Historic England

Organic Residue Analysis and Archaeology

Guidance for Good Practice

Summary

This document provides guidance for good practice in the recovery, analysis and publication of **organic** residues from archaeological sites. It has been written for a range of archaeological professionals, including local authority archaeology officers, archaeological units and consultants, project managers, museum curators, conservators and pottery specialists, with the aim of ensuring that approaches are suitable, cost-effective and informative. The objectives of the guidelines are to:

- inform practicing archaeologists of the principles and potential applications of organic residue analysis (ORA)
- provide clear and coherent guidance on organic residues recovery, sampling and analysis
- demonstrate the research potential of the approach

This guidance was commissioned by Historic England and written by University of Bristol-based organic residue specialists, with contributions from the Universities of Bradford and York, in consultation with pottery specialists, museum curators, field archaeologists, local authority curators and Historic England.

Supporting Information for this document can be found on the Historic England website. It contains further detail on terms and concepts used in ORA and analytical techniques used to identify organic residues, together with guidance on where future research themes involving ORA might usefully be targeted. A thematically organised bibliography and details of where to access literature relating to ORA is also included. It also incorporates a short section on reporting, publishing and digital archiving, and guidance for museum curators and conservators in archiving ceramics with potential to be used for ORA.

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HistoricEngland.org.uk/advice/technical-advice/archaeological-science/

Front cover
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Contents

How to Use the Guidance

The document is divided into seven sections, with links to relevant online *Supporting Information* given at appropriate points in the text. In Section 1, the principles underlying **archaeological biomarkers**, and, specifically, **lipids**, are covered in brief. Full details can be found in Section 1 of the *Supporting Information*. Section 2 lists the analytical techniques used to identify the origins of fats, waxes and oils extracted (with full details given in the *Supporting Information*). The types of commodities that can currently be identified through ORA are described in Section 3. Section 4 describes the types of archaeological questions that can be addressed using ORA, such as production, use and technological change in vessels, resource acquisition and exploitation, trade and exchange, and broad-scale dietary reconstruction and subsistence practices. Section 5 sets forth a step-by-step guide to the processes involved in planning a project involving ORA. Section 6 describes (in brief) areas where the application of organic residue analysis would help address specific archaeological questions regarded as priorities by pottery research groups and/or described within regional frameworks. Full details can be found in Section 2 of the *Supporting Information*.

Organic residue analysis is an analytical approach that can be used to address a wide range of archaeological questions on multiple levels, as summarised in the Overview of Organic Residue Analysis on the opposite page. ORA is a technical topic covering a number of complex scientific processes that may be unfamiliar to the non-specialist. Terms in **bold** text can be found in the Glossary, which provides clarification of the scientific terms used. Details on where to obtain advice on ORA are listed on page 47.

The majority of organic residue analyses are on absorbed residues from pottery, and these comprise the main focus of this guidance. However, visible residues adhering to pottery and amorphous residues (such as **resins**, tars and pitches) are also discussed (Figure 1).

Overview of Organic Residue Analysis (ORA)

ORA can address archaeological questions regarding diet and subsistence practices, as well as the ancient trade of goods and raw materials, technology (including vessel production, use and provenance), resource acquisition/exploitation, and the domestication of plants and animals, among other issues. It should be emphasised that the greatest potential for ORA on ceramics is from its application at the site level. ORA needs to be built into the project budget, for by far the greatest potential source of material is from planning-led archaeological work. Such data can then contribute to interpretations regarding the archaeological questions discussed above, for example, dietary reconstruction, technology and resource acquisition, all on local, national and global scales.

Some examples of the types of questions that ORA of archaeological pottery has successfully addressed are:

- What commodities were processed or stored in ancient vessels? ORA enables the characterisation of resources including:
    - a) terrestrial animal fats. The use of isotopic approaches enables us to distinguish between ruminant and non-ruminant fats, and carcass fats from dairy fats
    - b) aquatic fats (for example, from fish, shellfish and marine mammals)
    - c) plant oils and waxes
    - d) beeswax
    - e) resins, tars and bitumens
- Were there specific roles for pottery groups relating form, fabric or context with function (variations in vessel use)?
- What were the methods of cooking food – for example, boiling or roasting?
- What organic materials were used to decorate or repair vessels?
- Were any post-firing treatments (such as milk, resins or beeswax) applied to vessels?
- Can broader patterns of diet and subsistence be identified across temporal and spatial frameworks, for example:
    - a) can ORA help determine if the exploitation of terrestrial resources completely replaced intensive marine resource use at coastal sites once farming was introduced to Neolithic Britain?
    - b) how important was dairying in Britain over the course of later prehistory?

Figure 1

A   Parts of vessels from the late Iron Age excavated from Middleton Quarry, Norfolk.
Archaeological Project Services.

B and C – Encrusted residue adhering to potsherds.
© Wessex Archaeology and M Roffet-Salque.

D   Amorphous residues (*Pistacia* spp. and Pinaceae resins) recovered from the base of a lead-lined stone sarcophagus, ‘Spitalfields Lady’, London.
R Brettell and Museum of London
(Accession number: SRP98 SK15903).

Introduction

What are organic residues?

The term ‘organic residues’ is used widely in archaeology to describe a range of amorphous organic remains from diverse natural sources, associated with a variety of artefacts found at archaeological sites. Their amorphous nature means that they lack the clearly discernible morphological features that characterise other biological materials that survive in the archaeological record, such as bone, wood, leather and textiles, and archaeological plant material such as seeds and pollen.

Organic residues from pottery

The majority of organic residue analyses are carried out on **absorbed** organic **residues** from ceramic vessels. These residues generally come from the original contents either stored or processed in the vessels, either from use for a single product, or from an accumulation of individual uses in a vessel over its life history. Organic residues survive widely in association with ceramics and can endure over considerable timescales. Once fired, pottery is extremely durable, which accounts for its ubiquitous recovery at archaeological sites dating from the Neolithic period onwards in Britain. Consequently, absorbed organic residues identified in pottery are the main focus of this document, but other amorphous organic residues (such as **resins**, bitumens and tars) that may be recovered in association with other matrices or containers are also discussed.

Survival of these residues in pottery occurs in three ways:

- The actual contents are preserved as solidified or liquid substances *in situ* as vessel or other container fills. This is rare, but examples do exist, such as the contents of a Roman tin container used to store cosmetic cream (Evershed *et al* 2004).

- Somewhat more commonly, as carbonized, encrusted residues adhering to the interior or exterior surface of a vessel. On the outer walls of ceramics, blackened marks may derive from soot deposited by heating the vessel over fire, or from materials used as decoration or adhesives. Burnt matter on the inner surface is often believed to result from one or more cooking episodes that have gone awry, resulting in the charring of food or other biological material.

- The most common form of organic residues are those preserved within the vessel wall, known as absorbed residues. These have been found to survive in >80 per cent of domestic cooking pottery assemblages worldwide (Evershed 2008), although the percentage recovery from individual assemblages can vary tremendously and is often dependent on conditions of preservation.

The largely amorphous, or invisible, nature of these residues means that their composition has to be determined through the use of analytical chemical methodologies. Modern techniques have enabled the effective recovery, detection and characterisation of **biomolecules** and their decay products in archaeological materials, achieved through the application of what is known as the **archaeological biomarker** concept.

1 Underpinning Principles of ORA

1.1 Archaeological biomarker concept

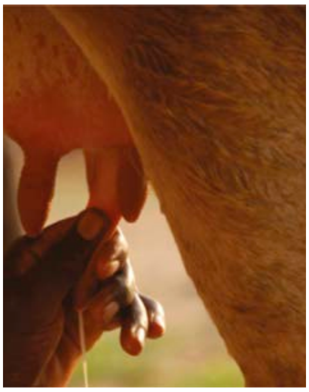
Biomarkers (biological markers) are indicator **compounds** that can be used as tracers for geological and environmental processes. These derive from the carbon skeletons of organisms that existed in past environments and often persist over long timescales. The concept of **archaeological biomarkers** encompasses approaches developed for the biomolecular analysis of organic archaeological remains, to provide information relating to past human activity. The term archaeological biomarkers covers a variety of organic compounds, including proteins, carbohydrates, **lipids**, nucleic acids and amino acids. The archaeological biomarker concept relies on matching the chemical structures and distributions identified in archaeological remains to those in modern organisms (Figure 2). These are known as 'chemical fingerprints'.

To interpret the results, it is crucial to understand the molecular structural changes that occur during the processes of degradation and decay. This document concentrates on **lipid** analysis, as lipids have been found to be the most durable and widely occurring biomarkers in the archaeological record.

Figure 2 (pages 2-3): Examples of chemical structures of lipids found in absorbed and amorphous organic residues.

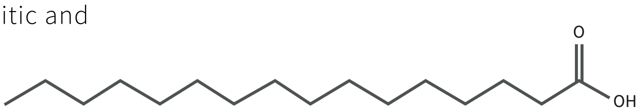
1 Palmitic and stearic acid are the most commonly identified compounds in lipid residues. These usually indicate a degraded animal fat origin although they are also found in aquatic and plant fats.
2 Compounds typical of an aquatic origin.
3 Terpenoids characteristic of frankincense and coniferous resin, respectively.
4 A hydroxy wax ester found in beeswax.
5 Typical biomarkers originating from plant leaf wax.

Images © Julie Dunne.

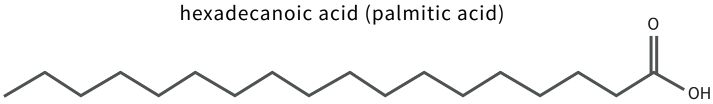


1 Animal fats

Identification of fat source by determining **stable carbon isotope** composition of palmitic and stearic acids

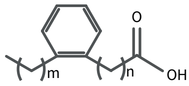


hexadecanoic acid (palmitic acid)

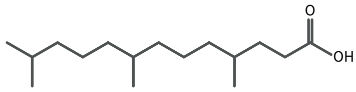


octadecanoic acid (stearic acid)

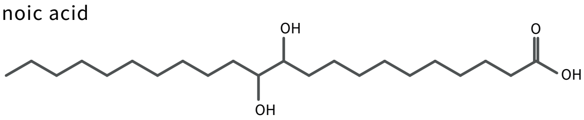
2 Aquatic products



ω-(*o*-alkylphenyl)alkanoic acid

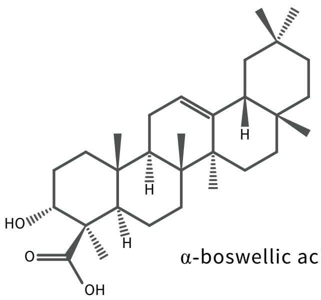


isoprenoid fatty acid
(4,8,12-TMTD)

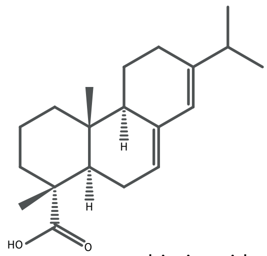


vicinal dihydroxy acid
11,12-dihydroxydocosanoic acid

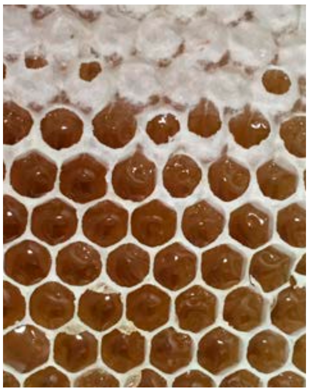
3 Resins, tars and pitches



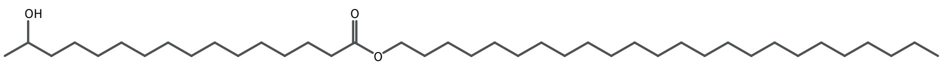
α-boswellic acid



abietic acid

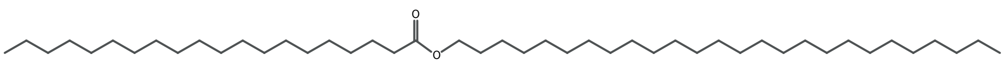


4 Beeswax

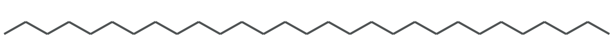


hydroxy wax ester (tetracosanyl 15-hydroxypalmitate)

5 Plant leaf waxes



wax ester (hexacosynanly eicosanoate)



n-alkane (*n*-nonacosane)

1.2 Lipids

Lipids are the fats, waxes and resins of the natural world. They are the most frequently recovered biomolecules from archaeological sites, for they can endure, often over long timescales, at their site of deposition. This is because they are **hydrophobic**, which means they will not readily dissolve in water. Lipids are mainly composed of carbon, hydrogen and oxygen, and form a wide variety of chemical structures, including:

- **fatty acids**
- **wax esters**
- **sterols** (including **cholesterol**)
- **phospholipids**
- **mono**-, **di**- and **triglycerides** (**triacylglycerols**)

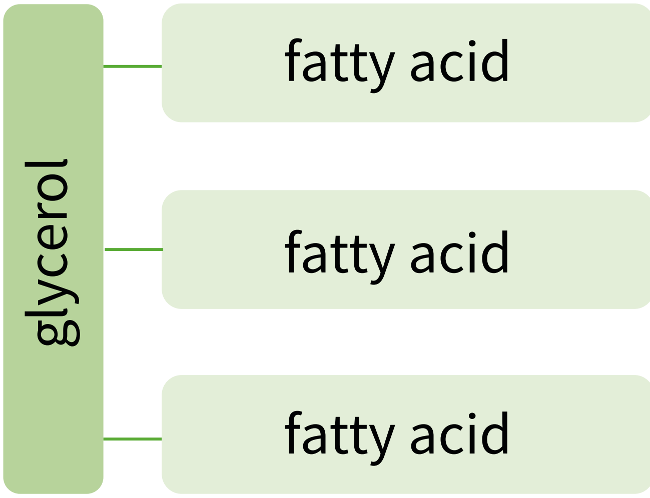


Figure 3
Simplified structure of a **triglyceride** (**triacylglycerol**), comprising a glycerol molecule joined by three **fatty acid** chains.

Together with carbohydrates and proteins, **lipids** are the main constituents of plant and animal cells, making up the building blocks of their structure and function.

The **triacylglycerols** (TAGs) are the main form of lipid found in living organisms. They are made from a glycerol molecule joined by three **fatty acid** chains (Figure 3).

Triacylglycerols make up more than 95 per cent of lipids in our diet and are found in most foods – in varying abundances – including meats, fish, dairy products, seeds, nuts and plant material (Figure 4). The fatty acids making up the triacylglycerols form the backbone of research in ORA.

Other lipids found in organic residues from vessels that might originate from food products include natural waxes produced by insects (that is, beeswax) and waterproof coatings on the outer surface of plants (known as **epicuticular waxes**).

Lipids from natural products that are *not* used for dietary purposes – originating from resins, tars and bitumen – have also been identified in both visible and absorbed residues. Moreover, beeswax has been found to have been used for non-dietary purposes. These commodities may have been stored or processed in vessels, or used in their manufacture as sealants, or for decoration or repair (adhesives). In addition, they may have been used in the manufacture of illuminants, incense, cosmetics, balms and medicines.

For detailed information on lipids see Section 1 in the *Supporting Information*.

