Neolithic and Eneolithic activities inferred from organic residue analysis of pottery from Mala Triglavca, Moverna vas and Ajdovska jama, Slovenia

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ABSTRACT – *The research discussed in this paper focused on the analysis and identification of organic residues either preserved as visible or absorbed organic remains on Neolithic and Eneolithic pottery from various archaeological and geographical contexts. These are connected with various food preparation strategies and past human activities,* i.e. *cave burials in Ajdovska jama (food as a grave good/offering), the rock shelter at Mala Triglavca (meat and dairy animal husbandry practices) and Moverna vas, which had a long occupation sequence (complex farming and animal management). The preservation of biomarkers mirrored past human activities and different pottery uses at various types of sites. The carbon stable isotope ratios of primary fatty acids in lipid pottery extracts confirmed the presence of adipose and dairy fats as well as biomarkers of plant fats, beeswax and birch bark tar.*

IZVLEČEK – *Predstavljeno raziskovalno delo se je osredotočalo na analizo in identifikacijo organskih ostankov na površini neolitske in eneolitske keramike ter ostankov lipidov absorbiranih v keramično matrico vzorcev iz različnih arheoloških in geografskih kontekstov. Ti so povezani z različnimi strategijami priprave hrane in preteklimi človeškimi aktivnostmi – pokopi v Ajdovski jami (hrana kot grobni pridatek), skalni previs Mala Triglavca (mesna in mlekarska živinoreja) ter naselbina Moverna vas z dolgo stratigrafsko sekvenco (kompleksno poljedelstvo in živinoreja). Različne tipe najdišč je bilo mogoče povezati z raznolikimi dejavnostmi in raznoliko uporabo keramičnih posod prek ohranjenih biomarkerjev. Analiza razmerja stabilnih izotopov glavnih maščobnih kislin v keramičnih ekstraktih je potrdila prisotnost mesnih in mlečnih maščob glavnih domestikatov kakor tudi navzočnost lipidnih biomarkerjev rastlinskega izvora, ostanke čebeljega voska in smole.*

KEY WORDS – *Neolithic; Eneolithic; lipid residue analysis; pottery; stable isotopes; birch bark tar; beeswax*

Introduction

Archaeological research has benefited greatly in the past twenty years from an exponential increase in interdisciplinary studies incorporating analytical sciences. Two major fields of archaeological investigation, predominantly in prehistoric periods, have been trying to understand past diets and the mobility of populations by analysing osteological material and ceramics. The porous surface of these two commonly found archaeological artefacts enables organic molecules such as lipids, proteins and nucleic acids to become entrapped and preserved through millennia.

Unglazed pottery has proved to be an ideal analytical medium: on the one hand, it readily absorbs organic compounds during cooking, food storage and consumption, while it also serves as an indicator of past lifestyles, kinship, animal husbandry practices,

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DOI:10.4312/dp.41.9

agriculture, trade or ritual practices (*Boast 2002; Gibson 2002*). Although organic molecules are prone to degradation processes during pottery use or during the post-depositional period, it has been found that adequate concentrations of lipids can be preserved either as absorbed residues or visible food crusts and retrieved through organic solvent extraction (*Heron, Evershed 1993; Evershed 2008; Craig 2004; Saul* et al. *2013*).

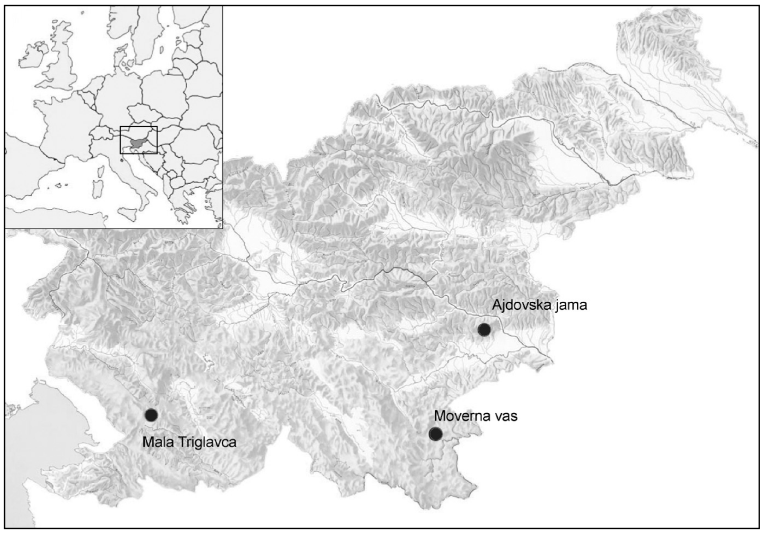


Fig. 1. A map of Slovenia showing the locations of archaeological sites investigated (adapted from National Museum of Slovenia).

Sites selection

Among the various Slovenian Neolithic and Eneolithic sites available, three were chosen for lipids analyses (Fig. 1). The two with the longest settlement sequence, *i.e.* Mala Triglavca and Moverna vas, are embedded in different environmental and cultural contexts. The third, Ajdovska jama, is a burial site with strong evidence of burial ceremonies and rituals.

The Mala Triglavca rock shelter is located on the Dinaric Karst in south-western Slovenia, 15km from the Northern Adriatic coast. The AMS ^{14}C dates show a long sequence of human activities from the 8th to the 3rd millennium calBC, combined with natural and geomorphological post-depositional disturbances. The Moverna vas open-air site is situated in the karstified Bela Krajina region in the south-eastern part of Slovenia. The settlement sequence spans approximately two millennia from the 5th to the 3rd millennium calBC. The Ajdovska jama cave site lies within the catchment of the Sava River in south-eastern Slovenia. The site is well known for its burials. The human remains at the site occurred as distinct clusters of mainly disarticulated bones belonging to at least 31 individuals. The cave was used for burial and related ritual practices in the late 5th and early 4th millennium calBC.

Moverna vas

The Neolithic and Eneolithic settlement sequence at Moverna vas consists of nine settlement phases. Phases 2 to 6 were recognised as Neolithic, and phases 7 to 9 as Eneolithic (*Budja 1989; 1994*). Bayesian modelling (Fig. 2) shows that the sequence spans approximately two millennia, with continuous occupation from 4945–4810 calBC to 4270–4135 calBC and discontinuous occupation until 2905–2800 calBC (at 68.2% probability) with possibly centuries-long breaks in occupation (*Budja 1994; Sraka 2013*). The chronology is largely based on AMS ^{14}C dates from carbonised organic residues adhering to interior pottery surfaces. Chemical analyses of these residues show that they are either charred remains of food

Sample Name	Material	Context	Phase	Lab code	^{14}C Conventional age (BP)	Calibrated age acc. to 68.2% prob. (calBC)	Calibrated age acc. to 95.4% prob. (calBC)	$\Delta^{13}C$ (meas. on AMS)	Reference
23MV	food crust	053.1	3	Poz-21396	5750±40	4450–4350	4460–4335	–24.9±0.5	Sraka 2013.App.
24MV	birch bark tar	050.2	4	Poz-21398	5550±40	4540–4455	4615–4370	–20.6±0.3	Sraka 2013.App.
25MV	birch bark tar	050.1	4	Poz-21399	5630±40	4715–4605	4770–4540	–24.2±0.2	Sraka 2013.App.
26MV	birch bark tar	022.1	5	Poz-21400	5610±40	4940–4805	4995–4785	–24.2±0.6	Sraka 2013.App.
27MV	birch bark tar	050.1	4	Poz-21401	5620±40	4495–4370	4530–4360	–20±0.4	Sraka 2013.App.
28MV	birch bark tar	050.2/056	2	Poz-21402	5990±40	4490–4365	4520–4355	–22.3±0.7	Sraka 2013.App.
29MV	food crust	planum 7	2	Poz-21403	5800±40	4505–4370	4540–4365	–21.6±1.8	Sraka 2013.App.
151MV	birch bark tar	031.4	6	Poz-21404	5670±40	4450–4350	4460–4335	–19.1±0.5	Sraka 2013.App.
152MV	birch bark tar	050.2	4	Poz-21420	5550±40	4680–4545	4705–4500	–22.9±0.5	Sraka 2013.App.

Tab. 1. ^{14}C dates obtained from organic remains on pottery for Moverna vas (see also Fig. 10).

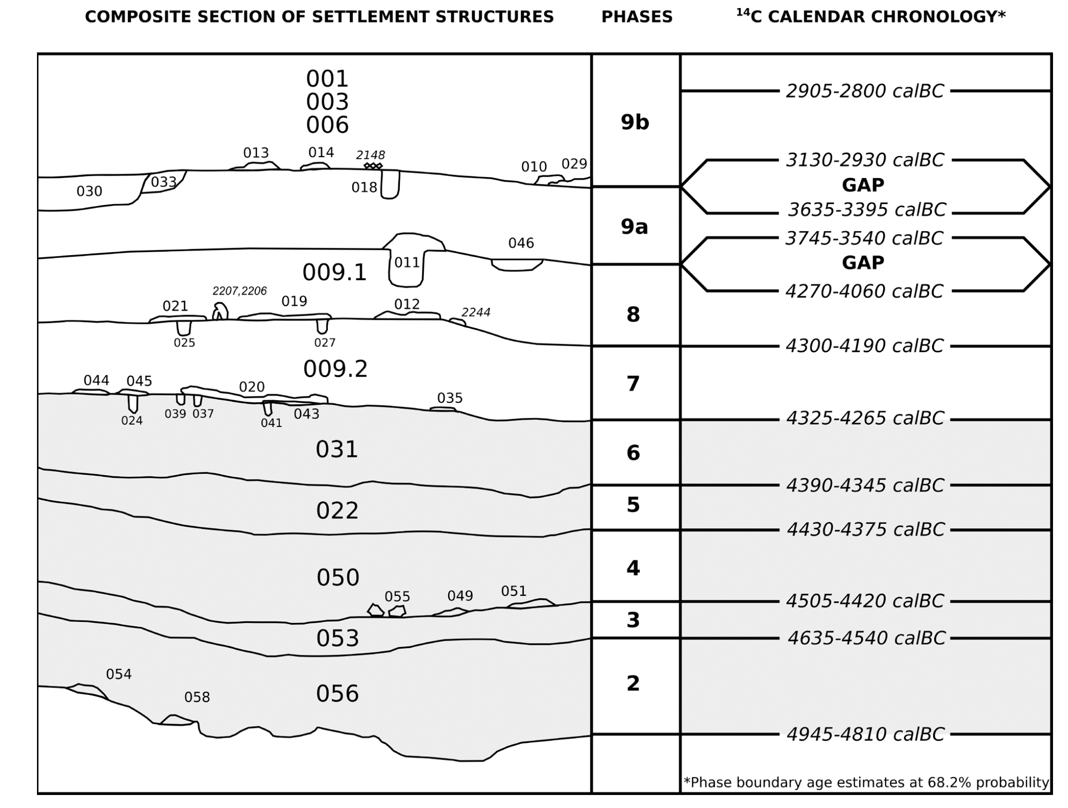


Fig. 2. Moverna vas stratigraphical sequence with Bayesian statistical modelling of radiocarbon dates.

(23MV, 29MV; App. 1; Tab. 1) or birch bark tar (24MV-28MV, 151MV, 152MV; App. 2; Tab. 1; Fig. 10). According to the results of chemical analyses, no freshwater reservoir effect is to be expected for these dates. Both the food crust and birch bark tar samples are considered as reliable samples with minimal inbuilt age (*Hedges* et al. *1992; Oinonen* et al. *2010*).

During the continuous occupation in the 5th millennium, two related changes in 4325–4265 calBC (at 68.2% probability) have been observed. While the transition from Neolithic to Eneolithic vessel types and pottery fabrics was observed in the pottery assemblages (*Tomaž 1997*), the changes in settlement pattern relate to settlement fragmentation and settlement extension within the site-catchment areas, as well as in previously uninhabited areas (*Budja 1995*).

The pottery samples selected for lipid analysis were embedded in Neolithic settlement phases 2 to 6 (*c.* 4945–4265 calBC) (Figs. 2, 4). The ceramic vessels of these settlement phases include various types of pot (Fig. 3.type 4, 5, 6, 8, 9), dishes with spouts (Fig. 3.type 1), pedestal dishes (Fig. 3.type 7), small pots (Fig. 3.type 3), bowls (Fig. 3.type 2), and ladles (Fig. 3.type 10). Most of the pottery was fired in an oxidising atmosphere and well made, with burnished surfaces and red or brown slips applied to the surface. The vessels were made with homogenous clay fabrics and abundant quartz grain inclusions, which in some samples can even be interpreted as added temper (*Tomaž 1997; 1999; Žibrat Gašparič 2008*).

Mala Triglavca

The Neolithic and Eneolithic sequence at the Mala Triglavca rock shelter consists of 23 occupational levels, ranging from *c.* 5600 to 3500 calBC. The lipid analyses of the pottery assemblage, which is comprised mainly of various types of bowls, beakers, dishes and pots have been published (*Šoberl* et al. *2008; Budja* et al. *2013*) and will be used here mainly in relation to other sites in the discussion. The pottery samples were taken from Neolithic occupational levels and can be linked according to their morphology and technology to the Vlaška pottery group (*Barfield 1972; Žibrat Gašparič 2004*). The oldest pottery fragments appear as early as 5616–5525 calBC. For the lipid analysis, we sampled 65 vessels from contexts that range from 5480 to 4261 calBC (68.2% probability). The results indicate an extensive mixing of ruminant and non-ruminant, and ruminant adipose and ruminant dairy fats in individual vessels. In some vessels, the presence of molluscs, crustaceans and freshwater fish was detected. Thirty per cent of the sampled pottery contained lipids characteristic of dairy fats, indicating

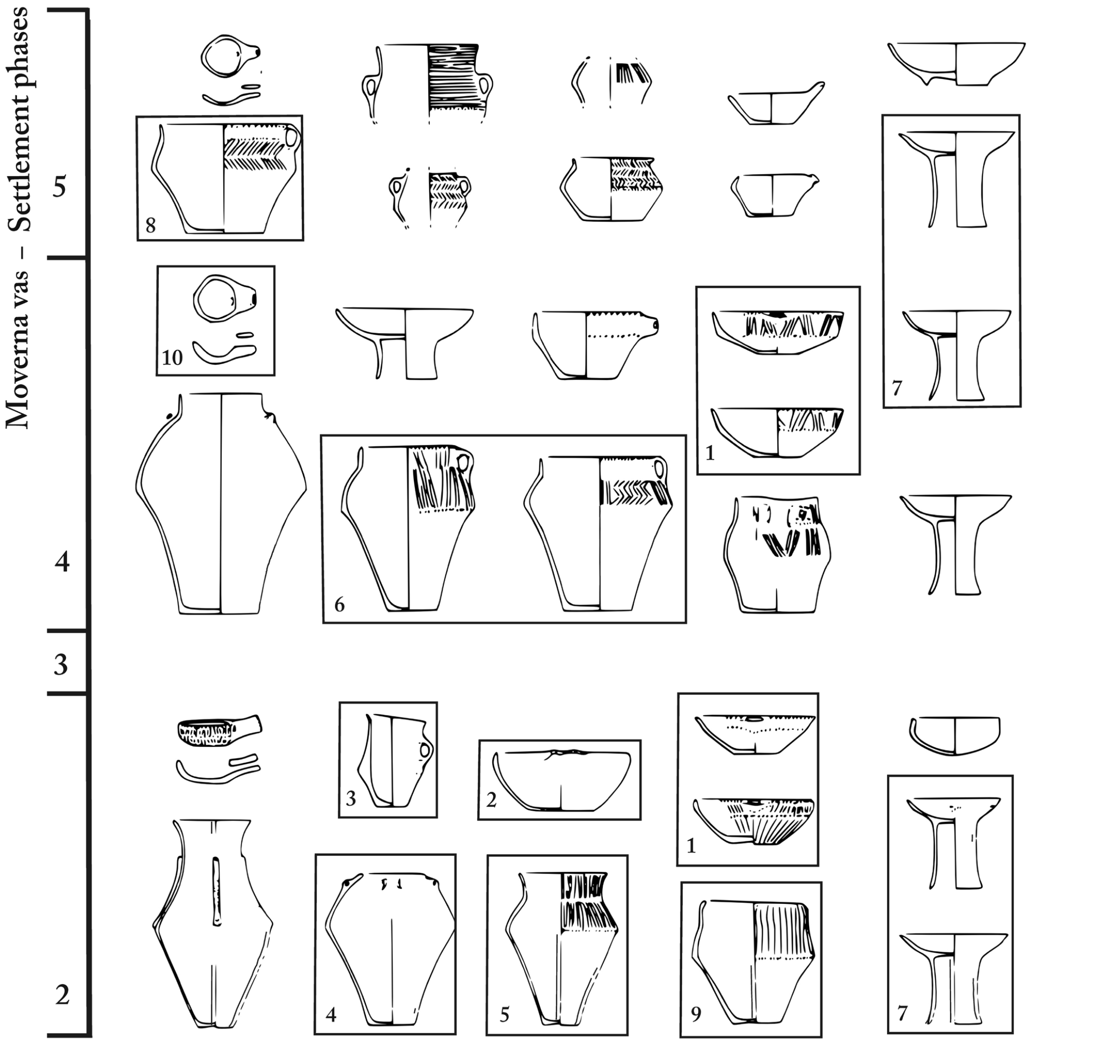


Fig. 3. Moverna vas pottery types represented within the different settlement phases. Types 1–10 were analysed for organic residues. See Appendix 1.

that the processing of dairy products in pottery vessels was quite extensive. The use of dairy products at Mala Triglavca is embedded in the time span between 5467–5227 calBC (for details see *Budja* et al. *2013*).

