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Chemical investigations of pottery and palaeoenvironmental material from Qasr Ibrim, Egypt.

Chemical Investigations of Pottery and Palaeoenvironmental Material from Qasr Ibrim, Egypt.

by

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School of Chemistry, March 2002

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ABSTRACT

Qasr Ibrim was situated close to what was once the border with Ancient Egypt and Nubia and is known to have gained economic, administrative and religious importance during its occupation (*c.* 1000BC to AD 1800). This, coupled with the extremely arid environment in the region which has aided in the macroscopic and molecular preservation of organic material, ensures that Qasr Ibrim is very important to Nubian archaeology. Immense changes occurred in Nubia between the Meroitic (*c.* AD 50 to 300), Early Post-Meroitic (*c.* AD 300 to 400) and Post-Meroitic periods (*c.* AD 400 to 600). For example, not only do we see the demise of the Meroitic State, but also the introduction of the *saqia* (an animal driven water-wheel) with the onset of the Early Post-Meroitic period.

During the processing of organic material in pottery vessels, lipids can be absorbed into the vessel wall, which can be extracted chemically and analysed by gas chromatography (GC), GC-mass spectrometry and GC-combustion-isotope ratio mass spectrometry. A large-scale (n=313) organic residue analysis of pottery was performed, and by using modern and archaeological botanical and faunal reference materials, it has been possible to determine the origins of the absorbed fatty acids in the pottery through their distributions and carbon stable isotope ($\delta^{13}C$) values. It was observed that approximately half of the sherds from the Meroitic and Post-Meroitic periods contained predominantly animal fats and approximately one fifth of the sherds contained predominantly plant lipids; furthermore, detailed GC-MS analysis has highlighted the mixing of commodities and re-use of vessels at Qasr Ibrim. During the Early Post-Meroitic period, *c.* 75% of the sherds yielded distributions indicative of palm kernel lipids, characterised by high abundances of $C_{12:0}$, $C_{14:0}$ and $C_{16:0}$, and low abundances of $C_{18:0}$ fatty acids (the fruit of the date palm, *Phoenix dactylifera*, and the dom palm, *Hyphaene thebaica*, have been recovered during excavations at the site), and may indicate the processing of palm fruit in order to extract their sugars. Furthermore, analysis of the $\delta^{13}C$ values of the fatty acids from the sherds and animal bones suggested the increasing importance of C_4 plants over time at the site.

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AUTHOR'S DECLARATION

I declare that the work described herein is my own, except where otherwise stated, and has not been submitted for a degree at this, or any other university. Any views expressed are those of the author and in no way represent those of the University of Bristol.

Signed. M. Copley Date. 26-4-02.

Mark Stafford Copley

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LIST OF ABRIEVIATIONS

acetyl-CoA	acetyl-Coenzyme A
BSTFA	*N,O* bis-(trimethylsilyl)trifluoroacetamide
CAM	*Crassulacean* acid metabolism
$\delta^{13}C$	$[(R_{sample} - R_{standard}) \times 1000]/R_{standard}$ where $R_x = {}^{13}C_x/{}^{12}C_x$ in per mil (‰)
$\Delta^{13}C$	$(\delta^{13}C_{18:0} - \delta^{13}C_{16:0})$
diacid	α,ϖ-dicarboxylic acid
DAG	diacylglycerol
DCM	dichloromethane
DNA	deoxyribonucleic acid
EPM	Early Post-Meroitic
FAME	fatty acid methyl ester
GC	gas chromatography
GC-C-IRMS	gas chromatography-combustion-isotope ratio mass spectrometry
GC-MS	gas chromatography-mass spectrometry
HPLC	high performance liquid chromatography
HTGC	high temperature gas chromatography
HTGC-MS	high temperature gas chromatography-mass spectrometry
IS	internal standard
LC-MS	liquid chromatography-mass spectrometry
MAG	monoacylglycerols
m/z	mass to charge ratio
PDB	Pee Dee Belemnite
PEP carboxylase	phosphoenol pyruvate carboxylase
RIC	reconstructed ion current
RuBP carboxylase	ribulose biphosphate carboxylase
TAG	triacylglycerol
TLE	total lipid extract
TMS	trimethylsilyl

Chapter 1.

Introduction

1.1 BIOMOLECULAR ARCHAEOLOGY

1.1.1 Ancient biomolecules

One of the newer, and rapidly expanding areas of archaeology is biomolecular archaeology. Apart from some modern DNA research, this is centred around the investigation of ancient biomolecules found in archaeological materials or in ancient sediments. Broadly, the core areas of research in biomolecular archaeology are concerned with hominid origins and evolution, palaeobiology and biogeography of human species, past human diet and food webs, and artefact studies concerning their use (Thomas, 1993). The main classes of biomolecule are listed below, firstly (left-hand-side) in increasing preservation potential (Logan *et al.*, 1991), and secondly (right-hand-side) in decreasing information *potential*.

1. Nucleic acids
2. Proteins
3. Carbohydrates
4. Lipids
5. Plant biopolymers

1. Nucleic acids
2. Proteins
3. Lipids
4. Carbohydrates
5. Plant biopolymers

The preservation of these organic molecules is not only dependent on their structural resistance to chemical and microbial attack, but also by the conditions of the burial environment (Evershed, 1993), e.g. pH, amount of water present, redox potential and temperature. It should be noted that over archaeological time periods, the burial conditions may change. Exceptional preservation of biomolecules, therefore, tends to occur in extremely cold environments, arid environments and under other unusual conditions such as with peat bogs (Eglinton and Logan, 1991). Furthermore, physical entrapment of organic compounds (e.g. in teeth, bone and pottery vessels) can aid in their survival during burial.

Lipids are utilised by plants and animals as energy stores, cell membrane components and in hormonal and metabolic processes (Kolattukudy, 1980; Gunstone *et al.*, 1986). There are many classes of lipid, all of which can survive in archaeological materials including: fatty acids, alkanes, ketones, alcohols, aldehydes, terpenoids, as well as triacylglycerols and wax esters. Some examples are shown in Figure 1.1. Lipids are hydrophobic; hence their chance of survival in intermittently waterlogged environments is greatly increased when compared with more hydrophilic substances, such as some amino acids and carbohydrates. Fatty acids are ubiquitous in animal fats and plant oils, and are principally found in the form of triacylglycerols (comprising three fatty acids ester linked to a glycerol backbone).

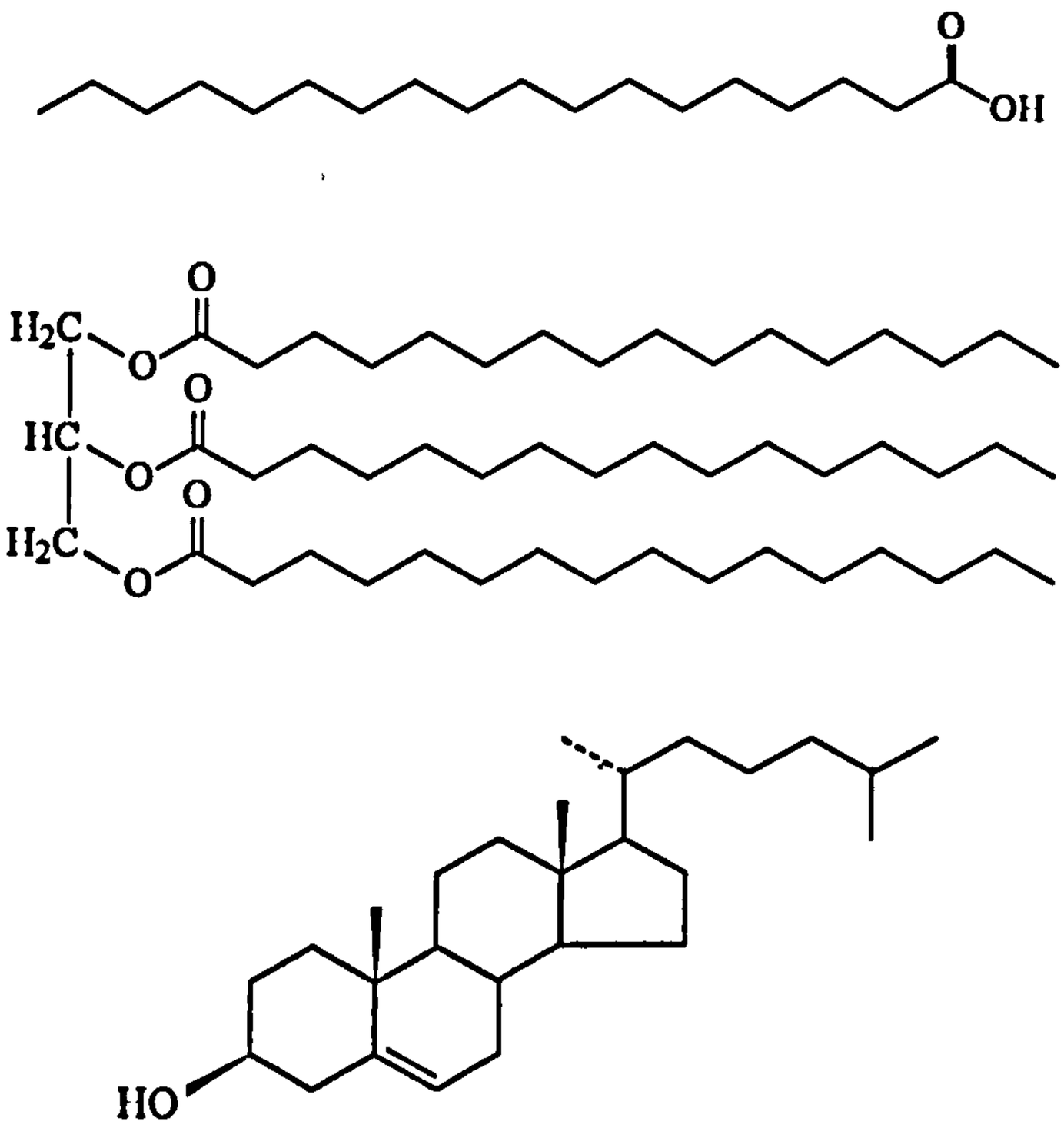

Figure 1.1 Some commonly found lipids in archaeological materials. From top to bottom: octadecanoic acid ($C_{18:0}$), tristearin (C_{54} TAG), and cholesterol (cholest-5-en-3-β-ol).

1.1.2 Chemical analysis of archaeological lipids in pottery and bone

The analysis of archaeological pottery has for some time been recognised as an important way of determining vessel use in antiquity (Rice, 1987:207-243), but whilst this has traditionally relied on analysis of the forms of the vessel (metrical data, ethnographic evidence etc.) to infer function, only through chemical analysis can actual use be determined. It is known that ceramic pots were not only used to store and cook food, but were also employed in the transportation of foodstuffs during trade/exchange, and had non-food related applications, such as oil lamps and semi-industrial uses.

During the preparation and cooking of food in unglazed pottery vessels, organic molecules from animals and plants (e.g. animal fats and plant oils) can be absorbed into the clay matrix of the fired pot. However, some of the earliest workers in the field have investigated charred surface residues, which have adhered to the interior or exterior of the vessel during use (Rottlander and Schlichtherle, 1979; Needham and Evans, 1987). This work has continued more recently with the use of pyrolysis mass-spectrometry (Oudemans and Boon, 1991; Oudemans and Boon, 1993). However, most modern investigations now utilise absorbed as well as the adhering residues. Absorbed residues in the vessel wall are afforded a level of protection from the burial environment; furthermore, it is known that possible migration of contaminating soil lipids into sherds is negligible (Heron *et al.*, 1991), confirming the integrity of lipids absorbed during archaeological usage.

Solvent extractable lipids from the absorbed residues were first analysed in the 1970's and 1980's. Some studies used high performance liquid chromatography (HPLC) to identify free fatty acids (Passi *et al.*, 1981), whilst others concentrated on the use of gas chromatography (GC) for the analysis of methylated free fatty acids, aided by the use of reference materials in an attempt at identification (Condamin *et al.*, 1976; Condamin and Formenti, 1978; Patrick *et al.*, 1985).

More recently, it has been recognised that animal fats and plant lipids are unlikely to be preserved in their original state due to diagenetic processes. For instance: hydrolysis of ester-linkages in acyl lipids liberates free fatty acids and alcohols (Evershed *et al.*, 1992); β-oxidation can occur in aerobic conditions which cleaves two carbon units off a carboxylic acid in a sequential fashion (Kindl, 1987); unsaturated fatty acids can be oxidised at the point of unsaturation yielding α,ϖ-dicarboxylic acids (Passi *et al.*, 1993) as well as other oxidation products such as dihydroxy fatty acids (Regert *et al.*, 1998); and thermal degradation or condensation of fatty acids is also known (Ramanathan *et al.*, 1959; Evershed *et al.*, 1995a). Furthermore, the diagenetic products of other classes of lipid have been studied, for example, cholesterol (Adachi *et al.*, 1997; Stott *et al.*, 1999). Therefore, through a combination of laboratory decay experiments and analyses of archaeological, ethnographic and modern material, diagnostic lipid components have been identified, and are discussed below.

Degraded animal fats

Laboratory decay of animal fats has been performed (Dudd *et al.*, 1998; Dudd, 1999:208-251), allowing degraded animal fats to be identified (Evershed *et al.*, 1990; Evershed *et al.*, 1992; Evershed *et al.*, 1997c) through the distributions of their free fatty acids, the tri, di- and monoacylglycerols, via GC, gas chromatography-mass spectrometry (GC-MS) and gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS). A high content of saturated carboxylic acids is indicative of an animal source, and when other lines of evidence are used, such as the positional isomers of alkenoic acids and the stable carbon isotope ($\delta^{13}C$) values of the $C_{16:0}$ and $C_{18:0}$ components (hexadecanoic and octadecanoic acid, respectively), it is possible to identify animal fats as either being ruminant or non-ruminant (Evershed *et al.*, 1997c; Dudd, 1999; Mottram *et al.*, 1999). Furthermore, through the use of a combination of criteria, including triacylglycerol distributions and the detection of more depleted $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids, the first direct evidence for the processing of dairy products in antiquity has been obtained (Dudd and Evershed, 1998).

Beeswax can be distinguished through the detection of characteristic palmitic wax esters and long-chain alcohols and has previously been detected in numerous vessels (e.g. Heron *et al.*, 1994; Charters *et al.*, 1995). Beeswax has also been detected in lamps where it was used as an illuminant (Evershed *et al.*, 1997d).

Degraded plant lipids

The detection of leaf wax lipids has enabled the identification of plant components in numerous archaeological vessels. These leaf wax lipids include wax esters, alcohols, alkanes and sterols (such as β-sitosterol, campesterol and stigmasterol) and ketones. Caution concerning the sole use of ketones as indicators of the processing of plants in pottery has to be applied, as some ketones originate in the dehydration/decarboxylation of acyl lipids when subjected to temperatures in excess of 300°C (Evershed *et al.*, 1995a; Raven *et al.*, 1997). However, the ancient processing of the leafy vegetables *Brassica oleracea* and *Allum porrum*, cabbage and leak respectively, have been identified through the identification of components of their epicuticular leaf waxes, i.e. nonacosan-15-one and nonacosan-15-ol from *Brassica olecracea* and hentricontan-16-one from *Allum porrum* (Evershed *et al.*, 1991; Evershed *et al.*, 1992), and confirmed through laboratory experiments (Charters *et al.*, 1997).

1.1.3 Carbon stable isotopes

Carbon exists in nature primarily in the form of two stable isotopes, ^{12}C and ^{13}C (with approximate abundances of 98.89% and 1.11% respectively). Plants discriminate against ^{13}C to differing degrees, depending upon which of the three photosynthetic pathways the plant utilises: C_3, C_4 or Crassulacean acid metabolism (CAM) (Smith and Epstein, 1971). The C_3 plants are so-called because after photosynthetic fixation of CO_2, they form a C_3 unit (3-phosphoglycerate) via the enzyme ribulose biphosphate carboxylase (RuBP carboxylase), i.e. they follow the Calvin-Benson or C_3 photosynthetic pathway. In contrast, C_4 plants utilise the Hatch-Slack photosynthetic pathway, whereby fixation of CO_2 yields oxaloacetate (a C_4 dicarboxylic acid) via the enzyme

phosphoenol pyruvate carboxylase (PEP carboxylase). CAM plants under normal circumstances use the C_4 pathway as their means of carbon fixation, although they are able to fix CO_2 via the C_3 pathway during the day.

The ^{13}C abundance in the metabolites of the two main pathways are different: the Calvin-Benson cycle includes a mass discriminatory bias against $^{13}CO_2$ during fixation. Therefore the relative values of ^{13}C are different, and this accounts for the majority of variation in plant $\delta^{13}C$ (the ratio of ^{13}C to ^{12}C, relative to a standard) values, with C_4 plants being more enriched in ^{13}C relative to C_3 plants (Farquhar *et al.*, 1989). However, stable isotope values are known to be dependent upon many factors which affect to what extent ^{13}C is discriminated against, such as altitude (Vitousek *et al.*, 1990; Beerling *et al.*, 1993), season (Lowdon and Dyck, 1973), latitude (Beerling *et al.*, 1993), as well as natural intra-species variation (Ehleringer, 1990; Lockheart *et al.*, 1997). When a herbivorous animal feeds on plants, the plant macronutrients will undergo various metabolic pathways to produce the animal's tissue biochemical components (see Appendix 2, Fig. A2.3 for the metabolism of dietary lipids, carbohydrates and proteins). Therefore, any variation in the $\delta^{13}C$ values of the plant biomolecules will be reflected in the stable isotope values of the fatty acids of the animal that consumes it.

A significant amount of work on archaeological human and animal bones, has been centred on the use of collagen to elucidate prehistoric diets (e.g. van Klinken *et al.*, 1994; van der Merwe and Tschauner, 1999). There has also been work on the lipid constituents of bone made possible through GC-C-IRMS of cholesterol and its congeners. Compared to collagen, these have been shown to be more indicative of short-term changes in diet and hence are a useful indicator of prehistoric human diets (Stott and Evershed, 1996; Stott *et al.*, 1997; Stott *et al.*, 1999; Jim *et al.* in prep.).

1.1.4 Previous biomolecular studies involving Qasr Ibrim and Nubia

There have been a few studies centred around the Nile Valley, most of which have relied solely on the analysis of bone collagen; however, other components of the desiccated human remains that survive in the arid environment have also been utilised. In a study of Nubian bone, muscle, skin and hair, temporal changes in the $\delta^{13}C$ values were found (White and Schwarcz, 1994). White and Schwarcz found that there was a significant shift in $\delta^{13}C_{COLLAGEN}$ values from C_3 to C_4 between the Meroitic Period and the subsequent Post-Meroitic Period and then back to predominantly C_3 in the following Christian periods (with the largest shift occurring in the Christian period). They also investigated whether differences existed between the $\delta^{13}C_{COLLAGEN}$ values for males and females. From analyses of multiple samples taken along the shaft of the hair, it was possible to observe very short-term changes in dietary consumption of C_3/C_4 foodstuffs, indicating seasonal changes in the diet just prior to death (White, 1993; White and Schwarcz, 1994). Furthermore, from the analysis of bone collagen, a suggested link has been made between the use of nitrogen stable isotope analysis and the aetiology of disease in past populations, for example osteopaenia (White and Armelagos, 1997).

Biomolecular studies emanating from samples from Qasr Ibrim, have included the analysis of nucleotides from radish seeds (O'Donoghue *et al.*, 1994; O'Donoghue *et al.*, 1996), and DNA sequences from sorghum (Shaw and Rowley-Conwy, 1996; Rowley-Conwy *et al.*, 1997). There has also been excellent preservation of archaeological seeds, both chemically and microscopically (van Bergen *et al.*, 1997), and this has helped in the understanding of decay processes of buried plant material (Evershed *et al.*, 1997b). The analysis of absorbed residues in lamps from various contexts from Qasr Ibrim revealed degradation products that are rarely seen in such abundance in archaeological materials (Regert *et al.*, 1998). In this latter study, high abundances of α,ω-dicarboxylic acids formed via various processes that include oxidation, hydration and bond cleavage were observed (see Section 3.1.2).

1.1.5 Large scale studies

Very few large-scale regional studies using lipids associated with potsherds have been completed. Evershed and co-workers have successfully investigated the use of vessels from Southern prehistoric Britain (e.g. Dudd, 1999), and of numerous medieval sites across central Britain (Evershed *et al.*, 1999). These studies have concentrated on the triacylglycerol distributions and the compound specific $\delta^{13}C$ values of the fatty acids for identification of lipid origin.

There have also been studies from archaeological sites in Canada, which have attempted to link fatty acid distributions in reference materials, laboratory degraded material and archaeological pottery (Malainey *et al.*, 1999a; Malainey *et al.*, 1999b; Malainey *et al.*, 1999c). However, they compared the fatty acid distributions of the extracts to reference materials using medium-chain fatty acids ($C_{12:0}$, $C_{14:0}$ and $C_{15:0}$) and unsaturated fatty acids. Neither of these lipid classes will survive in their original form or abundance, and since the use of the vessel during its lifetime and the burial conditions will differ significantly, the pottery samples cannot be directly compared with results of laboratory decay experiments.

In order to determine vessel use over a period of time, a large-scale analysis of pottery vessels needs to be employed. Most of the statistical methods that are applicable assume that the data is normally distributed. If only a small data set is available for analysis, then it's distribution may not be normal; this reduces the effectiveness of any statistical analyses on the data set (Shennan, 1988; Tabachnick and Fidell, 1996). Furthermore, where statistical comparisons of data sub-sets (e.g. ware type, period etc.) are to be undertaken, then an even larger sample size needs to be utilised. Therefore for statistically meaningful results, a large sample size needs to be obtained.

1.2 THE ARCHAEOLOGY OF NUBIA

The pottery samples that have been selected for this thesis come from specific contexts that date from archaeological periods in which a great deal of change was occurring, not only at Qasr Ibrim, but also in Nubia as a whole. In order to understand the rationale behind the sampling strategy, it is necessary to briefly discuss the history of Nubia, and then how Qasr Ibrim fits in to this.

1.2.1 Introduction

The term 'Nubia' was used by medieval Arabs to describe the area just south of Aswan, in Egypt, and extending to the White and Blue Niles in Sudan. The Greeks and Romans referred to it as 'Aethiopia'. The ancient Egyptians called this area 'Kush', which can be limited to denoting the period of Nubian history which had Kerma, Napata and Meroë as their 'capital' cities. Nubia lies to the south of Ancient Egypt and whilst the history of both areas are entwined, Nubia itself is distinct and quite different to its neighbour; and even though Ancient Egypt has been more comprehensively studied, it should be noted that Nubian Civilisation has stretched back in time to pre-Dynastic Egypt. A map of Nubia is shown in Figure 1.2 and contains many of the sites referred to in this section.

1.2.2 Geography

Without doubt, the Nile was the lifeblood of Nubia providing, in places, the only source of water for humans, animals and crops. The Nubian stretch of the river in the north cuts through areas of desert plains, and abrupt cliffs line its course in places (Hume, 1925:8). Although outcrops of Basement Complex can be observed in areas such as the Lower Nubian Plain, other geology commonly found in the area includes the Nubian sandstone (Adams, 1977:22-23), some of which exhibits a dark-gray colour on portions of the exposed sandstone, due to the presence of ferruginous sandstones (Butzer and Hansen, 1968:203, 205).

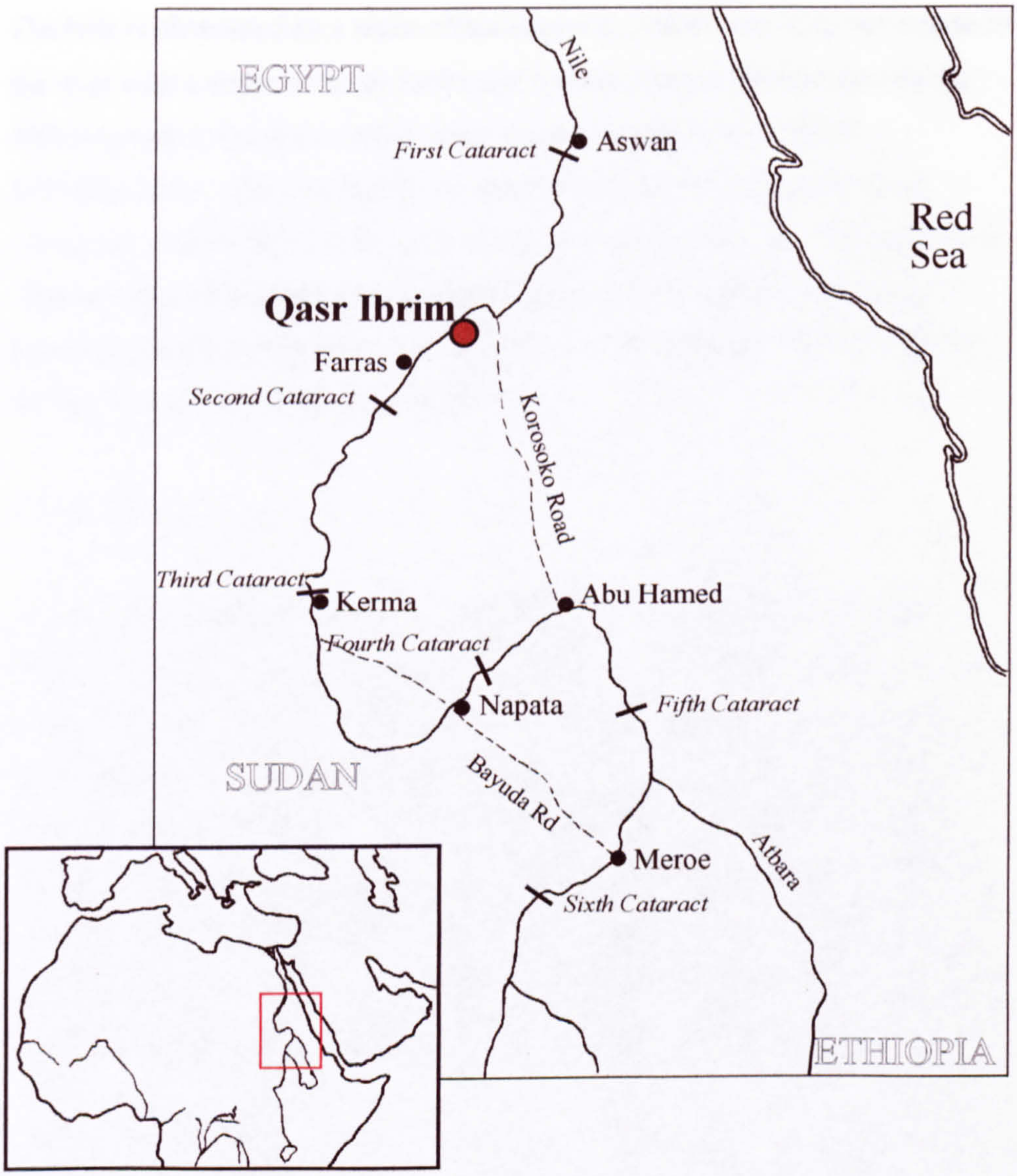

Figure 1.2 Map of Nubia, showing the location of Qasr Ibrim.

The Nile is obstructed by a series of six cataracts, which hem in on the course of the river with a series of rocky banks and islands, formed through the slightly differing rock formations, which create numerous rapids (e.g. Hume, 1934:236,313). The first of these is just south of Aswan, and the second cataract is south of Wadi Halfa in modern day Sudan (Fig. 1.2). The existence of these cataracts ensured that the river could not be easily used as a long-distance line of communication, as it could in Ancient Egypt, and necessitated the use of overland routes in some places.

1.2.3. Climate

As one would expect, with the vast area of land that Nubia covers, the climate of the area varies considerably. To the south (in what is termed 'Upper Nubia') where tropical conditions prevail, seasonal variation exists such that there is a dry winter and summer as well as a rainy season followed by a humid season. However, in the north (in 'Lower Nubia'), with its desert conditions, there is a short winter and a long and hot summer (Edwards, 1989:15). The rainfall in Lower Nubia is essentially zero and is very localised where it does occur; this contrasts greatly with that of Upper Nubia where 200-400 mm are typical south of Kartoum, near the junction of the Blue and White Niles.

1.2.4. A brief history of Nubia

Whereas the emergence of Egypt as a centralised entity, with its roots in the Archaic period (*c.* 3100-2800BC), has provided the civilisation with a sense of unity, the same cannot be said of Nubia (Welsby, 1996:11-12).

Before the Meroitic period: Kerma and Napata

The fortunes of the Kingdom of Kush have changed throughout its history. From the outset, with the establishment of a civilisation centred around the city of Kerma (*c.* 2400BC), the region has relied on its proximity to both Central Africa and Egypt for its prosperity. The Nile Valley provided a conduit for trade and exchange, primarily of luxury goods, between Egypt / the Mediterranean and the lands to the south of Egypt. Thus, the exportation of

luxury goods that included gold, ivory and slaves (Adams, 1995:775) comprised an important element of the Nubian economy . Indeed, it has been described as 'The Gateway to the South' by Ancient Egyptian and Arabic writers alike (Kirwan, 1962).

Egyptian interest and influence in Kush has waxed and waned depending upon its own domestic political necessities. For example, with the domestic problems faced by the Egyptians during the Second Intermediate period (*c.* 1750-1680BC), and later with the Hyksos causing troubles in the North of Egypt (Welsby, 1996:12), Egypt's interest and influence in Nubian affairs diminished. Egyptian activity in the region is displayed through the building of fortresses around the Second Cataract, probably for protection of the Nile trade. Similarly, later on (although the exact date is unknown), the establishment of Napata as the 'capital' of the Kingdom of Kush was made possible with the assistance of locally based priests from the Temples of the Egyptian god Amun (Adams, 1977:257); these priests would have had connections with the elite groups in Egypt.

Egypt's influence on Kushite culture can be observed in the fairly rapid acculturation that is evident at Kerma and later at Napata with the architecture, religion and burial practices of the Kushites (Adams, 1995:778-779). Despite this, some local traditions have continued right through the region's history and as such the cultural development of the area also needs to be assessed within a Sudanic/African context (Edwards, 1998b).

The Meroitic period (c. 600BC-AD350)

The rise of the city of Meroë, some 300 miles upstream of the Nile from Napata, heralds the beginning of the Meroitic period. Although the precise date of the establishment of Meroë as the major Kushite city, as opposed to Napata, is unknown, it is accepted that by *c.* 300BC Royalty were routinely buried there, rather than at Napata (Adams, 1995:781-3).

The Meroitic state was centred around a political body closely connected to Royal lineage and the religious elite, which dominated every aspect of the

culture. Meroitic religion included the continuation of the worship of Amun, although the Kushite pantheon incorporated indigenous gods such as Apedemak, and the 'imported' cults of Horus, Osiris and Isis (Welsby, 1996:77). Whilst large cities such as Meroë were undoubtedly major urban centres with people living sedentary lives, a significant proportion of the Meroitic peoples would have lived nomadic or semi-nomadic lives (Welsby, 1996:137), although the actual extent of uncentralised, nomadic existence has been debated (e.g. O'Connor, 1993).

Aspects of Kushite culture have similarities to that found in Egypt (e.g. the use of Egyptian deities) such that, in the past, some scholars have seen the Meroitic State as being organised along the 'Pharonic' model, as seen in Egypt. However, recent archaeological and ethnographic work has pointed more towards a 'Sudanic' model, with its roots within Africa rather than Egypt (e.g. (Edwards, 1996; Edwards, 1998b). This latter model sees the Meroitic State encompassing limited direct rule over the whole of Kush (Nubia); instead, a system of suzerainty is utilised. Hence, especially in the peripheral parts of the Meroitic State, power is illustrated through religious symbols and the redistribution of prestige goods. The presence of trade between Lower Nubia and Roman Egypt (which did not occur to the same extent in the south) indicates that although suzerainty may indeed be the prime mode of 'political' legitimacy, local elites did indeed trade prestige goods with Egypt outside of the normal centralised economy (e.g. Edwards, 1996).

The majority of the Nubian economy would have been based around agriculture and animal husbandry (Welsby, 1996:153). The level of the Nile is known to have risen and fallen over the years, with occasional flooding, and in a region that so heavily depends upon the Nile, this has had an effect on the subsistence patterns employed. Unlike in Egypt, a great deal of the Nile Valley in Nubia was not entirely amenable to agriculture, especially without the use of effective irrigation methods. Although the *shaduf* (a counterweighted contraption that could raise water by 3 m) allowed very limited irrigation (Welsby, 1996:156), it was not until the introduction of the *saqia* (Fig. 1.3), an animal driven waterwheel, in the Early Post-Meroitic period (Rose, 1995) that effective

Figure 1.3 The *saqia*, as used in the region in modern times.
The pottery vessels specifically designed for the *saqia*, known as a *qadus*, are found from the beginning of the Post-Meroitic period in Lower Nubia (from http://visitweb.com/nubian).

irrigation in Lower Nubia could take place, allowing a greater quantity and variety of crops to be grown; for detailed information on how the *saqia* works, see Menassa (1974).

A significant proportion of the economy of Nubia was probably geared towards trade, particularly with Egypt. Possibly channelled through Royal monopolies (Török, 1987:23), ivory, ebony, skins, ostrich feathers, gum arabic and slaves have all been traded or transhipped from Central Africa through Nubia to Egypt (Edwards, 1989:156-157). Furthermore, live animals, copper, timber and stone are also known to have been shipped northwards (Kirwan, 1962). Cotton was grown and exported by Lower Nubia to Roman Egypt, filling a demand for this commodity, as it is thought that cotton was not yet grown in Egypt (Adams, 1995:788). Similarly, it has been suggested that grain, and later other crops, were grown in Lower Nubia and sold to the Roman settlements and military that were stationed in the area just south of Aswan. Trade of this nature did very well during times of political stability, with the reverse naturally being true during troubled times (Adams, 1988). However, the precise extent of north-south contact in Lower Nubia has been questioned (Alexander, 1988), and it is likely that a substantial proportion of the Lower Nubian economy would have been concerned with local subsistence.

The Nile would have intuitively been the main line of communication between north and south, however there are also a number of overland caravan routes such as the Korosoko, Maheila and Bayuda roads (Fig. 1.2), with many more localised routes, running not only north-south, but also east-west. It has been stated that the Korosoko Road, which ran from Abu Hamed, north of the Fifth Cataract to Korosoko, far downstream in Lower Nubia, became the main link between the Sudan and Egypt (Adams, 1977:304), but there is no direct evidence of this or other land routes being utilised in early Nubian history (Edwards, 1989:158; Shinnie, 1991).

As was the case in much of Nubia's past, there was a sharp divide between Upper and Lower Nubia. The geology and climate (*c.f.* Sections 1.2.2, 1.2.3) of the region differs dramatically. This may partially explain the fact that in the

south there is continuous substantial occupation of Meroë from its foundation to the end of the Meroitic period, whilst in the north there appears to be a much lower level of occupation. The desert environment that exists in Lower Nubia ensured that permanent settlements had to be placed along the River Nile and thus their fortunes were inextricably linked with the level of the river (Welsby, 1996:153). It is believed that there was a complete depopulation of Lower Nubia during the early (pre-Roman) Meroitic period, with subsequent resettlement (Adams, 1976), however the extent of this depopulation and resettlement may be exaggerated, and there is some evidence for the continued settlement in Lower Nubia during the early Meroitic period, a period which sees the increase in Ptolemaic and Roman interest in the area just south of Aswan (Rose, 1996; and references therein).

A further difference between Upper and Lower Nubia may be seen in the architecture of the two regions. For the most part the south tends to be represented by large temples, tombs and Royal buildings, possibly indicating the more theocratic tendencies of the ruling elite. In contrast, Lower Nubian architecture is more in tune with a secular feudalism in which the fortress, palace, village and cemetery gain ascendancy (Adams, 1976).

The Post-Meroitic (c. AD350-550)

One of the reasons for the eventual collapse of the Meroitic State was the demise of international trade in the 3rd and 4th centuries AD (Edwards, 1998b). What is clear is that Meroë eventually came into conflict with the Axumites from the Abyssinian Highlands, and there is evidence for Axumite invasions of Meroë itself, although possibly only after another people, the Nuba from the west of the Nile, had already overrun Upper Nubia. Similarly, in the north, from written sources, it is stated that the withdrawal of the Romans to the First Cataract created a vacuum that left the region open to subsequent movements from the west of the Nile of the Nobatae, and from the east of the Nile the Blemmyes (Adams, 1977:383-390). Recently less emphasis has been placed on the migration of different peoples, and more emphasis on an evolving Meroitic culture that transformed itself into what is witnessed in the Post-Meroitic

period, for example in terms of burial customs, the economy and industries such as pottery production (Welsby, 1996:202-205).

However, much of the history of this period is unknown, and the extent of any continuities/discontinuities in Meroitic–Post-Meroitic culture has been debated (e.g. Trigger, 1965:134-140; Adams, 1976; Kirwan, 1982; Edwards, 1989:170-174). Little is known about the changes in settlement during this period, apart from the fact that urban centres declined and in Lower Nubia few substantial buildings were constructed (Edwards, 1989:178). With the demise of Meroë, there are some indications of cultural change, for example old funerary traditions re-emerge in Upper Nubia (Welsby, 1996:202), and the level of imports represented in the archaeology of Upper Nubia dwindles in this period, contrary to the experience of Lower Nubia (Edwards, 1989:181).

In summary, the history of Nubia is essentially divided somewhat between north and south. A strong theocracy existed in the south, borrowing from Egyptian religion and style of government, possibly ruling over a number of settlements, which made up the Meroitic people. However, in the north, the ruling elite possibly had looser ties to Meroë, and through trade and exchange had closer economic ties with Egypt. Furthermore, with the end of the Meroitic period, the lack of Egyptian goods in the south and their continued presence in the north is indicative of the political and economic changes that were prevalent in Nubia at the time.

1.3 Qasr Ibrim

1.3.1 The site

Qasr Ibrim lies within Lower Nubia, just over half way between the First and Second Cataracts, upstream of both Korosoko (the northern end-point of the Korosoko Road) and the Roman frontier (Fig. 1.2). It comprised a fortified citadel that gained religious, administrative, commercial and strategic

importance during its history (Horton, 1991:264). Qasr Ibrim used to be situated on a cliff top promontory that overlooked the Nile on the eastern side of the river. In the 1960's, with the building of the Aswan High Dam, intense excavation started at Qasr Ibrim, under the auspices of the Egypt Exploration Society, as part of a global initiative in the region. Today, a large proportion of the Nile Valley within southern Egypt and northern Sudan is flooded (i.e. Lake Nasser), therefore all of the Nubian archaeology that has not been excavated and is under water, is lost. Because Qasr Ibrim was situated on top of a prominent craggy outcrop it has survived this flooding (Fig. 1.4 and Plate I) although, with the levels of Lake Nasser rising still, increasing amounts of the site are being submerged (Rose, 1998a).

The site has been dated from *c.* 1000BC to 1800AD, and the main archaeological periods are given in Table 1.1. The earliest evidence for occupation is from the first millennium BC with the building of Napatan

Figure 1.4 Qasr Ibrim as it was in 1998. A portion of the fortification can be seen nearest to the camera, right of centre. The structure in the centre is a Christian 'cathedral'. Trench 10/14, from which the pottery samples used in this study were excavated, is to the right of the island.

Table 1.1 The main archaeological periods at Qasr Ibrim

Archaeological period	Dates (approximate)
Napatan	1000 BC - 700 BC[1]
Ptolemaic	100 BC - 23 BC[2]
Roman	23 BC - AD 50
Meroitic	AD 50[3] - 300
Early Post-Meroitic	AD 300 - 400
Post-Meroitic	AD 400 - 550
Christian	AD 550 - 1500
Islamic	AD 1500 - 1800

[1] some point after this date.
[2] exact status of 'Ptolemaic' Qasr Ibrim is unknown.
[3] some point before this date.

temples and defensive walls (e.g. Rowley-Conwy, 1988; Driskell *et al.*, 1989; Horton, 1991; Rose and Edwards, 1998). Following the Napatan period, little is known about any possible occupation until Meroitic peoples attempted to defend Qasr Ibrim against the Romans in 23BC (Adams, 1977:340-341; Rose, 1996:1). Even though it has been suggested otherwise (Alexander, 1988), it is possible that the Romans were only at Qasr Ibrim for a short period of time (Horton, 1991:268, 271). Regardless of the length of time of Roman Ibrim, there is clear evidence for contemporary Meroites in the 'hinterland' just beyond the fortifications at Ibrim during the Ptolemaic period (Rose and Rowley-Conwy, 1989; Rose, 1994; Rose, 1996).

From recent excavations, Meroitic levels at Qasr Ibrim seem to suggest the growing significance of Ibrim as a religious site, with the building and extension of existing temples (Alexander and Driskell, 1985; Horton, 1991:272; Horton, 1992; Rose and Edwards, 1998). The religious importance of the temples is essentially a continuation from Napatan times, when a 'Taharaqa Temple' (as well as other structures) was erected under the Kushite Ruler Taharaqa (*c.* 690-664BC), and appears to extend at least until the end of the Post-Meroitic period (Horton, 1991), after which the site became a focus for Christians. The regional importance of the site as a religious centre can be seen in the numerous ostraca, docklets and engraved footprints that contain the Meroitic script as left by

'Meroitic' pilgrims and in Demotic as left by the 'Egyptian' pilgrims (Edwards and Fuller, 2000). A portion of the Temple precinct is shown in Plate II. Not only was Qasr Ibrim a religious centre, but also, along with Faras and Jebel Adda, a commercial and administrative centre (Adams, 1995:788), probably aided by its prominence on the Nile and proximity to overland routes (Fig. 1.2). The population that stayed at Qasr Ibrim were unlikely to be representative of the Lower Nubian population as a whole. Whilst it is known that areas outside of the fortress were used for settlements, it would not have been possible to utilise the eastern bank of the Nile effectively due to the geology. Therefore, although there are a few burials on the eastern side (Rose, 1994:201), it is more likely that settlements existed on the western bank of the Nile, opposite Qasr Ibrim.

The focus of this study is centred on pottery samples obtained from Structure 265 (Plates III & IV), which was partially excavated during the 1998 season. This structure lies within trench 10/14, to the east of the Meroitic temple development and along 'Magazine Street'. Ottoman buildings overlie this complex amalgamation of structures of Meroitic and Post-Meroitic date, and it is clear that numerous modifications have occurred during its occupation (Rose and Edwards, 1998). Figure 1.5 shows the internal structures of Str. 265 and neighbouring buildings.

Numerous finds were recovered from the excavation (Plate V), including leather, fragments of Meroitic text, textiles, basketry, stone stamps (used in the sealing of mud seals in jars and amphorae), spindle whorls and other possible weaving-related artefacts, such as loom-weights, and vast quantities of ceramics and archaeobotanical remains (Rose and Edwards, 1998). It is known that buildings from this period were rectangular shaped, with frequently re-laid mud-laid floors. They may have had internal stairways and cellars; the walls were of mud-brick, which were plastered and whitewashed, and sometimes had niches where it appears 'lamps' were set.

Qasr Ibrim is important to Nubian archaeology not only due to its unusual function as a cult centre (not to mention its administrative and military functions

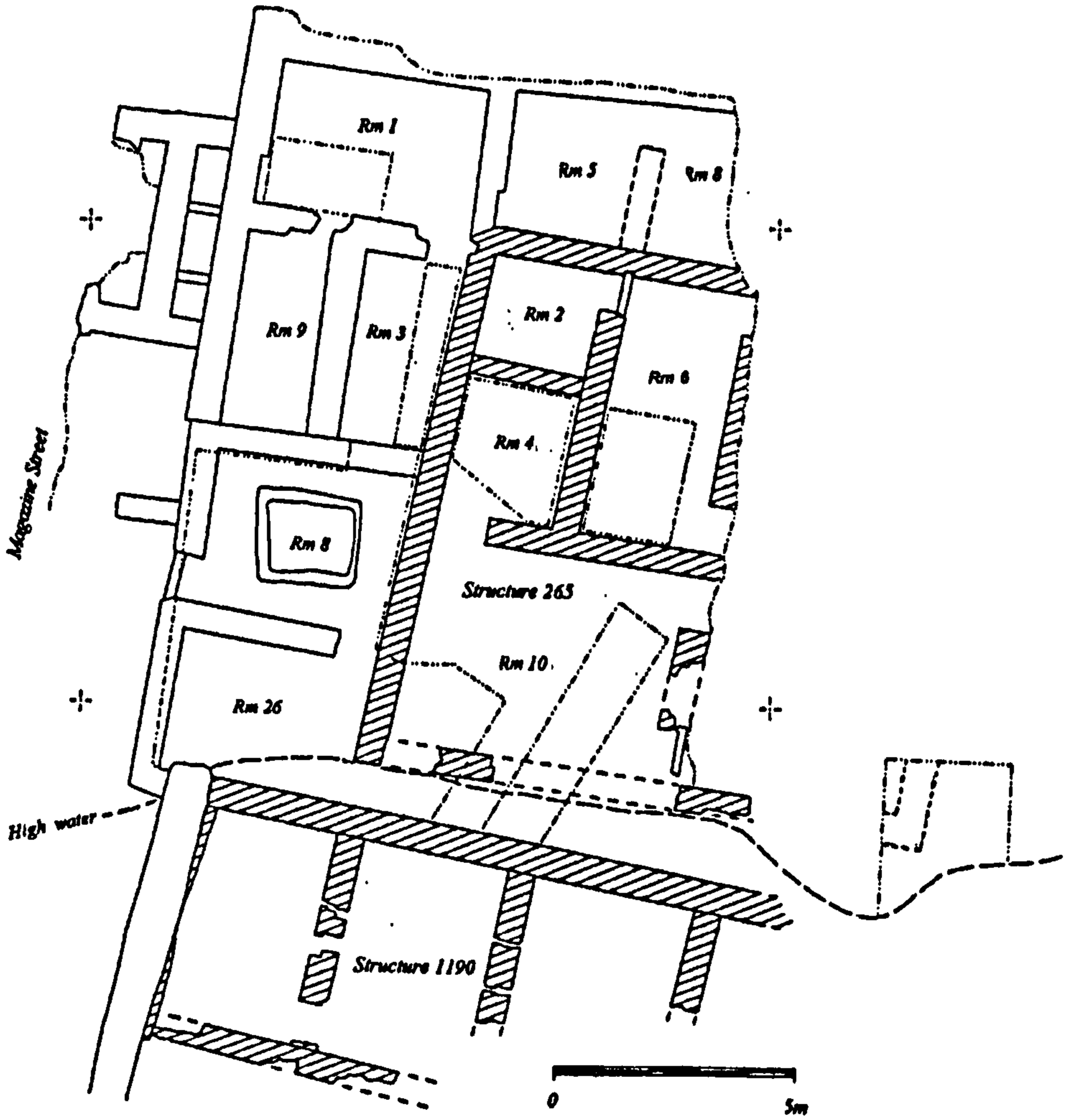

Figure 1.5 Structure 265. Rm denotes the room numbers from where the pottery samples were excavated (Source: David Edwards).

as well), but also due to the fact that with the arid environment, there has been excellent preservation of organic material. Furthermore, with the creation of Lake Nasser, Qasr Ibrim is the only major Nubian site left in Egypt (Anderson, 1982).

1.3.2 The pottery

Adams' two volumes that constitute 'Ceramic Industries of Medieval Nubia' (1986) form the basis of this section. Although the following draws heavily

from this, a few points should be made: (i) The quoted percentages of ware types present in the different periods are only rough estimates. (ii) Adams' volumes are particularly good for the Christian periods, but now, with more excavation of the earlier Meroitic and Post-Meroitic levels, scholars' understanding of the ceramic evidence for these periods is increasing. (iii) The situation at Qasr Ibrim nearly always differs to other areas in Lower Nubia, and is inheritantly different to that found in Upper Nubia, due to its role within the region, as discussed above. The result is that a re-assessment of the whole pottery assemblage in Lower Nubia, and particularly at Qasr Ibrim, is needed and is currently being undertaken (Rose, *pers. comm.*).

Nubian pottery can be divided into three main categories: Nubian hand-made, Nubian wheel-made and imported wares. During the Meroitic and Post-Meroitic periods, only a small percentage (*c.* 5-10%) of the total wares from the Meroitic and Post-Meroitic were hand-made (Adams, 1986b:38, Fig. 9). They were produced and utilised locally, and it is thought that part-time potters made them, probably women (Adams, 1986b:39). Hand-made pottery vessels were probably fired without the use of a kiln, but rather in a pit or were piled on the ground, covered in dung, which was used as a fuel. In the recent past, there were peoples in Egypt who similarly use durra stalks and cow/buffalo dung as fuel (Blackman, 1927:148); and similar practices are even known in Sudan today (Tobert, 1984). These bonfires can reach temperatures of 600-700°C (Adams, 1986b:33). Although these vessels have organic tempers included in their matrix, it is known through experimental work (Johnson *et al.*, 1988) that temperatures over 600°C are enough to thermally combust the organic material to CO and CO_2, and these temperatures are routinely achieved even in bonfires (e.g. Tobert, 1984).

Nubian wheel-made vessels were probably made at specific production centres by 'professional' potters and traded locally. They were fired in kilns that reached temperatures of around 850°C (Adams, 1986b:32). The prevalence of Nubian wheel-made wares fluctuated inversely with the hand-made wares, but typically represents 60-70% of the total number of ceramics found in Meroitic

and Post-Meroitic Nubia (Adams, 1986b:38, Fig. 9). It is sometimes thought that there was a slightly higher abundances of hand-made wares were present in more impoverished times, with the reverse being true during times of plenty, thus explaining the inverse relationship between the quantities of the two types found during excavation. Whether this is the case or not, it is likely that one of the functions of the pottery vessels included the exhibition of the social standing of the user; such that the wheel-made wares would be preferred over the hand-made wares (Adams, 1986b:36-37). The remainder of vessels excavated from Meroitic and Post-Meroitic phases are the imported wares from Egypt, which are said to make up 20-30% of the total ceramic assemblage. The following describe the main closed form (pots/jars) vessels that have been selected for this study (Fig 1.5); for a more comprehensive list of the entire ceramic attributes see Adams (1986).

The principal hand-made ware found in the Meroitic and Post-Meroitic periods was the H1, the 'Domestic Plain Utility Ware', with the most common forms being pots and jars. They are made from a Nile mud paste, the inclusions known to have been utilised were sand, chopped straw, grass and dung, and they tend to have relief decoration on the rim (Adams, 1986a:413).

There is a chronological development of Nubian wheel-made wares that sees the introduction and phasing out of specific ware-types (Fig. 1.6). The principal wares from the periods under study are: R32 (Meroitic), R25 (Early Post-Meroitic), and R1 (Post-Meroitic), which exist in a variety of different forms. The R32 'Meroitic Ordinary Red Wares' are constructed from Nile mud paste, the inclusions utilised were sand, chopped straw and dung, and they often have painted decoration on the surface (Adams, 1986a:454-455). The R25 'Early X-group Brown Ware' and the R1 'Classic X-group Red Ware' are made from Nile mud paste, the inclusions utilised were sand, ground sherd and occasionally chopped straw or dung, and they are rarely decorated (Adams, 1986a:468-469). Examples of some Post-Meroitic hand-made and wheel-made wares are shown in Plate VI.

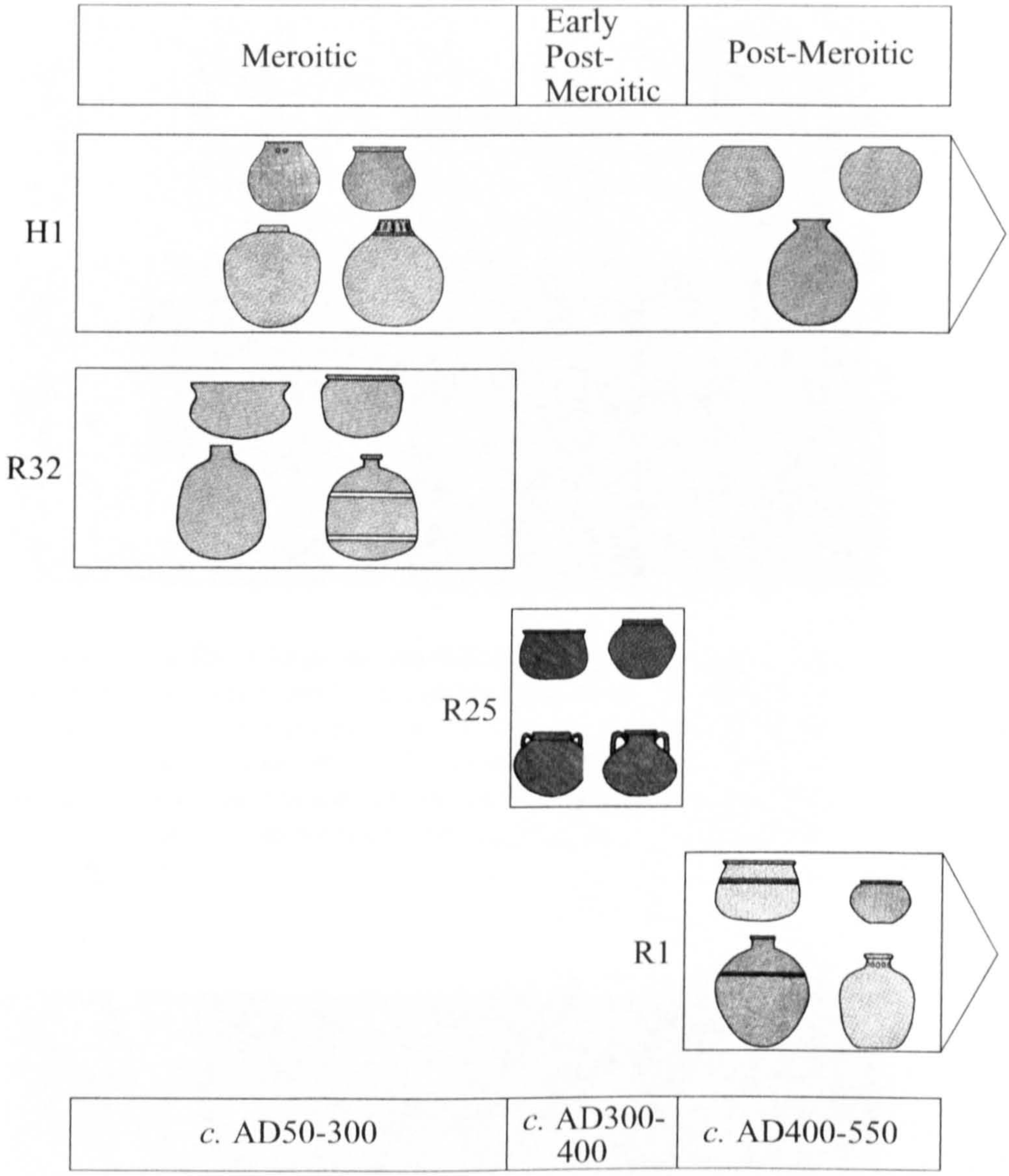

Figure 1.6 Pottery chronology at Qasr Ibrim. Selected vessel types are shown: at the top of each box are the pots, and the jars are shown towards the bottom of each box. Illustrations of the pots/jars are from Adams (1986a,b), and are not all on the same scale. The introduction and the phasing out of the various forms would not have been completed as abruptly as the figure suggests.

Plate I. Qasr Ibrim from the 'mainland'. The ancient approach to the site can be clearly seen in the foreground, which would have led to the main entrance; the course of the Nile would have past Ibrim on the far side. The Christian church dominates the site, but structure 265 lies on the side closer to the camera, near to the water's edge. Much of the ruins are now under water, and the only way of getting to the site is by boat.

Plate II. The main temple precinct. In the background is the 'cathedral', in the bottom right-hand corner of the picture is the entrance to one of the Temples. Some of the flagstones in the precinct area have pictures of feet etched into them, a practice known to have been done by religious pilgrims to the site.

Plate III. The area around Structure 265. The photograph was taken facing the 'mainland' and shows the internal structures of the building (Source: David Edwards).

Plate IV. Close up of the area under excavation. The mud-brick walls can be seen, along with doorways. There are niches in the walls, where 'lamps' may have been placed; 'oily' marks on the walls can even be witnessed in places (Source: David Edwards).

Plate V. Pottery and basketry from one of the contexts. Also of interest are the large quantities of organic material, which survives due to the arid environment, and can be seen on the left of the picture (Source: David Edwards).