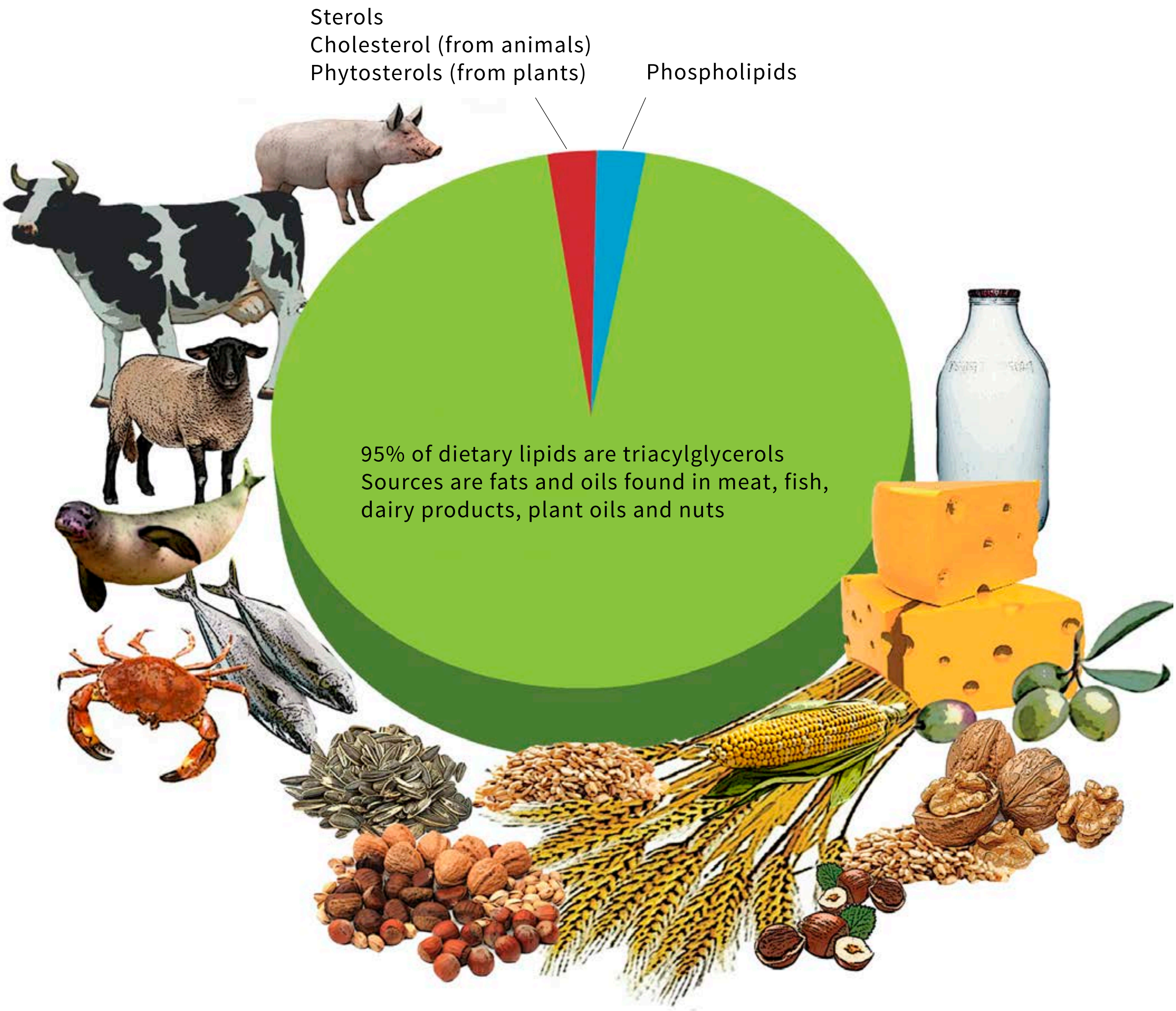


Figure 4

Sources of dietary lipids deriving predominantly from **triacylglycerols**.

2 Analytical Techniques

The main analytical techniques used to identify and characterise different compound classes from visible and absorbed lipid residues extracted from archaeological ceramics are:

- **gas chromatography - Flame Ionisation Detection** (GC-FID), which screens and quantifies preserved organic residues, separating the mixtures into individual components

- **gas chromatography-mass spectrometry** (GC-MS), which enables identification of the separated components and hence gives a 'biomolecular fingerprint'

- **gas chromatography-combustion-isotope ratio mass spectrometry** (GC-C-IRMS), which enables the determination of the **stable isotope** values of individual **fatty acids**

For further information on these analytical techniques and their application see Section 1.2 in *Supporting Information*.

Figure 5
A gas-chromatography autosampler.
© Lucy Cramp.

3 Commodities Identified from Organic Residues

Table 1 shows the commodities that have been identified in visible or absorbed organic residues from archaeological pottery.

Notice that there are two main factors that determine the identification of specific commodities from organic residues. Specific biomarkers that can unambiguously identify a natural product are crucial (for example, the distribution of *n*-nonacosane', *n*-nonacosan-15-ol and *n*-nonacosan-15-one found in *Brassica oleracea* (cabbage type cultivars) and identified at medieval West Cotton – Case study 2), but it is their recognisable survival over archaeological timescales that commonly poses the challenge.

Table 1 (pages 8-9)
Types of commodities that can be identified using ORA, showing their source, related biomarkers, level of specificity and some relevant examples.

	Sources	Biomarkers
terrestrial animal fats	**carcass** fats	saturated and monounsaturated fatty acids, **δ^{13}C** values of fatty acids
	dairy fats	saturated and monounsaturated fatty acids, including shorter-chain fatty acids, **δ^{13}C** values of fatty acids
aquatic fats	aquatic fish, shellfish and mammals	**dihydroxy acids** (DHYAs), isoprenoid acids (IPAs) and **long-chain ω-(*o*-alkylphenyl) alkanoic acids**, **δ^{13}C** values of fatty acids
plant	oils	saturated ($C_{16:0}$ and $C_{18:0}$) and unsaturated **fatty acids** (for example $C_{18:1}$ and $C_{18:2}$, hydroxy **fatty acids**, di**carboxylic acids)**
	waxes	**wax esters**, long-chain *n*-alkanes, *n*-alkanols and even-numbered long-chain fatty acids
	resins, tars and pitches	terpenoids including di-, tri- and sesquiterpenoids
bitumen	fossil organic matter	hydrocarbons, steranes, terpanes
insect	waxes (that is beeswax)	odd carbon numbered *n*-alkanes (C_{21}-C_{33}), even-numbered free **fatty acids** (C_{22}-C_{30}) and long-chain palmitate esters in the carbon range C_{40}-C_{52})

Specificity	Examples
Can distinguish differences between **ruminant** and **non-ruminant** animals (using **$\delta^{13}C$** values of **fatty acids**), but not usually between species in either category (for example ruminants such as cattle, sheep or goat are difficult to distinguish).	Ruminant animal fat (tallow), probably of sheep or goat, found in medieval lamps while non-ruminant animal fat, such as pig, was present in 'dripping dishes', suggesting that the latter were probably used as receptacles for fat collection during spit-roasting (Mottram *et al* 1999).
Dairy fats can be separated from carcass fats through **$\delta^{13}C$** values of **fatty acids** (**$\Delta^{13}C$ proxy**).	Absorbed lipid residues from 256 pottery vessels obtained from four southern British Bronze Age sites demonstrated (using **$\Delta^{13}C$ proxy)** that dairying was an important component of animal husbandry practices in southern Britain during the Bronze Age (Copley *et al* 2005b).
Species from marine/ocean-like ecosystems can usually be distinguished from freshwater based on **$\delta^{13}C$** values. Marine ecosystems are complex and higher-level specificity would be challenging.	ORA revealed an immediate shift away from the exploitation of aquatic resources in the earliest Neolithic, later followed by a gradual return to the inclusion of marine products over subsequent millennia, maximising in the Late Norse period in northern Britain (Cramp *et al* 2014).
Very specific biomarkers may exist for some oils (for example radish oil), although the majority of plant oil biomarkers only indicate the processing of oils generally.	Radish and castor oil used as illuminants in archaeological lamps at Qasr Ibrim, Egypt (Copley *et al* 2005d).
Can be very specific depending on source (for example biomarkers found for *Brassica* (see Case study 2)), although the majority of plant waxes are not specific and can only indicate processing of leafy plants.	*Brassica oleracea* (probably cabbage) identified in Saxon/Medieval vessels at West Cotton, Northamptonshire (Evershed *et al* 1991). The mixing of leafy plants and animal fats was identified in Roman mortaria (Cramp *et al* 2011).
Resins can often be provenanced to the botanical family of origin, and sometimes even to genus, enabling the pinpointing of their geographic origin.	ORA has provided direct evidence for the use of resinous exudates in mortuary rites in Roman Britain, including coniferous resin, mastic/terebinth resin from the Mediterranean and frankincense from southern Arabia or eastern Africa (Case study 5; Brettell *et al* 2014, Brettell *et al* 2015a,b). The distillation of resins, which produces tars or pitches, can also be chemically identified. For example, using GC-MS, pitch from the wreck of King Henry VIII's flagship (AD 1509–45), the *Mary Rose*, was identified as Stockholm tar, a good-quality pine tar obtained from the destructive distillation of *Pinus sylvestris* (Evershed *et al* 1985).
Biomarker ratios and **$\delta^{13}C$** values can provenance bitumen to area of geographic origin.	ORA identified bitumen, mainly from the Dead Sea, often mixed with fat, conifer resin and beeswax, used in mummification practices by Egyptian embalmers between 1000BC and 400AD (Connan 1999).
Composition of wax esters confirms beeswax.	Beeswax was mixed with tallow to make candles at 12th century Fountains Abbey, Yorkshire (Case study 1, Frith *et al* 2004). Combed ware pottery vessels from Ancient Greece were shown to have been used as beehives (Evershed *et al* 2003). Beeswax was used as a lamp illuminant in Minoan Crete (Evershed *et al* 1997) and has been identified as a widespread component of balms used for mummification in Pharaonic and Graeco-Roman Egypt (Buckley and Evershed 2001).

4 Archaeological Applications

4.1 Archaeological questions that ORA can address

ORA is generally known for its role in identifying what food products were processed or stored in ancient vessels. However, it can also address wider questions regarding the ancient trade of goods and raw materials, ancient technologies, resource exploitation and acquisition, ritual practices, the domestication of plants and animals, and dietary reconstruction and subsistence practices. ORA has the potential to inform archaeological interpretations on scales ranging from a site context to local, national and global scale questions, together with the investigation of chronological change, again across small- to broad-scale contexts.

It should be emphasised that the greatest potential for ORA is its application at site level, where analyses would be commissioned through planning-led archaeological work. A considerable amount of information can be gained from site-level analysis of an assemblage of 20–30 potsherds from a well-constrained chronological period (see Table 2). At multi-phase sites, ORA can be applied to identify changes in diet and economy over time. Such data can then contribute to broader knowledge (and also to possible research questions) on diet and subsistence practices on a regional and national level. Case study 3, Ascott-under-Wychwood, demonstrates the potential of this approach.

Some examples of particular research questions that ORA can address are set out in Table 2. It gives details of archaeological themes and questions that ORA might usefully investigate: diet, economy and subsistence; production, use, technological change and vessel specialisation; food preparation techniques and resource acquisition and exploitation; and trade and exchange. Also shown are themes that relate to archaeological questions that can be asked at site level and their subsequent fit within broader-scale questions. Some examples of target periods and regions where these themes might be directed are noted as well as specific case studies and examples of existing research.

4.2 Overall interpretation of organic residue information

It is always crucial to apply the analysis and subsequent interpretation of organic residues within the context of the archaeology and palaeoecology of the settlement, region and/or period from which they derive. Organic residue information should be integrated with other lines of archaeological evidence – such as faunal and archaeobotanical remains – in order to provide the most meaningful answers to research questions. For example, in studies of animal product processing in prehistoric Britain, only a restricted range of ruminants need to be considered as likely sources of lipid extracted from vessels; these include cattle, sheep/goat and deer, with pig being the most common non-ruminant. In other regions or periods the range of species might be different, guiding interpretations accordingly. Significantly, it should also be noted that ORA can be a valuable **proxy** where other lines of evidence are not present. For instance, in Ireland, as in many parts of south-west and northern England, acidic soils lead to poor preservation of faunal remains, precluding archaeozoological interpretations of animal husbandry practices in prehistory. However, as Case study 4 demonstrates, ORA has firmly established that dairying was a significant component of the earliest farming practices in Neolithic Ireland (Smyth and Evershed 2015a).

Table 2 (pages 12-13)
Archaeological themes and questions that ORA might usefully help to address.

Site level question	Site-level analysis	Fit within large-scale questions	Examples of target periods/ regions	Example (site specific and broader context, if applicable)
Diet, economy and subsistence				
diet, economy and subsistence	assemblages of cooking pots (>25 vessels)	large-scale subsistence shifts through time (for example introduction of farming, intensification of agriculture, episodes of invasion or immigration and cultural change)	periods with limited evidence for subsistence (for example prehistory, rather than historic period)	Case study 3 **Ascott-under-Wychwood** site level – high abundance of dairy fats processed in vessels (Copley and Evershed 2007); broader scale – dairying shown to be widespread activity at early Neolithic sites (Copley *et al* 2003)
			regions with poor bone preservation, specifically acidic regions of south-west and northern Britain, because of acidic soils	Case study 4 **Dairying in Neolithic Ireland** acidic soils meant poor preservation of faunal remains, precluding interpretations of animal husbandry practices; ORA on pots from 15 sites established dairying was significant component of earliest farming practices in Neolithic Ireland (Smyth and Evershed 2015)
Production, use, technological change and vessel specialisation				
manufacture, decoration and repair of vessels	encrusted and absorbed residues (as required)	technological change, resource exploitation, trade relationships	were British Bronze Age funerary vessels specifically manufactured solely for that purpose or are they re-used cooking vessels?	(a) birch bark tar used to repair Ecton Ware jar at West Cotton, Northampton (Charters *et al* 1993) (b) bitumen used as applied decoration on Late Neolithic ceramics fron Tell Sabi Abyad, Syria (Connan *et al* 2004) (c) beeswax as surface sealant (post-firing treatment) found on Bronze Age Mediterranean Red Lustrous Wheel Ware (Knappett *et al* 2005)
relationships between form and function (food and non-food products)	comparative analysis of one or more defined vessel types (for example jars, dishes, bowls)	tracing diversification and specialisation of vessel use (for example cheese strainers) through intra- and inter-site, and regional comparisons; assessment of broader trends in vessel use	vessel types, capacities, decoration and vessel use from Late Bronze Age, Early Iron Age and Middle to Late Iron Age domestic structures could be compared to examine changing patterns in food consumption in later prehistory	ORA carried out on British Iron Age 'saucepan pots', jars and bowls, to determine whether any of these particular vessel types were consistently used to process specific commodities; study revealed that milk products were processed in 'saucepan pots' at sites where these pots predominated, yet at sites where jars dominated, the latter type were preferentially associated with dairy products (Copley *et al* 2005a)
				ORA carried out on Roman mortaria from mixture of urban, rural and military settlements across Britain to determine what they were used for; analyses showed high abundances of plant epicuticular waxes in mortaria and (mainly) degraded ruminant carcass fats, suggesting that both animals and plant products were routinely processed in same mortaria, although not known if they were mixed together or processed on separate occasions (Cramp *et al* 2011).
vessel function (non-food)	vessel and container fills (liquid or solidified)	technological change, resource exploitation	as required	ORA found that whitish cream preserved in small Roman tin canister from London was made from animal fat, starch and tin oxide; likely of cosmetic or medicinal origin (Evershed *et al* 2004)
	assemblage of likely containers or matrices (for example lamps, candleholders)	technological change, resource exploitation	as required	Case study 1 **12th century Fountains Abbey, Yorkshire,** ORA of visible residues adhering to metal candlesticks found that 'recycled' beeswax from church candles was mixed with tallow to make domestic candles (Frith *et al* 2004)