Ajdovska jama

Excavations in Ajdovska jama proved that the cave was an eminent site at the end of the Neolithic period, with traces of temporary human activity until the High Middle Ages. Ajdovska jama is the funerary site with the oldest excavated burials in Slovenia, and a place where the remains of the dead were worshipped. The cave is also a natural karstic phenomenon, which might have had a symbolic meaning for prehistoric people.

The most typical grave goods of individual groups of burials that were excavated in the central hall and the left corridor included pottery (*i.e.* pot, dish, jug and ladle), jewellery (*i.e.* necklace or bracelet), and tools or weapons (*i.e.* axe, awl). The grave goods were found alongside the bodies of the deceased and prove that rituals were performed at the time of subsequent burials and visits to the cave. Food and meat were also placed beside the bodies as offerings. Analyses of plant and animal remains from the burials showed that cereals (*e.g.*, wheat, barley and a type of bean) and the meat of domestic animals (*i.e.* ovicaprids, cattle) as well as wild animals (*i.e.* rabbit, wild boar, red deer, fox) were cooked (*Horvat 1989*). Additional information on diet came from the analyses of carbon (^{13}C) and nitrogen (^{15}N) isotopes in collagen obtained from human and animal bones. The results show that the people of this community consumed mostly meat and plant protein coming from C_3 dietary source (*Ogrinc, Budja 2005; Bonsall* et al. *2007*). Bayesian chronological modelling of ^{14}C dates provided by Clive Bonsall *et al.* (*2007*) shows that the burials belong to a relatively

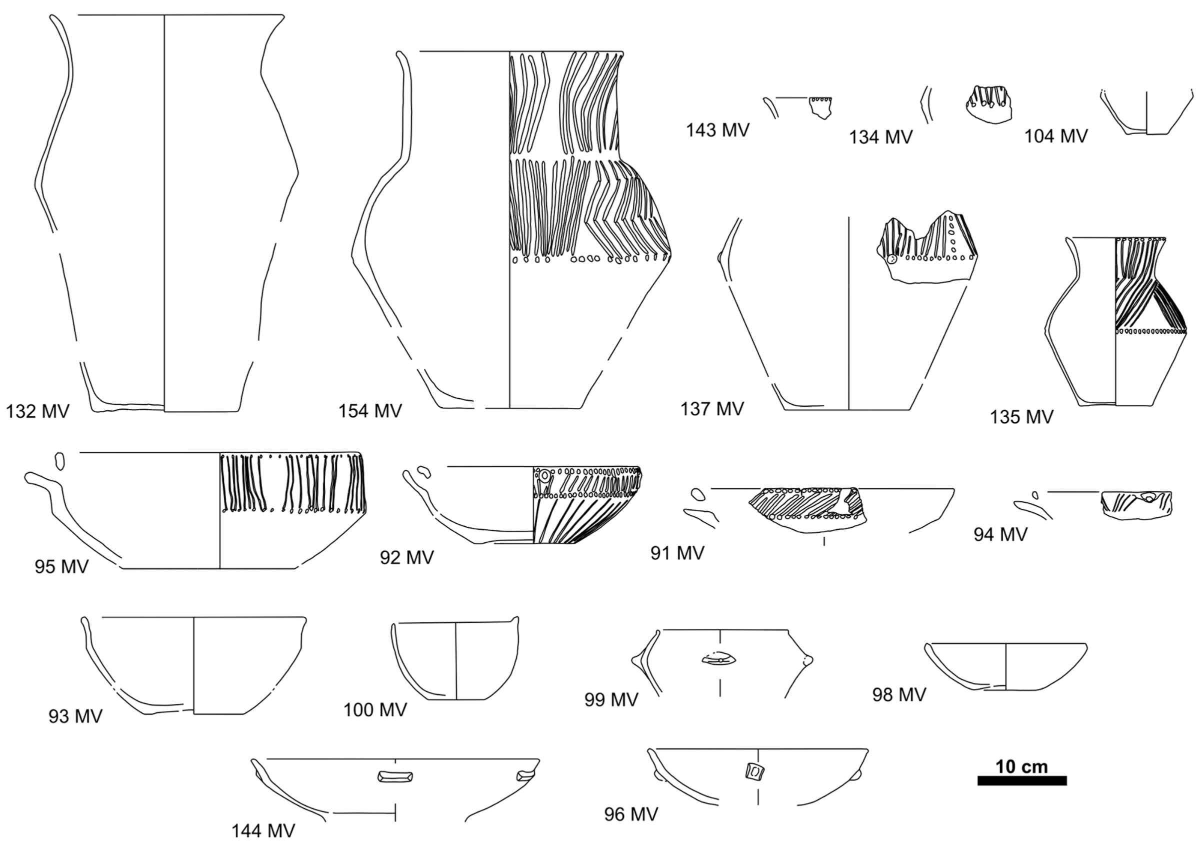


Fig. 4. Vessels from Moverna vas that were analysed for organic residues.

short time interval of a few human generations from 4340–4290 to 4295–4235 calBC (at 68.2% probability).

A collection of 52 pottery samples was selected for lipid residue analysis, including various types of pots, dishes, dishes with spouts, pedestalled vessels, bowls and jugs (Fig. 5). Most of the vessels were fired in an oxidising atmosphere and were made with various fine-grained quartz fabrics (*Horvat 1989; Osterc 1986*).

Organic residue analysis

Cooking and processing organic commodities enables insoluble lipid residues to be absorbed into porous ceramic matrix and preserved for several thousand of years in the form of surface or/and absorbed residues. Cooking vessels have proved to be the most convenient for analysing organic residues due to their constant everyday use and exposure to high temperatures during cooking. However, non-culinary related vessel use can also absorb lipids when fatty commodities are stored: from the use of various sealants to reduce the permeability of the unglazed ceramic surface (resin, tar, pitch, milk and beeswax) and from the use of adhesives to repair broken vessels (*Charters* et al. *1993a; Regert, Rolando 2002; Regert 2004*).

When determining the functionality of pottery, numerous archaeological methods can be employed, including written and pictorial evidence, the use of archaeological contexts, information obtained via ethnographical comparisons, pollen analysis of visible organic remains, use wear analysis and the analysis of preserved contents (*Orton* et al. *1993; Rice 1987; Tite 2008; Skibo, Feinman 1999*). Prudence Rice (*1987*) divided the principle functions of pottery into three categories: (i) storing dry substances; (ii) carrying liquids; and (iii) heating the contents over fire. Direct evidence for storage vessels apart from their larger size is not readily available, cooking pots offer some additional indices, *i.e.* carbonised visible remains adhering to outer or inner pot surfaces and signs of sooting.

Chemistry offers interpretative tools for use in cases with a complete absence or selective preservation of organic and biological remains, such as osteological assemblages or plant remains; or for activities that are archaeologically difficult to trace, such as beekeeping. The primary focus of lipid research in the past 20 years has been to identify various biomar-

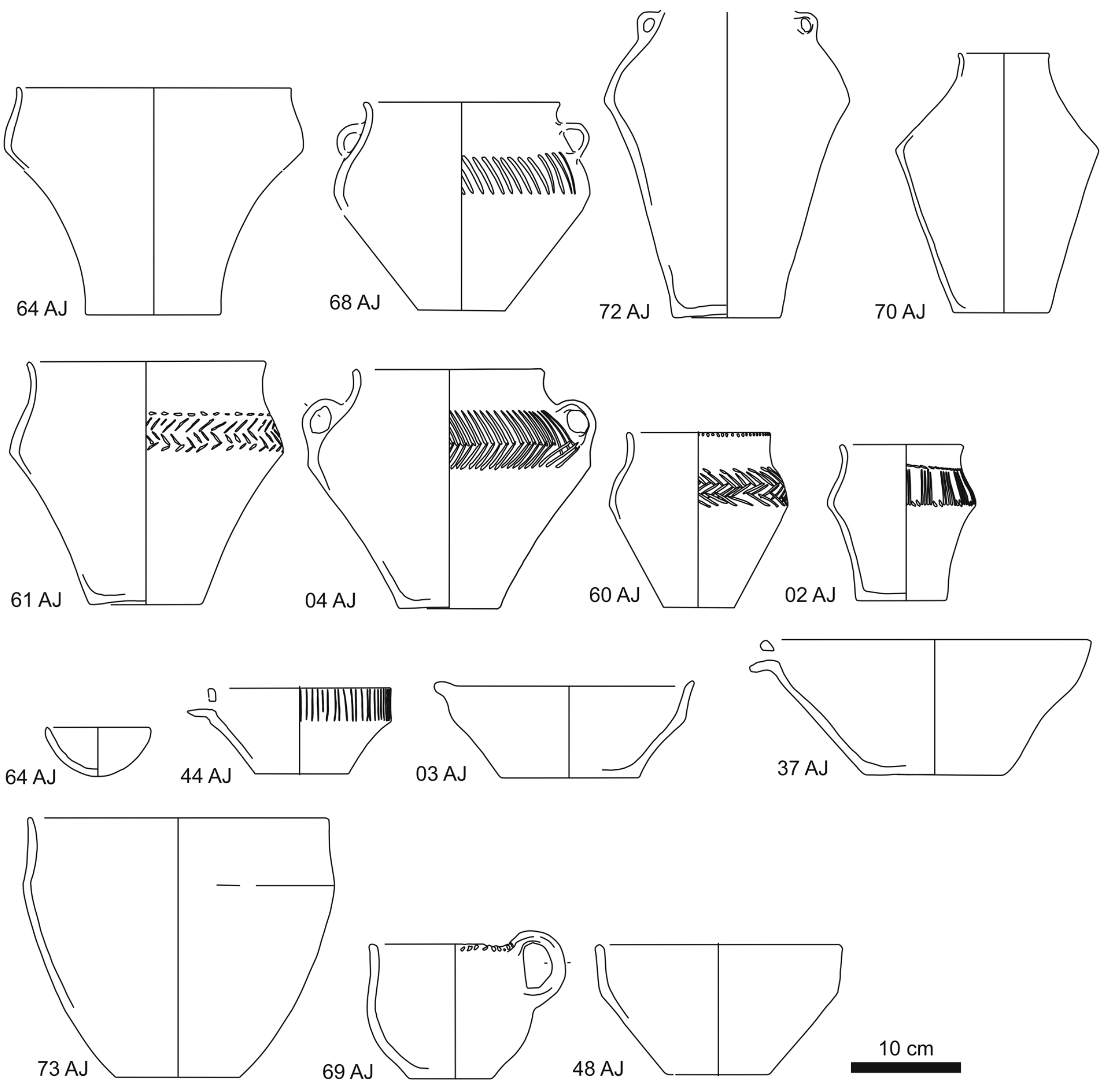


Fig. 5. Vessels from Ajdovska jama that were analysed for organic residues.

kers connected to specific domestic activities and ancient diets, and above all, identifying landmark transitions in ancient economies. These transitions include examples such as horse domestication (*Outram* et al. *2009*), the earliest dairying practices in Europe, the Near East and Africa (*Copley* et al. *2005c; Craig* et al. *2005; Evershed* et al. *2008b; Dunne* et al. *2012; Salque* et al. *2013; Cramp* et al. *2014*); the chronologically and typologically diverse importance of pig exploitation (*Mukherjee* et al. *2007*) and the evidence of geographical dependence on marine food sources (*Copley* et al. *2004b; Hansel* et al. *2004; Evershed* et al. *2008d; Craig* et al. *2011; Cramp* et al. *2014; Cramp, Evershed 2014*).

The components of the lipid extracts of such residues can be identified and quantified by solvent extraction and a combination of analytical techniques that can achieve molecular level resolution, *i.e.* high temperature-gas chromatography (HTGC), GC/mass spectrometry (GC/MS; *Evershed* et al. *1990*) and GC-combustion-isotope ratio MS (GC-C-IRMS; *Evershed* et al. *1994; 1999*).

Furthermore, modern cooking experiments have helped to understand the accumulation of lipids caused by different cooking practices, vessel use and preservation. If we take into consideration that an average concentration of preserved lipids in archaeological pottery is around 100μg g^{-1}, it is quite clear that only 1% or less of the original concentration survives post-depositional degradation (*Evershed 2008*). The initial lipid absorption also depends on the lipid content of processed food (animal *vs.* plant products) and modes of food preparation or storage. Variations in long-term lipid preservation can also occur due to differences in fabric types. Animal fats are by far the most common class of residue identified from archaeological pottery with compound-specific stable carbon isotope analysis, allowing the identification of different animal fats, *e.g.*, ruminant and non-ruminant adipose fats and dairy fats (*Dudd, Evershed 1998*), as well as the identification of the mixing of commodities (*Charters* et al. *1995; Evershed* et al. *1999*).

Materials and methods

Lipid analyses were performed with established protocols that are described in detail in earlier publications (*Evershed* et al. *1990; Charters* et al. *1993b*). The identification of individual compounds was based upon eluting order, a comparison of retention times to standards and by comparing the mass spectra with known fragmentation patters and NIST spectra library. In summary, after cleaning the potsherd surface, a 2g fragment was ground to a fine powder and extracted using a mixture of chloroform and methanol (2:1 v/v). An aliquot of the obtained total lipid extract (TLE) was trimethylsilylated and analysed directly by HTGC. Structure elucidation and molecular identification was achieved by GC-MS and HTGC-MS analyses. Fatty acid methyl esters were prepared by saponification of a TLE aliquot with BF_3/methanol to enable compound-specific stable isotopic determination by GC-C-IRMS. The addition of an internal standard of known concentration (n-tetratriacontane, 1mg mL^{-1}) enabled the calculation of extracted lipid concentration. The discussion of recovery rates refers to the proportional number of pottery extracts with an appreciable preserved lipid concentration (>5μg g^{-1}), which was determined as the lowest acceptable lipid concentration that can be reliably attributed to and interpreted as remnants of ancient food processing rather than modern contamination (*Evershed* et al. *1999; Evershed 2008a*).

