APAA C18 F	APAA C18 G	APAA C18 H	APAA C18 I	ratio E/H	APAA C20/18
NOR-1			0	0	0	0	0	0	0	0	0	0
NOR-2			0	0	0	0	0	0	0	0	0	0
NOR-3			0	0	0	0	0	0	0	0	0	0
NOR-4	x	x	2143	851	1216	1300	1133	744	0	624	0	0.17
NOR-5			0	0	0	0	0	0	0	0	0	0

Table.3. Results from the AQUASIM scan. Key: TMTD, 4,8,12-trimethyltridecanoic acid; SSR, 3S,7R,11R,15-phytanic acid; RRR, 3R,7R,11R,15-phytanic acid; SRR%, ((Area SSR/Area SSR+Area RRR)*100).

Sample	TMTD	Pristanic acid	Phytanic acid			APAAs
			SRR	RRR	SRR%	
NOR-1	present	present	3083	7797	28.3	absent
NOR-2	present	present	3199	10288	23.7	absent
NOR-3	present	present	1764	0	/	absent
NOR-4	present	present	27561	2877	90.6	C18; C20 trace
NOR-5	present	present	6322	8302	43.2	absent

Table.3. Results from the AQUASIM scan. Key: TMTD, 4,8,12-trimethyltridecanoic acid; SSR, 3S,7R,11R,15-phytanic acid; RRR, 3R,7R,11R,15-phytanic acid; SRR%, ((Area SSR/Area SSR+Area RRR)*100).

6.4.3. Griddle-cooking

Sample Code	APAA C18	APAA C20	APAA C18 A	APAA C18 B	APAA C18 C	APAA C18 D	APAA C18 E	APAA C18 F	APAA C18 G	APAA C18 H	APAA C18 I	ratio E/H	APAA C20/18
X01	x	x	979	2201	665	665	3519	3470	8816	7352	3533	0.48	0.06
X02	x	x	1171	1781	3281	6246	8959	1962	14112	57779	4950	0.16	0.58
X03	x	x	873	1390	2441	6889	6229	871	2677	4966	3233	1.25	0.06
X04	x	x	0	0	0	0	6623	3052	4262	1169	898	5.67	0.00

Sample	TMTD	Pristanic acid	Phytanic acid			APAAs
			SRR	RRR	SRR%	
X01	present	absent	0	0	0	C18, C20 trace
X02	present	present	28366.0	47056.0	37.61	C18, C20
X03	present	absent	0	0	0	C18, C20 trace
X04	absent	absent	27681.0	9123.0	75.21	C18

6.4.4. Latvia pit-cooking

Sample	APAA C18	APAA C18 A	APAA C18 B	APAA C18 C	APAA C18 D	APAA C18 E	APAA C18 F	APAA C18 G	APAA C18 H	APAA C18 I	ratio E/H
LTV-18A	x	0	0	1043	2100	9363	9546	2955	1263	1371	7.4
LTV-18B	x	0	0	296	676	3292	3815	1159	440	430	7.5

Sample	TMTD	Pristanic acid	Phytanic acid			APAAs
			SRR	RRR	SRR%	
LTV-18A	absent	present	300	251	54.4	C18

LTV-18B	absent	present	119	119	50.0	C18

6.4.5. Laboratory controlled experiment

Sample	APAA C18	APAA C20	APAA C18 A	APAA C18 B	APAA C18 C	APAA C18 D	APAA C18 E	APAA C18 F	APAA C18 G	APAA C18 H	APAA C18 I	ratio E/H	APAA C20/18
LBX-1A	x	0	3308	9227	10559	39220	139725	159607	127957	54808	35385	2.5	0
LBX-1B	x	0	1361	2738	2389	6001	24470	24839	6396	1372	3930	17.8	0
LBX-1C	x	0	2348	1863	5345	5632	27762	29821	18438	7037	5814	3.9	0
LBX-2A	x	0	0	0	1160	1258	3669	4954	2701	1614	2000	2.3	0
LBX-2B	x	0	1378	2053	2306	4756	8638	12171	9524	6137	5662	1.4	0
LBX-2C	x	x	28770	45530	53543	94839	133689	174366	111460	66586	62531	2.0	0.09
LBX-3A	x	0	6094	12084	18721	42754	92110	142084	83709	31451	22377	2.9	0
LBX-3B	x	0	5119	13040	3727	7781	18045	16891	4495	719	5148	25.1	0
LBX-3C	x	0	1353	2862	5083	11908	57414	57424	11819	4338	5285	13.2	0
LBX-4A	0	0	0	0	0	0	0	0	0	0	0	0	0
LBX-4B	0	0	0	0	0	0	0	0	0	0	0	0	0
LBX-4C	0	0	0	0	0	0	0	0	0	0	0	0	0
LBX-5A	0	0	0	0	0	0	0	0	0	0	0	0	0
LBX-5B	0	0	0	0	0	0	0	0	0	0	0	0	0
LBX-5C	0	0	0	0	0	0	0	0	0	0	0	0	0

Sample	TMTD	Pristanic acid	Phytanic acid			APAAs
			SRR	RRR	SRR%	
LBX-1A	yes	yes	446.0	1667	21.1	C18
LBX-1B	yes	yes	385.0	319	54.7	C18
LBX-1C	yes	yes	898.0	4582	16.4	C18
LBX-2A	no	yes	156.0	157	49.8	C18
LBX-2B	yes	yes	355.0	359	49.7	C18
LBX-2C	yes	yes	5160.0	6286	45.1	C18 C20
LBX-3A	yes	yes	4127.0	107	97.5	C18
LBX-3B	yes	yes	2642.0	644	80.4	C18
LBX-3C	yes	yes	590.0	581	50.4	C18
LBX-4A	no	no	0	0	0	0

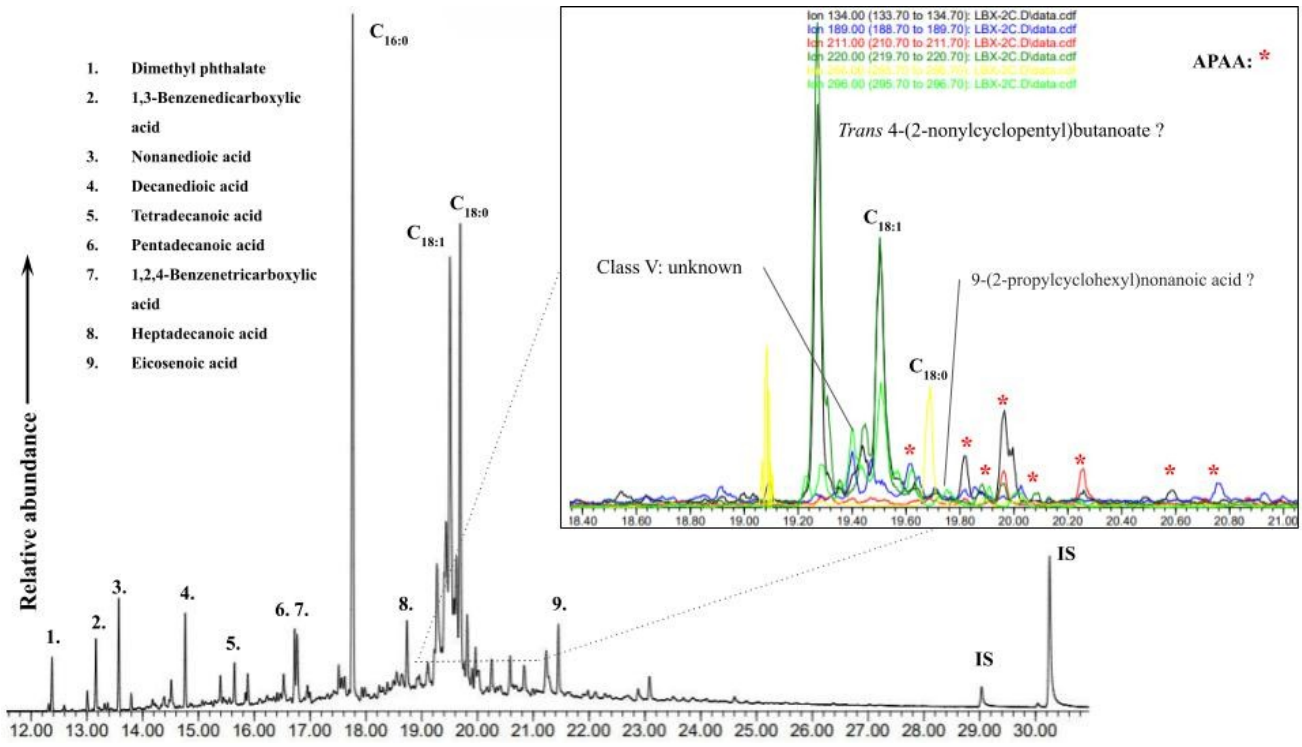
LBX-4B	no	no	0	0	0	0
LBX-4C	no	no	0	0	0	0
LBX-5A	no	no	0	0	0	0
LBX-5B	no	no	0	0	0	0
LBX-5C	no	no	0	0	0	0

