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Lipid biomarkers and compound specific $\delta^{13}C$ analysis indicate early development of a dual-economic system for the Arctic Small Tool tradition in northern Alaska

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ABSTRACT

Analysis of preserved lipids from archaeological sites in northwest Alaska indicates hunters exploited marine animal resources as early as 4500 years ago. Bone preservation at early prehistoric sites in northern Alaska is generally poor, contributing to uncertainty about the economic orientation of the earliest Arctic Small Tool tradition (ASTt) hunters. We used lipid analysis and compound specific stable isotope analysis (CSIA) of burned, cemented sand and organic residue features to detect the use of marine versus terrestrial animals at several coastal sites in northwest Alaska. Though the sample size for this initial study was small ($n = 5$), comparisons among samples from early ASTt, and later Norton and Thule sites indicate all three groups made use of marine animals for food and/or fuel. Recently obtained radiocarbon dates suggest ASTt hunters settled coastal regions of Alaska prior to moving inland to exploit terrestrial habitats. Our results provide empirical evidence that suggests the economy of the early ASTt population included a maritime component. In Arctic settings where bone preservation is poor, lipid analysis of cemented sand and organic residue features can provide an effective alternative for detecting the use and processing of marine versus terrestrial animals.

1. Introduction

Development of a dual economy, wherein resources from both land and sea were exploited, represents a defining moment in arctic prehistory, as this system arguably became the foundation for successful adaptations to the circumpolar environment (e.g., McCartney and Helmer, 1989). Yet, the origin of this dual economy, and the maritime adaptations that accompanied it, remain a mystery. The first people to colonize the North American Arctic and routinely make use of coastal habitats were hunters bearing tools of the Arctic Small Tool tradition (ASTt) (Ackerman, 1998; Giddings, 1964; Giddings and Anderson, 1986; McGhee, 1996). Archaeological and genetic research has established that this represents a migration from Siberia across the North American Arctic beginning around 5000 years ago (Dumond, 1987; Mochanov, 1969; Powers and Jordan, 1990; Raghavan et al., 2014; Rasmussen et al., 2010). Despite the implication that ASTt colonists had to cross the 80 km wide Bering Strait into Alaska, many questions remain unanswered concerning the timing and extent of their maritime subsistence adaptations, including their nautical capabilities. No ASTt sites are known from coastal contexts on the west side of the Bering Strait; and in Alaska, many archaeologists consider ASTt maritime adaptations as incidental or as an early developmental stage (Ackerman, 1998; Dumond, 1975, 1982; Giddings, 1964; Giddings and Anderson, 1986).

Recent research has demonstrated that the earliest ASTt sites across Alaska (locally referred to as the Denbigh Flint complex) and the eastern Arctic of Canada and Greenland appear in coastal contexts (e.g., Grønnow, 1994; Maschner et al., 2010; Savelle and Dyke, 2002; Slaughter, 2005; Tremayne, 2014) prompting renewed interest in the timing and development of ASTt maritime adaptations. If coastal sites predate settlement of interior habitats, then use of marine resources may have played a larger role in the ASTt colonization process than once thought. However, the mere

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presence of early sites in coastal settings does not assure a maritime adaptation, as terrestrial game was also available in these environments. Poor preservation of bone—inhibiting the reconstruction of past organic hunting technologies as well as restricting faunal analyses—is one factor fueling continued debate. Without bones, harpoons, or boating technology, we lack empirical evidence of sea mammal exploitation by early coastal groups in arctic Alaska. Were ASTt people on the coast following migrating caribou herds and muskox, or were marine resources their targeted prey?

In arctic settings where bone preservation is poor, lipid analysis of organic residues from archaeological features can provide an effective and practical alternative for detecting the use and processing of animal fats (Farrell et al., 2014; Heron, 2010; Kedrowski et al., 2009; Morgan et al., 1984). We used gas chromatography/mass spectrometry (GCMS) and compound specific stable isotope analysis (CSIA) of lipid extracts to detect marine animal versus terrestrial animal processing in several samples of sand cemented with fatty organic residues, spanning the ASTt through the late Thule cultures. Cemented sand features occur in the earliest (Kedrowski et al., 2009) through the latest northern Alaskan sites (Giddings and Anderson, 1986; Morgan et al., 1984; Schaaf, 1988). In younger sites, such features are assumed to be deposits of marine mammal oil (Harritt, 1994; Schaaf, 1988), while a recent study suggests that some of the earliest known cemented sand deposits in Interior Alaska resulted from burning bones of large terrestrial mammals (Kedrowski et al., 2009). In fact, little research has focused on the formation and organic composition of cemented sand deposits in this region. Thus, we saw an opportunity to (1) determine which animal products contribute to its formation, and (2) to use these data to elucidate subsistence activities of people at Cape Espenberg, Alaska over the last 4500 years (Fig. 1). This study seeks to address several questions. First, do any of the cemented sand deposits contain lipids from marine animals? Second, if marine lipids are present, are they mixed with lipids from terrestrial sources? Finally, do the residues indicate changes in the use of animal species over time at this locality?

2. Sites and cemented sand samples

Samples were collected from four archaeological sites at Cape Espenberg, located in Bering Land Bridge National Preserve, Alaska (Fig. 1). Cape Espenberg is comprised of a series of beach and dune ridges that formed sequentially beginning around 5000 years ago (Mason et al., 1997). The progradation of sand ridges and dunes allows archaeologists to exploit their "horizontal stratigraphy" to aid in the development of culture histories based on the chronological sequence of occupations (Giddings and Anderson, 1986; Mason, 1993). The cultural sequence at Cape Espenberg includes ASTt, followed by the ceramic bearing Norton tradition, and, finally, by the fully maritime-adapted Thule tradition (Schaaf, 1988).

Surveys in 2011 and 2013 at Cape Espenberg led to the discovery of 10 new ASTt and four new Norton sites, and extended the chronology for settlement of the oldest beach ridges (Tremayne, 2014). Cultural affiliation for each occupation was determined from diagnostic artifacts and/or radiocarbon dates. While some prefer to include Norton as a late ASTt manifestation (e.g., Anderson, 1980), we follow Dumond (1982, 1987) and make a distinction between aceramic ASTt components and ceramic-bearing Norton deposits. Osteofaunal remains documenting subsistence activities were not preserved at the ASTt or Norton sites, but their remains are abundant in Thule sites.