Visible surface residues were scraped from the ceramic surface with a clean scalpel, ground to a fine powder and extracted as described above; again an internal standard was added for lipid quantification.

A total of 179 potsherds and visible residues were selected for organic residue analysis: 52 samples from Ajdovska jama, 36 samples from Mala Triglavca and 91 samples from Moverna vas site. Potsherds were selected to represent different occupational phases and human activities at each site. Visible residues were sampled and analysed separately. To avoid duplication, where visible residues were present they were labelled with the number 1, while the originating potsherd extract was labelled with the number 2.

Results

Neolithic and Eneolithic pottery from Ajdovska jama, Mala Triglavca and Moverna vas showed a very good lipid preservation, with an overall 53.6% of potsherds analysed yielding an appreciable lipid concentration. The preservation of lipids in pottery is heavily influenced by the alterations that may occur during vessel use or due to post-burial conditions in the soil, as well as the use of ceramic vessels during their lifetime (*Evershed* et al. *1999; Evershed 2008a*). These factors could explain the variations observed in average lipid concentrations and recovery rates between sites: the pottery assemblage from Mala Triglavca yielded the least TLE extracts, with appreciable lipid concentrations (30.6%), followed by 48% of pottery from Ajdovska jama yielding lipids, while 65.9% of the analysed pottery from the Moverna vas site showed preserved organic residues. This trend is also repeated in observed median lipid concentrations from potsherd extracts as well as lipid concentration ranges. For a better demonstration of different concentration ranges, these are plotted as box-and-whiskers plots in Figure 5. Since, generally, only lipid concentrations higher than 5μg g^{-1} are considered as appreciable and can therefore be interpreted as archaeological, lipid concentrations below this threshold were ignored in the following comparisons (see Fig. 6 and Tab. 2).

The observed lipid concentration ranges are considerably narrower in the ceramic assemblages of Mala Triglavca and Ajdovska jama than those from Moverna vas. Since the latter site was a fully developed, permanent settlement, in contrast to the occasionally used rock shelter and burial cave, higher lipid concentrations could indicate frequent daily use of ceramic vessels resulting in an accumulation of residues.

The organic residues in the investigated pottery showed compound distributions typical of animal fats and plant material degraded to various degrees. The parent triacylgycerols (TAGs) present in fresh adipose fats and plant oils quickly degrade into their constituent fatty acids, with the palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acids persisting in highest abundance, and with minor contributions from shorter chain saturated fatty acid components. Many vessels yielded only free fatty acids, indicating that complete hydrolysis of the precursor TAG components had taken place. Two gas chromatograms representing differing degrees of degradation and compounds most commonly identified in the Neolithic and Eneolithic residues analysed can be seen in Figure 7.

Fatty acids are usually present in the greatest abundance in archaeological lipid extracts with even rather than odd carbon number preference, dominated by palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acids. While animal fats generally display a greater abundance of stearic acid, the plant derived lipids show a predominance of palmitic acid (*Dudd 1999; Copley* et al. *2005a; Romanus* et al. *2007*). The presence of odd carbon number free fatty acids (*e.g.*, $C_{15:0}$, $C_{17:0}$, $C_{19:0}$) together with their iso- and anteiso-branched variations may indicate ruminant animal sources, as these compounds are biosynthesised by the bacteria living in the rumen (*Mottram* et al. *1999; Evershed* et al. *2002*).

Despite their predominance, the $C_{16:0}$ and $C_{18:0}$ fatty acids possess only limited biomarker potential. Broad groups of commodities can be alluded to only by investigating the $C_{16:0}$ *vs*. $C_{18:0}$ fatty acid ratio (P/S ratio). Previous investigations of P/S ratios in modern reference materials have provided some additional proxies; however, interpretations of these have to be applied with great caution and only in combination with other data, *i.e.* TAG distributions and $\delta^{13}C$ values. Calculated P/S ratios for pottery extracts from investigated sites are shown in Figure 8.

Previous studies have reported a P/S ratio <1.3 as indicative of ruminant adipose fats; a P/S ratio of approx. 2.2–2.9 and 4.9 indicative of dairy fats or non-ruminant adipose fats, while a P/S ratio between 4.0 and 9.4 has been reported for commercial olive oils (*Dudd 1999; Copley* et al. *2005a; Romanus* et al. *2007*). P/S ratios calculated for TLEs of Slovenian pottery (Fig. 8) show a large proportion of 53 lipid extracts (68%) falling below the 1.3 mark, indicative of ruminant adipose fat; 24 extracts (31%) displaying the range 1.3–4.0, attributed to either ruminant dairy or non-ruminant adipose fats; only 1 extract (1%) displays a P/S ratio higher than 4.0, indicative of olive/plant oils.

Labels	Ajdovska jama	Mala Triglavca	Moverna vas
Min	5.02	5.53	5.25
Q1	8.26	10.81	18.03
Median	32.45	21.93	118.18
Q3	79.25	65.95	503.45
Max	557.13	173.35	3308.10
IQR	70.99	55.14	485.42
Upper Outliers	3	1	7

Tab. 2. Details of absorbed lipid concentration ranges in analysed pottery.

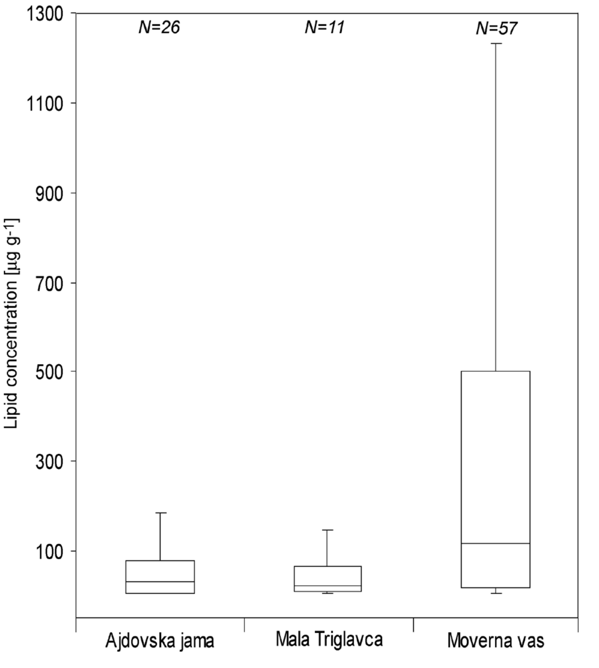


Fig. 6. Box-and-whisker plots showing the range of preserved appreciable lipid concentrations in pottery from Ajdovska jama, Mala Triglavca and Moverna vas. Only concentrations higher than >5µg g⁻¹ were used.

The presence of ruminant-derived lipids has also been confirmed by observed distributions of odd carbon number saturated fatty acids with their branched iso- and anteiso- homologues ($C_{15:0}$, $C_{15:0br}$, $C_{17:0}$, $C_{17:0br}$), biosynthesised by the bacteria living in the rumen (*Dudd* et al. *1998; Mottram* et al. *1999; Vlaemnick* et al. *2006*). These branched fatty acid biomarkers were found in ten potsherd extracts: 08MT, 18MT, 75MT, 87MT, 23MV-2, 98MV, 134MV, 149MV, 153MV and 154MV.

Apart from $C_{16:0}$ and $C_{18:0}$ fatty acids, a series of saturated long-chain fatty acids (LCFA) with a carbon number range between C_{20} and C_{30} has also been identified in fifteen potsherd extracts, representing 8% of the total assemblage: 03AJ, 37AJ, 69AJ, 70AJ, 18MT, 29MV-2, 91MV, 99MV, 102MV, 111MV, 114MV, 121MV, 147MV, 149MV AND 154MV. Such a series of LCFA has previously been associated with two potential sources, depending on the accompanying compounds. If found in combination with isoprenoid fatty acids such as phytanic or pristanic acid, 4,8,12-trimethyltridecanoic acid or ω-(*o*-alkylphenyl)-alkanoic acids, preserved lipids are most likely derived from marine organisms (*Copley* et al. *2004b;*

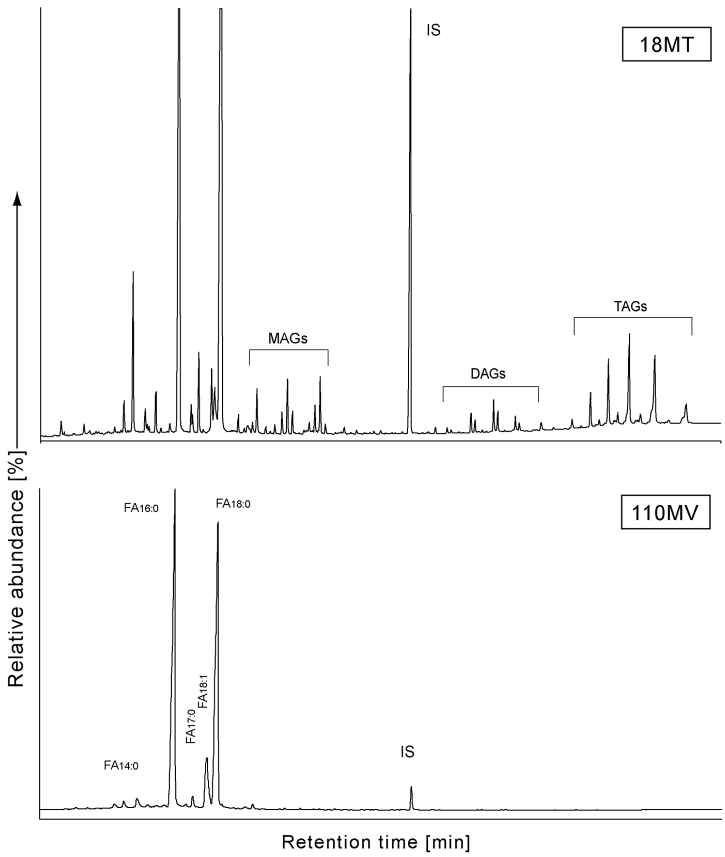


Fig. 7. Partial HTGC profile of the trimethylsilylated total lipid extract from potsherds 18MT (Mala Triglavca) and 110MV (Moverna vas) illustrating the contrasting distribution of compounds characteristic of partially and fully degraded animal fat. Key: FAX:Y are saturated free fatty acids of carbon length x and degree of unsaturation y. IS is the added internal standard; MAGs are monoacylglycerols; DAGs are diacylglycerols; TAGs are triacylglycerols.

Hansel et al. *2004; Evershed* et al. *2008c*). LCFA extracted from Slovenian Neolithic and Eneolithic pottery, however, occur in high frequency together with long-chain n-alcohols of C_{22} to C_{32} even-carbon number series and analogous odd-carbon number n-alkane series of C_{23} to C_{33} chain length, which are more characteristic of degraded plant waxes (*Tulloch 1976; Bianchi 1995*).

Mid-chain ketones have been identified in six residues derived from five body sherds and one rim sherd (02AJ, 04AJ, 72AJ, 29MV-2, 91MV, and 133MV), most frequently displaying a narrow distribution with C_{29}, C_{31}, C_{33} and C_{35} homologues. These biomarker compounds are known to form by the condensation of fatty acids, involving decarboxylation and dehydration reactions occurring at high temperatures, typically in excess of 300°C (*Evershed* et al. *1995; Raven* et al. *1997*). The carbon chain length of ketones previously found in pottery extracts usually ranges between C_{27} and C_{35}, which reflects the length of the precursor fatty acids. These compounds have also been reported as components of the epicuticular leaf waxes of higher plants (*Tulloch 1976; Kolattukudy* et al. *1976*). However, the presence of unsaturated ketones was identified in two lipid extracts (02AJ and 72AJ), suggesting the unsaturated fatty acid precursors common in plant oils. A similar series of ketones was also reported to be formed during vigorous pyrolysis at temperatures reaching 800°C (*Raven* et al. *1997*).

Waxes

Beeswax recovered from archaeological contexts can undergo various degrees of alteration; however, four major groups of compounds provide biomarkers for its presence: (i) long-chain alcohols (C_{24} to C_{32}); (ii) odd-carbon number n-alkanes (C_{25} to C_{33}); (iii) a series of palmitic wax esters (C_{40} to C_{54}); and (iv) hydroxy palmitic wax esters (C_{42} to C_{54}). Similarly, plant waxes contain a mixture of compounds, including odd-carbon number n-alkanes (C_{21} to C_{37}), monoesters ranging in chain length from C_{32} to C_{64} and long-chain alcohols with a chain length range between C_{22} to C_{34} (*Tulloch 1976; Heron* et al. *1994; Mills, White 1994; Charters* et al. *1995; Regert* et al. *2001*). A relatively large proportion of preserved lipid residues (27%) showed traces of wax esters with chain lengths of C_{40} to C_{48}, together with even-carbon number long chain alcohols (C_{22}–C_{32}) and odd-carbon number straight chain alkanes (C_{23}–A_{33}), which could either derive from diagenetically altered beeswax or degraded epicuticular plant waxes (Fig. 9).

Wax esters in these potsherd extracts were predominantly found together with free fatty acids and their acylglycerol moities, suggesting that the vessels were used to process both leafy plants and animal products; whether they were processed simultaneously or separately cannot be elucidated from this data. Interestingly, a beeswax residue was identified in cup extract 125MV without any contributions from animal fats or plant waxes. A further seven potsherd extracts from three vessels (25MV, 26MV and 28MV) contained birch-bark tar biomarkers in conjunction with wax esters, indicating mixing of commodities.

Recent experimental work reported by Dana Millson (*2011*) and Merryn Dineley (*2000; 2011*) has addressed the question of applying beeswax as a sealant, concluding that, although it is an effective waterproofing agent, it is not appropriate for use on cooking pots, causing the pot fabric to spall and flake off. Based on this, as honey would have been the earliest available natural sweetener, the beeswax residues identified in archaeological pottery could be interpreted as the remains of food processing that involved the addition of honey.

Birch bark tar

An unusual set of triterpenoid compounds was identified in 16 potsherd extracts from Moverna vas, representing seven vessels (24MV, 25MV, 26MV, 27MV, 28MV, 151MV and 152 MV) which had visible residues present on either the interior or exterior surface (Fig. 10). Visible residues are a common find on archaeological pottery, and routinely used for radiocarbon dating. It has been previously assumed/assessed that the exposed nature and structure of visible residues are usually not a good medium for preserving organic molecules (*Evershed* et al. *1992; Evershed 2008*). It was possible to assess this variation, because visible residues were sampled and extracted separately. Lipid concentration values differ quite significantly, with values for visible residues averaging at 1537.04µg g^{-1}, while absorbed residues displayed an average lipid concentration of 25.5µg g^{-1}. The two sets of residues were also differentiated by the biomarkers extracted: visible residues showed the presence of lupa-2,20(29)-dien-28-ol, allobetul-2-ene, lupenone, lupeol, betulone and betulin, which are characteristic of birch-bark tar. While betulin and lupeol are the predominant biomarkers present in birch-bark tar, other compounds are formed by degradation reactions, particularly the heating processes needed to produce the pitch. In particular, betulin is partly transformed into lupa-2,20(29)-dien-28-ol by dehydration, whereas lupeol leads to the formation of a triterpenoid hydrocarbon identified as lupa-2,2-(29)-diene (*Charters* et al. *1993; Pollard, Heron 1996; Regert, Rolando 2002; Regert* et al. *2003a: 2003b*).