6.4.6. APAA reference sources for figure.8.1

Product	APAA-C18 distribution									Source
	A (%)	B (%)	C (%)	D (%)	E (%)	F (%)	G (%)	H (%)	I (%)	
Rape seed oil	0.4	1.1	2.2	5.1	13.1	17.8	30.1	16.4	13.9	Bondetti et al 2021
Rape seed oil	0.4	1.1	2.5	5.1	14.6	18.2	27.1	16.9	14.1	Bondetti et al 2021
Rape seed oil	0.4	1.1	2.8	5.3	17	18.5	25.7	15.7	13.5	Bondetti et al 2021
Rape seed oil	0.4	1.1	2.8	5.1	16.2	18.6	26.5	16.2	13.2	Bondetti et al 2021
Rape seed oil	0.7	1.7	3.9	5.2	20.7	27.7	19.5	12.8	7.9	Bondetti et al 2021
Rape seed oil	1.2	2.8	4.4	8.4	22.2	25.6	16.5	10.5	8.4	Bondetti et al 2021
Rape seed oil	0.8	2.4	3.5	6.3	26	31.3	12.8	10.1	6.9	Bondetti et al 2021
Rape seed oil	0	0.9	3.2	5.1	34.6	37.4	10.2	5.1	3.5	Bondetti et al 2021
Red Deer1	1.4	3.7	5.2	9.3	27.8	31.4	12.1	9.3	0	Bondetti et al 2021
Organic Butter	1.8	4.8	8.5	13.9	22.6	20.8	11.2	7.5	8.8	Bondetti et al 2021
Elk	2.4	4.7	7.4	11.5	22.6	24.6	10.2	8.5	8.1	Bondetti et al 2021
Red Deer	1	2.1	3.3	6.9	28.9	33.1	10.7	8.1	6	Bondetti et al 2021
Beaver	1.8	3.7	4.1	7.2	29.6	31.8	8.7	6.4	6.7	Bondetti et al 2021
Pork	2.9	5.6	12.6	12.6	25.1	22.7	8.1	4.3	6.1	Bondetti et al 2021
Cod Liver Oil	0	0	3.5	16.9	22.4	22.8	17.1	9.5	7.8	Bondetti et al 2021
Salmon Fat	0	0	4.4	6.1	26.5	31.3	9.6	13.6	8.4	Bondetti et al 2021
Salmon Oil	0	1.4	3.9	7.4	28.3	31.9	11.9	8.7	6.5	Bondetti et al 2021
Duck	1.3	2.8	4.1	7.2	24.4	30.1	11.1	12.2	6.8	Bondetti et al 2021
Catfish	2.5	5.2	6.4	12.7	29.3	21.5	8.6	7.7	6.1	Bondetti et al 2021

Sturgeon	1.8	3.7	5.1	8.4	29.4	30.8	8.9	5.8	6.3	Bondetti et al 2021
Unio Shellfish	1.5	2.9	5	8.5	24.2	28.1	12.8	10.1	6.9	Bondetti et al 2021
Viviparus Shellfish	1.4	2.8	3.6	6.6	23.2	29.2	12.7	13	7.5	Bondetti et al 2021
Perch	2	4.4	6.2	10	23.1	23.9	12.3	9.9	8.2	Bondetti et al 2021
Chestnut Flour	3.2	4.9	5.3	8.1	25.6	28.3	10.5	6.8	7.4	Bondetti et al 2021
Hazelnut Oil	1.1	2.7	4.1	7.6	25.7	29.1	13.6	9.2	6.8	Bondetti et al 2021
Hemp Oil	1.6	3.9	6.1	11	28.8	28.1	9.4	5.1	6.1	Bondetti et al 2021
Rice Bran Oil	1.2	2.9	3.8	8.1	31.1	32.5	9.6	5.2	5.7	Bondetti et al 2021
Walnut Oil	1.3	2.5	3	7.1	28.1	30.9	12.9	7.7	6.6	Bondetti et al 2021
Leek leave	0.8	2.1	2.8	5.7	20.7	30.2	14.1	14.3	9.2	Bondetti et al 2021
Leek white part	1	2.4	3.2	6.5	28.8	33.1	10.8	7.5	6.8	Bondetti et al 2021
Onion	1.2	2.6	4	8.5	28.6	30.6	10.7	6.1	7.5	Bondetti et al 2021
Cabbage	0.9	2.4	2.4	4.1	15	28.6	18.1	19	9.5	Bondetti et al 2021
Almond	1.5	3.1	3.7	6.4	34.9	35.2	6.1	4	5.2	Bondetti et al 2021
Walnut	1.6	3	3.5	6.3	31.6	34	8.2	5.4	6.5	Bondetti et al 2021
Wheat	1.5	2.9	3.5	6.1	31.9	34.6	8.1	5.1	6.4	Bondetti et al 2021
Barley	1.3	2.6	3	5.6	32.3	35.3	7.9	5.5	6.4	Bondetti et al 2021
Carrot leave	0.8	2.5	2.3	4.5	24.3	33.3	12.4	12.4	7.5	Bondetti et al 2021
Carrot	1.3	3.3	3.3	6.5	28.1	32.1	10.9	7.4	7.1	Bondetti et al 2021
Olive oil	3.4	6.4	9	14	22.1	19.3	12.9	6.2	6.7	Bondetti et al 2021
Pistachio	1.7	4.2	6.7	10.8	31.6	29.5	6.3	4	5.2	Bondetti et al 2021
Viburnum	1.9	3.8	5.2	8.9	29.3	29.7	8.8	5.6	6.9	Bondetti et al 2021
Spinach	0.8	2.4	3.5	3.4	11.8	29	16.4	23.5	9.3	Bondetti et al 2021
Millet seed	1.5	3	7.1	7.1	29.9	31.5	8.6	5.1	6.4	Bondetti et al 2021
Quinoa seed	1.4	3	6.2	6.2	29.3	32.5	8.6	6.4	6.3	Bondetti et al 2021
Rice grain	2	3.9	4.9	8.2	31.4	31.4	7.9	4.6	5.8	Bondetti et al 2021
Sesame seed	1.7	3.4	4.1	7	31.6	33.4	7.9	4.7	6.2	Bondetti et al 2021
Acorn seed	1.7	3.6	4.5	8.1	26.6	29.2	10.9	7.5	7.9	Bondetti et al 2021

6.5. APCAA chromatogram



6.6. BPCA table

Samples	B2CA isomers	B3CA isomers	B4CA isomers
LBX-1A	/	/	/
LBX-1B	/	/	/
LBX-1C	/	/	/
LBX-2A	1,2; 1,3; 1,4	1,2,3; 1,2,4; 1,3,5	1,2,3,5; 1,2,4,5
LBX-2B	1,3; 1,4	1,2,4; 1,3,5	/
LBX-2C	1,3; 1,4	1,2,4; 1,3,5	/
LBX-3A	1,3; 1,4	/	/
LBX-3B	1,3; 1,4	/	/
LBX-3C	/	/	/
LBX-4A	1,3; 1,4	1,2,3; 1,2,4; 1,3,5	1,2,3,5; 1,2,4,5
LBX-4B	/	1,2,4; 1,3,5	1,2,4,5
LBX-4C	1,3; 1,4	1,2,4; 1,3,5	/
LBX-5A	/	/	/
LBX-5B	/	/	/