Plate VI. Examples of the potsherds selected for this study. The top four sherds are hand-made wares from context 10107 (from the Post-Meroitic period); whilst the eight on the bottom are wheel-made wares from 10107. The first two columns show the interior of the sherds, whereas the last two columns show the exterior of the sherds.

1.4 THE ARCHAEOBOTANICAL AND ZOOARCHAEOLOGICAL EVIDENCE

As regards to Nubian crop cultivation and animal husbandry in the past, there are a number of different sources of information. Classical writers such as Strabo, Pliny, and Herodotus have written about the region, essentially concerning the Meroitic period, and there is also the archaeological evidence from excavated material. Finally, there is the knowledge of the diversity and extent of modern-day flora and fauna species. These shall be examined with regard to Nubia as a whole, and then with particular focus on the archaeological material from Qasr Ibrim.

Strabo (*c.* 64BC-AD23) stayed in Egypt for a long period of time, and although he never visited Nubia, he did have acquaintances that had, and from whom he could gain information (Eide *et al.*, 1994:811). In *Geography* 17.2[1-2] he writes of the Nubians:

> "They lead a miserable life, go poorly clad for the most part, and are nomads. Their domestic animals are small: sheep, goats and cattle. The dogs too are small... They live on millet and barley, from which they also make a beverage. Butter and suet serve as their olive oil. Nor do they have fruit trees except for a few date-palms in the royal gardens. Some even eat grass, soft twigs, water-lily, and reed root. They make use of meat, blood, milk, and cheese."
> (Eide *et al.*, 1994:815)

There is some uncertainty as to whether the millet described by Strabo is common millet (*Panicum miliaceum* L.) or actually sorghum (Rowley-Conwy, 1991:206-207). Strabo also reported that date palms were found "in abundance" in Aithiopia [Nubia] (Strabo transl. Jones, XVII:2.2).

Pliny (*c.* AD23-79) wrote *Natural History*, which comprised 37 books. He did not visit Nubia, but compiled references from other sources. In *Natural History* 13[90] he writes:

> "Aithiopia [Nubia], which has a boundary common with Egypt, barely has any remarkable tree except the one that yields flax [cotton]... In addition, there are palm trees..."
> (Eide *et al.*, 1994:874)

As of yet, the studies in the region have not concentrated on the zooarchaeological evidence; hence little is known about the nature of animal husbandry in Nubia. The herding of caprids and bovids is thought to have been widely practiced, however little work has been completed outside of Meroë (Edwards, 1998b). At Meroë, a large quantity of cattle remains was observed (Carter and Foley, 1980), although given the religious significance of the site and of the cow, it is hard to interpret from this the actual importance of cattle to the ordinary Nubian (Edwards, 1989:149). Although large populations of cattle are also seen in earlier times, for example at Kerma (Chaix and Grant, 1993), it is likely that in Lower Nubia as a whole, sheep/goat would have predominated, with lesser numbers of cattle (Rowley-Conwy, *pers. comm.*). Fish are also likely to be important to the Nubian diet (e.g. Nile Perch), although their remains may be underestimated without careful excavation (Rowley-Conwy, 1994). At the early Meroitic habitation site of Gezira Dabarosa, the faunal record has indicated that sheep, goat and cattle are most prevalent. No fowl or swine were found, but large quantities of fish were discovered. With regard to the botanical remains, date kernels and carbonised wheat were also recovered (Hewes, 1964).

Strabo reported that the Nubians ate meat, blood, milk and cheese, as detailed above; while there is no direct evidence for all of this in the past, modern Sudanese people are known to drink milk and also to ferment the surplus, for use during the dry season (Abdelgadir *et al.*, 1998). Fermentation is essentially a method of preserving foodstuffs, and there is similar evidence for the processing of meat in the region (Dirar, 1994).

With the introduction of the *saqia* in the Early Post-Meroitic (Rose, 1995), it is likely that a greater quantity and variety of crops would have been able to be cultivated, especially in the desert north. If ancient/modern Egypt and modern Sudan is used as an analogy for ancient crops in Nubia, then beans, lentils and green vegetables were likely to have been utilised (Welsby, 1996:160). Cereals such as sorghum, wheat, barley and millet are also known.

Sorghum cultivation appears to predominate in the Meroitic period in Nubia (Welsby, 1996:160). *Sorghum bicolor* Moench. is known from Neolithic times in Egypt (Vartavan and Asensi Amorós, 1997:242), and domesticated forms are common from the Meroitic period (e.g. Rowley-Conwy, 1991:196). Modern Sudanese make bread and beer from sorghum, and it is seen as their staple food (Dirar, 1993:168, 224). Emmer wheat (*Triticum dicoccum* Schübl.) has been recovered from archaeological sites from the Neolithic in Egypt (Vartavan and Asensi Amorós, 1997:260), and although is not as common in Nubia, emmer bread can be made from the cereal (Täckholm *et al.*, 1941:242-252). Free-threshing wheats such as *Triticum durum* L. and *Triticum aestivium* L. increase in prevalence in Ptolemaic Egypt (Crawford, 1979) and are subsequently found in small quantities in Nubia (e.g. Rowley-Conwy, 1989:133).

Barley (*Hordeum* L.) has been found from Neolithic sites in Egypt (Vartavan and Asensi Amorós, 1997:127-133), and is commonly found in Nubian sites (e.g. Rowley-Conwy, 1989:133). It is used in the making of Egyptian bread and beer (Samuel, 2000:547) and also used as fodder (Murray, 2000a:512). Millet (*Panicum* L.) has been used as both food and fodder, and has been noted in Nubia from the Napatan period (e.g. Rowley-Conwy, 1989:134), and at least from Neolithic times in Egypt (Brewer *et al.*, 1994:31). Modern Sudanese people eat a porridge called *aceda* made from either sorghum or millet (Dirar, 1994; Dirar, 1993:112) and forms a major part of their diet.

Cotton cloth production is recognised as an important local industry in antiquity (Edwards, 1998b), and perhaps provided Nubia's only truly manufactured export (Welsby, 1996:160). Indeed, cotton (*Gossypium* L.) has been found at a Neolithic site approximately 10 miles northwest of Qasr Ibrim (Chowdhury and Buth, 1971). From the Ptolemaic period onwards,
the development of trade in wines from Egypt to Nubia is known, attested by the large quantities of excavated amphora (Adams, 1988). Various fruit, vegetables and pulses that were found in ancient Egypt are listed in Table 1.2.

Table 1.2 Commonly found fruit, vegetables and pulses in Ancient Egypt[1]

Common name	Latin name	First found in Egypt
Date palm	*Phoenix dactylifera*	Predynastic?
Dom palm	*Hyphaene thebaica*	Late Palaeolithic
Argun palm	*Medemia argun*	5th Dynasty
Sycamore fig	*Ficus sycomorus*	Predynastic
Common fig	*Ficus carica*	Predynastic
Pomegranate	*Punica granatum*	12th Dynasty
Persea	*Mimusops laurifolia*	3rd Dynasty
Egyptian plum	*Cordia myxa*	Middle Kingdom
Carob	*Ceratonia siliqua*	12th Dynasty?
Christ's Thorn	*Ziziphus spinachristi*	Predynastic
Grape	*Vitis vinifera*	Predynastic
Olive	*Olea europaea*	13th Dynasty
Onion	*Allium cepa*	13th Dynasty?
Leek	*Allium ampeloprasum* var. *porrum*	? (One single find)
Garlic	*Allium sativum*	18th Dynasty
Lettuce	*Lactuca sativa*	Roman?
Celery	*Apium graveolens*	18th Dynasty
Water Melon	*Citrullus lanatus*	Predynastic
Melon	*Cucumis melo*	Predynastic
Radish	*Raphanus sativus*	12th Dynasty?
Chufa, tiger nuts	*Cyperus esculentus*	Predynastic
Lentils	*Lens culinaris*	Predynastic
Pea	*Pisum sativum*	Predynastic
Chick	*Cicer arietenum*	18th Dynasty
Faba bean	*Vicia faba* var. *minor*	5th Dynasty

[1]After Murray (2000); Tables 24.2,.3,.4: pp. 614-616.

Due to its general importance to the region, evidence for the use of the palm has been investigated in more detail. Date palm (*Phoenix dactylifera* L.) and dom palm (*Hyphaene thebaica* (L.) Mart.) remains are frequently found on archaeological sites in Nubia, with the excavation of the kernels being reported throughout the region. Recovery of other parts of the tree (for example the leaves) are restricted to areas where the preservation is particularly good, as at Qasr Ibrim. In modern Nubia it has been recorded that palms have been offered as collateral for loans, given as gifts, used in the manufacture of baskets, ropes, rugs, furniture, and fencing, and used for animal feed and fuel (Treloar, 1884:18; Beadnell, 1909:218; Adams, 1977:53; Lucas and Harris; 1962:444; Gale *et al.*, 2000:347-348). Today, and in the recent past, the fruit is heavily utilised as a food and its importance to the Nubian economy and society would

be expected in the past as it is today. Interestingly, no direct evidence for the processing of dates has been proven through the archaeology of the region (Welsby, 1996:160).

Modern botanical surveys of Nubia have shown numerous varieties of plants to exist in the Nile region. These include fruit such as mango (*Mangifera indica* L.) and the tangerine (*Citrus aurantium* L.), the aubergine (*Solanum melongena* L.), cacti (e.g. *Opuntia ficus-indica* MILL.), as well as numerous weeds, ivies and water plants (Boulos, 1966). Of course, the regional extent of these plants in antiquity is unknown at present.

1.4.1 The environmental evidence from Qasr Ibrim

Qasr Ibrim provides one of the best palaeobotanical assemblages that exists in Nubia, primarily due to the arid environment that preserves the morphological features of the samples. Therefore botanical investigations at Ibrim form the major part of what is known about Nubia's (and especially Lower Nubia's) archaeobotanical record. Rowley-Conwy gives an account of the archaeobotanical remains at Qasr Ibrim, and unless otherwise stated, the following draws from his work (Rowley-Conwy, 1989; Rowley-Conwy, 1991).

In the Napatan period the most common cereal that is found was the six-row hulled barley (*Hordeum vulgare* L.), although in lesser quantities, other crops are also found: e.g. Emmer wheat (*Triticum dicoccum* L.), common millet (*Panicum miliaceum* L.), and wild sorghum (of unknown race). The only pulse so far discovered is the lentil (*Lens culinaris* L.), although this is probably an under-representation of Napatan pulses. There is also evidence for the date palm (*Phoenix dactylifera* L.), dom palm (*Hyphaene thebaica* (L.) Mart.), Egyptian balsam (*Balanites aegyptiaca* L.), fig (probably *Ficus cf sycomorus* L.), the ben-oil tree (*Moringa peregrina* L.), linseed (*Linum usitatissimum* L.), and castor bean (*Ricinus communis* L.). The vegetables include watermelon (*Citrullus lanatus* L.) and the cucumber (*Cucumis cf sativus* L.). Onion (*Allium cepa* L.), garlic (*Allium sativum* L.) and coriander (*Coriandrum sativum* L.) are also known.

During the Roman period, most of the Napatan flora are present, although the quantity of common millet increases. Cotton seeds (*Gossypium herbaceum* L.) first appear here, although it is likely that they were only locally grown in later periods. With the onset of the Meroitic period, the first cultivated sorghum (*Sorghum bicolor bicolor*) starts to appear, and becomes one of the dominant crops (along with barley). Towards the end of the Meroitic the wheats *Triticum durum* L. and *T. aestivium* L. are found but do not represent a significant proportion of the total assemblage.

During the Post-Meroitic, other plants were also being utilised. These include the termis bean (*Lupinus albus* L.), which became the most important pulse at Ibrim, the pea (*Pisum sativum* L.), sesame (*Sesamum indicum* L.), and pearl millet (*Pennisetum typhoides* L.) is also found. Furthermore, it is known that the modern Sudanese utilise these crops, and actually make a cake out of the sesame remains following oil extraction (Elfaki *et al.*, 1991; Dirar, 1993:447).

More recently, Alan Clapham has studied botanical remains from the Meroitic and Post-Meroitic contexts from which the pottery samples for this study were taken (Clapham, 1998). Many of the species detailed above were found; in addition safflower (*Carthamus* L.) and olive (*Olea europaea* L.) were identified (although were not probably cultivated). The food crops chickpea (*Cicer arientinum* L.) and cress (*Lepidium sativum* L.) were also found. There was also the presence of certain plants that were added to food, such as the spices coriander (*Coriander sativum* L.) and carob (*Cerotinia sililiqua* L.). In one context (18003), the hyacinth bean (*Lablab purpureus* L.) was found in abundances not seen in other contexts.

Archaeozoological remains at Qasr Ibrim have not been as intensely studied as the botanical remains. Although sheep/goat are likely to predominate in the valley economy, the analysis of a context associated with one of the Temples at the site has indicated that juvenile cattle bones are the most frequently found faunal remains, with an age range that may be indicative of a mixed meat/milk economy. It may be that the priests at the Meroitic Temple were selectively

removing the young cattle from the valley economy below (Rowley-Conwy, *pers. comm.*). Indications of the existence of other animals at Qasr Ibrim includes: one chicken specimen (Macdonald and Edwards, 1993), and from the presence of camel dung at Ibrim, one of earliest incidences of camel in Nubia (Rowley-Conwy, 1988).

1.5 AIMS

The Meroitic and Post-Meroitic periods of Nubian archaeology cover a time of political, economic and cultural change, with the end of the Kushite Empire, the emergence of a weaker Egypt and the potential resultant reduction in long-distance trade, as well as technological changes such as the introduction of the *saqia* and the establishment of new crops.

Due to the extremely arid nature of the Lower Nubian environment there has been exceptional macroscopic preservation of plant and animal material. This level of preservation is mirrored at the molecular level. Therefore, through the large-scale chemical analysis of carefully selected pottery vessels, and the use of plant and animal remains as references, it is possible to detect the commodities that were being processed in the vessels, and hence the following questions can be answered:

(i) Is there a change in vessel use over time? And does this correspond to changes in the Meroitic and Post-Meroitic economies?

(ii) Is there a distinction in use of vessels of different ware types (especially hand-made and wheel-made wares)?

(iii) Do the palaeoenvironmental reference materials show any trends over time?

Modern and archaeological faunal and botanical materials were initially analysed, through gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) of the total lipid extract (TLE), and through GC and gas chromatography-combustion isotope ratio mass spectrometry (GC-C-IRMS) of the fatty acid methyl esters (FAMEs). This not only enabled the state of molecular preservation to be assessed, but also lipid characteristics were identified, which could then be used in the statistical separation of the commodity groups. Once the criteria to differentiate between commodity groups were established, the analyses of the sherds could proceed, allowing classification of the lipid extracts.

Chapter 2.

Materials and Methods

2.1 GENERAL

2.1.1 Glassware and solvents

Reusable glassware was cleaned with Micro, rinsed with double distilled water and acetone, oven dried and then placed in furnace (500°C, 2 h). Disposable and reusable glassware were rinsed with solvent prior to use. HPLC grade solvents were utilised (Rathburn).

2.2 MATERIALS

Archaeological pottery, faunal and botanical samples were provided by the Egyptian Exploration Society (EES), 3 Doughty Mews, London WC1 2PG, from the excavations at Qasr Ibrim. Pottery samples were collected with the assistance of Dr. Pam Rose (EES) from the excavations of Dr. David Edwards (School of Archaeological Studies, University of Leicester) and Dr. Alan Clapham (EES) and Peter Rowley-Conwy (Durham University) provided the botanical specimens.

2.3 LIPID EXTRACTION AND DERIVATISATION

2.3.1 Sample preparation

The analytical protocol is outlined in Figure 2.1.

Pottery vessels: Approximately 2 g of pottery sherd was surface cleaned with an electric hand-drill, and crushed in a mortar and pestle. The powdered sherd was weighed and then 20 μg of internal standard (*n*-tetratriacontane) added.

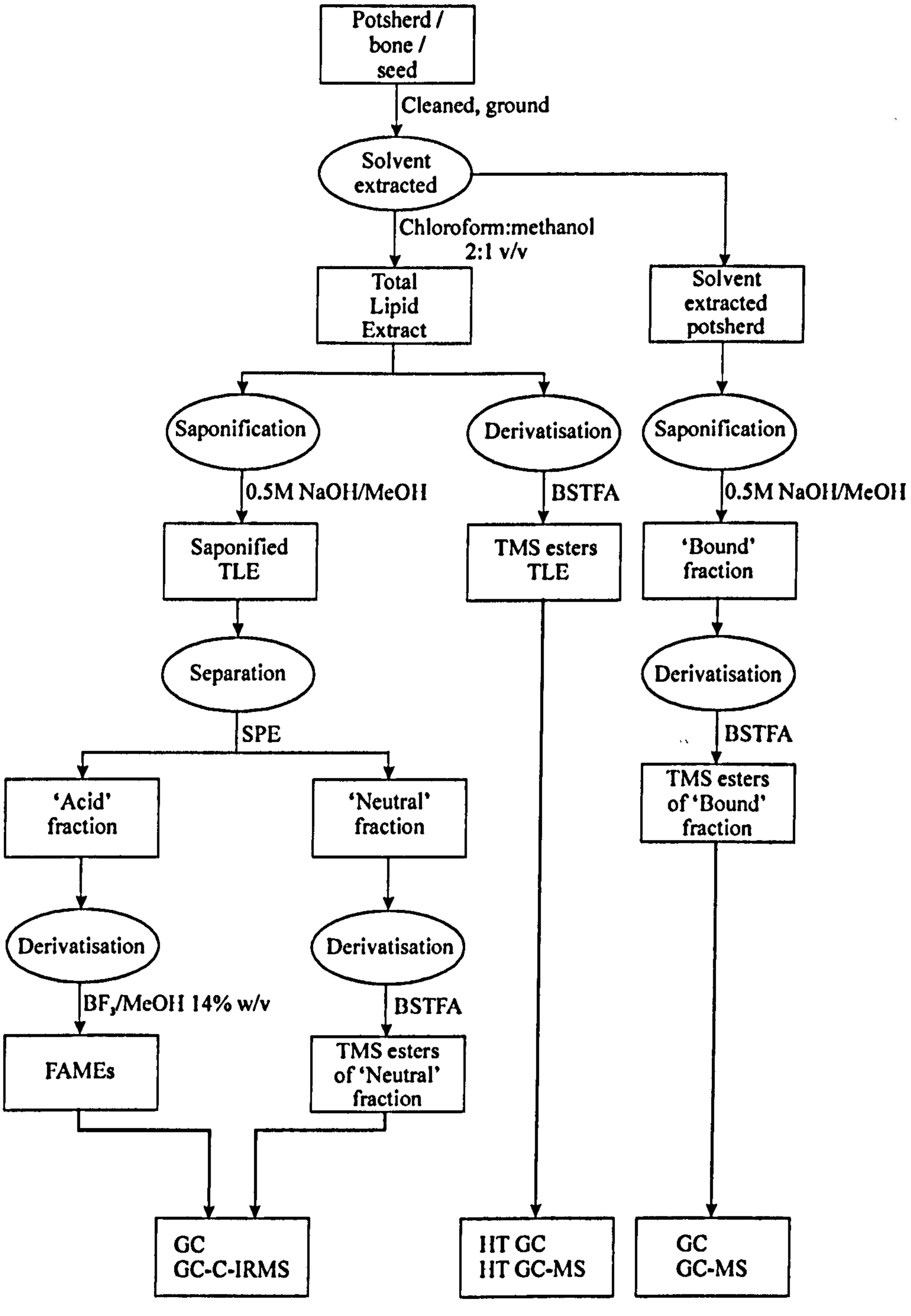

Figure 2.1 Protocol used for the extraction and analysis of lipids from the bone and plant reference materials and pottery vessels.

Seeds: Individual seed samples, where possible, were surface cleaned with an electric hand-drill, and then rinsed in fresh chloroform. Approximately 100-200 mg of seed (which generally equated to a few individual seeds) was crushed to a fine powder in a mortar and pestle with (where needed) liquid nitrogen. Either 20 or 100 μg of internal standard (*n*-tetratriacontane) was added to the crushed seed.

Bones: A pre-washed, solvent rinsed saw was used to obtain approximately 1-2 g of bone. This was then surface cleaned with an electric hand-drill, and rinsed in fresh chloroform. The cleaned bone samples were then crushed in a mortar and pestle with liquid nitrogen. One hundred micrograms of internal standard (*n*-tetratriacontane) was added.

2.3.2 Solvent extraction

Sherds, seeds and bones: All samples were solvent extracted on the same day that they were crushed. Lipids were extracted using chloroform/methanol solution (2:1 v/v, 10 ml, 2 x 15 min sonication). For the bone and seed samples, this was repeated three times and the extracts combined. This lipid extract was then centrifuged (25 min, 2000 rpm) decanted and then filtered through solvent washed silica gel. This total lipid extract (TLE) was then dried under a gentle stream of nitrogen, and stored at -20°C until required for derivatisation and analysis.

Boiled dates: Two dates were cut into half and, along with two whole dates, were vigorously boiled for 1.5 h in 250 ml of DCM extracted double distilled water. The volume of water was kept constant by the continual addition of more water. After the water had cooled, the remnants of the date flesh and kernels were removed, and the remaining organic material in the water was extracted with 3 x 100 ml of DCM. This DCM extract was then dried under a gentle stream of nitrogen, and stored at -20°C until required for derivatisation and analysis.

2.3.3 Hydrolysis and separation

An aliquot of the TLE (typically 1/3) was hydrolysed with 0.5M NaOH in MeOH solution (2 ml, 70°C, 1 h), cooled and then acidified to pH 3 with 3M HCl. Hexane was used to extract the hydrolysed lipids (3 x 3 ml), and these were then combined and the excess solvent evaporated under a gentle stream of nitrogen. The extracts were then separated into their 'acid' and 'neutral' fractions through use of aminopropyl solid phase columns (Isolute 3 ml, 500 mg sorbent mass) that had been flushed with hexane (6 ml). First the neutral fraction was eluted with chloroform/*iso*-propanol (2:1 v/v, 6 ml) and then the acid fraction was eluted with 2% acetic acid in diethyl ether (6 ml). These extracts were dried under a gentle stream of nitrogen and stored at -20°C until required for derivatisation and analysis.

2.3.4 Hydrolysis of the 'insoluble' fraction in pottery vessels

Following the solvent extraction of some of the ground pottery vessels, 1 g of the remaining insoluble residue was base treated with 0.5M NaOH in MeOH solution (5 ml, 70°C, 1 h) whirlimixing every 5 minutes. After being allowed to cool, the mixture was centrifuged (2000 rpm, 25 min) and the solvent decanted. This was acidified to pH3 (3M HCl), 1 ml of dichloromethane (DCM) extracted double distilled water was added, and then extracted with chloroform (3 x 3 ml). The solvent was evaporated under a gentle stream of nitrogen and the extract stored at -20°C until required for derivatisation and analysis.

2.3.5 Derivatisation

Trimethylsilyl (TMS) derivatives of the TLE and 'neutral' and 'bound' fractions were obtained by heating the sample with *N,O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA, Sigma) containing 1% trimethylchlorosilane (30 µl, 70°C, 1 h). The BSTFA was evaporated under nitrogen, and the samples were then ready for instrumental analysis.

Fatty acid methyl ester derivatives (FAMEs) of the saponified fatty acid fraction were prepared with the addition of 100 µl of boron trifluoride-methanol complex (14% w/v, Aldrich) and heated at 75°C for 1 h. In order to stop the reaction, the mixture was cooled and 2 ml of double distilled, DCM extracted water added. The FAMEs were then extracted with chloroform (6 ml), and the solvent evaporated under nitrogen and the samples were then ready for instrumental analysis.

2.4 Gas Chromatography and Gas Chromatography-Mass Spectrometry

Gas chromatographic (GC) analyses were performed on a Hewlet-Packard 5890 Series II gas chromatograph fitted with a flame ionisation detector. Data acquisition and processing were preformed using HP Chemstation software.

2.4.1 Total lipid extracts

The TLEs were introduced by on-column injection onto a DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 µm film thickness), the carrier gas was hydrogen and the GC oven temperature programmed from 50°C, following a 2 min hold after injection, to 350°C at 10°Cmin^{-1} followed by an isothermal 10 min hold at 350°C. Quantification of the total lipid extract was accomplished through the addition of a known quantity of internal standard (20µg of tetratriacontane). Electronic integration of the peak areas is known to give an overall precision of <4% (Braithwaite and Smith, 1996:41).

2.4.2 Fatty acid methyl esters

The FAMEs and alditol acetates were analysed using a CP wax 52 CB (polyethylene glycol) fused silica capillary column (50 m x 0.32 mm, 0.2 µm) or a BPX70 (70% cyanopropyl equivalent modified siloxane). In both cases,

the FAMEs were introduced via an on-column injector, and the carrier gas was hydrogen with the GC oven programmed from 40°C to 100°C at 10°C min^{-1}, then from 100°C to 240°C at 4°C min^{-1} and held at 240°C for 20 min.

2.4.3 The 'neutral' fraction

The neutral fraction was analysed using a CP Sil 5CB (100% polymethyl siloxane) fused silica capillary column (50m x 0.32 mm, 0.12 μm film thickness). The GC conditions were as follows; 40°C to 200°C at 10°C min^{-1}, then 200°C to 300°C at 4°C min^{-1} and held at 300°C for 10 min.

2.4.4 Gas chromatography-mass spectrometry

Gas chromatography-mass spectrometry (GC-MS) analyses were performed using a Finnigan 4500 quadrupole mass spectrometer (Finnigan MAT GmbH, Bremen, Germany) directly coupled to a Carlo Erba HrGC 5160 Mega series GC with on-column injection. The mass spectrometer was set to scan in the range of *m/z* 50-600 in a total cycle time of 1.0 s, or if there were triacylglycerols present, *m/z* 50-850 in a total cycle time of 1.5 s. The same GC operating conditions were used as described above (Sections 2.4.1 and 2.4.2). Further operating conditions were as follows: Ion source temperature, 170°C; emission current, 300 μA; electron ionisation potential, 70 eV; the GC-MS interface was maintained at a temperature of 350°C; helium was used as carrier gas. Data were acquired and processed using an INCOS data system, peak identifications were aided by Interactive Chemical Information Structure (ICIS) software.

2.5 Gas Chromatography-Combustion-Isotope Ratio Mass-Spectrometry

Analyses were performed on a Varian 3500 gas chromatograph (Varian Associates Inc., Walnut Creek, CA) coupled to a Finnigan MAT Delta-S isotope

ratio mass spectrometer (Finnigan MAT GmbH, Bremen, Germany) via a modified Finnigan MAT (Pt/CuO) combustion interface. Removal of water after combustion was made possible through the use of Nafion tubing (Perma Pure Products Inc., Toms River, NJ). The Cu/Ni/Pt reactor was maintained at a temperature of 860°C. The mass spectrometer source pressure was 9×10^{-5} with helium being the carrier gas.

Two types of fused-silica capillary columns were utilised. One, coated with a 100% polymethyl siloxane stationary phase (CPSil-5 CB, 50 m x 0.32 mm, 0.12 μm film thickness, Chrompak) was utilised for the analysis of cholesterol in the 'neutral fraction', and for the samples that only contained saturated fatty acids. With a CP wax52 CB or BPX70 column used during the analysis of both the saturated and unsaturated fatty acid components.

Standardisation of runs was achieved with CO_2 gas pulses of known $\delta^{13}C$ value ($\delta^{13}C_{(CO_2)} = -31.8$ ‰) injected directly into the ion source of the mass spectrometer, furthermore a standard (octadecanoic methyl ester) of known value was used to test the accuracy of the GC-C-IRMS over time.

The carbon isotope ratios ($\delta^{13}C$) are the ratios of ^{13}C to ^{12}C, and are expressed relative to the standard reference material Vienna Pee Dee Belemnite (vPDB) by:

$$\delta^{13}C = \frac{(R_{sample} - R_{standard})}{R_{standard}} \times 1000$$

where $R_x = {}^{13}C_x/{}^{12}C_x$ in per mil (‰).

In order to account for the additional methyl group that the FAMEs contain, the $\delta^{13}C$ values of the FAMEs were corrected by utilising the mass balance equation employed by (Jones *et al.*, 1991):

$$\delta^{13}C_{FA} = \frac{(((n+1)\,(\delta^{13}C_{FAME})) - \delta^{13}C_{BF3MeOH})}{n}$$

where $\delta^{13}C_{FA}$ = the corrected value for the fatty acid, *n* = carbon chain length, $\delta^{13}C_{FAME}$ = the value obtained for the free fatty acid of carbon chain length *n*, $\delta^{13}C_{BF3MeOH}$ = the correction factor for the derivatising agent (BF_3-methanol) which for this study was -41.3 ± 0.3‰.

In order to account for the addition of the three methyl groups during the TMS derivatisation of cholesterol, a correction factor was calculated for the batch of BSTFA that was used in this study:

$$27\delta_{OH} + 3X = 30\delta_{OTMS}$$

(Stott & Evershed 1996).

where δ_{OH} is the $\delta^{13}C$ value for underivatised cholesterol standard (measured off-line, -24.9‰); δ_{OTMS} is the $\delta^{13}C$ value for the derivatised cholesterol standard (-26.7‰). Therefore, for this study, 3X, the correction factor to account for the additional carbon contributed by the BSFTA, was –128.7‰ (i.e. X = -42.9‰). This value was used in the same equation to calculate the δ_{OH} of the sample.

2.6 Statistical Evaluation of the Data

A number of different statistical methods were employed on the data set gathered herein. The majority of the statistical analyses were performed by using Systat® 7.0 (SPSS Inc., Chicago, Ill.). The following describes how the data evaluation was approached and which statistical methods were utilised.

2.6.1 The broad approach to the data analysis

The continuous variables were tested to determine whether they were normally distributed, using the Kolmogorov-Smirnov one sample test of distribution (see below). If the variable was not normally distributed, then non-parametric methods were employed on any further statistical analysis involving this

variable. However, if the variable was normally distributed, then parametric statistical methods were used. Discrete variables were tested using the appropriate tests (e.g. χ^2, see below).

2.6.2 Statistical methods utilised

Kolmogorov-Smirnov test of normality The distribution of a continuous variable can be assessed graphically or statistically, e.g. examining the kutosis or skewness of a distribution (Tabachnick and Fidell, 1996:71). However, the underlying distribution can be statistically tested using the Kolmogorov-Smirnov test of normality (Fletcher and Lock, 1991:99), and it was this latter method that was used prior to the use of any further tests that assume that the underlying distribution of the variable is normal.

Kolmogorov-Smirnov two sample test This tests whether two samples are from the same population, and does not assume that the underlying distribution is normal; only that it is continuous.

T-test The two sample T-test assumes that the groups are both normally distributed, and tests the mean value between the two groups. If the variances of the groups are equal (i.e. the shape of the distributions are the same) then pooled variance T-test is used; where this is not the case, then separate variance T-test is used (SPSS, 1997:104).

ANOVA ANOVA (ANalysis Of VAriance) is useful in testing the difference in means across more than two groups. This statistical method was of particular use in determining changes that occur over the three archaeological periods from which the pottery was selected. In the statistical analyses, firstly the data set is submitted to ANOVA to determine whether there is a difference in the mean values over the three periods. Then using a Bonferroni type adjustment (Tabachnick and Fidell, 1996:402) pairwise mean comparisons can be made, therefore enabling differences in the mean values of a variable to be highlighted between each of the three periods, rather than across all three of them.

F-test of variance This test assumes that the two variables are normally distributed. It determines whether one group shows more variability than another group (Fletcher and Lock, 1991:82).

Chi-squared test of association The χ^2 test is used to test the association of two discrete variables (Fletcher and Lock, 1991:116). A Yates correction factor should sometimes be included in the analysis (Kirk, 1999:363), especially when a 2 x 2 contingency table is used in the test (Fletcher and Lock, 1991:118). The actual χ^2 value cannot be used to compare different tests, however where χ^2 is significant, Cramér's measure of association (V) can be used to compare the strength of associations between different tests (Kirk, 1999:565).

Hierarchical clusters Single linkage clustering creates dendrograms based on using the normalised Euclidean distance between the two closest members of clusters of variables. All variables were standardised to range prior to analysis ($Value_{standardised} = [Value_{sample} - Value_{minimum}] / [Value_{maximum} - Value_{minimum}]$; therefore $Value_{standardised}$ has a range of between 0 and 1).

Chapter 3.

The Environmental Reference Materials: Results and Discussion

3.1 ZOOARCHAEOLOGICAL REFERENCE MATERIALS

3.1.1 General

The reference materials were selected from a number of archaeological periods, covering the whole occupation of Qasr Ibrim, and comprised 22 sheep/goat (with only bone fragments these are indistinguishable), 14 cattle, 2 pig and 7 unidentified bone fragments. As discussed above (Section 1.4) the ovi-caprine and bovine bones make up the vast majority of zooarchaeological record, with porcines appearing in Christian contexts (Rowley-Conwy, *pers. comm.*). Therefore, the inclusion of pigs in this survey was for completeness, and not used in the subsequent analysis of Meroitic and Post-Meroitic pottery. A full list of the context numbers, bone type, and notes concerning the size/age of the animal bones is given in Appendix 1 (Table A1.1). Figure A2.1 in Appendix 2 shows the chemical structures and the mass spectra of the major compounds identified herein.

3.1.2 Total lipid extracts

Substantial concentrations of lipid were extracted from the bones (up to 4.25 mg g^{-1}) as demonstrated in the box-plots in Figure 3.1. There is no significant

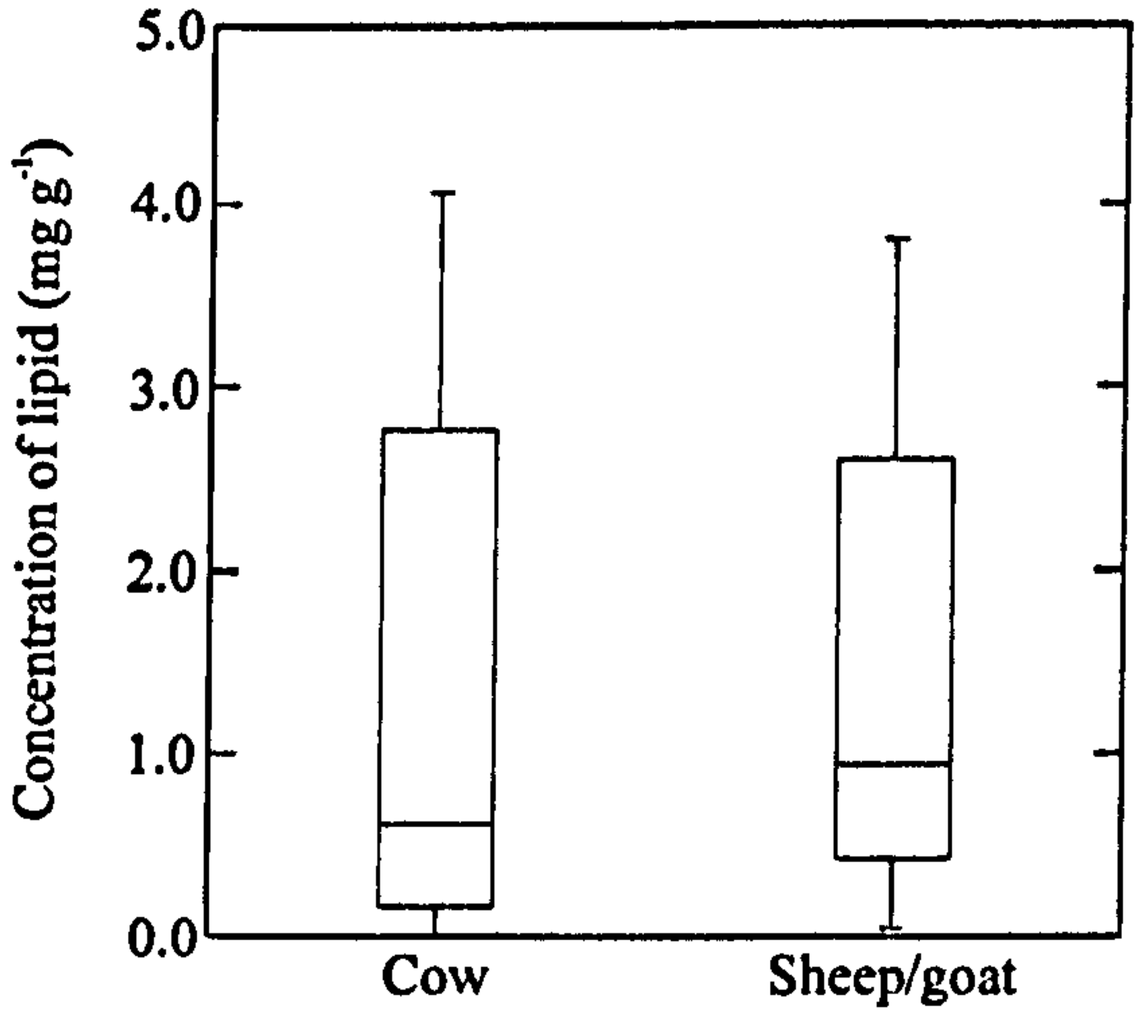

Figure 3.1 Box plots of the lipid concentrations from the animal bones. The horizontal line within the boxplot is the median value, 50% of the bones lie within the boxes shown.

difference between sheep/goat and cow, nor any correlation between period and the concentration of lipid.

An example of a partial gas chromatogram of the TLE of a cattle bone (sample B27pelv) is shown in Figure 3.2, and an ovi-caprine bone (sample 17035) in Figure 3.3. From the chromatograms, it can be seen that the extracts are primarily composed of saturated fatty acids in the range of $C_{14:0}$ to $C_{20:0}$, with $C_{16:0}$ and $C_{18:0}$ predominating. There is only one unsaturated fatty acid present ($C_{18:1}$), and in much lower abundance than the saturated acids; these distributions are typical of all the cattle and sheep/goat bones. The $C_{15:0}$, *iso* and *anteiso* branched $C_{15:0}$, $C_{17:0}$ and *iso* and *anteiso* branched $C_{17:0}$ fatty acids are also present in all of the samples, and most contain the branched chain $C_{16:0}$ homologue but $C_{18:0br}$ is rarely present.

In living animals, the majority of these fatty acids would have been found in the form of triacylglycerols (TAGs). However, it can be seen that complete hydrolysis of the TAGs has occurred during burial, as evidenced by the lack of TAGs, diacylglycerols (DAGs) and monoacylglycerols (MAGs): only free fatty acids are present, accompanied by very high abundances of glycerol. This is typical for all of the bone reference materials; only in one (sample 10048·2; cattle) are any TAGs present (C_{48}, C_{50}, C_{52}) and then only in very low abundances. There were rarely any DAGs, and only traces of MAGs were detected in five of the samples. In sample 10048·2, the lack of any substantial abundance of C_{54} TAG (3 x C_{18}) presumably is due to the fact that a large proportion of this compound would comprise various isomers of octadecenoic acid, which are particularly susceptible to oxidative decay.

The majority (33/45; 72%) of the bones yielded 9,10-dihydroxyoctadecanoic acids, and the mass spectrum of its TMS derivative is given in Figure 3.4, showing characteristic ions at *m/z* 215 and 317 corresponding to fragmentation between the two hydroxyl functional groups. These vicinal dihydroxy compounds are believed to be formed by the oxidation of the carbon-carbon double bond by dihydroxylation (Regert *et al.*, 1998), yielding two isomers, as can be seen in Figure 3.3, corresponding to their *threo* and *erythro*

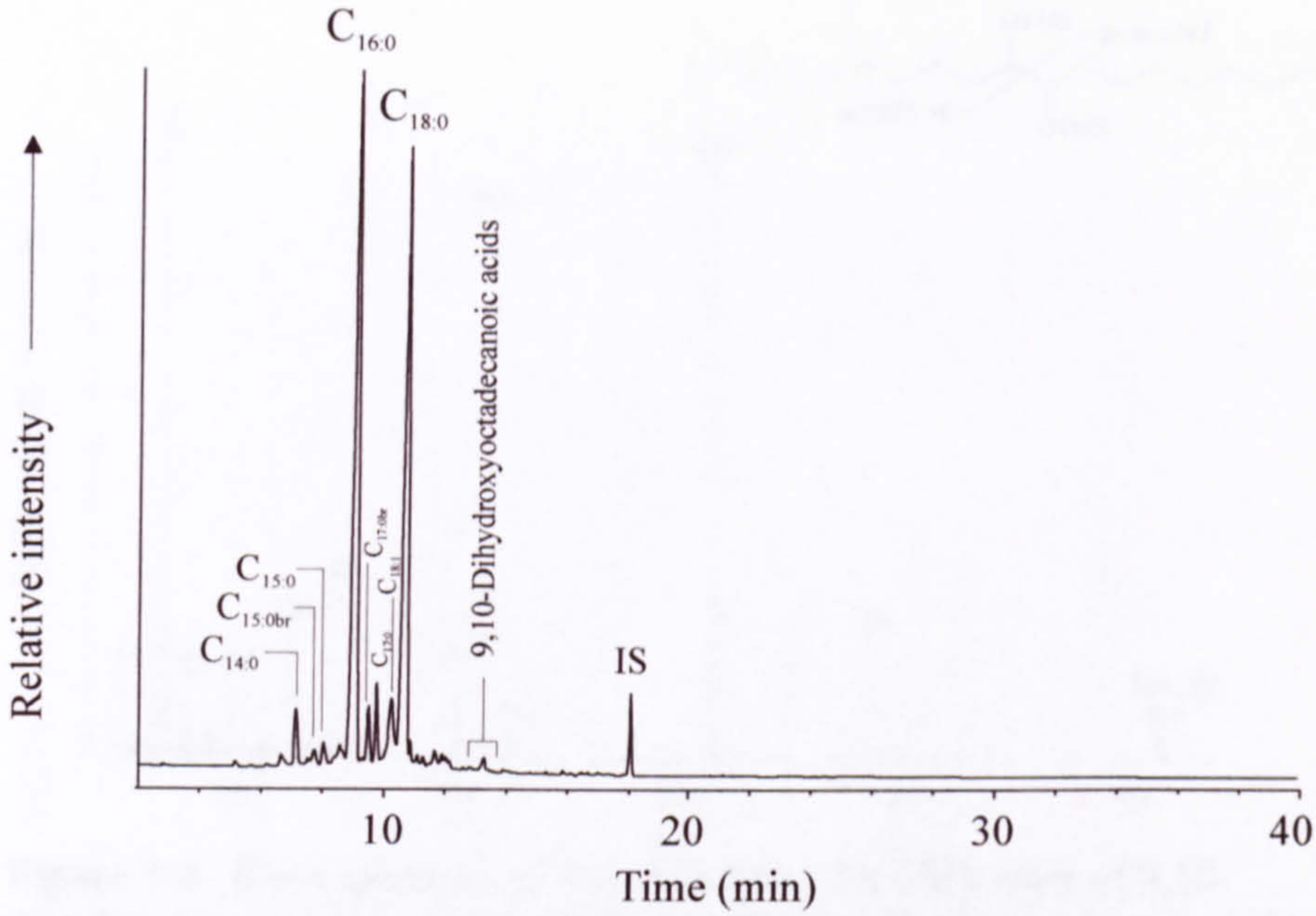

Figure 3.2 Partial high temperature gas chromatogram of TLE of sample B27pelv (cattle bone). $C_{x:y}$ represents carboxylic acids of carbon chain length x and degree of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

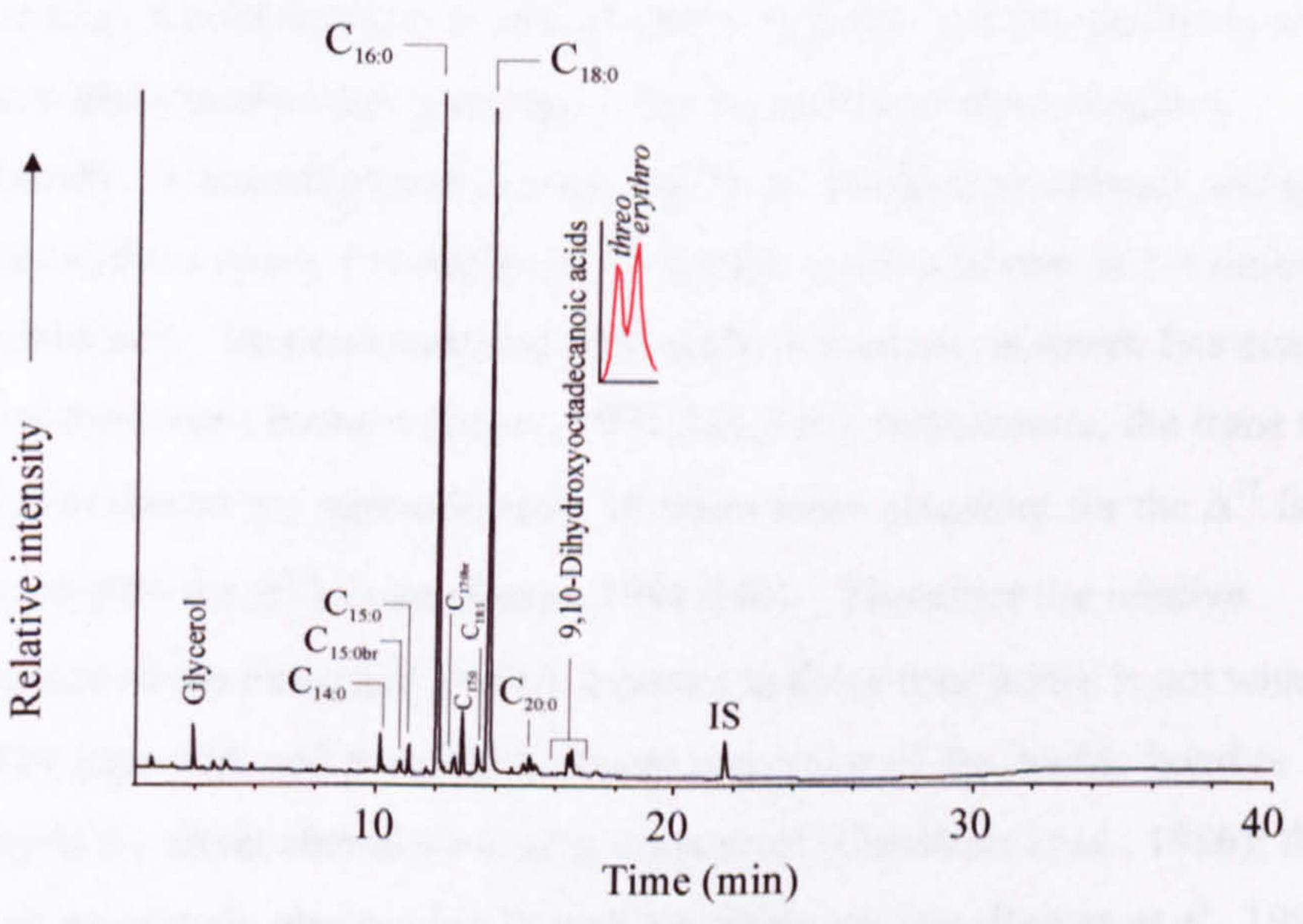

Figure 3.3 Partial high temperature gas chromatogram of TLE of sample 17035 (sheep/goat bone). $C_{x:y}$ represents carboxylic acids of carbon chain length x and degree of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). The *threo* and *erythro* isomers of 9,10-dihydroxyoctadecanoic acid (eluting in that order) can be seen to be of approximately the same relative abundance. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

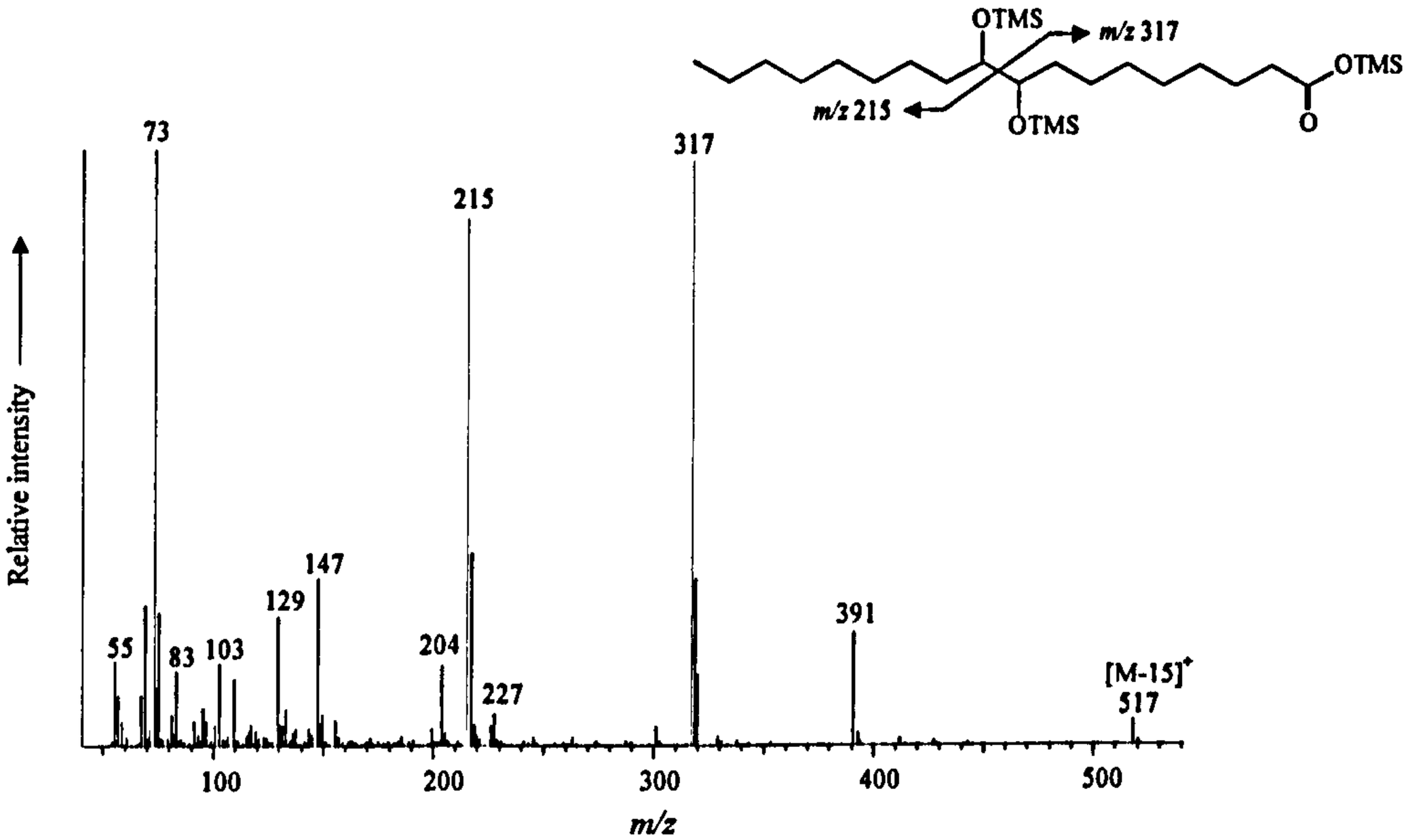

Figure 3.4 Mass spectrum of the TMS ester, *bis* TMS ether of 9,10-dihydroxyoctadecanoic acid. TMS derivatives of 9,10-dihydroxyhexadecanoic acid are characterised by abundant ions at *m/z* 73, 187 and 317.

diastereoisomers (eluting in that order). Hence, hydroxylation of the Δ^9 double bond in $C_{18:1}$ would result in hydroxyl groups at the 9- and 10- positions with the *threo* and *erythro* corresponding to the *cis* and *trans* stereoisomers, respectively. Generally these isomers are found in equal abundance, except in four cases (three cows, 1 sheep/goat) where the *erythro* isomer is 2-4 times more abundant. Monounsaturated fatty acids in modern ruminant fats contain more *cis* than *trans* isomers (Enser, 1991:331,346); furthermore, the trans fatty acids in ruminants are approximately 10 times more abundant for the Δ^{11} isomer compared with the Δ^9 isomer (Enser, 1991:346). Therefore the relative abundances of the *threo* and *erythro* isomers in these four bones is not what would be expected, and possibly indicates migration of the double bond or changes in the stereochemistry during diagenesis (Gunstone *et al.*, 1986); this has been previously observed in British Neolithic vessels (Regert *et al.*, 1998) and in lamps from Qasr Ibrim (Bland, 1999:173).

Figure 3.5 shows a partial gas chromatogram for the TLEs of a sheep/goat sample (IB82) with an unusual lipid distribution. The fatty acid composition

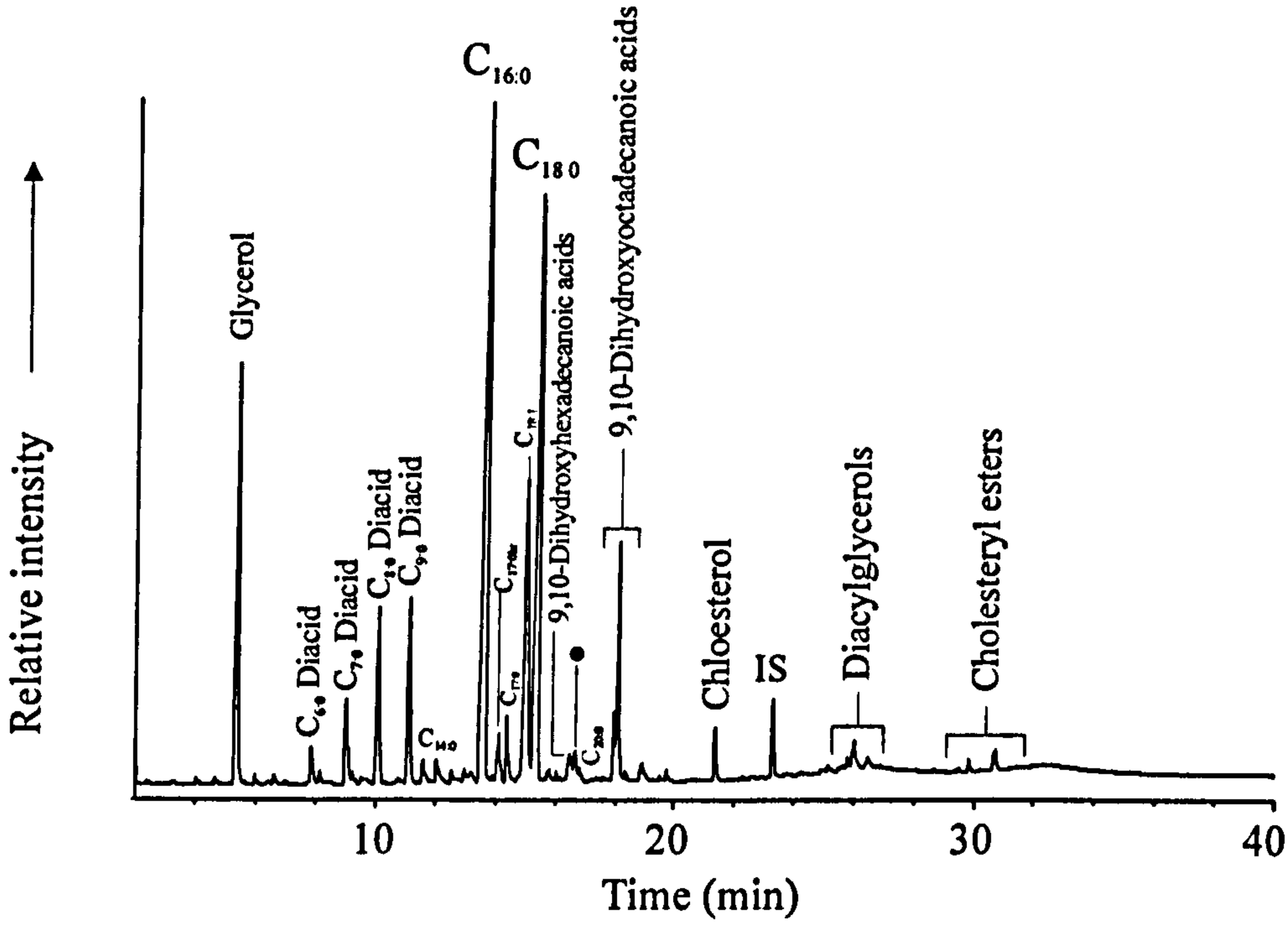

Figure 3.5 Partial high temperature gas chromatogram of TLE of sample IB82 (sheep/goat). $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. $C_{x:0}$ Diacid are α,ω-dicarboxylic acids of carbon chain length x. The dot represents a mixture of hydroxyoctadecenoic acids. IS denotes the internal standard (*n*-tetratriacontane). The *erythro* isomer of 9,10-dihydroxyoctadecanoic acid was found to be approximately 4 times as abundant as the *threo* isomer (see text). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

is similar to that of the previous examples, however, there are several lipids present that are indicative of oxidative decay. There is a high abundance of glycerol (formed from the hydrolysis of the ester linkages in the acyl moieties), although some DAGs and traces of MAGs are present, indicating that complete hydrolysis has not yet occurred. 9,10-dihydroxyoctadecanoic acids are also present, but the *erythro* isomer is *c.* 4 times as abundant as the *threo*. Also present in this sample is 9,10-dihydroxyhexadecanoic acid (with characteristic ions *m/z* 73, 187, 317 and $[M - 15]^+$ of 489; this compound is not found in any other of the bone samples. Oxidation of the unsaturated fatty acids is indicated by the presence of homologues of α,ω-dicarboxylic acids ('diacids') from $C_{6:0}$

to $C_{9:0}$, which are present in high abundances, with $C_{8:0}$ and $C_{9:0}$ predominating. The single other bone to yield appreciable quantities of diacids (sample B1-12; sheep/goat) only contained the $C_{8:0}$ and $C_{9:0}$ diacids. These α,ϖ-dicarboxylic acids have been observed in Nubian mummies, with $C_{9:0}$ being the major component (Gulaçar, 1989; Gulaçar *et al.*, 1990), and in a study of Nubian lamps; although the $C_{9:0}$ diacid still predominated, slightly greater abundances of diacids of lower molecular weight were also present (Bland, 1999:177).