Triacylglycerols

Triacylglycerols, as the most abundant components of fresh animal fats and plant oils, can be useful indicators of lipid preservation and the extent of degradation. A comparison of TAG distributions with those of modern reference fats has shown that specific distributions can be linked to different lipid sources and enable the preliminary differentiation of their origins from the two major classes of domestic animals (ruminant and non-ruminant/porcine) and between ruminant dairy and adipose fats. Ruminant animals show a characteristic distribution of TAGs, with carbon numbers ranging from C_{44} to C_{54} with a maximum concentration at C_{52}, whereas non-ruminant animals display a slightly shorter distribution with carbon numbers between C_{46} and C_{54} with a low concentration at C_{46} and C_{54} and a maximum again at C_{52}. Dairy fats show the widest TAG distribution, with carbon numbers ranging from C_{42} to C_{54}, usually with two maxima at C_{50} and C_{52} (*Evershed* et al. *1997; Dudd, Evershed 1998; Mottram* et al. *1999*). Triacylglycerol remains (including samples with only trace amounts preserved) were identified in thirty pottery lipid extracts, representing 17% of the total assemblage investigated. Quantifiable TAG distributions as detected in lipid extracts from Slovenian potsherds are represented in Figure 11.

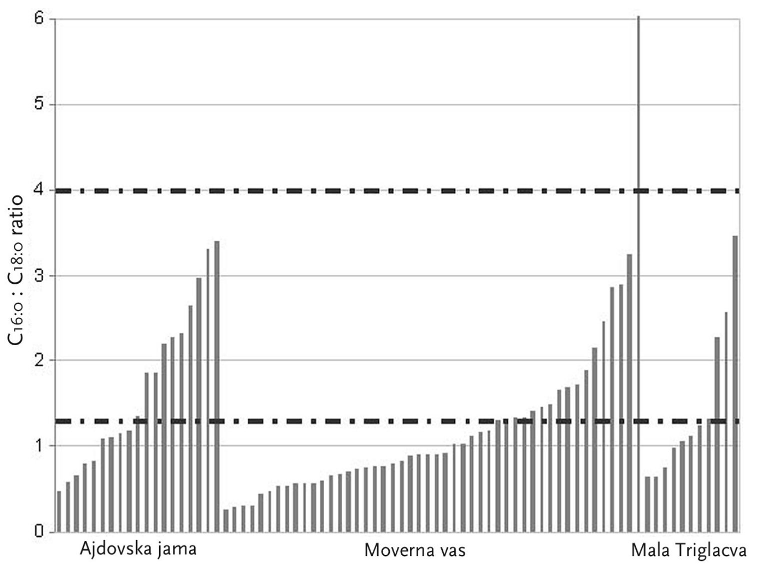


Fig. 8. Histograms representing the relative abundance ratios of palmitic ($C_{16:0}$) vs. stearic ($C_{18:0}$) fatty acid as detected in analysed potsherds. The dotted lines (set at 1.3 and 4) represent criteria as reported in the literature and used to separate ruminant adipose fats from non-ruminant, ruminant dairy fats and plant oils (**Dudd 1999; Copley** ***et al.*** **2005a; Romanus** ***et al.*** **2007*****).***

TAG distributions detected in potsherd extracts seem to be predominantly derived from ruminant adipose fats, with only 5 extracts (18MT, 79MT, 159MT, 96MV, 143MV) possibly deriving from ruminant dairy

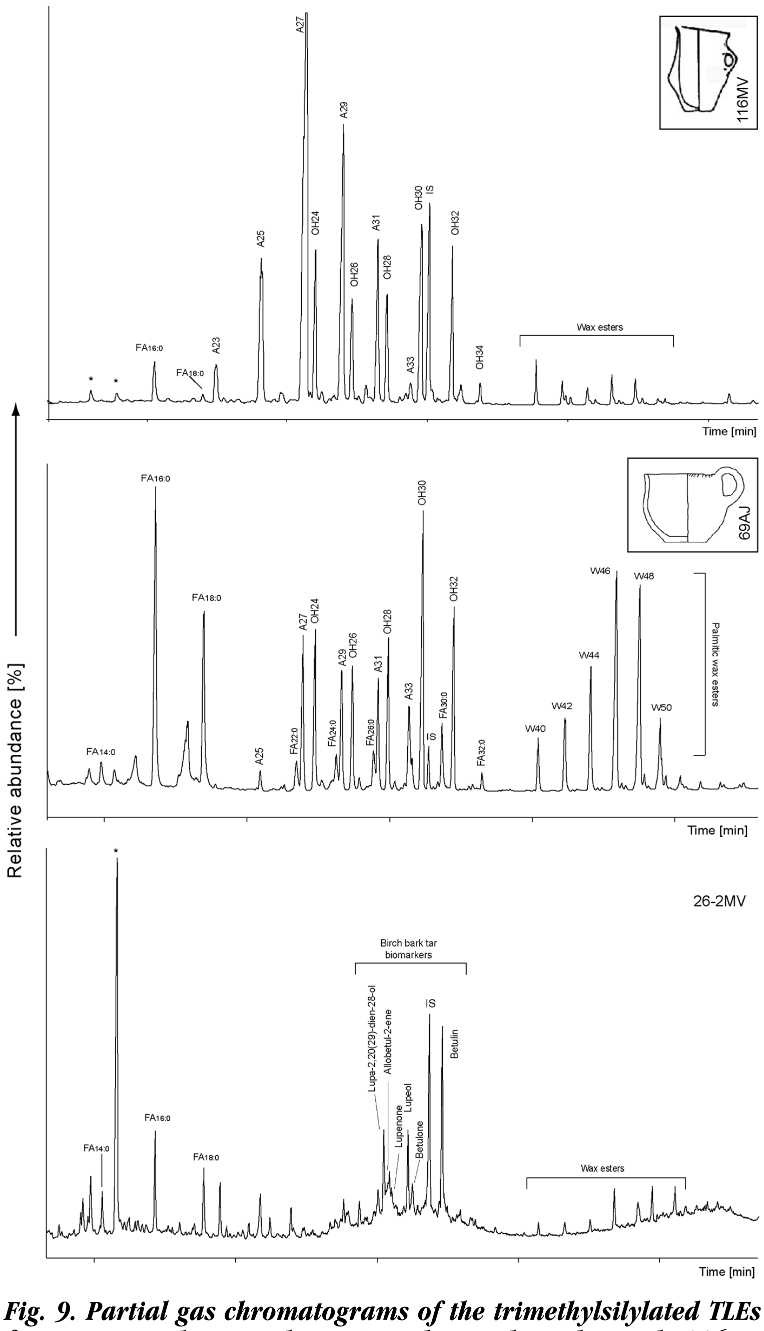


Fig. 9. Partial gas chromatograms of the trimethylsilylated TLEs from pottery showing the various biomarkers detected: 116MV epicuticular waxes residue; 69AJ a mixture of beeswax and plant residue; 26-2MV mixture of birch bark tar and plant residue. Key: FAx:y are fatty acids where x is the carbon chain length and y is the degree of unsaturation; OHx are long-chain alcohols of carbon chain length x; Ax are aliphatic alkanes of carbon chain length x; Wx are wax esters with carbon chain length x; IS is internal standard; * are plasticisers.

residues and with no TAG distribution indicating the presence of porcine fats. However, laboratory experiments have shown that TAG distributions can be skewed by degradation occurring during use or post-deposition, causing the wide TAG distribution characteristic of fresh ruminant dairy fat to become considerably narrowed due to the preferential degradation of compounds with lower carbon numbers, and thus come to resemble the narrower distribution seen in ruminant adipose fat TAGs distribution (*Dudd, Evershed 1998; Aillaud 2001*). Conclusions from TAG distributions have to be drawn with caution and serve only as preliminary results, complemented with compound-specific carbon isotope ratio measurements.

Compound-specific stable carbon isotope analysis

Compound-specific stable carbon isotope values ($\delta^{13}C$) were obtained for the palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acid methyl ester derivatives (FAMEs) from 52 Neolithic and Eneolithic potsherd residues with sufficient lipid concentrations. In order to elucidate the origin of preserved lipids accurately, archaeological $\delta^{13}C$ values were compared with modern reference fats from animals reared on isotopically similar diets to those of animals in prehistory. To eliminate any isotopic variations occurring in animals through differences in dietary intake or environmental factors, the difference between $\delta^{13}C_{18:0}$ – $\delta^{13}C_{16:0}$ values ($\Delta^{13}C$) is plotted in Figure 12. $\Delta^{13}C$ values ranging from –3.3 to –6.3‰ indicate ruminant dairy fats; values from 1.0 to 2.8‰ represent ruminant adipose fats, while values from –0.7 to + 1.9‰ indicate porcine adipose fats (*Dudd, Evershed 1998; Evershed* et al. *2008; Craig* et al. *2011; Dunne* et al. *2012; Salque* et al. *2013*).

Distributions of stable carbon isotopic values of lipids preserved in Neolithic and Eneolithic pottery show differences between individual archaeological sites: the residues recovered from Ajdovska jama pottery were predominantly of ruminant adipose (5 extracts) and ruminant dairy origin (4 extracts), while those from the Moverna vas pottery assemblage were mainly of ruminant adipose origin (17 extracts). The ubiquitous presence of dairy lipid residues in vessels from Mala Triglavca has already been reported, with 63% and

17% of vessels being used to process or store dairy products, respectively (*Šoberl* et al. *2008; Budja* et al. *2013*). The lowest occurrences were of porcine derived lipids, being found on only one potsherd from Ajdovska jama and three potsherds from Moverna vas. The mixing of various commodities throughout the life of vessels can also be seen by $\delta^{13}C$ values plotting close to, or between, the ranges of modern reference fats. It has been assumed that the pottery extracts with minor concentrations of leaf waxes or beeswax components present still reflect the isotopic signature of predominant fatty acids present in the residues.

Discussion

Pottery use in different contexts

Pottery has been traditionally regarded as a passive bearer of culture; however, with the rise of contextual archaeology, pottery has come to be seen more as an active factor, brought about by human agency and used in the construction of social identity (*Boast 2002; Gibson 2002*). Ceramic vessels could be used for many primary functions, such as the preparation, storage and cooking of food, brewing, tanning, dairying, dyeing, fulling, textile washing, transporting and salt preparation.

Whatever pottery was used for, it was an important artefact, as demonstrated by its appearance at domestic sites, as well as within ritually structured deposits. Pottery deposited in funerary settings could consist of previously used vessels or one made deliberately for that purpose. Although pottery is very robust and able to survive, it is also very sensitive and responsive to cultural, social, economic and ideological changes. These can be mirrored in a variety of ways: decoration, design, typology, modes of use and deposition.

Ajdovska jama

The ceramic vessels recovered from the Ajdovska jama site formed part of Neolithic burial rituals, acting as grave goods or simply containers for food offerings. Lipid preservation in ceramic vessels was good, with almost half of the potsherds (48%) yielding an appreciable amount of organic residue. The variety of animal remains deposited with the burials is well reflected in preserved fatty acid composition and isotopic signatures, indicating that ceramic vessels predominantly contained ruminant animal products (meat and dairy), while only one vessel showed the presence of porcine fat. The presence of mid-chain ketones in three pots (02AJ, 04AJ, 72AJ), formed by the condensation of precursor fatty acids at high temperatures in the presence of clay, suggests these vessels were used as cooking pots (Fig. 5). The extraction of uncommon unsaturated mid-chain homologues in 02AJ pot could be the result of processing plant material which contains high concentrations of unsaturated fatty acids. This was also suggested by the extracted organic residues, which in combination with the excavated plant remains (burnt, scattered cereal grains and pulses), show a high contribution of a plant-based diet, with approx. 25% of vessels containing some plant biomarkers. A mixed diet of plants and animals was also attested in bulk stable isotopic determinations ($\delta^{13}C$ and $\delta^{15}N$) of collagen extracted from osteological remains recovered from the burials (*Ogrinc, Budja 2005; Bonsall* et al. *2007*). However, enriched $\delta^{13}C$ values of palmitic acid extracted from lipid residues in ceramics (Fig. 12) could indicate a C_4 or marine component in the animal-based diet, as they are strongly diet-dependent (*Copley* et al. *2003*). Since a marine dietary contribution seems unlikely in the case of Ajdovska jama, enriched $\delta^{13}C_{16:0}$ values could have been introduced via animals eating plants from a waterlogged environment (*Salque* et al. *2012*). This observed difference between Ajdovska jama, Mala Triglavca and Moverna vas isotopic values

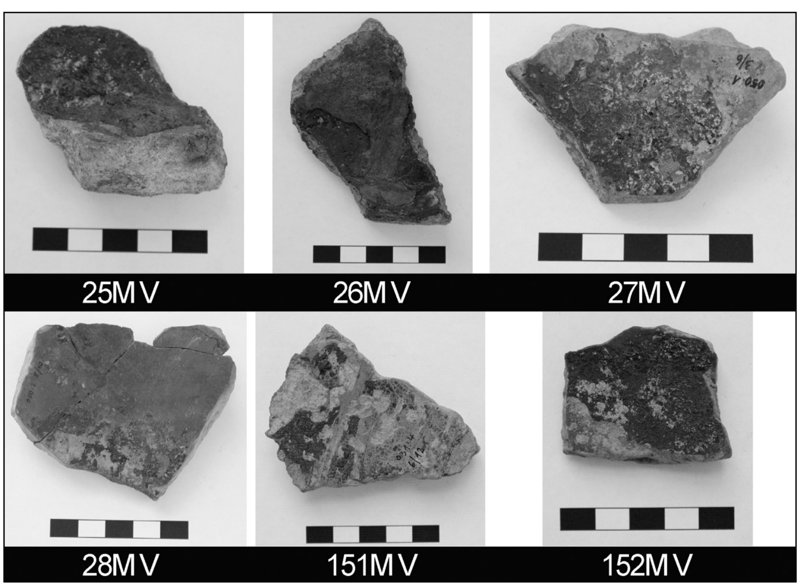


Fig. 10. Potsherds from Moverna vas with visible residues, remnants of birch bark tar application or production.