LBX-5C	/	/	/
LTV-06A	1,2	1,2,4	/
LTV-06B	1,3	1,2,4	/
LTV-18A	1,4	1,2,4; 1,3,5	/
LTV-18B	1,4	1,2,4; 1,3,5	/

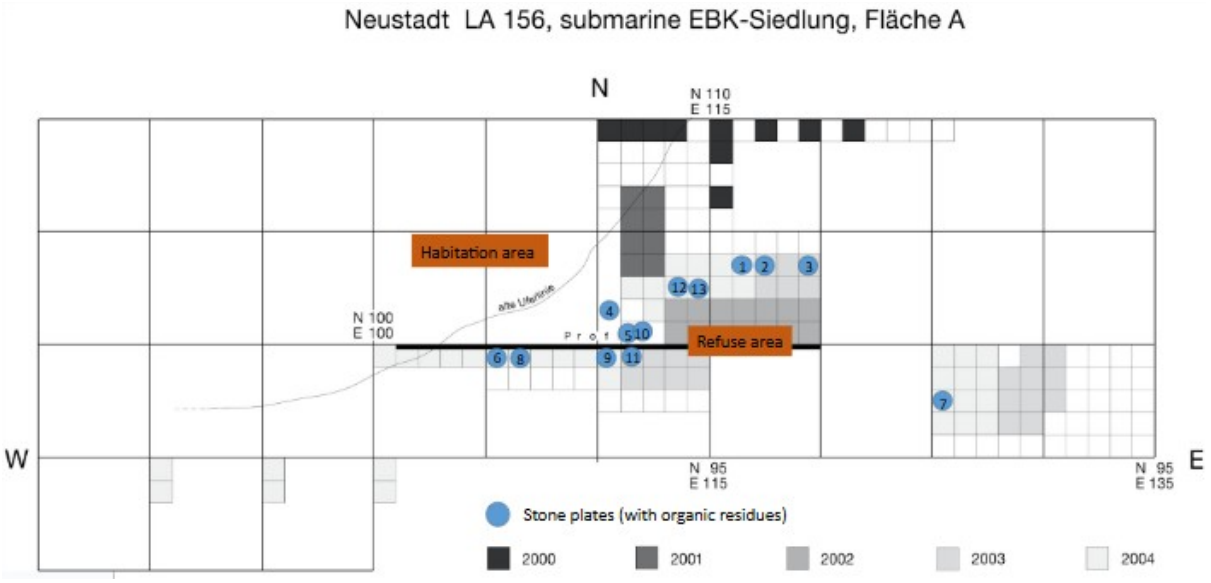
7.0 Appendix Seven: **Radiocarbon dates for the archaeological sites (Chapter 7)**

7.1. Neustadt LA 156

The occupation phases for Neustadt LA 156 are not well resolved due to the difficulty of establishing stratigraphic divisions during underwater excavations (Glykou, 2016). Radiocarbon dating has established a transition from EBK to TRB over a 600 year time span.

Sample	Method	Phase	Material	Age BP	Age cal BC (2σ)	Reference
N-1178	^{14}C	EBK	Food crust	5548 ± 26	4450–4350	(Hartz and Lübke, 2006)
N-1178a	^{14}C	EBK	Food crust	5625 ± 31	4500–4350	(Hartz and Lübke, 2006)
N-3251	^{14}C	EBK	Food crust	5823 ± 35	4800–4550	(Hartz and Lübke, 2006)
N-3251a	^{14}C	EBK	Food crust	5832 ± 32	4800–4600	(Hartz and Lübke, 2006)
N-629	^{14}C	EBK	Food crust	5460 ± 90	4450–4050	(Hartz and Lübke, 2006)
N-629a	^{14}C	EBK	Food crust	5350 ± 80	4350–4000	(Hartz and Lübke, 2006)
N-1025	^{14}C	EBK	Food crust	5754 ± 30	4700–4500	(Hartz and

						Lübke, 2006)
N-1178	^{14}C	EBK	Charcoal/ pottery	5374 ± 54	4350–4050	(Hartz and Lübke, 2006)
N-868	^{14}C	EBK	Charcoal/ pottery	5298 ± 41	4250–4000	(Hartz and Lübke, 2006)
N-2751	^{14}C	EBK	Charcoal/ pottery	5597 ± 39	4500–4350	(Hartz and Lübke, 2006)
N-957	^{14}C	EBK	Charcoal/ pottery	5467 ± 39	4450–4250	(Hartz and Lübke, 2006)
N-441–442 GE 146	^{14}C	TRB	Food crust	5548 ± 26	4450–4350	(Hartz and Lübke, 2006)
N-1457	^{14}C	TRB	Food crust	5190 ± 29	4050–3950	(Hartz and Lübke, 2006)
N-1494	^{14}C	TRB	Food crust	5354 ± 31	4350–4050	(Hartz and Lübke, 2006)
N-1495	^{14}C	TRB	Food crust	5424 ± 32	4350–4250	(Hartz and Lübke, 2006)
N-2636 GE 50	^{14}C	TRB	Food crust	5418 ± 27	4350–4250	(Hartz and Lübke, 2006)
N-1495	^{14}C	TRB	Charcoal/ pottery	5122 ± 63	4050–3700	(Hartz and Lübke, 2006)
N-2131	^{14}C	TRB	Charcoal/ pottery	5214 ± 57	4250–3850	(Hartz and Lübke, 2006)
GE 142	^{14}C	TRB	Charcoal/ pottery	5128 ± 65	4150–3700	(Hartz and Lübke, 2006)
KIA-30590	^{14}C	TRB	Cattle bone	5235±31	4051-3981	(Glykou, 2016)
KIA-39767	^{14}C	TRB	Cattle bone	5055±28	3942-3798	(Glykou, 2016)
KIA-29092	^{14}C	TRB	Cattle bone	5010±34	3912-3713	(Glykou, 2016)



The map of the habitation and refuse areas of Neustadt LA 156 (after Hartz, pers comm)

7.2. Grube-Rosenfelde LA 83

Sample	Method	Phase	Material	Age BP	Age cal BC (2σ)	Reference
KIA-14651	^{14}C	EBK	Hazelnut shell	5951 ± 35	4824 ± 56	(Hartz and Lübke, 2006)
KIA-16637	^{14}C	EBK	Aurochs bone	6045 ± 31	4923 ± 56	(Hartz and Lübke, 2006)
KIA-22668	^{14}C	EBK	Wood	5729 ± 31	4640-4520	(Hartz 2005)
KIA-22669	^{14}C	EBK	Hearth charcoal	6304 ± 27	5320-5260	(Hartz 2005)
KIA-22670	^{14}C	EBK	Hazelnut shell	5931 ±32	4830-4750	(Hartz 2005)

7.3. Ormen Lange 48 Site: A-Y

Sample	Method	Phase	Age BP	Age cal BC (2σ)	Reference
TUa-3576	^{14}C	EM	9410 ± 55	8760 - 8620	(Bjerck et al. 2008)
TUa-3297	^{14}C	EM	9515 ± 70	9120–8740	(Bjerck et al. 2008)
T-16928	^{14}C	EM	9445 ± 130	9150–8550	(Bjerck et al. 2008)
T-17186	^{14}C	EM	9480 ± 125	9130–8630	(Bjerck et al. 2008)
T-17001	^{14}C	EM	9485 ± 110	9120–8630	(Bjerck et al. 2008)
TUa-4589	^{14}C	EM	9380 ± 70	8750–8560	(Bjerck et al. 2008)