Cemented sand deposits are a common feature at many sites at Cape Espenberg (Harritt, 1994; Schaaf, 1988). Typically, these deposits occur as small patches of dark brown to black, cobble to pebble-sized nodules of consolidated sediments and fatty organic material (Fig. 2), although 8 cm thick beds extending across a meter have been reported (Harritt, 1994:132). Charcoal, bone fragments, and lithic microdebitage are embedded in samples confirming a formation due to cultural processes. Cemented sand deposits are also common in activity areas of Thule houses interpreted as kitchen alcoves (Darwent et al., 2013, Supplemental Fig. 12). Because this material occurs in a cultural zone where sea mammal fat was definitely used, based on faunal remains dominated by sea

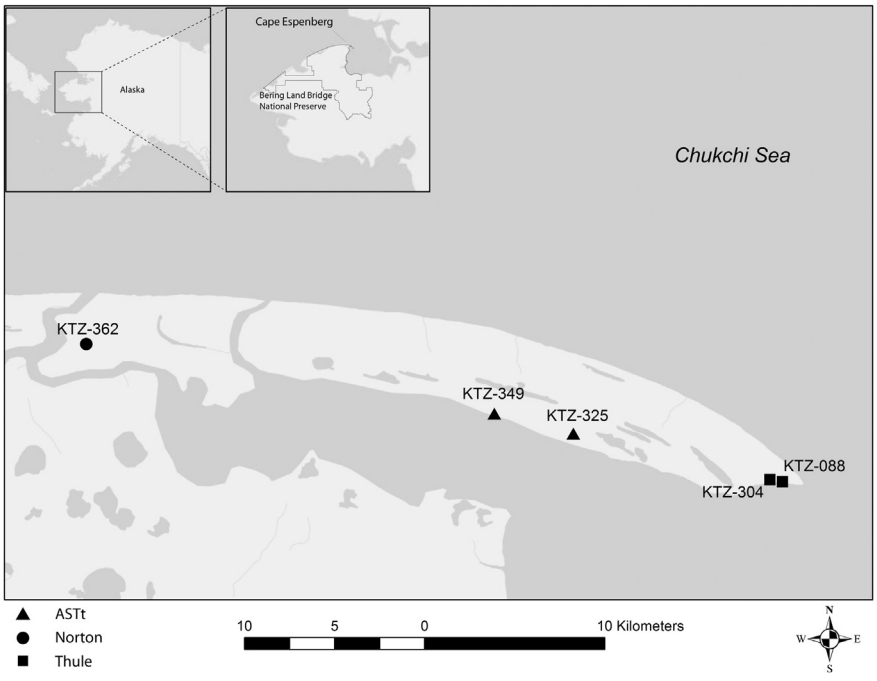

Fig. 1. The location of the study area within Alaska and the sites where samples were collected at Cape Espenberg.

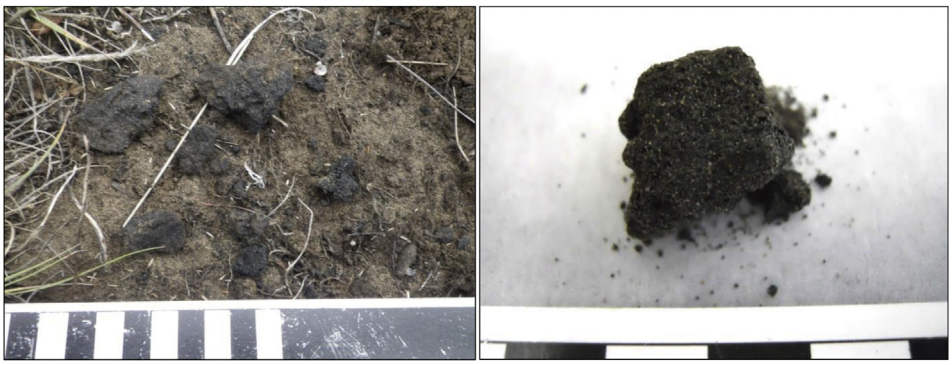

Fig. 2. Images of cemented sand from KTZ-325: in the field (left) and in the lab (right) (scale is in centimeters).

mammals and associated hunting technology, archaeologists have assumed they formed from fat rendering or spillage of rendered seal oil. While this assumption may be warranted for Thule houses, no studies have demonstrated that this holds true for older sites, nor ruled out the possibility that terrestrial mammal fats can create similar features. Ethnographic accounts of caribou bone processing have revealed that significant amounts of grease can be produced and stored for later use (e.g., Binford, 1978; Burch, 2006:138). We are not aware of caribou bone grease being used for fuel in the same way that sea mammal oil was, for burning for light and heat, but fresh bones of large terrestrial mammals were used as a fuel source by Paleolithic hunters (Kedrowski et al., 2009; Théry-Parisot, 2002), and by early ASTt inhabitants of northern Greenland (Darwent, 2001, 2003).

Two recent studies explain how deposits of cemented sand and fatty organic residues could form from terrestrial or marine fats, and in different combustion contexts (Grønnow et al., 2014; Kedrowski et al., 2009). Kedrowski et al. (2009) compared the fatty acid profile and physical appearance of cemented sand deposits recovered from the Swan Point site in central Alaska to those created in a laboratory experiment where red deer (*Cervus elaphus*) bones were burned on a sandy matrix. The fatty acid profile and physical appearance of the experimentally produced cemented sand were similar to those observed in the Swan Point archaeological samples (Kedrowski et al., 2009).

A different study by Grønnow et al. (2014) used archaeological and experimental work to suggest that early ASTt hunters in Greenland may have burned blubber and oil in box hearths—small, stone-lined hearths. Experimentally reproduced box hearths, and those from well-preserved ASTt sites in Greenland, both contained layers of sand and gravel cemented together with fatty material (Grønnow et al., 2014:405–409). These authors propose that box hearths were functional precursors to later lamps, providing a concentrated source of heat and light suitable for small, enclosed spaces. They argue that burning blubber may have been a crucial prerequisite for survival in high Arctic settings, especially during winter months (Grønnow et al., 2014:405–407). If the earliest ASTt hunters in northern Alaska already used marine mammal blubber in this way, it would seem to imply the existence of an integrated and well-developed maritime adaptation among initial ASTt colonizers.