spatial patterning and activity-specific (for example pottery associated with different structures or activities)	analysis or comparison of assemblages associated with particular context (for example industrial area, domestic settlement, cemetery, ceremonial centre)	ritual use of pots and feasting	pottery from different kinds of early Neolithic contexts, especially house and settlement contexts, but also causewayed enclosures	ORA showed Late Neolithic Grooved Ware preferentially associated with pig consumption; also demonstrated clear connection between pottery from ceremonial sites and pig exploitation, suggesting ritualistic aspect to pork consumption (Mukherjee *et al* 2008)
	site variation	inter- and intra-site variation, and regional comparisons to assess broader trends in vessel use	diet and subsistence in Roman Britain (eg at inter- and intra-site levels: evidence of diet for people of high and low status in Roman Britain; differences between rural and urban settlements; does diet differ at military sites); data derived from ORA can also be integrated into broader-scale projects, such as those concerned with Roman agricultural practices	study of British Bronze Age vessels, intra-site variation in the use and deposition of pottery vessels detected at Trethellan Farm, Cornwall, where 'ancillary' buildings less likely than 'residential' structures to contain potsherds yielding lipids (Copley *et al* 2005b)
Food preparation techniques				
food preparation techniques	investigation of sherds from different parts of the vessel profile (for example base, middle and rim) from characterised vessel types	technologies and cultural culinary practices, such as boiling and roasting	comparison of use of particular vessel types found at a range of settlement sites (eg Roman black-burnished cooking ware) used both by military garrisons and civilians, found widely across much of Roman Britain from 2nd century to end of Roman occupation; ORA could examine possible variations in, and changes to, diet and cooking methods across the province through time	jars from Romano-British sites have distinctive patterning in ketone distribution, likely arising from cooking practices (Cramp *et al* 2012)
Resource acquisition/exploitation, trade and exchange				
resource exploitation, trade and exchange	analysis of amorphous masses of organic matter (for example lumps of 'resin')	some commodities, such as resins, can sometimes be provenanced to their geographic origin, providing evidence for reconstruction of trade and exchange routes	as required	Case study 5 ORA provided direct evidence for use of resinous exudates in mortuary rites in Roman Britain, providing information on practical and symbolic aspects of Roman mortuary rites – including coniferous resin, mastic and terebinth resin from the Mediterranean, and frankincense from southern Arabia or eastern Africa – giving insights into Britain's relationship with the Roman Empire (Brettell *et al* 2014, Brettell *et al* 2015a, b)
	investigation of assemblages of likely containers or matrices	diet and subsistence strategies, exploitation of resources, trade relationships	as required	Case study 2 *Brassica oleracea* (cabbage) identified in Saxon/ Medieval vessels at West Cotton, Northamptonshire (Evershed *et al* 1991)

5 Project Design, Planning and Sampling

This section provides good practice guidance on how to design and plan a project involving organic residues and explains the protocols necessary for a targeted sampling strategy that will best maximise results.

5.1 Project design

The starting point in any archaeological project involving ORA is the definition of a clear research question or series of questions that ORA can help to answer. In the case of planning-led archaeological work these questions would generally be formatted at the 'brief', or written scheme of investigation (WSI) stage and written into the project design (PD). At this stage, both the pottery specialist and the ORA laboratory where the analysis is to be carried out should be involved in planning the sampling procedure. These procedures should also be written in to the WSI/PD. In cases where fieldwork indicates that ORA will be important to the understanding of a site, arrangements for sampling could be made at post-excavation stage (again with input from the pottery specialist and the ORA laboratory). Provision could also be made for the retention of samples for future analysis.

Specific research questions towards which ORA can contribute might cover both temporal and spatial timeframes and relate to issues such as diet and resource use, settlement patterns and trade networks. Questions also might cover themes such as technology, for example, relating to form and function (see Table 2, Section 4, Sections 5.2.1 and 6 in this document, and Section 2, Research agenda and strategy in *Supporting Information* for examples of typical research questions). Once the research questions are determined they can be further refined into hypotheses that can be tested through an appropriate sampling strategy and the application of appropriate techniques or combinations thereof.

5.1.1 Questions to consider when starting a new ORA project

These questions must be addressed in consultation with other stakeholders, such as ceramic specialists and project managers.

- What research questions do I want to ask and will ORA help?
- What types and how many samples are required?
- How will I collect samples and what steps will I take to reduce the introduction of contaminants?
- What is the sampling protocol?
- What analytical techniques will be required?

5.2 Sampling strategy

5.2.1 Defining research questions

A well-constructed sampling strategy first will address the aims and objectives of the archaeological project, and, where possible, contribute to research questions identified in regional and national research agendas (see also Section 2 on research themes employing ORA in the *Supporting Information*). The aims and objectives will determine the sampling strategy: for example, number, type and context of samples required to answer the research questions. Research questions can be multifaceted: for example, as well as identifying the commodities stored or processed in vessels, different forms and functions could also be compared. Some examples of effective research questions are described in more detail below:

Example 1

The primary question addressed by one project was to 'ascertain the extent of dairy consumption in the British Bronze Age'. To this end, molecular and isotopic analyses were undertaken of absorbed lipid residues from 256 pottery vessels obtained from four southern British Bronze Age sites (Potterne, Brean Down, Black Patch and Trethellan Farm) to find out what commodities were being processed. However, questions of 'form, function and intra-site variability' were also addressed. At Trethellan Farm and Black Patch, sherds were selected from several different buildings in order to investigate possible intra-site variation in vessel use and deposition. The relationship between commodity group and vessel characteristics was also examined. This study demonstrated that dairying during the Bronze Age was an important component of animal husbandry practices at all four of the southern Britain sites sampled. It also established that dairy products were generally processed in vessels with smaller rim diameters than those used for cooking animal meat products. Finally, intra-site variation in the use and deposition of pottery vessels was detected at Trethellan Farm, where the 'ancillary' buildings were less likely than the 'residential' structures to contain potsherds yielding lipids (Copley *et al* 2005b).

Example 2

The 'timing and nature of the transition from the exploitation of marine and freshwater resources to an agricultural lifestyle in the Western Baltic' was investigated using a combination of molecular and isotopic analysis of absorbed and surface residues from pottery. Lipids were extracted from 133 ceramic vessels and 100 carbonised surface residues from 16 sites covering late forager (EBK), transitional, and early farming (TRB) phases. These sites dated to immediately before and after the first evidence of domesticated animals and plants in the Western Baltic and showed a continued exploitation of marine resources after domesticates arrived in the region. However, the residues also confirmed that dairying was practised at coastal and inland sites as soon as domestic animals appeared in the sequence. This study identified both aquatic biomarkers and ruminant fats and, significantly, confirmed that the Neolithic economic 'package' did not immediately replace the forager lifestyle in the Western Baltic (Craig *et al* 2011).

Example 3

In this project, pottery vessels from the British Iron Age sites of Maiden Castle, Danebury Hillfort, Yarnton Cresswell Field and Stanwick were submitted for ORA. Iron Age sites predominantly yield pottery vessels that can be classified into three main classes: 'saucepan pots', jars and bowls. Hence, *c* 60 vessels (20 from each vessel type) were sampled from each site to establish 'what commodities were being processed in each container and whether any of the particular vessel types were consistently used to process specific commodities'.

Results showed that both dairying and ruminant carcass products were a significant component of animal husbandry economies during the Iron Age. This supported the faunal evidence, which indicated a dominant role for ruminant animals in the local economies, with sheep, goats and cattle present in significantly higher numbers than pigs and wild animal species. The study also revealed that milk products were processed in 'saucepan pots' at sites where these pots predominated, but that at sites where jars dominated, jars, instead, were preferentially associated with milk products, demonstrating vessel specialisation by the users (Copley *et al* 2005a).

ORA might also be used to address research questions such as comparison between decorated and non-decorated vessels, between domestic and ritual contexts, and variation between sites and regions.

5.2.2 Planning a sampling strategy

Where possible, determine the sampling strategy for any project at the pre-project planning stage, in cooperation with the ORA laboratory that will carry out the analysis and any other stakeholders, such as pottery specialists working on the pottery assemblage.

The nature of the archaeological questions to be addressed will govern the sampling protocol itself (including type, size and number of samples together with modern reference materials that may be required) and the choice of analytical method. Some general information on sampling protocol is set out below.

5.2.2.1 How do I determine what types and how many samples are required?

It should again be emphasised that the greatest potential for ORA on ceramics is from its application at site level. The research questions to be addressed (see Section 5.1 Project design) will dictate the number and type of samples required. This should be agreed after consultation with the ORA laboratory that will be carrying out the analysis. As a general guideline, a minimum sample of *c* 20-30 sherds of at least c 2-3 g size (each representing a different vessel) per group will ensure that statistically reliable data are obtained. This is because recovery rates of lipids from sherds can vary widely, and sufficient data is needed to support wider interpretations. For example, in southern England, the rate of preservation of lipids is 58 per cent on average, whereas in Scotland it is 89 per cent on average (Copley *et al* 2005c; Cramp *et al* 2014). For a comparison between two assemblages (for example two different vessel forms, or cooking pots from an earlier and a later phase of the site) a minimum of 40-60 potsherds is therefore required. If felt necessary, a pilot study of *c* 10 samples representing the ranges of pottery types or contexts could be undertaken to assess the recovery potential of the assemblage.

5.2.2.2 How should I collect samples and what steps will I take to reduce the introduction of contaminants?

Guidance to maximise optimal sampling conditions for (i) sampling in the field and (ii) sampling archived material are set out below. Adherence to these sampling guidelines will help to preserve existing residues and protect them from possible sources of contamination.

5.2.2.3 Sampling ceramics in the field

- If possible, avoid direct contact with plastic or cling film; air dry sherds lifted from the burial environment, wrap in aluminium foil (or acid-free tissue paper) and place in brown paper envelopes or ziplock plastic bags if site conditions are wet. Once wrapped, store the sherds in boxes

- As far as possible, try to avoid handling the sherds. If available, use nitrile (powder-free) gloves to handle the sherds. If gloves are not available, wash hands before sampling to reduce contamination by skin lipids and cosmetic products

- In some cases, it may be considered useful to sample a small portion of soil (a few grams) from close by or adhering to the sherd (to test for soil contamination)

- Keep samples away from modern contaminants such as your coffee, lunch, suntan lotion etc

- For newly excavated material, avoid washing sherds intended for organic residue analyses. Washing can dislodge surface residues, remove more labile constituents of absorbed residues and introduce contaminants

- Avoid applying substances, such as glues, paraloid etc to sherds intended for analysis

5.2.2.4 Sampling of archived material (that is, held in museums)

- Obtain sampling permissions from the relevant museum or archive at the project-planning stage

- If possible, ascertain details of the storage history of the potsherds. For example, when were they accessioned? How were they stored (for example, in plastic bags or wood lined drawers)? What were the environmental conditions (for example, temperature and humidity)? What chemicals might they have come into contact with?

- It is preferable to select potsherds that have not been subject to cleaning or other treatments. However, excellent results have been obtained from numerous cleaned potsherds from assemblages in museum collections (for example, Case study 4) and therefore this should not preclude sampling from existing collections

5.3 Information to be supplied to ORA laboratories carrying out the analysis

- brief account of the nature and history of the site

- aims and objectives of the project

- full details of samples (site, find number, context, pottery type, fabric)

- details of preservation environment (for example, anoxic, oxic)

- storage record and any conservation history

- health and safety considerations (for example, asbestos tempered sherds, presence of fungal spores and mould, contaminated soil)

6 Research Agenda and Strategy

Regional or period-based research framework documents are valuable tools for helping set aims and objectives relating to archaeological projects. Other useful sources include publications by pottery groups such as the *Prehistoric Ceramics Research Group Research Framework: Agenda and Strategy*, *A Research Strategy and Updated Agenda for the Study of Roman Pottery in Britain* and *A Research Framework for Post-Roman Ceramic Studies in Britain*.

Details of these regional and thematic research frameworks can be found in Table 1 in the *Supporting Information*. Section 2 gives detailed information and suggestions for the application of ORA to help address specific archaeological research questions identified as priorities in ceramic and regional research frameworks.

Although the web-based guidance does not attempt to provide a prescriptive list of research aims, direction is given to areas where specific 'themes' for (site-level) research can be targeted in order to address site or region specific questions. These themes can be prioritised at site level in order that research becomes targeted nationally (and internationally). This is intended both to enhance the value of individual projects and also, importantly, to maximise the collective research potential of datasets.

Such themes include, but are not limited to:

- archaeological deposits and finds assemblages
- chronology
- form and function of pottery
- overarching archaeological research themes across both temporal and spatial scales, such as the investigation of aspects of continuity or change in dietary practices across transition periods in prehistory

7 Frequently Asked Questions

Q **The site is being excavated on a very tight budget and I only have enough money for analysis of (say) 20 sherds. Is it worth doing?**

A If you have specific research questions that ORA can address, then 20 potsherds will be an adequate sample size. The sherds should be selected in consultation with the ORA laboratory carrying out the analysis, who will advise on how to maximise the research potential.

Q **What size does each sample need to be?**

A To extract lipids from pottery a small sherd, *c* 2–3g, is required for processing. This is approximately the size of a £1 coin. However, if possible, it is preferable to provide larger sherds, from which a small piece will be taken, and the remainder returned intact. If sampling amorphous organic remains such as resins or burnt residues, a very small amount can be sufficient, perhaps tens of milligrams. If it is pure organic material, a pinhead-size piece might suffice, although a larger piece would be preferable, as this makes repeat analysis possible.

Q **Are the techniques destructive?**

A The technique requires grinding the ceramic sherds to powder to extract lipids. Therefore, it is, by necessity, destructive. However, only a small portion of pottery is required (see above) and any sherd remaining after sampling can be returned, if required.

Q **Which parts of a vessel are better to sample?**

A If possible, rim sherds are usually preferred for analysis. Research has established that, based on the 'fat floats on water principle', cooking vessels accumulate large concentrations of lipids near the rims of vessels and gradually lower amounts towards and in their bases (Charters *et al* 1993).

Q **Does it matter if the sample has been washed?**

A If sampling directly from site, it is preferable that the sample is not washed (see Section 5.2.2 on sampling strategies). However, if sampling from existing museum collections, it is likely that the potsherds will already have been cleaned. It should be noted that excellent results have been obtained from numerous cleaned potsherds from assemblages obtained from museum collections, therefore this should not preclude sampling from existing collections (see Case study 4).