7.4. Havnø

Sample	Method	Material	Age BP	Age cal BC (1σ)	Reference
AAR-10620	^{14}C	*Bos taurus*	4998 ± 47	3810-3660	(Robson 2015)
AAR-10621	^{14}C	*Ostrea edulis*	6478 ± 42	4670-4370	(Robson 2015)
AAR-10622	^{14}C	*Ostrea edulis*	6295 ± 40	4460-4220	(Robson 2015)
AAR-11930	^{14}C	*Bos taurus*	5015 ± 41	3890-3660	(Robson 2015)
AAR-13466	^{14}C	*Bos taurus*	5100 ± 44	3980-3780	(Robson 2015)

AAR-13467	^{14}C	*Ostrea edulis*	5715 ± 40	3870-3580	(Robson 2015)
AAR-14672	^{14}C	*Ostrea edulis*	6230 ± 37	4420-4130	(Robson 2015)
AAR-14673	^{14}C	*Ostrea edulis*	6209 ± 37	4370-4080	(Robson 2015)
AAR-15644	^{14}C	*Ostrea edulis*	5646 ± 33	3760-3590	(Robson 2015)
AAR-17177	^{14}C	*Ostrea edulis*	6367 ± 35	4510-4280	(Robson 2015)
AAR-17178	^{14}C	*Cerastoderma edule*	6117 ± 47	4300-4000	(Robson 2015)
AAR-17179	^{14}C	*Ostrea edulis*	5927 ± 39	4080-3780	(Robson 2015)
AAR-19082	^{14}C	*Ostrea edulis*	5137 ± 27	3170-2870	(Robson 2015)
AAR-19083	^{14}C	*Ostrea edulis*	5223 ± 14	3300-2960	(Robson 2015)
K-6918	^{14}C	*Ostrea edulis*	5130 ± 65	3290-2820	(Robson 2015)
K-6919	^{14}C	*Ostrea edulis*	4820 ± 80	2860-2360	(Robson 2015)
OXA-27064	^{14}C	*Ovis/Capra*	5329 ± 35	4310-4050	(Robson 2015)
SUERC-42620 (GU25952)	^{14}C	*Homo sapiens*	5067 ± 29	3950-3710	(Robson 2015)
SUERC-42621 (GU25953)	^{14}C	*Homo sapiens*	4101 ± 29	2860-2470	(Robson 2015)
SUERC-42625 (GU25954)	^{14}C	*Homo sapiens*	4233 ± 29	2870-2400	(Robson 2015)
SUERC-42626 (GU25955)	^{14}C	*Homo sapiens*	5880 ± 29	4210-3810	(Robson 2015)
SUERC-42627 (GU25956)	^{14}C	*Homo sapiens*	5869 ± 29	4210-3800	(Robson 2015)
Ua-35185	^{14}C	*Bos taurus*	3610 ± 45	2140-1880	(Robson 2015)
Ua-35186	^{14}C	Unknown (burnt fragment)	5455 ± 40	4360-4240	(Robson 2015)
Ua-35187	^{14}C	Unknown (splinter)	5630 ± 45	4540-4360	(Robson 2015)

UBA-20175	^{14}C	*Ovis/Capra*	4883 ± 29	3710-3640	(Robson 2015)
UBA-20176	^{14}C	*Bos taurus*	4777 ± 26	3640-3520	(Robson 2015)
UBA-20177	^{14}C	*Bos taurus*	4927 ± 28	3760-3650	(Robson 2015)
UBA-20178	^{14}C	*PhocaHalichoerus*	5648 ± 28	3810-3630	(Robson 2015)
UBA-20179	^{14}C	*Canis familiaris*	5574 ± 31	3770-3580	(Robson 2015)
UBA-20320	^{14}C	*Ovis aries*	4848 ± 41	3710-3530	(Robson 2015)

8.0 Appendix Eight: Stable carbon isotopes of n-hexadecanoic (C16:0) and n-octadecanoic (C18:0) acids for reference fats from contemporary animal fats for Figure 7.5

Sample	Species	Location	δ ^{13}C16:0 (‰)	δ ^{13}C18:0 (‰)	Δ ^{13}C (C18:0- C16:0)	Reference
Ruminant adipose	*Bos taurus*	Kärkölä	-27.6	-29.0	-1.4	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Bos taurus*	n/a	-27.2	-28.7	-1.5	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Bos taurus*	Koski as.	-27.0	-29.4	-2.4	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Bos taurus*	n/a	-27.8	-29.7	-1.9	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Bos taurus*	Estonia, Võru	-28.0	-30.1	-2.2	(Courel *et al.*,

		county				2020)
Ruminant adipose	*Ovis aries*	Estonia, Metsakivi	-30.3	-31.8	-1.6	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Kuhmo	-30.2	-32.2	-1.9	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Alces alces*	Kuhmo	-28.8	-30.3	-1.5	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Alces alces*	Oripää	-29.8	-32.2	-2.4	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Alces alces*	Kuhmo	-29.2	-31.4	-2.2	(Pääkkönen, Evershed and Asplund, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi	-30.0	-32.0	-1.9	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-31.2	-32.7	-1.5	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-31.3	-33.0	-1.8	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-31.6	-33.0	-1.4	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-33.4	-34.7	-1.2	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-31.1	-33.3	-2.2	(Courel *et al.*, 2020)

Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-30.1	-30.7	-0.6	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-32.8	-34.5	-1.6	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-29.4	-31.4	-2.0	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-32.2	-32.6	-0.4	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-30.8	-32.1	-1.3	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-30.7	-32.3	-1.5	(Courel *et al.*, 2020)
Ruminant adipose	*Alces alces*	Estonia, Alatskivi/Metsakivi	-32.6	-33.9	-1.2	(Courel *et al.*, 2020)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-27.8	-31.6	-3.8	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-27.5	-31.2	-3.7	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-28.5	-32.7	-4.2	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-30.1	-33.8	-3.7	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-29.2	-32.8	-3.6	(Craig *et al.*, 2012)

Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-28.9	-33.1	-4.2	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-30.5	-33.1	-2.6	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-29.6	-33.2	-3.6	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-29.0	-32.4	-3.4	(Craig *et al.*, 2012)
Ruminant adipose	*Cervus elaphus*	Poland, Slowinski National Park	-29.5	-33.1	-3.6	(Craig *et al.*, 2012)
Porcine	*Sus scrofa domesticus*	n/a	-27.4	-25.8	1.6	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa domesticus*	n/a	-28.1	-26.7	1.5	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa domesticus*	n/a	-27.4	-26.9	0.6	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa domesticus*	n/a	-27.2	-25.6	1.5	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa domesticus*	Ilomantsi	-26.5	-25.7	0.8	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa ferus*	Ilomantsi	-27.0	-26.9	0.0	(Pääkkönen, Evershed and Asplund,

						2020)
Porcine	*Sus scrofa ferus*	Estonia, Alatskivi	-26.9	-25.8	1.1	(Courel *et al.*, 2020)
Dairy	*Capra hircus*	Laitila	-27.5	-31.2	-3.7	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Capra hircus*	Laitila	-27.5	-32.5	-5.1	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Capra hircus*	Laitila	-27.8	-31.8	-3.9	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	Laitila	-27.3	-31.0	-3.6	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	Laitila	-27.1	-30.9	-3.9	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	Laitila	-27.0	-31.8	-4.8	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	Laitila	-26.9	-31.4	-4.6	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	n/a	-27.8	-31.9	-4.1	(Pääkkönen, Evershed and Asplund, 2020)
Dairy	*Bos taurus*	Estonia, Metsakivi	-25.8	-34.5	-8.7	(Courel *et al.*, 2020)