Samples for this study were collected from two ASTt sites (KTZ-325 and KTZ-349), one Norton site (KTZ-362), one early Thule house (Feature 21, KTZ-304) and one late Thule house (Feature 33, KTZ-088). Sample descriptions and associated radiocarbon ages are listed in Table 1. The ASTt and Norton samples were collected by Tremayne during survey and testing of the oldest beach ridges. Prior to this work, no ASTt sites at Cape Espenberg were known to have associated cemented sand deposits. The Thule samples were collected during excavation of house features in 2010, and were made available by the Cape Espenberg Project (Hoffecker and Mason, 2010, 2011).

3. Classification of lipid sources

Recent experimental and archaeological work has made it possible to identify marine lipids in archaeological contexts (Copley et al., 2004; Craig et al., 2011; Evershed et al., 2008; Hansel et al., 2004; Heron et al., 2010, 2013). Widely-accepted biomarker criteria for heating marine lipids in pottery vessels currently includes a combination of at least one of three isoprenoid fatty acids—4,8,12-trimethyltridecanoic acid (4,8,12-TMTD), 4,8,12,16-tetramethylpentacecanoic acid (pristanic acid), and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid)—together with, ω-(*o*-alkylphenyl)alkanoic acids of at least 18 and 20 (and preferably, also 22) carbons (Evershed et al., 2008). Combining marine biomarkers with compound specific stable isotope analysis (CSIA), can provide even stronger evidence for the processing of marine fauna. Several studies have taken this latter approach to identify the processing of marine products in pottery cooking vessels (Copley et al., 2004; Craig et al., 2011, 2013; Cramp et al., 2014; Taché et al., 2015). These latter studies have identified marine products using combinations of one or more isoprenoid fatty acids, ω-(*o*-alkylphenyl)alkanoic acids ranging from 16 to 22 carbons long, and δ^{13}C C16:0 and δ^{13}C C18:0 values.

While recent organic residue studies have made great progress identifying the processing of marine products in archaeological contexts, they have tended to focus on pottery sherds. A recent study by Heron et al. (2010), however, analyzed lipids extracted from cemented sand and organic residue, charcoal, and sediments from Iron Age slab-lined pits in Arctic Norway. These authors used the presence of isoprenoid fatty acids, ω-(*o*-alkylphenyl)alkanoic acids, and bulk δ^{13}C values to identify the processing of marine oils in the slab-lined pits. Earlier studies of fatty deposits in features from arctic sites focused on overall distributions of unsaturated and saturated fatty acids, which can be far more equivocal due to differences in patterns of degradation (Morgan, 1973; Morgan et al., 1983, 1984).

To classify lipid sources in the current study we considered a variety of data including ratios of certain saturated fatty acids, δ^{13}C values for C16:0 and C18:0, and the presence of several marine biomarker compounds. Marine biomarker compounds include the isoprenoid fatty acids 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD) and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid), as well as, ω-(*o*-alkylphenyl)alkanoic acids 18, 20 and 22 carbons long. We also noted the presence of α,ω-dicarboxylic acids

Table 1
Contextual information for cemented sand samples recovered from sites on Cape Espenberg, Alaska.

Sample ID	Site#	Feat#	Lab#	Associated date (BP)	Calibrated date range	Sample description
Late Thule	KTZ-088	33	Beta-286170[a]	120 ± 40	1675–1942 AD	Dark brown consolidated sand
Early Thule	KTZ-304	21	Beta-286169[a]	640 ± 30	1281–1400 AD	Dark brown to black consolidated
			Beta-286168[a]	680 ± 30	1263–1394 AD	sand and organic material
Norton	KTZ-362	NA	AA102994[b]	2154 ± 39	360–110 BC	Brown consolidated sand
ASTt-1	KTZ-325	NA	Beta-305875[b]	3880 ± 30	2460–2300 BC	Dark brown consolidated sand and organic material
			Beta-305874[b]	4100 ± 30	2860–2570 BC	
ASTt-2	KTZ-349	NA		Diagnostic ASTt tools		Black charred material

[a] Darwent et al., 2013.
[b] Tremayne 2014.

ranging from carbon chain lengths of C7–C12 and the presence of long-chain fatty acids. The utility of these data for classifying source material is discussed briefly below.

3.1. Saturated fatty acids

Fatty acids are the most abundant class of lipids encountered in archaeological materials and are designated here using the shorthand notation C*x*:*y*, where *x* indicates the carbon chain length and *y* is the degree of unsaturation (number of C–C double bonds). Proportions of certain saturated fatty acids can play a role in suggesting major contributions from broad resource categories such as terrestrial mammal, plant or fish, but should be used cautiously and as part of a larger framework of identification due to complexities of degradation and potential mixing of sources. Though sources of fresh plant and animal fats can be distinguished based on their fatty acid content, over time these compounds degrade at different rates and through a variety of mechanisms (Heron and Evershed, 1993; Morgan et al., 1973). In particular, unsaturated fatty acids are far more susceptible than saturated fatty acids to oxidation due to increased reactivity at the site of double bonds. As the number of double bonds increase in polyunsaturated fatty acids, so do their rates of degradation (Christie, 2003:93). Hence, ancient fatty materials are typically dominated by several common saturated fatty acids (especially C16:0 and C18:0) with lower amounts of some mono- or even di-unsaturated fatty acids, while polyunsaturated acids are typically absent.

To account for some of these transformations, Eerkens (2005) proposed using ratios of fatty acids with similar chain lengths and degrees of unsaturation to separate very general resource categories. Using discriminant analysis of modern and experimentally aged residues, Eerkens demonstrated that ratios of C16:0 to C18:0 versus C12:0 to C14:0, and C16:1 to C18:1 versus C15:0 + C17:0 to C18:0 correctly classified a sample of residues as terrestrial mammals, fish, seeds, greens or roots about 72% of the time (2005:91). Additional experimental studies have supported the utility of Eerkens' (2005) saturated fatty acid ratios (C16:0 to C18:0 versus C12:0 to C14:0) for separating terrestrial animal or plant resources (Buonasera, 2013; Kedrowsi et al., 2009). Here, we used Eerkens' (2005:91) saturated fatty acid ratios (C16:0 to C18:0 versus C12:0 to C14:0) to assist in the identification of the original sources of archaeological residues (Table 2). Since marine mammal fats were not part of the original criteria, we also compiled literature values for modern marine fats and wild ruminant animal fats and plotted these with respect to the archaeological samples (Fig. 3).