Q **Are there any special recommendations for the treatment of material to be sampled, such as storage and handling?**

A It is important to keep handling of materials intended for sampling to a minimum, where possible. For detailed information, see Section 1.3 in the *Supporting Information* on factors affecting the preservation and decay of organic residues.

Q How long will the analyses take?

A The time required to analyse samples will depend on the number of samples to be analysed and the specific techniques required for their analysis. The analyses are quite lengthy and may take several weeks to months. The laboratory carrying out the analyses of your material will give you an indication of how long this will take.

Q How much does ORA cost?

A Organic residue analysis is not one analysis, but can involve complex sample preparation steps and multiple instrumental analyses depending upon the question being asked and material(s) being analysed. Therefore, the cost per sample can vary depending on which types of analysis are carried out. The cost may vary slightly between the main laboratories that carry out the sampling analyses, but as a rough guideline can range from £50 to £300 per sample. Quotations for analyses can be obtained from the main laboratories, whose staff may be willing to apply a reduction in cost for larger numbers of samples. If analyses are required for assemblages that fit within regional research guidelines frameworks for relevant research questions that are highlighted (see Section 2 in the *Supporting Information*), then it might be worth contacting any of the university laboratories to enquire whether the analyses might be carried out as part of ongoing research projects.

ORA is also available through the NERC Services and Facilities at the University of Bristol for Research Council UK (RCUK) remit researchers, including PhD students. RCUK remit researchers include those employed at an eligible UK academic or research institution or holding a NERC Fellowship. For further details please refer to the NERC funding guide.

Q What sort of materials can ORA identify? Can ORA detect specific commodities such as x, y or z?

A See Table 1 for details of the range of commodities that can (reliably) be identified currently. Specificity is limited over archaeological timescales by the survival of biomarkers that would unambiguously identify a compound. However, if you have a specific question about a product or commodity not covered in these guidance, please contact one of the ORA laboratories for technical advice.

Q Can alcoholic beverages or narcotic substances be detected?

A There are significant challenges involved in the chemical identification of organic residues that suggest the processing of alcoholic beverages or narcotic substances in archaeological vessels. If you have specific research questions involving ORA of vessels you suspect were used to process these commodities, please contact one of the ORA laboratories to discuss this (for contact details see below).

Q Can the storage or processing of cereals be identified in organic residues?

A Lipids denoting plant processing have been identified in vessels (for example, *Brassica oleracea*); however, no specific biomarkers have yet been identified for cereals known to have been exploited in British prehistory. Furthermore, starchy grains, such as barley and wheat, have a lower lipid content than fattier animal products such as meat or milk, which would mask their signal and make identification difficult.

Q **Can organic residues be extracted from vessels made from other materials, for example, chalk, chlorite, steatite, wood?**

A The porous nature of the ceramic fabric, which is an important factor in the preservation of absorbed organic residues in archaeological potsherds, suggests the possibility of lipid recovery from other inorganic materials with similar internal structures. Lipids have been extracted from stone vessels – for example, chalk, chlorite and steatite – suggesting that stone fabric has the capacity to absorb and preserve organic material in a similar way to ceramic (for example, Clelland *et al* 2009, Namdar *et al* 2009, Tanimoto *et al* 2011). Organic vessels, such as wood or leather bowls, are unlikely to yield meaningful lipid profiles from commodities processed in them, as the lipids present in the commodity could not be distinguished from the lipids present in the wood or leather itself.

Q **Preservation of other organic material at the site being excavated, for example, bone, is very poor. Is it worth doing ORA?**

A Yes, organic residues can survive well in conditions such as acidic soils that are not conducive to the survival of other types of organic material. ORA can act as a **proxy** (Case study 4) and provide crucial and viable evidence where other organic remains do not survive.

Q **Can ceramics from the historic period be analysed?**

A To date, ORA has mostly been carried out on ceramics from periods where written evidence of commodity use is either sparse or non-existent. Some medieval pottery has been analysed (Case study 6), demonstrating its research potential. Hence, ORA could be successfully used on historic period, unglazed ceramics, for example, to test the written record on diet or resource exploitation.

8 Case Studies

CS1: 'Recycled' beeswax candles from Fountains Abbey

J Dunne and C Heron

Keywords
Fountains Abbey, candleholders, organic residue analysis, fatty acids, tallow, beeswax

Summary
ORA identified visible residues adhering to metal candlesticks comprised 'recycled' beeswax from church candles mixed with tallow to make domestic candles.

Background

In this case study, ORA provided a rare opportunity to identify visible residues found adhering to metal candleholders from 12th century Fountains Abbey (Figure CS1.1). Today, the Cistercian Abbey of Fountains, part of the Studley Royal Park, is a World Heritage Site in the River Skell Valley near Ripon. The Abbey holds an extensive collection of artefacts, including 12th century lead and iron candleholders that had white flaky deposits, suspected to be candle wax, in their sockets.

Figure CS1.1
The ruin of Fountains Abbey, North Yorkshire.

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Results

GC and GC-MS analysis of the white deposits adhering to five candleholders (Figures CS1.2 and CS1.3) identified two types of residue composition (Figure CS 1.4; Frith *et al* 2004). The first lipid profiles, dominated by $C_{16:0}$ and $C_{18:0}$ free fatty acids, were consistent with degraded animal fat. Triacylglycerols (TAGs) with acyl carbon numbers in the range $C_{46:0}$ to $C_{50:0}$, and their degradation products, diacylglycerols(DAGs) and monoacylglycerols (MAGs) were also present.

The presence of $C_{15:0}$ and $C_{17:0}$ odd-carbon-numbered fatty acids, known bacterial markers that result from microbial activity in the rumen, strongly suggest a ruminant animal source. The profile is consistent with a degraded tallow product known to have been used in medieval candle making.

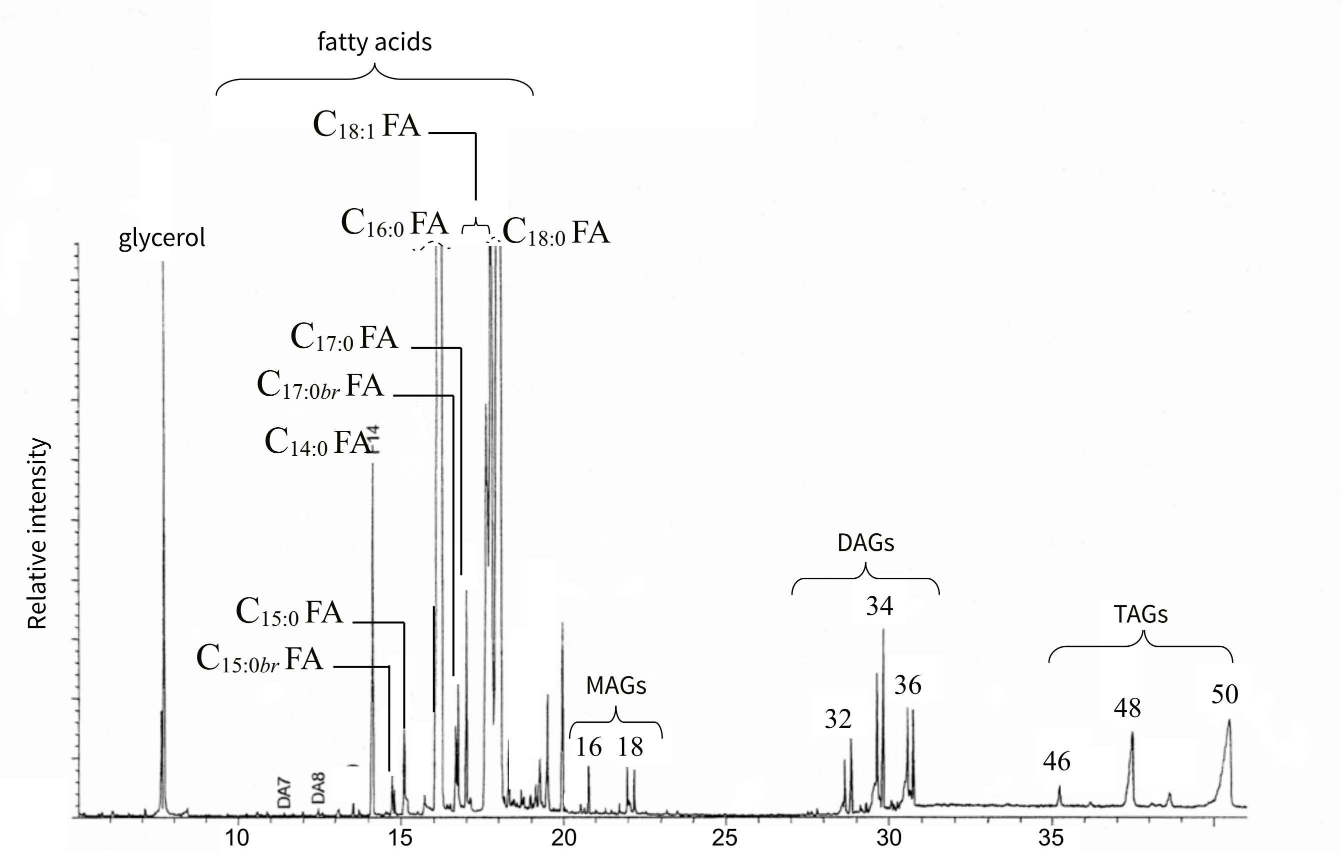
Tallow is obtained by skimming the fat from the top of vats where animal carcasses, commonly beef and mutton, are boiled. Mutton tallow was particularly valued for its qualities of 'gloss and hardness'. The Abbey gained a large proportion of its wealth from wool production from the 18,000 sheep kept on its estate in the early medieval period, suggesting that mutton was the likely source of the tallow made for the candleholders.

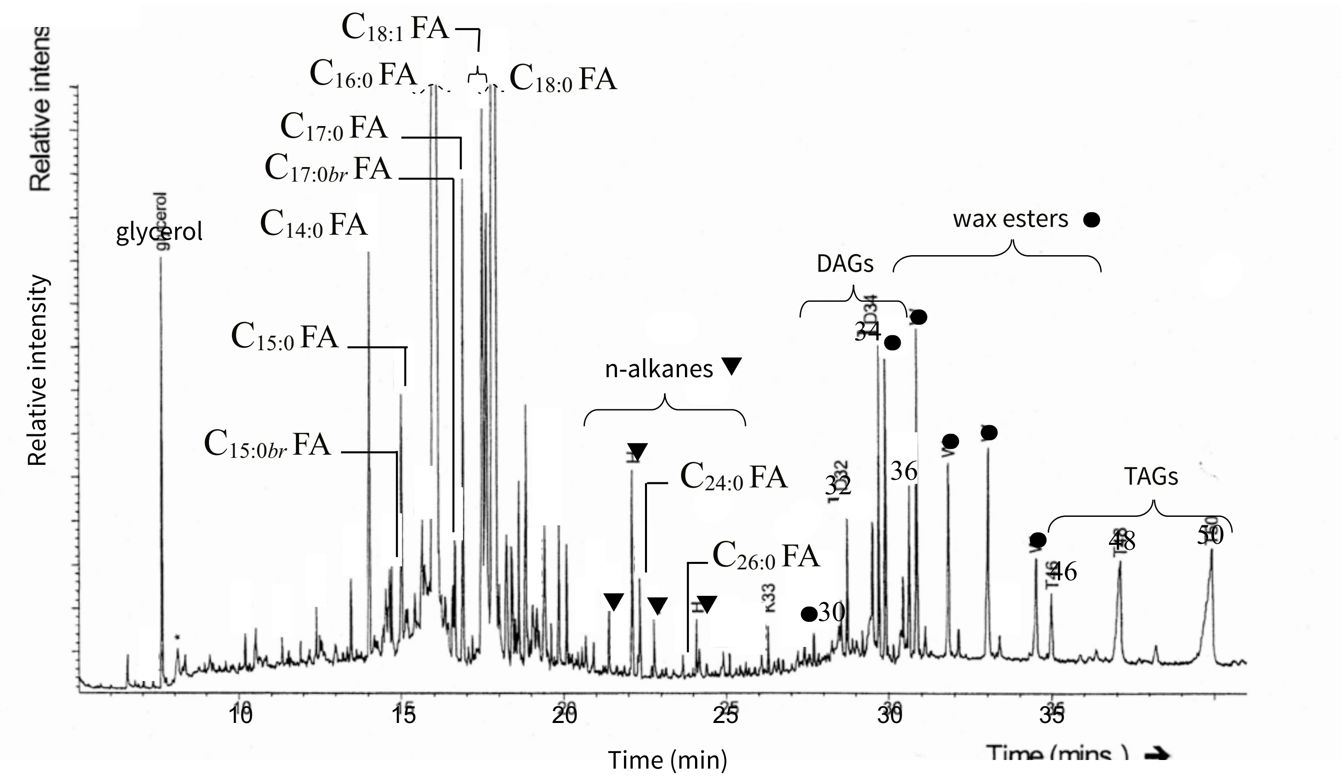
The second type of lipid profile comprised the same components indicative of degraded tallow, with the addition of a series of compounds known to be diagnostic biomarkers of beeswax. These included *n*-alkanes (C_{21} to C_{33}) maximising at C_{27}, long-chain free saturated fatty acids (C_{20} to C_{30}) and palmitate wax esters in the range C_{40} to C_{50}. Low abundances of hydroxymonoesters and a suite of long chain alcohols were also present, all consistent with a beeswax candle source.

Three of the candleholders had residues composed solely of tallow, but as the remainder included diagnostic biomarkers denoting the presence of beeswax, it is likely that beeswax and tallow were 'mixed' to make candles. This mixture suggests that the candleholders were used partly for domestic purposes, as only candles made of pure beeswax would have been used in the church.

Figure CS1.2: Lead-socketed candleholder with bowl and short handle.
Note the deposits – light in colour in comparison to the metal – adhering to the interior surface of the socket and the bowl (Sample 671502).

Figure CS1.3: Iron 'cupped-stick' candleholder, used as a wall fixture.
The socket was plugged with a flaky, white deposit similar to that found in Figure CS 1.2.
Both images © English Heritage Trust/Bob Smith.





CS1.4 Gas chromatograms showing lipid profiles from two candle holders.
Note compounds marked $C_{x:y}$, denoting free fatty acids of carbon length x and degree of unsaturation y, and MAGs (monoacylglycerols), DAGs (diacylglycerols) and TAGs (triacylglycerols) with numbers above the peaks denoting the number of acyl carbons. These distributions suggest animal fat processing. Wax esters are indicated with filled circles and long-chain *n*-alkanes with filled triangles, both of which are found in beeswax. The top chromatogram suggests a tallow origin and the bottom chromatogram indicates a mixture of tallow and beeswax.

Discussion and conclusion

Wax candles were regarded as sacred in the early Christian Church and their use in ecclesiastical services was obligatory. This is true today – only beeswax candles can be used in certain Catholic services. The Gwentian Code of the Ancient Laws of Wales, first written in the 1200s, begins with the passage: 'The origin of bees is from Paradise and because of the sin of man they came thence, and God conferred His grace on them, and therefore the mass cannot be said without the wax.'