Dairy	*Bos taurus*	Southern UK	-31.0	-34.6	-3.6	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-28.0	-32.3	-4.3	(Dudd, 1999)
Dairy	*Ovis aries*	Southern UK	-29.6	-34.0	-4.4	(Dudd, 1999)
Dairy	*Ovis aries*	Southern UK	-29.2	-33.6	-4.4	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-27.6	-32.4	-4.8	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-29.1	-33.9	-4.8	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-29.8	-35.1	-5.3	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-28.1	-33.3	-5.2	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-28.8	-34.3	-5.5	(Dudd, 1999)
Dairy	*Bos taurus*	Southern UK	-28.3	-34.2	-5.9	(Dudd, 1999)
Freshwater	*Abramis brama*	Lake Niinivesi, Äänekoski	-36.2	-34.8	1.4	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Abramis brama*	Lake Niinivesi, Äänekoski	-34.3	-34.7	-0.4	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Abramis brama*	Lake Niinivesi, Äänekoski	-35.8	-35.3	0.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Abramis brama*	Lake Kellojärvi, Kuhmo	-34.1	-32.6	1.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Lota lota*	Lake Kellojärvi, Kuhmo	-34.6	-32.6	2.0	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Leuciscus idus*	Lake Pajalampi, Kuhmo	-33.1	-31.5	1.6	(Pääkkönen, Evershed and Asplund,

						2020)
Freshwater	*Leuciscus idus*	Lake Pajalampi, Kuhmo	-33.6	-32.0	1.6	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Esox lucius*	Lake Pönkälampi, Kuhmo	-33.5	-33.1	0.4	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Esox lucius*	Lake Niinivesi, Äänekoski	-32.0	-31.5	0.4	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Esox lucius*	Lake Kellojärvi, Kuhmo	-33.7	-32.5	1.2	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Murtojärvi, Kuhmo	-32.3	-32.5	-0.2	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Pönkälampi, Kuhmo	-35.3	-34.9	0.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Niinivesi, Äänekoski	-34.5	-33.4	1.1	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Valkea-Kotinen, Hämeenlinna	-35.2	-33.5	1.6	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Valkea-Kotinen, Hämeenlinna	-33.5	-32.8	0.7	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Valkea-	-33.2	-32.5	0.7	(Pääkkönen,

		Kotinen, Hämeenlinna				Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Pajalampi, Kuhmo	-35.4	-35.0	0.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Hujakko, Äänekoski	-32.2	-32.8	-0.6	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Lake Pajalampi, Kuhmo	-35.5	-37.1	-1.6	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Sander lucioperca*	Lake Kellojärvi, Kuhmo	-34.7	-33.9	0.8	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Sander lucioperca*	n/a	-30.5	-30.2	0.3	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Rutilus rutilus*	Lake Hujakko, Äänekoski	-29.6	-32.1	-2.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Rutilus rutilus*	Lake Pajalampi, Kuhmo	-34.2	-33.1	1.1	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Rutilus rutilus*	Lake Peipus/Peipsi	-29.6	-29.6	0.0	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Coregonus albula*	Lake Vanajanselkä	-28.5	-27.0	1.4	(Courel *et al.*, 2020)
Freshwater	*Coregonus*	Lake Lentua,	-37.1	-38.0	-1.0	(Pääkkönen,

	albula	Kuhmo				Evershed and Asplund, 2020)
Freshwater	*Coregonus albula*	Lake Puruvesi	-29.4	-28.1	1.3	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Coregonus albula*	Lake Puruvesi	-28.2	-29.7	-1.5	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Tinca tinca*	Estonia, Kääpa	-32.7	-31.1	1.6	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Perca fluviatilis*	Estonia, Kääpa	-31.8	-31.0	0.8	(Courel *et al.*, 2020)
Freshwater	*Perca fluviatilis*	Estonia, Kääpa	-33.9	-31.6	2.3	(Courel *et al.*, 2020)
Freshwater	*Esox lucius*	Estonia, Kääpa	-32.8	-31.6	1.1	(Courel *et al.*, 2020)
Freshwater	*Gobio gobio*	Estonia, Kääpa	-36.2	-35.4	0.8	(Courel *et al.*, 2020)
Freshwater	*Gobio gobio*	Estonia, Kääpa	-36.2	-35.1	1.0	(Courel *et al.*, 2020)
Freshwater	*Rutilus rutilus*	Estonia, Võõpsu	-35.5	-34.8	0.7	(Courel *et al.*, 2020)
Freshwater	*Rutilus rutilus*	Estonia, Võõpsu	-36.1	-35.8	0.3	(Courel *et al.*, 2020)
Freshwater	*Rutilus rutilus*	Estonia, Võõpsu	-37.2	-36.0	1.2	(Courel *et al.*, 2020)
Freshwater	*Rutilus rutilus*	Estonia, Võõpsu	-33.9	-33.7	0.1	(Courel *et al.*, 2020)
Freshwater	*Esox lucius*	Estonia, Võõpsu	-32.6	-31.7	0.9	(Courel *et al.*, 2020)

Freshwater	*Esox lucius*	Estonia, Võõpsu	-32.8	-31.7	1.2	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Pori	-28.5	-28.8	-0.3	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Castor fiber*	Satakunta	-27.9	-29.1	-1.2	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Castor fiber*	Satakunta	-30.1	-31.1	-1.1	(Pääkkönen, Evershed and Asplund, 2020)
Freshwater	*Castor fiber*	Mehikoorma	-30.1	-30.3	-0.2	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-31.4	-32.0	-0.7	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-31.1	-31.6	-0.5	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-31.2	-31.6	-0.4	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-30.1	-30.2	-0.1	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-29.4	-29.7	-0.3	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-31.0	-30.7	0.2	(Courel *et al.*, 2020)
Freshwater	*Castor fiber*	Estonia, Mehikoorma	-31.3	-32.0	-0.7	(Courel *et al.*, 2020)
Marine	*Clupea harengus membras*	Bay of Bothnia, Sweden	-26.7	-25.9	0.9	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Clupea*	Sea of Bothnia	-27.4	-30.1	-2.7	(Pääkkönen,

	harengus membras					Evershed and Asplund, 2020)
Marine	*Clupea harengus membras*	Sea of Bothnia	-27.4	-27.0	0.4	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Clupea harengus membras*	Sea of Bothnia	-26.9	-29.2	-2.3	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Clupea harengus membras*	Sea of Bothnia	-26.5	-25.3	1.2	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Clupea harengus membras*	Baltic Sea	-23.7	-23.2	0.6	(Courel *et al.*, 2020)
Marine	*Esox lucius*	Rymättylä, Finnish Archipelago Sea	-23.0	-23.2	-0.2	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Esox lucius*	Askainen, Finnish Archipelago Sea	-23.9	-23.2	0.7	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Esox lucius*	Askainen, Finnish Archipelago Sea	-23.0	-22.1	1.0	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Perca fluviatilis*	Sea of Åland	-23.0	-22.4	0.5	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Perca fluviatilis*	Sea of Åland	-23.4	-22.2	1.3	(Pääkkönen, Evershed and Asplund, 2020)

Marine	*Perca fluviatilis*	Sea of Åland	-23.6	-21.8	1.8	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Perca fluviatilis*	Sea of Åland	-23.6	-23.0	0.6	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Sander lucioperca*	Rymättylä, Finnish Archipelago Sea	-23.4	-22.7	0.7	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Sander lucioperca*	Luonnonmaa, Finnish Archipelago Sea	-25.0	-24.5	0.5	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Sander lucioperca*	Parainen, Finnish Archipelago Sea	-22.8	-22.6	0.2	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Rutilus rutilus*	Seili, Finnish Archipelago Sea	-21.7	-23.2	-1.5	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Rutilus rutilus*	Seili, Finnish Archipelago Sea	-22.7	-23.3	-0.6	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Salmo salar*	Askainen, Finnish Archipelago Sea	-23.6	-23.2	0.4	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Salmo salar*	Peimari, Finnish Archipelago Sea	-24.3	-22.9	1.4	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Sprattus sprattus*	Sea of Bothnia	-24.8	-24.3	0.5	(Pääkkönen, Evershed and Asplund,