3.2. Biomarkers

Isoprenoid fatty acids 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD) 2,6,10,14- tetramethylpentadecanoic acid (pristanic acid), and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid) are present in high amounts in marine animal fats; they are rarely encountered, and in only very low amounts, in terrestrial mammals, and are not present in plant oils (Ackman and Hooper, 1968; Ackman, 1989; Copley et al., 2004; Evershed et al., 2008). Furthermore, Evershed et al. (2008) and Copley et al. (2004), report that isoprenoid fatty acids have not been encountered in thousands of sherds from inland sites that have been processed in their labs.

In marine systems, phytol, present in the chlorophyll of photosynthesizing organisms, is biologically modified to 4,8,12,TMTD, phytanic acid, and pristanic acid as it moves through the marine food web (Ackman, 1989:23). The presence of one or more of these isoprenoid fatty acids is used to detect the processing of marine products in archaeological pottery and features (Copley et al., 2004; Cramp et al., 2014; Farrell et al., 2014; Hansel et al., 2004; Heron et al., 2010).

In addition to isoprenoid fatty acids, the presence of ω-(*o*-alkylphenyl)alkanoic acids with 18, 20 and 22 carbons provides evidence that marine products were processed in the presence of heat. Experiments indicate that these compounds form when triunsaturated and other unsaturated fatty acids are exposed to temperatures above 270 °C in an anoxic environment (Evershed et al., 2008:105). Unlike terrestrial mammal fats, marine fats/oils

Table 2
Fatty acid ratios used to distinguish different food types, from Eerkens (2005:91).

Ratio	State	Terrestrial mammals	Fish	Roots	Greens	Seeds and nuts	Berries
$\frac{(C15:0 + C17:0)}{C18:0}$	Fresh	<0.2	0.2–0.5	>0.2	0.1–1.0	<0.6	<0.2
	Degraded	<0.2	0.2–0.5	>0.2	0.1–1.0	<0.6	<0.2
$\frac{C16:1}{C18:1}$	Fresh	0.02–0.2	0.2–0.5	0.05–0.7	>0.7	<0.03	<0.08
	Degraded	0.08–0.8	0.8–2.0	0.2–2.8	>2.8	<1.2	<0.32
$\frac{C16:0}{C18:0}$	Fresh	<3.5	4–6	3–12	5–12	0–9	2–6
	Degraded	<7	8–12	6–24	10–24	0–18	4–12
$\frac{C12:0}{C14:0}$	Fresh	<0.15	<0.15	>0.15	>0.05	>0.15	>0.15
	Degraded	<0.15	<0.15	>0.15	>0.05	>0.15	>0.15

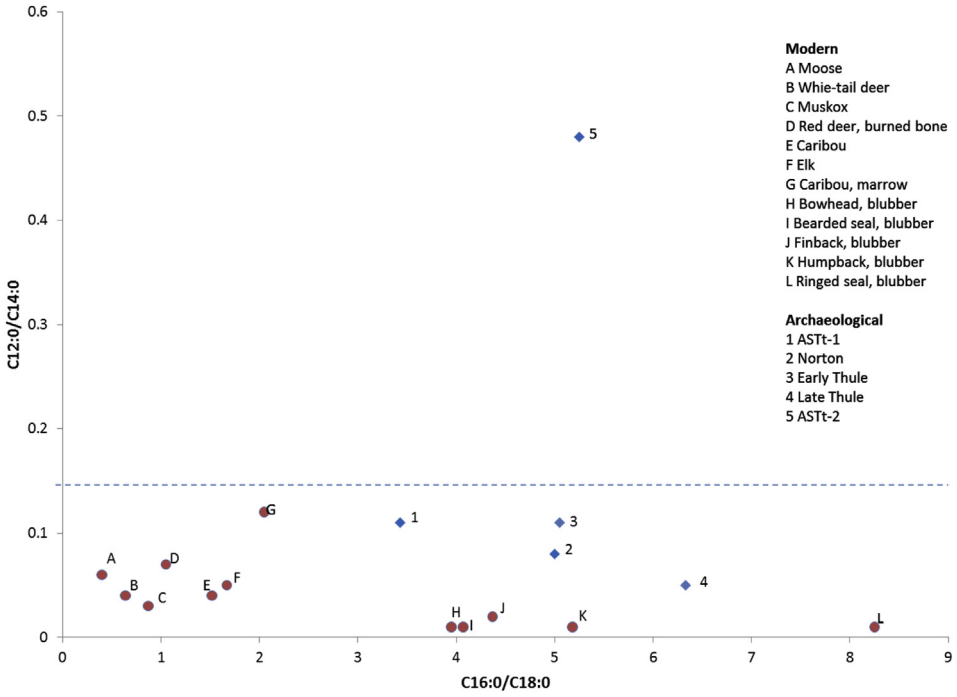

Fig. 3. Ratios of saturated fatty acids in modern marine mammal and wild ruminants plotted with archaeological samples. The dotted line represents an experimentally derived cut-off for plant lipids (C12:0/C14:0 > 0.15) or terrestrial animals and fish (C12:0/C14:0 < 0.15) (Eerkens, 2005). Modern references are literature values for adipose tissues and, where specified, bone lipids. References for species plotted here are as follows: Borobia et al., 1995 (finback and humpback whales); Budge et al., 2008 (bowhead whale, C16:0/C18:0); Dugan et al., 2007 (muskox); Garton et al., 1971 (caribou, elk, moose, white-tail deer); Innis and Kuhnlein, 1987 (bearded seal and ringed seal, C16:0/C18:0); Kedrowski et al., 2009 (lipids from red deer bones burned as fuel); Meng et al., 1969 (caribou marrow lipids). The ratio of C12:0/C14:0 was estimated for bowhead whales based on other baleen whales (Borobia et al., 1995). The ratio of C12:0/C14:0 was estimated for bearded seals and ringed seals based on values for reported for seals from the Baltic in Käkelä et al. (1993).

have high amounts of polyunsaturated fatty acids that are 20 and 22 carbons long. Heating these fats in pottery vessels produces ω-(*o*-alkylphenyl)alkanoic acids 20 and 22 carbons long (Evershed et al., 2008).