Beeswax was considered the purest of all animal or vegetable waxes and many early Christian writers regarded bees as being virgin – for example, Ennodius of Pavia (d 521 AD) spoke of the chastity of the working bee as typical of the virgin birth of the Saviour. Bees were also highly revered as an example of an ordered Christian community.

The availability of beeswax in the environs of the Abbey is borne out by documentary evidence; the chartulary of Fountains Abbey holds a grant dated October 1st 1284: 'Grant by Edmund, son of Richard, King of Germany … to the Abbot and Convent of Fountains … grants that they may have the honey and bees found in their woods.'

In conclusion, the lipid profiles suggest that candle remnants used in the church were recycled for domestic use with the addition of tallow. Certainly, candles made solely from tallow have a putrid stench and emit offensive smoke, a point noted by Samuel Pepys in the 17th century. Beeswax candles emit a much brighter light than tallow, and, in practical terms, the coating of tallow candles with beeswax makes them able to be held in the hand without melting. Improving the smell was likely also a consideration. Long-chain ketones, previously identified as biomarkers for cooking at high temperatures in archaeological pots, were found in the residues containing beeswax. Experiments showed that higher temperatures are attained in beeswax candle flames than in tallow flames, supporting the suggestion of mixed-composition candles (Frith *et al* 2004).

CS2: Epicuticular waxes identifies cabbage cooked in late Saxon pottery

J Dunne and R Evershed

Keywords

West Cotton, late Saxon, potsherd, organic residue analysis, leaf waxes, *Brassica oleracea,* cabbage

Summary

ORA identified epicuticular wax components denoting the cooking of cabbages in late Saxon/ early medieval potsherds from West Cotton, Northamptonshire.

The site and background

This case study focuses on residues extracted from late Saxon/early medieval potsherds excavated during the Raunds Area Project in Northamptonshire. The multi-period study investigated rural landscape development in Northamptonshire and the wider Midland region, and covered extensive excavations from Neolithic and Bronze Age sites beside the River Nene, Iron Age and Roman settlement at Stanwick and Saxon and medieval settlement in north Raunds and West Cotton. The excavations at West Cotton were of national importance for understanding the origin and development of villages in the late Saxon period (Chapman 2010).

West Cotton was the smallest of three deserted medieval hamlets located on the margin of the floodplain within the Raunds area. All three were probably late Saxon planned centres on regular one-acre plots. An 11th-century manor was identified, comprising a timber hall with ancillary buildings and a watermill (Figures CS2.1 and CS2.2). Later, a 13th century manor house was added, followed by a series of stone houses built over the top of the old manor house (Chapman 2010).

Results

Organic residue analysis focused on more than 100 potsherds (Figures CS2.3 and CS2.4) from a variety of site contexts, using GC and GC-MS to identify what commodities were processed in the vessels. Of these, lipid profiles extracted from five sherds revealed a mixture of compounds that included three major components: *n*-nonacosane, *n*-nonacosane-15-one and *n*-nonacosan-15-ol, all known to be the main constituents of the epicuticular leaf waxes of *Brassica oleracea* (cabbage). These are long-chain aliphatic compounds present in nature as constituents of epicuticular leaf waxes of higher plants and known to protect the plant from bacterial and fungal pathogens and decrease moisture loss. This suggests that the five potsherds with these biomarkers are from vessels that were used to cook *Brassica* vegetables.

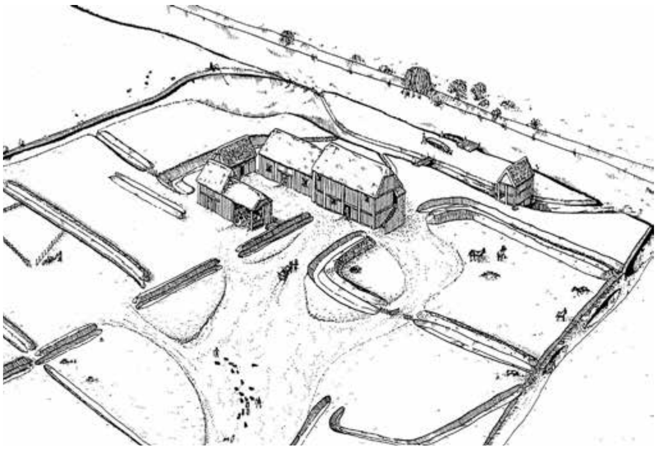


Figure CS2.1 (top)
General view of West Cotton, showing the timber hall (centre) and the mill leat (foreground).
Northamptonshire Archaeology; now MOLA Northampton.

Figure CS2.2 (above)
Reconstruction of late Saxon West Cotton, 11th century.
Northamptonshire Archaeology; now MOLA Northampton.

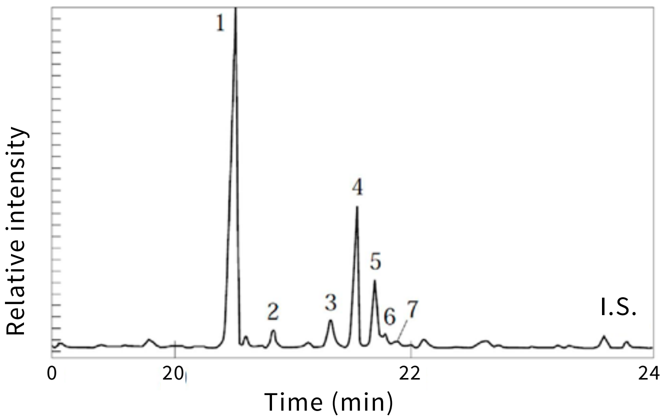


Figure CS2.3 (top)
Shelly ware cooking pots, showing some sooting on their lower bodies.
Northamptonshire Archaeology; now MOLA Northampton.

Figure CS2.4 (above): Partial gas chromatogram showing the lipid extract from the medieval jar (RP54).
In each case the peaks represent Brassica leaf wax components: 1 = *n*-nonacosane; 2 = *n*-hexacosanol; 3 = *n*-heptacosanol; 4 = *n*-noncosan-15-one; 5 = *n*-nonacosan-15-ol; 6 = *n*-hentriacontane; and 7 = *n*-octacosanol; I.S. = internal standard, *n*-tetratriacontane (Charters *et al* 1997).
Reprinted from Journal of Archaeological Science, 24, Charters, S, Evershed, R P, Quye, A, Blinkhorn, P W and Denham, 'Simulation experiments for determining the use of ancient pottery vessels: the behavior of epicuticular leaf wax during boiling of a leafy vegetable', Pages No.1-7, Copyright (1997), with permission from Elsevier.

Interestingly, these waxy components were also identified in more complex lipid profiles, including fatty acids, and mono-, di- and triacylglycerols, from some of the other vessels sampled. These lipids denote a typical degraded animal fats profile, suggesting that the cabbage was cooked with carcass fats, possibly as part of a stew.

Discussion and conclusion

The earliest records of cultivated cabbages are found in ancient Greek and Roman sources, and date from *c* 600 BC. Today, there are more than 200 cultivated varieties of *B. oleracea*, including cabbage, cauliflower, kale, broccoli, savoy and Brussels sprouts (Brouk 1985). Comparison to modern varieties of *B. oleracea* shows that the distribution of lipid components found in the West Cotton vessels best fits that of cabbage. Another possibility is turnip (*B. rapa*), which was also cultivated during the late Saxon period; however, analysis of modern turnip leaves shows that predominantly they contain free fatty carboxylic acids and not the components described above.

At West Cotton, there is no evidence of cultivated cabbage or turnip from the archaeobotanical record, but the detection of the leafy parts of plants is known to be difficult, and seeds and pollen are often absent as they are harvested before seeding. Consequently, this case study shows that lipid analyses can demonstrate the utilisation of leafy vegetables at sites where there is an absence of other archaeobotanical evidence (Evershed *et al* 1991).

CS3: Ascott-under-Wychwood – the earliest evidence for dairying in Britain

A Barclay

Keywords

Neolithic, carinated bowl, southern Britain, dairying

Summary

ORA of early Neolithic pottery sealed under a long barrow revealed a very high prevalence of dairy fats, correlating with a high proportion of cattle bones in the faunal assemblages.

The site

The Cotswold long barrow at Ascott-under-Wychwood (Figure CS3.1) was constructed in the late 38th century cal BC, during the first centuries of the Neolithic in southern Britain. It was likely only to have been in use into the 37th century cal BC, lasting for about three to five generations. It was built on the site of a substantial midden deposit. The site is important because it provides the earliest known substantial evidence for a Neolithic lifestyle from a sealed and secure context from southern England, predating the construction of the numerous causewayed enclosures and long barrows that are found across areas of Britain. Such sealed sites are rare, making them key to understanding the social mechanisms by which a farming lifestyle was introduced and spread across Britain and Ireland from mainland Europe (Benson and Whittle 2007).

The ceramic assemblage

The Ascott-under-Wychwood long barrow covered a midden deposit that contained one of the earliest and largest assemblages of Early Neolithic

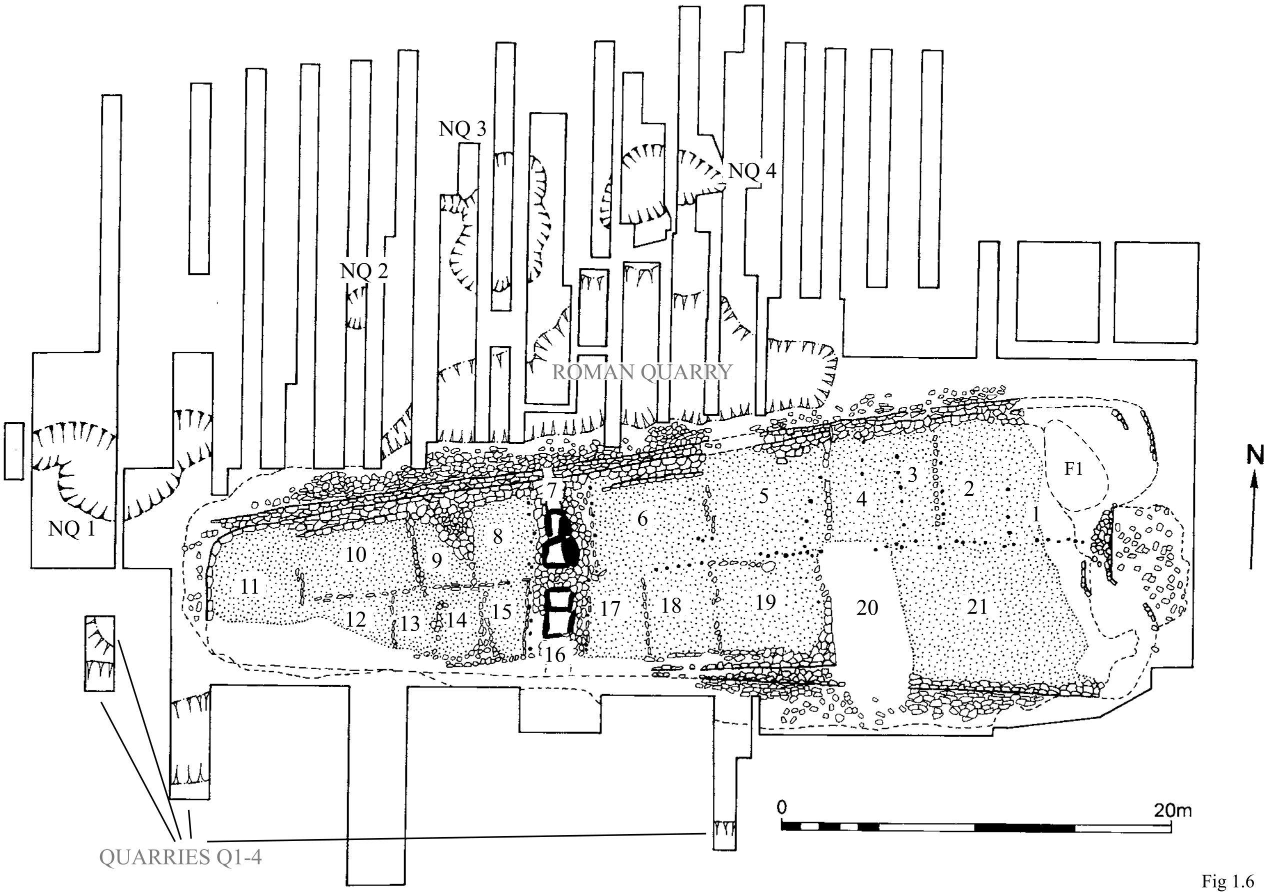


Figure CS3.1
Site plan for Ascott-under-Wychwood.
From Benson and Whittle 2007, copyright: Ian Dennis, Cardiff University, fig 1.6.

pottery ever found in Britain. The assemblage was recovered from occupation deposits of 39th or early 38th century BC date, arguably pre-dating the building of the long barrow by at least 50 years (Benson and Whittle 2007).

This assemblage has been characterised as Carinated Bowl (Barclay and Case 2007), a type of pottery that can be traced across much of Britain and Ireland during the first 250 years of the Neolithic (*c* 4000–3750 BC), and with stylistic origins within the Chasséen and Michelsberg pottery traditions of Belgium and Northern France. Significantly, the pottery – which displays a wide variation in size, form, fabric and colour – appears to represent an assemblage used in everyday life rather than comprising pots made specifically for mortuary rituals.