						2020)
Marine	*Sprattus sprattus*	Sea of Bothnia	-26.2	-25.4	0.8	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Sprattus sprattus*	Sea of Bothnia	-26.4	-27.0	-0.5	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Coregonus lavaretus*	Luonnonmaa, Finnish Archipelago Sea	-23.5	-23.7	-0.2	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Coregonus lavaretus*	Finnish Archipelago Sea	-25.6	-23.9	1.6	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Coregonus lavaretus*	Luonnonmaa, Finnish Archipelago Sea	-24.5	-24.1	0.4	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Coregonus lavaretus*	Rymättylä, Finnish Archipelago Sea	-23.7	-22.9	0.8	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Rutilus rutilus*	Seili, Finnish Archipelago Sea	-22.5	-22.4	0.1	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Platichthys flesus*	Baltic Sea	-24.3	-24.4	-0.2	(Courel *et al.*, 2020)
Marine	*Halichoerus grypus*	Isokari, Uusikaupunki, Finnish Archipelago Sea	-23.7	-23.6	0.1	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Halichoerus grypus*	Merikarvia, Sea of Bothnia	-25.3	-25.4	-0.1	(Pääkkönen, Evershed and

						Asplund, 2020)
Marine	*Halichoerus grypus*	South Kälö, Korppoo, Finnish Archipelago Sea	-22.9	-23.3	-0.3	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Halichoerus grypus*	Estonia, Pärnu Bay, Gulf of Riga	-23.3	-23.4	-0.2	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Halichoerus grypus*	Brändö, Åva, Finnish Archipelago Sea	-23.0	-23.6	-0.6	(Pääkkönen, Evershed and Asplund, 2020)
Marine	*Halichoerus grypus*	Estonia, Leppneeme harbour	-24.0	-24.4	-0.4	(Pääkkönen, Evershed and Asplund, 2020)
Porcine	*Sus scrofa domesticus*	Southern UK	-25.0	-24.2	0.8	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-26.6	-25.0	1.6	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-26.7	-25.0	1.7	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-25.7	-24.6	1.1	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-26.2	-25.1	1.1	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-25.3	-24.0	1.3	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-25.9	-24.6	1.3	(Dudd, 1999)
Porcine	*Sus scrofa domesticus*	Southern UK	-25.6	-24.3	1.3	(Dudd, 1999)

Porcine	*Sus scrofa domesticus*	Switzerland, Langerbruck	-28.9	-27.6	1.2	(Spangenberg, Jacomet and Schibler, 2006)
Porcine	*Sus scrofa domesticus*	Switzerland, Langerbruck	-25.8	-26.7	-0.9	(Spangenberg, Jacomet and Schibler, 2006)
Porcine	*Sus scrofa ferus*	switzerland	-24.7	-24.7	0.0	(Spangenberg, Jacomet and Schibler, 2006)
Porcine	*Sus scrofa ferus*	Germany	-25.3	-25.0	0.3	(Debono Spiteri, 2012)
Porcine	*Sus scrofa ferus*	Germany	-28.3	-28.2	0.1	(Debono Spiteri, 2012)
Ruminant adipose	*Ovis aries*	Southern UK	-29.6	-31.4	-1.8	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-28.6	-30.3	-1.7	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-28.9	-30.6	-1.7	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-29.4	-31.3	-1.9	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-29.5	-31.3	-1.8	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-28.6	-31.5	-2.9	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-29.0	-30.3	-1.3	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-28.4	-30.2	-1.8	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-30.6	-32.4	-1.8	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-30.4	-32.5	-2.1	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-30.7	-32.7	-2.0	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-29.6	-32.0	-2.4	(Dudd, 1999)
Ruminant adipose	*Ovis aries*	Southern UK	-29.2	-30.6	-1.4	(Dudd, 1999)
Ruminant adipose	*Bos taurus*	Southern UK	-29.1	-32.0	-2.9	(Dudd, 1999)

Ruminant adipose	*Bos taurus*	Southern UK	-30.0	-32.5	-2.5	(Dudd, 1999)
Ruminant adipose	*Bos taurus*	Southern UK	-28.9	-31.7	-2.8	(Dudd, 1999)
Ruminant adipose	*Bos taurus*	Southern UK	-30.1	-31.8	-1.7	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-28.5	-29.9	-1.4	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-29.0	-30.7	-1.7	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-29.6	-29.9	-0.3	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-29.0	-32.3	-3.3	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-28.2	-33.4	-5.2	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-30.9	-34.0	-3.1	(Dudd, 1999)
Ruminant adipose	*Cervus elaphus*	Southern UK	-31.0	-33.8	-2.8	(Dudd, 1999)
Freshwater	*Anguilla anguilla*	Denmark	-28.5	-28.7	-0.2	(Craig *et al.*, 2011)
Freshwater	*Esox lucius*	Denmark	-35.1	-35.3	-0.2	(Craig *et al.*, 2011)
Freshwater	*Tinca tinca*	Denmark	-28.0	-29.1	-1.1	(Craig *et al.*, 2011)
Freshwater	*Tinca tinca*	Denmark	-24.5	-26.6	-2.1	(Craig *et al.*, 2011)
Freshwater	*Tinca tinca*	Denmark	-37.5	-36.8	0.7	(Craig *et al.*, 2011)
Freshwater	*Coregonus lavaretus*	Switzerland, Lake Constance	-33.2	-32.1	1.1	(Spangenberg, Jacomet and Schibler, 2006)
Freshwater	*Carassius carassius*	UK	-30.3	-28.3	2.0	(Lucquin *et al.*, 2016)
Freshwater	*Esox lucius*	UK	-28.3	-25.9	2.4	(Lucquin *et al.*, 2016)
Freshwater	*Perca fluviatilis*	UK	-31.1	-28.5	2.6	(Lucquin *et al.*, 2016)

Freshwater	*Perca fluviatilis*	UK	-35.9	-35.3	0.6	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-35.4	-36.0	-0.6	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-35.0	-35.1	-0.1	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-34.7	-35.7	-1.0	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-34.7	-35.7	-1.0	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-34.6	-35.8	-1.3	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-34.3	-33.9	0.4	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-33.8	-33.8	-0.1	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-33.6	-33.4	0.1	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-33.1	-34.1	-0.9	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-32.8	-34.3	-1.5	(Cramp *et al.*, 2014)
Freshwater	*Perca fluviatilis*	UK	-32.4	-33.0	-0.6	(Cramp *et al.*, 2014)
Freshwater	*Rutilus rutilus*	UK	-34.4	-34.5	-0.1	(Cramp *et al.*, 2014)
Freshwater	*Rutilus rutilus*	UK	-33.8	-35.0	-1.2	(Cramp *et al.*, 2014)
Freshwater	*Rutilus rutilus*	UK	-33.0	-34.1	-1.1	(Cramp *et al.*, 2014)
Freshwater	*Rutilus rutilus*	UK	-32.9	-33.9	-1.0	(Cramp *et al.*, 2014)
Freshwater	*Rutilus rutilus*	UK	-33.0	-33.5	-0.5	(Cramp *et al.*,

						2014)
Freshwater	*Rutilus rutilus*	UK	-32.4	-32.7	-0.3	(Cramp *et al.*, 2014)
Marine	*Gadus morhua*	UK	-25.5	-26.8	-1.3	(Dudd, 1999)
Marine	*Melanogrammus aeglefinus*	UK	-26.8	-24.5	2.3	(Dudd, 1999)
Marine	*Pleuronectes platessa*	UK	-24.7	-24.1	0.6	(Dudd, 1999)
Marine	*Anguilla anguilla*	Denmark	-20.7	-21.3	-0.6	(Craig *et al.*, 2011)
Marine	*Anguilla anguilla*	Denmark	-20.8	-21.9	-1.1	(Craig *et al.*, 2011)
Marine	*Anguilla anguilla*	Denmark	-18.6	-18.7	-0.1	(Craig *et al.*, 2011)
Marine	*Anguilla anguilla*	Denmark	-19.9	-21.6	-1.7	(Craig *et al.*, 2011)
Marine	*Gadus morhua*	Denmark	-23.0	-22.5	0.5	(Craig *et al.*, 2011)
Marine	*Gadus morhua*	Denmark	-23.0	-24.4	-1.4	(Craig *et al.*, 2011)
Marine	*Gadus morhua*	Denmark	-22.3	-24.8	-2.5	(Craig *et al.*, 2011)
Marine	*Phoca largha*	Denmark	-20.3	-20.3	0.0	(Craig *et al.*, 2011)
Marine	*Phoca largha*	Denmark	-13.1	-14.6	-1.5	(Craig *et al.*, 2011)
Marine	*Phoca vitulina*	Germany	-18.9	-20.5	-1.6	(Craig *et al.*, 2011)
Marine	*Platichthys flesus*	Denmark	-18.8	-20.1	-1.3	(Craig *et al.*, 2011)
Marine	*Pleuronectes platessa*	Denmark	-20.1	-21.8	-1.7	(Craig *et al.*, 2011)