α,ϖ-dicarboxylic acids are formed through the oxidative cleavage of the carbon-carbon double bond (Simic *et al.*, 1992), therefore the $C_{9:0}$ diacid derives from the Δ^9 unsaturated C_{18} acid (Gillan and Johns, 1982). Oxidation is likely to have occurred either in the burial environment, or during storage following excavation because firstly, these diacids are not present in living animals, and secondly, whilst it is possible that they could be formed through the boiling of the bones in a pot (e.g. during cooking), this is unlikely to be the case because one sample (IB82) had desiccated skin still attached to the bone; furthermore, boiling of the bones would result in the loss of a substantial quantity of lipid (not witnessed here), and would probably even change the appearance (colour) of the bone (Pearce and Luff, 1994).

Further evidence for oxidation is seen in a group of compounds eluting at *c.*16.5 min, just before $C_{20:0}$ in Figure 3.5. These are a mixture of hydroxyoctadecenoic acids that are unresolvable by GC, and have been detected in the laboratory decay, under oxic conditions, of olive oil (Aillaud and Evershed, *unpublished results*), and are believed to be formed through the oxidation of the carbon-carbon double bond. Figure 3.6 shows the summed mass spectrum for the co-eluting peaks (at *c.* 16.5 min). These hydroxy acids have characteristic fragmentation patterns, from which it is possible to gain information relating to the position of the hydroxyl functionality. For instance, 9-hydroxyoctadec-10-enoic acid has an abundant ion at *m/z* 227, whereas with

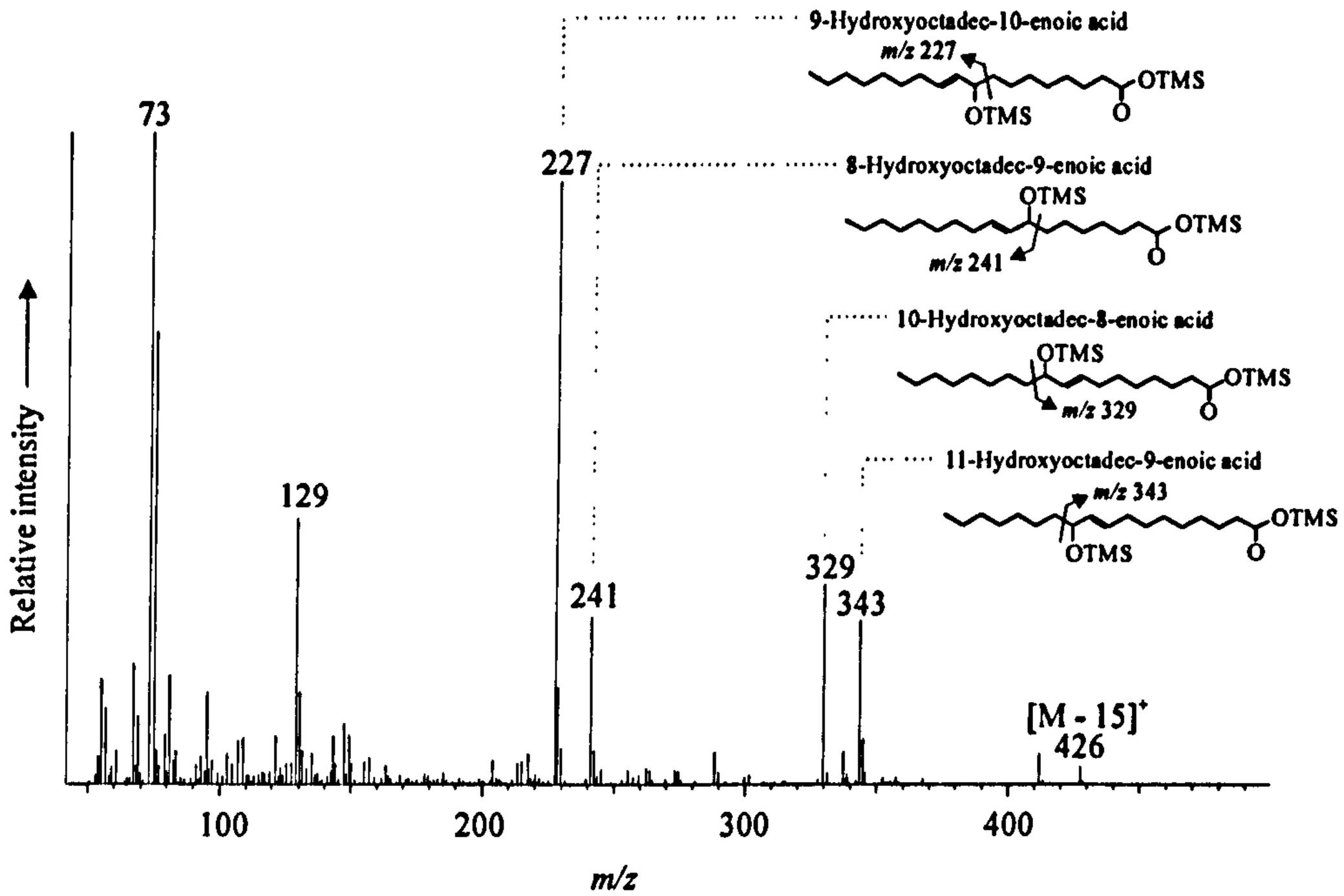

Figure 3.6 Summed mass spectrum of the peaks co-eluting at *c.* 16.5 min in Figure 3.5. The fragment ions shown indicate the position of the hydroxyl groups within the TMS derivatives of the hydroxy alkenoic acid.

10-hydroxyoctadec-8-enoic acid the ion is at *m/z* 329, for 9-hydroxyoctadec-9-enoic acid it is *m/z* 241, and lastly, for 11-hydroxyoctadec-9-enoic acid it is *m/z* 343 (Fig. 3.6). This mixture of hydroxyoctadecenoic acids was observed in the majority of the animal bones investigated, although always in low abundances. Figure 3.5 also shows that a substantial quantity of cholest-5-en-3β-ol (cholesterol) and cholesterol fatty acyl esters are present in the bone. This was also the case for nine (20%) other bone samples studied. The mass spectrum is shown in Figure 3.7; *m/z* 368 corresponds to the $[M - RCO_2H]^+$ fragment ion and has previously been recorded to be more prominent in cholesteryl esters containing saturated fatty acids, whereas the $[M - RCO_2]^+$ is more abundant when the acyl moiety is an unsaturated fatty acid (Goad and Akihisa, 1997:183). Cholesteryl esters are found in the blood and have previously been detected in archaeological human bones (Evershed *et al.*, 1995b; Stott and Evershed, 1996). There is a trace of cholest-5-en-7-one-3β-ol (7-ketocholesterol) in only one sample (10384; cow); this is known to be an oxidative degradation product of

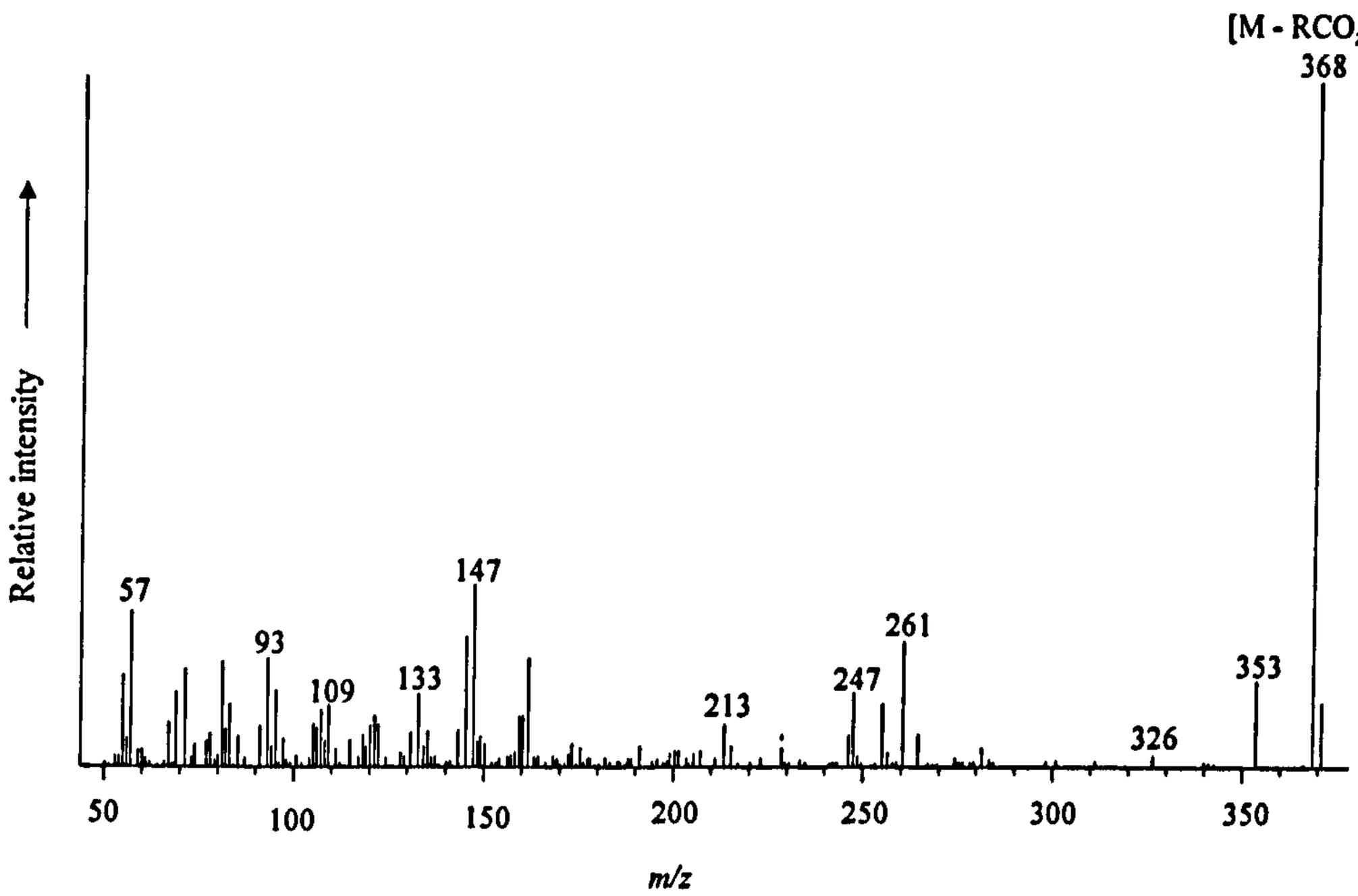

Figure 3.7 Mass spectrum of a cholesteryl ester. The $[M - RCO_2H]^+$ ion is indicative of the sterol moiety; however the $M^{+\cdot}$ is not observed in the electron ionisation (EI) mass spectra of fatty acyl esters of sterols bearing a Δ^5 double bond.

cholesterol and has previously been detected in fossilised whale bones (Stott *et al.*, 1997)

3.1.3 Fatty acids

Figure 3.8 shows the fatty acid distributions (as methyl esters) of cow (B27pelv) and sheep/goat (17035) bones. The fatty acids display an analogous distribution to that outlined above, and their percentage compositions are listed in Appendix 1 (Table A1.2) along with the other bones studied herein. The fatty acid composition of modern animals observed in the literature is given in Appendix 1 (Table A1.3) for comparison. The relative abundances of the major fatty acids extracted from the zooarchaeological reference materials are summarised in Table 3.1.

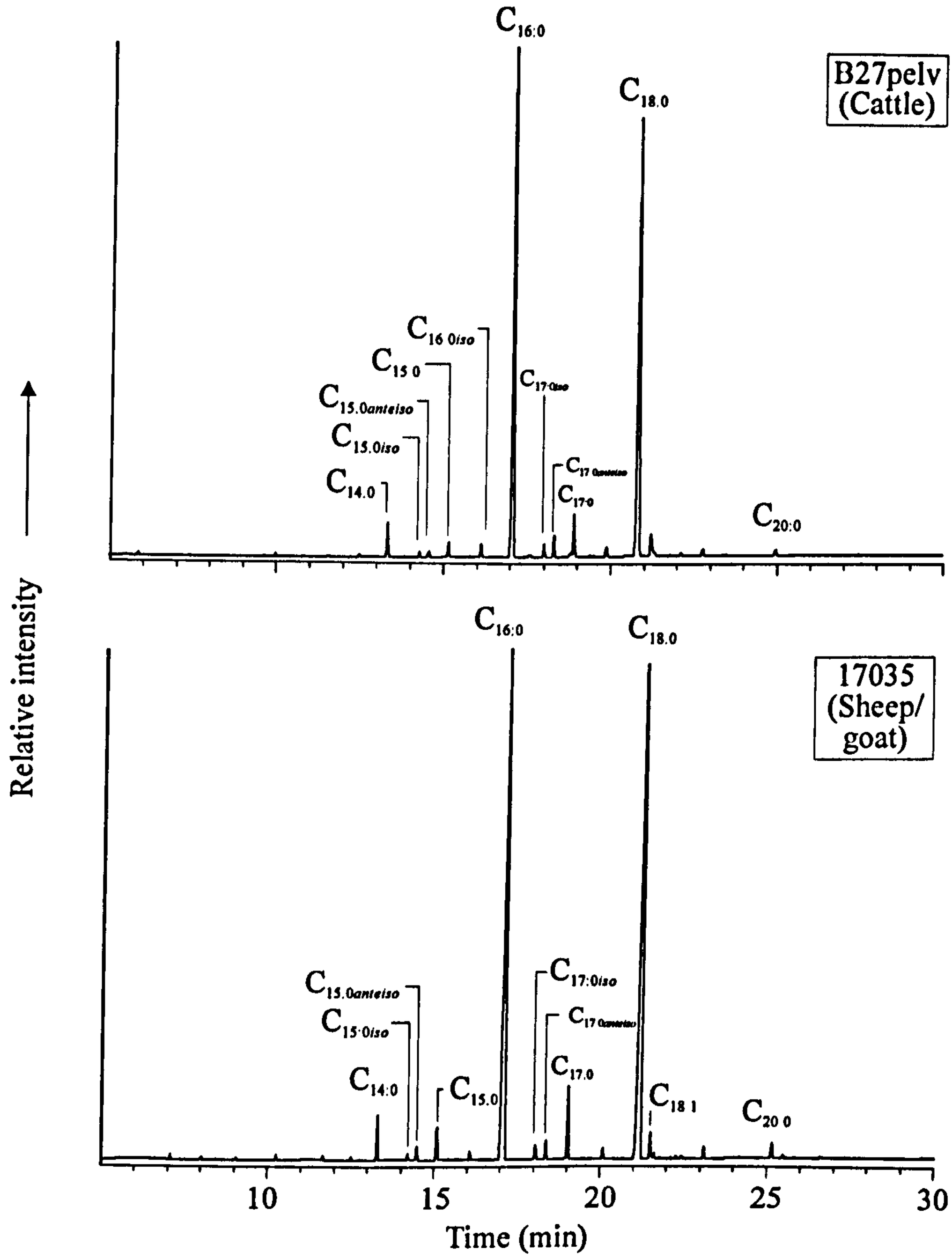

Figure 3.8 Partial gas chromatograms of FAMEs of bovine and ovi-caprine bones. $C_{x:y}$ refers to fatty acids of carbon chain length x and level of unsaturation y. $C_{15:0iso}$ and $C_{15:0anteiso}$ refer to the branched chain $C_{15:0}$ isomers. A CP wax 52 CB (polyethylene glycol) fused silica capillary column (50 m x 0.32 mm, 0.2 μm) was used.

Table 3.1 Composition and ratio[1] of the fatty acids extracted from the reference animal bones

	$C_{16:0}$ %		$C_{18:0}$ %		$C_{16:0}/C_{18:0}$	
	Mean	SD	Mean	SD	Mean	SD
Sheep/goat	56.4	11.7	36.6	13.0	1.54	0.82
Cow	50.8	12.3	47.1	12.7	1.08	0.56
Pig	60.4	1.1	28.1	1.6	2.15	0.08

[1] % of total fatty acids

In a recent study of modern and archaeological fats in pottery, it was found that ovine fats generally displayed a greater abundance of $C_{18:0}$ than $C_{16:0}$, and a high proportion of *trans* Δ^{11} $C_{18:1}$, whereas bovine fats exhibited a greater abundance of $C_{16:0}$ than $C_{18:0}$, less *trans* Δ^{11} $C_{18:1}$ and less C_{54} TAG when compared with the ovids (Dudd, 1999). A large proportion of the $C_{18:1}$ fatty acid, and all of the TAGs, have been degraded in the Qasr Ibrim animal bones, so several of the criteria established for distinguishing between the fats of different domesticates based on analyses of modern animals raised in Britain could not be utilised in the case of these archaeological specimens. Furthermore, the $C_{16:0}/C_{18:0}$ ratio is ≈1 for the bovine bones (mean = 1.08; s.d. = 0.56), and for the ovi-caprine it is >1 (mean = 1.54; s.d. = 0.82). The difference in means between the sheep/goat and cow for $C_{16:0}/C_{18:0}$ is 0.46, but this is not significant at the 95% confidence interval (CI); (T Test; $t = -1.822$; $df = 28.1$; $p = 0.079$).

β-oxidation (Kindl, 1987:209-210) is the primary mechanism for the degradation of saturated fatty acids in archaeological fat residues, and there is no reason to suppose that hexadecanoic acid will be more susceptible to β-oxidation than octadecanoic acid. Furthermore, although it is known that $C_{16:0}$ is more soluble in water than $C_{18:0}$ (Bell, 1973), the exceptionally arid conditions at Ibrim will have ensured that there is no leaching, during burial hence the $C_{16:0}/C_{18:0}$ relative abundance ratios should be preserved during burial and subsequent storage following excavation.

In living animals there are several reasons for varying fatty acid compositions, namely:

(i) The age of the animal affects the ratio of saturated : unsaturated fatty acids in both cattle and sheep. With increasing age, the proportion of $C_{18:0}$ decreases and $C_{18:1}$ increases (e.g. Leat, 1977; Pyle *et al.*, 1977; Enser, 1991:332-334).

(ii) The sex of the animal can affect the quantity of fatty deposits, and hence the relative abundances of the fatty acids (e.g. Pyle *et al.*, 1977; Enser, 1991:334).

(iii) The diet of the animal affects the fatty acid composition of its depot fats. Sheep diets low in roughage and high in carbohydrates inhibit the extent of hydrogenation that occurs in the rumen, therefore under these conditions, unsaturated acids would increase in the depot fats. Furthermore, it is known that the feeding of high concentrations of cereals to sheep increases the abundance of branched chain fatty acids (Enser, 1991:335). With cattle, an increase of branched chain acids occurs with forage diets rather than grain diets; the opposite occurs in sheep (Enser, 1991:345). Forage-fed cattle also tend to have more *trans* unsaturated monoenoic and saturated acids, and lower abundances of linoleic acid, when compared with cereal-fed cattle (Enser, 1991:349).

The mean values for the ratio of ($C_{15:0iso}$ + $C_{15:0anteiso}$) to $C_{16:0}$ fatty acids are shown in Figure 3.9; the calculated ratios for bovine adipose tissue for cows fed on forage and cereal diets are based on Enser (1991:345), and they fall well below the data points for the archaeological bones. Therefore, the elevated values shown in Figure 3.9, cannot be due to differences in the diet of the animals. It is known that C_{15}, C_{17} and their branched chain counterparts are bacterial makers (e.g. Goossens *et al.*, 1986), and hence it is likely that the higher $C_{15:0br}/C_{16:0}$ abundance ratios are due more to bacterial activity during burial rather than due to the animal's diet or metabolic processes (i.e. rumen bacterial contributions). Of interest are the high ratios in bones from the Napatan and Meroitic periods, and much lower values in the later periods.

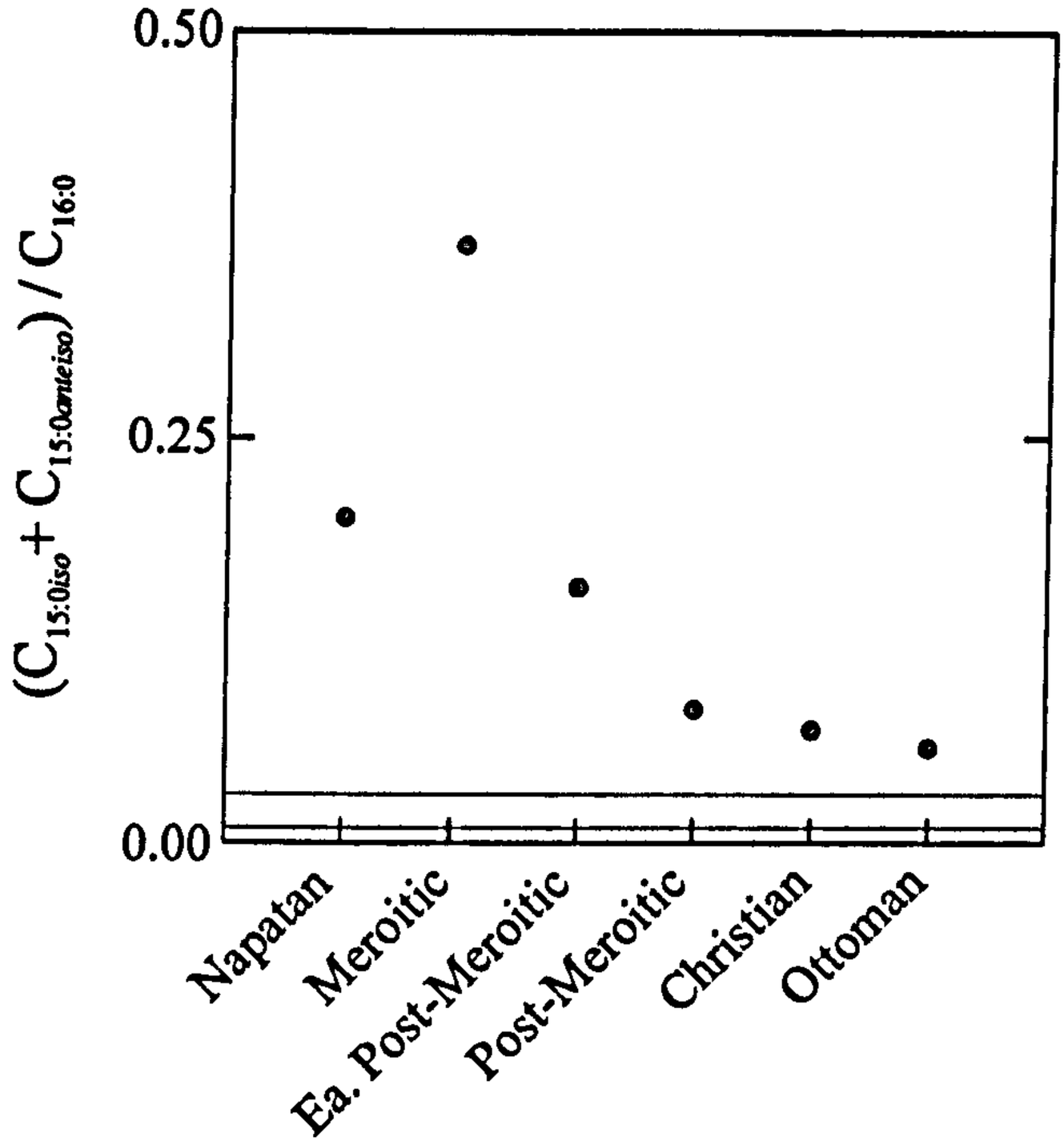

Figure 3.9 The mean ratios of $C_{15:0br}$ to $C_{16:0}$ over time. The reference lines at the bottom of the graph refer to the ratios in modern bovine adipose fats. The top line is the level for forage fed cattle (0.029). The bottom line is the level calculated (0.010) for cereal fed cattle (after Enser 1991:345).

At Qasr Ibrim, where there is an arid environment, it is possible that bacterial activity would be low, but constant over time. Hence the extent of microbial degradation might be expected to increase uniformly over time. This was seen in the Napatan bones, which exhibited high abundances of $C_{15:0br}$ (standardised to $C_{16:0}$) compared to the bones from later periods. However, the sharp increase in the relative abundance of $C_{15:0br}$ in the bones from the Meroitic period (and subsequent decrease in bones from later periods) may be due to the fact that all of the Meroitic bones analysed were relatively small (but well preserved) making them perhaps more susceptible to microbial degradation during burial.

3.1.4 Carbon stable isotopes ($\delta^{13}C$) values

The $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids were obtained from their methyl esters via GC-C-IRMS. The values for the sheep/goat and cattle are shown in Figure 3.10; these exhibit a wide range of values from the region of the graph indicative of pure C_4 $\delta^{13}C$ values (*c.* -15‰) to pure C_3 $\delta^{13}C$ values (*c.* -27‰). The mean $\delta^{13}C_{16:0}$ for ovi-caprids is -23.9‰ (s.d. 2.4‰) and the mean $\delta^{13}C_{18:0}$ is -24.5‰ (s.d. 2.1‰); whereas for the bovids the mean $\delta^{13}C_{16:0}$ is -18.7‰ (s.d. 3.9‰) and the mean $\delta^{13}C_{18:0}$ is -20.4‰ (s.d. 3.9‰). The $\delta^{13}C$ values are an indicator of the relative importance of C_3 and C_4 plants to the diet of the animals, and it can be seen that the sheep/goats were eating a predominately C_3 diet, 8 (40%) are likely to have consumed almost solely C_3 plant material, with the remainder either eating C_3 plants with less depleted $\delta^{13}C$ values, or possibly a mixed diet of C_3 and C_4 plants. One of the cattle samples

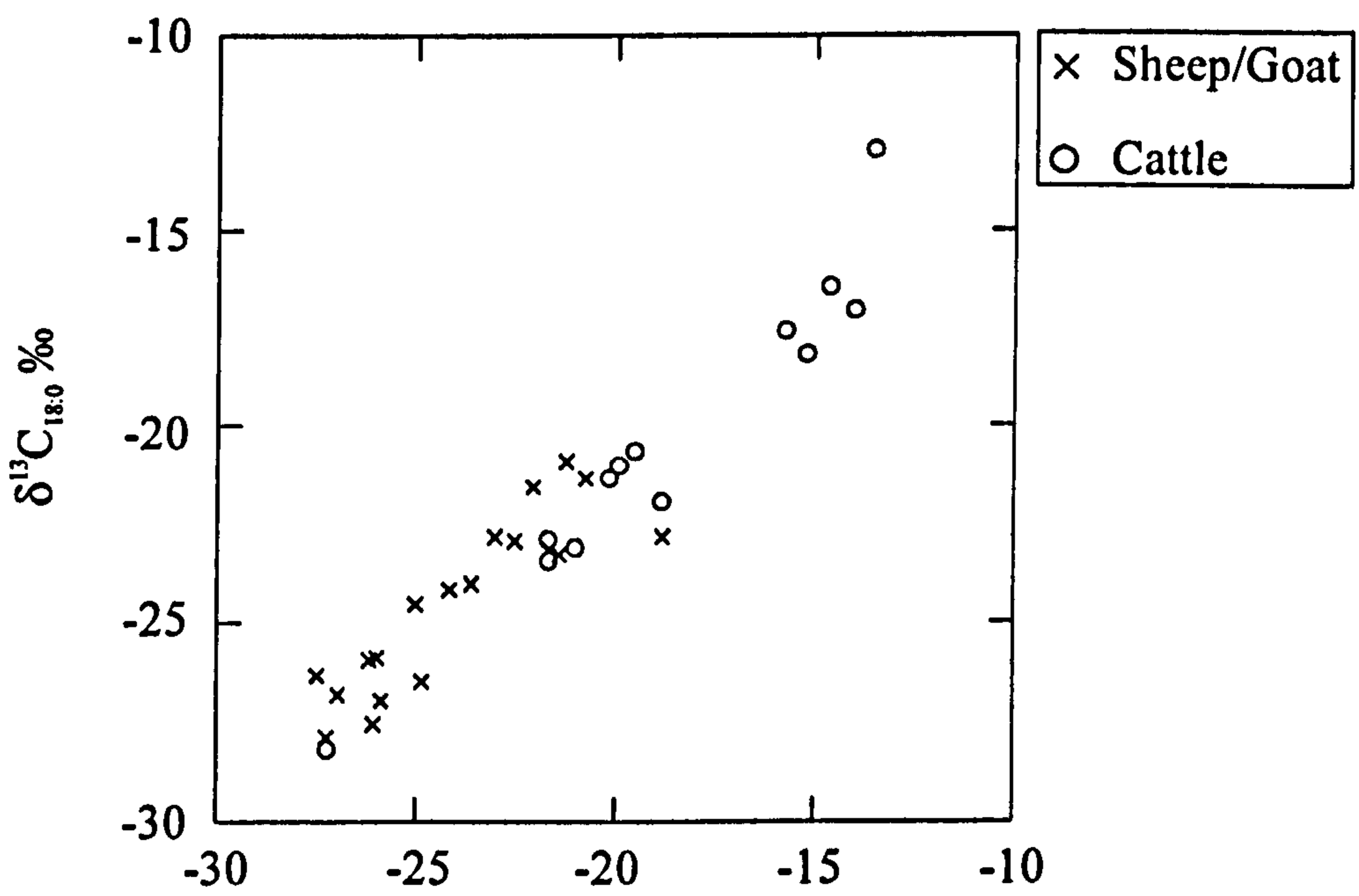

Figure 3.10 The $\delta^{13}C$ values of the major fatty acids from the ovi-caprine and bovine bones.

shows a distinctly pure C_3 signature for its fatty acids and 5 (38%) show values indicative of a purely C_4 based diet; the remainder (7; 54%) show a mixed C_3 and C_4 diet.

Figure 3.11 shows the variation in $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ fatty acids from the sheep/goat and cattle bone over time. The ovi-caprids all plot within the C_3 or predominantly C_3 area of the graph, with no apparent correlation between period and the stable isotope values. However, for the cattle, a temporal difference appears to exist. The fatty acids from the Napatan bones display $\delta^{13}C$ values that are indicative of a pure C_3 or predominantly C_3 origin, with $\delta^{13}C$ values in the range of -20‰ to -27‰ for the $C_{16:0}$ component and -25 to -28‰ for the $C_{18:0}$ component. In contrast, the bones from the Christian period are C_4/predominantly C_4 or mixed C_3 and C_4, with $\delta^{13}C$ values in the region of -13 to -20‰ for the $C_{16:0}$ fatty acid and -13 to -19‰ for the $C_{18:0}$ fatty acid. The fatty acids from the bones from the early Post-Meroitic and Ottoman periods also plot within the mixed C_3/C_4 range (-27 to -30‰ for both components). Although a larger sample suite would confirm this, even at this stage it appears that there was a shift towards the importance of C_4 plants in the diet of cattle in the Christian period, and there are also indications of a trend away from the use of a pure C_3 forages or fodders after the Napatan period.

The differences in the $\delta^{13}C$ values of the major fatty acids appear to reflect differing feeding strategies of the two species. Ovi-caprids are browsers, and therefore consume a wide variety of plants. They are often represented in urban archaeological sites through their bone and droppings, and caprids especially are well suited to eat the debris associated with human occupation. Bovids are grazers and, in an environment such as at Qasr Ibrim, would be fed on fodder such as sorghum, wheat and barley. In the Napatan period the most common cereal found during excavation was barley (a C_3 plant). In the Meroitic, domesticated sorghum (a C_4 plant) appears and is found in similar quantities as barley. In the Post-Meroitic, millet is found (but not in the same abundance as sorghum/barley). In the Christian and Ottoman periods, sorghum

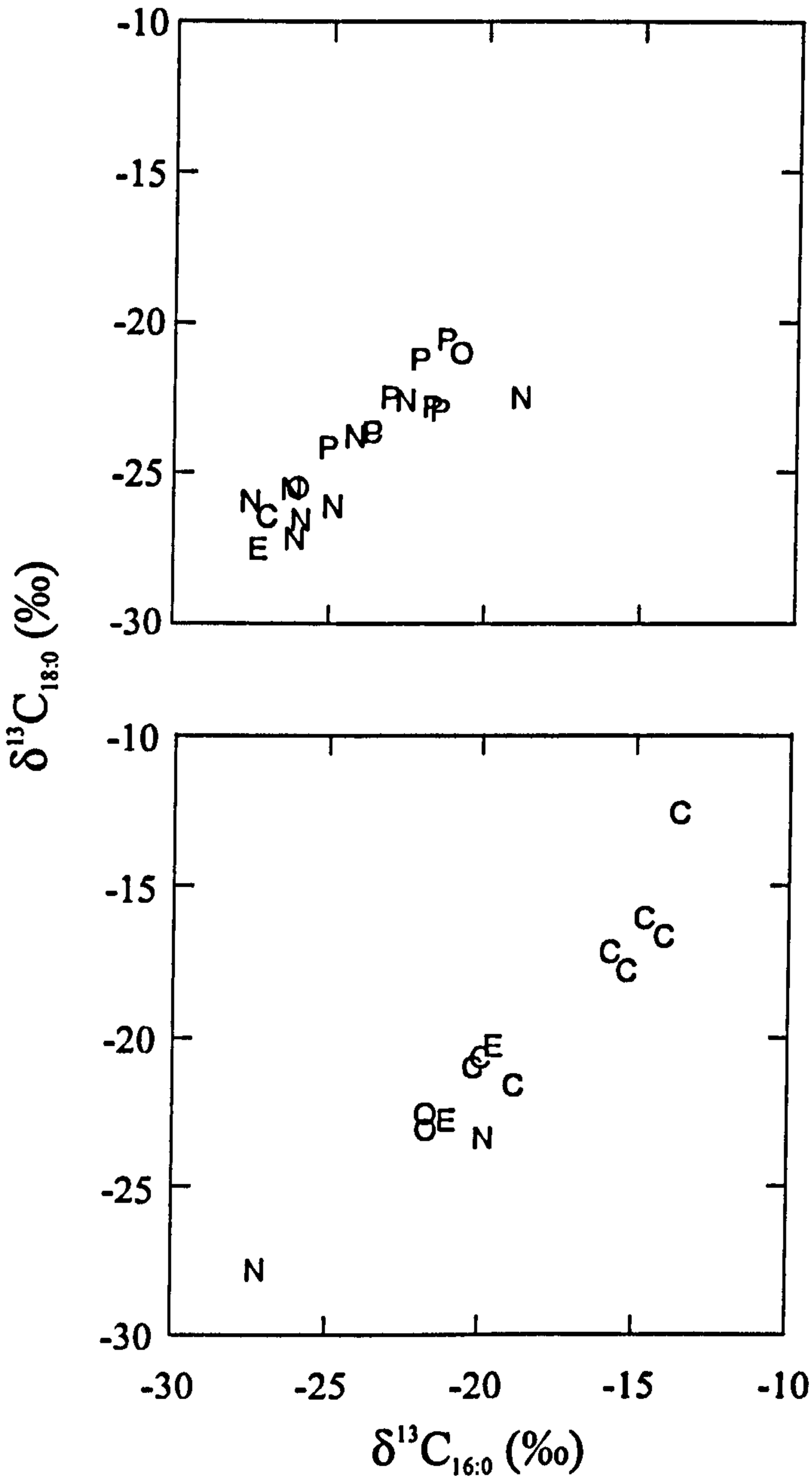

Figure 3.11 The $\delta^{13}C$ values for the major fatty acids of the ovi-caprine and bovine bones, by period. Sheep/goat (top) and cattle (bottom) show different $\delta^{13}C$ values over time, with more cattle displaying C_4 signatures in the later periods. N = Napatan (*c.* 1000-700BC); E = Early Post-Meroitic (*c.* AD300-400); P = Post-Meroitic (*c.* AD400-550); C = Christian (*c.* AD550-1500); O = Ottoman (*c.* AD1500-1800).

race bicolor and durra are found, and overall the C_4 crops predominate in these later periods (Rowley-Conwy, 1989; Rowley-Conwy, 1991). From the $\delta^{13}C$ values of the bone fatty acids, it appears that the introduction of new cultivars is represented in their diet. Although it is known that wheat and barley are winter crops, and sorghum a summer crop (C_4 crops are more efficient users of water and are physiologically more adept at surviving the hot, dry summer), from just the fatty acid analyses, it is not possible as yet to state anything about seasonal changes in the cattle's diet.

Nine of the TLEs of the bones contain cholesterol or cholesterol fatty acyl esters. The $\delta^{13}C$ values of the cholesterol (obtained by GC-C-IRMS of the TMS derivative following hydrolysis of the TLE and separation of the 'acid' and 'neutral' fractions) are plotted with the bone $C_{16:0}$ fatty acid in Figure 3.12. In all cases the $\delta^{13}C$ value of cholesterol is enriched relative to the $C_{16:0}$ fatty acid (the $C_{18:0}$ fatty acid shows the same trend).

Lipid biosynthesis involves the enzymatic decarboxylation of pyruvate (2-oxopropanoic acid) to acetyl-Coenzyme A, during which there is a kinetic isotope effect that results in the carboxyl carbon of the acetyl-CoA being more depleted with respect to the pyruvate precursor. However, the methyl carbon of the acetyl-CoA undergoes no fractionation with respect to the pyruvate precursor (e.g. De Niro and Epstein, 1977). During fatty acid biosynthesis, the ratio of methyl:carboxyl carbons in even chain acids is 1:1. However, the ratio of methyl:carboxyl carbons in cholesterol is 5:4; therefore cholesterol will be enriched relative to the fatty acids (assuming that they were biosynthesised from the pyruvate of the same $\delta^{13}C$ value). See Appendix 2, Figure A2.4 for a diagrammatic comparison of the carbon flow during the biosynthesis of $C_{16:0}$ and cholesterol.

The $\delta^{13}C$ values of the cholesterol are enriched relative to the $\delta^{13}C$ values of the fatty acids in the bones from Qasr Ibrim. Furthermore, the $\Delta^{13}C_{cholesterol\text{-}C_{16:0}}$ in the cattle bones ranges from 0.6 to 12.7‰ and does not show any linear

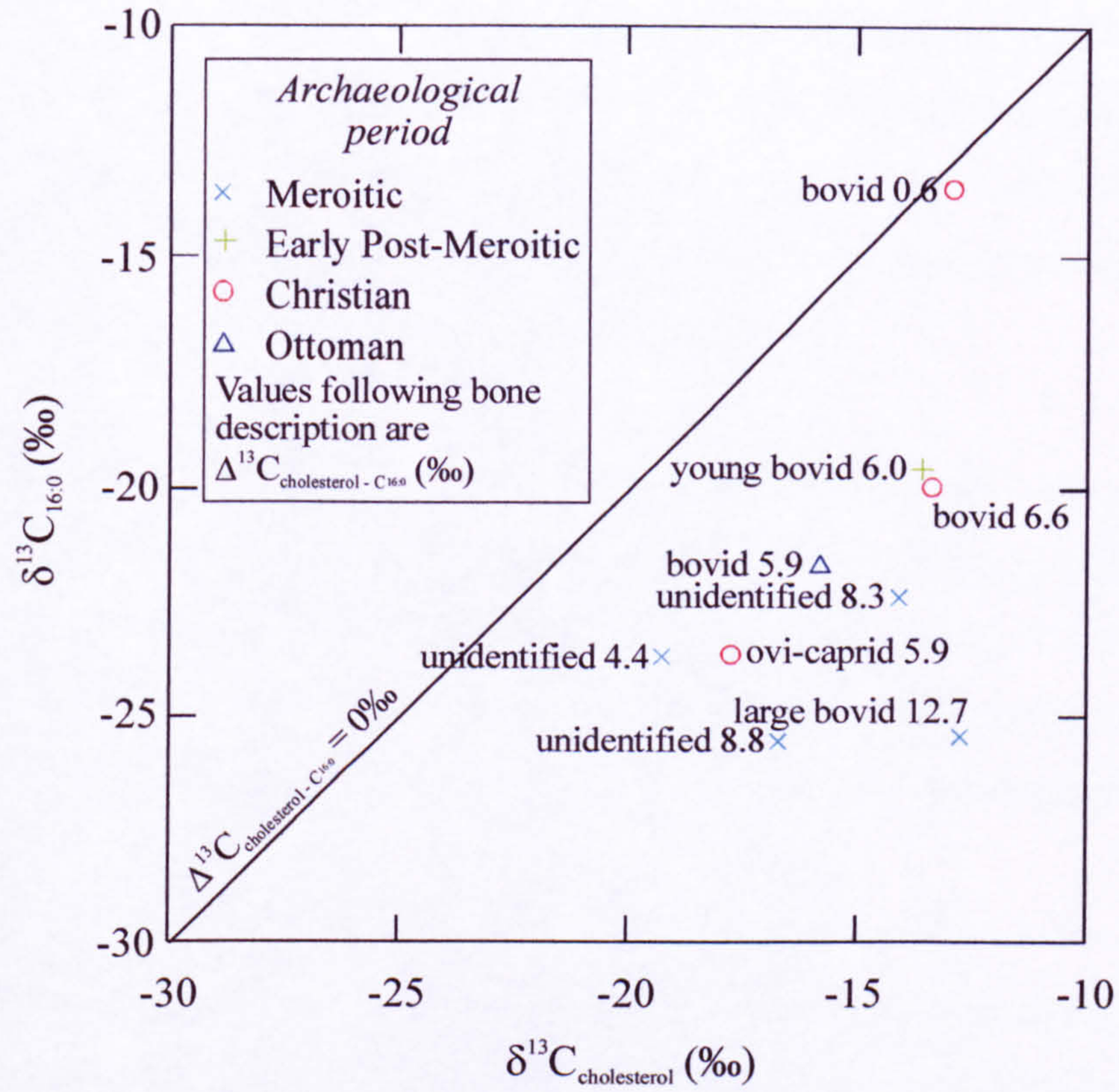

Figure 3.12 Comparison of cholesterol and fatty acid $\delta^{13}C$ values. The $\Delta^{13}C_{cholesterol\text{-}C16:0}$ spacing ranges from 0.6 to 12.7‰.

relationship between the $\delta^{13}C$ values of the two components. This may partially be explained through turnover rates and seasonality: From a study of the turnover rates of cholesterol and fatty acids in rats (Jim and Evershed, unpublished data), it was found that when the diets were changed from a C_3 to C_4 (and *vice versa*) it took approximately 290 days for there to be a 99% turnover in the cholesterol, whereas for the $C_{16:0}$ fatty acid it took only approximately 200 days, i.e. in rats, the fatty acid has a faster turnover rate than cholesterol. The exact turnover rate for these lipids in bovids would be expected to be different to those observed in the rats, however cholesterol would still be expected to have a longer turnover time (Jim *pers. comm.*).

On this basis, fatty acids may be seen as indicators of shorter-term fluctuations in diet and may reflect the whole diet. Whereas the $\delta^{13}C$ values of the fatty acids vary from typical C_3 to C_4 values (Fig. 3.12), the majority of the $\delta^{13}C$

values of the cholesterol for the bones are within the C_4 range. This could be due to the fact that C_4 crops are traditionally grown in the summer months and C_3 crops in the winter months, so that if the cattle were slaughtered during the early winter, a higher proportion of their fatty acids would have been turned over (from C_4 to C_3), compared to their cholesterol.

Since cholesterol is only present in nine of the animal bones, it is not possible to draw any firm conclusions regarding any differences that may exist between cattle and sheep/goats. Similarly, the feeding of C_4 plants to cattle in the summer and C_3 plants in the winter must remain speculative until controlled feeding experiments previously undertaken using rats have been performed on cattle.

The $\Delta^{13}C$ (defined as $\delta^{13}C_{18:0}$-$\delta^{13}C_{16:0}$) value is useful in the separation of cattle and sheep/goat fatty acids (discussed below). Since no pottery vessels were sampled from the Christian period, it was necessary to ensure that there is not a difference in the $\Delta^{13}C$ values for fatty acids extracted from the cattle bones from the Christian period compared to those from the cattle bones that were excavated from the other archaeological periods (Fig. 3.13).

No difference in the means is found with the $C_{16:0}/C_{18:0}$ ratio (T test; $t = 1.711$; $df = 12$; $p = 0.113$) nor with the $\Delta^{13}C$ values (T test; $t = -0.603$; $df = 12$; $p = 0.559$); therefore, the two sets of data were combined and used in subsequent analyses. A statistical difference is observed between the $\Delta^{13}C$ values of sheep/goat and cattle (T test; $t = 2.797$; $df = 31$; $p = 0.009$), thus providing the basis for differentiating the origins of fatty acids extracted from the pottery vessels that exhibit $C_{16:0}/C_{18:0}$ abundance ratios that are indicative of animal fats (see below Section 3.3).

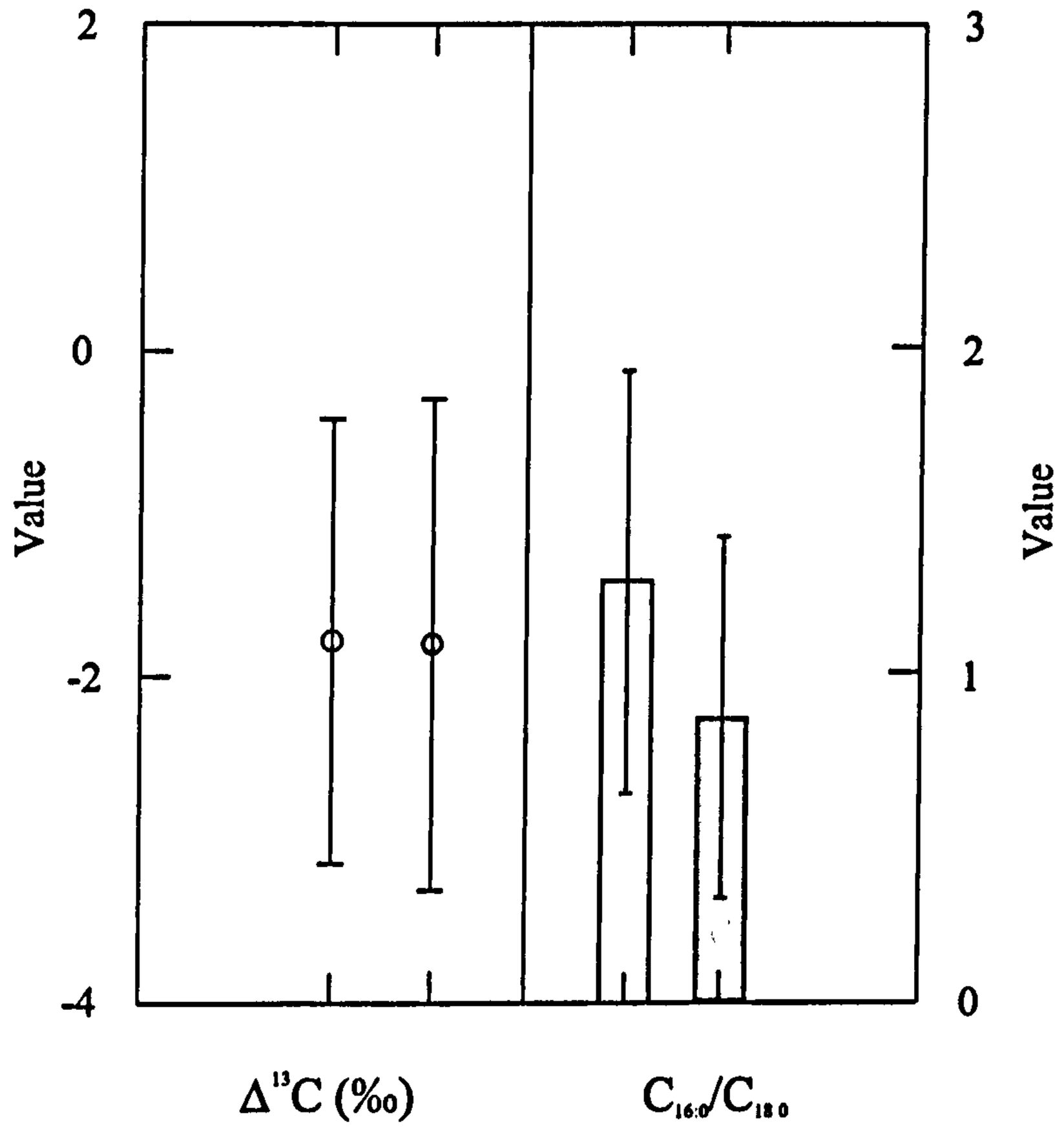

Figure 3.13 Graph of the $C_{16:0}/C_{18:0}$ and $\Delta^{13}C$ values for the cattle bones. All the cattle bones are included in the unshaded bar and circle, whilst the shaded bar and circle represent the values for the samples that did not give a 'pure' C_4 signal. One standard deviation error bars are included. No statistical difference was observed between the two.

3.1.5 The porcine bones

The pig bones gave an average $C_{16:0}/C_{18:0}$ ratio of 2.15 (s.d. 0.08) and $\Delta^{13}C$ value of -1.12‰ (s.d. 0.70‰), with the $\delta^{13}C_{16:0}$ being –23.7‰ (s.d. 1.3‰) and the $\delta^{13}C_{18:0}$ –24.9‰ (s.d. 1.3‰). This is similar to values previously obtained for modern porcine fats (Dudd and Evershed, 1998:177), and is in keeping with their varied diet.

3.2 BOTANICAL REFERENCE MATERIALS

3.2.1 General

The botanical reference materials include 12 samples (of 7 species) previously studied by Bland (1999); a further 13 samples (of 5 species) were analysed by the author, and the results were combined for statistical analysis. As with the animal bones, the archaeobotanical samples were taken from contexts that span the whole period of occupation of the site, and collectively comprise the main components of the palaeobotanical record. A full list of the samples, their provenance, fatty acid compositions and $\delta^{13}C$ values is given in Appendix 1 (Tables A1.4 to A1.7).

3.2.2 Total lipid extracts

As would be expected from a plant source (Gunstone *et al.*, 1986), the total lipid extracts of the modern botanical specimens are dominated by TAGs, principally in the range of C_{48} to C_{54} (e.g. Fig. 3.14; top). Very low abundances of free fatty acids are present in the modern propagules, while the TLEs indicate that the modern and ancient specimens contain very low abundances of the plant sterols β-sitosterol (stigmast-5-en-3β-ol), stigmasterol [(24*R*)-stigmasta-5,22-dien-3β-ol], and campesterol [(24*R*-24-methylcholest-5-en-3β-ol]. Modern date and dom kernels exhibit very unusual TAG distributions (the HT-GC profile of the dom palm is shown in Fig. 3.14; bottom), with acyl carbon number ranging from C_{36} to C_{46}, with particularly high abundances of C_{40} to C_{46}. The modern date (Fig. 3.15) has significantly lower abundances of the C_{36} to C_{38} and more of the C_{42} to C_{46} homologues. This latter point is partially explained by the fact that the dom kernel has a slightly greater abundance of the $C_{12:0}$ fatty acid than the date, and probably also reflects that the majority of the $C_{12:0}$ fatty acid was esterified in the C_{36} and C_{38} TAGs in the dom, rather than in the higher molecular weight TAGs extracted from the date kernel.

HT-GC analysis of the ancient plant TLEs shows that the majority of the TAGs have been hydrolysed to DAGs, MAGs and principally their free fatty acids (e.g. Fig. 3.15). Mono- and diunsaturated fatty acids are present in the majority of the archaeobotanical materials, indicating protection of the interior of the seed from oxidation during burial. Preferential hydrolytic degradation of the shorter-chain acyl lipids can be seen in the archaeological date kernels, possibly due to the fact that the ester linkages of these moieties are more exposed to hydrolysis due to steric effects as has been observed in the degradation of milk fat residues (Dudd and Evershed, 1998).

3.2.3 Fatty acids

The partial GC profiles of the modern date and dom kernel analysed as their FAME derivatives are shown in Figure 3.16, and can be compared with ancient and modern hyacinth bean (Fig. 3.17) and archaeological cotton and fig (Fig. 3.18). There is a striking difference between the fatty acid composition of the palm fruit kernels and the other samples, as evidenced by the high abundances of $C_{12:0}$, $C_{14:0}$ and $C_{18:1}$ in the palm kernels, whereas $C_{16:0}$ predominates in the other seed/propagule samples. The distribution of fatty acids dominated by the $C_{12:0}$ and $C_{14:0}$ components is chemotaxonomically highly diagnostic since it is only found in the kernels of palm fruit.

Compared to the modern reference materials, lower abundances of diunsaturated fatty acids are present in the archaeological specimens (clearly seen in the hyacinth beans). Furthermore, whereas monounsaturated fatty acids were only detected in low abundances in the archaeological bone samples, monounsaturated fatty acids are present in significant abundances in the archaeobotanical samples (e.g. Figs. 3.15, 3.18). Not only have the TAGs been incompletely hydrolysed to their free fatty acids (which was the case with the animal bones), but the survival of significant proportions of monounsaturated fatty acids also points towards a further level of preservation afforded by the seeds, due to physical and/or chemical protection (e.g. possibly due to the presence of natural antioxidants in the seeds themselves).