The research question

At a site level, ORA on this assemblage provided an important opportunity to examine what foodstuffs were served and consumed in a variety of ceramic vessels; and also to look at how the earliest farming communities in southern England used their everyday pottery. On a broader scale, this case study contributed to research on where and when dairying was practised by

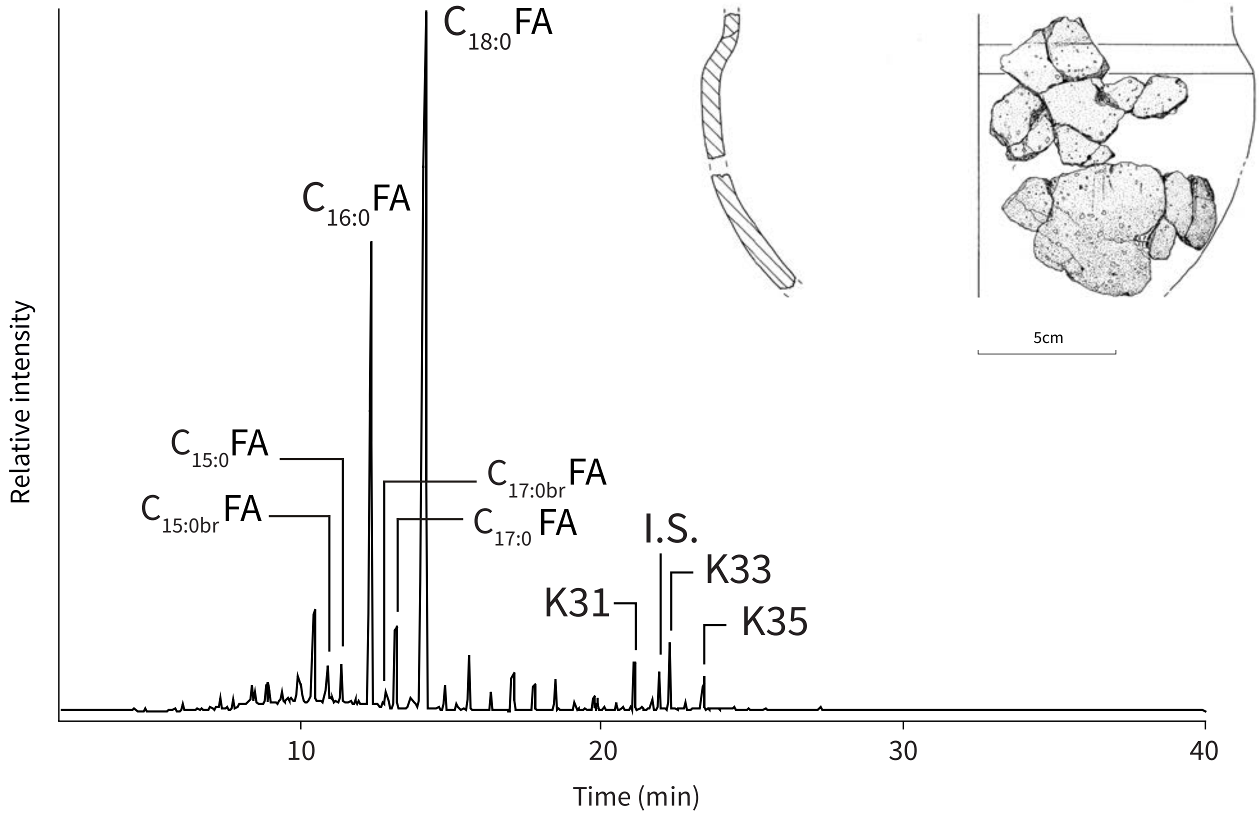


Figure CS3.2
Part profile of vessel from Ascott-under-Wychwood on partial HTGC profile of the trimethylsilylated total lipid extract from sample AuW1, illustrating the distribution of components characteristic of animal fat that has undergone heating and extensive degradation. Key: $C_{x:0}$ are saturated free fatty acids of carbon length x, and KX are mid-chain ketones of carbon length X. I.S. is the internal standard (C_{34} *n*-alkane).
Modified from Barclay and Case 2007, copyright Ian Dennis, Cardiff University, fig 10.3 (vessel profile) and Copley and Evershed 2007, fig 11.1 (chromatogram)

early Neolithic farmers. The substantial faunal assemblage recovered from the site also meant that a direct comparison could be made between the nature of the assemblage and the pottery lipid residues.

The sampling strategy

The Ascott-under-Wychwood ceramic assemblage was excavated in the 1960s and subsequently stored in a museum. Consequently, this study tested the potential for lipid analysis of ceramics stored over long periods in museum collections. In this instance, many of the sherds had evidence of the application of glue during post-excavation processing, necessitating particularly vigorous cleaning of their outer surfaces during sampling. Vessels ranged in size from small cup-like bowls to large bowls, and included thin-walled fine wares and thick-walled coarse wares. Most vessels were carinated, and some had been smoothed while others were burnished. Forty-eight potential vessels were recovered from the site; 39 came from pre-barrow contexts; and 32 potsherds were selected for residue analysis (Copley and Evershed 2007).

Results

Of the 32 vessels selected for residue analysis, 11 (34 per cent) had absorbed lipid residues (Figure CS3.2). This percentage represents a slightly lower recovery rate than has been achieved from other British sites of the same age, emphasising the need for a minimum sample size in order to ensure a statistically reliable amount of data. Based on the **δ^{13}C** values, analysis showed that eight vessels predominantly contained ruminant dairy fats, two contained ruminant carcass fats and one contained porcine fats (Figure CS3.3). Significantly, 82 per cent of the vessels with residues had traces of dairy fats, one of the highest values so far recorded in any assemblage of early Neolithic date in Britain. The identification of mid-chain **ketones** in four sherds and charred residue on another provided direct evidence that pots were used for the cooking as well as for serving food.

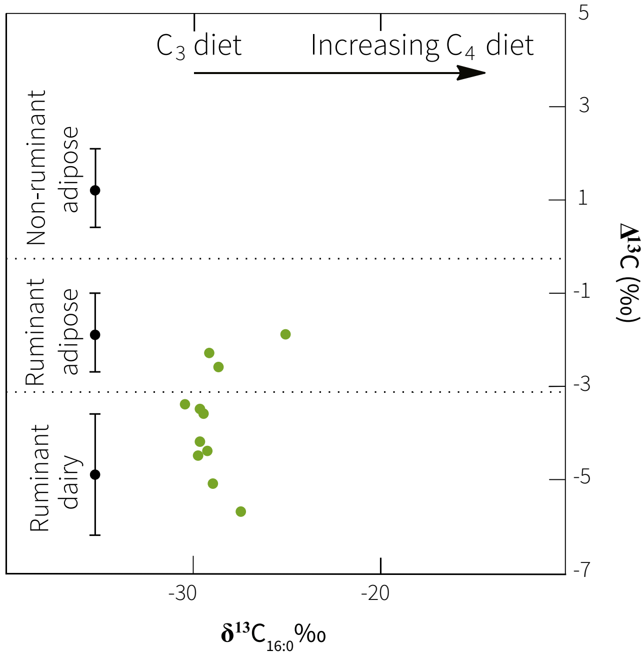


Figure CS3.3
Plot of the Δ^{13}C values ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) plotted against $\delta^{13}C_{16:0}$ values of the fatty acid methyl esters prepared from lipid extracts from the Ascott-under-Wychwood assemblage. The predominance of dairy fats demonstrates the importance of dairying to these early British farmers.
From Copley and Evershed 2007.

Comparison with other lines of evidence

The prevalence of ruminant animal fats (dairy and carcass) in the organic residues correlates with the largely domestic faunal assemblage found, in which cattle dominated, but with *c* 10 per cent from sheep/goats and pigs (Mulville and Grigson 2007). Furthermore, the difference in nitrogen **isotope** values between the humans and the domesticates suggests a high level (> 50 per cent) of animal protein consumption (Hedges *et al* 2007).

Discussion and conclusion

At a site level the varied nature of the assemblage enabled investigation of relationships between pottery form and function. However, no significant correlations were found with either vessel fabric or colour. Analysis of sherd wall thickness showed that lipid residues were not found in thin-walled small bowls or cups, but were present in medium-and thick-walled bowls. This suggests that cups and small bowls were not generally used to process animal products, whereas just under half the medium and large vessels did contain fats. Interestingly, the presence of mid-chain ketones in four sherds provided direct evidence that pots were used for cooking as well as for serving food.

These results suggest that the consumption of dairy products, likely originating from cattle, was a significant component of the diet of the early farmers at Ascott-under-Wychwood. Carcass fats were also processed from sheep/goats and pigs.

On a broader scale, this evidence correlates with studies of pottery from a range of other key early Neolithic sites in Britain, and confirms that dairying was a widespread activity in this period; and therefore was probably already well established when farming was introduced into Britain in the 5th millennium BC (Copley *et al* 2003, Cramp *et al* 2014).

CS4: Organic residues – an important animal proxy for the Irish Neolithic

J Smyth

Keywords

Organic residues, Ireland, Neolithic, dairying, fatty acids

Summary

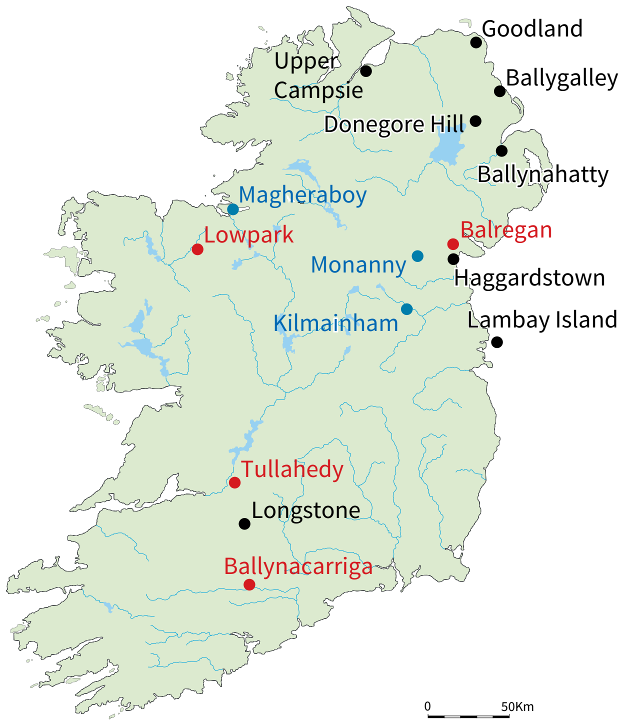
ORA, used as a proxy for animal bone, identifies continuous and sustained exploitation of dairy fats by Irish Neolithic farmers.

Background

This case study investigated the chronology and spread of early farming methods on the island of Ireland. Preservation of animal bones is poor in the acidic soils of Ireland. Hence ORA was used as a proxy for understanding animal husbandry practices. Organic residues (lipids) were extracted from assemblages of early, middle and late Neolithic pottery, including Grooved Ware, to determine what commodities were being stored, cooked and served in pots. The study also sought to ascertain whether vessels found across domestic, funerary and ceremonial contexts were used in similar ways.

Sampling strategy

This study also demonstrates the potential of a well-planned sampling strategy. The specific set of research questions considered dietary practices across a defined chronological period and geographic location, together with comparisons between different contexts (site types). Careful site and sherd sampling were applied to maximise the chances of obtaining meaningful, statistically robust results. Wherever possible, sites with assemblages of more than 30 vessels were targeted. If lipid recovery rates were to be as low as 30 per cent, the detection of lipids from 10 sherds would provide a statistically acceptable minimum, although adopting this criterion meant that several archaeologically interesting sites were passed over, in particular megalithic monuments, which generally have small pottery assemblages. Rim sherds and upper body sherds were sampled where possible, as cooking vessels



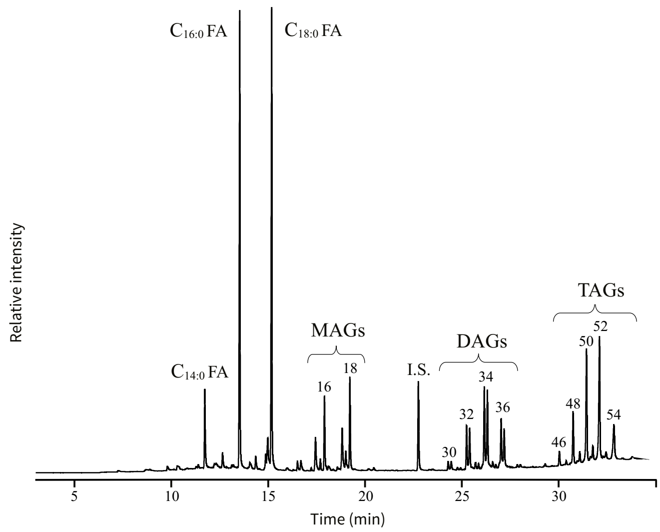


Figure CS4.1 (top): The locations of Neolithic sites sampled.
Blue = Early Neolithic; black = Middle Neolithic; red = Late Neolithic.

Figure CS4.2 (above): Typical chromatogram of a lipid extract from an Irish Neolithic pot, containing lipid components characteristic of a degraded animal fat.
$C_{x:y}$ FA = free fatty acids of carbon chain length x, and degree of unsaturation y; MAGs = monoacylglycerols; DAGs = diacylglycerols and TAGs = triacylglycerols. Numbers above peaks denote number of acyl carbons. I.S is the internal standard (C_{34} *n*-alkane).

accumulate large concentrations of lipids near the rims of vessels and gradually lower amounts towards and in their bases.

The Irish Neolithic can be divided into early, middle and late phases, which are defined by changes in domestic architecture, monuments and artefact types (including pottery); analysis thus sought to examine whether there were discernible differences in vessel use and contents through these phases. Assemblages from different site types were also selected – such as causewayed enclosures, houses and pit complexes – and from different parts of the island, to explore whether site function and geography had an impact on what pottery was being used for which functions.

In total, more than 450 sherds from 15 sites were analysed (Figure CS4.1). Eight of these assemblages came from recent, commercially-excavated sites, while pottery from the remaining seven sites had been recovered from research excavations dating from the 1950s onwards, and subsequently stored in museums. Significantly, there was no noticeable difference in residue recovery rates from newly excavated sherds and those stored in museums.

Results

GC screening revealed extremely good preservation of absorbed lipids (*c* 90 per cent), with nearly 60 per cent of sherds showing profiles consistent with degraded animal fats (Figure CS4.2). Intact triacylglycerols (TAGs) and their degradation products, diacylglycerols (DAGs) and monacylglycerols (MAGs) were observed in over 90 per cent of these animal fats, further demonstrating the high level of lipid preservation. The presence of C_{42}, C_{44} and C_{46} TAGs was often noted, compounds only detectable in milk fat; and of the 212 samples carried forward for compound-specific carbon stable isotope analysis, nearly 90 per cent had $\delta^{13}C$ values consistent with those from reference milk fats. Indeed, ruminant milk fats were the dominant fat observed in residues from vessels dating to all phases of the Irish Neolithic (Figure CS4.3).

We now know that the first farming communities on the island were already involved in milking their livestock and processing those products in pots, that is, that dairying was not a farming practice 'discovered' or developed at a later, more advanced stage of agricultural development (Sherratt 1981). Furthermore, the results indicate that this subsistence regime did not substantially shift during nearly a millennium and a half.

Discussion and conclusion

The results from this project have highlighted the considerable potential of archaeological lipid analysis for exploring the nature of human–animal relations in the past. With poor bone preservation in Irish acidic soils, species identification is often extremely challenging and the construction of kill-off profiles, from which the management of dairy herds is traditionally inferred, is near impossible. For example, at the house sites of Kilmainham and Monanny, faunal assemblages consisted mostly of small fragments of indeterminate burnt animal bone, occasionally identified as from 'medium-sized' or 'large-sized' mammals. Consequently, the identification of ruminant and non-ruminant meat and milk fats using ORA has served as an important proxy for animal exploitation in the Irish Neolithic.

Thus, lipid analysis is particularly well suited to pottery assemblages from sites located on acidic soils. Comparable results from British Neolithic sites show lipid recovery rates of 54 per cent of sherds from southern England and of 87 per cent of sherds from Scotland. Such a difference is more than likely due to similar acidic soils covering much of Ireland and Scotland, which seem to better preserve lipids. Indeed, such is the potential for high lipid yields in Irish unglazed vessels (from the Neolithic to the early medieval period), that statistically robust results can be obtained from fewer than the guideline 30 potsherds (see Cleary and Kelleher 2011, 387–9). Unglazed pottery from sites across Britain and Ireland thus forms part of a substantial, as yet largely untapped, archaeological resource (Smyth and Evershed 2015a).