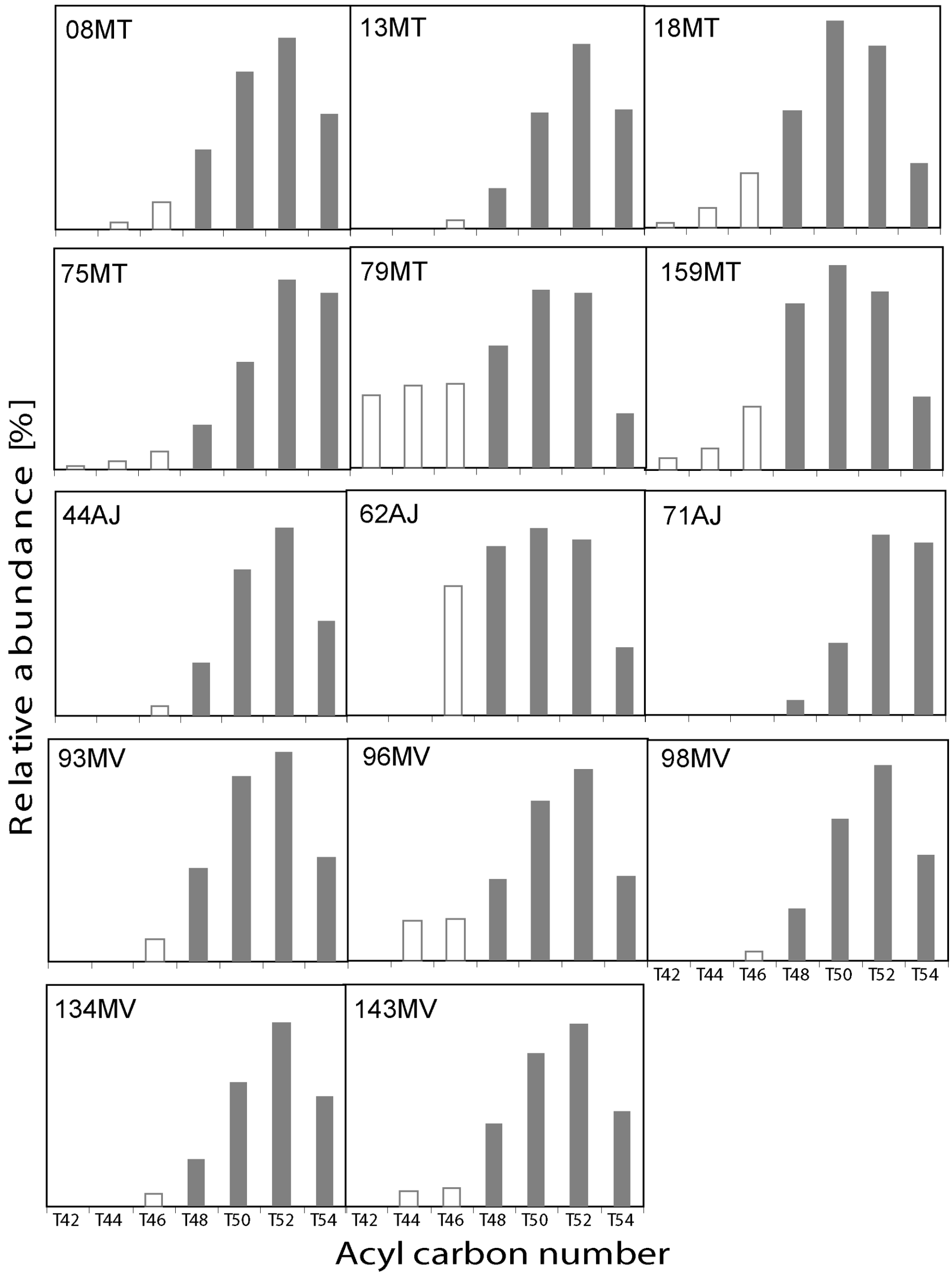
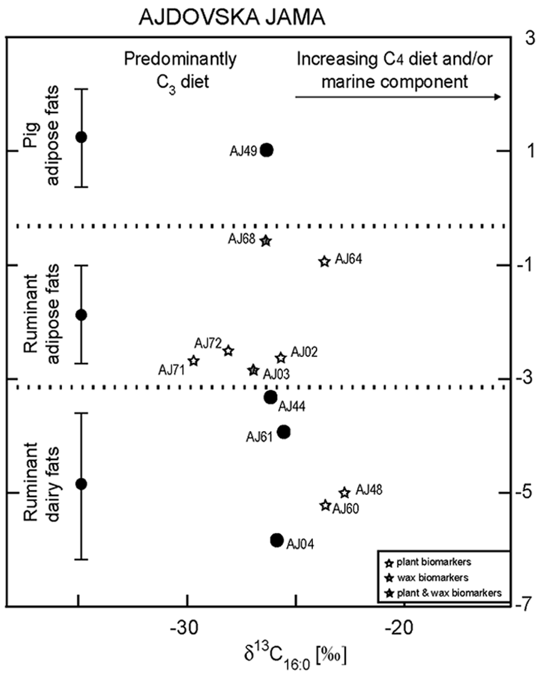


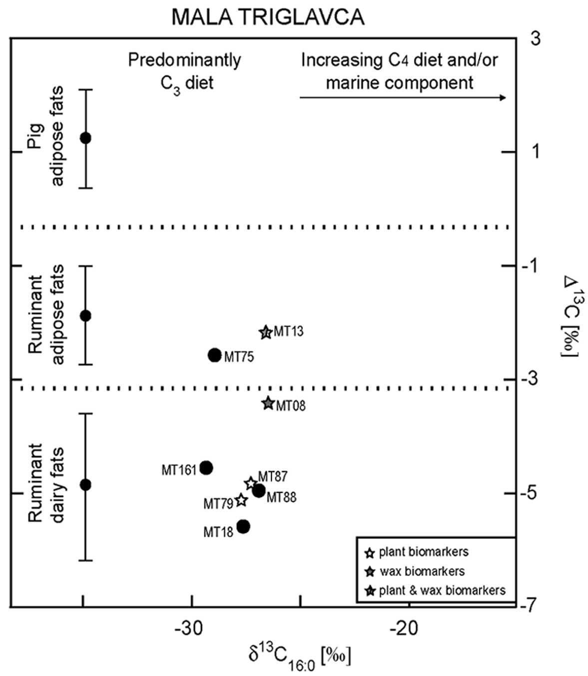
Fig. 11. Histograms showing triacylglycerol (TAG) distributions detected in pottery lipid extracts from Ajdovska jama, Mala Triglavca and Moverna vas. TX denotes the number of acyl carbon atoms in individual TAGs, grey bars represent TAGs identified in modern adipose and dairy fats while those in white are usually found only in dairy fats.

was also confirmed statistically with a two-tailed student's T-test which returned probability values of 0.003 and 0.016.

Mala Triglavca

Rock-shelters such as Mala Triglavca were used as gathering places for Mesolithic populations, and some could have been subsequently transformed into shelters and pens for domestic animals during the Neolithic. An analysis of herd structure and mortality on faunal remains (sex and age of animals) can be used to produce 'kill-off curves' in order to distinguish between meat or dairy animal exploitation (*Payne 1973*). Kill-off curves from Neolithic sites in the Northern Adriatic region have been interpreted in two ways: while Preston Miracle, Stašo Forenbaher and Laura Pugsley believe herds of domestic animals were kept predominantly for dairying, Dimitrij Mlekuž suggests simple, non-optimised animal husbandry (*Miracle, Forenbaher 2005; Miracle, Pugsley, 2006; Mlekuž 2005; 2006; Bonsall* et al. *2013; Rowley-Conwy* et al. *2013*).





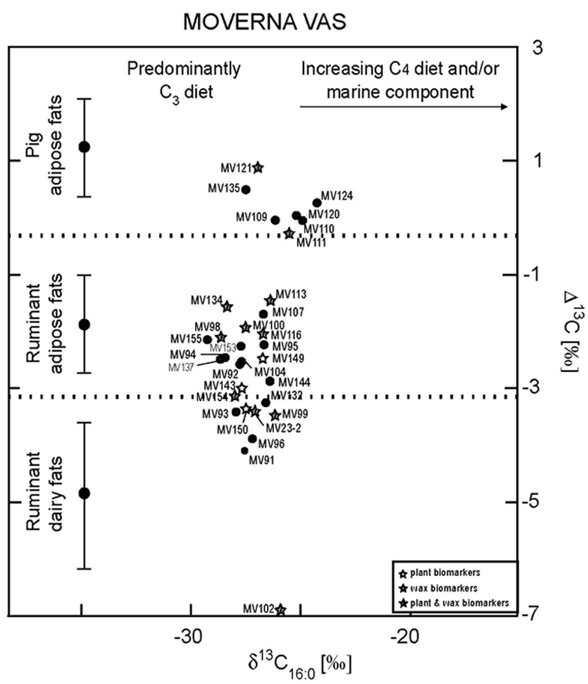


Fig. 12. Scatter plots showing $\Delta^{13}C$ values ($\delta^{13}C_{18:0}$ – $\delta^{13}C_{16:0}$) and $\delta^{13}C$ values of $C_{16:0}$ fatty acids extracted from Neolithic and Eneolithic pottery from sites Ajdovska jama, Mala Triglavca and Moverna vas. The ranges for the modern reference fats obtained from Europe, Africa, Kazakhstan and the Near East are plotted to the left of the diagram with mean ±1 s.d. (*Salque *et al.* 2013*). The $\delta^{13}C$ values obtained for modern reference animal fats have been adjusted for the post-Industrial Revolution effects of fossil fuel burning, by the addition of 1.2‰.

The findings of absorbed organic residues from Mala Triglavca rock-shelter pottery have been discussed elsewhere (*Šoberl* et al. *2008*) together with a more recent analysis of another pottery assemblage from the same site (*Budja* et al. *2013*). Both studies show lipid biomarkers of a mixed animal and plant economic model. Domesticates were simultaneously exploited for meat and milk. The question of the contribution of game to the diet of the Neolithic occupants of this rock-shelter, as attested in zoological remains, has yet to be addressed and investigated. Ceramic vessels displayed the lowest concentration range of preserved lipids, as well as an absence of mid-chain ketones, suggesting perhaps less intense food processing (no heat involved) or a faster turnover in pottery use. While porcine fats were completely absent, the $\delta^{13}C$ values of most abundant fatty acids ($C_{16:0}$ and $C_{18:0}$) fall within a range expected for ruminant dairy (75%) and ruminant adipose fats (25%; Fig. 12). Traces of odd-carbon number aliphatic alkanes, even carbon number long-chain alcohols and wax esters were detected in three potsherds (08MT, 13MT, 79MT), indicating a degree of mixed commodities (meat as well as vegetables) being processed within these vessels.

Moverna vas

The Neo-Eneolithic settlement of Moverna vas had fully developed animal husbandry with agriculture, as well as diverse pottery production. The complexity of this settlement was mirrored perfectly in the lipid biomarkers extracted from the ceramic vessels. The 99 potsherds analysed covered a diverse vessel typology, from large cooking and storage pots to small 'drinking' cups and highly specialised pedestal dishes. Intensive use of ceramic vessels in food preparation and storage is reflected in the highest lipid concentration range (Fig. 6) and the presence of mid-chain ketones identified in three potsherds (29MV-2, 91MV, 133MV) (Fig. 4). Extensive mixing of commodities (meat and plants) is apparent from extracted lipid biomarkers, in which not only free fatty acids were identified together with their parent acylglycerol moieties, but also a suite of other compounds, *i.e.* aliphatic alkanes, long-chain fatty acids, long-chain alcohols, triterpenoids and wax esters. In the absence of faunal remains, compound specific stable isotope analysis of palmitic and stearic fatty acid enabled us to approximately reconstruct animal husbandry practices. While $\delta^{13}C$ values for $C_{16:0}$ and $C_{18:0}$ extracted from ceramic vessels found at Ajdovska jama and Mala Triglavca sites showed the ubiquitous presence of ruminant meat and dairy products, the potsherds from Moverna vas settlement contained predominantly ruminant and porcine adipose lipids (Fig. 12). Only seven potsherds revealed the presence of milk residues (23-2MV, 91MV, 93MV, 96MV, 99MV, 102MV, 150MV) (Fig. 4). An elusive association of porcine products with prehistoric pottery has been observed in the past, especially in British Neolithic pottery (*Mukherjee* et al. *2007*) as well as two recently investigated Slovenian Neolithic pottery assemblages from Maharski prekop and Resnikov prekop. While porcine derived lipids were detected in extracted residues at Resnikov prekop, the same class of foodstuff was completely absent from Maharski prekop (*Ogrinc* et al. *2012; Mlekuž* et al. *2012; 2013*). The discrepancy in preserved porcine lipids and faunal statistics may be the result of alternative ways of preparing porcine meat that did not necessarily involve pottery, but perhaps spit-roasting, as suggested by Umberto Albarella and Dale Serjeantson (*2002*).

Pottery use within typology

The ubiquity of pottery finds in all archaeological sites shows indirectly that this was a commodity produced en masse and used daily, not simply made for display or burials. From the perspective of pottery typology, it is only assumed which vessels were used for storing and/or cooking food.

Investigations of British Neolithic and Bronze Age pottery revealed correlations between specific commodity groups and three main differential criteria: (a) pottery size/rim diameter; (b) pottery typology, and (c) various household activities (*Copley* et al. *2005c; 2005d*). Similarly, biomarkers for a specific commodity, dairy products in this case, were detected in Neolithic ceramic sieves in Europe, which were in turn interpreted as cheese strainers (*Salque* et al. *2013*). Lipid residue analyses of pottery from Ajdovska jama, Mala Triglavca and Moverna vas have shown some correlations between lipid concentrations and pottery typology (Figs. 4, 5 and 13), while only two correlations between specific commodities and vessel types have been observed. Mid-chain ketones, which are used as biomarkers for exposure to high temperature (cooking), were observed in only three vessel types, all characterised either by larger volumes or openness of rims: pots, bowls and dishes. Pots and pedestal dishes were also unique ceramic types associated with birch-bark tar biomarkers.

Rice (*1987*) divided the principle functions of pottery into three categories: (i) storing dry substances; (ii) carrying liquids; and (iii) heating contents over fire. The investigation of potsherd samples taken from different parts along the profile of the same ceramic vessel compared to laboratory cooking simulation experiments has shown correlations between concentrations of absorbed lipids, their spatial distribution and different modes of pottery use.

The hydrophobic nature of lipids and their lower density results in the highest lipid concentrations to be absorbed near the top of the vessel, where the original water line would have been (*Charters* et al. *1997*). Other lipid distributions observed in the bases of vessels are thought to indicate an analogous preparation of food, namely roasting, or the application of surface sealing treatments (*Charters* et al. *1993b; 1997*).

The average lipid concentration profiles in Figure 13 were divided into rim, body and base sherds to assess potential variations in distinguishing pottery use. Investigated pottery from Ajdovska jama, Mala Triglavca and Moverna vas shows distinct differences in vessel use: while average lipid concentration in cups peaks in the upper parts of the vessels, suggesting preferential absorption of immobilised lipids, an opposite concentration distribution was observed for pots and jugs, where the highest concentration in the lower vessel parts could indicate 'dry' cooking.

Average lipid concentration poses an interesting question as well; accepting that lipid accumulation correlates with the longevity of vessel's use, it is clear from Figure 13 that pedestal dishes, small cups and

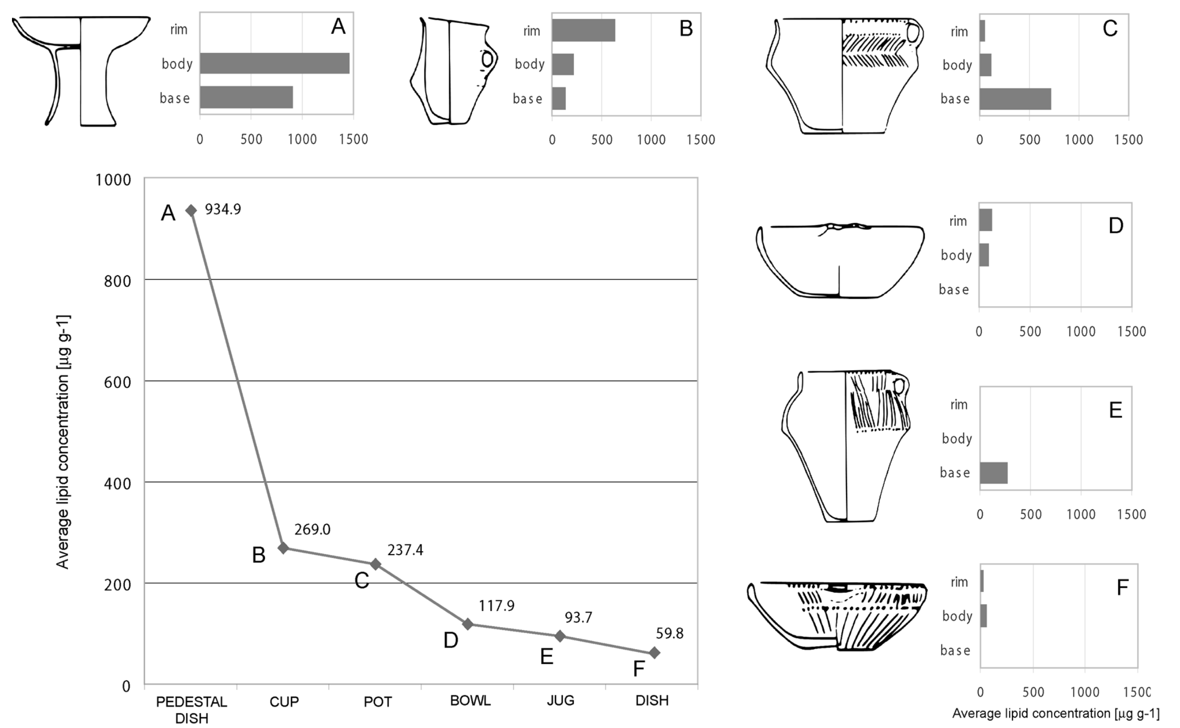


Fig. 13. Diagrams showing the mean concentration of extracted lipids according to ceramic typology and potsherd location.

pots with the highest preserved lipid concentrations (935, 269 and 237µg g^{-1}, namely) were probably used regularly to process or store fatty foodstuffs over an extended period. On the other hand, bowls, jugs and dishes were perhaps used only as storage vessels for less fatty commodities, or were perhaps dedicated serving vessels. An estimate of the longevity of cooking pots in regular use ranges between three months, one year and even longer periods of two to ten years (*Foster 1960; David 1972; DeBoer 1974; Rice 1987; Longacre 1991*).