Marine	*Pleuronectes platessa*	Denmark	-19.2	-20.4	-1.2	(Craig *et al.*, 2011)
Marine	*Zoarces viviparus*	Denmark	-19.7	-21.3	-1.6	(Craig *et al.*, 2011)
Marine	*Zoarces viviparus*	Denmark	-17.2	-18.2	-1.0	(Craig *et al.*, 2011)
Marine	*Clupea harengus*	Germany	-23.5	-21.1	2.4	(Craig *et al.*, 2011)
Marine	*Gadus morhua*	Germany	-21.6	-22.0	-0.4	(Craig *et al.*, 2011)
Marine	*Anguilla anguilla*	UK	-18.6	-15.9	2.7	(Lucquin *et al.*, 2016)
Marine	*Gadus morhua*	UK	-24.9	-24.5	0.4	(Lucquin *et al.*, 2016)
Marine	*Myoxocephalus scorpius*	Denmark	-21.5	-21.6	-0.1	(Lucquin *et al.*, 2016)
Marine	*Ostrea edulis*	UK	-24.4	-25.0	-0.6	(Lucquin *et al.*, 2016)
Marine	*Platichthys flesus*	Denmark	-19.2	-20.4	-1.2	(Lucquin *et al.*, 2016)
Marine	*Scomber scombrus*	UK	-25.5	-25.7	-0.2	(Lucquin *et al.*, 2016)
Marine	*Salmonidae sp.*	UK	-26.0	-25.9	0.0	(Lucquin *et al.*, 2016)
Marine	*Salmonidae sp.*	UK	-25.8	-25.7	0.1	(Lucquin *et al.*, 2016)
Marine	*Clupea harengus*	UK	-27.6	-27.3	0.3	(Cramp *et al.*, 2014)
Marine	*Clupea harengus*	UK	-27.0	-25.8	1.3	(Cramp *et al.*, 2014)
Marine	*Clupea harengus*	UK	-27.0	-25.3	1.7	(Cramp *et al.*, 2014)
Marine	*Clupea*	UK	-26.5	-26.1	0.4	(Cramp *et al.*,

	harengus					2014)
Marine	*#N/A*	UK	-25.7	-27.1	-1.4	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-24.9	-24.8	0.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-24.5	-24.0	0.4	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-24.0	-23.5	0.5	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.2	-23.8	1.4	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.3	-23.6	1.7	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.7	-23.8	1.9	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.6	-23.4	2.2	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.3	-23.2	2.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.1	-22.9	2.2	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-26.3	-23.2	3.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.1	-22.0	3.2	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-24.1	-23.6	0.5	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.5	-24.3	1.3	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.6	-25.8	-0.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-25.5	-24.6	0.9	(Cramp *et al.*,

						2014)
Marine	#N/A	UK	-25.4	-24.7	0.7	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.4	-24.2	0.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.3	-23.5	0.8	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.3	-23.3	1.0	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.2	-23.3	0.9	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.9	-23.2	0.7	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.0	-23.0	1.0	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.9	-22.9	1.1	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.9	-22.8	1.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.4	-22.8	1.5	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-24.1	-22.0	2.0	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.1	-22.3	0.8	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.2	-22.2	0.9	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.8	-23.0	-0.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.8	-23.0	-0.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.0	-23.1	-0.1	(Cramp *et al.*,

						2014)
Marine	#N/A	UK	-22.9	-21.4	1.5	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.7	-22.0	-0.3	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.5	-23.0	-0.5	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.5	-22.0	0.6	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-23.2	-20.7	2.5	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.8	-21.6	0.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.1	-21.4	0.7	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.0	-21.3	0.6	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-22.1	-21.0	1.1	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-20.3	-20.8	-0.5	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.0	-19.7	1.3	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.9	-19.3	2.7	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.9	-19.3	2.6	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-21.3	-19.1	2.2	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-20.8	-19.2	1.7	(Cramp *et al.*, 2014)
Marine	#N/A	UK	-20.8	-19.3	1.5	(Cramp *et al.*,

						2014)
Marine	*#N/A*	UK	-20.8	-18.3	2.5	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-19.1	-17.9	1.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-18.9	-17.8	1.0	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-18.5	-17.4	1.0	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-23.9	-23.4	0.5	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-23.4	-22.3	1.1	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-22.8	-21.9	0.9	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-21.4	-22.3	-0.9	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-21.3	-20.7	0.6	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-21.3	-20.6	0.7	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-20.2	-18.5	1.8	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-17.6	-16.1	1.5	(Cramp *et al.*, 2014)
Marine	*#N/A*	UK	-16.6	-16.1	0.5	(Cramp *et al.*, 2014)
Marine	*Dicentrarchus labrax*	UK	-23.1	-24.2	-1.1	(Bell *et al.*, 2007)
Marine	*Esox lucius*	Denmark	-20.1	-21.8	-1.7	(Courel *et al.*, 2020)

9.0 Appendix Nine: Stable carbon isotopes of n-hexadecanoic (C16:0) and n-octadecanoic (C18:0) acids for reference fats from archaeological pottery/lamps

Sample	Phase	Type	Location	δ ^{13}C16:0 (‰)	δ ^{13}C18:0 (‰)	Δ ^{13}C (C18:0-C16:0)	Reference
NOR-01	EM	FCR	Ormen Lange	-30.505	-30.364	0.14	This study
NOR-02	EM	FCR	Ormen Lange	-30.474	-29.968	0.51	This study
NOR-03	EM	FCR	Ormen Lange	-30.903	-30.189	0.71	This study
NOR-04	EM	FCR	Ormen Lange	-28.385	-28.888	-0.50	This study
NOR-05	EM	FCR	Ormen Lange	-30.525	-30.277	0.25	This study
R01A	EBK/TRB	FCR	Neustadt LA 156	-30.686	-30.565	0.12	This study
R01B	EBK/TRB	FCR	Neustadt LA 156	-27.003	-26.568	0.43	This study
R02A	EBK/TRB	FCR	Neustadt LA 156	-27.917	-28.725	-0.81	This study
R02B	EBK/TRB	FCR	Neustadt LA 156	-30.067	-30.503	-0.44	This study
R03A	EBK/TRB	FCR	Neustadt LA 156	-27.078	-28.555	-1.48	This study
R03B	EBK/TRB	FCR	Neustadt LA 156	-26.592	-27.844	-1.25	This study
R06A	EBK/TRB	FCR	Neustadt LA 156	-30.106	-30.591	-0.48	This study
R06B	EBK/TRB	FCR	Neustadt LA 156	-29.827	-30.576	-0.75	This study