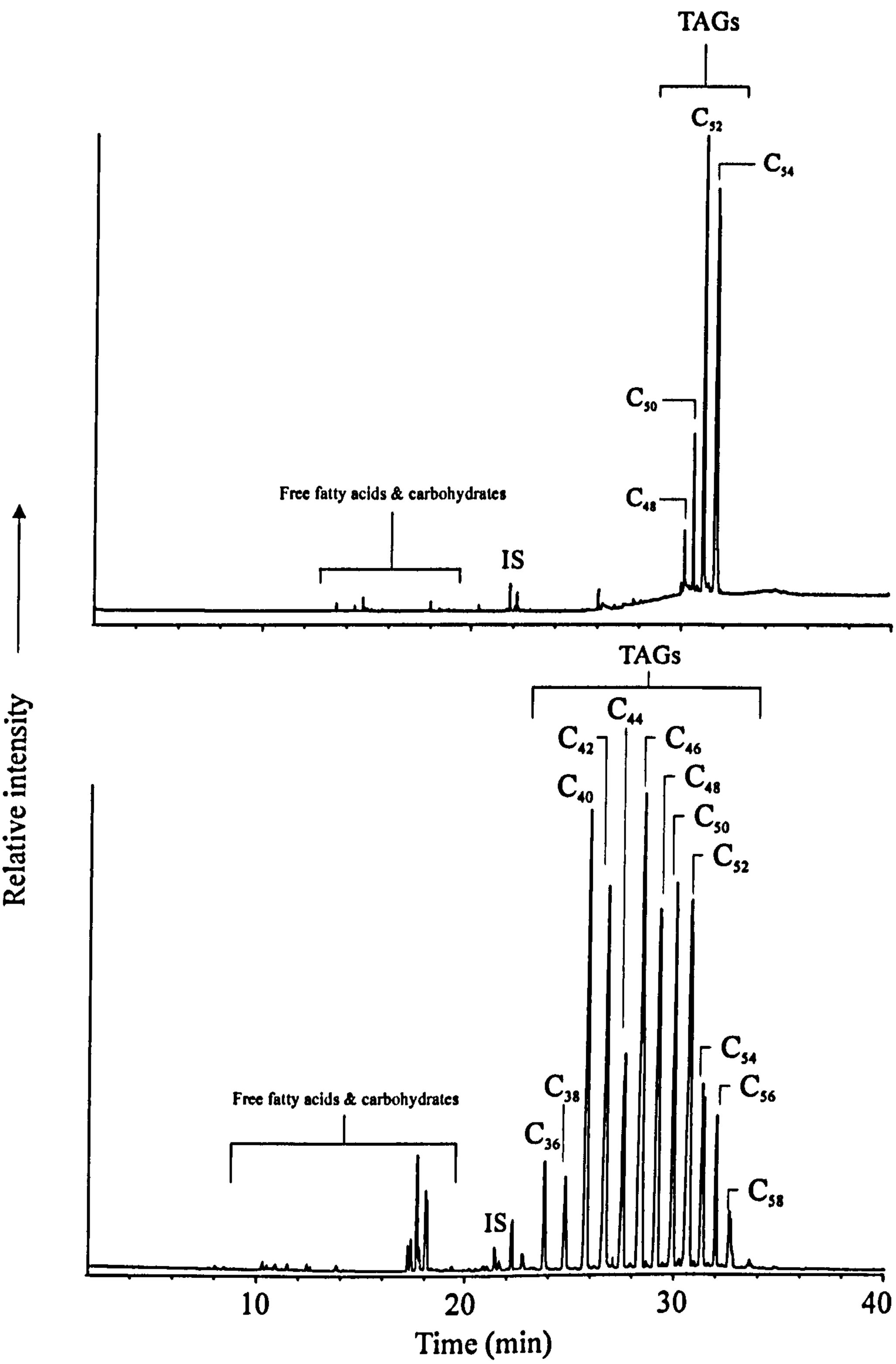

Figure 3.14 Partial gas chromatograms of the TLEs of modern hyacinth bean (top) and modern dom palm kernel (bottom). C_x refers to triacylglycerols (TAGs) with acyl carbon number x. IS denotes the internal standard (*n*-tetratriacontane). The greater range of free fatty acids in the dom kernel, with respect to the hyacinth bean, reflects the presence of lower molecular weight TAGs. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

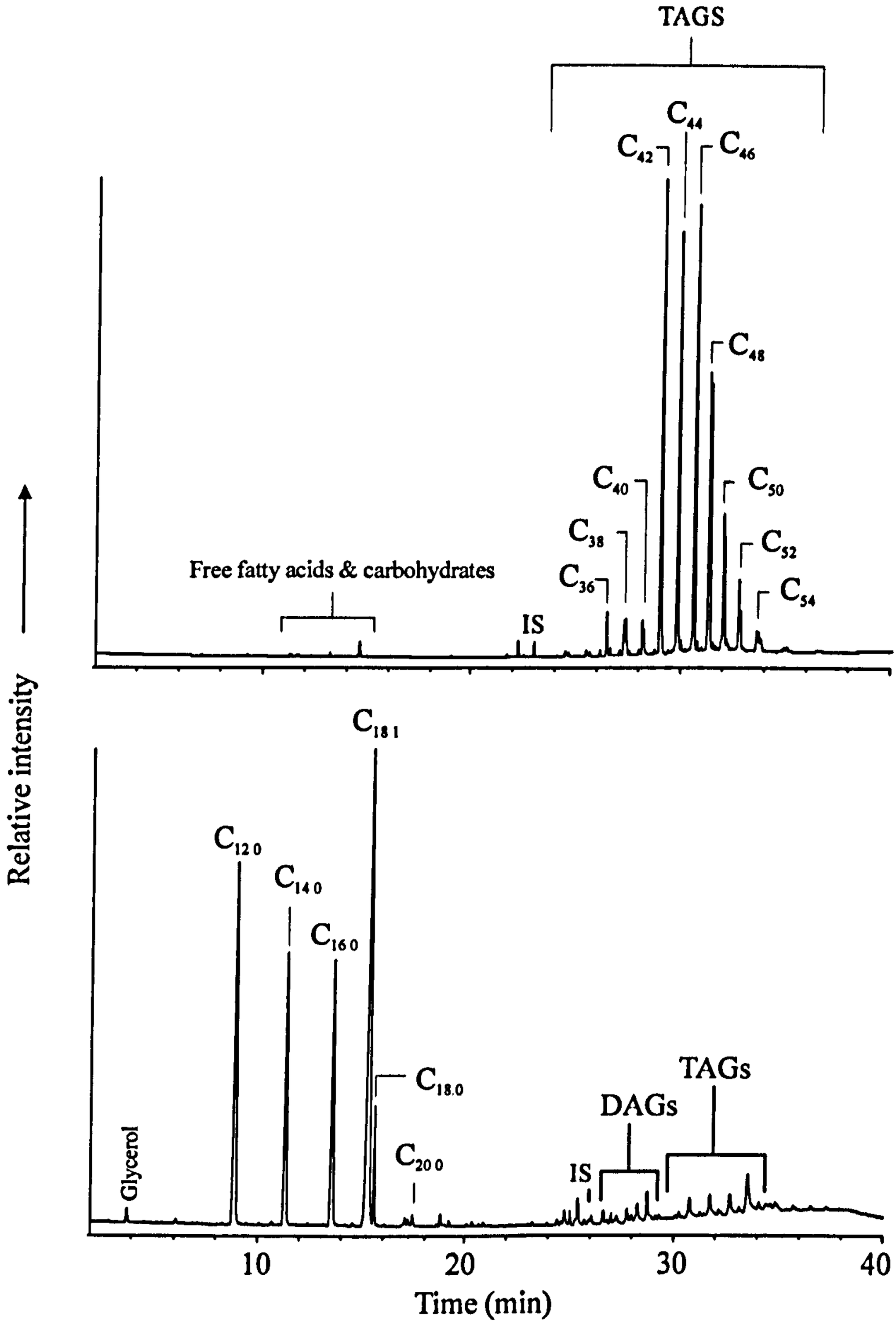

Figure 3.15 Partial gas chromatograms of the TLEs of modern and archaeological date kernels. The top chromatogram is of a modern date kernel, and the bottom is an archaeological date kernel. $C_{x:y}$ refers to carboxylic acids of carbon chain length x and level of unsaturation y. C_z refers to triacylglycerols (TAGs) with acyl carbon number z. DAGs refer to diacylglycerols, and IS is the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

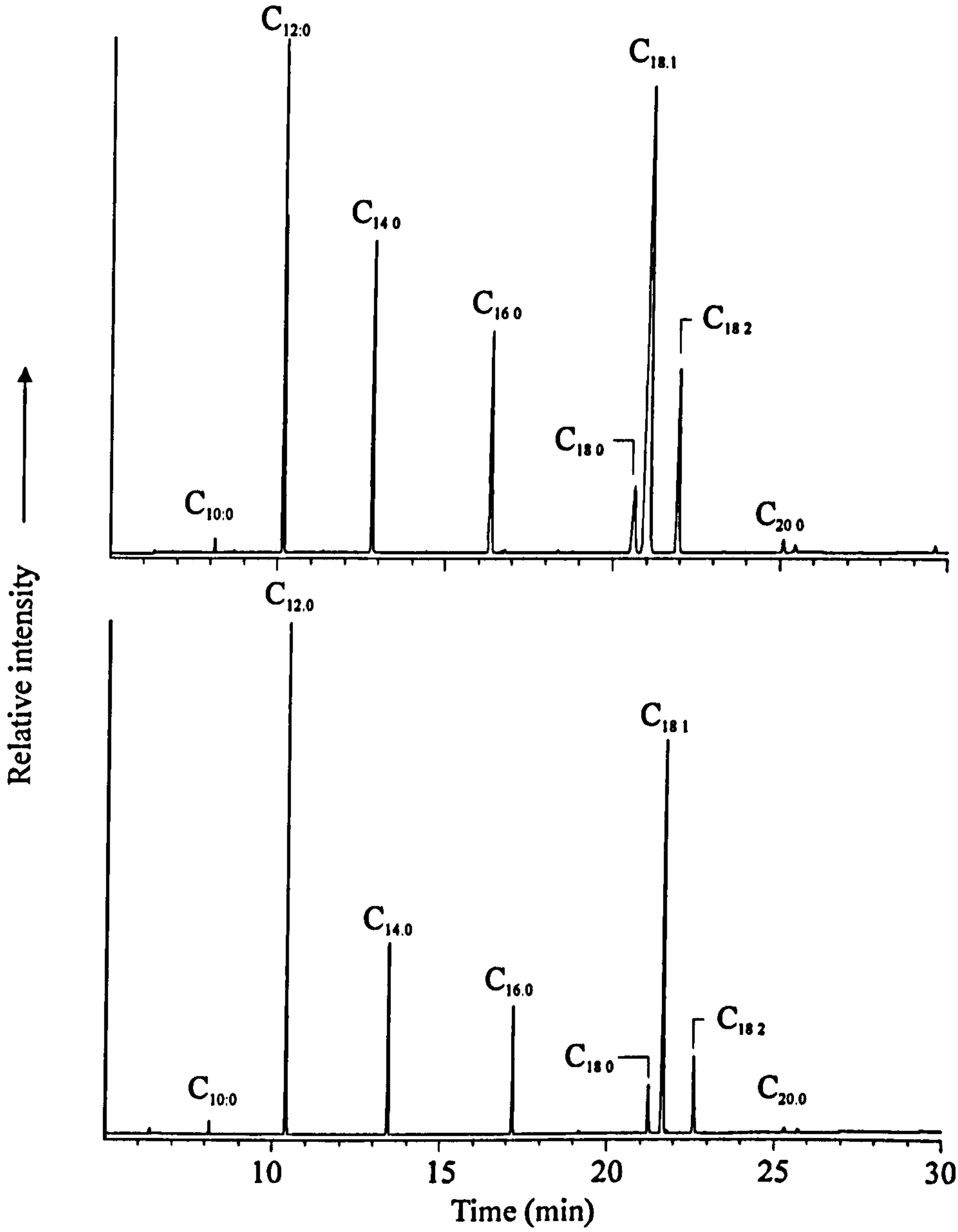

Figure 3.16 Partial gas chromatograms of the FAMEs of modern palm kernels. The top chromatogram shows the fatty acid distribution of a modern date kernel (*Phoenix dactylifera*), and the bottom that of a modern dom kernel (*Hyphaene thebaica*). $C_{x:y}$ refers to fatty acids of carbon chain length x and level of unsaturation y. A CP wax 52 CB (polyethylene glycol) fused silica capillary column (50 m x 0.32 mm, 0.2 μm) was used.

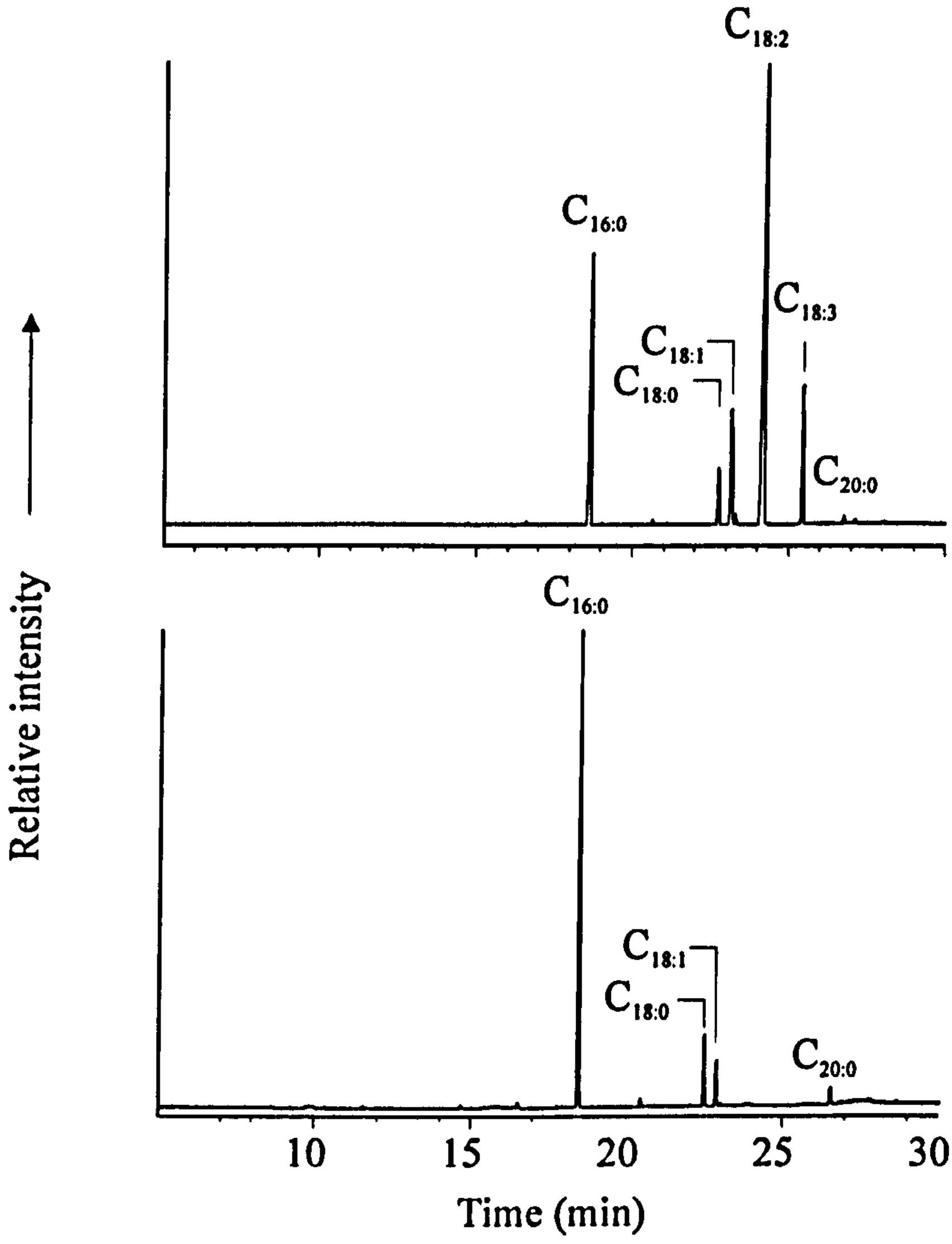

Figure 3.17 Partial gas chromatogram of the FAMEs of modern and archaeological hyacinth beans. The fatty acids of modern (top) and archaeological (bottom) *Lablab purpureus*. $C_{x:y}$ refers to fatty acids of carbon chain length x and level of unsaturation y. A CP wax 52 CB (polyethylene glycol) fused silica capillary column (50 m x 0.32 mm, 0.2 μm) was used.

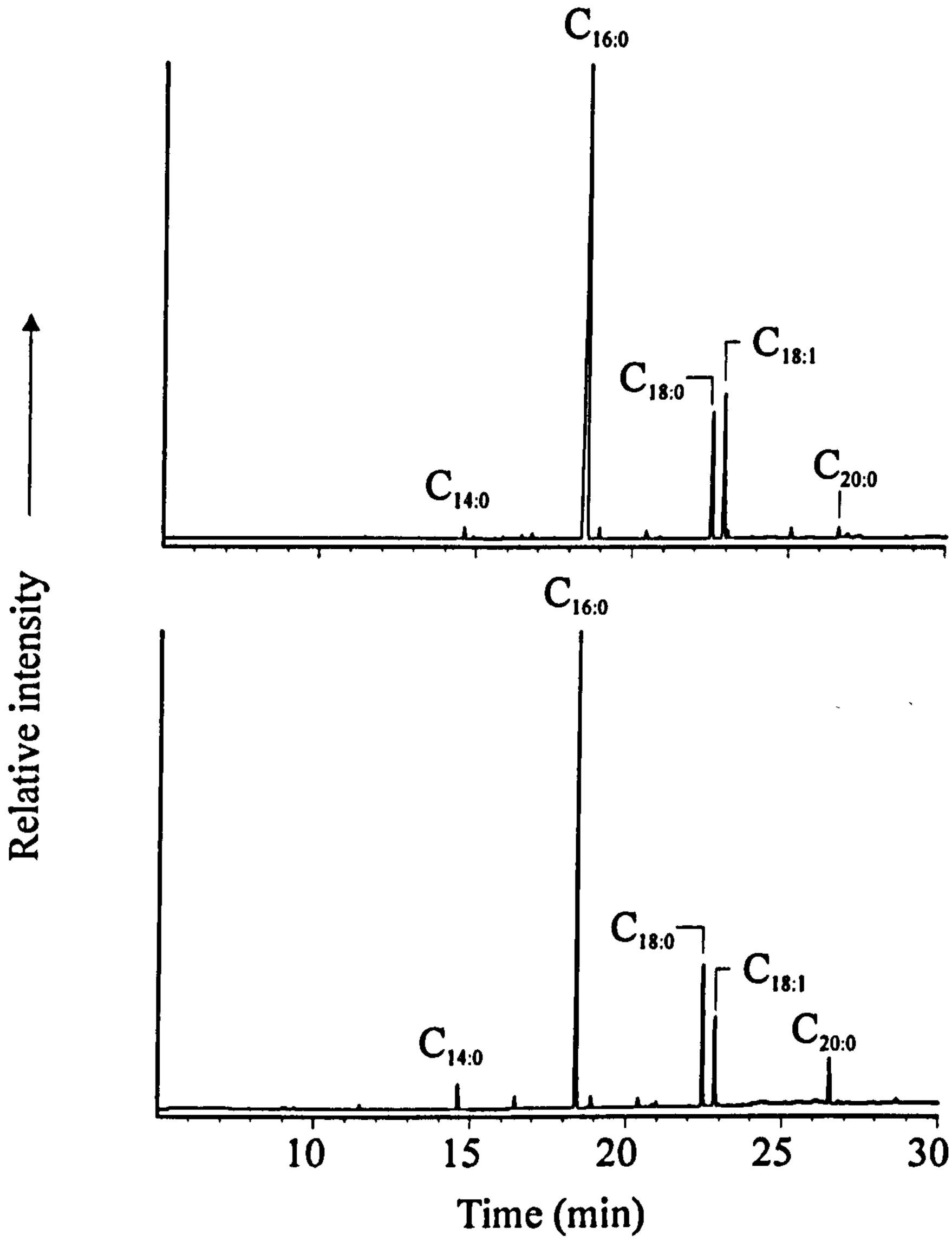

Figure 3.18 Partial gas chromatograms of the FAMEs of cotton and fig. The top chromatogram shows the fatty acid distribution of an archaeological cotton seed (*Gossypium herbacea*), and the bottom that of an archaeological fig (*Ficus sycomorus*). $C_{x:y}$ refers to fatty acids of carbon chain length x and level of unsaturation y. A CP wax 52 CB (polyethylene glycol) fused silica capillary column (50 m x 0.32 mm, 0.2 μm) was used.

From the GC profiles of the plant reference materials, it can be seen that the abundance of the $C_{16:0}$ fatty acid is always higher compared to the $C_{18:0}$ fatty acid. The mean value for the ratio of $C_{16:0}$ to $C_{18:0}$ was 4.3 (s.d. 1.7). There is a significant difference between the $C_{16:0}/C_{18:0}$ ratio of the sheep/goat and the plant remains and between cattle and the plant remains (ANOVA; *F ratio* = 20.379; $p < 0.0005$ in each case), thereby enabling the statistical differentiation between plant lipids and animal fats absorbed in pottery vessels.

3.2.4 Carbon stable isotope ($\delta^{13}C$) values

The $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acids are given in Appendix 1 (Tables A1.6-A1.7). Castor was the only CAM (*Crassulacean* Acid Metabolism) plant analysed, and the $\delta^{13}C$ values are no different than those of C_3 plants, i.e. -32.9 to -33.7‰ for the modern varieties. The C_3 cultivars have mean $\delta^{13}C_{16:0}$ values of -30.4‰ (s.d. 2.4‰) and mean $\delta^{13}C_{18:0}$ values of -30.0‰ (s.d. 2.8‰). These values lie within the range expected for C_3 plants, whereas the C_4 plants have less depleted $\delta^{13}C$ values where the mean $\delta^{13}C_{16:0}$ value is -17.2‰ (s.d. 0.8), and the mean $\delta^{13}C_{18:0}$ value is -17.8‰ (s.d. 0.2). However, the distributions of the $\Delta^{13}C$ values for the plants known to possess the two different photosynthetic pathways are not significantly different (Kolmogorov-Smirnov two sample test; $p = 0.287$) and when combined have a mean $\Delta^{13}C$ value of 0.2‰ and s.d. 0.5‰.

3.3 Statistical Differentiation of the Environmental Reference Materials

Figure 3.19 shows the $\delta^{13}C$ values of the hexadecanoic ($C_{16:0}$) and octadecanoic ($C_{18:0}$) acids for the botanical and faunal remains. The C_4 plants and animals eating C_4 plants plot together, as do the C_3 plants and the animals consuming predominantly C_3 diets. Hence, by plotting the values for the fatty acids extracted from the pottery vessels, it would be possible to determine whether or not they originate from C_3 or C_4 plants/animals that had consumed C_3 or C_4 plants.

The triangular plot of the relative abundances of $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$ fatty acids is particularly useful for displaying the number of sherds containing palm kernel lipid residues, due to the high abundances of the $C_{12:0}$ fatty acid. The plots for the botanical and faunal remains, along with the mean values for the groups, are given in Figure 3.20.

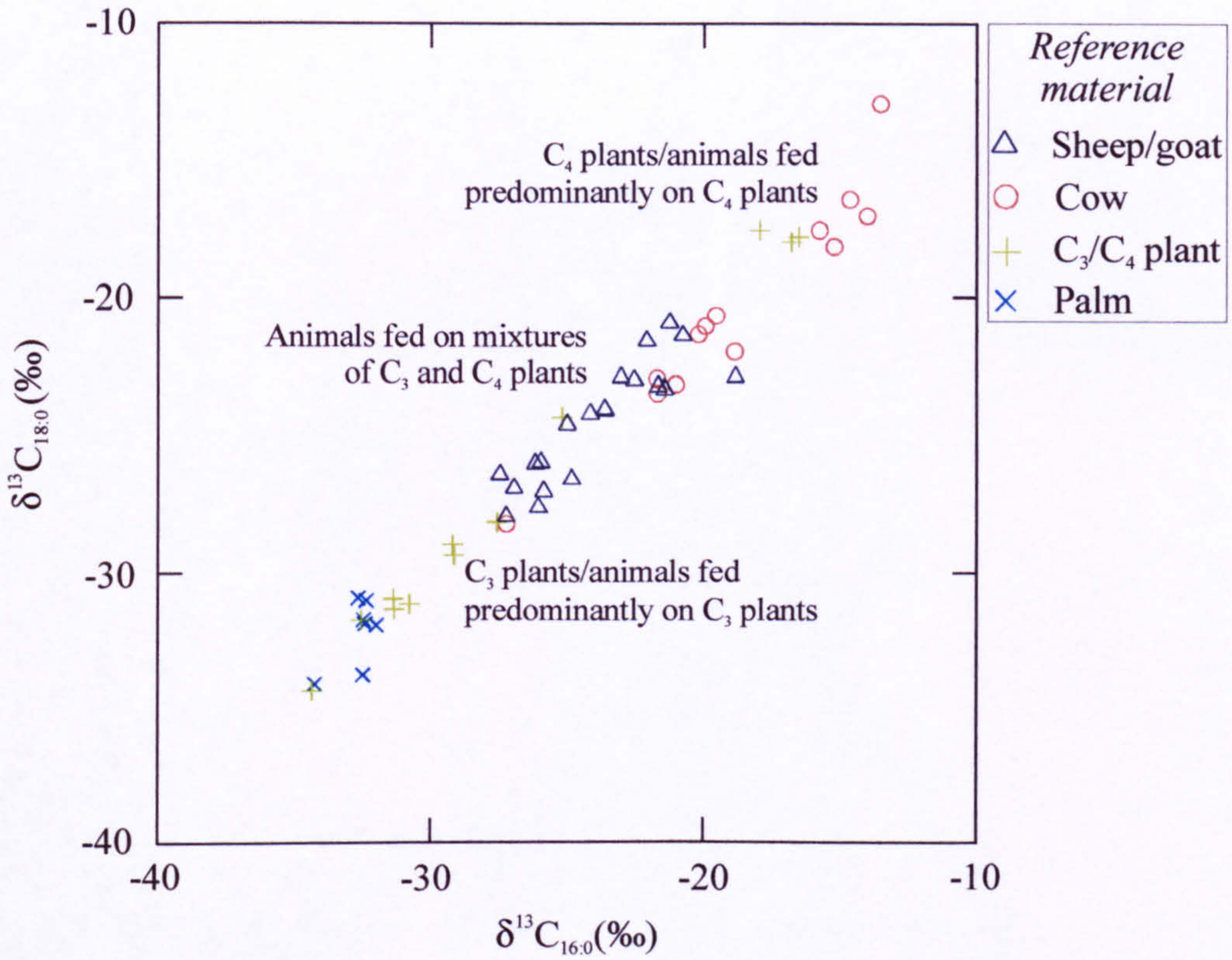

Figure 3.19 Plot of the $\delta^{13}C$ values of all of the environmental reference materials.

The plant material (excluding the palm kernels) and the animal bones plot close together along the *y*-axis, indicating the relatively low abundances of $C_{12:0}$ that they contain. However, the palm kernels plot to the left of the graph, due to the high abundances of $C_{12:0}$.

Furthermore, by plotting $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) against the ratio of the abundances of $C_{16:0}/C_{18:0}$ (Fig. 3.21), general trends can be observed. As has already been discussed, the plant reference materials have higher abundances of $C_{16:0}$ than $C_{18:0}$, compared to the animal bones, and there is a significant difference between the mean values for cattle, sheep/goat and plant (ANOVA; $df = 42$; $F\ ratio = 20.379$; $p < 0.0005$). Furthermore, the difference between the $\Delta^{13}C$ values for cattle & plant and sheep/goat & cattle are also significant (ANOVA; $df = 42$; $F\ ratio = 11.377$; $p < 0.0005$).

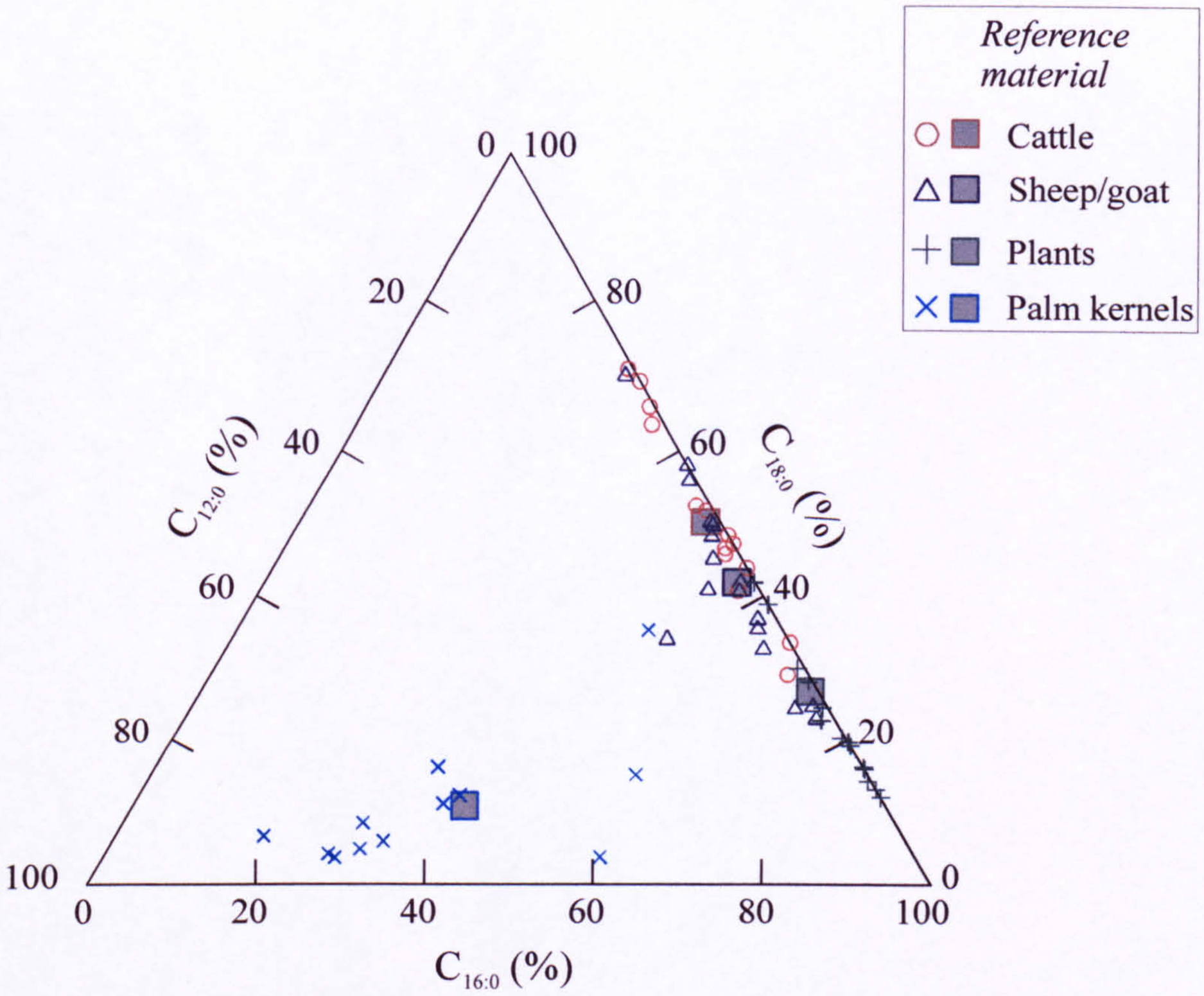

Figure 3.20 Triangular plot of the reference materials. The values for $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$ have been normalised. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

The insert in Figure 3.21 is the separate plot for sheep/goat, over which a modal smoother has been applied. Different types of smoothers can be used to fit a line through a set of points, and a modal smoother calculates the running mode for sub-groups of data points, which are displayed visually as a line. Modal smoothers are particular useful for highlighting the presence of sub-populations (Scott, 1992), and here there appears to be an approximate linear relationship

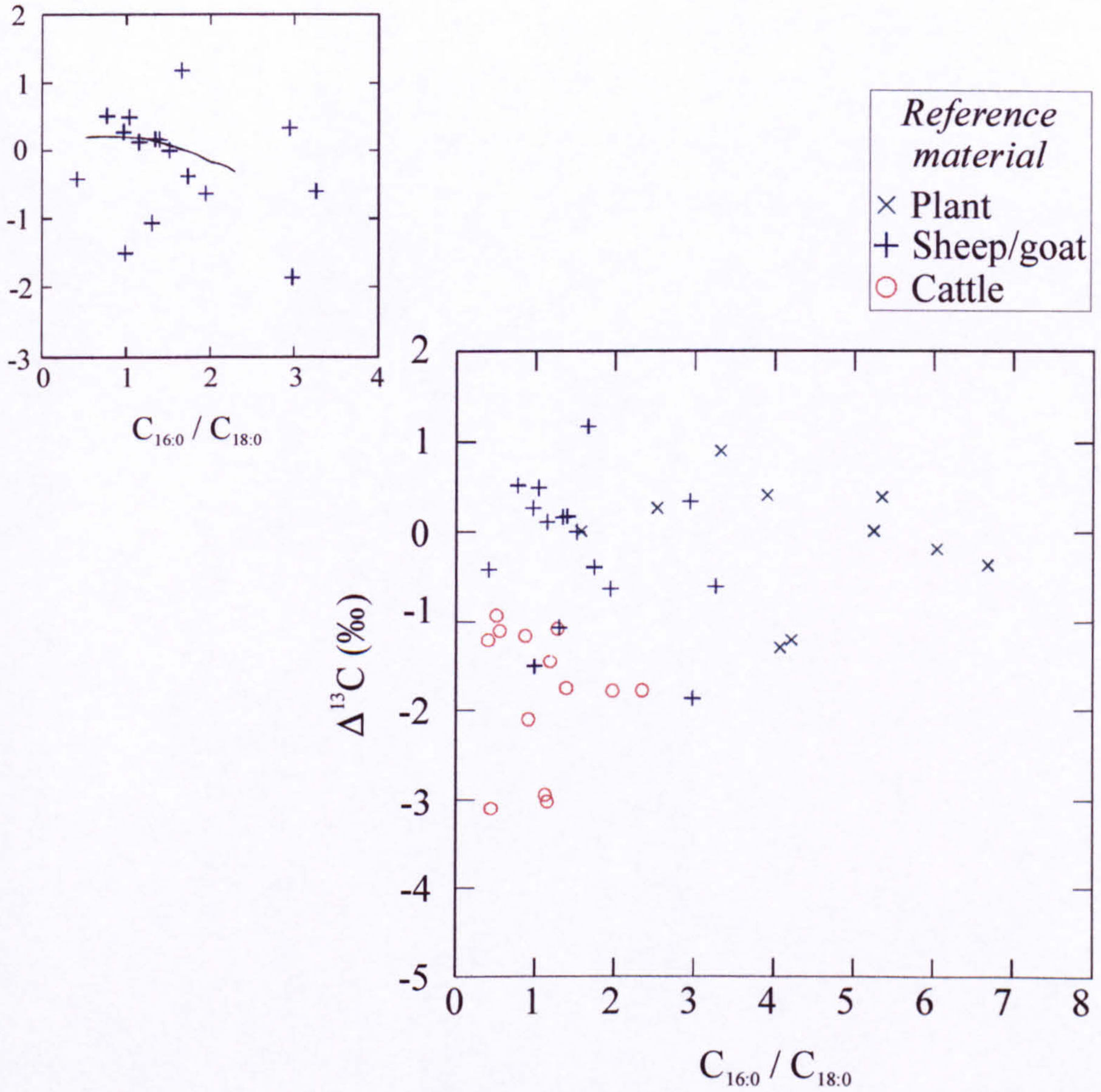

Figure 3.21 Plot of $\Delta^{13}C$ against $C_{16:0}/C_{18:0}$ for all the reference materials. The smaller graph in the inset includes only the sheep/goat bones, with a modal smoother drawn, indicating a possible trend.

between $C_{16:0}/C_{18:0}$ and $\Delta^{13}C$ for some of the ovi-caprine bones (indicated by the modal smoother extending through a portion of the plot in Fig. 3.21). However, this relationship cannot be detected statistically through regression analysis, presumably partly due to the fact that some of the data have high leverage over the analysis. With a larger sample population it might be possible to ascertain whether or not there is an underlying link between the two variables for sheep/goat.

Figure 3.22 shows the three groups with p=0.683 confidence ellipses drawn around them. The shaded areas within the ellipses have been designated the names 'cattle', 'sheep/goat', 'mixed animal', 'mixed plant/animal' and 'plant'. This nomenclature is designed to conveniently describe the particular areas of the bivariate ellipses. For instance, samples plotting within the 'mixed animal' field could of course statistically be cattle, sheep/goat or cattle *and* sheep/goat.

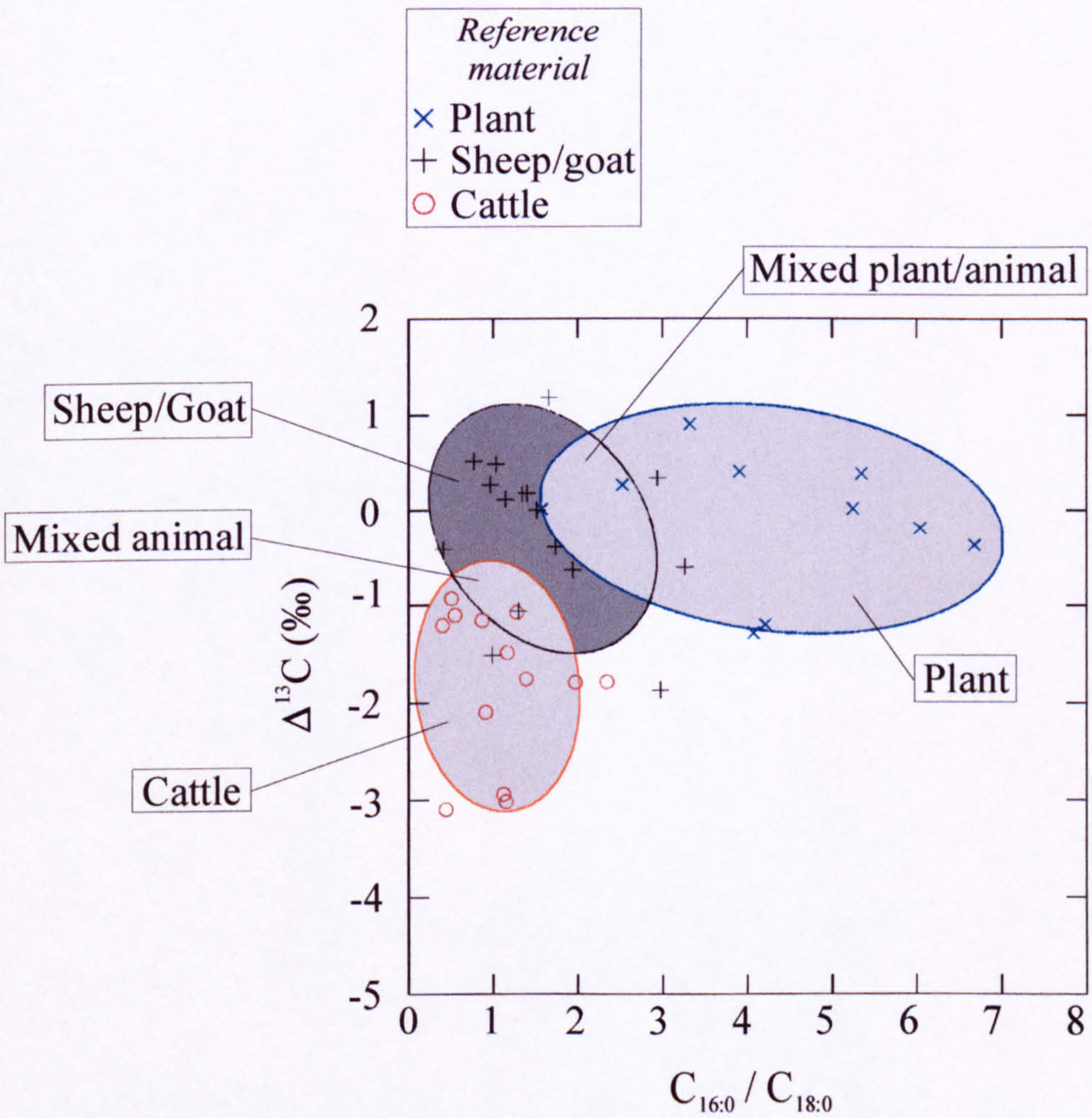

Figure 3.22 Plot of the $\Delta^{13}C$ values against the $C_{16:0}/C_{18:0}$ ratios with group designations.

A complication would arise if a vessel had been used to process both an animal that had fed on C_3 plants and a C_4 plant (or *vice versa*). Figure 3.23 shows the three confidence ellipses for the different reference materials, overlain with theoretical mixing curves. Due to the fact that plants and animals have differing $C_{16:0}/C_{18:0}$ abundance ratios and $\Delta^{13}C$ values, the mixing of the two commodities will result in characteristic curves. Mixing curve **a** would be obtained from mixing equal quantities of lipid from a C_3 plant (represented here by the mean value of all the C_3 reference materials) and fats derived from cattle

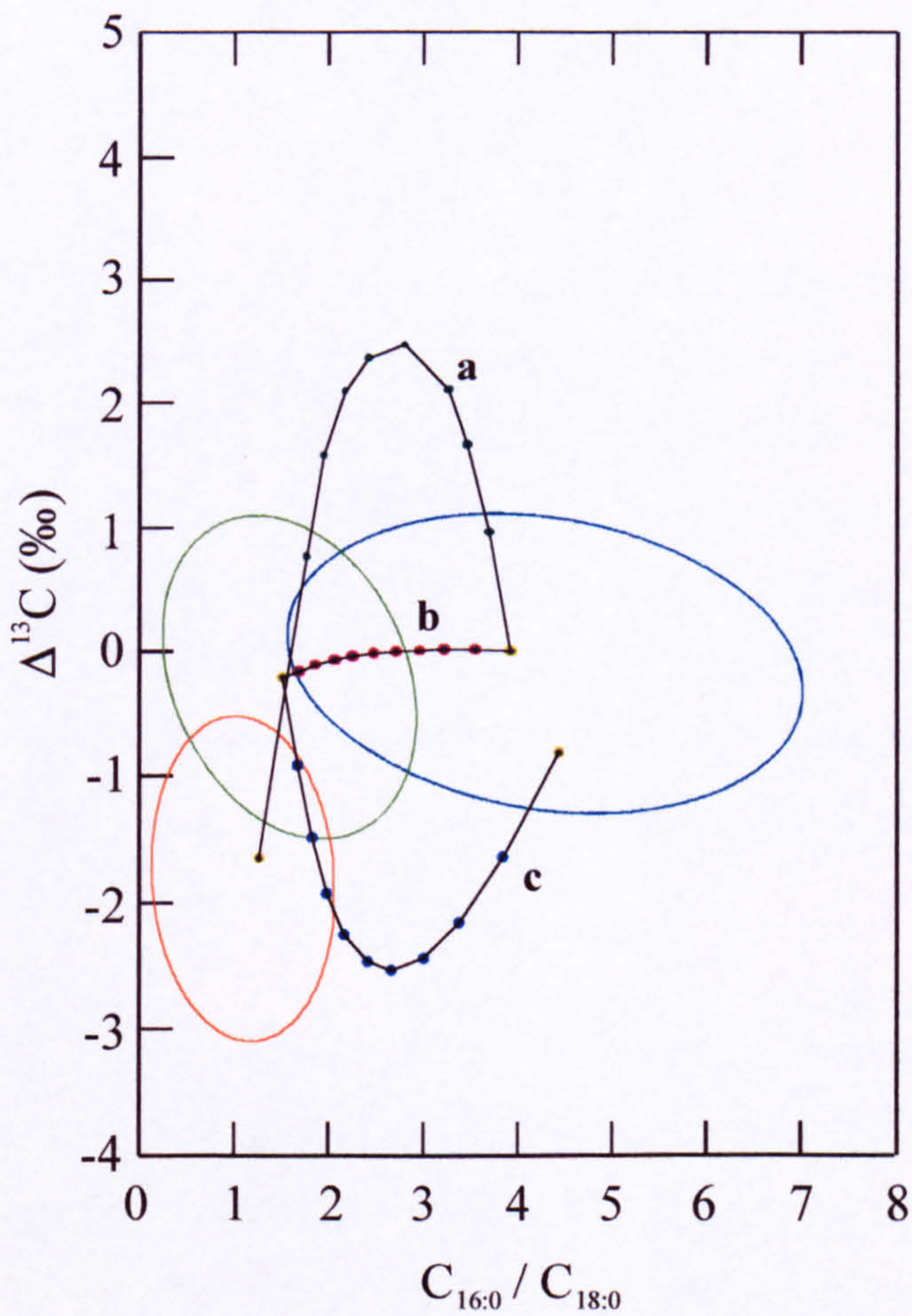

Figure 3.23 Theoretical mixing curves. Curve **a** is the mixing of C_4 cow fat and C_3 plant material. **b** is the mixing of C_3 sheep/goat and C_3 plant. **c** is the mixing of C_3 sheep/goat and C_4 plant material. For both fatty acids the following formula was used: $\delta^{13}C_{mix} = (\delta^{13}C_1 \times R_1/(R_1+R_2)) + (\delta^{13}C_2 \times R_2/(R_1+R_2))$, where $R_1 = C_1 \times P_1$ and $R_2 = C_2 \times P_2$. [$\delta^{13}C_{mix}$ = compound specific stable isotope value for the mixture; C_x = quantity of fatty acid in commodity x per gram of x; P_x = proportion of commodity x in mixture].

that had been reared on a C_4 diet (assigned the mean value of the bovid bones with fatty acids with a C_4 signature). Each point along the curve represents a 10% increase/decrease in each of the commodities, from 100% cow & 0% plant to 0% cow & 100% plant. The curve is more pronounced when compared with mixing curve **b**, which would result from the mixing of fatty acids from C_3 plants and sheep/goats (which have been shown to have a predominantly C_3 diet). Mixing curve **c** is the result of the mixing of mainly C_3 sheep/goat fats and the lipids from a C_4 cultigen (the mean values for sorghum). It can be seen that instead of having a maxima with a positive $\Delta^{13}C$ value, a minima with a negative $\Delta^{13}C$ value is obtained, this is due to the fact that plants have significantly higher abundances of the $C_{16:0}$ than $C_{18:0}$ fatty acid and therefore have less influence on the $\delta^{13}C_{18:0}$ values (and hence the $\Delta^{13}C$ values vary widely).

Although these curves are calculated from varying percentages of plant/animal lipids, in reality, it is very unlikely that equal proportions (by weight) of animal and plant products would have been processed in the vessels. It is possible that where the cooking of meat and vegetables has occurred, more meat products (by weight) would have been cooked in the vessel, compared with vegetables, pulses or beans. Hence in Figure 3.23, vessels that had been used in this latter manner would plot within the fields associated with the animal reference materials. Therefore, it can only be stated that 'predominantly' animal fats were detected in these particular sherds.

The mixing of C_3 and C_4 commodities can to a certain extent be detected via very positive or very negative $\Delta^{13}C$ values. However, if bovine fats from an animal whose diet had included predominantly C_4 plants were mixed with large quantities of C_3 plant lipids, then they would plot in the sheep/goat region of the graph. Similarly, the mixture of C_4 plants and the fat from a sheep/goat raised on a predominantly C_3 diet would plot, in part, within the 'cattle' ellipse, and investigation of the individual $\delta^{13}C$ values would not distinguish between residues of cow and mixed plant/sheep/goat origin. Therefore, the separation of the animal fats into ovi-caprine and bovine fats has to be undertaken with

caution, due to the possibility of mixing of fatty acids from different commodities during vessel use.

In view of the above, the results from the pottery vessels described in the following Chapter will be presented in two formats. Lipids shall be assigned as having the following sources:

(i) Predominantly animal, plant/animal, or predominantly plant, based on their $C_{16:0}/C_{18:0}$ abundance ratios.

(ii) Predominantly sheep/goat, predominantly cattle, mixed animal, plant, or predominantly mixed C_3 plant/animal (*taking account of the potential mixing of lipids from C_3/C_4 animals and plants*), based on the $C_{16:0}/C_{18:0}$ abundance ratios and the $\delta^{13}C$ values of the individual fatty acid components.

(iii) C_3 and C_4 plants, based on the $\delta^{13}C$ values of the fatty acids and *n*-alkanes.

(iv) 'predominantly sheep/goat' and 'predominantly cattle' fats from animals fed on C_3, C_4 and mixed diets, based on the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values.

3.4 SUMMARY

The arid environment at Qasr Ibrim has aided in the survival of lipids in animal bone and plant material over archaeological time periods. Not only did these samples yield high abundances of free fatty acids, but the majority also contained lipids that are generally not observed in such high abundances in European zooarchaeological/palaeobotanical materials (e.g. glycerol and α,ϖ-dicarboxylic acids).

- The zooarchaeological materials contain very large concentrations of preserved lipid, including fatty acids and cholesterol
- There is no significant difference between the bovine and ovi-caprine bone $C_{16:0}/C_{18:0}$ abundance ratios or their $\delta^{13}C$ values
- The $\delta^{13}C$ values potentially point to differences in feeding patterns between the two species, and for the bovids, this may also include a temporal change
- The consumption by the animals of C_3 and/or C_4 plants does not alter the $\Delta^{13}C$ value ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) significantly and therefore this latter criterion can be used in statistical analyses
- The palaeobotanical materials show a high abundance of more unsaturated components in their lipid distributions, indicating the increased level of preservation found in the seed compared with the archaeofaunal remains, although high abundances of 9,10-dihydroxyoctadecanoic acids, glycerol and hydroxyoctadecenoic acids are evident
- The $C_{16:0}/C_{18:0}$ abundance ratios for the plant material are statistically different from the faunal samples, as are the $\Delta^{13}C$ values of the bovine fats/plant remains and the ovi-caprine fats/plant remains
- When examining the plot of $\Delta^{13}C$ v $C_{16:0}/C_{18:0}$, interpretations of the mixing of C_3 and C_4 plants and animals must proceed with caution, although it is possible to differentiate between predominantly cattle and predominantly sheep/goat fats
- Kernels from the palm fruit display a unique lipid distribution enabling the detection of the processing of the fruit in the pottery from arid sites where the more water soluble fatty acids are not leached during burial

Chapter 4.

The Pottery Vessels: Results

4.1 GENERAL

4.1.1 The pottery samples

The pottery samples were selected primarily from structure 265 (Section 1.3), encompassing the Meroitic (*c.* AD 50-300), Early Post-Meroitic (*c.* AD 300-400) and Post-Meroitic (*c.* AD 400-550) phases at Qasr Ibrim. The sherds prefixed with 'ML' refer to the Hinterland survey, where samples were selected from recorded vessels from outside the fortress walls. These latter sherds dated to the early Meroitic and were contemporaneous with the Ptolemaic occupation of the Qasr Ibrim (Rose, 1996) and therefore represent a distinct ceramic assemblage at the site. The number of potsherds sampled and their provenance is given in Table 4.1. The sherd numbering system used herein encompasses the context number and the number of the sherd from that context. For example 9·24 is sherd 24 from context 18009, and similarly 110·42 is sherd 42 from context 10110.

Sherds from each context were compared visually for colour, fabric and size, such that, as far as possible, no two sherds originated from the same vessel. However, the hand-made wares exhibited such homogeneity that ensuring this was almost impossible. Furthermore, the possibility existed that sherds from a single vessel could quite conceivably be recovered from disparate archaeological contexts due to scattering and displacement during the initial phase of discard and post-depositional changes.

Table 4.1 Numbers of potsherds sampled from each period and context

Period[1]	Context	Room No.[2]	Number of sherds sampled	
Meroitic *Room 26/8* 18002 18031 18013 18014 18036	Hinterland Survey	n/a[3]	52	
	18002	26/8	1	
	18010	3	2	
	18013	26/8	23	
	18014	26/8	10	
	18023	2, 6	27	
	18031	26/8	1	
	18036	26/8	4	
			Total	*(120)*
Early Post-Meroitic *Room 1* 18001 18003 18009 18022 18024	18001	1	5	
	18003	1	12	
	18009	1	27	
	18022	1	30	
	18024	1	9	
	18042	Robbing trench fill of main N-S wall	7	
	18047	Dump around Room 8 cellar	4	
			Total	*(94)*
Post-Meroitic	10107	7	55	
	10110	6	44	
			Total	*(99)*

[1] The matrices on the left-hand side of the table are from (Edwards, 1998a). Only contexts from where sherds were sampled are shown. Broadly, these show that, for example, context 18036 was 'under' 18014, which in turn was 'under' 18013, etc.
[2] Figure 1.5 shows where the individual room numbers actually fit into Structure 265
[3] The 'p-numbers' for the individual illustrated sherds are given in Appendix 1 (Table A1.8)

4.1.2 Assessment procedure

In order to identify the origins of the lipids absorbed in the selected potsherds the following data treatments were performed:

1. Lipid compositions of all of the potsherds were investigated. Triangular plots are used to graphically display the range of lipid compositions detected in the sherds. The extracts exhibiting characteristic palm kernel lipids were then removed from the data set and assessed separately.

2. $\Delta^{13}C$ values ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) were calculated for the reduced data set (i.e. not including the sherds containing the palm kernel lipids), and these values were plotted against their $C_{16:0} / C_{18:0}$ abundance ratios. Classification of the origins of the extracts was based on comparisons with the results of analyses of the environmental material described in Section 3.3.
3. Detailed investigation of the $\delta^{13}C$ values of the individual fatty acid components enabled the relative importance of C_3 and C_4 plants to the people (and animals) at Qasr Ibrim to be elucidated.
4. GC/MS allowed the identification of specific plant/animal biomarkers (e.g. sterols), and the state of preservation of the lipids to be assessed (e.g. through the identification of TAGs, α,ω-dicarboxylic and hydroxy acids, as witnessed in the palaeoenvironmental material).

Figure A2.1 in Appendix 2 shows the chemical structures of the major compounds discussed herein.