Birch-bark tar – a multi-purpose widespread prehistoric commodity

Similar to beeswax, resins have also been shown to have had various potential applications in prehistory: as adhesives (*Charters* et al. *1993a; Regert* et al. *2003b*), for medicinal use (*Lucquin* et al. *2006; Evans, Heron 1993*), as a waterproofing agent (*Evershed* et al. *1985; Robinson* et al. *1987; Romanus* et al. *2009*) in pitch and tar production (*Eerkens* et al. *2002*) or perhaps in wine production (*McGovern* et al. *2009*). The presence of birch-bark tar has been widely reported from various prehistoric archaeological contexts, where it was mainly used as hafting adhesive on arrowheads or as a material used to repair broken ceramic vessels as early as the Neolithic (*Pollard, Heron 1996; Regert, Rolando 2002; Regert* et al. *2003b; Regert 2004; Lucquin* et al. *2006*). Birch-bark tar is sometimes also found as free lumps in sediment or in the form of visible residues on the exterior or interior surfaces of ceramic vessels (*Charters* et al. *1993; Bosquet* et al. *2001; Urem-Kotsou* et al. *2002; Regert* et al. *2003; Lucquin* et al. *2006*). Birch-bark tar was only identified as a visible organic residue on pottery from Moverna vas, where it was linked specifically to pedestal dishes and pots. Pedestal dishes have been previously reported together with this natural product from Neolithic funerary contexts in Brittany (*Lucquin* et al. *2006*), where the authors interpreted these vessels as 'incense burners' or portable hearths. As the smell of burning tar is quite unpleasant, it has been suggested that it might have been used to mask strong odours, such as decomposing bodies in funerary contexts (*ibid.*). This theory could explain the find of a small, black, amorphous lump of tar within the burial sediments in Ajdovska jama, where burial rituals were similar to those described above (*Šercelj, Culiberg 1984*). The presence of this natural substance has also been reported from the Urnfield culture cemetery at the Slovenian Academy of Sciences and Arts in Ljubljana (*Puš 1976; Hadži, Cvek 1976; Hadži, Orel 1978*) and the Neolithic pile dwelling site at Maharski prekop (*Hadži, Orel 1978*). The availability of birch trees in prehistory has also been confirmed by palinological analysis of contemporary regional sediments, where a decline has been recorded after 6400 cal BP with the increase of 'anthropogenic indicators' (*Andrič 2007*).

Conclusions

The organic residue analysis of the Neolithic and Eneolithic pottery from Ajdovska jama, Mala Triglavca and Moverna vas showed very good lipid preservation, enabling us to reconstruct the past pottery use and address potential contextual differences. The choice of three sites with very diverse archaeological contexts has proven to be justified, as the vessels retained varying lipid concentrations, probably depending on their originating contexts.

Pottery from Mala Triglavca rock shelter yielded the lowest lipid recovery (30.6%) as well as the lowest lipid concentration range, which together with the absence of mid-chain ketones identified might suggest less intense food processing, without heating, or a faster turnover in pottery use. Lipid biomarkers confirm the archaeozoological data, *i.e.* the presence of domesticated ovicaprids, which were exploited for both meat and dairy products, occasionally mixed with plant-based foods as indicated by biomarkers.

The Ajdovska jama pottery that played a part in prehistoric funerary rituals proved to retain 48% of vessels with identifiable lipid residues. Identified biomarkers reflect the animal remains that were deposited with the deceased, suggesting a mixed plant and animal based diet. The compound specific stable isotopic analysis of primary fatty acids suggests that the lipids derive from ruminant animals (meat and dairy) and one porcine fat residue, most likely a remnant of wild boar. A high occurrence of plant biomarkers (25% of the pottery assemblage) in conjunction with recovered palaeobotanical remains suggests that a large proportion of the cave visitors' diet or food offerings were plant based.

The highest lipid recovery rate (65.9%) as well as the broadest lipid concentration range of Moverna vas settlement pottery can be interpreted as an indication that prehistoric ceramic vessels were used frequently to process or store foodstuff of animal as well as vegetable origin. The complexity of biomarkers in ceramic vessels mirrors perfectly the complexity of a fully developed Neolithic and Eneolithic settlement economy. Ceramic vessels were used to process animal products (ruminant and porcine adipose fats) and plant-based foodstuffs, as well as more rare commodities such as beeswax (perhaps indicating the presence of honey) and birch-bark tar. Pots and pedestal dishes were unique ceramic types associated with birch-bark tar biomarkers, which were identified only on pottery from Moverna vas. Other prehistoric finds of birch-bark tar from Slovenian archaeological sites include a lump of tar in sediment from Ajdovska jama, and occurrences with pottery have been reported from other Neolithic and Bronze Age sites in Slovenia.

The analysis of pottery typology and lipid residues showed that some vessels types can be linked to specific foodstuffs or food preparation techniques. Mid-chain ketones, biomarkers for exposure to high temperature, were observed only in vessel types of larger volume or openness of rim (*i.e.* pots, bowls and dishes). High lipid concentrations detected in pedestal dishes, cups and pots suggest intensive use with fatty foods, while bowls, jugs and dishes were perhaps used only as storage or serving vessels for less fatty commodities.

ACKNOWLEDGEMENTS

This research was financially supported by the European SOCRATES exchange programme, which enabled LŠ to undertake the analysis at the Organic Geochemistry Unit in Bristol, UK in October 2006 and April 2007. Thanks are due to the UK Natural Environment Research Council for their support of mass spectrometry facilities to the Bristol node of NERC Life Sciences Mass Spectrometry Facility and also to Ian Bull and Rob Berstan for their invaluable assistance with the instrumental analysis.

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Appendix

App. 1. Details of ceramic assemblages investigated.

Sample # CHEM	Sample # ARCH	Site code	SF #	SE/rel. depth	Sector/quadrant	Description
01AJ	1	Ajdovska jama 1982	129a	42	LH/tunnel 1	body of a pot, bucket- shaped
02AJ	2	Ajdovska jama 1982	684	43	CDV/XXII, group 4	body of a pot
03AJ	3	Ajdovska jama 1982	567/2	43	CDV/XXII, group 4	body of a dish
04AJ	4	Ajdovska jama 1982	660 (2519)	43	CDV/XVI, group 4	body of a pot
05AJ	5	Ajdovska jama 1982	682	43	CDV/XVI, group 4	body of a dish
06AJ	6	Ajdovska jama 1982	146	44	LH/tunnel 1	body of a pot
07AJ	7	Ajdovska jama 1982	43	43	LH/tunnel 2	body of a pot
31AJ	8	Ajdovska jama 1976	208	43	LH/group 6	body of a pedestal dish
32AJ	9	Ajdovska jama 1982	70	/	DH/33	body with base of a dish, black residue on exterior
33AJ	10	Ajdovska jama 1986/87	633	43	CDV/XVIa, group 4	body of a pedestal dish, modern glue residue
34AJ	11	Ajdovska jama 1986	640	42	CDV/XXII	upper body of a pedestal dish
35AJ	12	Ajdovska jama 1967	262	43	LH/group 6	base of a pedestal dish, traces of a red slip on exterior
36AJ	13	Ajdovska jama 1967	284	43	LH/group 6	body of a dish
37AJ	14	Ajdovska jama 1988	730	43	CDV/XXIVa, group 4	base with body of a spouted dish, yellowish residue perhaps on interior
38AJ	15	Ajdovska jama 1986	577	43	CDV/XVI, XIIa, group 4	rim of a spouted dish, black residue on interior, shiny surface – perhaps consolidant?
39AJ	16	Ajdovska jama 1986	576	42	CDV/ XXIX/XXVIII; XXVIII	rim of a dish, modern glue residue
40AJ	17	Ajdovska jama 1967	274	42	LH	body of a spouted dish, modern glue present
41AJ	18	Ajdovska jama 1985-87	575	43	CDV/XXII,XXIIa, XXIX, group 4	rim of a dish
42AJ	19	Ajdovska jama 1985/86	635	43	CDV/XXIIa, group 4	rim of a bowl, modern glue present
43AJ	20	Ajdovska jama 1986	574	42	CDV/XVI	rim of a dish, unusual surface – organic residue, limescale or consolidant?
44AJ	21	Ajdovska jama 1967	273	43	LH/group 6	body of a spouted dish
45AJ	22	Ajdovska jama 1967	245	42	LH	body of a spouted dish , modern glue present
46AJ	23	Ajdovska jama 1982	122	42	LH/tunnel 1	rim of a dish with an appliqué decoration
47AJ	24	Ajdovska jama 1982	119	44	LH/group 1	body of a dish, ribbed decoration
48AJ	25	Ajdovska jama 1986	584	42	CDV/XXIX	body of a bowl (by fireplace SE56), ribbed decoration
49AJ	26	Ajdovska jama 1985	583	43	CDV/XXII, group 4	rim of a bowl
50AJ	27	Ajdovska jama 1986	586	/	CDV/ X/IX,XV,XXIX	body of a ribbed dish
51AJ	28	Ajdovska jama 1982/83	109	42	LH/tunnel 1	body of a spouted dish
52AJ	30	Ajdovska jama 1982	126	42	LH/tunnel 1	body of a bowl, bucket-shaped
53AJ	32	Ajdovska jama 1967	258	43	LH/group 6	body with base of a spouted bowl
54AJ	33	Ajdovska jama 1985	440	43	CDV/XVI,XXII, group 4	rim of a jug
55AJ	34	Ajdovska jama 1967	267	43	LH/group 6	body of a jug with a horizontal rib and incised decoration
56AJ	35	Ajdovska jama 1967	206	43	LH/group 6	base of a jug
57AJ	36	Ajdovska jama 1967	257	43	LH/group 6	body of a jug
58AJ-1	29	Ajdovska jama 1987	739	44	CDV/XXII,XXIIa,XVIa	body of a pot, burnt residue on interior and exterior
58AJ-2	29	Ajdovska jama 1987	739	44	CDV/XXII,XXIIa,XVIa	body of a pot, burnt residue on interior and exterior
59AJ	31	Ajdovska jama 1984	116	42	LH/tunnel 1	body of a pot, bucket-shaped
60AJ	37	Ajdovska jama 1985-87	743	43	CDV/XXII,XXIIa,XVI, group 4	rim of a pot with incised decoration

Sample # CHEM	Sample # ARCH	Site code	SF #	SE/rel. depth	Sector/quadrant	Description
61AJ	38	Ajdovska jama 1986/87	756	43	CDV/XXIIa, group 4	two rims of a pot – one with a horizontal rib, one with criss-cross incisions
62AJ	39	Ajdovska jama 1985	737	43	CDV/XXII, group 4	body of a pot with horizontal incisions and a base of the handle (similar in Moverna vas assemblage)
63AJ	40	Ajdovska jama 1982	120	44	LH/group 1	three body fragments of a pot with a zoomorphic head
64AJ	41	Ajdovska jama 1984	385	83	CDV/XVIII	rim with body of a pot
65AJ	42	Ajdovska jama 1987	641	43	CDV/XXIIa, group 4	body of a pot
66AJ	43	Ajdovska jama 1985	564	43	CDV/XXII, XVI, group 4	rim of a pot with vertical incisions
67AJ	44	Ajdovska jama 1985/86	455	43	CDV/XVI,XXII,XXIIa, group 4	base of a pot, painted decoration
68AJ	45	Ajdovska jama 1982	125	44	LH/group 1	body of a pot
69AJ	46	Ajdovska jama 1982	107	42	LH/tunnel 1	body of a pot
70AJ	47	Ajdovska jama 1967/82	670	44	LH/group 1	body of a large pot (pythos)
71AJ	48	Ajdovska jama 1982	198	43	LH/group 2	body of a pot
72AJ	49	Ajdovska jama 1985-87	606	44	CDV/XXII,XVI, group 3	body of a pot, red slip on exterior, burnt interior
73AJ	50	Ajdovska jama 1982	129	43	LH/tunnel 1	body of a pot, bucket-shaped
74AJ	51	Ajdovska jama	703	?	CDV/22	body of a pot, ribbed decoration
8MT	/	Mala Triglavca 2006	PN1690	55	A/L92/a	body, unwashed, undefined type
9MT	/	Mala Triglavca 2006	PN1722	55	A/L92/a	body, unwashed, undefined type
10MT	/	Mala Triglavca 2006	PN1697	55	A/K93/c	body, undefined type
11MT	/	Mala Triglavca 2006	PN1752	55	A/K91/c	body, undefined type
12MT	/	Mala Triglavca 2006	PN1694	55	A/L93/a	body, undefined type
13MT	/	Mala Triglavca 2006	PN1714	55	A/K92/c	rim, undefined type
14MT	/	Mala Triglavca 2006	PN1748	55	A/L91/a	body, undefined type
15MT	/	Mala Triglavca 2006	PN1561	47	A/L91/d	body, undefined type
16MT	/	Mala Triglavca 2006	PN1680	55	A/L92/d	body, undefined type
17MT	/	Mala Triglavca 2006	PN1644	47/51	A/L90/a	body, undefined type
18MT	/	Mala Triglavca 2006	PN1829	61	A/L91/a	body, undefined type
75MT	78	Mala Triglavca	R043	2.90-3.05m	5,6,7	perforated rim, undefined type
76MT	81	Mala Triglavca	R044	2.90-3.05m	5,6,6	base with body, burnt interior, undefined type
77MT	85	Mala Triglavca	R045	2.90-3.05m	5,6,7	body of a pot
78MT	86	Mala Triglavca	R046	2.90-3.05m	5,6,7	body of a bowl, incised decoration
79MT	101	Mala Triglavca	R052	2.90-3.05m	5,6,7	body of a bowl, polished exterior
80MT	103	Mala Triglavca	R054	2.90-3.05m	5,6,7	rim and body (two fragments), incised decoration, undefined type
81MT	112	Mala Triglavca	R060	2.90-3.05m	5,6,7	body of a bowl, burnt interior
82MT	140	Mala Triglavca	R076	2.90-3.05m	5,6,7	base with body, undefined type
83MT	169	Mala Triglavca	R097	2.70-3.00m	4	body of a dish
84MT	335	Mala Triglavca	R178	2.70-2.90m	3	base with body, undefined type
85MT	377	Mala Triglavca	R187	2.60-2.75m	5,6,7	rim with body (two fragments) of a bowl
86MT	440	Mala Triglavca	R208	2.50-2.70m	4	rim with body, traces of a black slip, undefined type
87MT	459	Mala Triglavca	R214	2.50-2.70m	4	body of a cup, modern glue residue
88MT	503	Mala Triglavca	R230	2.60-2.80m	3	rim with body, burnt interior, undefined type
89MT	512	Mala Triglavca	R236	2.30-2.50m	4	rim with body (two fragments), undefined type
156MT	20	Mala Triglavca 1984	R016	3.05-3.25m	5	undefined type
157MT	27	Mala Triglavca 1984	R021	3.05-3.25m	5	pot
158MT	36	Mala Triglavca 1984	R024	3.05-3.25m	5,6,7	bowl
159MT	40	Mala Triglavca 1984	R027	3.05-3.25m	5,6,7	bowl
160MT	59	Mala Triglavca 1984	R034	3.05-3.25m	5,6,7	bowl
161MT	99	Mala Triglavca 1984	R050	2.90-3.05m	5,6,7	ladle
162MT	159	Mala Triglavca 1981	R090	2.70-3.00m	4	bowl
163MT	222	Mala Triglavca 1981	R123/124	2.70-3.00m	4	handle fragment