RO6C	EBK/TRB	FCR	Neustadt LA 156	-31.322	-31.473	-0.15	This study
R07A	EBK/TRB	FCR	Neustadt LA 156	-28.514	-29.827	-1.31	This study
R08A	EBK/TRB	FCR	Neustadt LA 156	-29.659	-29.855	-0.20	This study
R09A	EBK/TRB	FCR	Neustadt LA 156	-28.806	-29.198	-0.39	This study
R11A	EBK/TRB	FCR	Neustadt LA 156	-29.495	-29.324	0.17	This study
R12A	EBK/TRB	FCR	Neustadt LA 156	-29.12	-29.496	-0.38	This study
R13A	EBK/TRB	FCR	Neustadt LA 156	-29.797	-29.878	-0.08	This study
R14A	EBK/TRB	FCR	Neustadt LA 156	-30.673	-31.001	-0.33	This study
R14B	EBK/TRB	FCR	Neustadt LA 156	-29.16	-29.919	-0.76	This study
R14C	EBK/TRB	FCR	Neustadt LA 156	-30.08	-30.832	-0.75	This study
B092	EBK	Pottery	Neustadt LA 156	-19.9	-20.1	-0.20	(Craig *et al.*, 2011)
B093	EBK	Pottery	Neustadt LA 156	-19.3	-19.8	-0.50	(Craig *et al.*, 2011)
B094	EBK	Pottery	Neustadt LA 156	-28.4	-27.3	1.10	(Craig *et al.*, 2011)
B095	EBK	Pottery	Neustadt LA 156	-26.5	-27.2	-0.70	(Craig *et al.*, 2011)
B096	EBK	Pottery	Neustadt LA 156	-28	-28.1	-0.10	(Craig *et al.*, 2011)
B097	EBK	Pottery	Neustadt LA 156	-29.2	-30.4	-1.20	(Craig *et al.*, 2011)
B098	EBK	Pottery	Neustadt LA	-25.8	-26.5	-0.70	(Craig *et al.*,

			156				2011)
B099	EBK	Pottery	Neustadt LA 156	-29.2	-31.7	-2.50	(Craig *et al.*, 2011)
B100	EBK	Pottery	Neustadt LA 156	-35.2	-33.3	1.90	(Craig *et al.*, 2011)
B101	EBK	Pottery	Neustadt LA 156	-28.3	-30.7	-2.40	(Craig *et al.*, 2011)
B102	EBK	Pottery	Neustadt LA 156	-24.4	-27.4	-3.00	(Craig *et al.*, 2011)
B103	EBK	Pottery	Neustadt LA 156	-28.2	-27.9	0.30	(Craig *et al.*, 2011)
B105	EBK	Pottery	Neustadt LA 156	-27.5	-27.8	-0.30	(Craig *et al.*, 2011)
B106	EBK	Pottery	Neustadt LA 156	-22.9	-22.7	0.20	(Craig *et al.*, 2011)
B107	EBK	Pottery	Neustadt LA 156	-29.8	-32.3	-2.50	(Craig *et al.*, 2011)
B108	EBK	Pottery	Neustadt LA 156	-25.1	-25.2	-0.10	(Craig *et al.*, 2011)
B109	EBK	Pottery	Neustadt LA 156	-30.5	-34.4	-3.90	(Craig *et al.*, 2011)
B110	EBK	Pottery	Neustadt LA 156	-20.3	-21.6	-1.30	(Craig *et al.*, 2011)
B111	EBK	Pottery	Neustadt LA 156	-23.3	-24.5	-1.20	(Craig *et al.*, 2011)
B112	EBK	Pottery	Neustadt LA 156	-22.5	-22.2	0.30	(Craig *et al.*, 2011)
B113	EBK	Pottery	Neustadt LA 156	-21.4	-22.2	-0.80	(Craig *et al.*, 2011)
B145	EBK	Pottery	Neustadt LA 156	-28.8	-33.6	-4.80	(Craig *et al.*, 2011)
B146	EBK	Pottery	Neustadt LA	-31.7	-35.9	-4.20	(Craig *et al.*,

			156				2011)
B147	EBK	Pottery	Neustadt LA 156	-31.9	-36	-4.10	(Craig *et al.*, 2011)
B148	EBK	Pottery	Neustadt LA 156	-29.6	-33.2	-3.60	(Craig *et al.*, 2011)
B149	EBK	Pottery	Neustadt LA 156	-28.1	-33.1	-5.00	(Craig *et al.*, 2011)
L25i	Rzcewo	Bowl/lamp	Nida	-32.1	-33.1	-1.00	(Heron *et al.*, 2015)
L12i	Rzcewo	Bowl/lamp	Nida	-32.2	-32.3	-0.10	(Heron *et al.*, 2015)
GR-1-S	EBK	Bowl	Grube-Rosenhof LA 58	-22.3	-22.4	-0.10	(Robson *et al.*, 2022)
GR-2-S	EBK	Bowl	Grube-Rosenhof LA 58	-23.4	-23.4	0.00	(Robson *et al.*, 2022)
GR-5-S	EBK	Bowl	Grube-Rosenhof LA 58	-20.8	-20	0.80	(Robson *et al.*, 2022)
GR-14-S	EBK	Bowl	Grube-Rosenhof LA 58	-19.4	-19.4	0.00	(Robson *et al.*, 2022)
RO-1-S	EBK	Bowl	Ronæs Skov	-16.9	-16.4	0.50	(Robson *et al.*, 2022)
RO-8-S	EBK	Bowl	Ronæs Skov	-19.3	-18.1	1.20	(Robson *et al.*, 2022)
RO-9-S	EBK	Bowl	Ronæs Skov	-18.4	-18.1	0.30	(Robson *et al.*, 2022)
RO-12-S	EBK	Bowl	Ronæs Skov	-22.3	-23	-0.70	(Robson *et al.*, 2022)
N1457i	TRB	Pottery	Neustadt LA 156	-27.3	-33.2	-5.90	(Craig *et al.*, 2011)
N1494f	TRB	Pottery	Neustadt LA	-21.3	-22.4	-1.10	(Craig *et al.*,

			156				2011)
N1494i	TRB	Pottery	Neustadt LA 156	-19.2	-19.5	-0.30	(Craig *et al.*, 2011)
N1908i	TRB	Pottery	Neustadt LA 156	-28.3	-33.6	-5.30	(Craig *et al.*, 2011)
N2162i	TRB	Pottery	Neustadt LA 156	-22.5	-26.6	-4.10	(Craig *et al.*, 2011)
N217i	TRB	Pottery	Neustadt LA 156	-29.3	-32.5	-3.20	(Craig *et al.*, 2011)
N22i	TRB	Pottery	Neustadt LA 156	-30.6	-34.5	-3.90	(Craig *et al.*, 2011)
N2449i	TRB	Pottery	Neustadt LA 156	-28.2	-32.8	-4.60	(Craig *et al.*, 2011)
N2451i	TRB	Pottery	Neustadt LA 156	-27.7	-32.8	-5.10	(Craig *et al.*, 2011)
N2641i	TRB	Pottery	Neustadt LA 156	-22.5	-21.5	1.00	(Craig *et al.*, 2011)
N2784i	TRB	Pottery	Neustadt LA 156	-19.9	-21.4	-1.50	(Craig *et al.*, 2011)
N2804i	TRB	Pottery	Neustadt LA 156	-25	-27.9	-2.90	(Craig *et al.*, 2011)
N3037i	TRB	Pottery	Neustadt LA 156	-28.5	-34.4	-5.90	(Craig *et al.*, 2011)
N3233i	TRB	Pottery	Neustadt LA 156	-28.5	-34.4	-5.90	(Craig *et al.*, 2011)
N3309f	TRB	Pottery	Neustadt LA 156	-20	-20.7	-0.70	(Craig *et al.*, 2011)
N3309i	TRB	Pottery	Neustadt LA 156	-20	-20.4	-0.40	(Craig *et al.*, 2011)
N3406f	TRB	Pottery	Neustadt LA 156	-25.6	-30.1	-4.50	(Craig *et al.*, 2011)
N3406i	TRB	Pottery	Neustadt LA	-26.5	-30.8	-4.30	(Craig *et al.*,

			156				2011)
N385i	TRB	Pottery	Neustadt LA 156	-26.9	-32.4	-5.50	(Craig *et al.*, 2011)
N387f	TRB	Pottery	Neustadt LA 156	-25.6	-26.5	-0.90	(Craig *et al.*, 2011)
N421i	TRB	Pottery	Neustadt LA 156	-26.8	-31.9	-5.10	(Craig *et al.*, 2011)
N441s	TRB	Pottery	Neustadt LA 156	-29.3	-33.6	-4.30	(Craig *et al.*, 2011)
N442i	TRB	Pottery	Neustadt LA 156	-30.2	-33.3	-3.10	(Craig *et al.*, 2011)

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