4.1.3 Lipid preservation

Figure 4.1 shows four different lipid distributions that are typical of the assemblage. Chromatogram A displays a profile indicative of a palm kernel origin due to the unusually high abundances of $C_{12:0}$ and $C_{14:0}$, and as a result it plots in the bottom left-hand corner of the triangular plot (Fig. 4.2), as discussed

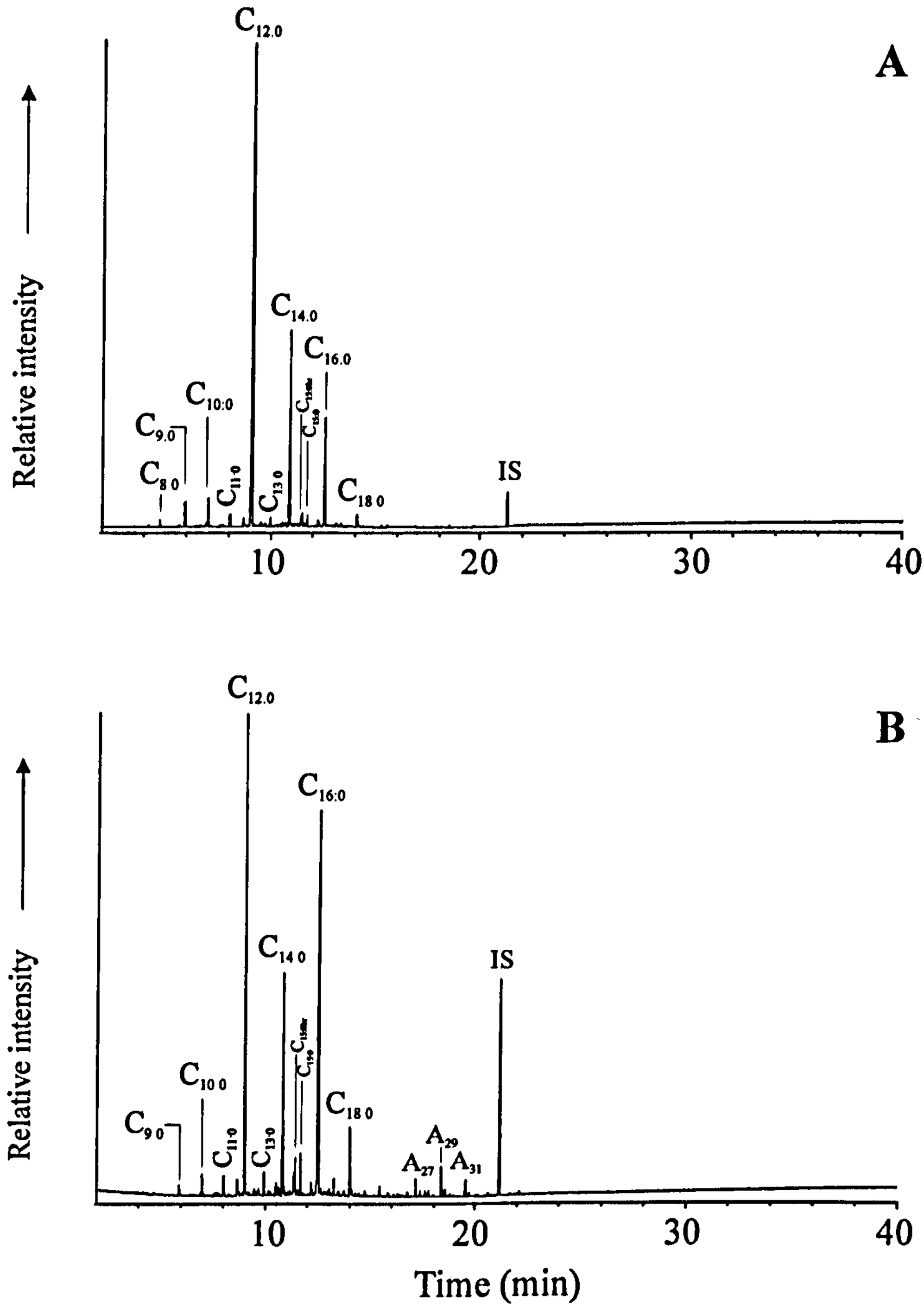

Figure 4.1 Examples of partial gas chromatograms of sherds from Qasr Ibrim. C_x refers to fatty acids of carbon chain length x, and A_y to *n*-alkanes of carbon chain length y. Chromatogram A shows a palm kernel lipid distribution (with very high abundances of $C_{12:0}$), and B a lipid distribution typical of degraded palm kernel lipids or a mixture of oils/fats and palm kernel lipids. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

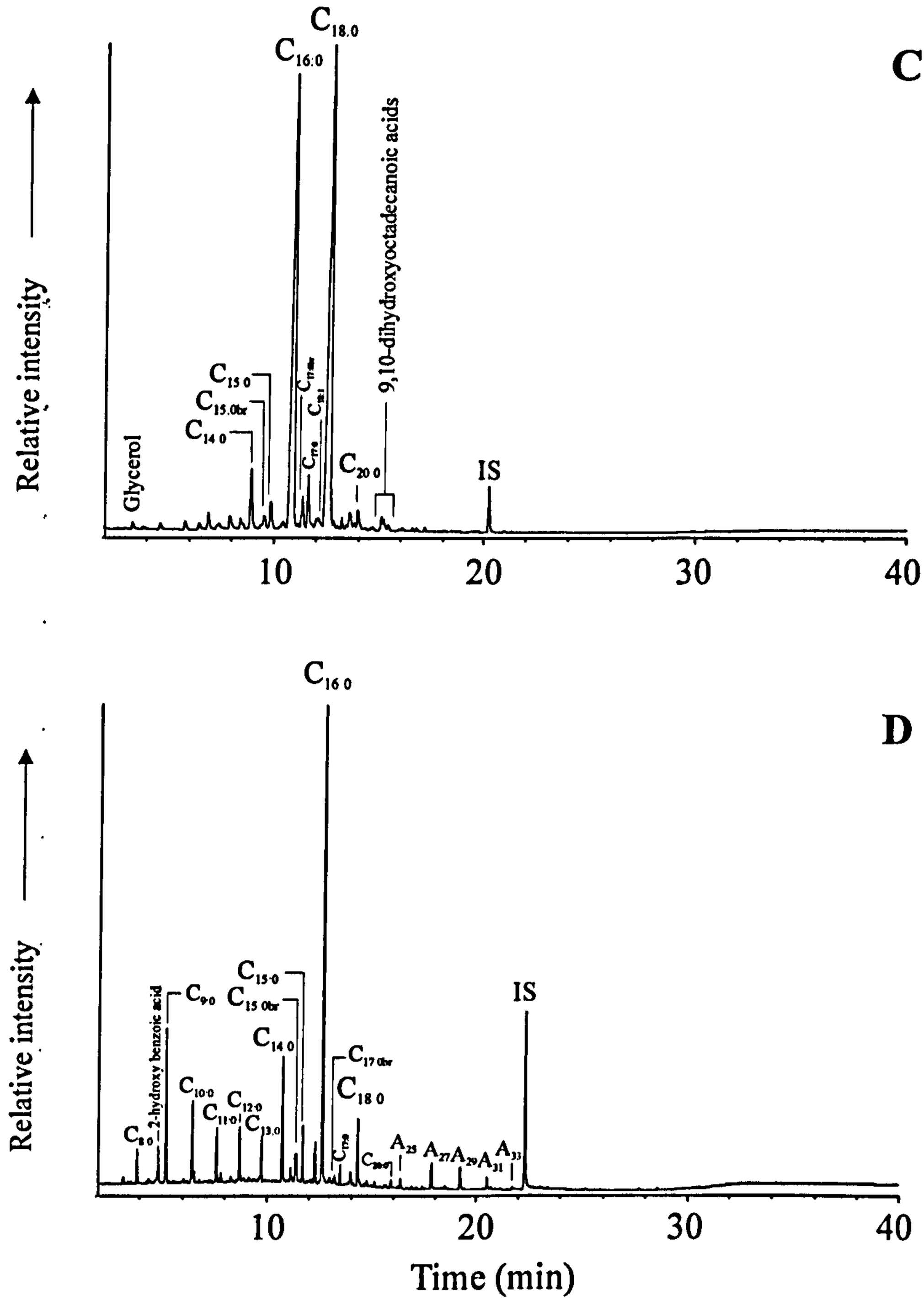

Figure 4.1 cont.
Chromatogram C shows a fatty acid distribution indicative of an animal fat (with relatively high abundances of $C_{18:0}$), and D shows a lipid distribution typical of a plant origin (with a relatively high abundance of $C_{16:0}$ and a low abundance of $C_{18:0}$). C_x refers to fatty acids of carbon chain length x, and A_y to *n*-alkanes of carbon chain length y. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

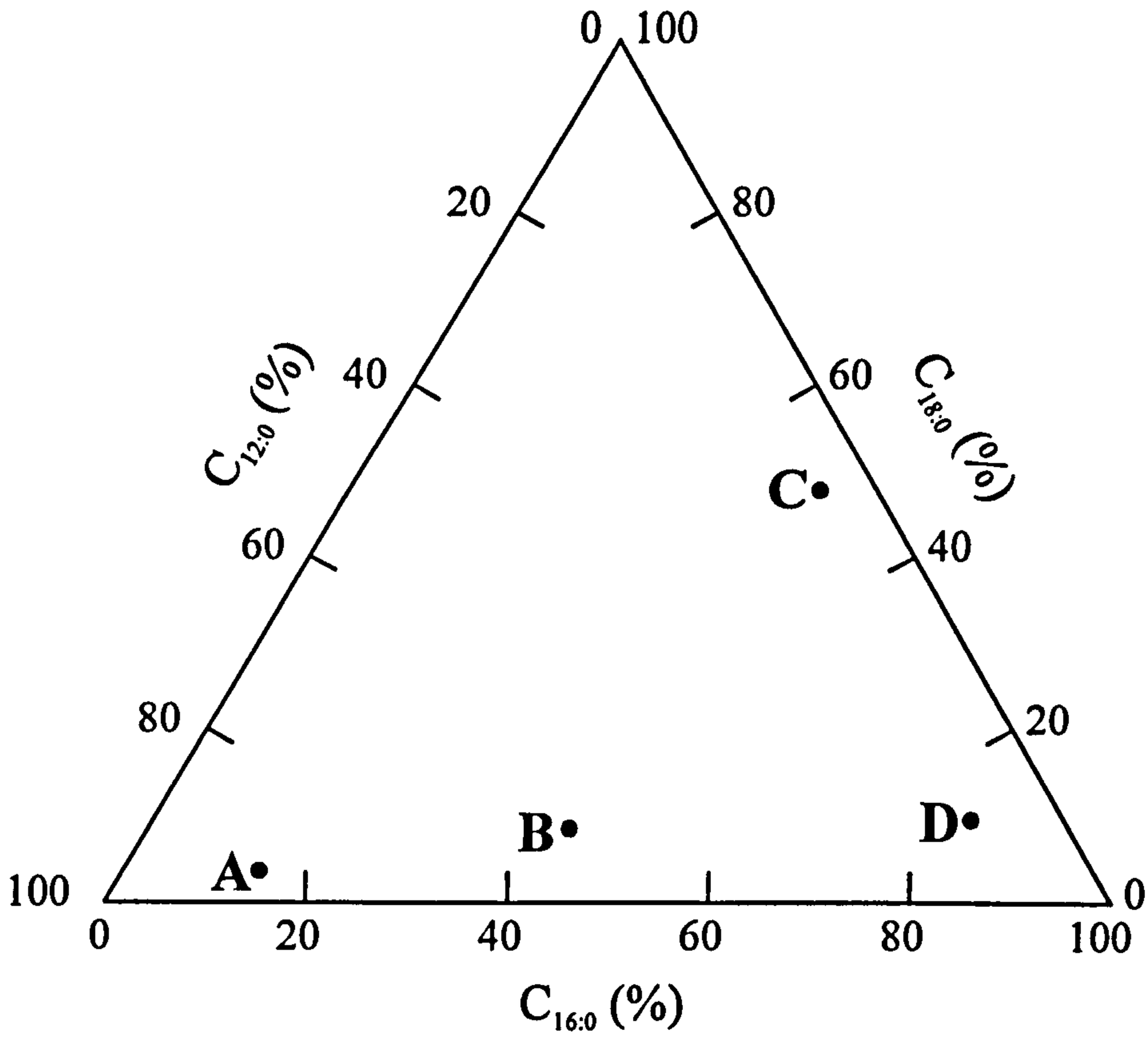

Figure 4.2 Triangular plot of the fatty acid composition of the sherds shown in Figure 4.1.

in Section 3.3. The sherd represented in chromatogram B is also indicative of a palm kernel origin, although due to the high relative abundances of $C_{16:0}$ and $C_{18:0}$, it is possible that this represents either (i) a palm kernel origin, where loss of the lower molecular weight fatty acids has occurred during the processing of the commodity, or (ii) a mixture of palm kernel lipids and another oil/fat (that has occurred either contemporaneously or during subsequent vessel re-use); this sherd extract plots at the bottom of the triangular plot, almost in the centre of the $C_{16:0}$-axis. The extract shown in chromatogram C is indicative of an animal fat (based on the high abundances of $C_{16:0}$ and $C_{18:0}$), whereas chromatogram D is indicative of a plant origin (based on the significantly higher abundance of $C_{16:0}$ relative to $C_{18:0}$). These two sherd extracts plot along the $C_{18:0}$-axis, essentially

due to the low abundance of the $C_{12:0}$ fatty acid as seen for the environmental material, discussed in Section 3.3.

A large percentage of the potsherds yielded appreciable concentrations of lipid, up to 1260 μg g^{-1} of potsherd. Two hundred and nine out of 313 of the sherds (67%) contained > 5 μg g^{-1}, and 57 sherds (18%) had little (<1 μg g^{-1}) or no extractable lipid. Figure 4.3 shows the concentration of extractable lipid for all of the sherds, plotted onto a triangular graph. It can be seen that the sherds that tended to yield the highest concentrations of lipid plot within the areas

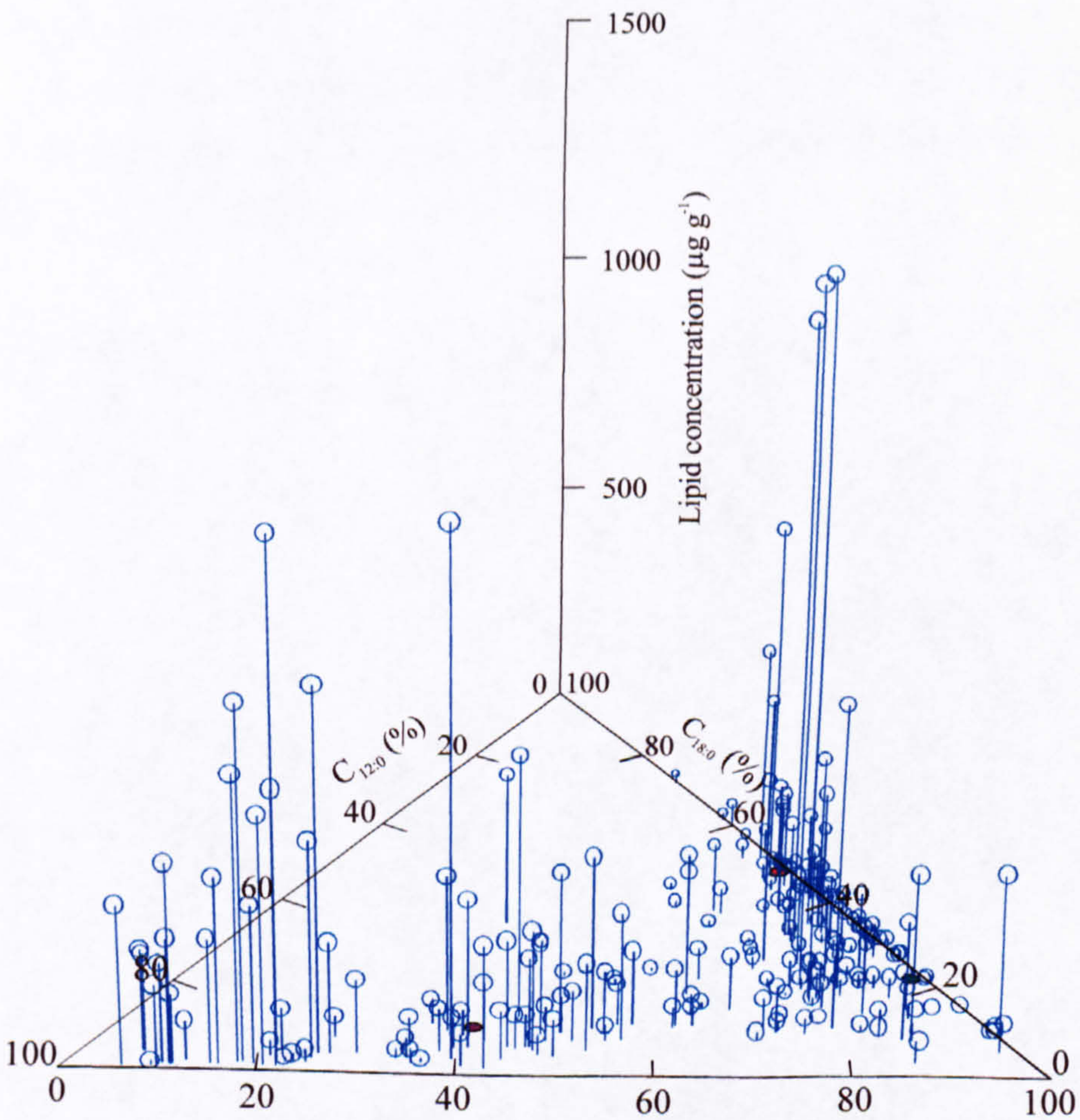

Figure 4.3 Triangular plot showing concentration of extracted lipid. The mean values for the reference materials are plotted onto the 2D portion of the triangular plot: Plant: ● Animal: ● Palm kernel: ●.

associated with the 'animal' and 'palm kernel' reference materials. The reason for the lower concentration of lipid in the extracts that plot the plant material may be that animal products tend to have a higher acyl lipid content (% dry wt.) than plants. This could lead to the record being biased towards animal fats (*c.f.* the mixing curves in Fig. 3.23). The elevated values for some of the samples plotting in the 'palm fruit' region of the graph is intriguing and is discussed in Section 5.1.

Only three out of 313 (1%) sherds (107·7; 107·14 and 14·8) yielded detectable quantities of TAGs and DAGs, and then only in traces amounts. However, in 73 sherds (23%) C_{18} MAGs are detectable with 45 of these (14% of the total) also displaying C_{16} MAGs; in all cases those showing acyl substitution in the *sn*-1 or 3 position were more abundant than the *sn*-2 isomers. The overall low abundance of intact acyl lipids indicates that extensive hydrolytic degradation has occurred during vessel use or burial.

Unsaturated carboxylic acids, principally $C_{18:1}$, were detected in only 17 of the sherds (5%). Considering the abundance of $C_{18:1}$ (and other unsaturated compounds) that are found in modern plants and animals, it is clear that extensive oxidation of the unsaturated components has occurred during both vessel use and burial. The products of oxidative decay are seen in 14 extracts where a range of α,ϖ-dicarboxylic acids are present (with $C_{9:0}$ predominating); correspondingly 9,10 dihydroxyoctadecanoic acids were detected in 33 of the sherds (11%). These distributions of components are analogous to those witnessed in the archaeological bone samples (Section 3.1). Furthermore, relatively short-chain fatty acids ($\leq C_9$) were detected in 59 of the sherds (19%). These short-chain fatty acids are, in part, likely to be the result of degradative processes, although the exact mechanism is not known. The fact that they survive in the pottery vessels at all is due to the arid environment that exists at Qasr Ibrim, as they are rarely found in deposits that undergo periodic water logging (e.g. in Britain).

Whilst extensive hydrolytic and oxidative degradation of the lipids has occurred during burial, due to the fact that the region is extremely arid, there would not have been any significant loss of lipids from the sherds via groundwater leaching. This unusual state of preservation justifies using the relative abundances and carbon ranges of fatty acids as robust diagnostic criteria to classify the origins of residues and uses of vessels (see below). Such criteria cannot be used at sites prone to waterlogging or inundation by percolating groundwater because there would be preferential loss of the lower molecular weight fatty acids (Bell, 1973). Therefore, the $C_{16:0}/C_{18:0}$ relative abundance ratios would be affected.

4.2 THE MEROITIC POTTERY VESSELS

Figure 4.4 shows a typical chromatogram that was observed for the TLEs of Meroitic pottery. Fifty-five of the 120 sherds (46%) exhibit significant

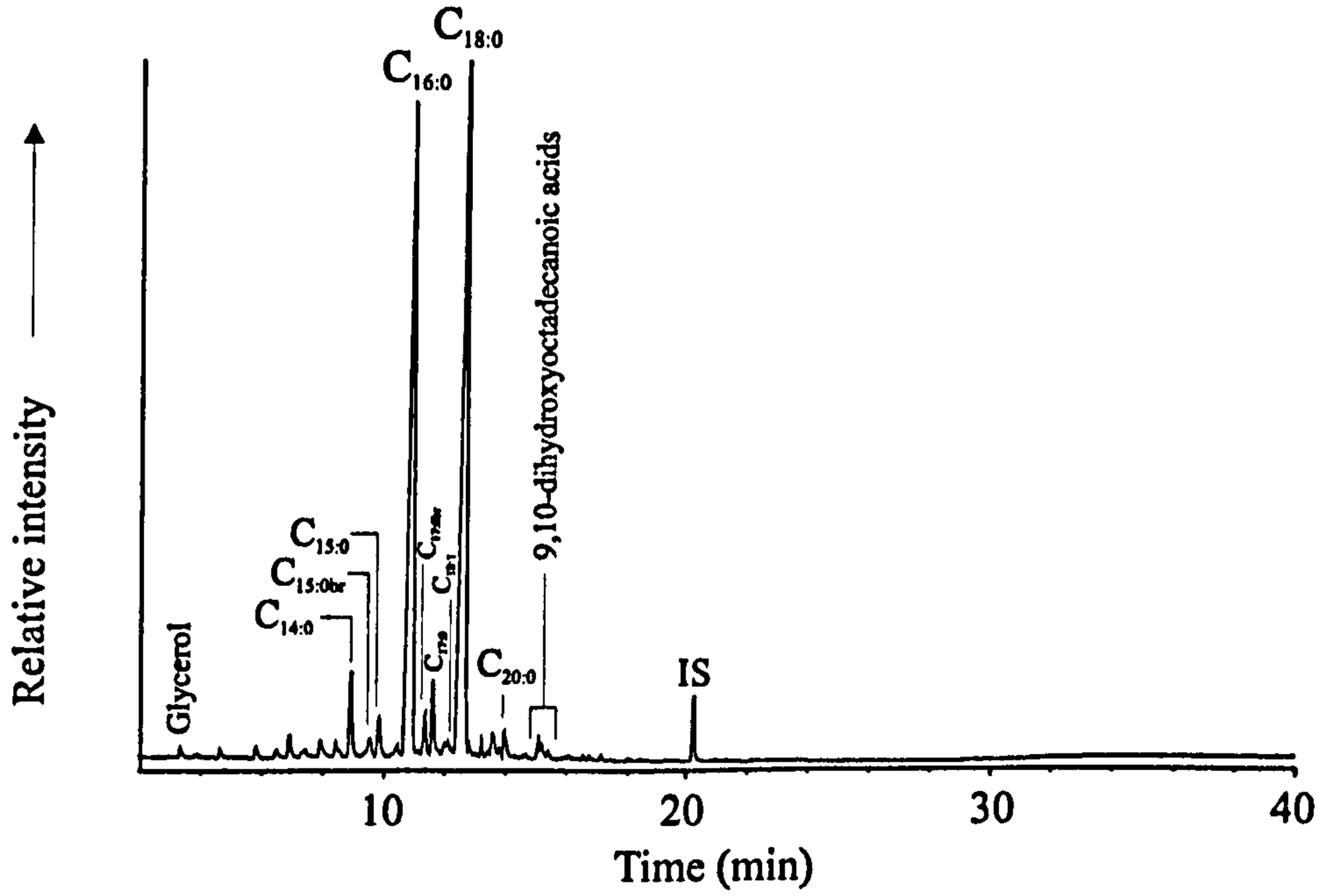

Figure 4.4 Partial gas chromatogram of the TLE of sherd 23·18. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

quantities of extractable lipid (>5μg g^{-1}), not unexpectedly dominated by saturated fatty acids.

One of the sherds (13·18; Fig. 4.5) has unusually high abundances of several short-chain fatty acids. Fatty acids in the carbon range $C_{6:0}$ to $C_{18:0}$ were detected in the extract from these sherds. $C_{5:0}$ may also be present, although this could not be verified by GC/MS because it elutes just after the solvent peak, and in order to protect the filament in the MS, data is not acquired for the first 2 min of the GC run. The fatty acids are most likely to be degradation products from a parent compound, because short-chain fatty acids are generally not found in such high relative abundances in living organisms. Although the exact processes involved are unknown, the high $C_{16:0}/C_{18:0}$ abundance ratio is indicative of a plant origin for the fatty acids. Compared with the majority of

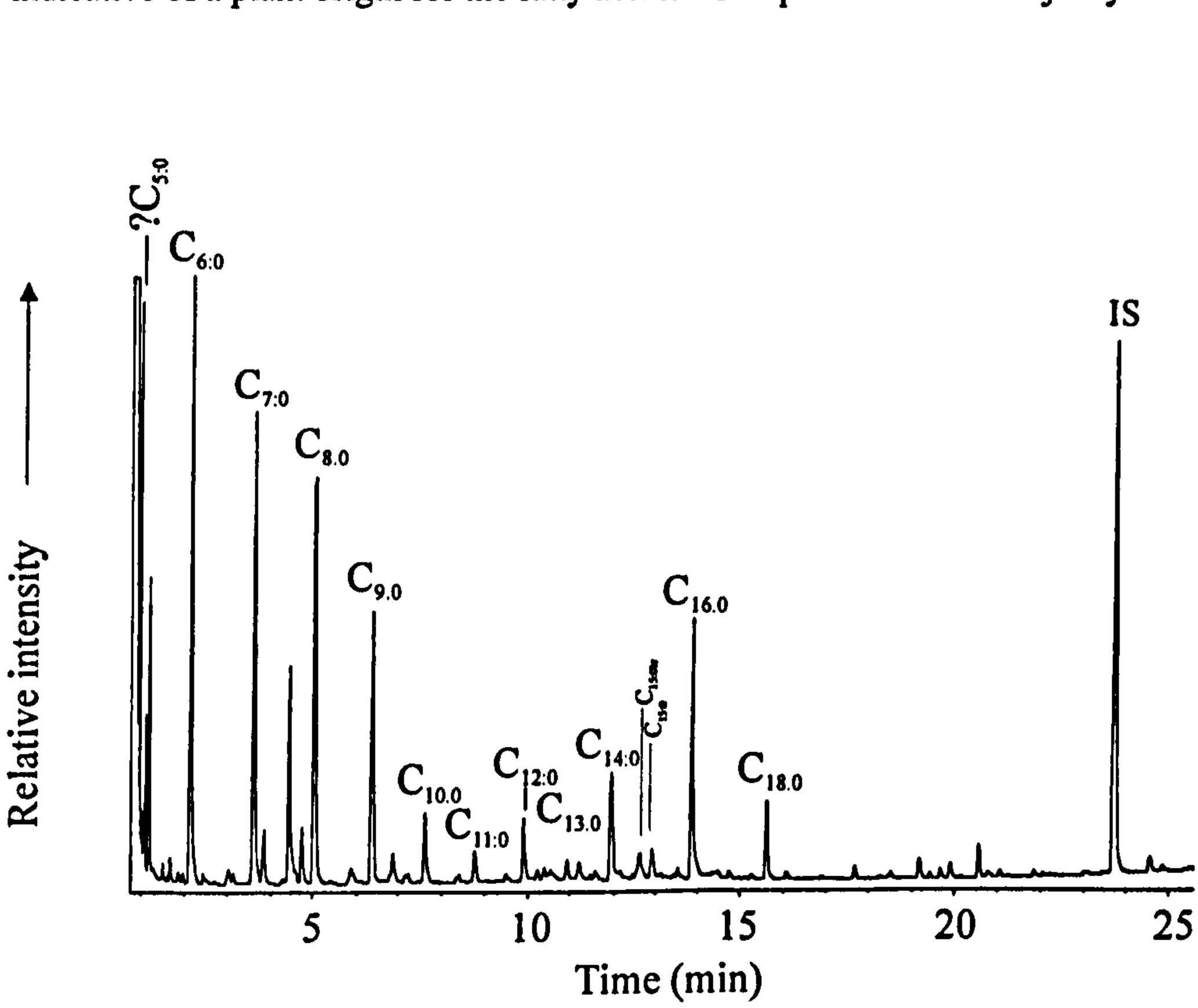

Figure 4.5 Partial gas chromatogram of the TLE of sherd 13·18. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). Eluting just after the solvent peak, $?C_{5:0}$ indicates probable $C_{5:0}$ fatty acid (see text). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

animal fats, plant oils have higher abundances of polyunsaturated fatty acids, and further work will need to be undertaken in order to determine whether this is a determining factor in the production of these very short chain fatty acids.

4.2.1 Fatty acid distributions

Figure 4.6 shows the $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$ triangular plot for all of the Meroitic sherds. One sherd plots to the left of the graph, indicating the presence of virtually pure palm kernel lipids in the extract. Furthermore, there is also evidence for the mixing of palm kernel lipids and other commodities in the vessels; three of the sherds that plot around the $C_{12:0}$ = 20% level in Figure 4.6. Upon closer inspection of the relative abundances of the $C_{14:0}$ component from these latter sherds, it can be stated that these too contain characteristic palm kernel lipids mixed with the fat/oil from another source (*c.f.* chromatogram B in Figure 4.1).

The triangular plot of the extracts grouped by context numbers (Fig. 4.7) shows that the sherds from context 18023 tend not to plot in the areas of the graph that are indicative of animal fats. However, the sherds from context number 18014 contain plant/mixed plant and animal lipids.

When the residues are grouped by ware type (Fig. 4.8), it can be seen that the three hand-made wares (H1) all plot towards the bottom right-hand corner of the triangular graph, thereby indicating a predominantly plant origin for the residues. The R25/32 wares cannot be distinguished as either being R32 or R25 (the latter is found more frequently in Early Post-Meroitic contexts), and the single R25/32 sherd from the Meroitic period plots firmly within the palm kernel lipid region of the triangular plot. The R32 wares plot within regions of the scatterplot indicative of a plant/animal origin for the extracts.

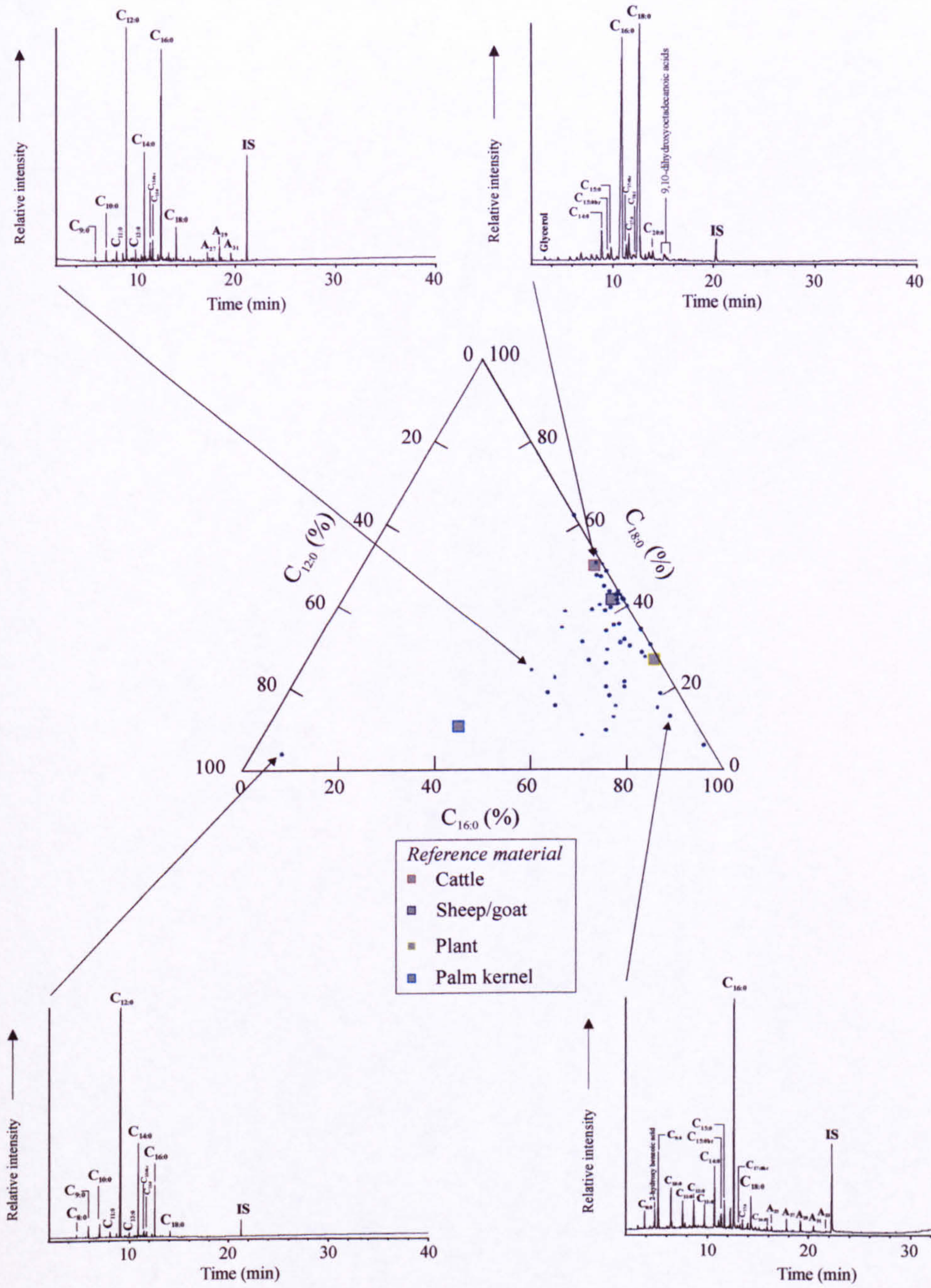

Figure 4.6 Triangular plot of the fatty acid composition of the sherds recovered from the Meroitic period. Partial gas chromatograms of the TLE from sherds that are typical of the lipid distributions from the Meroitic period (C_x refers to fatty acids of carbon chain length x, and A_y to *n*-alkanes of carbon chain length y). The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

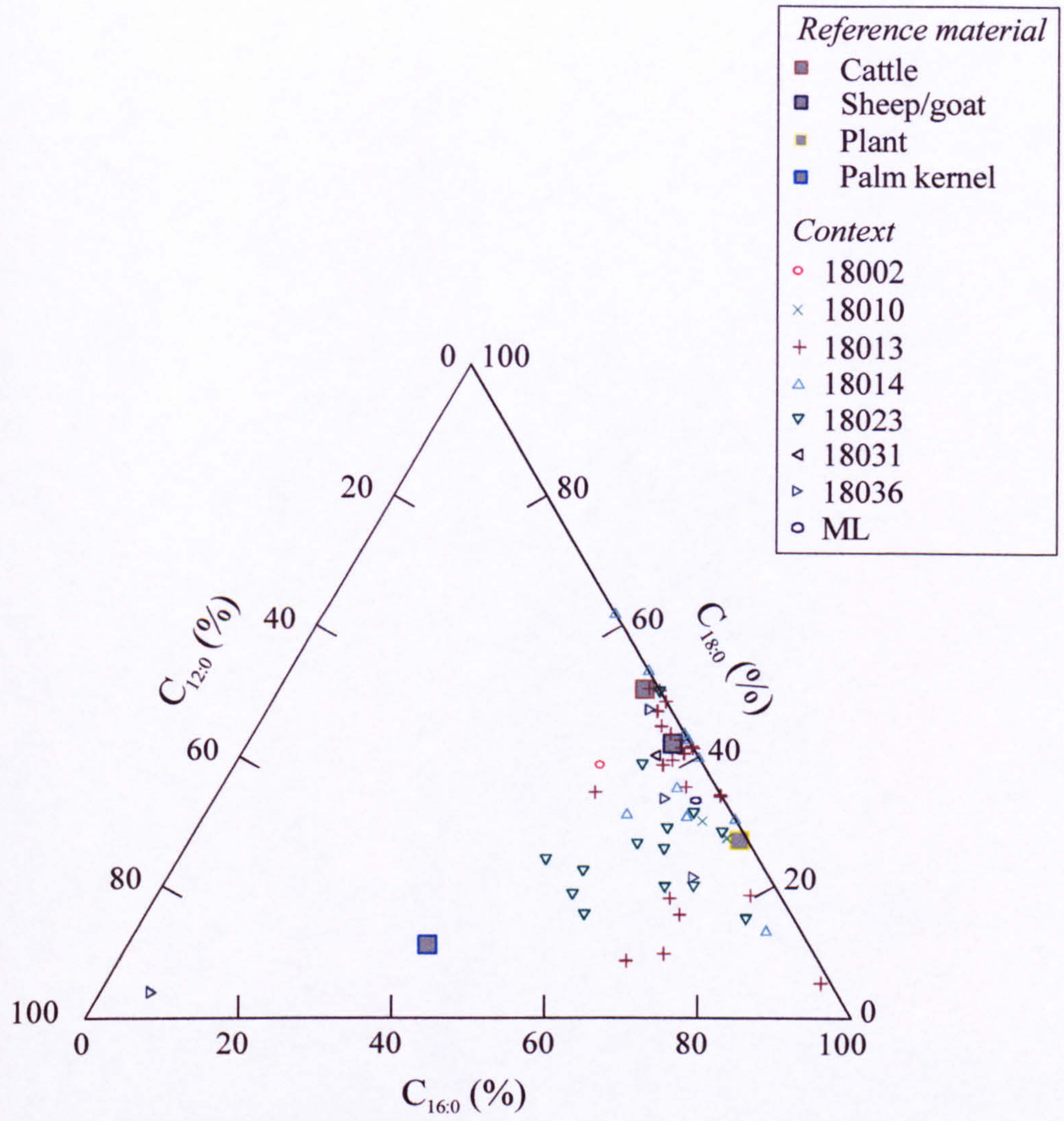

Figure 4.7 Triangular plot of the fatty acid composition of the sherds recovered from the Meroitic period, distinguished by context number. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

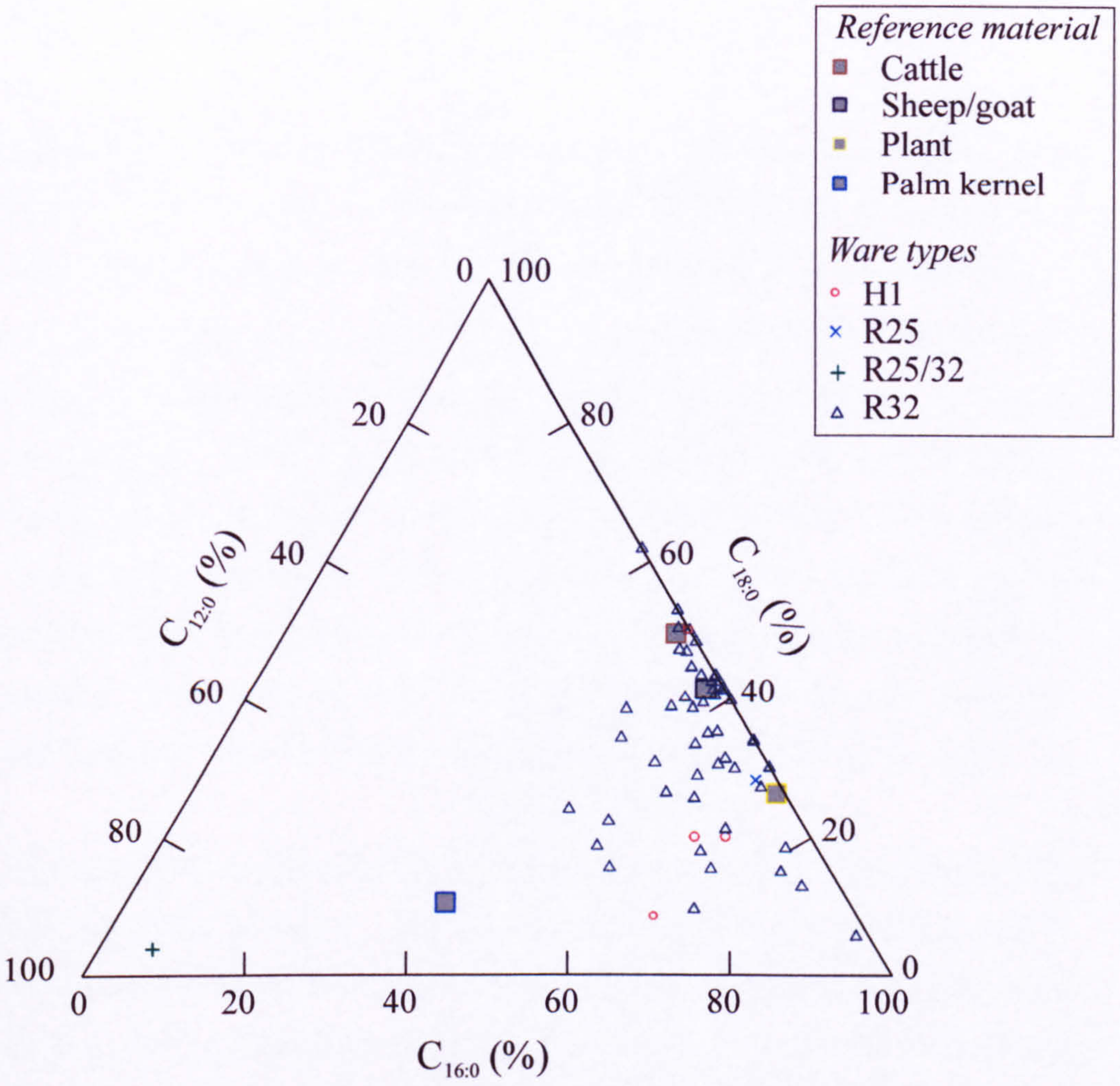

Figure 4.8 Triangular plot of the fatty acid composition of the sherds recovered from the Meroitic period, distinguished by ware type. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

4.2.2 Fatty acid stable isotope ($\delta^{13}C$) values

As reported above, four sherds contained fatty acids indicative of palm kernel lipids. These were removed from the data set and subjected to further data treatments (Section 4.7). The $\Delta^{13}C$ values of the remainder were plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, together with p=0.683 confidence ellipses established through the analysis of the reference environmental material (Section 3.3). The triangular plots suggest that many of the absorbed lipids from Meroitic sherds have an animal origin. This is substantiated in Figure 4.9, where only 7 vessels plot within the 'predominantly plant' field (12%), with a further two (4%) plot just to the right of this field and are also likely plant derived. In contrast, 25 of the extracts (44%) plot within the sheep/goat and cattle ellipses and hence can be attributed to having an animal fat origin.

Also worthy of further clarification are the four sherds that plot towards the top of the graph in Figure 4.9. The extracts are suggestive of a predominantly animal source for the lipids, due to the fact that they occur in the region where the $C_{16:0}$ to $C_{18:0}$ abundance ratio is between 1 and 2. However, these sherds do not plot within the reference ellipses and have $\Delta^{13}C$ values of greater than +1‰. The fatty acids from all these sherds have $\delta^{13}C_{16:0}$ values in the region of -28‰, and $\delta^{13}C_{18:0}$ values in the region of -25‰. The most likely explanation for these unusual $\Delta^{13}C$ values is that it represents the mixing of an animal fat with a plant oil that has significantly more depleted $\delta^{13}C$ values, as illustrated by the theoretical mixing curves shown in Figure 3.23.

Figure 4.10 is a plot of the $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ fatty acid components from the sherds. It shows that the majority of the vessels contained C_3 fatty acids with $\delta^{13}C$ values in the region of *c.* -27 to -31‰. Some of the sherds from context 18023 may also have mixtures of C_3 and C_4 fatty acids, as indicated through their higher $\delta^{13}C$ values, which were in the region of *c.* -20‰.

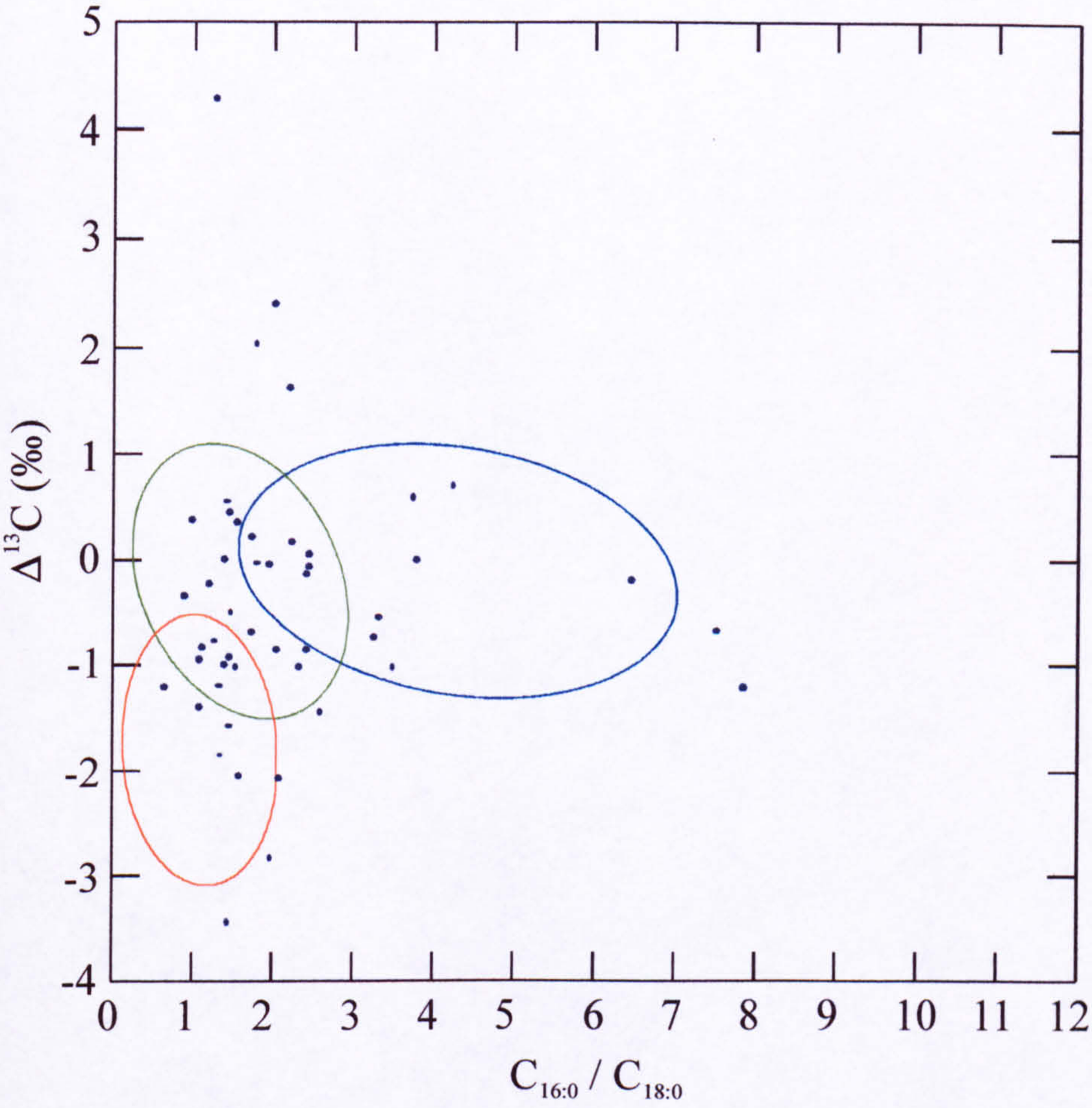

Figure 4.9 $\Delta^{13}C$ ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) of the Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

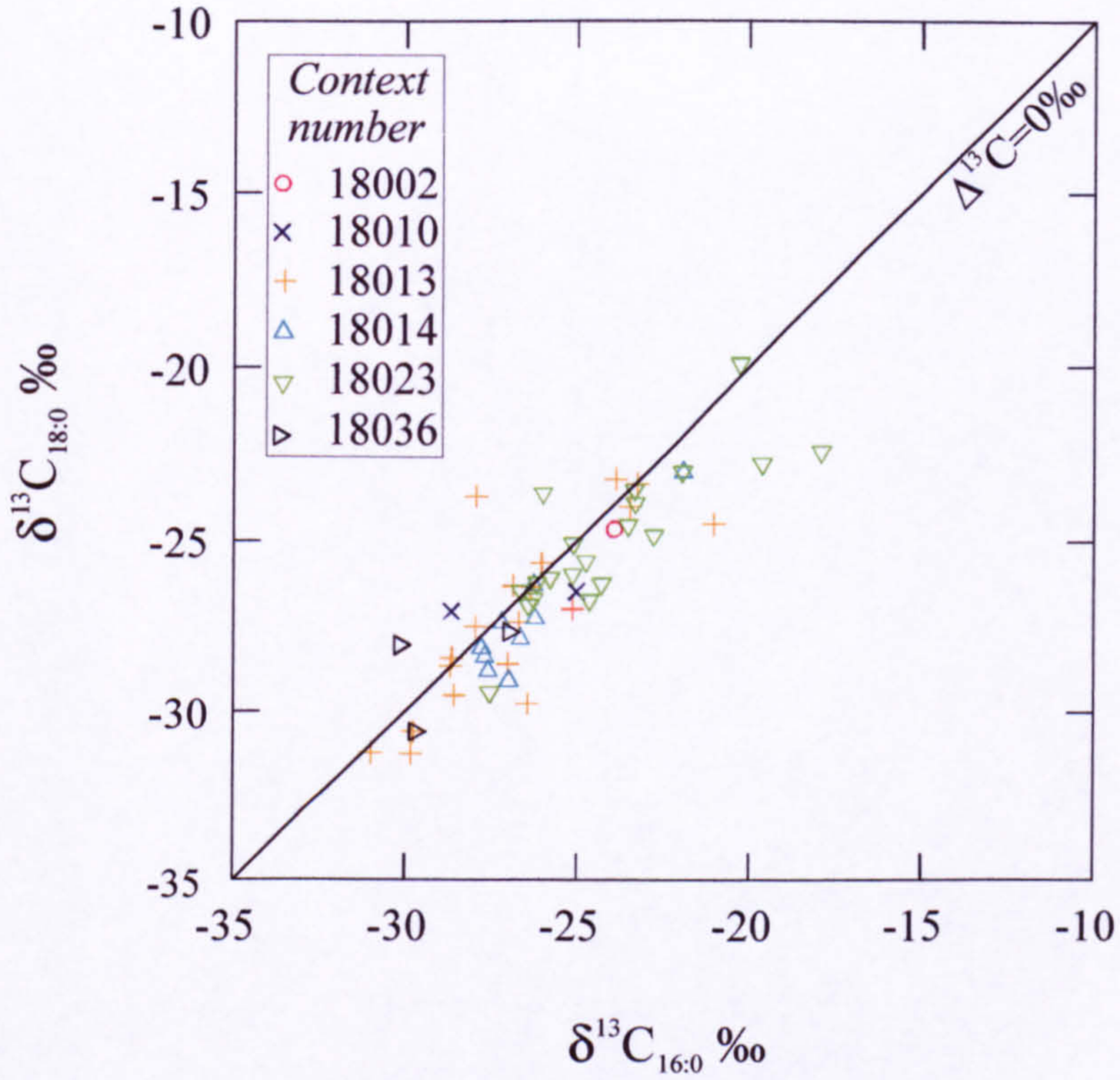

Figure 4.10 The $\delta^{13}C$ values of the major fatty acids extracted from the Meroitic sherds.

Figure 4.11 confirms that the sherds from the main contexts sampled for this period contain more animal than plant lipids, and that there does not appear to be any difference in vessel use within the varying contexts.

Although the pottery assemblage is dominated by the R32 wheel-made wares (Fig. 4.12), it is apparent that they were used to process all types of commodities, and were not preferentially utilised for any one purpose. The three hand-made wares all plot within, or close to, the plant group. However, not too much should be read into this due to the small numbers of sherds of this ware type represented here.