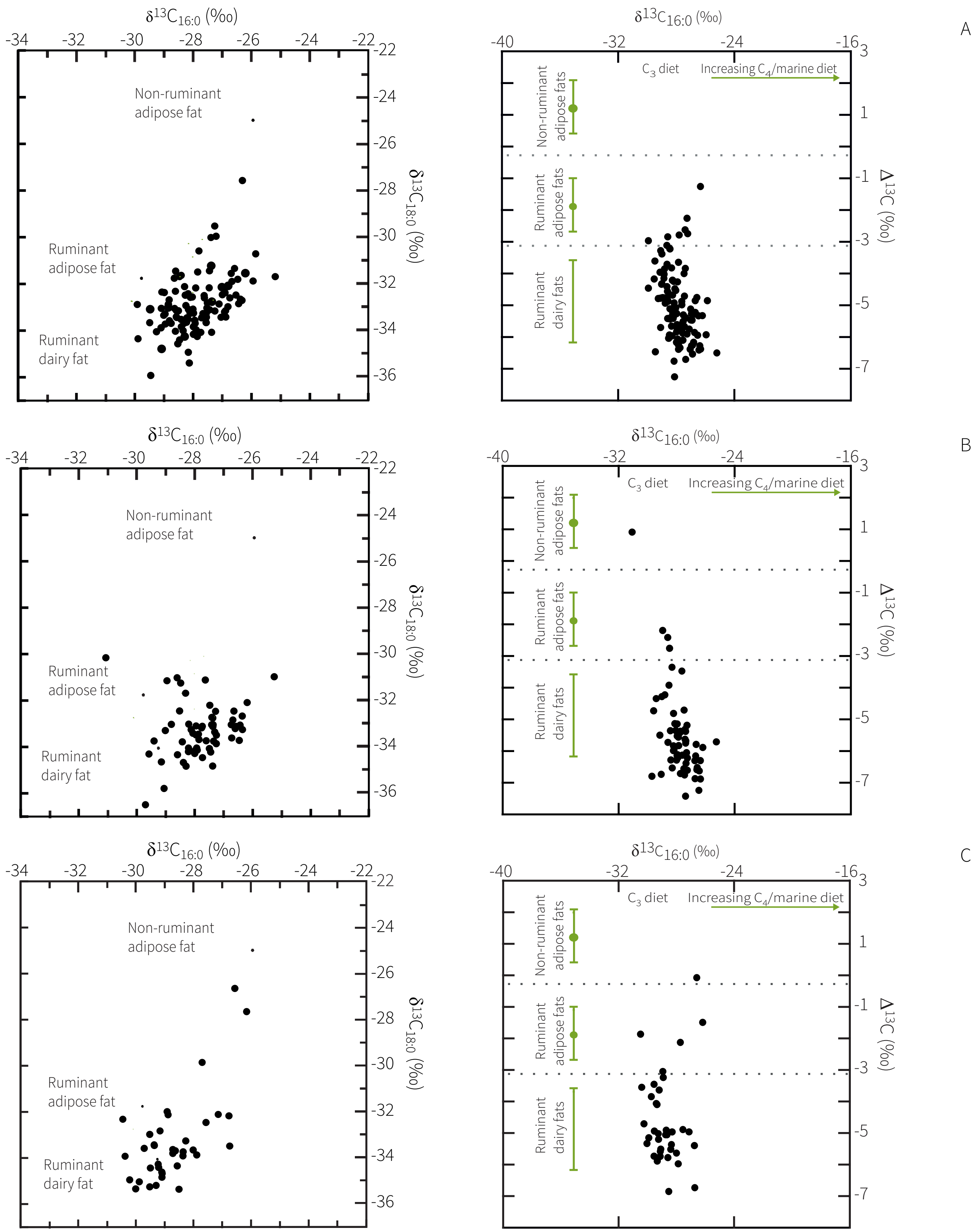


Figure CS4.3: Ruminant milk fats were the dominant fat observed in residues from vessels dating to all phases of the Irish Neolithic

(left) Scatter plots showing $\delta^{13}C$ values of methylated individual $C_{16:0}$ and $C_{18:0}$ fatty acids;
(right) $\Delta^{13}C$ values ($=\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) of the same fatty acids plotted against $\delta^{13}C_{16:0}$ values.

A – Early Neolithic sites, B – Middle Neolithic sites, and C – Late Neolithic sites.
Smyth and Evershed 2015b, fig 5. Reproduced with permission of the Royal Irish Academy.

CS5: Resins from Roman mortuary contexts

R Brettell

Keywords
resinous substances; GC-MS; mortuary rites; Roman Britain

Summary
ORA of amorphous residues from Romano-British mortuary contexts identified a variety of resinous exudates, including pine resins, terebinth and frankincense.

Background
In this case study, ORA was used to identify 'grave dust' found in Romano-British mortuary contexts. There has long been speculation about the use of resinous substances as part of Roman mortuary rites and the spread of this practice to Britain (Pearce 2013). In the 1970s, a correlation with plaster body-coatings and interment in stone sarcophagi and/or lead lined coffins was proposed and a debate commenced over the significance of this 'package' (for example, Sparey Green 1977; Philpott 1991). Nonetheless, definitive evidence for the presence of exotic plant exudates in Roman Britain remained elusive.

Figure CS5.1
Location of the sites around Britain from which positive results were obtained by organic residue analysis for the presence of resinous substances in Roman period sarcophagus and/or lead-lined coffin burials.

Natural resins, gums and gum-resins are sticky, often highly scented, substances secreted when the bark of certain trees or shrubs are 'wounded'. Widely employed throughout human history, their characterisation in the archaeological record predominantly rests on the survival of the more degradation-resistant compounds present in the resin fraction. Fortunately, these higher molecular mass di- and triterpenoids and their derivatives are all diagnostic of botanical source and amenable to analysis by gas chromatography-mass spectrometry (GC-MS).

A project was, therefore, undertaken to identify key marker compounds in residues from 2nd–4th century AD mortuary contexts in Britain using GC-MS in order to determine the source of any resinous substances employed and to illuminate the meaning of this rite.

Materials and method
The recovery of visible resinous fragments in the archaeological record is rare, as, owing to their amorphous nature, they are easily overlooked during excavation or may become powdered and essentially invisible, even within a closed environment. Thus, a protocol was established (Figure CS5.2) that focused on the collection and analysis of multiple samples of the comminuted debris from the burial containers alongside visible fragments and residues adhering to plaster body casings or skeletal elements. Archaeological samples were then sought from Roman period mortuary contexts in Britain. The focus was on individual(s) who had been interred in stone or lead containers and/or encased in plaster, plus a number of normative inhumations evaluated as comparatives.

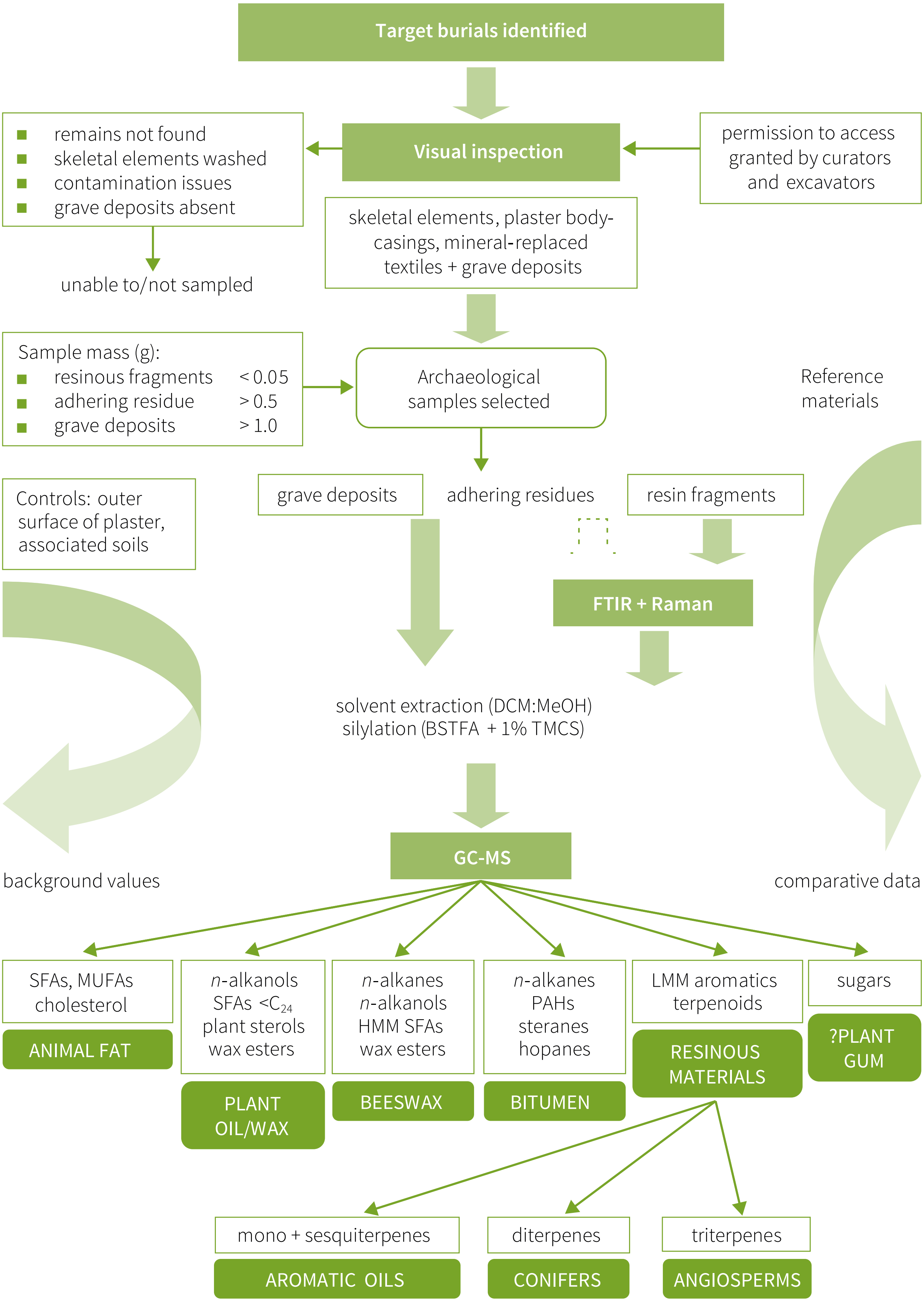


Figure CS5.2
The sampling and analytical protocol established for this project. SFA – saturated fatty acid, MUFA – monounsaturated fatty acid, PAH – polyaromatic hydrocarbon, HMM – high molecular mass, LMM – low molecular mass.

Results and discussion

Suites of terpenic compounds characteristic of resinous substances of archaeological interest were identified in 16 of the stone sarcophagus and/or lead-lined coffin burials analysed (Brettell *et al* 2014, 2015a) (Figure CS5.1). These findings showed that:

- coniferous resins from the widespread European sub-family, Pinaceae, were present in seven plaster burials from Poundbury Camp, Dorchester, Dorset (Figure CS5.3a)

- *Pistacia* spp. (mastic/terebinth) resins from the Mediterranean had accompanied an infant buried near Arrington, Cambridgeshire and an adult encased in plaster from York, North Yorkshire (Figure CS5.3b)

- two young adult females interred in elaborate lead-lined sarcophagi, one in the northern cemetery of London (the 'Spitalfields lady') and the other in a rural burial ground at Purton, Wiltshire, contained both mastic and Pinaceae resins (Figures CS5.3a and CS5.3b)

- a combination of Pinaceae and possibly *Liquidambar orientalis* (storax) had adorned the remains of a mature adult male, the founder burial at the Eagle Hotel site, Winchester, Hampshire

- *Boswellia* spp. gum-resins (frankincense) had been transported from southern Arabia or eastern Africa to be deposited with two individuals, including a child clothed in a tunic reserved for members of the elite, in a rural burial ground at Alington Avenue, Dorchester (Figure CS5.3c) and in two plaster burials from York

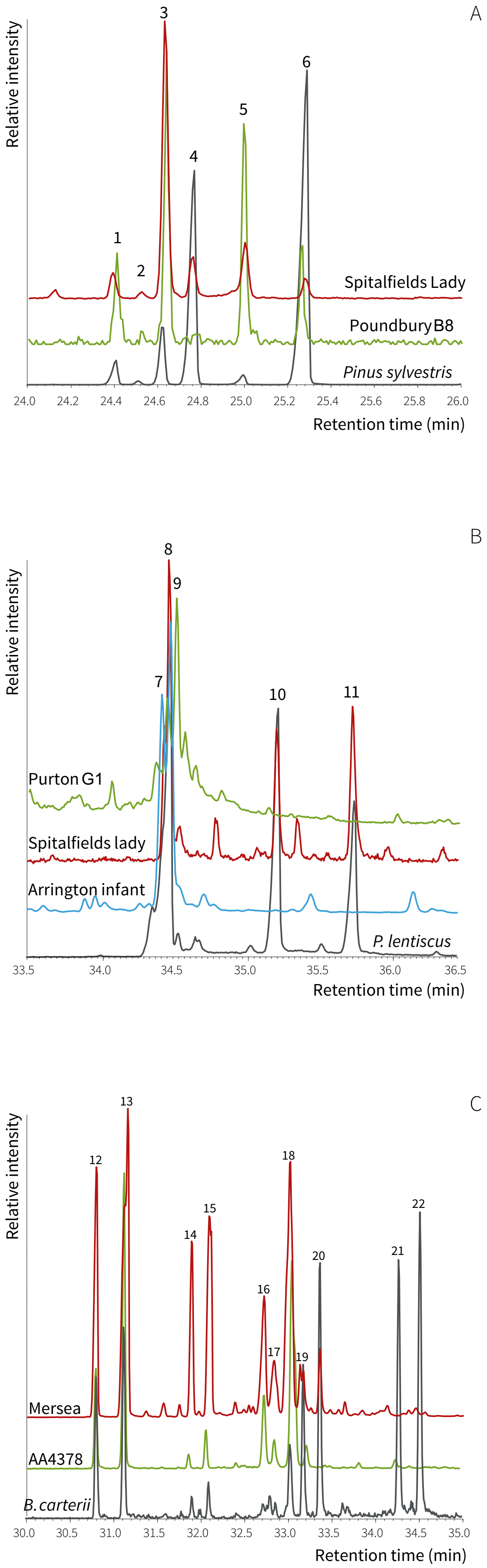
In addition, yellow-white amorphous masses discovered during re-analysis of the cremation burial at the centre of the Mersea Island barrow, Essex proved to be frankincense mixed or 'cut' with a small amount of Pinaceae resin (Figure CS5.3c). Dated to the 2nd century AD, this was a remarkable find. The considerable abundance of resinous material present (and exceptional preservation conditions within the ossuary) demonstrated that an unburnt offering had been added to the glass cinerary urn before the barrow was sealed (Brettell *et al* 2015b).

Indeed, the survival of resin acids in many of the samples from the inhumation burials indicates that these exudates had been deposited in their natural state. This would have enabled the scent of their more volatile components to mask the odour of decay and, in combination with their anti-microbial properties, have impeded decomposer organisms. Such considerations would have been particularly pressing during the extended funerary rites of the Roman elite, where conspicuous consumption of these luxury goods would have provided an additional signifier of social status (Pearce 2013).