Detection of α,ω-dicarboxylic acids (sometimes referred to *di-acids*) can provide further evidence that substantial amounts of unsaturated fatty acids were once present in a residue (Buonasera, 2013; Passi et al., 1993; Regert et al., 1998). Evershed et al. (2008:106) found that α,ω-dicarboxylic acids between eight and 11 carbons long were formed "in appreciable amounts" during experimental heating of marine oils. These compounds are formed from the oxidation of C—C double bonds and their length may be representative of double bond positions in the original unsaturated fatty acids (Evershed et al., 2008; Passi et al., 1993).

3.3. Bulk and compound specific $\delta^{13}C$ analysis

Marine animal fats are significantly more enriched in ^{13}C than terrestrial animal fats, allowing for discrimination between these resources (Copley et al., 2004; Craig et al., 2007, 2011; Cramp et al., 2014). Bulk $\delta^{13}C$ analysis is a blunter tool than CSIA as it provides a value for the entire residue, which can include charred plant matter and bone as well as fatty organic residue. Application of CSIA to ancient lipids compares $\delta^{13}C$ values for C16:0 and C18:0 to those from modern reference fats, adjusted to account for contributions of industrial carbon (Craig et al., 2011:17914; Regert, 2011:196). Palmitic (C16:0) and stearic (C18:0) acids are used in these comparisons because they are typically the most abundant lipids encountered in ancient organic residues. The stable carbon isotope values for C16:0 and C18:0 can also provide a means of detecting and estimating mixtures of marine and terrestrial animal fats (Craig et al., 2011). Terrestrial animal fats contain substantially greater quantities of C18:0 than marine fats, so in cases where the two are mixed and show intermediate values, $\delta^{13}C$ values for C18:0 should be more affected (and appear less enriched) than $\delta^{13}C$ values for C16:0.

4. Analytical protocol

4.1. Extraction and derivatization

Samples were extracted using a modified Bligh and Dyer method (Bligh and Dyer, 1959; Buonasera, 2007) and total lipid extracts were derivatized to methyl esters. Laboratory protocol used for extraction and derivatization are provided in supplementary information. Prepared samples were analyzed via GC/MS within 24 h. Portions of these samples were sent to the Stable Isotope Facility (SIF) at the University of California, Davis for CSIA analysis of Fatty Acid Methyl Esters (FAMEs).

4.2. Gas chromatography/mass spectrometry

Samples were analyzed on an Agilent 6890N gas chromatograph coupled with a 5973MSD, and signals were integrated using HP Chemstation software. These instruments were available in the Chemistry Department at UC Davis for interdepartmental research. The GC was fitted with a VF-5ms, 30 m × 0.25 mm column (Varian CP8944), the carrier gas was He, and the inlet temperature was set to 285 °C. Samples (1 μL) were autoinjected and split 1:10. After a 1 min isothermal hold at 40 °C, the temperature was ramped to 120 °C at 20 °C per minute, then to 280 °C at 4 °C per minute with a 10 min isothermal hold at 280 °C. Ions were generated using electron impact (EI, 70 eV) and the mass spectrometer was operated in scan mode, detecting ions between 50 and 650 *m*/*z*. Lipid compounds were identified by comparing mass spectra to the NIST Standard Reference Database. Fatty acids were also identified and quantified

based on comparisons to standard reference compounds run the same day on the same instrument (Supelco SP-37 FAME mixture).

Detection of ω-(*o*-alkylphenyl)alkanoic acids 18, 20, and 22 carbons long was accomplished by analyzing extracted mass spectra for selected ions. The compounds were identified by the presence of a dominant ion at *m/z* 105 along with M+ ions for C18 (*m/z* 290), C20 (*m/z* 318) and C22 (*m/z* 346) ω-(*o*-alkylphenyl)alkanoic acids (Evershed et al., 2008; Hansel et al., 2004; Heron et al., 2010). The dominant ion at *m/z* 105 represents a dialkyl benzene fragment, $C_8H_9^+$, common to all ω-(*o*-alkylphenyl)alkanoic acids (Michael, 1966).

4.3. Bulk stable isotope analysis and CSIA of the lipid extract

Portions (12–15 mg) of several cemented sand samples (Early Thule, Late Thule, and ASTt-1) were dried and weighed into tins and submitted to the Stable Isotope Facility (SIF) at UC Davis for bulk $\delta^{13}C$ and $\delta^{15}N$ analysis (http://stableisotopefacility.ucdavis.edu/). Samples were analyzed using an Elementar Vario EL Cube or Micro Cube elemental analyzer interfaced with a PDZ Europa 20–20 isotope ratio mass spectrometer. During analysis, samples were interspersed with several replicates of at least two laboratory standards. Laboratory standards were selected to be compositionally similar to samples, and were previously calibrated against NIST Standard Reference Materials. Provisional sample values were corrected based on the known values of the laboratory standards. The long-term standard deviations reported by the UC Davis SIF are 0.2 permil (parts per thousand) for ^{13}C, and 0.3 permil for ^{15}N. Final values are expressed in permil relative to international standards for Vienna Pee Dee Belemnite (V-PDB) and Air for carbon and nitrogen, respectively. Isotopic composition was calculated as follows: δ (‰) = (R sample − R standard/R standard) × 1000, where R is equal to the ratio of the heavy to the light isotope (either $^{13}C/^{12}C$, or $^{15}N/^{14}N$) in the sample compared with that of the standard.

Aliquots of the derivatized methyl ester extracts for all samples (Early Thule, Late Thule, Norton, ASTt-1, and ASTt-2) were also submitted to the SIF at UC Davis for compound specific stable isotope analysis. Compounds were analyzed on a Trace GC Ultra gas chromatograph coupled to a Delta V Advantage isotope ratio mass spectrometer through a GC–C–III interface. Samples were injected, splitless, on a VF-5ms column (30 m × 0.25 mm ID, 0.25 μm film thickness). Once separated, FAMES were quantitatively converted to CO_2 in a in a CuO/NiO/Pt oxidation reactor at 950 °C, dried, and introduced to the IRMS. Corrections to provisional IRMS values were made based on working standards composed of FAMEs calibrated against NIST standard reference materials. As with bulk samples, the $\delta^{13}C$ values for FAME samples are expressed in permil as ratios of ^{13}C to ^{12}C relative to the ratio for the standard reference, V-PDB. Final $\delta^{13}C$ values for sample FAMES were corrected for the isotopic contribution of methanol, incorporated during fatty acid derivatization, using a mass balance equation (Regert, 2011:196).