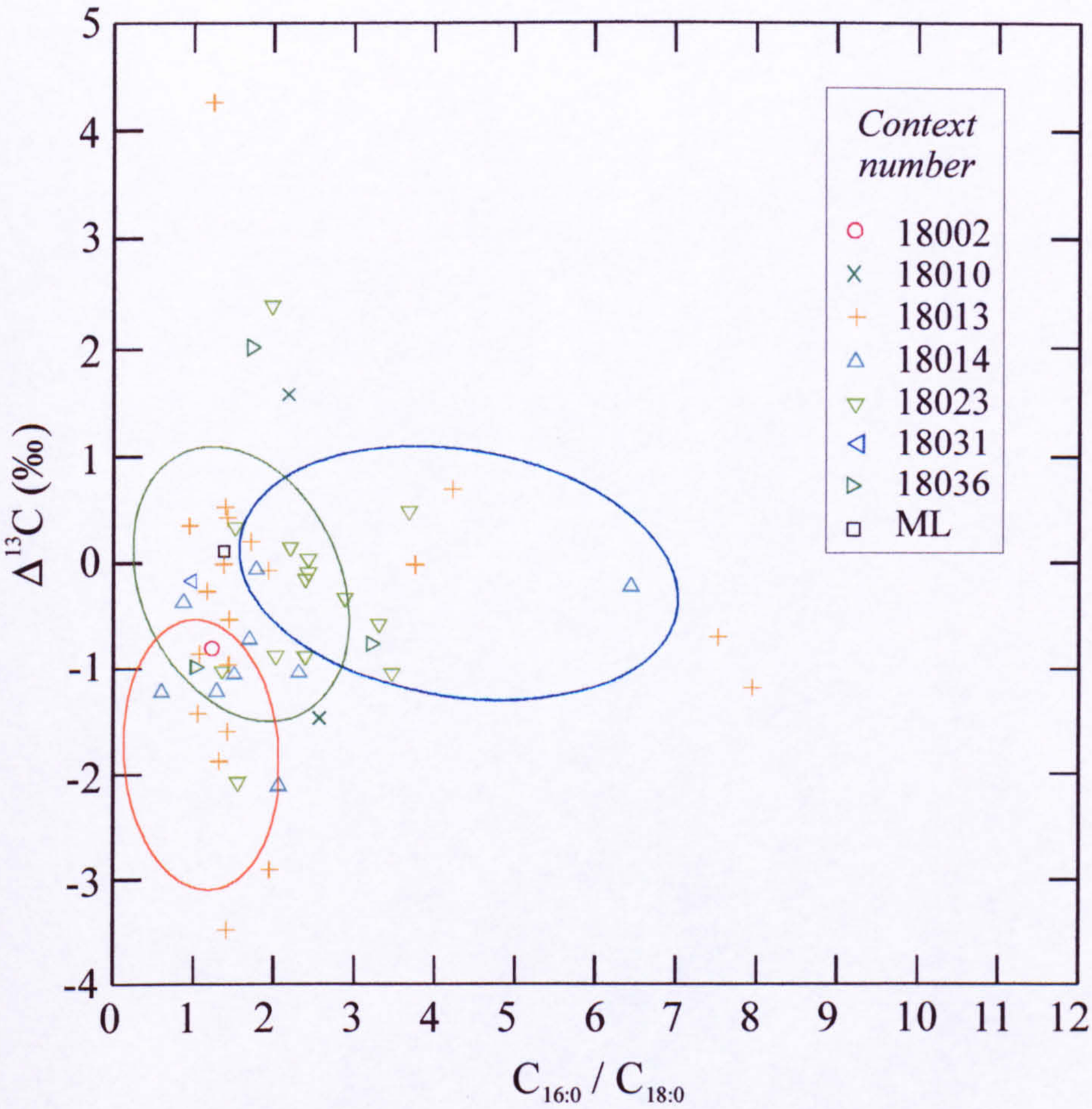

Figure 4.11 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by context number. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

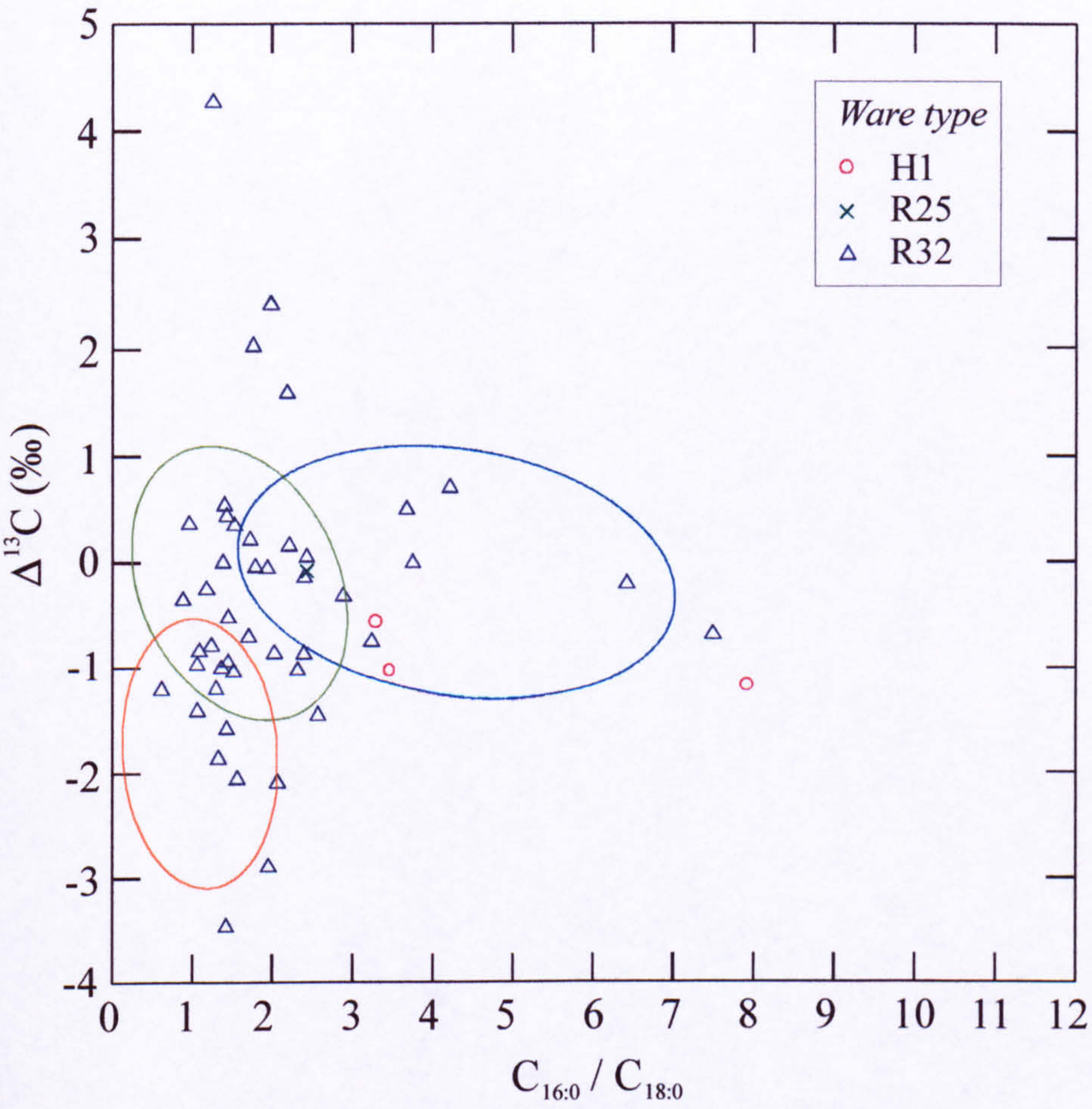

Figure 4.12 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by ware type. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

4.2.3 Other diagnostic compounds

In addition to the fatty acids, other diagnostic compounds, such as *n*-alkanes, sterols, stanols, levoglucosan and derivatives of benzoic acid were detected via HTGC/MS in some of the sherds excavated from the Meroitic contexts. These diagnostic compounds are an additional indicator of vessel use, because they are only found in specific types of commodity and organic material. Figure 4.13 indicates the sherds that yielded these diagnostic compounds. Interestingly, many of the sherds that contain fatty acid distributions indicative of an

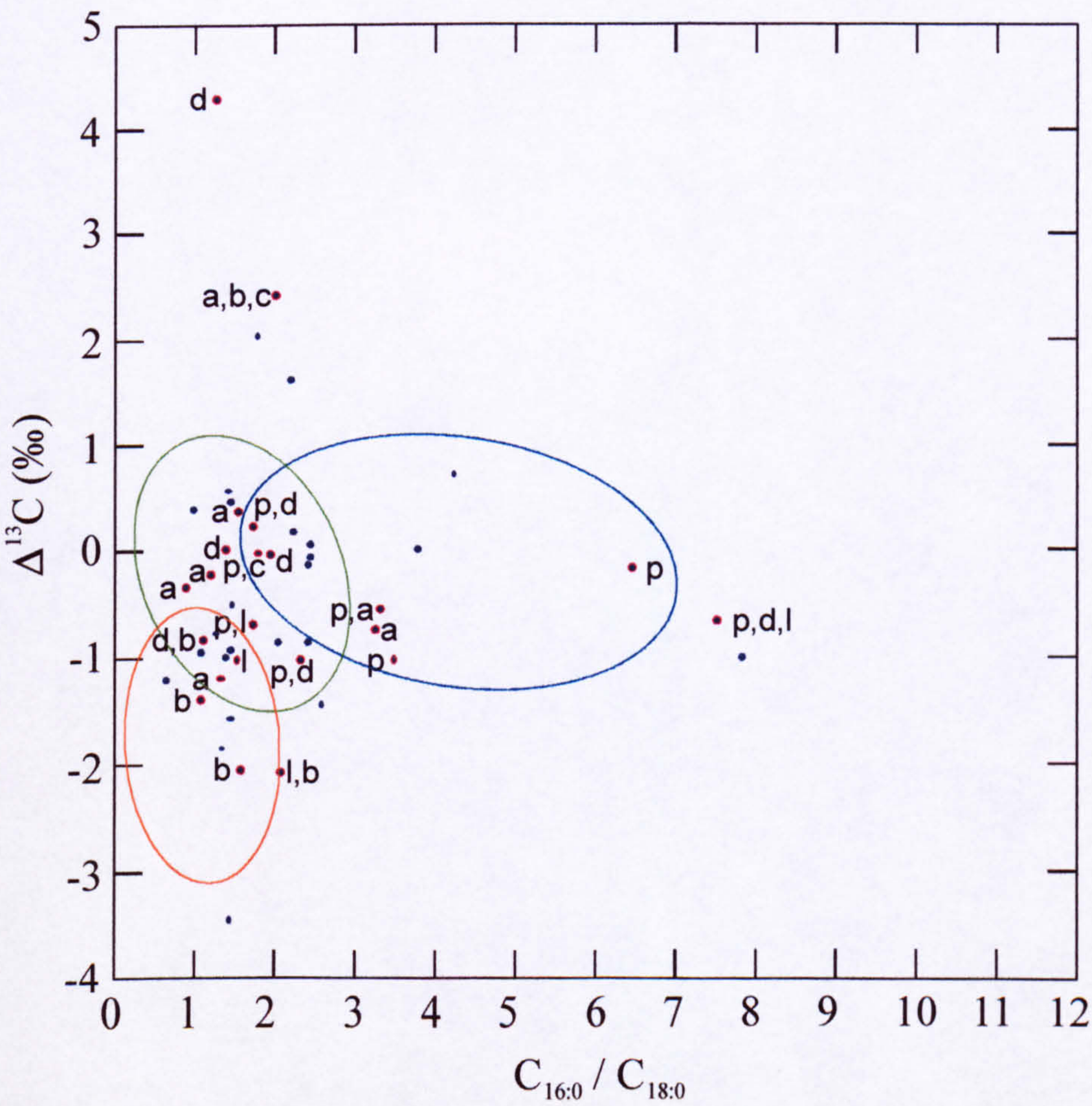

Figure 4.13 $\Delta^{13}C$ ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) of the Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, with labelled diagnostic compounds. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3). Key: 'p': plant sterols; 'a': *n*-alkanes; 'l': levoglucosan; 'b': benzoic acid derivatives; and 'd': coprostanol and other stanols.

animal fat also yielded diagnostic plant compounds. This indicates the mixing of animal and plant products in the vessels during use in antiquity. Each of these classes of diagnostic compound are investigated in more detail:

n-Alkanes are commonly found in higher plant epicuticular waxes, and although found in the stems, fruits and surface of grains, the greatest abundances of these compounds are generally contained on the leaves (e.g. Kolattukudy, 1976:582; Walton, 1990:114). Due to their apolar nature, they appear to survive well in pottery (e.g. Evershed *et al.*, 1991; Evershed *et al.*, 1997d). *n*-Alkanes were detected in seven of the sherds from the Meroitic period (Figure 4.13), and six of these contained significant concentrations of *n*-alkanes such that their distributions could be investigated (Fig. 4.14), showing that the C_{27} to C_{31} odd molecular weight components predominate. Four of these sherds also yielded fatty acids characteristic of predominantly animal fats, thus indicating mixing of commodities has occurred in the vessels. This latter point also highlights the

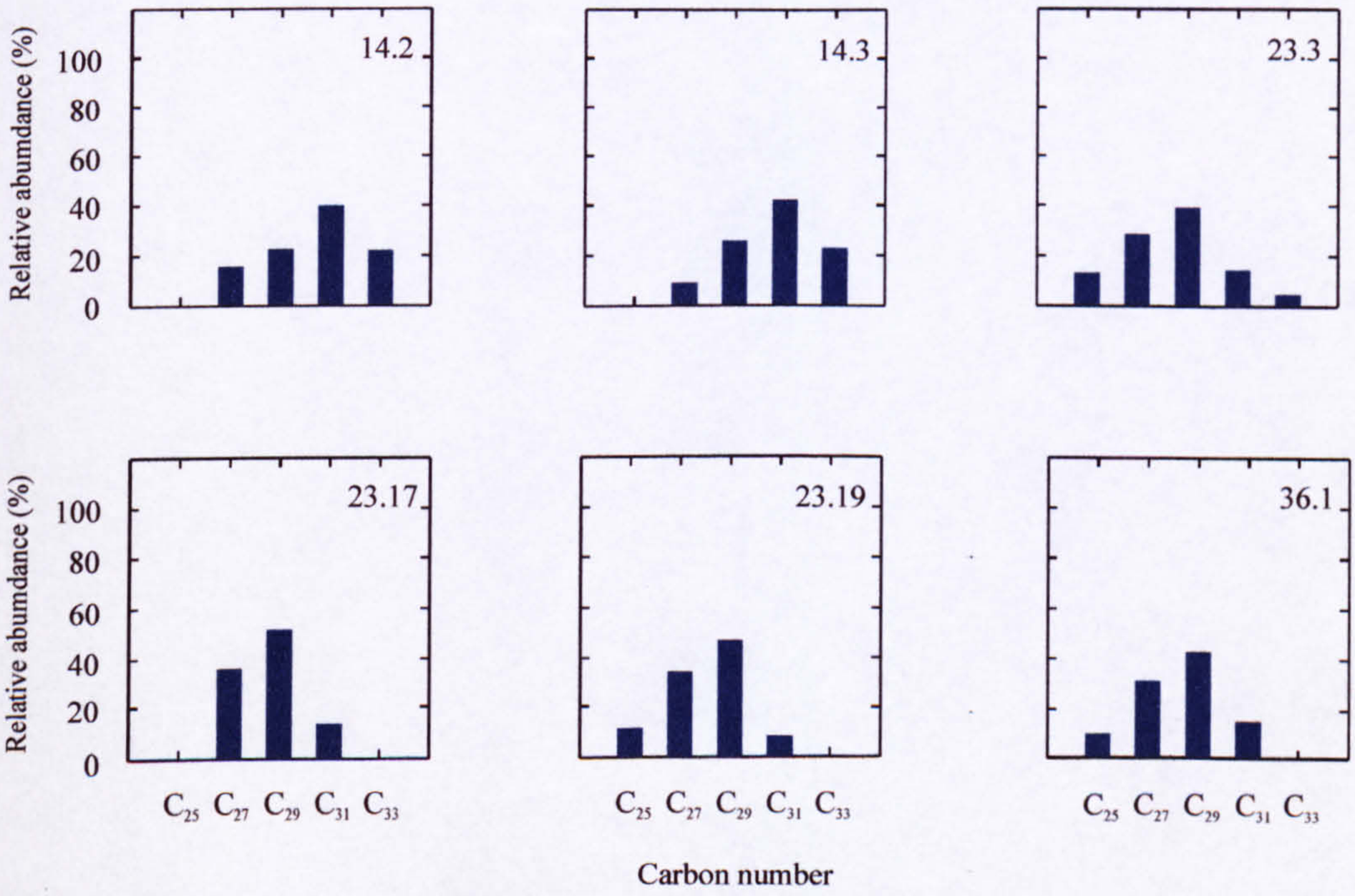

Figure 4.14 The *n*-alkane distributions extracted from the Meroitic sherds. Only the sherds that yielded significant abundances of *n*-alkanes are shown. The sherd numbers are shown in the top right corner. C_X refers to *n*-alkanes of carbon number x.

fact that classification of the sherds involves the use of confidence ellipses, and that statistically they may include relatively low abundances of plant material mixed with relatively higher abundances of animal products (as discussed in Section 3.3). Hence detailed mass spectrometric analysis is vital to the overall determination of vessel use. The *n*-alkane distributions are discussed in detail in Section 5.3.

Sterols are also a useful indicator of the processing of plant material in the vessels. β-sitosterol (stigmast-5-en-3β-ol), stigmasterol [(24*R*)-stigmasta-5,22-dien-3β-ol], and campesterol [(24*R*-24-methylcholest-5-en-3β-ol] are typical plant sterols that survive in the botanical reference materials from Qasr Ibrim (Section 3.2.2). β-Sitosterol was detected in six of the vessels, but only in very low concentrations. Cholesterol (choles-5-en-3β-ol), a lipid biomarker that is present in animal products, was not detected in any of the vessels, but its degradation product, coprostanol (5β-cholestanol), is present in five of the sherds from this period, an example of which is shown in Figure 4.15. The microbial transformation of cholesterol to coprostanol is known to occur in the human gut (Bjorkhem and Gustafsson, 1971) and is formed through several oxidation and reduction stages to form coprostanol (Ren *et al.*, 1996). Figure A2.5 in Appendix 2 illustrates the reduction of cholesterol to 5α- and 5β-stanols. Coprostanol has been detected in human adipocere (Adachi *et al.*, 1997), bog bodies (Evershed and Connolly, 1994), in archaeological bone (Evershed *et al.*, 1995b; Stott and Evershed, 1996), archaeological coprolites (Lin *et al.*, 1978) and archaeological sediments (Pepe *et al.*, 1989; Pepe and Dizabo, 1990; Bethell *et al.*, 1994), and has even been used as an indicator of ancient manuring practices (Evershed *et al.*, 1997a; Simpson *et al.*, 1998). However, whilst 5β-stanols have been noted in lacustrine sediments (Gaskell and Eglington, 1975), such transformations do not appear to be produced by micro-organisms living in sedimentary systems (Bull *et al.*, 1998:80).

Relatively high concentrations of coprostanol (up to 20 μg g^{-1}) were detected in five of the Meroitic vessels. Four of these have fatty acids that are indicative of

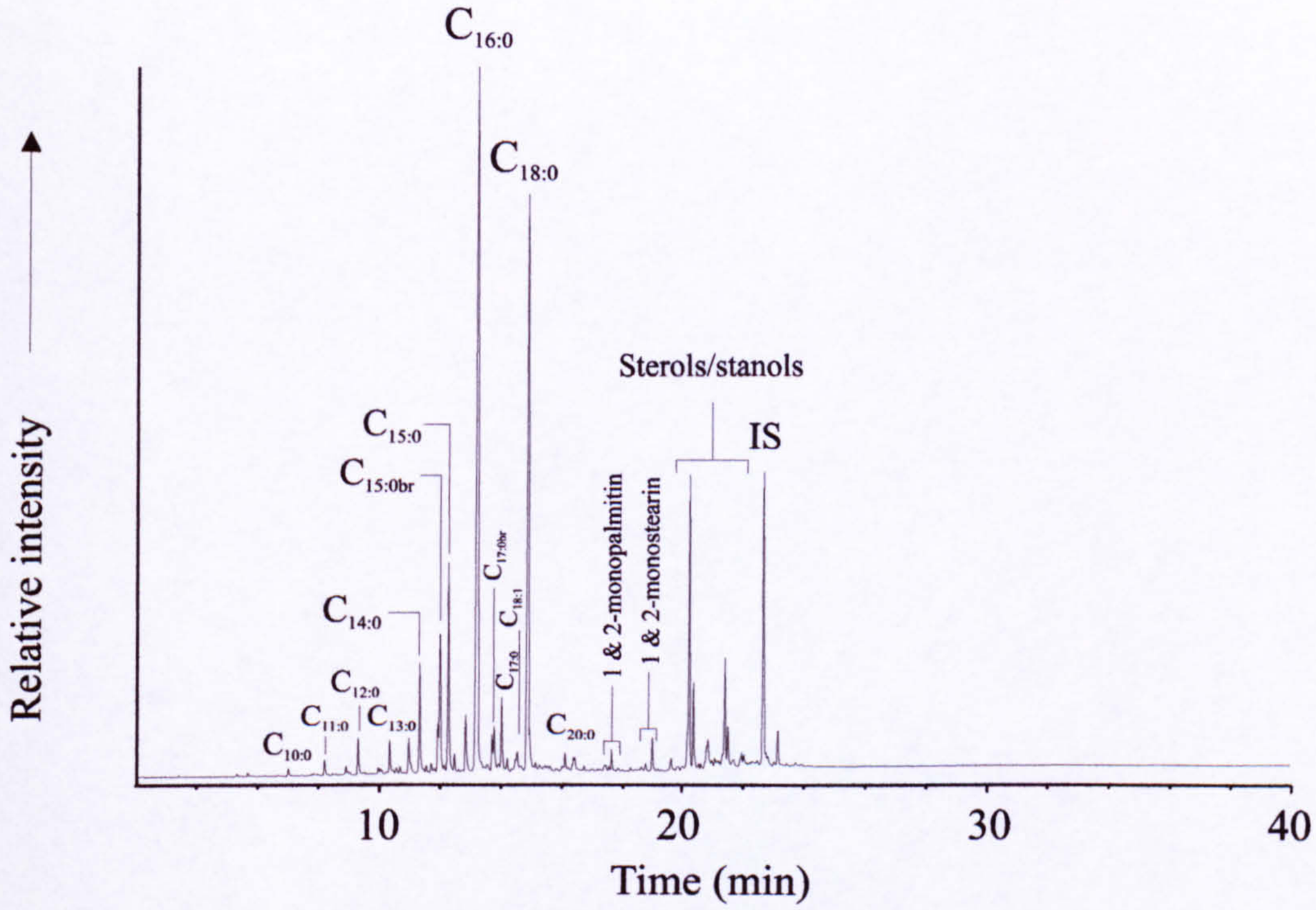

Figure 4.15 Partial gas chromatogram of the TLE of sherd 13·4. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). Identification of the individual sterols/stanols is given in Figure 4.16. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

the processing of animal products; an example (sherd 13·4) is shown in Figure 4.15. A further sherd plots at $C_{16:0}/C_{18:0}$ abundance ratio ≈ 7, and hence does not contain fatty acids that have an animal origin. The fact that all five sherds were from one context (18013) may indicate that the context itself was partially associated with degraded sterols (e.g. from faecal material). A faecal contribution of some description is corroborated through HTGC/MS of the TLEs. Not only was coprostanol detected, but other stanols and sterols are also present in some of the sherds (Fig. 4.16): i.e. 5β-epicholestanol, 5β-campestanol, 5β-epicampestanol, 5β-stigmastanol, 5β-epistigmastanol, as well as 5α-cholestanol, 5α-stigmastanol and β-sitosterol. 5β stanols have been shown to be particularly useful in indicating the presence of faecal matter in a range of archaeological contexts, for instance in field systems and cess pits (Evershed *et al.*, 1997a; Bull *et al.*, 1998). Furthermore, human faeces

typically yield high relative abundances of coprostanol, and lower relative abundances of 5β-stigmastanol. A ratio of 1.5:1 (coprostanol:5β-stigmastanol) has been calculated as the minimum relative abundance of coprostanol needed to ensure that the faecal material is of a human/porcine rather than of a ruminant animal origin (Bethell *et al.*, 1994); for human/porcine faeces the ratio is *c.* 5.5:1, while for ruminant faeces, the ratio is *c.* 1:4. Three of the sherds from context 18013 yielded coprostanol:5β-stigmastanol abundance ratios of between 3.1 and 3.5; significantly higher than the minimum 1.5 that is suggestive of human faecal material. The other two sherds did not contain detectable quantities of 5β-stigmastanol, and so relative abundance ratios could not be determined, however it is likely that these two sherds also contain human faecal material, given the high abundances of coprostanol in the sherds.

There are two explanations for the presence of faecal material in these sherds. Firstly, there is the possibility that the room that the sherds were excavated from was, at least partially used as a deposit for waste material, leading to gross contamination of the deposit. However, since coprostanol and the other 5β-stanols are present in only five of the 23 sherds from context 18013, this appears to be unlikely. Furthermore, there is no archaeological evidence for the use of the context as a deposit for waste material. A second explanation for the presence of faecal material in these sherds is that they originated from vessels that were used to contain waste material, i.e. they were used as chamber pots. This explanation is perhaps the more likely given the particularly large abundances of coprostanol, and the number of sherds that contain these stanols. As no soil samples were available for analysis, which would have allowed to test for the presence of faecal material in the soil, the exterior portion of the sherd was removed with a modelling drill and solvent extracted. The results showed that coprostanol and the other stanols were not present in the exterior portion of the five sherds, but were only absorbed in the interior of the five sherds; thus adding credence to the possibility of these being used as chamber pots. It is assumed that this was the final use of the vessels; indeed re-use of the vessels is attested to by the fact that the sherds also contain high

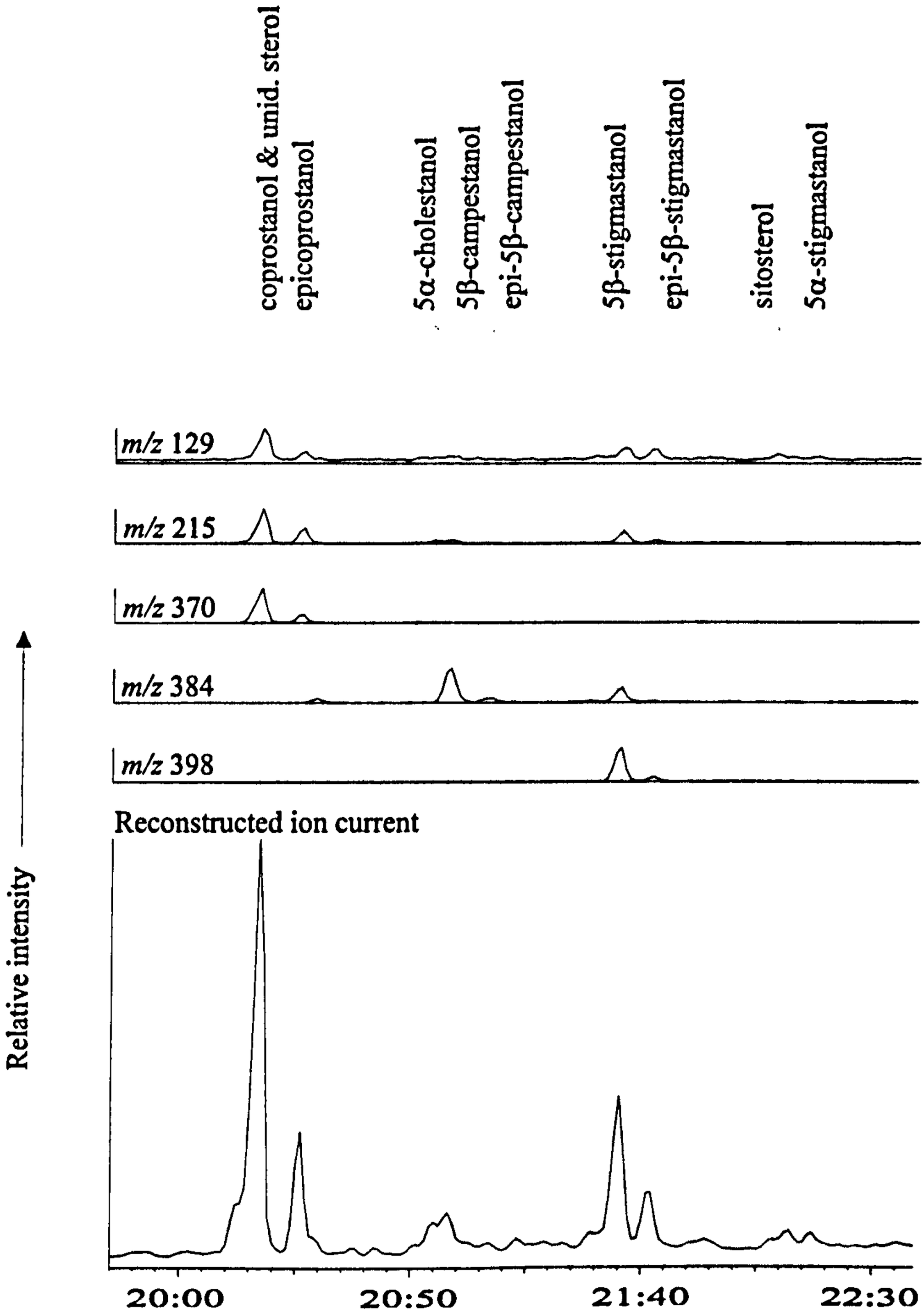

Figure 4.16 Partial HTGC-MS RIC and *m/z* 129, 215, 370, 384 and 398 mass chromatograms of the TLE of sherd 13·4. *m/z* 215 is a characteristic ion for the 5β-stanols. The other *m/z* are characteristic of the particular stanols/sterols indicated. A high coprostanol to 5β-stigmastanol ratio is indicative of human or pig faeces.

abundances of fatty acids characteristic of the use of the vessels in the processing of animal and/or plant products (Fig. 4.13).

Levoglucosan (1,6-anhydro-β-D-glucopyranose) is not found in abundance in nature, but is a major product of the pyrolysis of hexosans (Lomax *et al.*, 1991). For its structure, refer to Figure A2.1 in Appendix 2. A viscous oil is produced through the pyrolysis of cellulose (Prosen *et al.*, 1993), and therefore the processing of plant material (namely cellulose) in pottery vessels could lead to the formation of levoglucosan. This particular compound was found in three of the Meroitic sherds (Fig. 4.17), which were all from different contexts and were of different ware-types (2 were wheel-made wares, 1 was a hand-made ware). Two of the sherds contain fatty acids indicative of a predominantly animal origin, whereas the third is indicative of a predominantly plant origin. The presence of levoglucosan in these sherds cannot be the result of the firing of pottery vessels with organic temper at a lower than normal firing temperature, leading to incomplete combustion of the organic matter. Firstly, if this were the case, statistically this compound would be expected in more of the vessels; and secondly, since hand-made wares are fired at lower temperatures than wheel-made wares, these former ware types would be expected to contain relative more levoglucosan.

Therefore, the presence of levoglucosan in the three Meroitic sherds is most likely indicative of the heating in the vessels of large quantities of material containing cellulose. The fact that levoglucosan is detectable at all is indicative of the burial conditions in which they have been preserved, as mild acidic conditions will hydrolyse levoglucosan to glucose, which will be readily fermented (Prosen *et al.*, 1993), and it would be expected to be easily leached from the pottery vessels by the groundwater.

Although often in very low abundances, derivatives of benzoic acid were detected in five sherds. Some aromatic hydrocarbons have been detected in

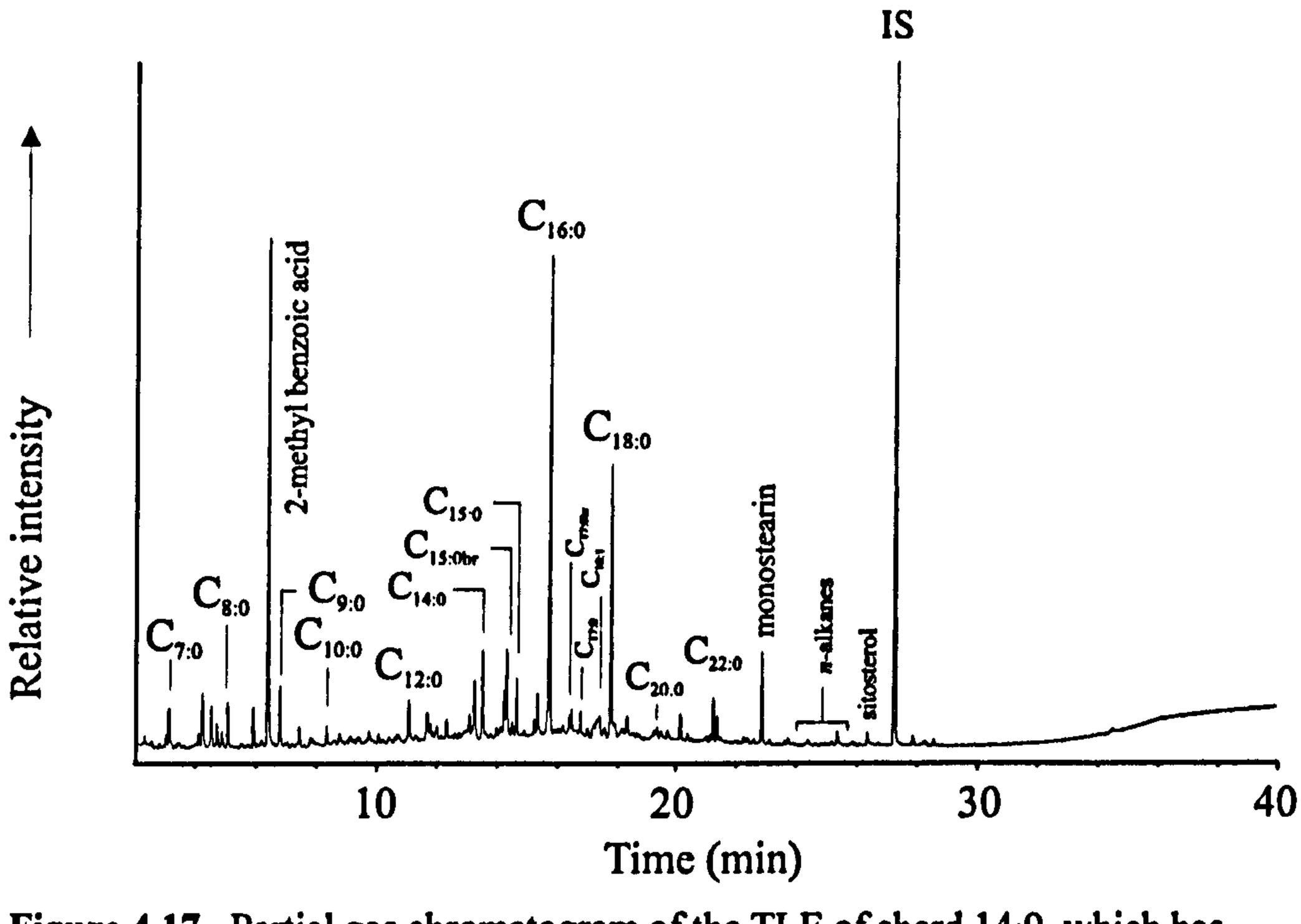

Figure 4.17 Partial gas chromatogram of the TLE of sherd 14·9, which has high abundances of derivatives of benzoic acid. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. IS denotes the internal standard (*n*-tetratriacontane). The $C_{16:0}/C_{18:0}$ abundance ratio, presence of *n*-alkanes, β-sitosterol and 2-methyl benzoic acid all suggest a higher plant origin for this lipid extract. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

higher plants (e.g. banana leaf), however, they are extremely rare in modern plant material (Kolattukudy, 1980:582). Although hydroxy benzoic acids have been previously detected in Egyptian mummies (Buckley & Evershed, 2001), it is known that 4-hydroxy benzoic acid is a degradation product of lignin (e.g. Maman *et al.*, 1996) and has been detected in lacustrine sediments (e.g. Stefanova and Disnar, 2000), and attributed to have been of lignin origin. Therefore, detection of these compounds in the sherds is a further indication of the processing of higher plants in the pottery vessels. A number of benzoic acid derivatives were detected in the sherds: 2-methylbenzoic acid (e.g. Fig. 4.17); 3- or 4-hydroxybenzoic acid and 3-methyl, 4-hydroxybenzoic acid. Sherd 14·9 (Fig. 4.17) not only yielded 2-methyl benzoic acid but also a homologous series of *n*-alkanes and β-sitosterol, which are all plant components.

4.2.4 Overall classification of the sherds from the Meroitic period

Classification of the sherds was completed using the plots of the $\Delta^{13}C$ v $C_{16:0}/C_{18:0}$ abundance ratios and the triangular graphs. Table 4.2 gives these classifications for the Meroitic sherds. The following conclusions can be drawn from the classification of the sherds, based on lipid content: (i) 51% of the vessels that exhibited extractable lipids contain mainly animal fats in them. (ii) This contrasts significantly with the 22% that have predominantly plant lipids (this figure includes the 7% of the sherds that contain characteristic palm kernel lipids). (iii) After classification of the extracts from the sherds as either being 'predominantly cattle' or 'predominantly sheep/goat', it was observed that the incidence of sheep/goat fats was approximately 2.3 times that of the cattle fats. (iv) A total of 14 of the vessels (24%) showed direct evidence for the mixing of plant material and animal products through the detection of diagnostic plant compounds. This mixing may have occurred at the same time during use, or through multiple re-use of the vessel.

Table 4.2 Classification of the Meroitic vessels based on lipid content[1]

Context	*Predominately Cattle*[2]	*Predominately Sheep/goat*[2]	*Mixed animal*[2]	*Possibly sheep/goat*[2]	*Cattle?*[2]	**Predominately Animal**[2]	**Mixed animal/plant**[2,3]	**Predominately Plant**	**Palm kernel**
18002	*0*	*0*	*1*	*0*	*0*	1	0	0	0
18010	*0*	*0*	*0*	*1*	*0*	1	1	0	0
18013	*3 (2)*	*6 (2)*	*2 (1)*	*0*	*2*	13 (5)	3 (1)	4	0
18014	*2 (1)*	*3 (3)*	*2 (2)*	*0*	*0*	7 (6)	1 (1)	1	0
18023	*1*	*3*	*1*	*0*	*0*	5	6 (1)	3	3
18031	*0*	*1*	*0*	*0*	*0*	1	0	0	0
18036	*0*	*0*	*1*	*0*	*0*	1	1	1	1
ML	*0*	*1*	*0*	*0*	*0*	1	0	0	0
TOTAL	*6 (3)*	*14 (5)*	*7 (3)*	*1*	*2*	**30 (11)**	**12 (3)**	**9**	**4**
(%)	*10(5)*	*24(9)*	*12(5)*	*2*	*3*	**51 (19)**	**20 (5)**	**15**	**7**

[1]The values not in italics are the final classifications of the sherds, those in italics represent the prevalence of specific animal fats (c.f. Section 3.3)

[2]The numbers in brackets indicate the number and percentage of sherds that contained traces of other diagnostic plant lipids as well as fatty acid distributions that are indicative of animal fats

[3]Vessels plotting above the ovi-caprid/plant ellipses as discussed in Section 4.4.3

4.3 THE EARLY POST-MEROITIC POTTERY VESSELS

The majority of the sherds from the Early Post-Meroitic (EPM) contexts yielded lipid distributions that were remarkably different to those from the Meroitic period. Figure 4.18 shows two typical chromatograms of the TLEs obtained from EPM pottery. The lipid distribution exhibited in sherd 3·10 is indicative of a plant origin, due to the high abundance of $C_{16:0}$ relative to the $C_{18:0}$ component, whereas sherd 9·7 contains lipids whose distribution is diagnostic of palm kernel lipids, characterised by high abundances of $C_{12:0}$ and $C_{14:0}$ and low abundances of $C_{18:0}$. The exceptional preservation of lipids in the pottery from Qasr Ibrim is illustrated by the fact that a total of 85 sherds out of 94 (93%) contained significant concentrations of lipid (i.e. >5 $\mu g\ g^{-1}$).

4.3.1 Fatty acid distributions

The triangular plot of all of the sherds from this period is displayed in Figure 4.19. The graph is dominated by extracts containing high relative abundances of $C_{12:0}$, varying $C_{16:0}$ and low abundances of $C_{18:0}$, and hence plot to the left of the triangular diagram; this fatty acid composition is indicative of a palm kernel source for the majority of the sherds. In fact, upon close inspection of the fatty acid distributions, it is apparent that a total of 64 sherds yielded characteristic palm kernel lipids. A further 21 sherds contain lipids that do not exhibit a palm fruit origin; these can be seen to plot on the bottom right-hand side of the triangular plot in Figure 4.19.

After the sherds are grouped according to context number (Fig. 4.20), some interesting differences emerge. Context 18009 is almost completely dominated by palm kernel lipids. In this context, 25 out of 26 (96%) of the sherds that contain lipids, yield fatty acid distributions indicative of a palm kernel origin. Similar results were also detected for the sherds excavated from contexts 18022 (28/29; 97%) and 18024 (8/9; 89%).

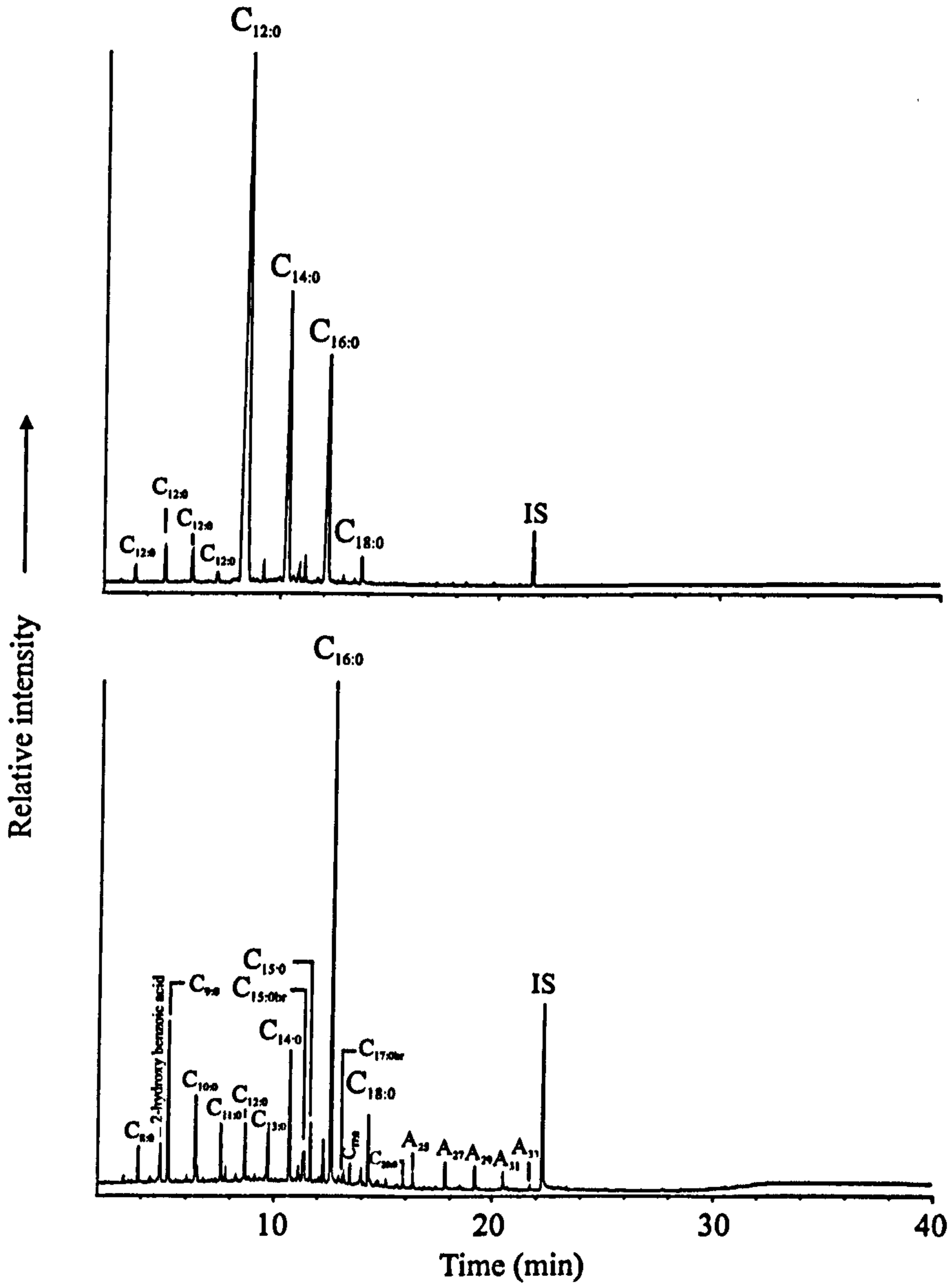

Figure 4.18 Partial gas chromatograms of the TLE of two typical Early Post-Meroitic vessels. Sherds 9·7 (top) is indicative of a palm kernel source, and sherd 3·10 (bottom) is indicative of a plant source. $C_{x:y}$ represents fatty acids of carbon chain length x and level of unsaturation y. A_z represents *n*-alkanes of carbon chain length z. IS denotes the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

Such a high proportion of sherds that contain these characteristic palm kernel lipids is in complete contrast to the other contexts from this period which are dominated by animal/plant lipids. For example, there is a tight cluster of sherds

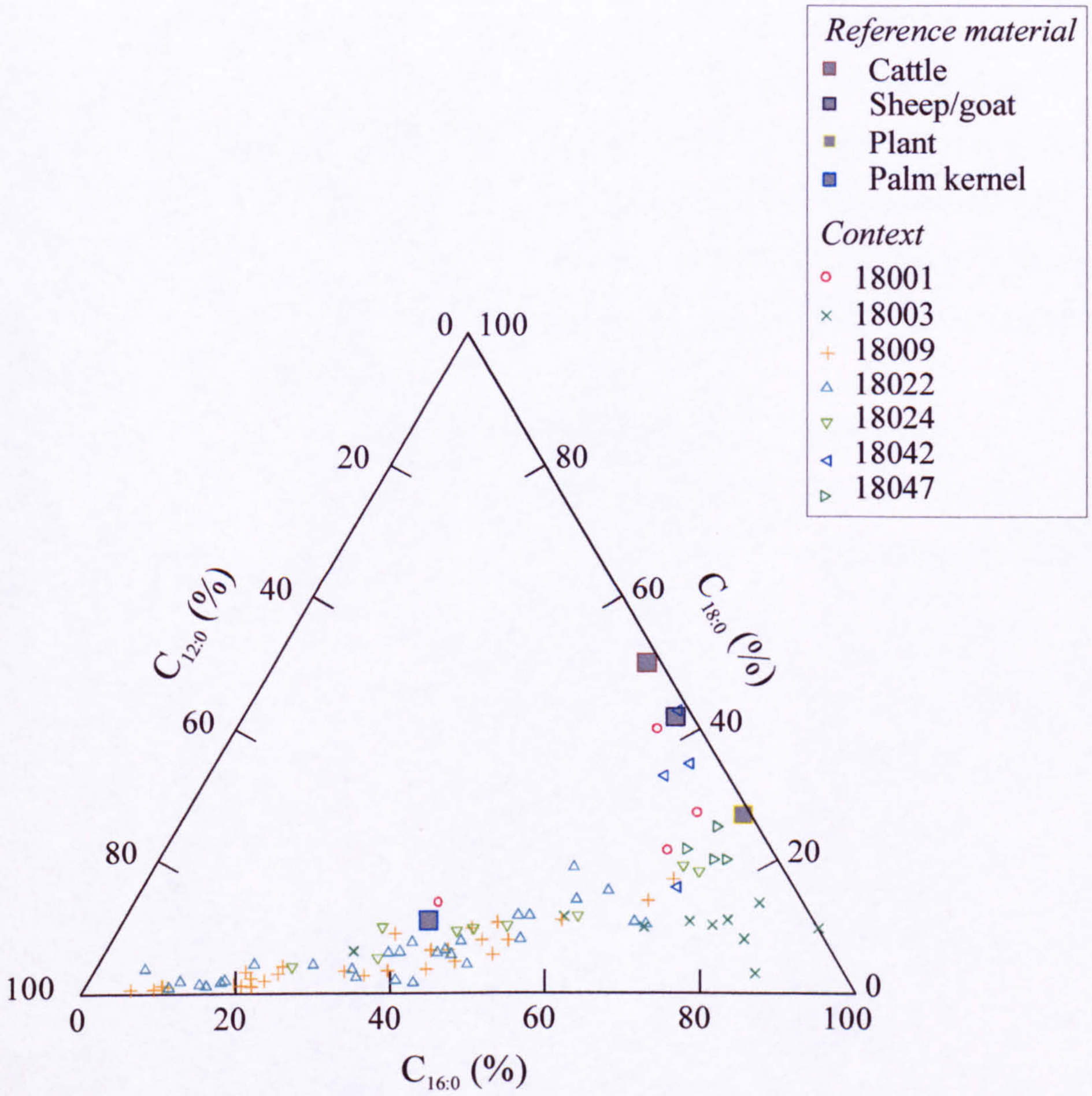

Figure 4.20 Triangular plot of the Early Post-Meroitic vessels, by context number. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

A further interesting point concerning these vessels is that there appears to be a difference in the composition of lipids extracted from the hand-made wares and wheel-made wares which may relate to their use (Fig. 4.21). Only one out of nine (11%) of the H1 wares display a fatty acid distribution indicative of palm kernel lipids. The remainder (89%) exhibit distributions that are indicative of predominantly animal fats and/or predominantly plant lipids. In sharp contrast, 83% of the wheel-made wares (i.e. ware types R1, R25 and R32) contain a palm kernel lipid distribution.

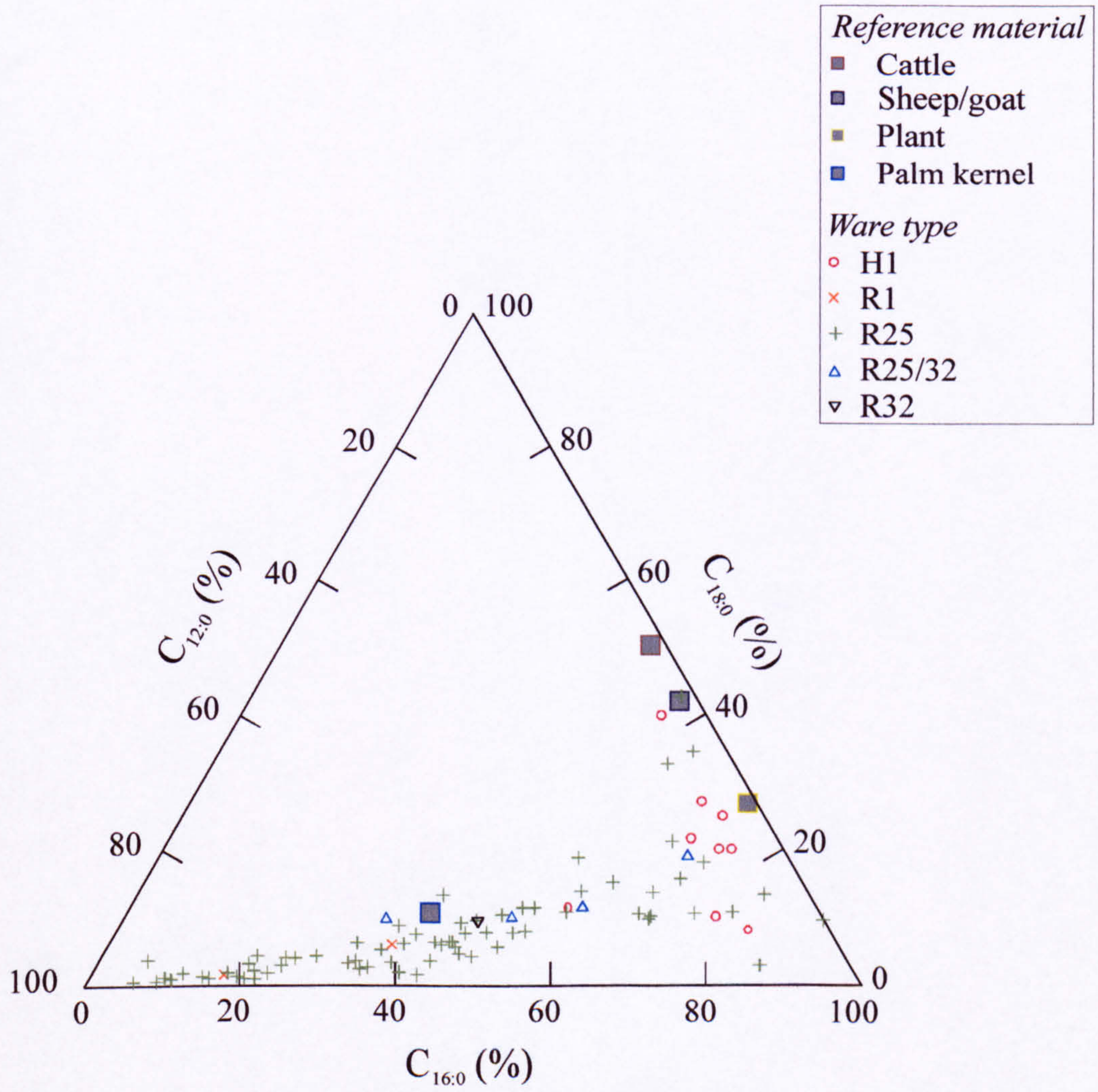

Figure 4.21 Triangular plot of the Early Post-Meroitic vessels, by ware type. The coloured boxes represent the mean values for the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

Interestingly, a total of 29 (45%) of the sherds containing palm kernel lipids also yielded other diagnostic lipids (Fig. 4.22). Twenty-seven of these latter sherds contain *n*-alkane distributions indicative of higher plants. These *n*-alkanes were always found in low abundances, in the C_{27} - C_{33} range with the odd-chain homologues predominating, and usually maximising at C_{29} or C_{31}. The *n*-alkane distributions for each of the sherds are shown at the end of this section, and discussed in detail in Section 4.5.

Derivatives of benzoic acid were detected in two of the sherds (3·10 and 9·7), and in both cases in the form of 4-hydroxy benzoic acid, a known degradation product of lignin (Maman *et al.*, 1996).

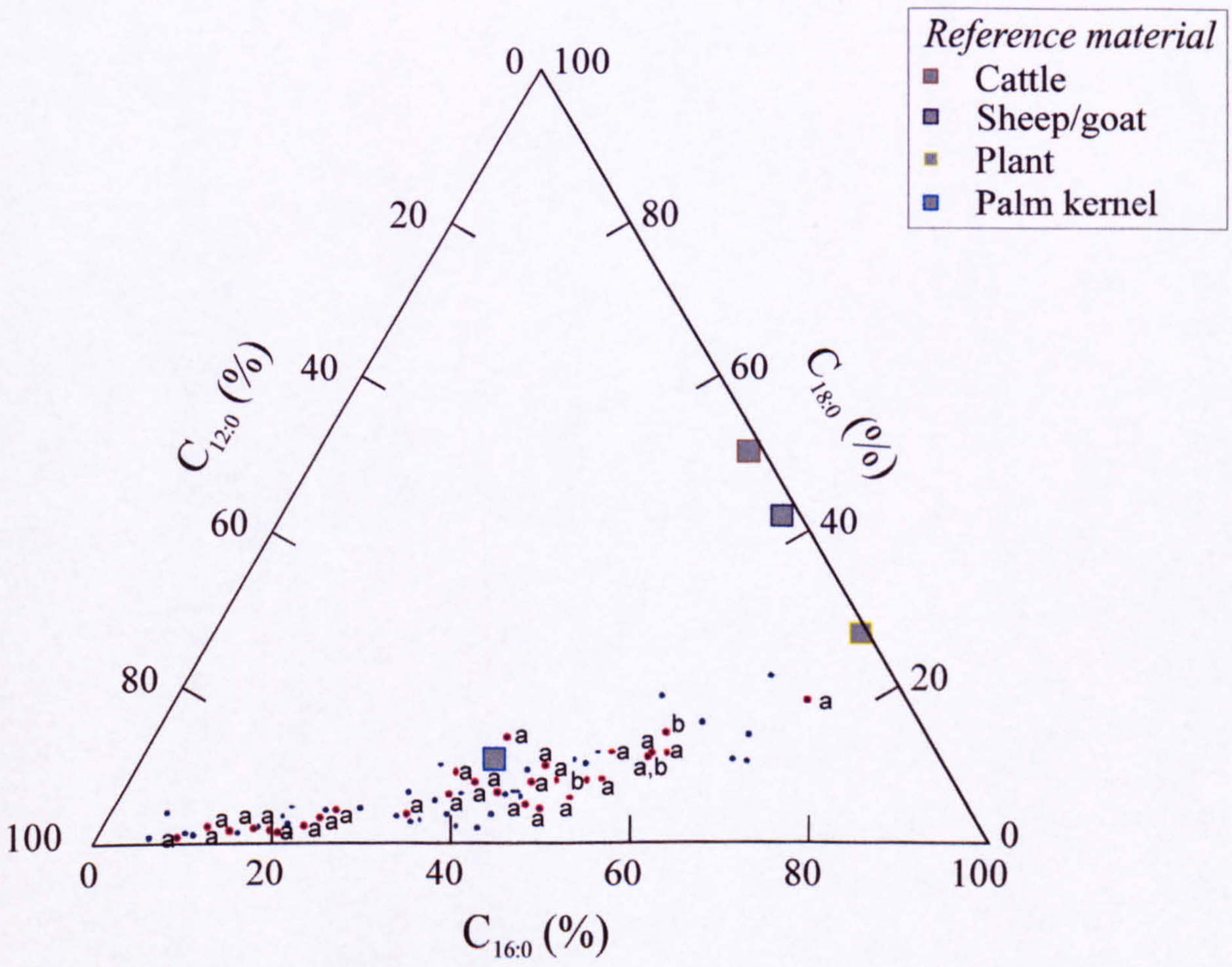

Figure 4.22 Triangular plot of the 'palm kernel' vessels from the Early Post-Meroitic period, with labelled diagnostic compounds. 'a' denotes *n*-alkanes; and 'b' denotes 4-hydroxybenzoic acid. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

4.3.2 Fatty acid stable isotopes ($\delta^{13}C$) values

The sherds that yielded typical palm kernel fatty acid distributions were removed from the data set and analysed separately (Section 4.7). The $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values of the remaining 21 sherds are plotted against their $C_{16:0}/C_{18:0}$ abundance ratios in order to classify the extracts according to their origin (Fig. 4.23). It can be seen that very few of the sherds contain predominantly animal fats as they would plot to the left of the graph. Only three sherds (4%) actually plot within the ellipses associated with animal fats,

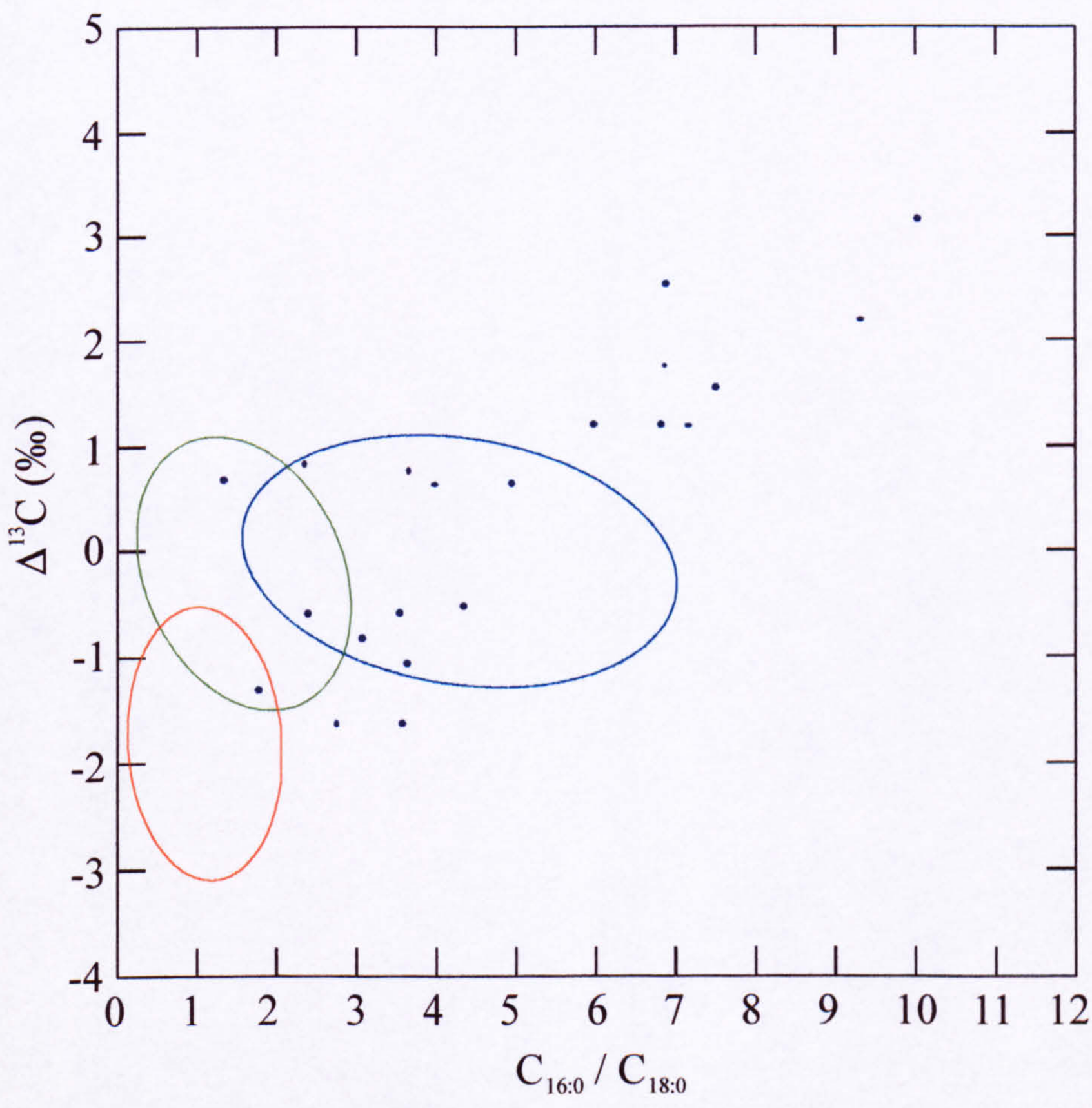

Figure 4.23 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Early Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

with one extract indicative of a predominantly sheep/goat fat, and the other two indicative of animal fats or mixed animal/plant products. Although some of the remaining eighteen sherds do not plot within the confidence ellipse generated from the plant reference materials, given their relatively high $C_{16:0}/C_{18:0}$ abundance ratios, these extracts too are likely to originate from a plant source.