Sample # CHEM	Sample # ARCH	Site code	SF #	SE/rel. depth	Sector/quadrant	Description
164MT	238	Mala Triglavca 1981	R133	2.70-3.00m	4	cup
165MT	239	Mala Triglavca 1981	R134	2.70-3.00m	4	bowl
166MT	442	Mala Triglavca 1981	R210	2.50-2.70m	4	pot
23MV-1	/	Moverna vas 1988	1238	053.1	5/13	visible black, charred residue, inside
23MV-2	/	Moverna vas 1988	1238	053.1	5/13	small bowl
24MV-1	/	Moverna vas 1988	2478	050.2	4/3	visible black, charred residue, inside
24MV-2	/	Moverna vas 1988	2478	050.2	4/3	base with body of a pot, organic residue on interior
25MV-1A	/	Moverna vas 1988	/	050.1	6/1-116	visible black, compact residue, inside
25MV-1B	/	Moverna vas 1988	/	050.1	6/1-116	visible brown, compact residue, inside
25MV-2	/	Moverna vas 1988	/	050.1	6/1-116	base with body of a pot, organic residue on interior
26MV-1	/	Moverna vas 1988	/	022.1	4/15	dark brown, compact residue, inside
26MV-2	/	Moverna vas 1988	/	022.1	4/15	base with body of a pot, organic residue on interior
27MV-1A	/	Moverna vas 1988	6	050.1	5/6	black, compact residue, inside - only spots
27MV-1B	/	Moverna vas 1988	6	050.1	5/6	black, compact residue on section
27MV-1C	/	Moverna vas 1988	6	050.1	5/6	black, compact residue outside - only spots
27MV-2	/	Moverna vas 1988	6	050.1	5/6	foot of a pedestal dish with red slip, organic residue on the foot
28MV-1A	/	Moverna vas 1988	5	050.2	5/10	black, compact residue, inside - only spots
28MV-1B	/	Moverna vas 1988	5	050.2	5/10	black, compact residue on section
28MV-1C	/	Moverna vas 1988	5	050.2	5/10	black, compact residue outside - only spots
28MV-2	/	Moverna vas 1988	5	050.2	5/10	foot of a pedestal dish with red slip, organic residue on the foot
29MV-1A	/	Moverna vas 1984	212	323-332cm	2/1-8/7, N-profile	pale brown residue on various parts, mixed with black residue - possibly soil, outside
29MV-1B	/	Moverna vas 1984	212	323-332cm	2/1-8/7, N-profile	"black, compact residue in form of a lump, located just below the rim and running over the rib; looks charred and visible fibres, outside"
29MV-2	/	Moverna vas 1984	212	323-332cm	2/1-8/7, N-profile	rim of a biconical cup, organic residue on exterior
30MV-1	/	Moverna vas 1984	/	/	2/9/7, T8-7/7	white, compact residue in spots, inside
30MV-2	/	Moverna vas 1984	/	/	2/9/7, T8-7/7	base of a miniature bottle with incised decoration, whiteish residue on interior
90MV	1	Moverna vas 1988	R6	056.3	5/7	body of a spouted dish, type 1
91MV	2	Moverna vas 1988	R17	056	4/11	body of spouted dish (two fragments), type 1, burnt interior
92MV	3	Moverna vas 1988	R 174	056.3	3/10-14	rim with body of a spouted dish, type 1, impressed and combed decoration
93MV	4	Moverna vas 1988	R176	056.3	3/7	body of a spouted dish, type 1, appliqué decoration on exterior
94MV	5	Moverna vas 1988	R226	056.1	6/13	body of a spouted dish, type 1
95MV	6	Moverna vas 1988	R 467	050.2	?	body of a spouted dish, type 1, horizontal rib and combed decoration
96MV	7	Moverna vas 1988	R8	056.2	3/13	body of a bowl with red slip, traces of residue on exterior, type 2
97MV	8	Moverna vas 1988	R19	056.2	4/3	body of a bowl (two fragments), type 2
98MV	9	Moverna vas 1988	R23	056.3	5/6	rim of a bowl (two fragments), type 2
99MV	10	Moverna vas 1988	R27	056.3	5/10	body of a bowl, type 2
100MV	11	Moverna vas 1988	R36	056.2	3/7	rim with body of a bowl (three fragments), type 2
101MV	12	Moverna vas 1988	R179	056.2	3/5,9	body of a bowl, type 2
102MV	?	Moverna vas 1988	?	056.3	3/7	rim of a bowl, type 2
103MV	13	Moverna vas 1988	R20	056.3	4/13	body of a small pot with red slip, type 3
104MV	14	Moverna vas 1988	R239	056.3	5/13	body of a small pot (two fragments), type 3
105MV	15	Moverna vas 1988	sample 99	050.1-2	3/7	body of a small pot (three fragments), type 3, horizontal rib

Sample # CHEM	Sample # ARCH	Site code	SF #	SE/rel. depth	Sector/ quadrant	Description
106MV	16	Moverna vas 1988	sample 103	056.2	3/14	body of a small pot (three fragments), type 3 horizontal rib
107MV	17	Moverna vas 1988	sample 104	056.2	3/9	body of a small pot with red slip, type 3
108MV	18	Moverna vas 1988	sample 105	056.2	3/3	base of a small pot with red slip, type 3
109MV	19	Moverna vas 1988	sample 106	056.2	3/3	body of a small pot, horizontal rib, type 3
110MV	20	Moverna vas 1988	sample 107	056.2	3/6	rim with body of a small pot with red slip, type 3
111MV	21	Moverna vas 1988	sample 108	056.2	3/6	base of a small pot with red slip, type 3
112MV	22	Moverna vas 1988	sample 109	056.2	3/8	rim of a small pot with red slip, type 3
113MV	23	Moverna vas 1988	sample 110	056.2	3/2	whole profile of a small pot with red slip, type 3
114MV	24	Moverna vas 1988	sample 111	056.2	3/2	base of a small pot, type 3
115MV	25	Moverna vas 1988	sample 112	056.2	3/7	body of a small pot with red slip, type 3
116MV	26	Moverna vas 1988	sample 113	056.2	3/1-16	body of a small pot with fragmented handle, type 3
117MV	27	Moverna vas 1988	sample 114	056.2	3/14	body of a small pot with red slip (two fragments), horizontal rib, type 3
118MV	28	Moverna vas 1988	sample 115	056.2	3/14	body of a small pot with red slip, type 3
119MV	29	Moverna vas 1988	sample 116	056.2	3/14	body of a small pot with red slip, horizontal rib, type 3
120MV	30	Moverna vas 1988	sample 117	056.2	3/14	body of a small pot with red slip, type 3
121MV	31	Moverna vas 1988	sample 118	056.2	3/14	body of a small pot, incides decoration on interior, type 3
122MV	32	Moverna vas 1988	sample 119	056.2	?	rim and base of a small pot with grey slip, type 3
123MV	33	Moverna vas 1988	sample 120	056.2	3/15	body of a small pot with red slip, type 3
124MV	34	Moverna vas 1988	sample 121	056.2	3/5	body and base of a small pot with red slip, type 3
125MV	35	Moverna vas 1988	sample 132	056.3	5/5	body of a small pot, horizontal rib, type 3
126MV	36	Moverna vas 1988	sample 133	056.3	5/5	rim of a small pot with red slip, type 3
127MV	37	Moverna vas 1988	sample 134	056.3	5/10	body of a small pot with red slip, type 3
128MV	38	Moverna vas 1988	sample 183	056.2	4/7.3/3	body of a pot (two fragments), type 4
129MV	39	Moverna vas 1988	sample 225	056.3	3/12	body of a pot (two fragments), type 4
130MV	40	Moverna vas 1988	sample 102	056.3	5/11	body of a pot, type 5, combed decoration on exterior
131MV-1	41	Moverna vas 1988	sample 128	056.2	3/5	brown visible residue on exterior
131MV-2	41	Moverna vas 1988	sample 128	056.2	3/5	body of a pot, type 5, horizontal rib and combed decoration on exterior, organic residue on exterior
132MV	42	Moverna vas 1988	R38	056.3	3/7	body of a pot, type 5,
133MV	43	Moverna vas 1988	R130	056.2	3/3	rim of a pot, type 5,
134MV	44	Moverna vas 1988	R131	056.2	3/3	rim and body of a pot, type 5, horizontal rib and combed decoration on exterior
135MV	45	Moverna vas 1988	R181	056.3	5/1	body of a pot (two fragments), type 5, combed decoration on exterior
136MV	46	Moverna vas 1984	R222	1/8/7	1/5-8	body of a pot, type 5, incised decoration on exterior
137MV	47	Moverna vas 1984	R 262	2/5/7	2/9	body of a pot (two fragments), type 5, impressed and combed decoration on exterior
138MV	48	Moverna vas 1988	R212	050.1	5/11	body of a pot (three fragments), type 6
139MV	49	Moverna vas 1988	sample 101	056.3	3/14	body of a pot, type 6, horizontal rib and combed decoration on exterior
140MV	50	Moverna vas 1988	R8	056.3	3/10	body of a pedestal dish with red slip, type 7
141MV	51	Moverna vas 1988	R122	056.2	3/2	rim of a pedestal dish with red slip, type 7
142MV	52	Moverna vas 1988	R124	056.2	3/11	base with foot of a pedestal dish, type 7, red slip
143MV	53	Moverna vas 1988	R127	056.2	3/5	rim and body of a pedestal dish, type 7, red slip
144MV	54	Moverna vas 1988	R224	056.3	3/5	base with foot of a pedestal dish, type 7, red slip
145MV	55	Moverna vas 1988	R264	050.1.2	3/3	body of a pedestal dish, type 7, red slip
146MV	56	Moverna vas 1984	R56	1/8/7	1/5-6	base and foot of a pedestal dish, type 7, red slip
147MV	57	Moverna vas 1988	sample 100	056.2	3/7	base and foot of a pedestal dish, type 7, red slip
148MV	58	Moverna vas 1988	sample 123	056.2	3/2	rim and body of a pedestal dish (two fragments), type 7, red slip
149MV	59	Moverna vas 1988	sample 125	056.2	3/10	rim and body of a pedestal dish (two fragments), type 7, red slip
150MV	60	Moverna vas 1988	sample 126	056.2	3/5	rim of a pedestal dish, type 7, red slip
151MV-1	61	Moverna vas 1988	sample 135	031.4	6/12	black, compact residue, inside

Sample # CHEM	Sample # ARCH	Site code	SF #	SE/rel. depth	Sector/ quadrant	Description
151MV-2	61	Moverna vas 1988	sample 135	031.4	6/12	body of a pedestal dish, type 7, red slip, organic residue on interior
152MV-1	62	Moverna vas 1988	sample 136	050.2	5/3	black, compact visible residue, covering interior surface uniformly
152MV-2	62	Moverna vas 1988	sample 136	050.2	5/3	body of a pedestal dish, type 7, red slip, organic residue on interior
153MV	63	Moverna vas 1988	R232	022	5/11	body of a pot, horizontal rib and incised decoration, type 8
154MV	64	Moverna vas 1984	R115	1/8/7	1/0-5	body of a pot (two fragments), combed decoration, type 9
155MV	65	Moverna vas 1988	sample 129	056.2	3/5	rim of a ladle, type 10

App. 2. Summary of lipid residue analysis and their interpretations

Sample # CHEM	Vessel part	Biomarkers detected	Lipid concentration [μg g^{-1}]	P/S ratio	$\delta^{13}C_{16:0}$ [‰]	$\delta^{13}C_{18:0}$ [‰]	Interpretation
01AJ	body	/	2.38	/	/	/	n/a
02AJ	body	FA14, FA16, FA17, FA18, K29-K35, K33:1, K35:1	198.63	1.19	-25.26	-27.88	mixed animal fat, plant residue, cooking
03AJ	body	FA14, FA16, FA17, FA18, FA20-FA28; OH24-OH34; A27-A31, traces of WE	83.19	0.46	-26.44	-29.27	ruminant adipose fat, plant residue, waxes
04AJ	body	FA14, FA15, FA16, FA17, FA18, K27-K35	161.18	1.15	-25.45	-31.25	ruminant dairy fat, cooking
05AJ	body	/	0		/	/	n/a
06AJ	body	/	11.86	1.35	/	/	n/a
07AJ	body	FA14, FA16, FA18, OH24-OH32	7.9	1.10	/	/	plant residue
31AJ	body	/	4.43		/	/	n/a
32AJ	body	/	0		/	/	n/a
33AJ	body	/	2.16		/	/	n/a
34AJ	body	/	0		/	/	n/a
35AJ	base	FA16, A25-A31; OH24-OH32, traces of WE	12.94		/	/	plant residue
36AJ	body	/	2.66		/	/	n/a
37AJ	body	FA16, FA22-34; A25-A33; OH22-OH34, WE	99.93	3.30	/	/	plant residue, waxes
38AJ	rim	/	0		/	/	n/a
39AJ	rim	/	2.73		/	/	n/a
40AJ	body	/	3.97		/	/	n/a
41AJ	rim	/	2.79		/	/	n/a
42AJ	rim	/	3.89		/	/	n/a
43AJ	rim	/	0		/	/	n/a
44AJ	body	FA14, FA16, FA17,FA 18, DAGs, TAGs	80.45	0.59	-25.71	-28.99	mixed ruminant fat
45AJ	rim	/	1.08		/	/	n/a
46AJ	rim	/	0		/	/	n/a
47AJ	body	/	4.03		/	/	n/a
48AJ	body	FA14, FA16, FA18:1, FA18, FA20	7.05	3.40	-22.41	-27.40	ruminant dairy fat, plant residue
49AJ	rim	FA16, FA18, FA20	48	2.32	-25.91	-24.88	porcine fat
50AJ	body	/	5.15		/	/	n/a
51AJ	body	/	0		/	/	n/a
52AJ	body	/	0		/	/	n/a
53AJ	body	/	4.16		/	/	n/a
54AJ	rim	/	1.76		/	/	n/a
55AJ	body	/	7.34		/	/	n/a
56AJ	body	/	6.72		/	/	n/a
57AJ	base	A25-A33, OH24-OH34, FA22-FA30	267.04		/	/	plant residue, waxes
58AJ-1	body	/	0		/	/	n/a
58AJ-2	body	/	0		/	/	n/a
59AJ	body	/	4.66		/	/	n/a
60AJ	rim	FA14, FA16, FA18	21.96	2.27	-23.25	-28.47	ruminant dairy fat, plant residue
61AJ	rim	FA16, FA17, FA18, FA20, MAGs, DAGs, TAGs	47.46	0.65	-25.15	-29.05	ruminant dairy fat
62AJ	body	FA14, FA16, FA17, FA18, MAGs, A27-A33, OH24-OH32, DAGs, WE, TAGs	73.65	1.09	/	/	animal fat, waxes
63AJ	body	/	0		/	/	n/a
64AJ	rim	FA16, FA18	9.36	1.85	-23.29	-24.24	ruminant adipose fat
65AJ	body	FA16, FA18, DAGs, traces TAGs	13.37	2.64	/	/	mixed animal fat
66AJ	rim	/	0		/	/	n/a
67AJ	base	/	5.02		/	/	n/a