5. Results and discussion

5.1. Fatty acids and fatty acid ratios

The Early Thule sample contained the highest concentration of lipids, and ASTt-2 contained the lowest concentration of lipids. Correspondingly, Early Thule also had the most complex total ion chromatogram (TIC). The remaining extracts, ASTt-1, Norton, and Late Thule, also contained considerable amounts of lipids. The samples contained a range of fatty acids including saturated n-chain fatty acids from C8:0 to C24:0, though not all extracts contained fatty acids below 10 or above 18 carbon lengths. With the exception of ASTt-2, all samples had ratios of C12:0 to C14:0 that placed them within the range of animal fats and well below the range of plant oils (Fig. 3, Tables 2 and 3). All samples displayed ratios of C16:0 to C18:0 well above those typically found in ruminant fats and in or near the range of various marine fats (or plant oils) (Fig. 3, Tables 2 and 3).

Marine fats contain large proportions of unsaturated fatty acids 20 carbons long. While polyunsaturated fatty acids are rapidly degraded, smaller quantities of monounsaturated fatty acids do sometimes survive in favorable conditions. All of the extracts in this study contained monounsaturated fatty acids C16:1 and C18:1. In addition, the Early Thule sample contained C20:1 and the Late Thule sample contained C20:1 and C22:1. ASTt-2 contained relatively high amounts of C18:1 and was the only extract to contain C18:2. ASTt-2 also contained a low amount of C24:0. This sample was essentially charcoal and had a much lower concentration of lipids than all other extracts. It is possible that more recent organics were absorbed from the surrounding soil, influencing the overall profile.

5.2. Biomarker compounds

All residues, except for ASTt-2, contained methyl esters of the isoprenoid fatty acids, 4,8,12-trimethyltridecanoic acid (4,8,12-TMTD) and 3,7,11,15-tetramethylhexadecanoic acid (phytanic acid). Low amounts of 20 and 22 carbon ω-(*o*-alkylphenyl)alkanoic acids were detected in two samples, Early Thule and ASTt-1. These samples seem to contain a greater amount of charred or blackened organic material than the Late Thule or Norton samples, and may have had better conditions for the formation of ω-(*o*-alkylphenyl) alkanoic acids. The TIC for ASTt-1 is shown in Fig. 4. Mass

Table 3
Summary of criteria used to classify cemented sand residue sources.

Sample ID	C12:0/C14:0	C16:0/C18:0	$\delta^{13}C$ (bulk)	C/N	$\delta^{13}C16:0$	$\delta^{13}C18:0$	Biomarkers	Classification
Early Thule	0.11	5.05	−22.24	>100	−19.48	−17.32	4,8,12 TMTD; phytanic acid; α,ω-dicarboxylic acids, C8–C11; ω-(*o*-alkylphenyl)alkanoic acids; C20:1; C20:0	marine fat
Late Thule	0.05	6.33	−23.13	>100	−19.48	−18.42	4,8,12-TMTD; phytanic acid; α,ω-dicarboxylic acids, C7–C11; C20:1; C20:0; C22:1	marine fat
Norton	0.08	5.00			−19.02	−19.84	4,8,12 TMTD; phytanic acid; α,ω-dicarboxylic acids, C7–C11	marine fat
ASTt-1	0.11	3.43	−23.09	>100	−18.70	−17.19	4,8,12-TMTD; phytanic acid; α,ω-dicarboxylic acids, C7–C12; ω-(*o*-alkylphenyl)alkanoic acids; C20:0; C22:0	marine fat
ASTt-2	0.48	5.25			−21.30	−24.99	C20:0; C24:0	undetermined

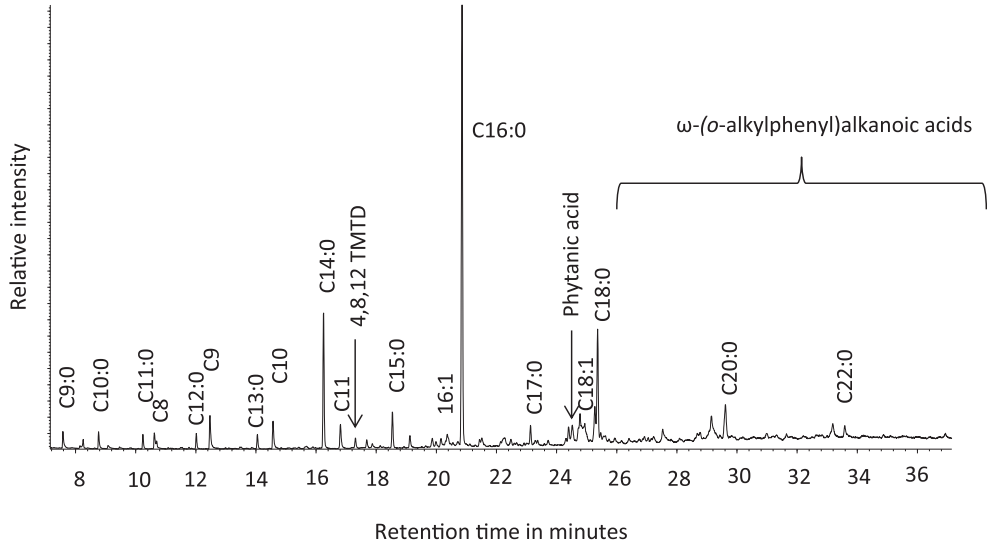

Fig. 4. Portion of total ion chromatogram (TIC) for ASTt-1. Fatty acids are designated as C*x*:*y*, where *x* is the carbon chain length and *y* is the degree of unsaturation (number of C–C double bonds). Dicarboxylic acids (diacids) from seven to 10 carbons long are designated as C7–C11.

chromatograms of selected ions (*m*/*z* 105, 290, 318, 346) for ASTt-1, indicative of ω-(*o*-alkylphenyl)alkanoic acids 18, 20, and 22 carbons in length, are shown in Fig. 5. Based on laboratory experiments, Evershed et al. (2008) demonstrated that a series of long-chain ω-(*o*-alkylphenyl)alkanoic acids form from heating long-chain polyunsaturated fatty acids, which are present in high amounts in marine oils. However, this comes with the important caveat that materials must have been heated to temperatures above 270 °C and in a largely anoxic environment—something that might occur within the pore spaces of pottery cooking vessels, or in closed cooking features, but not necessarily in an open fire. A study by Heron et al. (2010) analyzed lipids in cemented organic residues, soil, charcoal, and fire affected rocks from slab-lined pits in Arctic Norway, believed to have functioned in the processing of marine fats (probably marine mammal blubber) into oil. These authors noted differences in the presence and quantity of fatty acids, biomarkers, and other lipids recovered from various types of materials (charcoal, cemented organic material, soil, or rocks) sampled in the pits, and suggested that preservation may be strongly influenced by material type. They found that abundant and complex lipid extracts were recovered from cemented organic materials and from most of the charcoal samples. Soil and rocks lining the bottom of the pits, however, had much lower amounts of lipids and less complex lipid profiles.