Figure 4.24 shows that when the context numbers are considered, certain clusters of sherds become evident. All nine sherds from context 18003 yielded

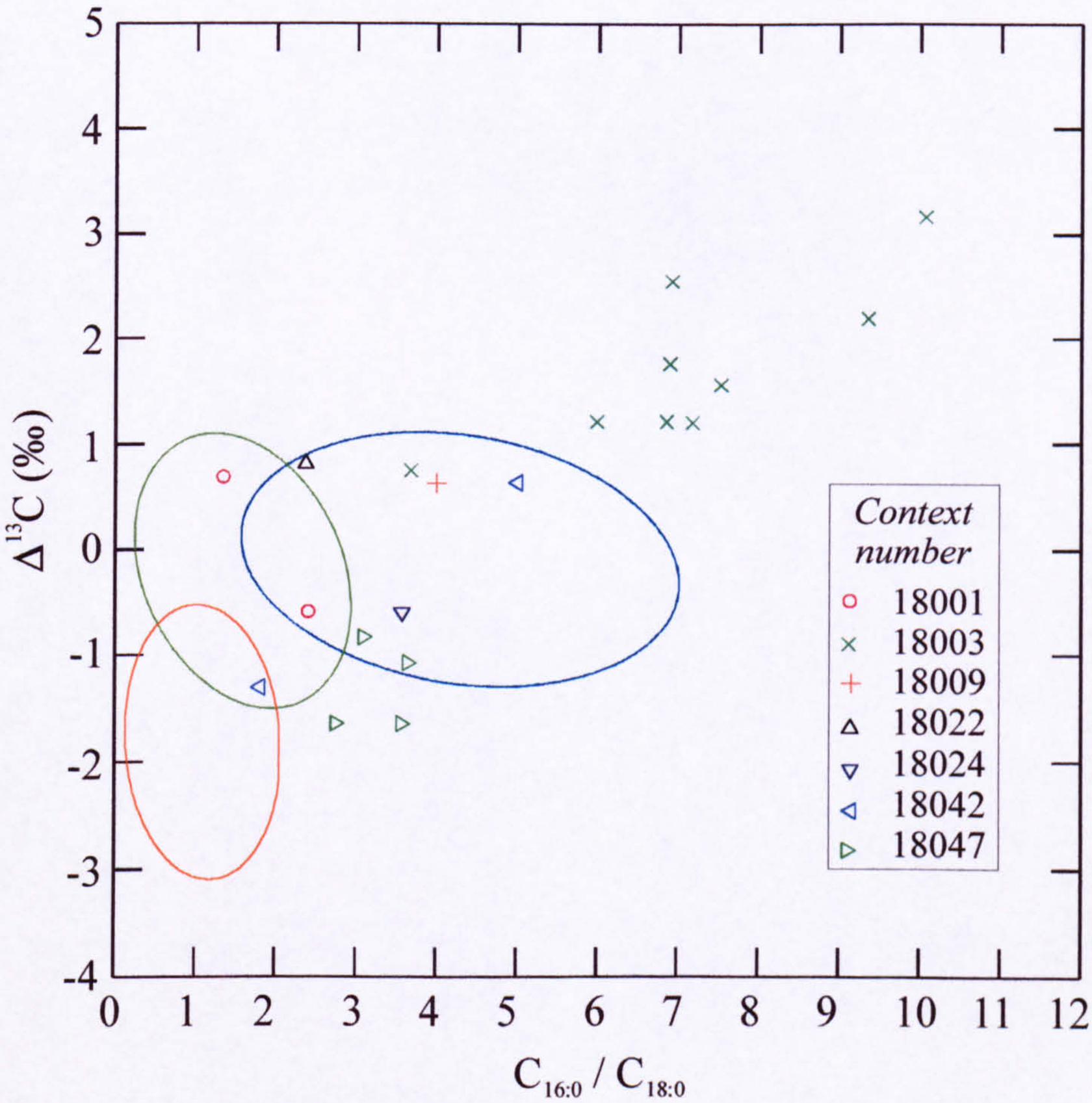

Figure 4.24 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Early Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by context number. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

much higher abundances of the $C_{16:0}$ than $C_{18:0}$, and for the majority of the sherds (8/9), their $\Delta^{13}C$ values are within the unusual range of 1.2 to 3.2 ‰ (Fig. 4.24). Furthermore, the four sherds from 18047 cluster together at $C_{16:0}/C_{18:0} \approx 3.3$, indicating the possibility that these are from the same origin.

When the actual $\delta^{13}C$ values for the fatty acids from the sherds are investigated (Fig. 4.25), in all but one of the sherds, they are within the C_3 range. The sherd with the most enriched fatty acid $\delta^{13}C$ value exhibits a $\delta^{13}C_{16:0}$ value of -23.2‰, and a $\delta^{13}C_{18:0}$ value of -22.6‰, this is most likely to represent the fat from an animal (sheep/goat) whose diet consisted of a mixture of both C_3 and C_4 plants. For the other sherds, the fatty acids display more depleted $\delta^{13}C$ values, and due to the spread of $\delta^{13}C$ values (shown in Fig. 4.25), it is likely that this represents the mixing of fatty acids of different C_3 isotope values.

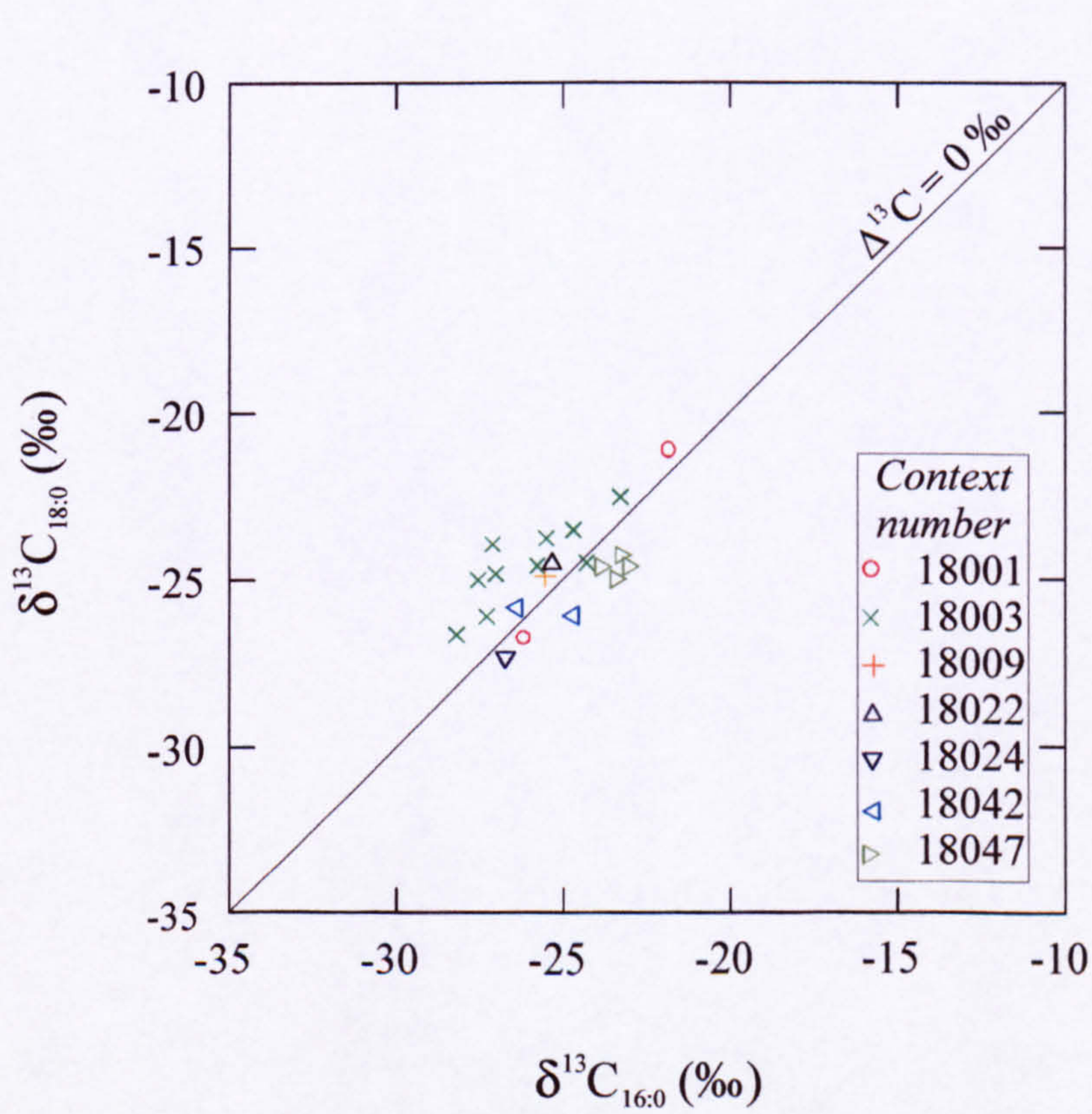

Figure 4.25 The $\delta^{13}C$ values of the major fatty acids extracted from the EPM period sherds.

The four sherds from 18047 plot very closely to each other in Figure 4.25, however it is not likely that the same commodity was actually being processed in all of these vessels. Their $C_{16:0}/C_{18:0}$ abundance ratios vary from 2.8 to 3.7, and as their $\delta^{13}C$ values are also variable, suggesting that these lipids originated from different plants.

Although differences in the use of wheel-made and hand-made wares were observed between the sherds containing palm kernel lipids and those containing animal/plant products (Fig. 4.21), no such distinction in vessel use was detected between the sherds predominantly used to process animal products or plant products (Fig. 4.26). Both the hand-made and wheel-made wares plot within

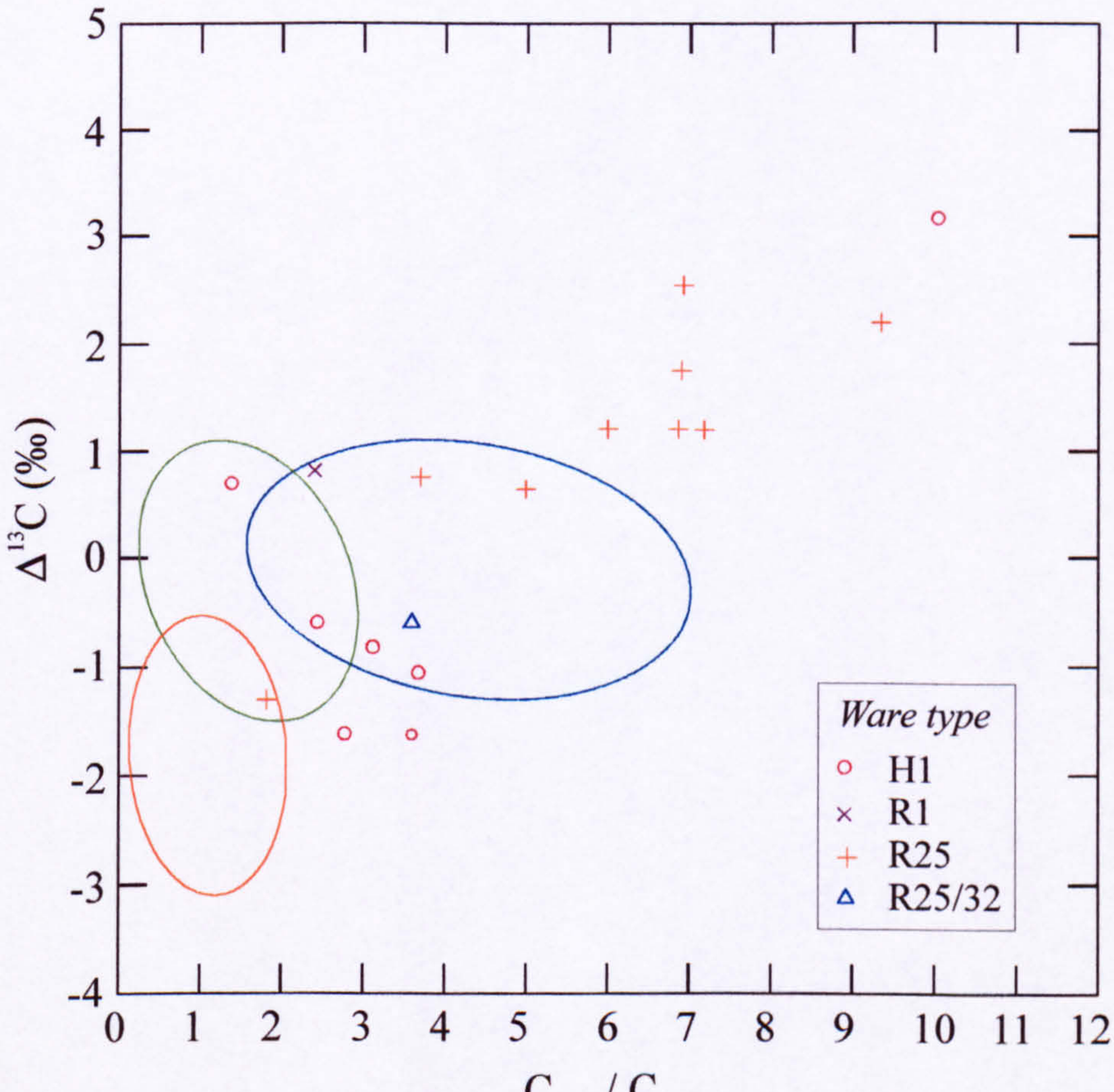

Figure 4.26 $\Delta^{13}C$ ($\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) of the Early Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by ware type. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

the predominantly sheep/goat field as well as within the predominantly plant and mixed plant/animal fields.

4.3.3 Other diagnostic compounds

Figure 4.27 indicates the other characteristic compounds that were detected in the sherds from the EPM period. Six out of the twenty-one sherds (29%) contain other diagnostic compounds. *n*-Alkanes, which are indicative of higher plant epicuticular waxes, were detected in all six of these sherds. In total (i.e. including the sherds containing *n*-alkanes shown in Figure 4.22), 24 sherds

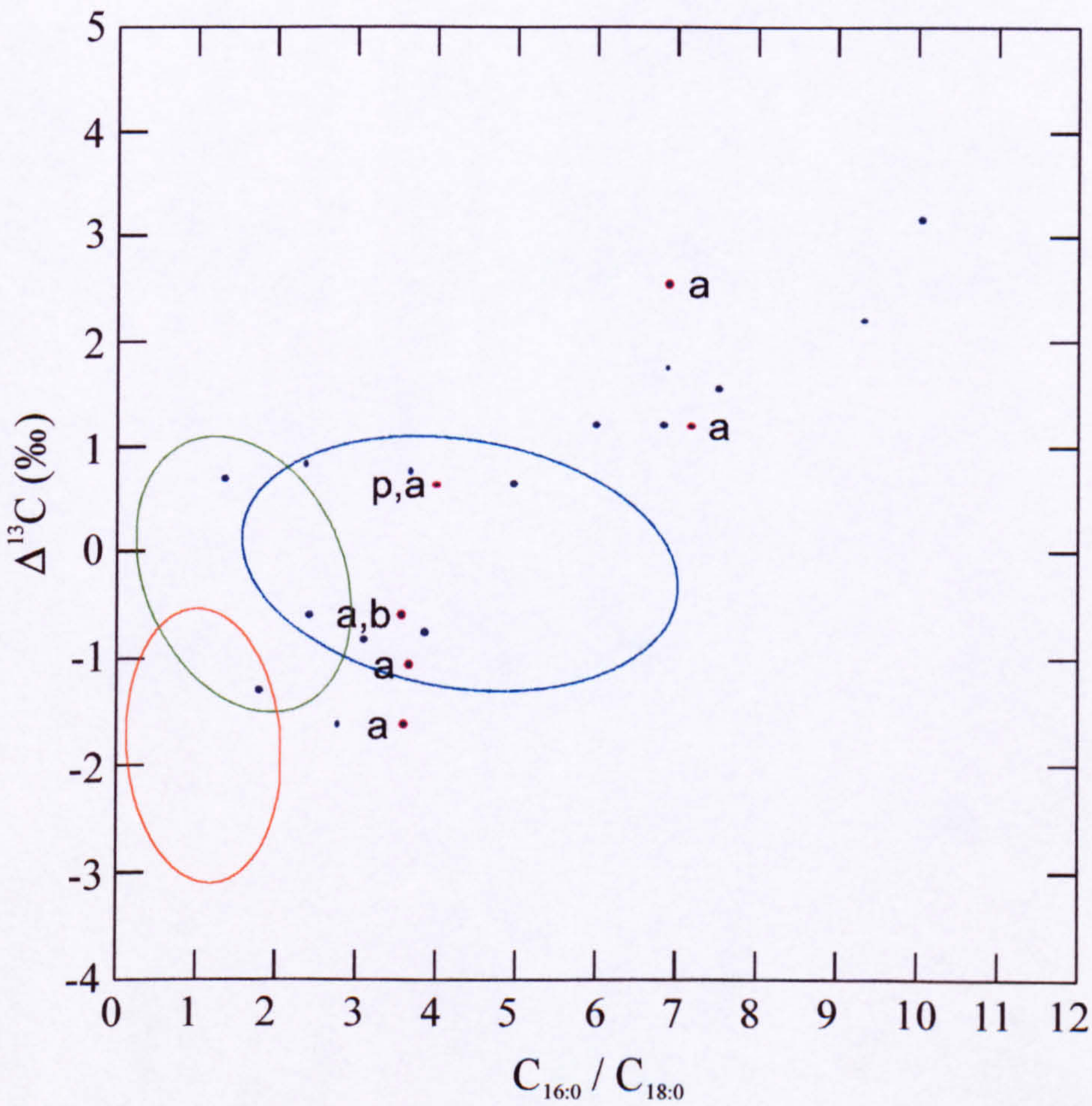

Figure 4.27 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Early Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, with labelled diagnostic compounds. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3). Key: 'a': *n*-alkanes, 'b': benzoic acid derivatives, and 'p': plant sterols.

from the EPM period contained *n*-alkanes, with the C_{27} to C_{31} homologues predominating (Fig. 4.28). These *n*-alkane distributions are discussed in detail in Section 4.5. In addition, one sherd (24·5) also yielded both 3-hydroxy and 4-hydroxy benzoic acid, and in a further sherd (9·27) β-sitosterol was also detected. All of the sherds with these compounds plot within the fields of the graph indicative of a predominantly plant origin for the extracts, i.e. with a high $C_{16:0}/C_{18:0}$ abundance ratio. None of the sherds that exhibit fatty acid distributions typical of an animal fat also contain any of these diagnostic plant compounds, indicating that the mixing of animal products

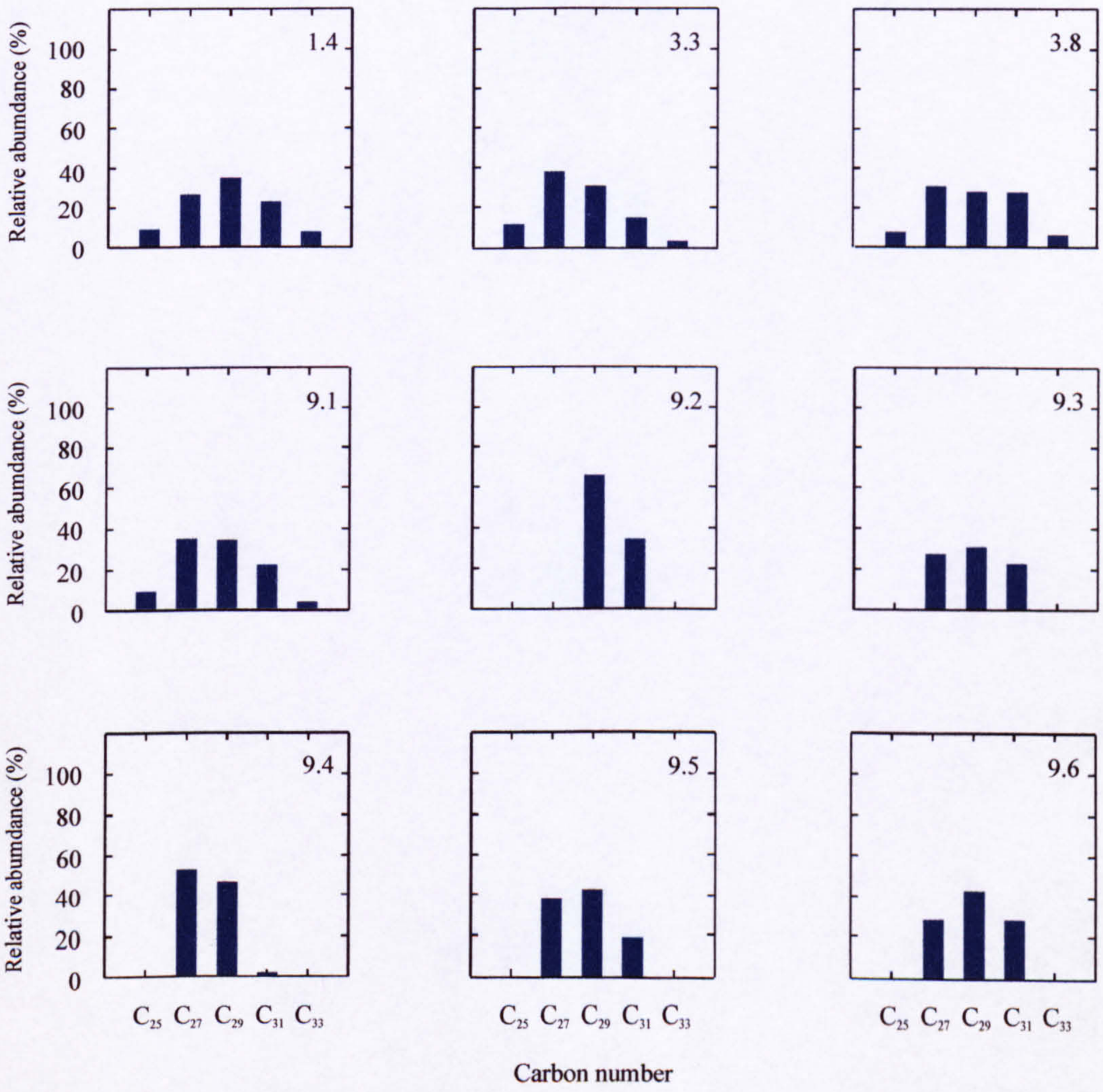

Figure 4.28 The *n*-alkane distributions extracted from the Early Post-Meroitic sherds. Only the sherds that yielded significant abundances of *n*-alkanes are shown. The sherd numbers are shown in the top right corner. C_X refers to *n*-alkanes of carbon number x.

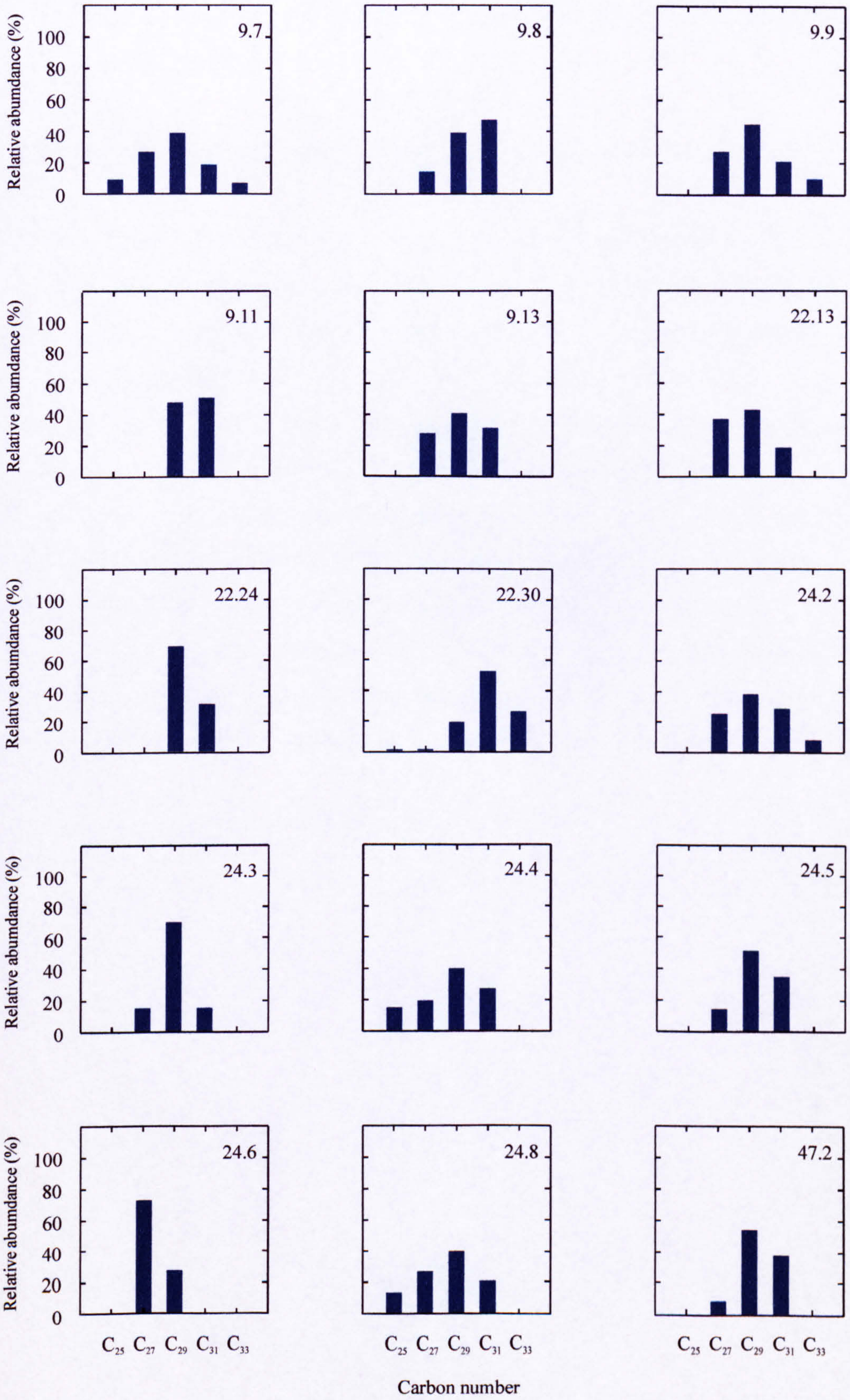

Figure 4.28 cont. The *n*-alkane distributions extracted from the Early Post-Meroitic sherds. Only the sherds that yielded significant abundances of *n*-alkanes are shown. The sherd numbers are shown in the top right corner. C_X refers to *n*-alkanes of carbon number x.

with low abundances of plant material is unlikely to have occurred to any extent during the EPM period.

4.3.4 Overall classification of the sherds from the Early Post-Meroitic period

After classification of the sherds to commodity group (utilising their $\Delta^{13}C$ values, $C_{16:0}/C_{18:0}$ abundance ratios and fatty acid distributions), the following conclusions can be drawn: (i) The vessels from the EPM period showed an overwhelming bias towards being used in the processing of palm fruit. Approximately 75% of the sherds that yielded significant concentrations of lipid (>5 $\mu g\ g^{-1}$) from this period exhibit a characteristic palm kernel lipid distribution. (ii) Table 4.3 also illustrates the fact that contexts 18009, 18022 and 18024 account for 61/64 (95%) of the sherds that contain characteristic palm kernel lipids. (iii) A further 21% of the sherds contain fatty acid distributions indicative of a plant origin. (iv) Only 5% of the sherds contain fatty acids indicative of a predominantly animal origin. Table 4.3 shows the final classification of the sherds from the Early-Post Meroitic period.

Table 4.3 Classification of the Early Post-Meroitic vessels based on lipid content[1]

Context	*Predominately Cattle*	*Predominately Sheep/goat*	*Predominately Mixed animal*	*Cattle?*	**Predominately Animal**	**Mixed animal/plant**	**Predominately Plant**	**Palm kernel**
18001	*0*	*1*	*0*	*0*	1	1	0	2
18003	*0*	*0*	*0*	*0*	0	0	9	0
18009	*0*	*0*	*0*	*0*	0	0	1	25
18022	*0*	*0*	*0*	*0*	0	0	1	28
18024	*0*	*0*	*0*	*0*	0	0	1	8
18042	*0*	*0*	*1*	*0*	1	0	2	1
18047	*0*	*0*	*0*	*0*		0	4	0
TOTAL	*0*	*1*	*1*	*0*	**2**	**1**	**18**	**64**
(%)	*0*	*1*	*1*	*0*	**2**	**1**	**21**	**75**

[1]The values not in italics are the final classifications of the potsherds, those in italics represent the prevalence of specific animal fats.

4.4 THE POST-MEROITIC VESSELS

The sherds from the Post-Meroitic period were excavated from two contexts (10107 and 10110) in structure 265. Generally, the lipid distributions of the extracts are similar to those seen in the Meroitic period, with $C_{16:0}$ and $C_{18:0}$ predominating. Figure 4.29 shows the chromatograms of the TLEs of two typical sherds from this period. The top chromatogram is indicative of an animal fat mixed with some plant lipids (as indicated by the high relative abundances of $C_{18:0}$ and the presence of *n*-alkanes). The bottom chromatogram is indicative of a plant origin for the extract, witnessed not only through the high relative abundances of the $C_{16:0}$ component, but also the presence of *n*-alkanes and derivatives of benzoic acid (see Section 4.4.3). Sixty-nine out of 99 of the sherds (70%) contained significant concentrations of extractable lipid (> 5 $\mu g\ g^{-1}$), thus enabling their fatty acid distributions to be determined.

4.4.1 Fatty acid distributions

The majority of the sherds exhibit typical animal/plant lipid distributions, and only a few of the sherds contain lipids characteristic of a palm kernel origin. This can be seen in Figure 4.30 in which the majority of the sherds plot close to the y-axis, indicating the relatively low abundances of $C_{12:0}$ in these sherds. The four sherds that plot towards the centre of the triangular plot have approximately equal relative abundances of $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$. They are therefore indicative of either the processing of palm fruit, mixed with another commodity, or the processing of palm fruit accompanied by a preferential loss of the shorter-chain $C_{12:0}$ and $C_{14:0}$ fatty acids during vessel use or burial. In fact, on close examination of the saturated fatty acid distributions, eleven out of 69 of the sherds (16%) display fatty acid compositions indicative of a palm kernel origin.

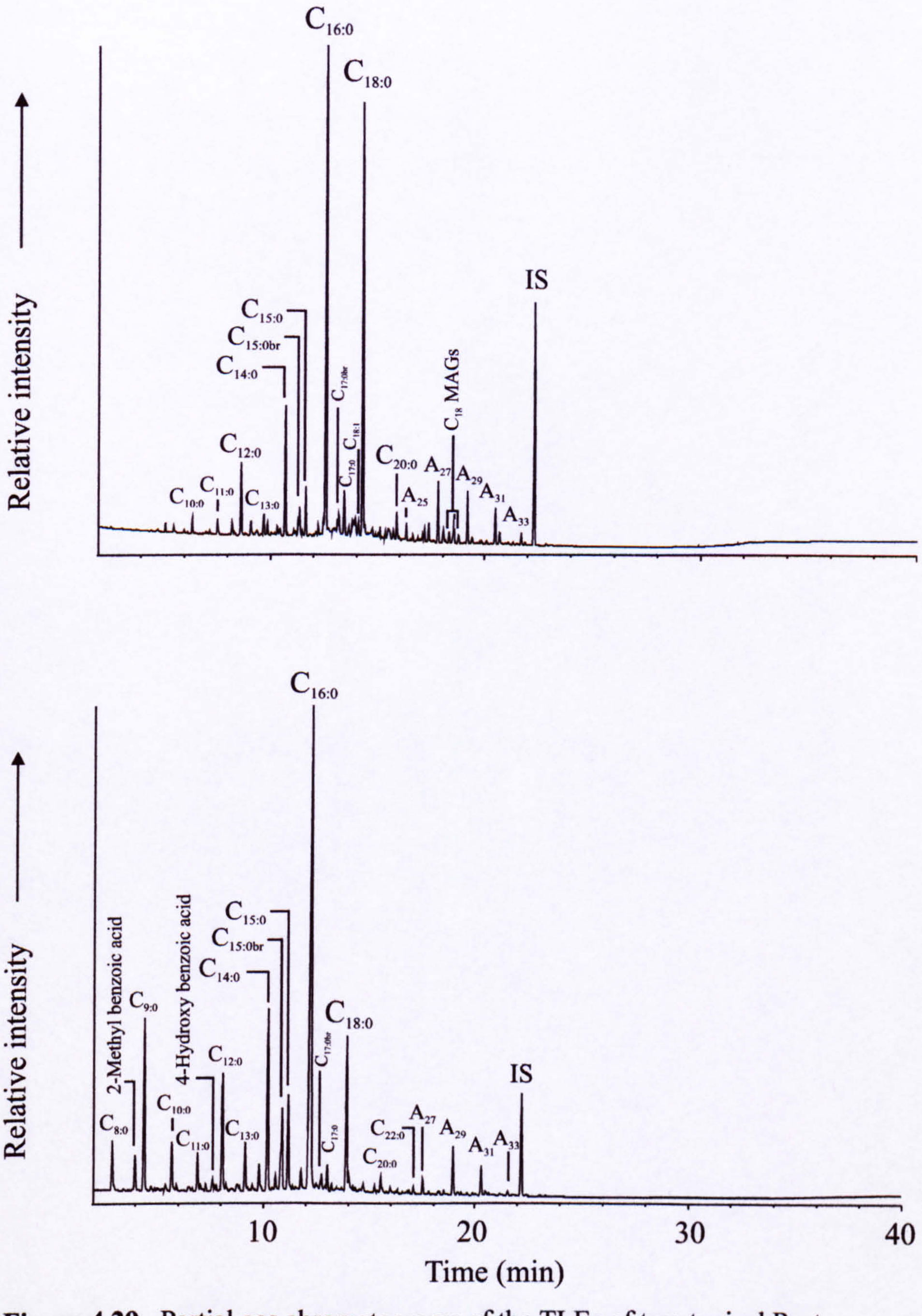

Figure 4.29 Partial gas chromatograms of the TLEs of two typical Post-Meroitic vessels. In sherd 107·16 (top) the ratio of $C_{16:0}$ to $C_{18:0}$ is indicative of either animal fats or a mixture of animal and plant lipids, whereas for 110·15 (bottom) it is indicative of a plant source. $C_{x:y}$ represents fatty acids of carbon chain length x and level of unsaturation y. C_{18} MAGs represent Sn-1,3 and Sn-2 monostearin. A_z represents *n*-alkanes of carbon chain length z. IS denotes the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness)was used.

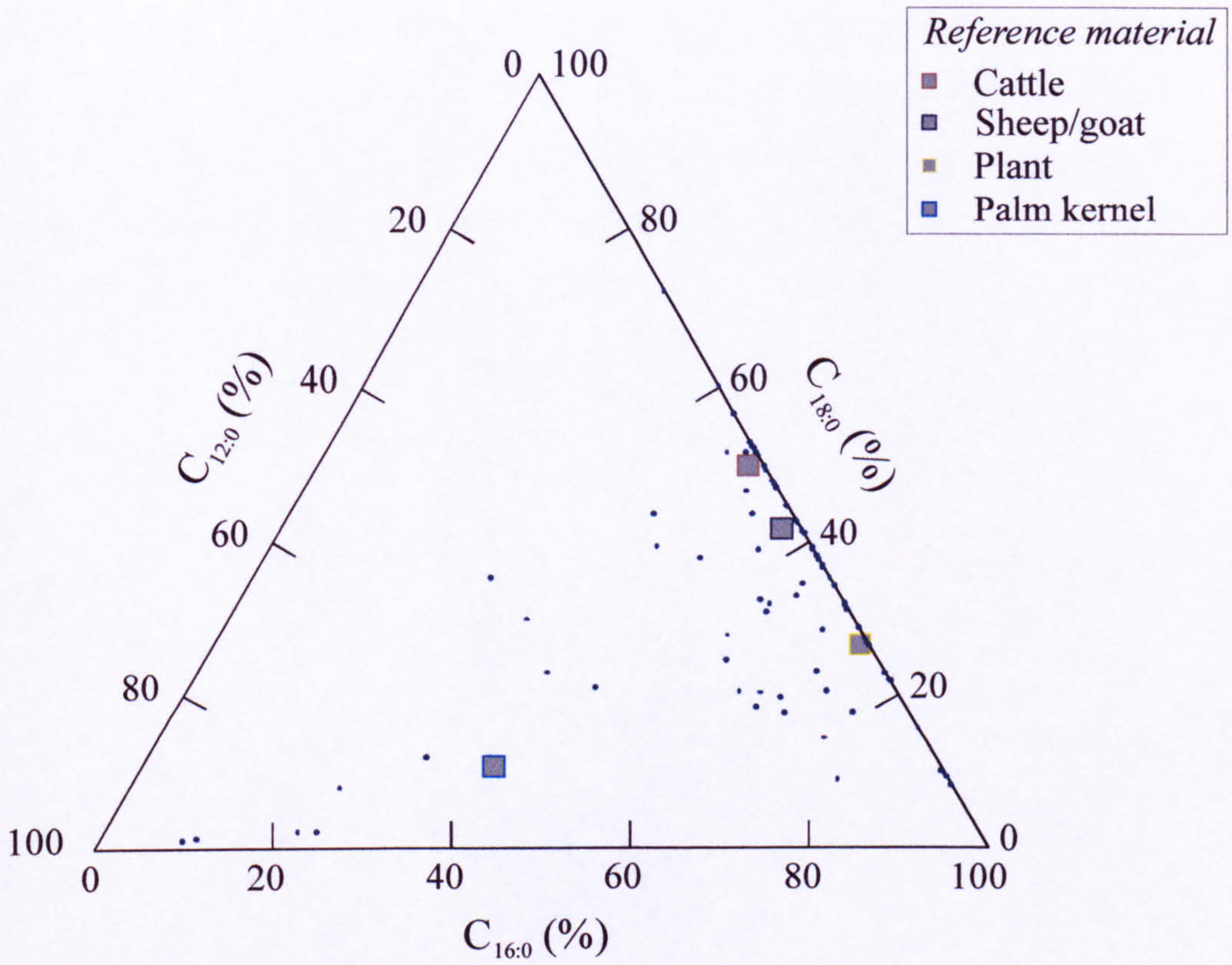

Figure 4.30 Triangular plot of all the Post-Meroitic vessels. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

A further point worthy of attention is that eight out of eleven (73%) of the sherds that display distributions indicative of a palm kernel origin were from context 10110 (Fig. 4.31); the remaining sherds from this context and the majority of those from context 10107 plot relatively uniformly in the plant/animal region of the triangular plot.

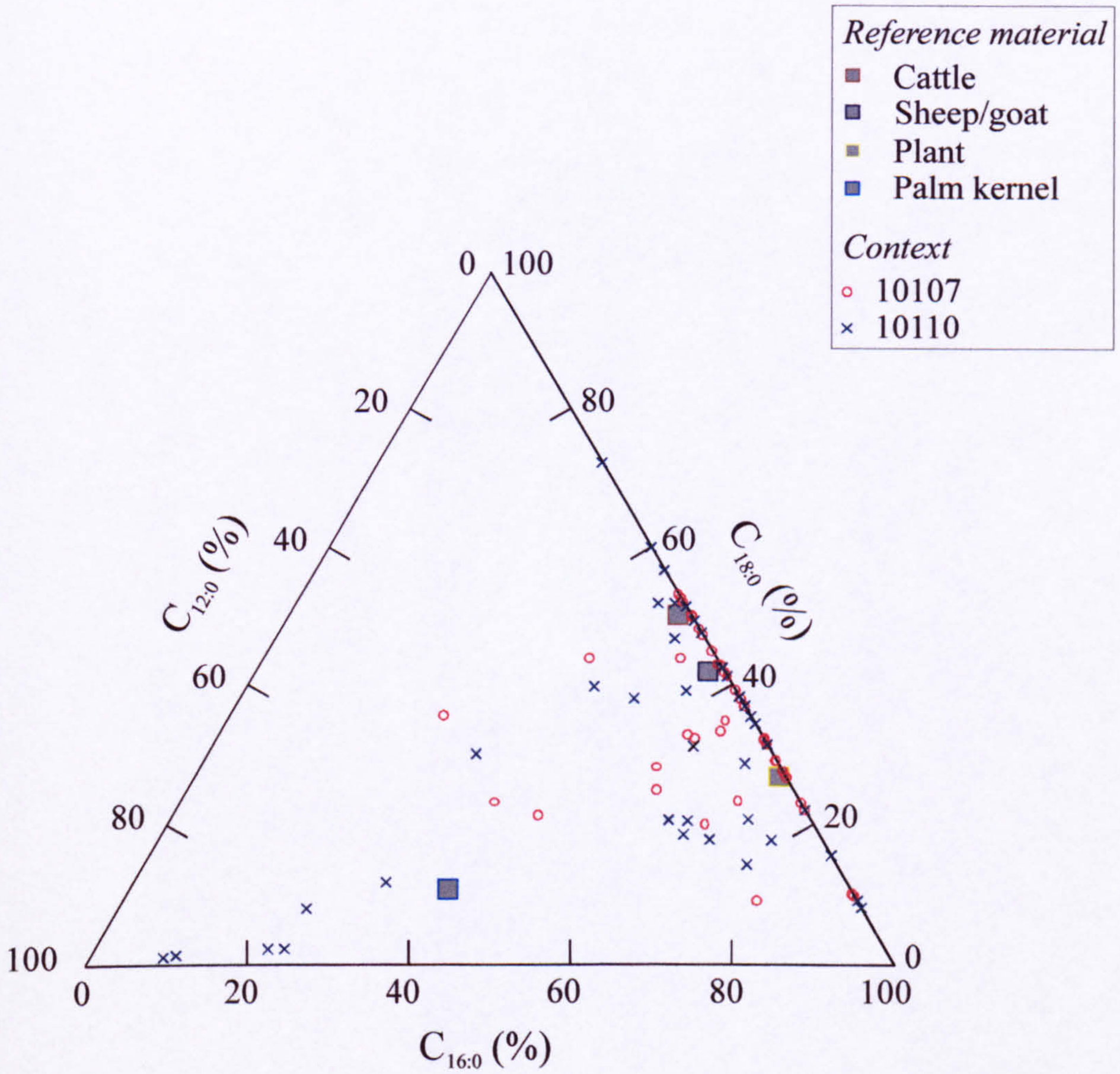

Figure 4.31 Triangular plot of the Post-Meroitic vessels, by context number. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

Differences in the use of specific ware types is illustrated in Figure 4.32, which plots the sherds from the EPM period, categorised by ware type. Out of all of the sherds that plot within the palm kernel region of the graph, only one was a hand-made ware (8%); all of the rest were wheel-made wares. This illustrates the preference for the use of wheel-made wares in the processing of palm fruit. No such difference was observed for the rest of the sherds, as these are uniformly spread across the areas of the graph associated with animal and plant products.

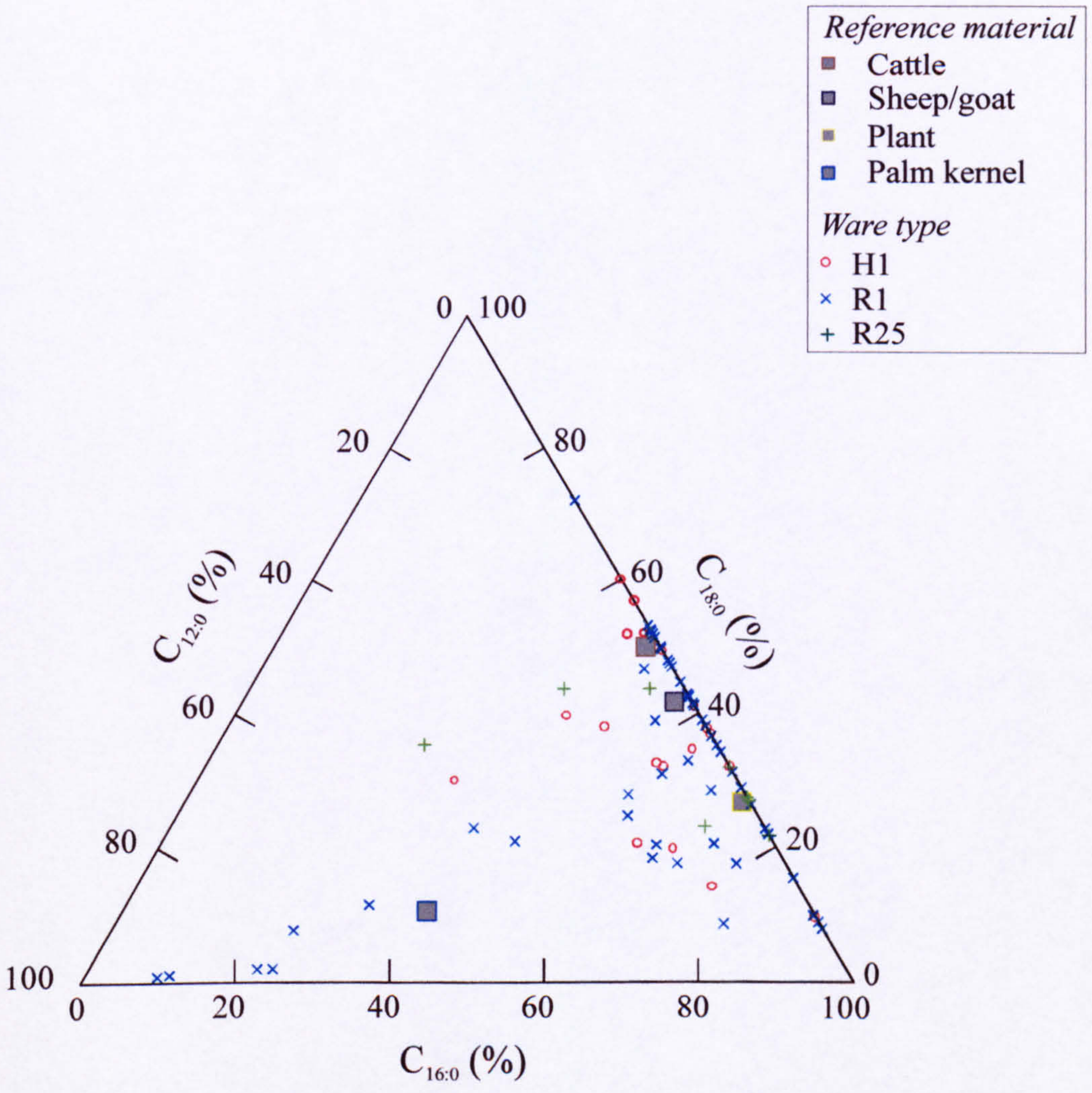

Figure 4.32 Triangular plot of Post-Meroitic vessels by ware type. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

4.4.2 Fatty acid stable isotopes ($\delta^{13}C$) values

The sherds that exhibited characteristic palm kernel lipid distributions were removed from the data set for later data treatments (Section 4.7). The $\Delta^{13}C$ values of the remaining sherds are plotted against their $C_{16:0}/C_{18:0}$ abundance ratios (Fig. 4.33). Thirty-one (45%) of the sherds plot within the fields that are indicative of predominantly animal fats, whereas 17 (25%) plot within or close to the ellipse associated with predominantly plant material. Four of these

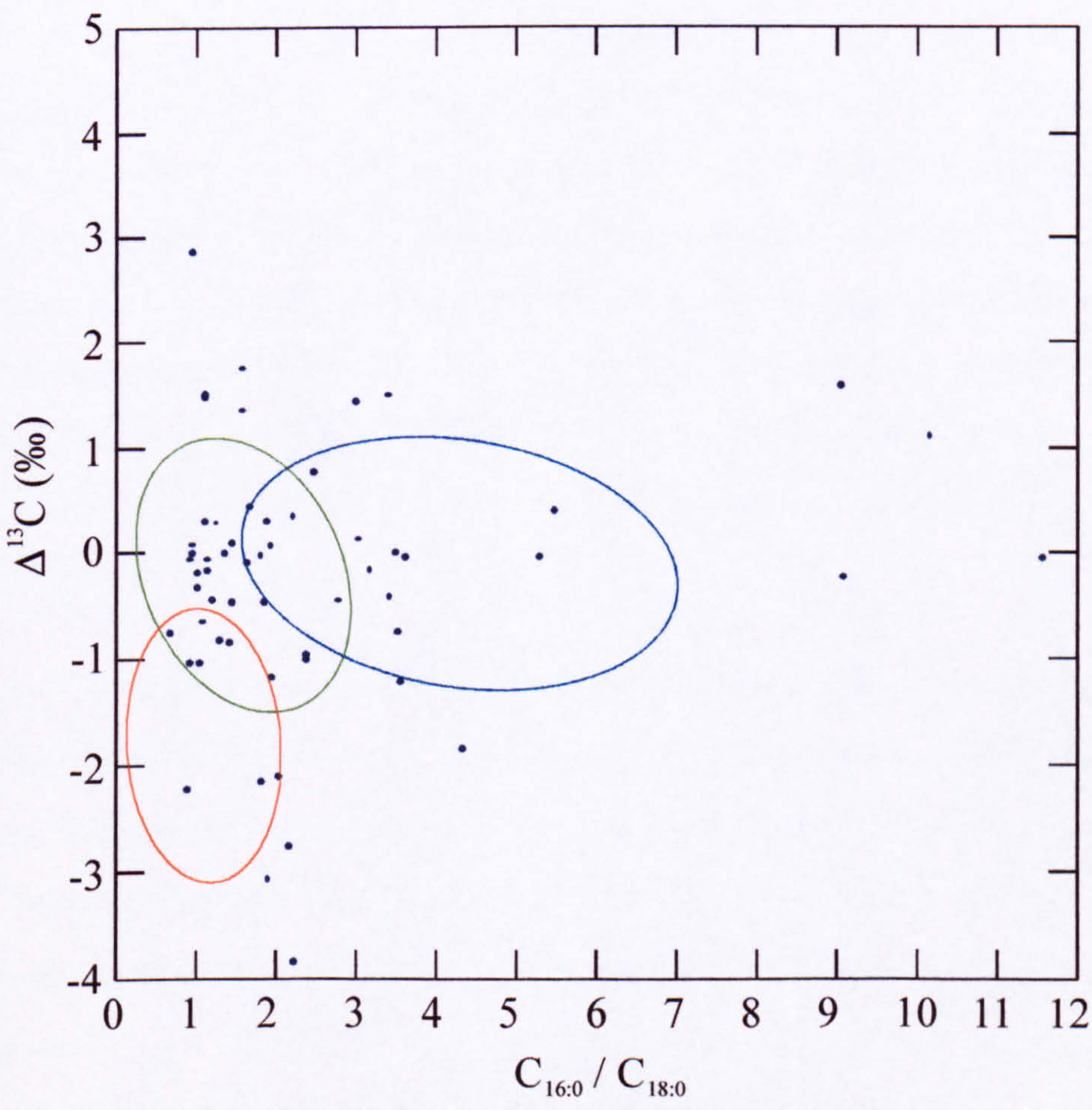

Figure 4.33 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

latter sherds have $C_{16:0}/C_{18:0}$ abundance ratios greater than 8, and appear as a distinct group in the bottom right-hand corner of Figure 4.32, at $C_{18:0} \approx 10\%$. While this points toward a plant origin for these extracts, it is not possible to determine the actual species of plant.

As was the case with some of the Meroitic vessels (Section 4.2.2), a small number of sherds plot just above the ovi-caprid reference ellipse. In this case, the most positive $\Delta^{13}C$ value is obtained from sherd 107·40 (*c.* 2.8‰), for which $\delta^{13}C_{16:0} = -25.6‰$, and $\delta^{13}C_{18:0} = -22.8‰$. This less depleted value for the $C_{18:0}$ fatty acid could be due to the mixing of C_3 and C_4 lipids of different $C_{16:0}/C_{18:0}$ ratios, as predicted in Section 3.3. For instance, if small quantities of a C_3 plant lipid had been mixed with the fat of an animal which had been raised on a predominantly C_4 diet, the addition of the more isotopically depleted lipid would affect the $\delta^{13}C$ values of the $C_{16:0}$ more than the $C_{18:0}$ (refer to the mixing curves in Section 3.3); this would account for the larger $\Delta^{13}C$ value. The other three sherds in this group all exhibit typical C_3 values for their individual fatty acid components, possibly indicating the mixing of different C_3 commodities. No diagnostic plant lipids were detected in these particular sherds (Section 4.4.3), which would have provided further evidence for the mixing of plant and animal products.

The two sherds that plot close to the field associated with the bovine reference materials have $\Delta^{13}C$ values of *c.* -3‰, and although these plot just outside of the ellipse, they are still likely to be indicative of predominantly bovine fats with particularly negative $\Delta^{13}C$ values. Both these sherds have similar $\delta^{13}C$ values; although the sherd that plots at $\Delta^{13}C \approx -3.1‰$ has less depleted stable isotope values ($\delta^{13}C_{16:0} = -15.5‰$; $\delta^{13}C_{18:0} = -18.6‰$). This is unlikely to be due to the mixing of plants and animals whose diet included plants of different ^{13}C values, due to the fact that the $\delta^{13}C_{16:0}$ is isotopically light (compared with the results of the botanical remains discussed in Section 3.1.4). Therefore, it is likely that these fatty acids derived from bovine fat of an animal raised on a C_4 diet.

The third sherd that plots just below the bovine reference ellipse in Figure 4.33 is intriguing. It is known that the milk of ruminant animals have $\Delta^{13}C$ values within the *c.* -4.0 to -5.0‰ range (Copley *et al.* in prep.). Therefore, the sherd that exhibits a $\Delta^{13}C$ value of -3.9‰ could well be indicative of the presence of lipids derived from dairy products. Alternatively, the $\delta^{13}C$ values of the individual components are within the range indicative of a bovid fed a diet of mixed C_3 and C_4 plants ($C_{16:0}$ = -20.5‰; $C_{18:0}$ = -24.4‰).

When the sherds are divided according to their respective context numbers (10107 and 10110), and then plotted on the graph of the $\Delta^{13}C$ v $C_{16:0}/C_{18:0}$ abundance ratios, some differences are observed (Fig. 4.34). Whilst all of the sherds containing lipids indicative of predominantly bovine fats are from context 10107, the sherds that contain extracts indicative of a predominantly plant origin are derived from both contexts. The number of sherds yielding fatty acids typical of ovi-caprid fats from the two contexts are not significantly different. Furthermore, the sherds that exhibit more positive $\Delta^{13}C$ values are from context 10107.

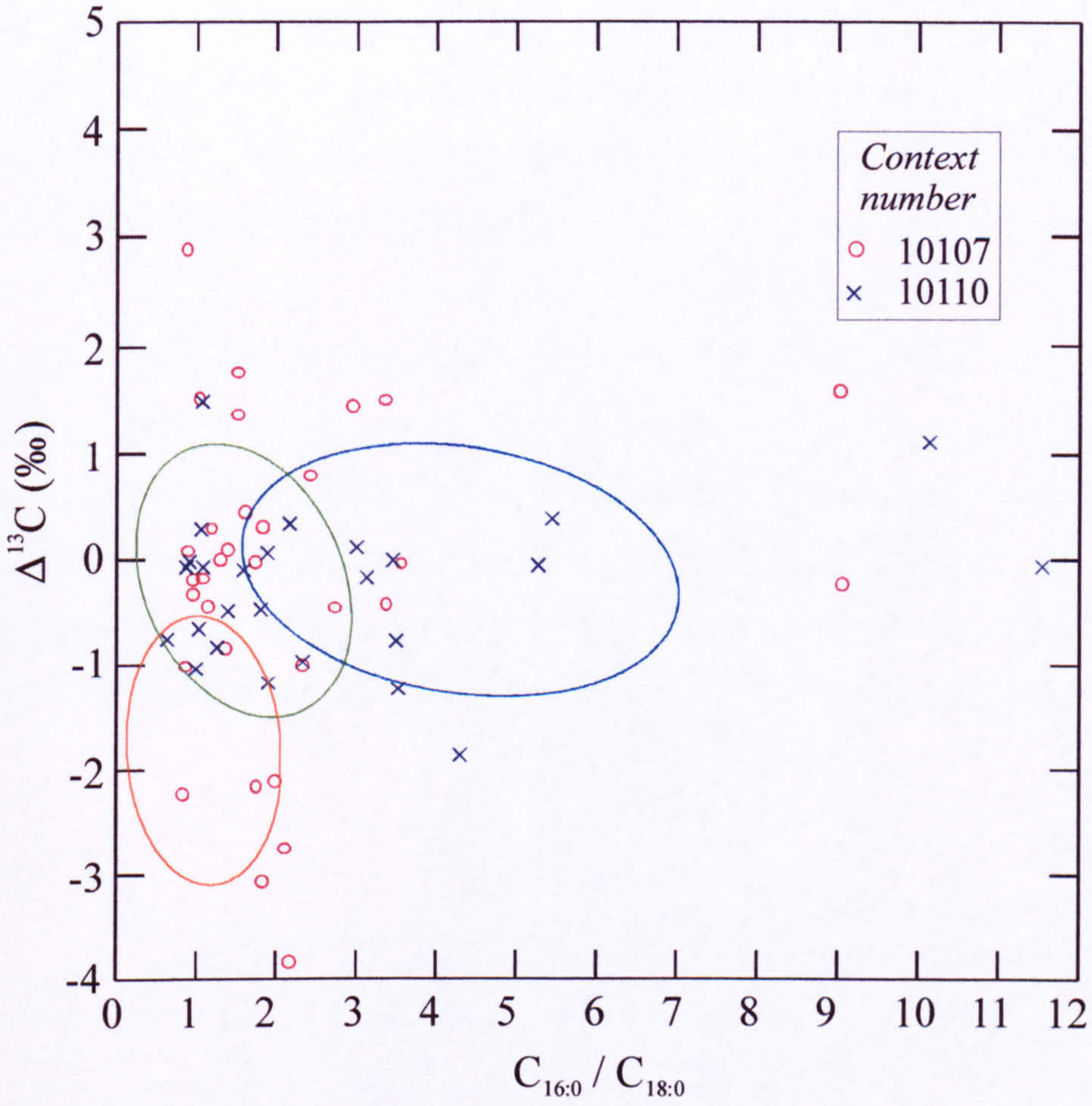

Figure 4.34 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by context number. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

Figure 4.35 plots the stable isotope values of the two fatty acid components. The majority of the sherds plot within the ranges indicative of predominantly C_3 isotope values, although four sherds (all from context number 10110) have predominantly C_4 fatty acids present.