An even more important role was, however, played out in the ritual sphere. In the Roman period, aromatic exudates were viewed as divine gifts and so were believed to purify the body and to facilitate the deceased's journey to the otherworld (Philpott 1991, 118). Their presence in mortuary contexts across the province of *Britannia* from the early–mid 2nd century AD to the mid–late 4th century AD clearly supports the considerable ritual significance of these scented substances and highlights the spread of Roman eschatology across the Empire.

Conclusion

In this case study, ORA provided direct evidence for the use of resinous exudates in mortuary rites in Roman Britain. The ramifications of this research are manifold, as they hold implications for both the social and economic integration of Britain within the Roman Empire and attest to the ritual role of resins in the mortuary sphere. These results also provide a salutary reminder of the potential for 'dirt' to retain invisible marker compounds of archaeological significance.



Peak	Compound
1	Pimaric acid
2	Sandaracopimaric acid
3	Isopimaric acid
4	Didehydroabietic acid
5	Dehydroabietic acid
6	Abietic acid
7	Moronic acid
8	Oleanonic acid
9	Oleanonic aldehyde
10	*Iso*masticadienonic acid
11	Masticadienonic acid
12	24-norolean-3, 12-diene
13	24-norursa-3, 12-diene
14	3-epi-β-amyrin
15	3-epi-α-amyrin
16	24-norursa-3, 12-dien-11-one
17	β-amyrin
18	α-amyrin
19	α-boswellic acid
20	β-boswellic acid
21	3-O-acetyl-α-boswellic acid
22	3-O-acetyl-β-boswellic acid

Figure CS5.3: Partial extracted ion current (*XIC*) chromatograms of the trimethylsilylated lipid extracts from archaeological samples and comparative modern reference materials.

A *XIC* (m/z 241) of (red) debris from the pelvic region, base of lead coffin, 'Spitalfields Lady', London
(green) residue from inner surface of plaster body-coating, stone sarcophagus, Burial 8, Poundbury Camp, Dorchester
(grey) modern reference *Pinus* spp. resin.

B *XIC* (m/z 511) of (green) debris associated with the femora, lead-lined sarcophagus, Grave 1, Purton, Wiltshire
(red) debris from the upper torso region, base of the lead coffin, 'Spitalfields Lady', London
(blue) resinous orange fragments, cranial region, infant burial in lead-lined coffin, Arrington, Cambridgeshire
(grey) modern reference *Pistacia* spp. resin.

C *XIC* (m/z 218) of (red) amorphous material from within Mersea Island cremation urn
(green) debris from base of lead coffin, Burial 4378, Alington Avenue, Dorchester
(grey) modern reference *Boswellia carterii* gum-resin.

CS6: Tracing the impact of Vikings on dietary patterns on the Outer Hebrides

L Cramp

Keywords
Bornais, Outer Hebrides, Shetland, Late Iron Age, Norse, marine biomarkers

Summary
ORA of Iron Age and Viking/Norse period vessels from the Outer Hebrides indicates a gradual shift towards an increased proportion of marine foods in the diet.

Background
This case study focuses on ORA carried out on ceramics from the site of Bornais (Bornish), on the machair plain of the western coast of South Uist, Outer Hebrides (Figure CS6.1). This multi-phase site, spanning the Middle Iron Age to the Late Norse period (200–1400 AD), covers a long sequence of occupation including a Late Iron Age wheelhouse and several substantial buildings, and a large bow-shaped hall dating to the 11th century (Sharples 1999, 2000, 2005; Sharples and Smith 2009). Ceramics, and other material culture, took on new forms over the lifespan of the site; however, it has been argued that the continued use of pottery *per se* (the Viking period is often aceramic, with soapstone used instead) and the similarity between Late Iron Age and Viking settlement patterns here indicate significant continuity of the indigenous Pictish population alongside Norse settlers. The investigation therefore aimed to test whether continuity or change in dietary patterns could be identified over this cultural transition.

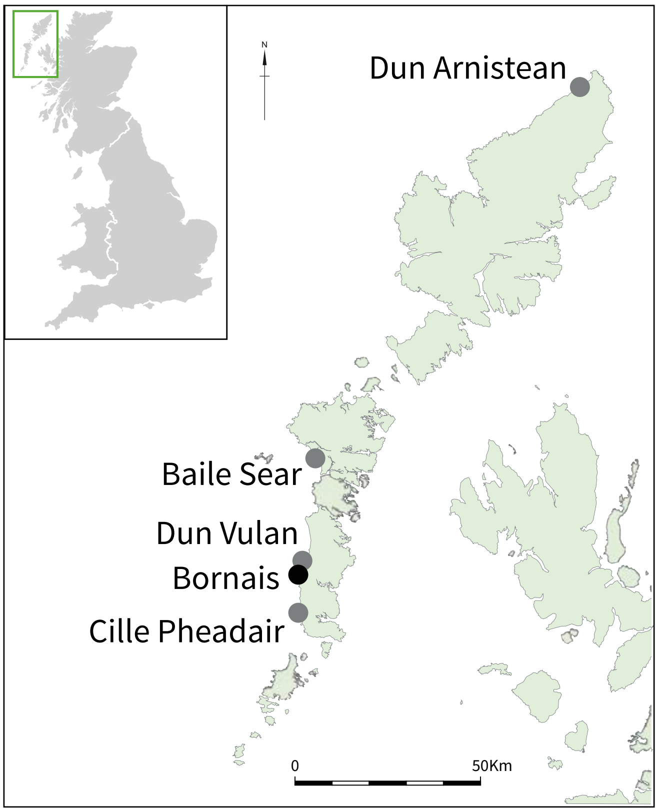


Figure CS6.1
The location of Bornais, and other nearby Iron Age and Viking/Norse sites on the Outer Hebrides.

Sampling
A diachronic investigation of 131 vessels spanning the Late Iron Age and Pictish period (5th–8th century AD) through the Norse period (10th–14th century) was undertaken to explore different dietary and cultural patterns that might have accompanied interactions of native Picts with Viking incomers. Sherds derived from the most common cooking pots, that is jars and bowls (for example, Figure CS6.2A), although 13 distinctive Middle and Late Norse 'platters' were included for comparison. These platters are thought to have been ceramic replicas of Viking steatite baking plates (Figure CS6.2B).

Results
Faunal evidence from the Outer Hebrides suggests that the advent of the Viking period on the islands is associated with increased exploitation of marine products. While the Norse platters investigated contained anomalously low quantities of lipid, which would be consistent with a use for preparing lipid-poor foods such as bread, most residues from cooking pots actually indicate processing of ruminant carcass fats – including a high proportion of dairy fats, with some evidence for processing of fish or other aquatic products alongside. The findings do not, however, strongly support a sudden increase in the importance of marine products from the Iron Age to Viking period. The biomarkers of aquatic origin, including long-chain APAAs, long-chain

vicinal dihydroxy fatty acids and isoprenoid acids, were frequently mixed with terrestrial products, suggesting that vessels were versatile or that ingredients were frequently prepared together; and with almost no evidence for any specialised fish- or blubber-processing pots. Although a broad increasing trend in marine product processing can be identified, the frequency of marine biomarkers actually drops in the Middle Norse period, so the trend is not unidirectional (Figure CS6.3).

Discussion and conclusion

When viewed alongside a comparative dataset of Iron Age and Late Norse residues from the site of Jarlshof, on Shetland, the transition to a more marine-based economy on South Uist appears gradual and somewhat sporadic. This might be explained by one of two phenomena: first, that during the Norse period on South Uist non-ceramic containers or materials were used for processing fish and other marine commodities, for example hanging fish to dry or smoking the fish on racks, or soaking fish in barrels, leaving no trace of an increase of this commodity as ceramic residues. This explanation contrasts with ceramic and soapstone vessels analysed from Jarlshof, which display extensive and intensive processing of aquatic commodities in the Late Norse material (Cramp *et al* 2015). Second, that there was possibly a higher degree of population continuity on the Outer Hebrides than on Shetland, which would explain a less-emphatic shift in dietary

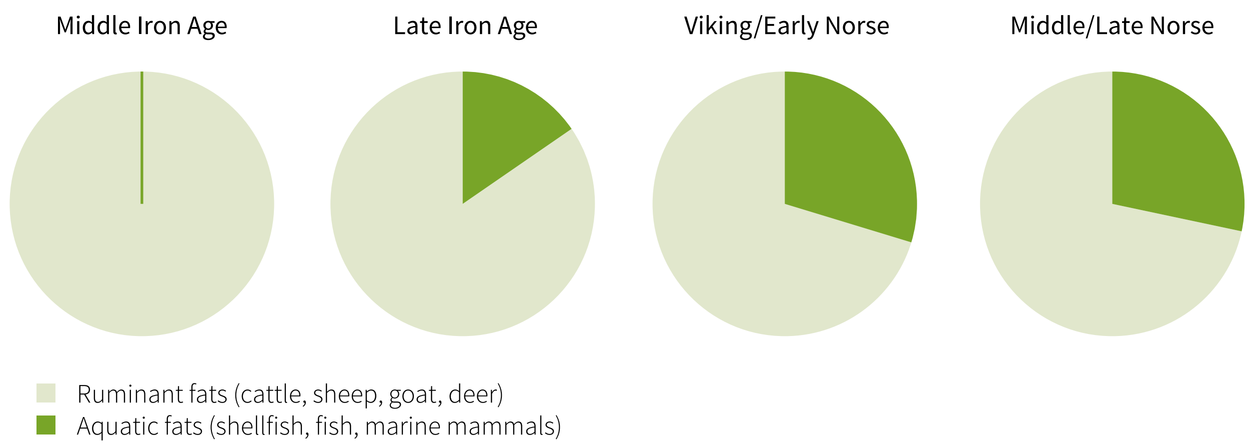
Figure CS6.2 (above): Pottery from Bornais

A. Late Iron Age I pot (5th–6th century AD)
B. Norse platter (11th–12th century AD)

Images courtesy of Niall Sharples.

Figure CS 6.3 (below): Pie charts showing the increase in aquatic residues in pottery from sites on the Outer Hebrides dating to the Iron Age and Late Norse period.

Sites include Bornais (Late Iron Age-Late Norse) as well as other nearby sites, including Dun Vulan, Baile Sear and Dun Arnistean (Middle Iron Age), and Cille Pheadair (Mid/Late Norse).



These findings can also be viewed within the wider picture of the importance of marine resources at coastal or island locations throughout British prehistory. There has been much debate about the apparent ‘abandonment’ of aquatic resources in the Neolithic period in Britain, which is supported by extensive studies of organic residues from the Neolithic and later periods. It is only at Late Iron Age and Viking sites such as Bornais and Jarlshof that we start to see a significant re-appearance of marine resources in pots, several millennia after terrestrial resources became the key source of animal protein (Cramp *et al* 2015).

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The *Supporting Information* includes further references.

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10 Glossary

α-, β-: used in organic chemistry nomenclature to specify structure

δ^{13}C: the ratio of ^{13}C:^{12}C, in parts per thousand, expressed relative to an international standard (Vienna Pee Dee Belemnite or vPDB)

Δ^{13}C: $\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$

δ^{15}N: the ratio of ^{15}N:^{14}N in parts per thousand, expressed relative to an international standard (AIR)

ω-(o-alkylphenyl) alkanoic acid class of lipid which forms when unsaturated **fatty acid**s are heated. Where these have a carbon-chain length of 20 - 22 carbon atoms, their presence can be used as an indicator for aquatic products

alkane a saturated hydrocarbon (consisting of hydrogen and carbon atoms)

archaeological biomarker substances occurring in organic residues that provide information relating to human activity in the past

biomolecules biological molecules such as lipids, proteins and DNA

carboxylic acid a chemical compound that is characterised by the –COOH functional group; carboxylic acids that have a straight or monomethyl-branched chain, with or without double bonds, are also called **fatty acids**

carcass meat from the dead body of an animal

cholesterol the principal **sterol** synthesised by animals

compound general term for a material with a specific chemical structure

diacylglycerol glyceride comprising two **fatty acids** bonded to a glycerol molecule

dairy milk or milk products such as butter, cheese and yoghurt

dihydroxy acid a **fatty acid** containing two hydroxyl groups

epicuticular wax a substance that covers the outermost layer of the plant cuticle, consisting mainly of straight-chain aliphatic hydrocarbons with a variety of substituted functional groups; it helps to decrease surface wetting and moisture loss

fatty acid a carboxylic acid with a long aliphatic tail (chain), which is either **saturated** or **unsaturated**

gas chromatograph an instrument used to separate, quantify and identify compounds by their retention time

gas chromatography-mass spectrometry an instrumental technique used to separate, quantify and identify compounds by their retention time and characteristic molecular weight and fragment pattern

gas chromatography-combustion-isotope ratio mass spectrometry an instrumental technique used to ascertain the relative ratio of the light stable **isotopes** of carbon, hydrogen, nitrogen or oxygen in individual compounds

hydrophobic the physical property of a molecule (known as a **hydrophobe**) that is seemingly repelled from a mass of water

isotope one of two or more forms of the same element (for example, carbon) with the same chemical properties but different atomic mass due to differing numbers of neutrons in the nucleus

ketone an organic compound that contains a non-terminal carbonyl group

lipid a group of organic solvent-soluble naturally occurring organic molecules (for example, originating from living organisms) that are mainly composed of carbon, hydrogen and oxygen

monoacylglycerol glyceride comprising one **fatty acid** bonded to a glycerol molecule

monounsaturated fatty acids that have one double bond in the **fatty acid** chain (with the remainder being single-bonded)

non-ruminant animals which are 'monogastric' – they have a single-compartment stomach, for example pig

organic relating to a living, or once-living entity. In chemistry, classes of compound based on carbon and usually hydrogen

phospholipid class of lipids that are a major component of all cell membranes

polyunsaturated fatty acids that have two or more double bonds in the **fatty acid** chain (with the remainder being single-bonded)

proxy a parameter that makes use of variables that can be empirically determined to estimate some other variable that cannot be measured directly

resin a hydrocarbon excretion of many plants, particularly conifers; usually a viscous liquid containing a variety of terpenes; often used in varnishes and adhesives

ruminant an animal that can acquire nutrients from plant-based food by fermenting it in a specialised stomach (with four compartments) prior to digestion for example cattle, sheep and goat.

saturated a **fatty acid** containing the maximum number of hydrogen atoms with only single bonds between the carbon atoms

stable isotope an isotope that does not undergo radioactive decay

sterol group of (mainly **unsaturated**) solid alcohols of the steroid group, such as **cholesterol** and sitosterol, present in the fatty tissues of plants and animals

triacylglycerol (triglyceride) an ester derived from glycerol and three **fatty acids**

unsaturated fatty acid in which there is at least one double bond within the **fatty acid** chain

wax ester an ester of a **fatty acid** and a fatty alcohol

11 Where to Get Advice

Within Historic England, the first point of contact for general archaeological science enquiries, including those relating to organic residue analysis, should be the HE science advisors, who can provide independent, non-commercial advice. For contact details see:

HistoricEngland.org.uk/scienceadvice

Specific advice can be sought from laboratories carrying out organic residue analysis, based at the Bradford, Bristol and York universities:

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A short video outlining the principles and chemical techniques involved in organic residue analysis and its application in archaeology has been produced by Oliver Craig at the University of York: https://www.youtube.com/watch?v=nxNMm78tvrI

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