The samples analyzed in the present study varied in composition and probably in their exposure to heat. Based on appearance, some samples contained greater proportions of sediments or charred organic material than others. With this in mind, it is interesting to note that the samples with the most abundant and complex lipid profiles, and which also contained 20 and 22 carbon

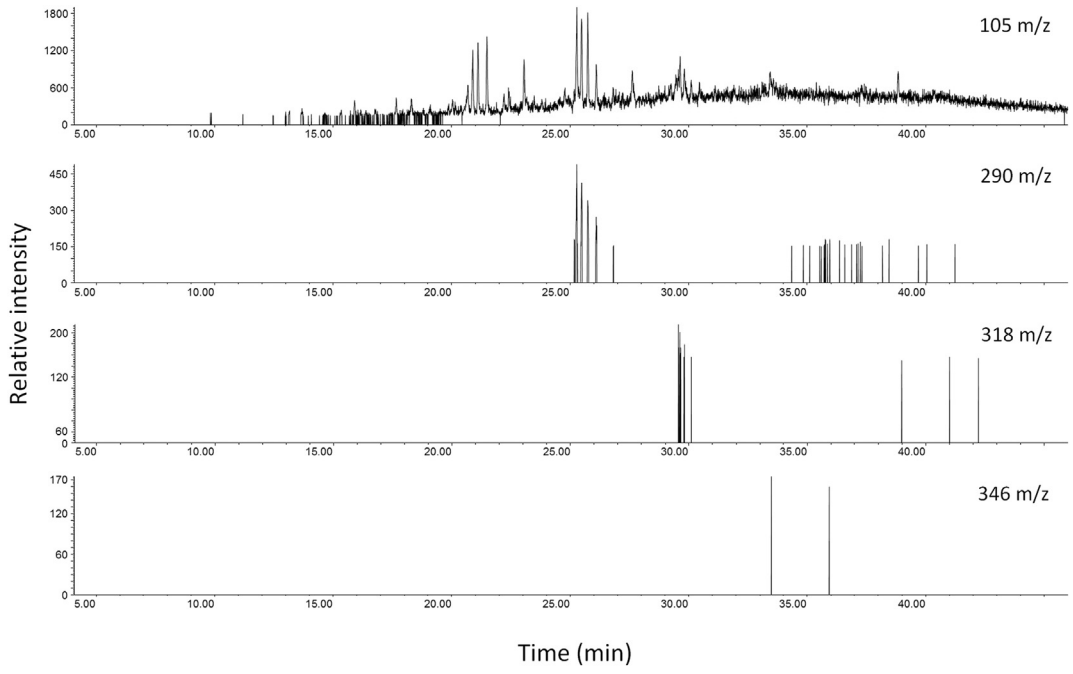

Fig. 5. Mass chromatograms of selected ions (*m*/*z* 105, 290, 318, 346) for ASTt-1. These ions are indicative of ω-(*o*-alkylphenyl)alkanoic acids 18, 20, and 22 carbons in length.

ω-(*o*-alkylphenyl)alkanoic acids, were dark brown-black cemented samples (Early Thule and ASTt-1). The Late Thule and Norton samples were a lighter brown and, although they did contain isoprenoid fatty acids and other biomarkers of marine products, 20 and 22 carbon ω-(*o*-alkylphenyl)alkanoic acids were not detected in these samples. Finally, ASTt-2, which was black and appeared to be completely charred, contained no biomarker compounds and had very low amounts of lipids overall.

Variation in overall content and the presence/absence of biomarkers may have much to do with the materials selected for analysis and/or the functions of individual features. It is important to recognize that sand cemented with fatty organic material could have formed through a variety of activities including food preparation, bone burning, burning of blubber or oil, or the processing and storage of oil and grease. These various activities have different implications for the integration of marine products into early ASTt economies. For example, bone burning could have occurred on a more or less opportunistic basis, while rendering and burning oil from marine animals is a more specialized process that implies greater familiarity and integration of marine resources into early ASTt economies. In light of this, future research should focus on identifying the variable contexts and processes that could have produced cemented sand and organic features in Arctic settings. One way that future studies could gain additional insight into cultural formation processes, is by combining lipid analysis with micromorphological techniques (Mentzer, 2014; Villagran et al., 2013).

5.3. Bulk $\delta^{13}C$

Bulk samples contained very low amounts of nitrogen. The C/N ratio for all samples was well over 100 and reliable $\delta^{15}N$ values could not be determined (Table 3). This is not a surprising outcome if the organic material was composed almost entirely of lipids. Heron et al. (2010) reported similar results for archaeological samples from slab-lined pits and modern reference samples of marine mammal blubber and oil. The bulk $\delta^{13}C$ values of the three samples assayed (Early Thule, Late Thule, and ASTt-1) fall in the range of those reported previously for marine fats and oils (Heron et al., 2010; Stott et al., 1997).

5.4. CSIA

Fig. 6A shows a plot of $\delta^{13}C$ values for C16:0 and C18:0. These values are also listed in Table 3. All samples fall within or very close to the range of marine fats shown in Fig. 6B. Fig. 6B (from Craig et al., 2011:17914) shows the $\delta^{13}C$ values of C16:0 and C18:0 for modern reference fats plotted with 95% confidence ellipses. Though most samples in the present study (Fig. 6A) cluster in or near the upper range of modern marine mammal fats, ASTt-2 falls at the low end of the ellipse for marine lipids shown in Fig. 6B. With respect to the other samples, ASTt-2 is less enriched in ^{13}C for both C18:0 and C16:0. Again, this could be related to the low amount of lipid recovered from this sample. In situations where very small amounts of organic residues remain, effects of low contributions from the surrounding medium will be magnified. Considered together with the higher C12:0 to C14:0 saturated fatty acid ratio for this sample, and the presence of relatively large proportions of di- and mono-unsaturated fatty acids, ASTt-2 may derive, at least in part, from more modern lipid sources present in the soil.