Sample # CHEM	Vessel part	Biomarkers detected	Lipid concentration [µg g-1]	P/S ratio	$\delta^{13}C_{16:0}$ [‰]	$\delta^{13}C_{18:0}$ [‰]	Interpretation
68AJ	body	FA16, FA18:1, FA18, traces WE	11.39	2.96	-25.91	-26.47	porcine fat, plant residue
69AJ	body	FA14, FA15, FA16, FA17, FA18, A25-A33, OH24-OH32, FA22-FA32, WE, DWE	557.13	2.20	/	/	plant residue, waxes
70AJ	body	FA14, FA16, FA18, A25-A33, OH24-OH34, FA22-FA30, WE, traces DWE	75.66	1.85	/	/	plant residue, waxes
71AJ	body	FA16, FA18:1, FA18, OH, DAGs, TAGs	42.94	0.82	-29.11	-31.78	ruminant adipose fat, plant residue
72AJ	body	FA16, FA18, K29-K35, K33:1, K35:1	47.37	0.80	-27.59	-30.08	mixed ruminant fat, plant residue, cooking
73AJ	body	/	1.46		/	/	n/a
74AJ	body	/	0		/	/	n/a
08MT	body	FA14, FA15br, FA15, FA16, FA17br, FA17, FA18:1, FA18, MAGs, DAGs, TAGs, traces of WE	173.35	0.99	-26.51	-29.91	ruminant dairy fat, plant residue
09MT	body	traces of FA16 & FA18, K31-K35	1.9		/	/	n/a
11MT	body	traces of FA16 & FA18, K31-K35	0.81		/	/	n/a
12MT	body	/	0.42		/	/	n/a
13MT	rim	FA16, FA18, MAGs, DAGs, TAGs, traces of WE, A and OH	11.56	0.63	-26.68	-28.85	ruminant adipose fat, plant residue
14MT	body	/	0.24		/	/	n/a
15MT	body	/	0		/	/	n/a
16MT	body	/	0		/	/	n/a
17MT	body	/	1.01		/	/	n/a
18MT	body	FA14, FA15br, FA15, FA16, FA17br, FA17, FA18:1, FA18, FA20-FA24, MAGs, DAGs, TAGs	88.09	0.75	-27.68	-33.26	ruminant dairy fat
75MT	rim	FA14, FA15, FA16:1, FA16, FA17, FA17br, FA18:1, FA18, FA20, MAGs, DAGs, TAGs	90.54	0.64	-28.96	-31.52	ruminant adipose fat
76MT	base	/	2.92		/	/	n/a
77MT	body	/	0		/	/	n/a
78MT	body	/	2.58		/	/	n/a
79MT	body	FA14, FA16, FA17, FA18:1, FA18, FA20, traces of A & OH, DAGs, TAGs	27.23	1.24	-27.80	-32.91	ruminant dairy fat, plant residue
80MT	rim	/	3.45		/	/	n/a
81MT	body	/	1.34		/	/	n/a
82MT	base	/	0		/	/	n/a
83MT	body	/	3.12		/	/	n/a
84MT	base	/	1.1		/	/	n/a
85MT	rim	/	1.34		/	/	n/a
86MT	rim	/	1.38		/	/	n/a
87MT	body	FA14, FA16, FA17br, FA17, FA18:1, FA18, FA20, MAGs, traces of A & OH, traces of DAGs & TAGs	21.93	1.05	-27.35	-32.18	ruminant dairy fat, plant residue
88MT	rim	FA14, FA16, FA18, FA20	9.93	2.56	-26.97	-31.91	ruminant dairy fat
89MT	rim	/	0		/	/	n/a
156MT	rim	FA16, FA18:1, FA18, traces of MAGs & DAGs	10.06	2.27	/	/	n/a
157MT	rim	/	4.06		/	/	n/a
158MT	rim	/	0.98		/	/	n/a
159MT	rim	FA16, FA18, MAGs, DAGs, TAGs	12.65	1.32	/	/	mixed animal fat
160MT	complete	FA16, FA18, traces of MAGs, DAGs & TAGs	5.53	3.46	/	/	n/a
161MT	rim	FA16, FA18:1, FA18, MAGs, traces of DAGs	43.81	1.11	-29.38	-33.95	ruminant dairy fat
162MT	n/a	/	2.77		/	/	n/a
163MT	body	FA16, FA18	4.02		/	/	n/a
164MT	rim	FA16, FA18	2.47		/	/	n/a
165MT	rim	/	1.53		/	/	n/a
166MT	rim	/	4.67		/	/	n/a
23MV-1	rim	FA16, FA18, traces OH	361.4	0.30	/	/	animal fat
23MV-2	rim	FA14, FA16, FA17br, FA17, FA18, FA20, OH24-OH34, A29-A35, WE	434.99	0.25	-26.99	-30.46	mixed fat, waxes
24MV-1	base	pitch markers	1789.97		/	/	birch bark tar

Sample # CHEM	Vessel part	Biomarkers detected	Lipid concentration [µg g-1]	P/S ratio	$\delta^{13}C_{16:0}$ [‰]	$\delta^{13}C_{18:0}$ [‰]	Interpretation
24MV-2	base	traces of pitch markers	0.77		/	/	n/a
25MV-1A	base	pitch markers, traces of WE	720.94		/	/	birch bark tar, waxes
25MV-1B	base	pitch markers, traces of WE	911.02		/	/	birch bark tar, waxes
25MV-2	base	pitch markers, traces of WE	31.06		/	/	birch bark tar, waxes
26MV-1	base	pitch markers	1539.77		/	/	birch bark tar
26MV-2	base	FA10, FA12, FA14, FA16, FA18, pitch markers, WE	24.3	1.48	/	/	birch bark tar, waxes
27MV-1A	base	FA16, FA18, pitch markers	3308.1	1.33	/	/	birch bark tar
27MV-1B	base	FA16, FA18, pitch markers	118.18	1.34	/	/	birch bark tar
27MV-1C	base	FA16, FA17, FA18:1, FA18, pitch markers	740.32	1.71	/	/	birch bark tar, plant residue
27MV-2	base	/	0		/	/	n/a
28MV-1A	base	FA16, FA18, pitch markers, WE	3056.3	1.32	/	/	birch bark tar, plant residue, waxes
28MV-1B	base	FA16, FA18, pitch markers, WE	542.58	1.30	/	/	birch bark tar, plant residue, waxes
28MV-1C	base	FA16, FA18, pitch markers, WE	1261.79	1.68	/	/	birch bark tar, plant residue, waxes
28MV-2	base	FA16, A, OH, traces of WE	5.25		/	/	plant residue
29MV-1A	rim	/	18.03		/	/	n/a
29MV-1B	rim	FA16:1, FA16, FA18:1, FA18	8.06	1.89	/	/	plant residue
29MV-2	rim	FA14, FA16:1, FA16, FA17, FA18:1, FA18, FA20-FA24, K31-K35	27.57	1.02	/	/	mixed animal and plant residue, cooking
30MV-1	base	/	0		/	/	n/a
30MV-2	base	FA16, MAG16, DAG32, TAG54	17.69		/	/	plant residue
90MV	body	/	0		/	/	n/a
91MV	body	FA16, FA17, FA18-FA24, K29-K35	156.62	0.31	-27.53	-31.65	ruminant dairy fat, cooking
92MV	rim	FA14, FA16, FA18, FA20, MAGs, traces of DAGs and TAGs	29.41	1.02	-27.75	-30.35	mixed ruminant fat
93MV	body	FA14, FA16, FA17, FA18:1, FA18, MAGs, DAGs, TAGs	119.17	0.77	-27.92	-31.36	mixed ruminant fat
94MV	body	FA16, FA18, FA20	15.2	0.57	-28.41	-30.89	ruminant adipose fat
95MV	body	FA16, FA18, MAGs, traces of DAGs and TAGs	5.5	1.19	-26.65	-28.90	mixed animal fat
96MV	body	FA14, FA16, FA18, FA20, MAGs, DAGs, TAGs	9.51	0.81	-27.18	-31.09	dairy fat
97MV	body	FA16,FA18	10.52	0.92	/	/	mixed animal fat
98MV	rim	FA12, FA14, FA16, FA17br, FA17, FA18, MAGs, traces of A & OH, DAGs, traces of WE, TAGs	57.85	0.69	-28.62	-30.75	ruminant adipose fat, plant residue, waxes
99MV	body	FA16, FA18, A25-A33, OH24-OH34, FA24-FA32, WE, DWE	429.65	6.20	-26.09	-29.62	dairy fat, plant residue, waxes
100MV	rim	FA16, FA18, A25-A31, MAGs, OH24-OH34, DAGs, WE, DWE	449.81	0.57	-27.47	-29.44	ruminant adipose fat, waxes
101MV	body	/	0		/	/	n/a
102MV	rim	traces of FA16 & FA18, A25-A33, OH24-OH34, FA24-FA34, WE, DWE	399.78	2.86	-26.20	-33.48	plant residue, waxes
103MV	body	FA16, FA18, MAGs, traces of A&OH, DAGs, traces of WE	4.27		/	/	plant residue
104MV	body	FA14, FA16, FA17, FA18	214.36	0.65	-27.66	-30.22	ruminant adipose fat
105MV	body	/	0		/	/	n/a
106MV	body	FA16, FA18, traces of A&OH	4.51	2.15	/	/	plant residue
107MV	body	FA16, FA18	29.38	0.67	-26.68	-28.39	mixed animal fat
108MV	base	/	0		/	/	n/a
109MV	body	FA16, FA17, FA18	126.6	0.90	-26.14	-26.19	porcine fat
110MV	rim	FA16, DA17, FA18:1, FA18, FA20	1219.85	1.17	-24.90	-24.96	porcine fat
111MV	base	FA12, FA14, FA16, FA17, FA18:1, FA18, MAG16, A27-A33, OH24-OH34, FA24-FA28, WE	177.63	2.47	-25.40	-25.73	porcine fat, plant residue, waxes
112MV	rim	traces of A&OH, traces of DAGs, WE	5.22		/	/	plant residue
113MV	rim	traces of FA16 & FA18, A23-A33, OH24-OH34, WE	47.2		-26.36	-27.85	plant residue, waxes
114MV	base	FA16, FA18, A23-A33, OH22-OH34, FA24-FA30, WE	94.85	2.89	/	/	plant residue, waxes

Sample # CHEM	Vessel part	Biomarkers detected	Lipid concentration [μg g-1]	P/S ratio	$\delta^{13}C_{16:0}$ [‰]	$\delta^{13}C_{18:0}$ [‰]	Interpretation
115MV	body	/	0		/	/	n/a
116MV	body	FA16, FA18, A23-A33, OH22-OH34, FA26, WE	145.68	3.24	-26.68	-28.75	plant residue, waxes
117MV	body	/	0		/	/	n/a
118MV	body	/	0		/	/	n/a
119MV	body	/	0		/	/	n/a
120MV	body	FA16, FA17, FA18:1, FA18, FA20	197.58	0.81	-25.18	-25.15	porcine fat
121MV	body	A25-A33, OH24-OH34, FA24-FA32, WE	970.81		-26.89	-26.03	porcine fat, plant residue, waxes
122MV	body	traces of FA16 & FA18	2.18		/	/	n/a
123MV	body	/	0		/	/	n/a
124MV	body	FA14, FA16, FA17, FA18:1, FA18, FA20	503.45	1.65	-24.24	-23.99	porcine fat
125MV	body	A23-A31, OH24-OH32, traces of WE	12.57		/	/	waxes
126MV	rim	traces of A&OH	1.4		/	/	n/a
127MV	body	traces of A&OH	1.74		/	/	n/a
128MV	body	/	0		/	/	n/a
129MV	body	/	1.07		/	/	n/a
130MV	body	/	0		/	/	n/a
131MV-1	body	/	0		/	/	n/a
131MV-2	body	/	0		/	/	n/a
132MV	body	FA16, FA17, FA18	13.45	0.60	-26.57	-29.84	mixed ruminant fat
133MV	rim	FA14, FA16, FA17br, FA17, FA18, K	17.61	0.88	/	/	mixed animal fat, cooking
134MV	rim	FA14, FA16, FA17br, FA17, FA18, MAGs, OH22-OH32, A29-A31, DAGs, WE, TAGs	289.58	0.54	-28.35	-29.94	ruminant adipose fat, plant residue, waxes
135MV	body	FA16, FA17, FA18, MAGs, traces of DAGs & TAGs	47.71	1.41	-27.47	-26.99	porcine fat
136MV	body	/	1.06		/	/	n/a
137MV	body	FA14, FA16, FA18	14.27	0.46	-28.64	-31.15	ruminant adipose fat
138MV	body	/	0		/	/	n/a
139MV	body	/	0		/	/	n/a
140MV	body	FA16, FA18, traces of A&OH; traces of DAGs&WE	4.14	1.12	/	/	mixed residue
141MV	rim	/	0		/	/	n/a
142MV	base	/	0		/	/	n/a
143MV	rim	FA16, FA18, MAGs, OH22-OH32, A27-A33, DAGs, TAGs	6.17	0.91	-27.66	-30.71	ruminant adipose fat, plant residue
144MV	base	FA16, FA18, FA20	9.76	1.45	-26.38	-29.28	ruminant adipose fat
145MV	body	traces of FA16 & FA18, A27-A31, OH22-OH32, traces of DAGs, WE & TAGs	6.82		/	/	mixed residue
146MV	base	/	0		/	/	n/a
147MV	base	FA12, FA14, FA15, FA16, FA17, FA18:1, FA18, FA20-FA30, MAGs, A25-A33, OH22-OH32, DAGs, WE	37.61	0.53	/	/	mixed animal and plant residue, waxes
148MV	rim	/	0		/	/	n/a
149MV	rim	FA14, FA16, FA17br, FA17, FA18, FA20-FA26, MAGs, A23-A33, OH22-OH32, DAGs, traces of TAGs	20.49	0.76	-26.71	-29.22	ruminant adipose fat, plant residue
150MV	rim	FA16, FA17, FA18, MAGs, A27-A33, OH24-OH32, DAGs, traces of TAGs	9.63	0.73	-27.42	-30.82	ruminant fat, plant residue
151MV-1	body	traces of FA16 & FA18, pitch markers	2918.51		/	/	birch bark tar
151MV-2	body	FA16, FA18, pitch markers	91.85	0.44	/	/	animal fat, birch bark tar
152MV-1	body	traces of FA16 & FA18, pitch markers, traces of TAGs	2818.76	0.91	/	/	mixed animal fat, birch bark tar
152MV-2	body	/	0		/	/	n/a
153MV	body	FA14, FA15, FA16, FA17br, FA17, FA18:1, FA18, FA20, MAGs, DAGs, traces of TAGs	55.01	0.76	-27.70	-29.97	ruminant adipose fat
154MV	body	FA14, FA15br, FA15, FA16, FA17br, FA17, FA18, FA20-FA28, MAGs, A23-A33, OH22-OH32, traces of DAGs & TAGs, WE	607.11	0.29	-27.96	-31.10	ruminant fat, plant residue, waxes
155MV	rim	FA14, FA16, FA17, FA18:1, FA18, traces of DAGs & TAGs	7.15	0.56	-29.17	-31.37	ruminant adipose fat