6. Conclusions

The combined data generated by this study strongly indicate that lipids in the Early Thule, Late Thule, Norton, and ASTt-1 samples were derived primarily or entirely from marine animals. Isoprenoid biomarkers 4,8,12 TMTD, and pristanic acid are present in each of these samples, together with a range of diacids from C7 to C12. Isoprenoid fatty acids are present in high amounts in marine animals, are rare in terrestrial animals, and are not present in terrestrial plants (Ackman and Hooper, 1968; Craig et al., 2011; Evershed et al., 2008). The diacids indicate that the residues once contained substantial quantities of unsaturated fatty acids (Buonasera et al., 2013; Evershed et al., 2008; Passi et al., 1993; Regert et al., 1998). Two of these samples, ASTt-1 and Early Thule, also contained 20 and 22 carbon ω-(*o*-alkylphenyl)alkanoic acids, that form when long-chain polyunsaturated fatty acids are heated to high temperatures in an anoxic environment (Evershed et al., 2008; Hansel et al., 2004) suggesting early ASTt/Denbigh people in Alaska may have used sea mammal fats as fuel. Several of these samples also contain fatty acids 20 and 22 carbons in length. Long-chain unsaturated and saturated fatty acids are unusual in

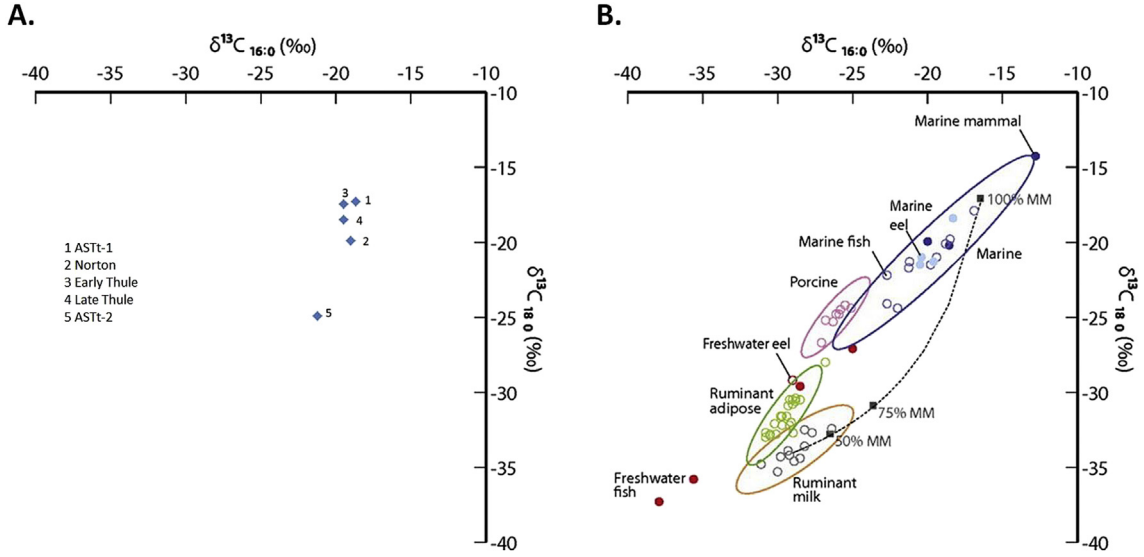

Fig. 6. (A) Compound specific $\delta^{13}C$ values of individual C16:0 and C18:0 fatty acids for archaeological samples. (B) Compound specific $\delta^{13}C$ values of individual C16:0 and C18:0 fatty acids extracted from authentic reference fats and 95% confidence ellipses; reprinted with permission from Craig et al., 2011, Ancient lipids reveal continuity in culinary practices across transition to agriculture in northern Europe. PNAS 108, 44, Fig. 4. MM is hypothetical mixing line for marine mammal and ruminant fat.

terrestrial animals but are present in marine animals, with long-chain polyunsaturated fatty acids being particularly abundant (Ackman, 1989; Evershed et al., 2008; Morgan et al., 1983).

Further, the Late Thule, Early Thule, Norton, and ASTt-1 samples have ratios of saturated fatty acids that place them in the realm of animal fats (C12:0/C14:0) and in the range of marine animals (C16:0/C18:0). Though ASTt-1 has a lower C16:0/C18:0 ratio than the other samples, it is still well above the highest ratio for ruminant lipids. Evidence for mixing with ruminant fats should be especially apparent in compound specific $\delta^{13}C$ values for C16:0 and C18:0. Because ruminant fats contain much higher quantities of stearic acid (C18:0) than marine fats, even a small contribution from ruminant fats should result in substantially lower $\delta^{13}C$ values for C18:0 (Craig et al., 2011). The CSIA values for Early Thule, Late Thule, Norton, and ASTt-1 are in agreement with those obtained for marine mammals and do not indicate mixing with ruminant lipids (Craig et al., 2011).

In sum, organic residue data from four of five cemented sand samples in this study are consistent with a marine origin. Results from the fifth sample appear to represent significant contamination from modern soil lipids, and as a result are less clear regarding origin. Because these cooking/heating features in northern Alaska span ca. 4500–120 calBP, the results indicate that ASTt inhabitants of the Cape Espenberg area were exploiting marine resources at or very soon after their arrival in Alaska. Although the sample size is small, this result is consistent with the hypothesis that ASTt migrants brought a dual economy with them from Asia. Further, the results provide additional evidence that marine resources continued to play an important role in coastal adaptations throughout the culture-historical sequence, from Norton through late Thule contexts. Analysis of a greater number of cemented sand features from the earliest ASTt sites could help to flesh out the nature and degree of marine animal exploitation by early ASTt hunters in northern Alaska. While additional samples from later time periods and different localities, would help to further clarify the development of marine mammal exploitation in northern Alaska. In a region where bone preservation is poor, the organic residue evidence from these cemented sand features can provide an important line of insight into ancient hunting and gathering practices.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jas.2015.05.011.

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