Three of these sherds (those that plot furthest away from the $\Delta^{13}C = 0$ line) plot just above or just below the 'predominantly animal' fields in Figure 4.34, thus supporting the probability that these represent the mixing of C_3 and C_4 fats and oils in the manner described above. The fourth sherd has a $C_{16:0}/C_{18:0}$ abundance ratio that is characteristic of a plant source, and therefore represents the processing of predominantly C_4 plants, with smaller quantities of C_3 plants.

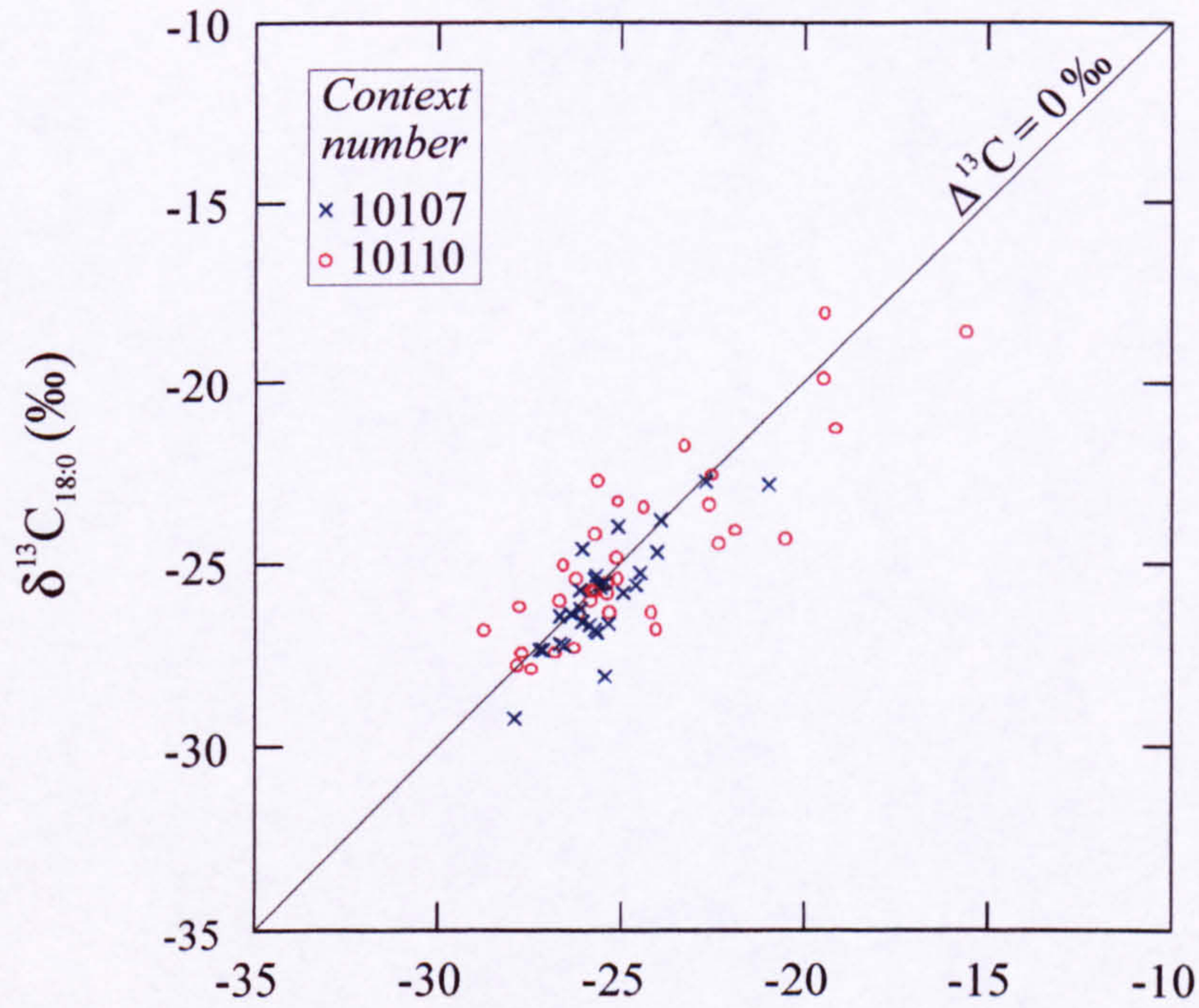

Figure 4.35 The $\delta^{13}C$ values of the major fatty acids extracted from the Post Meroitic vessels.

No apparent differences were observed in the use of the different ware types in the processing of animal and plant products in vessels from this period (Fig. 4.36). Nine out of fourteen hand-made wares (64%) yielded lipids characteristic of predominantly animal fats, whereas only three (21%) contain typical plant-like fatty acid compositions. The wheel-made wares exhibit no difference in the origins of their absorbed lipid residues.

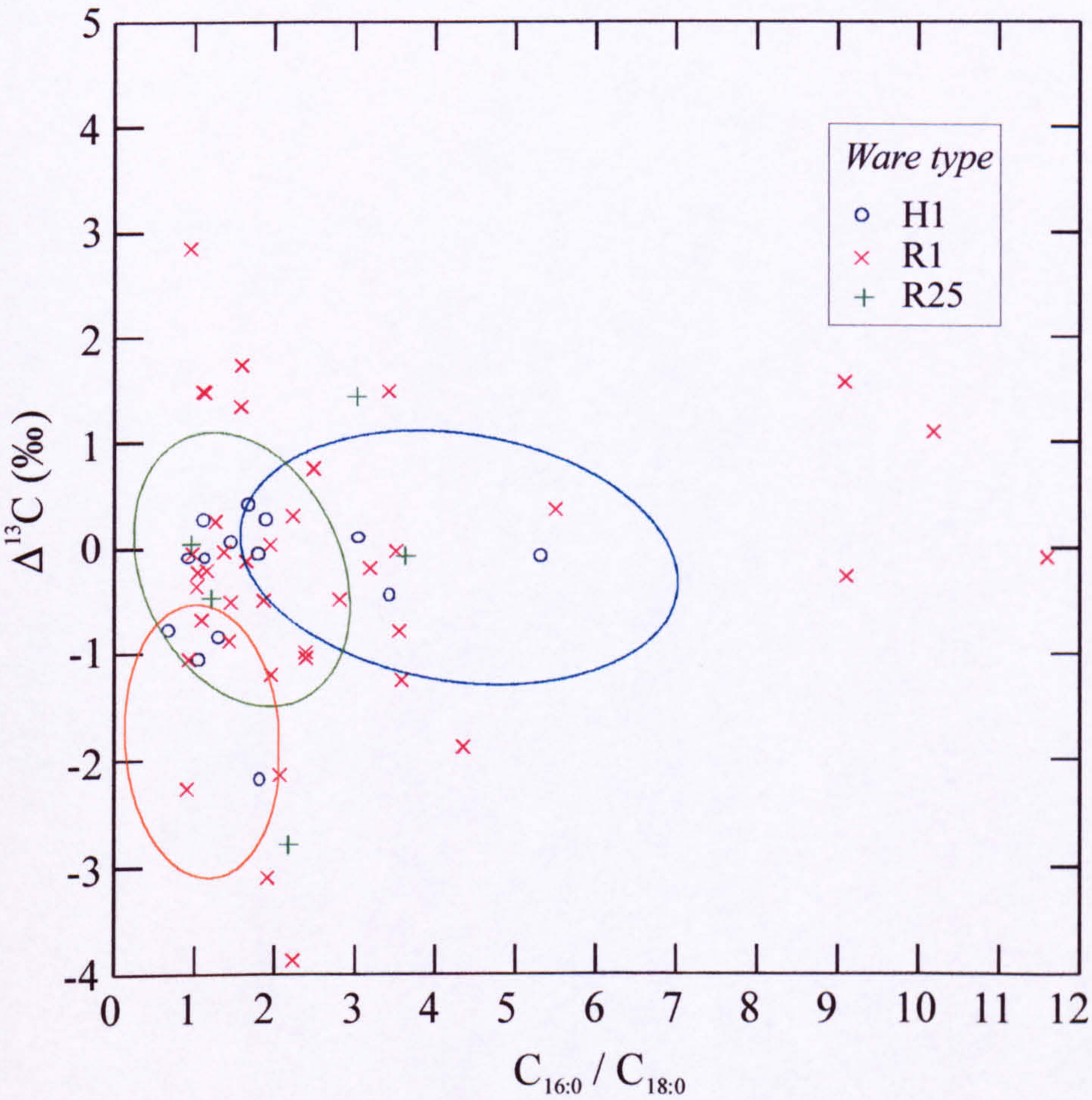

Figure 4.36 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by ware type. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

4.4.3 Other diagnostic compounds

Compounds that are commonly found in plant material were detected not only in some of the vessels that exhibit fatty acid distributions of plants, but also in sherds that contain predominantly animal fats (Fig. 4.37). Six of the sherds that plot within the ellipses associated with animal fats also yielded compounds (e.g. *n*-alkanes, derivatives of benzoic acid and β-sitosterol) that are found in plants or degraded plant material. The *n*-alkane distributions for the individual sherds indicate that the C_{27} to C_{31} *n*-alkanes predominate (Fig. 4.38), and this

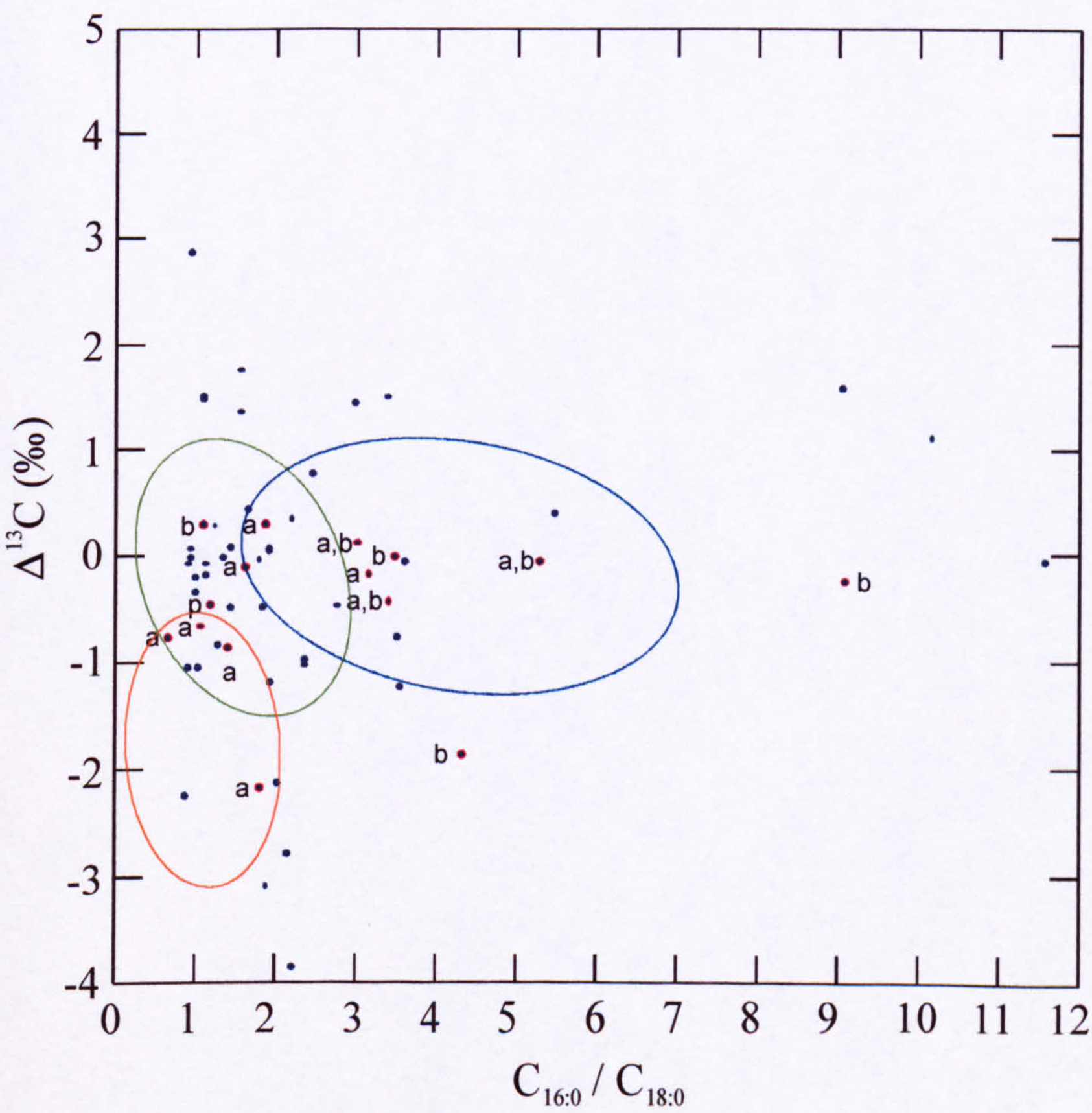

Figure 4.37 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the Post-Meroitic sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, with labelled diagnostic compounds. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3). Key: 'a': *n*-alkanes, 'b': benzoic acid derivatives, and 'p': plant sterols.

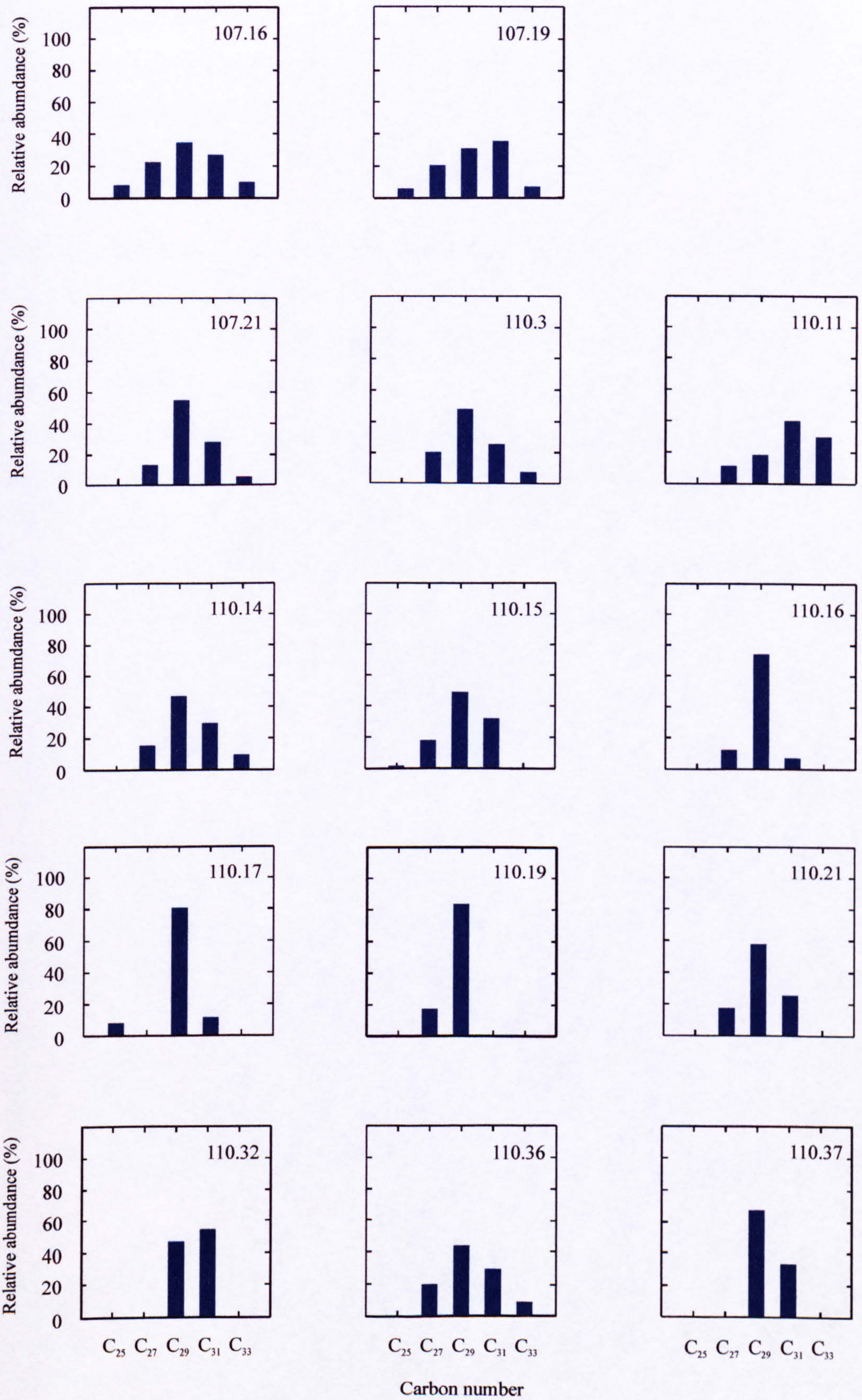

Figure 4.38 The *n*-alkane distributions extracted from the sherds from the Post-Meroitic period. Only the sherds that yielded significant abundances of *n*-alkanes are shown. Sherd numbers are given in the top right corner. C_x refer to *n*-alkanes of carbon length x.

component shall be discussed in more detail in Section 4.5. Derivatives of benzoic acid (2-methylbenzoic acid or 3-hydroxybenzoic acid) were detected in a total of seven sherds, six of which also yielded fatty acid abundances characteristic of plants.

Figure 4.39 shows the profile of the lipid extract from sherd 110·15. This sherd not only yielded a distribution of saturated fatty acids indicative of a plant origin for the extract, but also other diagnostic plant lipid compounds, including *n*-alkanes and benzoic acid derivatives. This sherd also yielded an unusually high abundance of glycerol, which must have resulted from the hydrolysis of acyl glycerol lipids. Glycerol was detected in high abundances in the majority of the animal bone reference materials (Section 3.1), and some of the archaeological plant propagules (Section 3.2). While it might be expected that

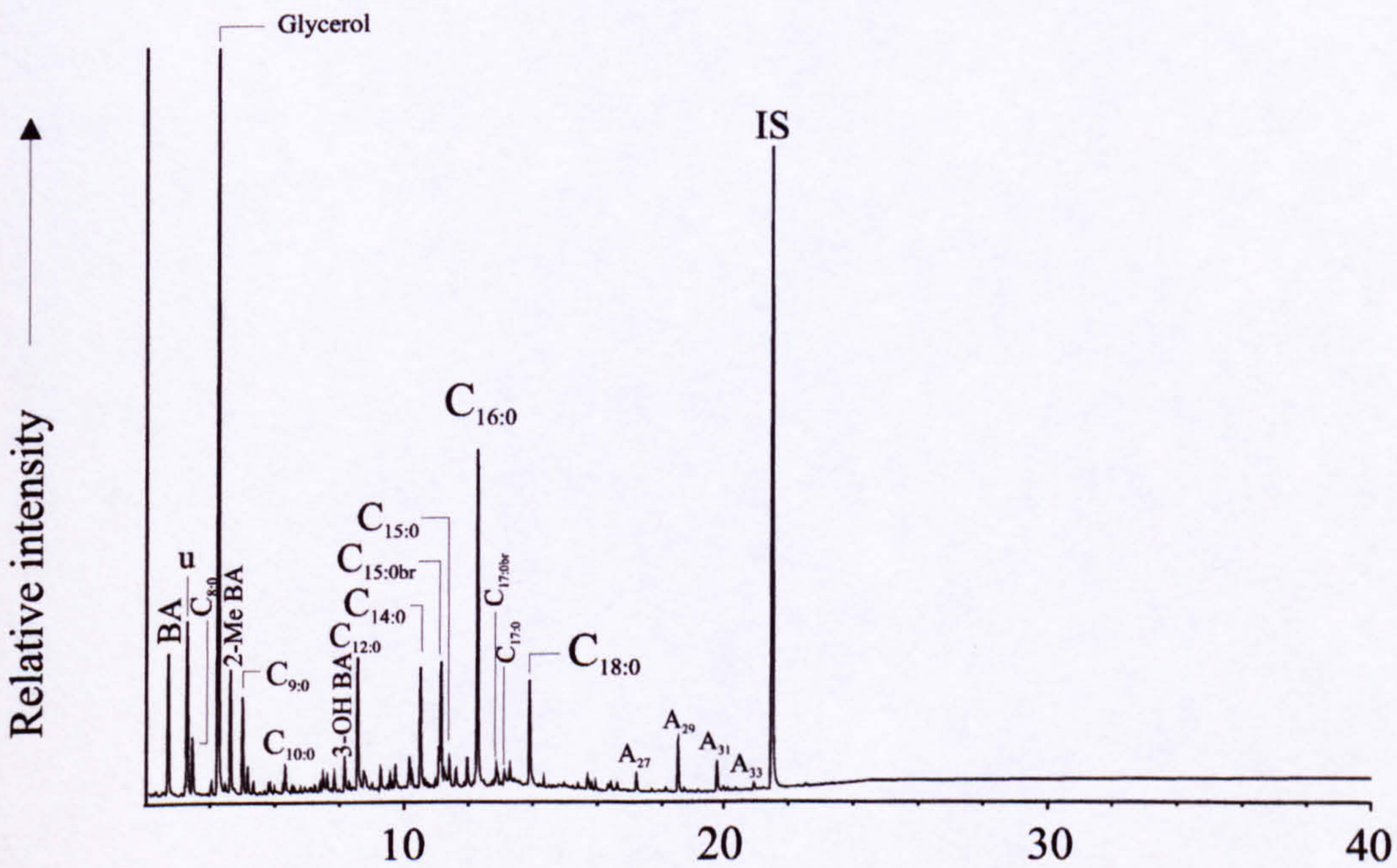

Figure 4.39 Partial gas chromatograms of the TLE of an unusual Post-Meroitic vessel. Sherd 110·15. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. A_z represents *n*-alkanes of carbon chain length z. BA refers to Benzoic acid, 2-Me BA refers to 2-methyl benzoic acid, and 3-OH BA to 3-hydroxy benzoic acid. u denotes high abundance of an unidentified compound. IS is the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

some glycerol would survive in the archaeological seeds and bones, due to the high level of physical protection afforded to their biochemical components in the burial environment, it is a unique feature of the arid nature at Qasr Ibrim that glycerol survives at all in the pottery as it is completely leached from pottery in waterlogged burial environments.

4.4.4 Overall classification of the sherds from the Post-Meroitic period

Following the classification of the sherds according to commodity type, based on their $\Delta^{13}C$ values and their $C_{16:0}/C_{18:0}$ abundance ratios and fatty acid compositions, the following conclusions can be drawn: (i) Thirty-one out of sixty-nine of the sherds (45%) from the Post-Meroitic period yielded lipids that are characteristic of predominantly animal fats. (ii) The mixing of commodities in these vessels is indicated through the detection of diagnostic plant lipids in six of these sherds (9%). (iii) Not all of the sherds were used in the processing of animal products; 17 sherds (25%) contain lipids indicative of a predominantly plant origin, and a further 11 sherds (16%) exhibit characteristic palm kernel lipids. (iv) After the sherds that yield predominantly animal fats are classified as being either 'predominantly cattle' or 'predominantly sheep/goat' fat residues, it was observed that the incidence of sheep/goat lipids in the sherds is approximately 6 times that of cattle residues. Table 4.4 shows the final classifications for the sherds from the Post-Meroitic period.

Table 4.4 Classification of the Post-Meroitic vessels based on lipid content[1]

Context	*Predominantly Cattle*[2]	*Predominantly Sheep/goat*[2]	*Mixed animal*[2]	*Cattle?*[2,3]	Predominantly Animal[2]	Mixed animal/plant[2,4]	Predominantly Plant	Palm kernel
10107	*3 (1)*	*10 (1)*	*2 (1)*	*3*	18 (3)	7 (1)	7	3
10110	*0*	*8 (1)*	*5 (2)*	*0*	13 (3)	3	10	8
TOTAL	*3 (1)*	*18 (2)*	*7 (3)*	*3*	**31 (6)**	**10 (1)**	**17**	**11**
(%)	*4 (1)*	*26 (3)*	*10 (4)*	*4*	**45 (9)**	**14 (1)**	**25**	**16**

[1]The values not in italics are the final classifications of the potsherds, those in italics represent the prevalence of specific animal fats (*c.f.* Section 3.3)
[2]The numbers in brackets indicate the number of sherds that contained traces of other diagnostic plant lipids as well as fatty acid distributions that are indicative of animal fats
[3]Includes one sherd that contains dairy fat
[4]This group includes the vessels plotting above the ovi-caprid/plant ellipses as discussed in Section 4.4.3

4.5 $\delta^{13}C$ VALUES OF THE *n*-ALKANES

As mentioned previously (Section 4.2.3), *n*-alkanes are commonly found in higher plant epicuticular waxes (e.g. Kolattukudy, 1976:582; Walton, 1990:114) and can be a useful criterion in the detection of the processing of plants in archaeological ceramics. *n*-Alkanes are a class of lipid that are relatively resistant to degradation and tend to survive vessel use and burial. Where there were sufficient abundances of *n*-alkanes, the neutral fractions of the TLEs (Section 2.3) were isolated and the $\delta^{13}C$ values of the *n*-alkanes determined by GC-C-IRMS. Of the 48 sherds that contain *n*-alkanes, 29 yielded sufficient concentrations for isotopic analysis.

The *n*-alkanes displayed unimodal carbon number distributions, ranging from C_{25} to C_{33}, generally maximising at C_{29}, with a pronounced odd-over-even predominance (Fig. 40). In some of the sherds, only low abundances of *n*-alkanes were detected, in which case only the C_{27} and C_{29} homologues were present. For this reason, the $\delta^{13}C$ values of all of the *n*-alkane components could not be determined for every sherd. The *n*-alkane distributions for the potsherds are displayed in Figure 4.41. The majority of the sherds displayed distributions that maximised at C_{29}, although in two of the sherds (24·6 and 9·4) the C_{27} was the most abundant component. In a further two sherds (110·11 and 9·8, shown in Figs. 4.38 and 4.28, respectively) the C_{31} was the most abundant *n*-alkane present; these latter *n*-alkane distributions are not typical of higher plants (e.g. Kolattukudy, 1976:582; Walton, 1990:114; Maffei, 1996).

GC/MS analysis of the lipid extracts from archaeological potsherds from Northern European sites has aided in the detection of *n*-nonacosane, together with nonacosan-15-one and nonacosan-15-ol, in a distribution indicative of the processing the leaves of the cabbage *Brassica oleracea* (Evershed *et al.*, 1991; Evershed *et al.*, 1992), similarly *Allum porrum* (leek) contains a high abundance of hentricontan-16-one a compound which has also been identified in some archaeological potsherds (Evershed *et al.*, 1992). However, long chain alcohols and ketones were not observed in any of the pottery vessels from Qasr Ibrim.

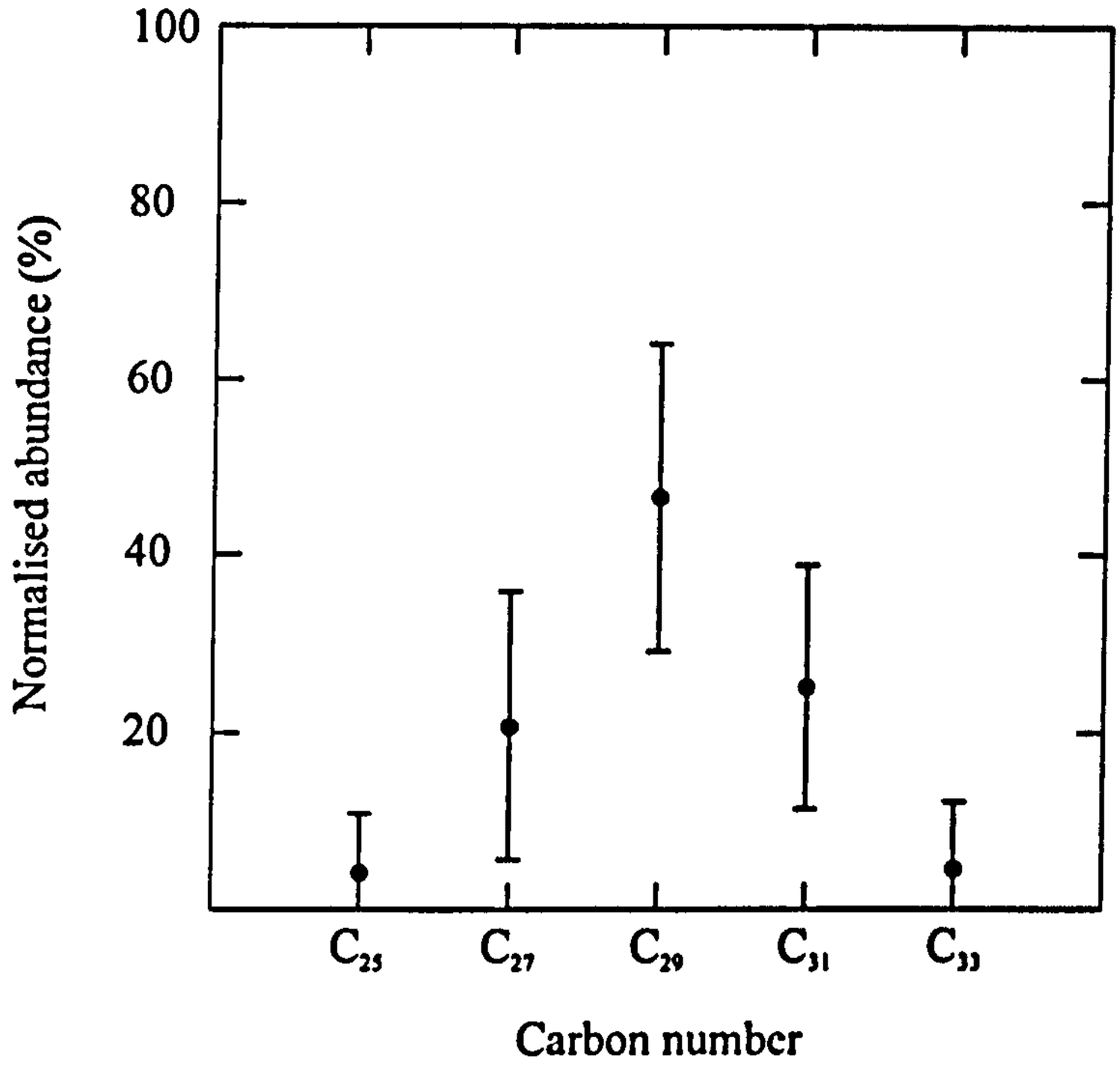

Figure 4.40 Summary of the variation of the *n*-alkane distributions from the pottery vessels from all periods. Error bars are 1 standard deviation from the mean value.

Therefore, based on the *n*-alkane distributions alone it is not possible to assign an origin to a specific plant species or group.

A clear distinction is seen between the mean $\delta^{13}C$ values for the *n*-alkane components (Fig. 4.42). The values for the C_{25} and C_{27} alkanes are significantly enriched (on average *c.* 4 - 5‰) relative to the C_{29} and C_{31} components; this phenomenon has been observed previously in the *n*-alkanes extracted from modern leaves (Lockheart *et al.*, 1997). Furthermore, the mean $\delta^{13}C$ values generally become more depleted with increasing carbon chain length: C_{25} = -23.1‰; C_{27} = -24.2‰; C_{29} = -28.5‰; C_{31} = -29.0‰ & C_{33} = -29.0‰. This has also been noted previously in analyses of plant leaf wax *n*-alkanes, and has been suggested to arise either due to the fact that different homologues of *n*-alkanes were synthesised at different times of the year, or through the utilisation of different carbon pools for biosynthesis of specific

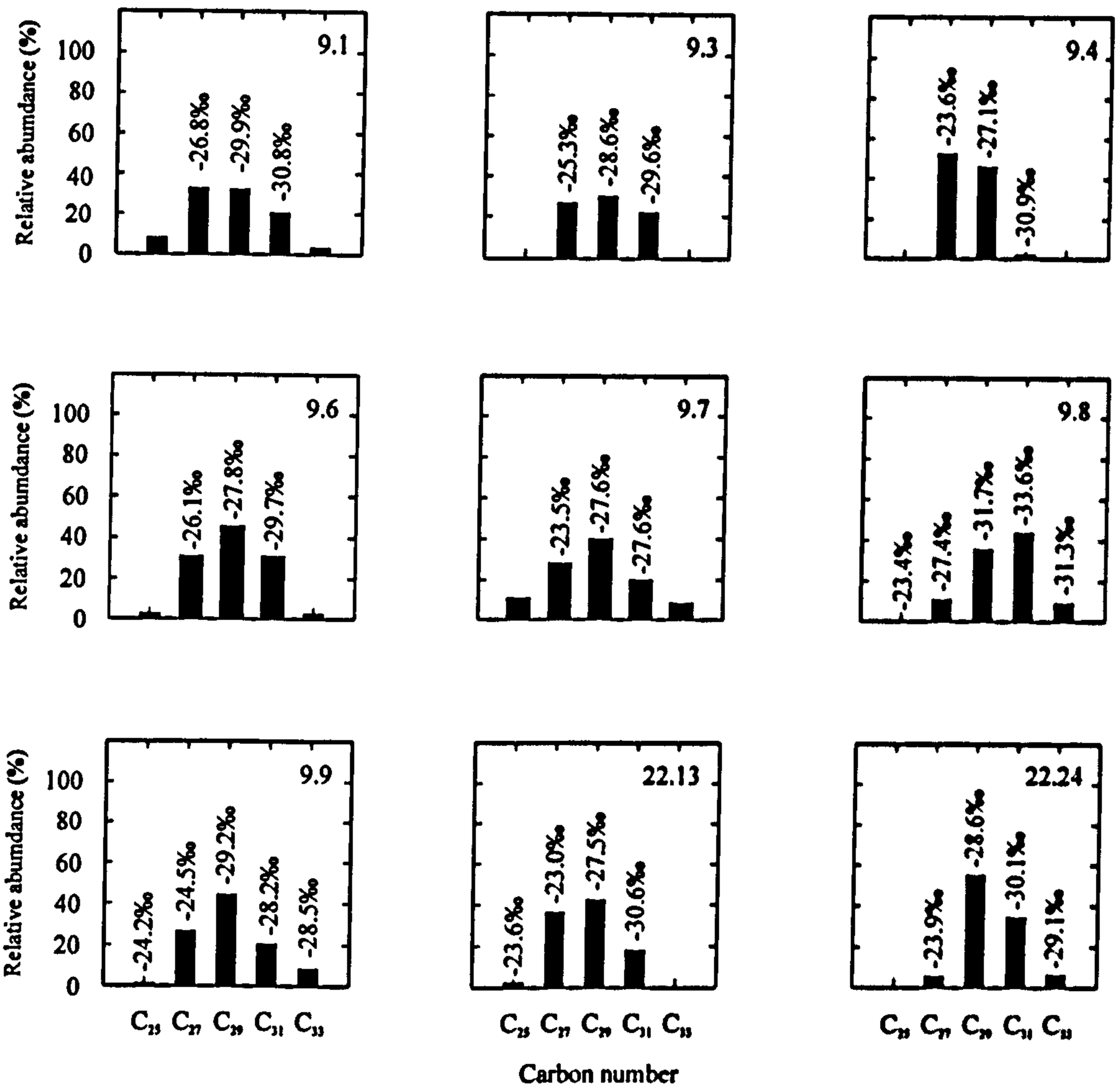

Figure 4.41 Distributions and $\delta^{13}C$ values of the *n*-alkanes.

n-alkanes (Lockheart, 1997:220-221). A note of caution should be applied here because although the mean values for the C_{25} and C_{33} alkanes are clearly different, these components were only present in sufficient abundances for compound specific stable isotope analysis in four of the sherds.

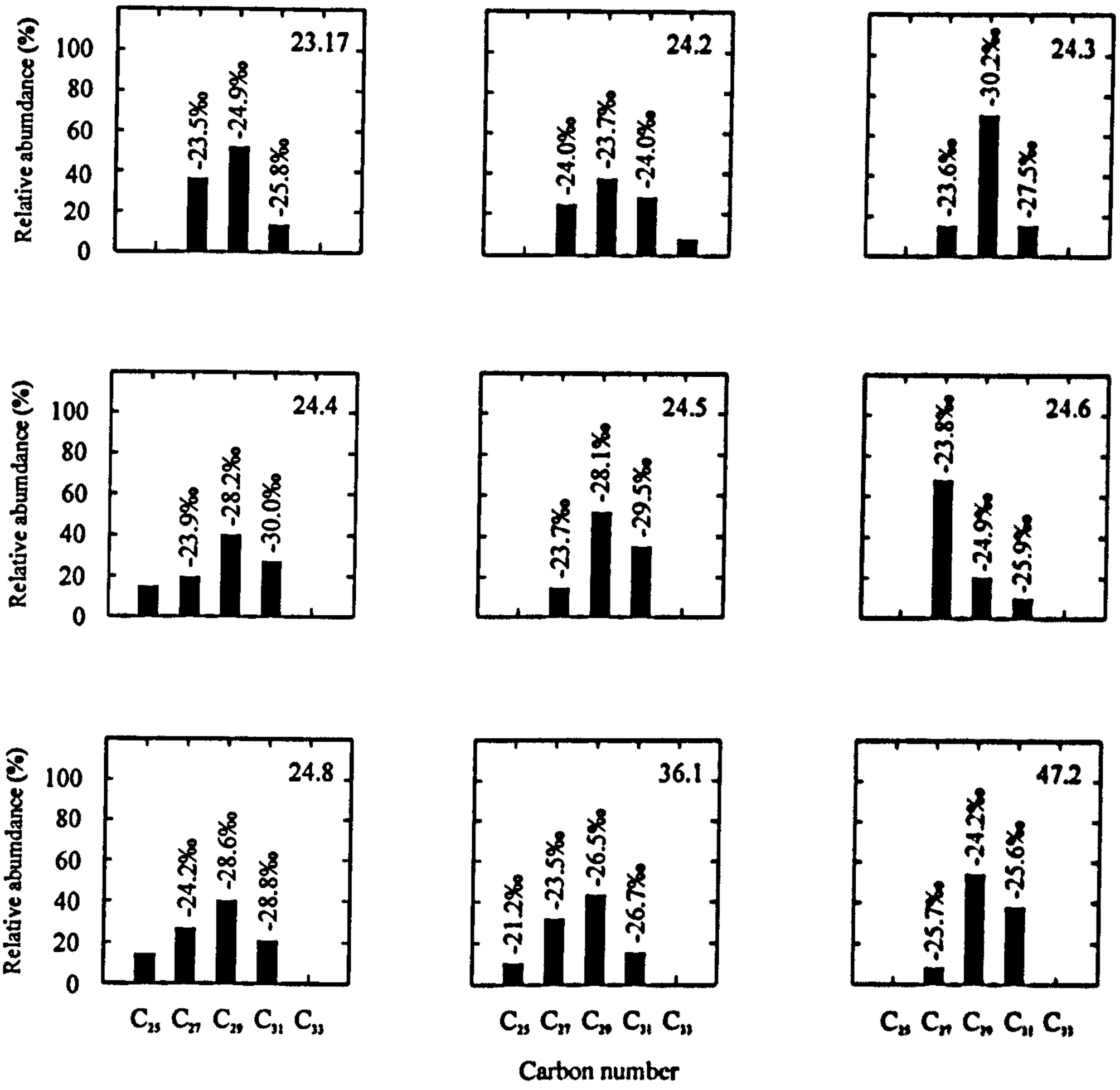

Figure 4.41 cont. Distributions and $\delta^{13}C$ values of the *n*-alkanes.

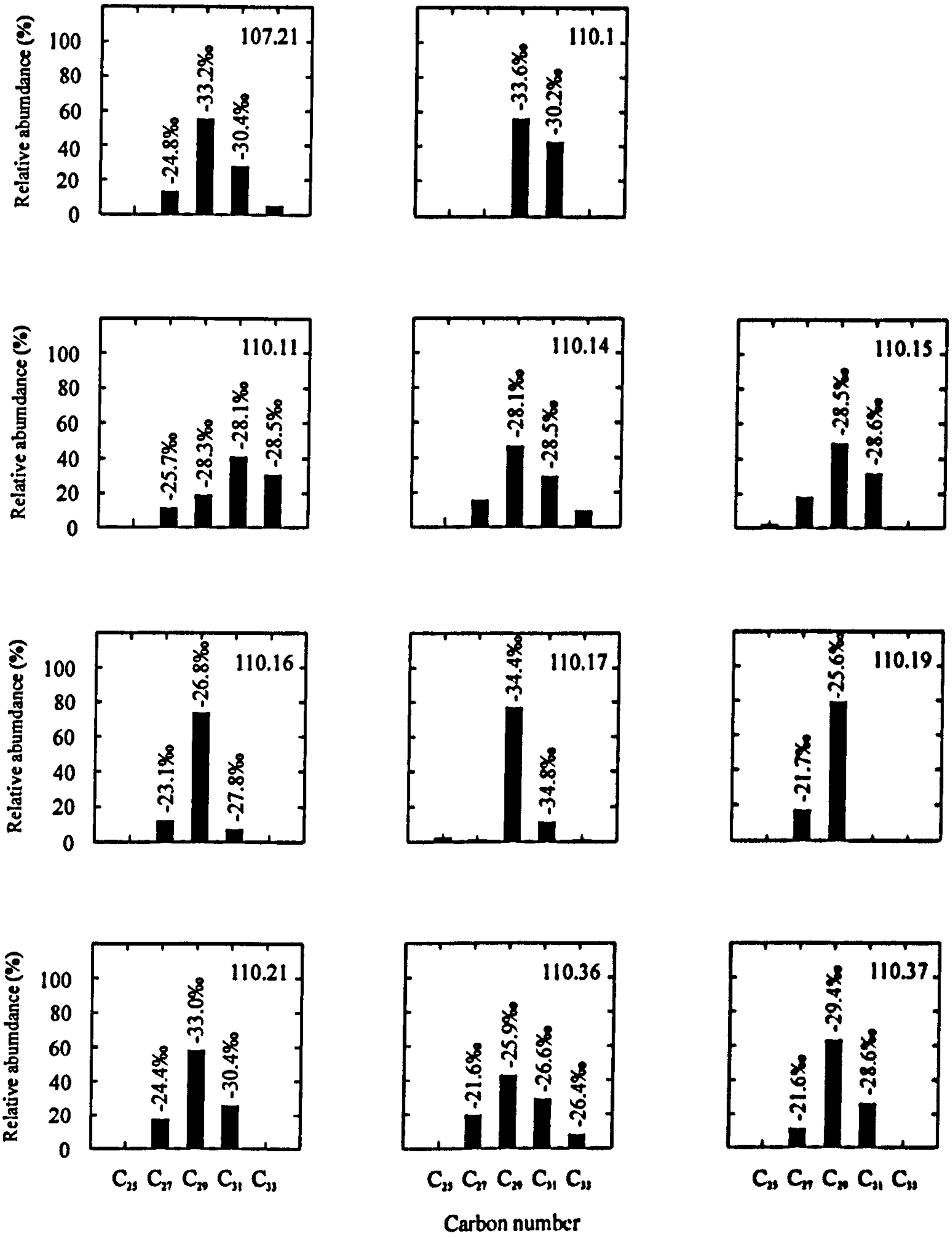

Figure 4.41 cont. Distributions and $\delta^{13}C$ values of the *n*-alkanes.

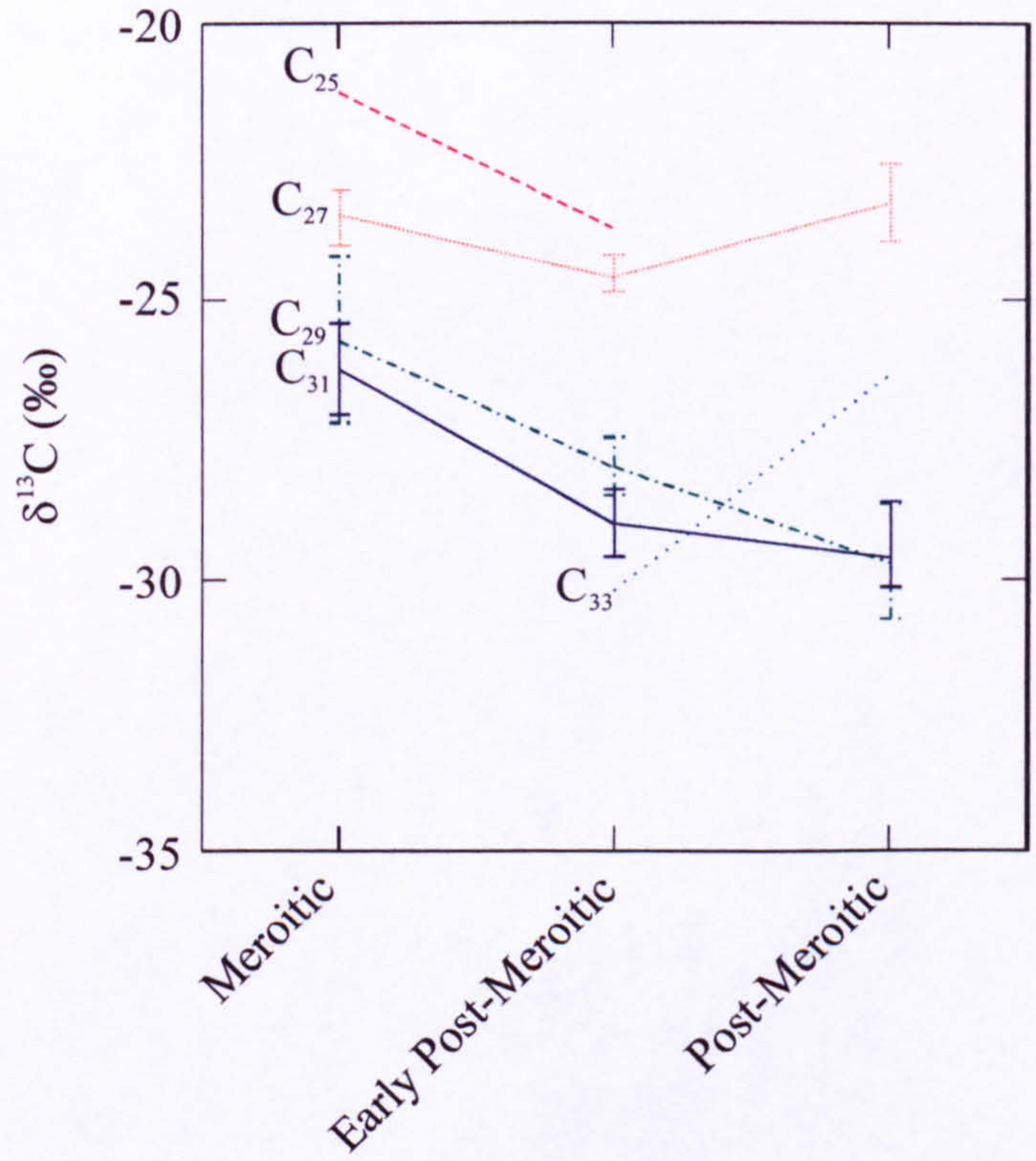

Figure 4.42 Mean $\delta^{13}C$ values for the *n*-alkanes by period. C_X refers to *n*-alkane of carbon chain length x. 1 S.E. bars are shown for the C_{27}-C_{31} components.

Interestingly, different in the $\delta^{13}C$ values are seen for the different archaeological periods (Figure 4.42), with the C_{29} and C_{31} *n*-alkanes being approximately 4‰ more depleted in the Post-Meroitic compared with the Meroitic period. The C_{29} *n*-alkane showed significant differences in its mean $\delta^{13}C$ value over all the three periods (ANOVA; *F ratio* = 3.422; $p = 0.048$). The C_{31} *n*-alkane also exhibited more depleted $\delta^{13}C$ values in the later periods, however, possibly due to the number of sherds analysed, no significant difference was observed between the Meroitic and Early Post-Meroitic/Post-Meroitic periods. In contrast, the C_{27} homologue showed no significant difference with period. The significance of these data will be considered later in conjunction with the $\delta^{13}C$ values of the fatty acids (Section 5.2.3).

4.6 THE INSOLUBLE FRACTION

4.6.1 Background

The insoluble fraction, also referred to previously as the 'bound' fraction (e.g. Regert *et al.*, 1998), here is defined as the lipid fraction released by base treatment of the crushed, solvent extracted sherd (Chapter 2).

It has already been noted that lipid compositions may change during burial, for instance, through hydrolysis of acyl moieties and oxidation of double bonds (e.g. Evershed *et al.*, 1992; Dudd *et al.*, 1998). In addition to this, lipids may undergo degradation during the lifetime of use of the vessels, for instance through their repeated heating. Furthermore, all plant and animal products are susceptible to oxidation during processing and storage (Davidek *et al.*, 1990; Penfield and Campbell, 1990; McGinley, 1991).

Evidence for oxidative degradation has already been detected in the animal bones; for example, dicarboxylic and dihydroxy acids have both been detected, accompanied by an overall decrease in the relative abundances of the unsaturated fatty acids (see also Section 3.1). Interestingly, however, relatively little evidence has been obtained for the presence of diacids and dihydroxy acids in the TLE of the pottery vessels. Previous investigations of the insoluble fraction have revealed the presence of degraded, and in particular, oxidised lipids (Regert *et al.*, 1998; Bland, 1999:177-189). For example, 9 and 10-hydroxyoctadecanoic acids, 9 and 10-hydroxyoctadecenoic acids, dihydroxyoctadecanoic acids and α,ω-dicarboxylic acids were readily detected in the insoluble fraction of Neolithic cooking vessels from Chalain, France (Regert *et al.*, 1998). Furthermore, Bland (1999) investigated the insoluble fraction from archaeological lamps from Qasr Ibrim, finding α,ω-dicarboxylic acids and vicinal dihydroxy fatty acids to be present in high abundances. Bland argued that the restricted range of positional isomers in monounsaturated fatty acids seen in plants compared with animal fats would account for the restricted range of isomeric forms of vicinal dihydroxy fatty acids present in insoluble

lipid fraction of the archaeological pottery vessels. On this basis, characterisation of dihydroxy fatty acids enabled the detection and identification of the burning of specific plant oils in Nubian lamps from Qasr Ibrim (Bland, 1999). For example, castor oil (deduced from 9,12-dihydroxy $C_{18:0}$ and 12-hydroxy $C_{18:1}$), and radish oil (deduced from 11,12-dihydroxy $C_{20:0}$ and 12,13-dihydroxy $C_{22:0}$) were both demonstrated to have been used as illuminants in the archaeological lamps.

4.6.2 The insoluble fraction of the pottery vessels from Qasr Ibrim

In view of the above, as an additional aid to the classification of the organic residues from the pottery vessels analysed as part of this thesis, the insoluble fraction of the sherds was investigated. Fifty of the sherds exhibiting a range of free lipid distributions (i.e. characteristic palm kernel lipids, predominantly animal fats and predominantly plant lipids) were targeted initially. Of the 50 sherds selected, 15 of these yielded TLEs that exhibited predominantly plant lipid distributions, and a further 15 contained fatty acids indicative of predominantly animal products. The remaining twenty sherds exhibited fatty acid distributions characteristic of palm kernel lipids.

The insoluble fraction of the majority of the vessels containing absorbed palm kernel lipids (16/20; 80%) yielded low abundances of α,ω-dicarboxylic acids typically in the range of C_6 to C_{14}, with C_9 predominating. The only dihydroxyalkanoic acid detected in these sherds was 9,10-dihydroxyoctadecanoic acid, which was seen in 13/20 (65%) of the sherds analysed, although only in low abundances (typically <5 $\mu g\ g^{-1}$ potsherd).

A range of 3-hydroxy fatty acids were detected in the majority of sherds from this group, typically ranging in carbon number from C_7 to C_{18} (Fig. 4.43). Although they were detected only in low abundances, the distribution of the $C_{12:0}$ to $C_{18:0}$ even chain 3-hydroxy homologues directly reflected that of the free fatty acids of equivalent carbon chain length detected in the TLE. Short chain 3-hydroxy acids (C_7-C_{11}) were also detected, always with the C_9 component predominating albeit in lower abundance than their longer chain counterparts (e.g. Fig. 4.43). Similarly, low abundances of 2-hydroxy acids were detected in the C_7 to C_{16} carbon number range. No other hydroxy acids were detected in the insoluble lipid fraction of these sherds.

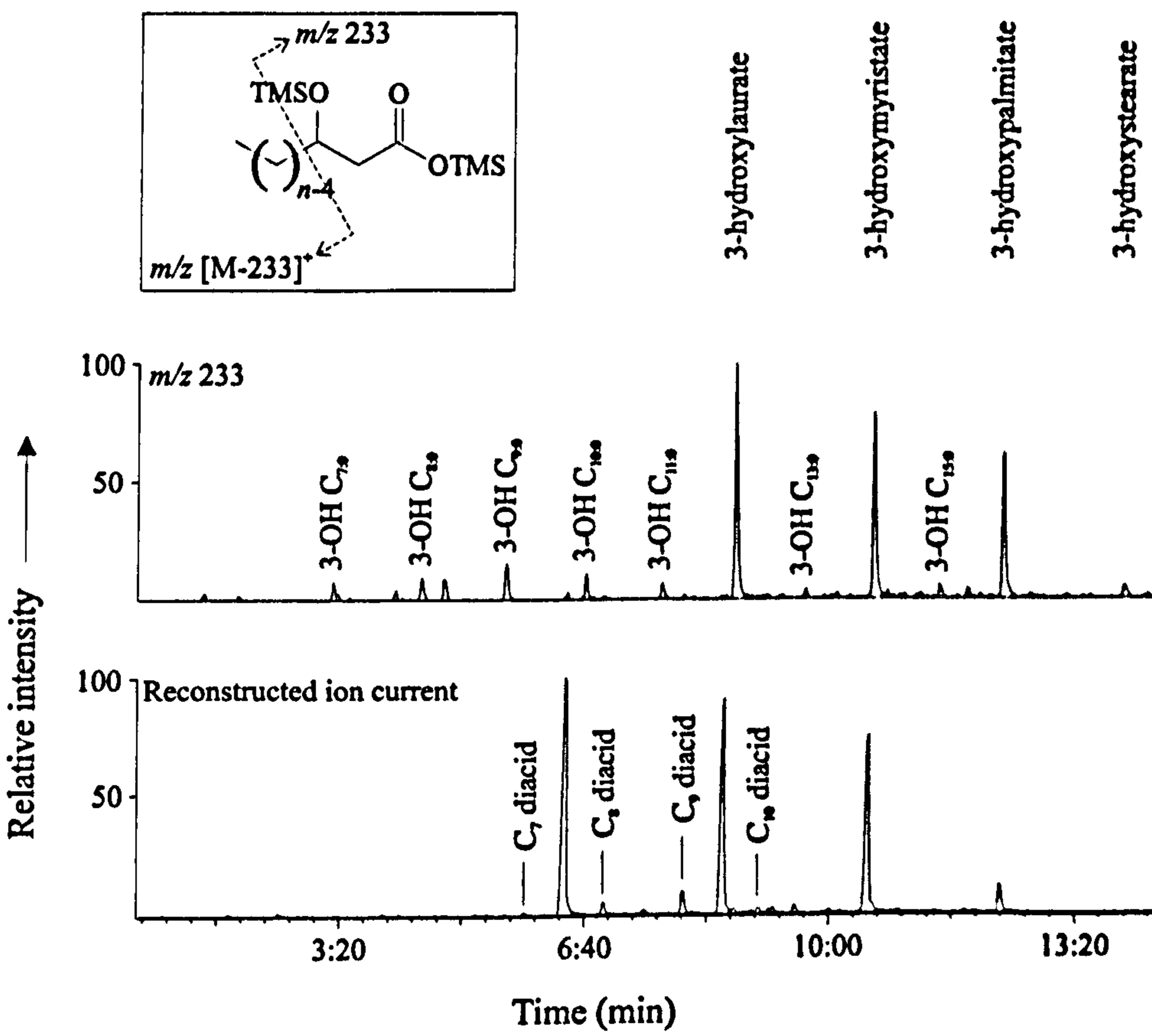

Figure 4.43 Partial GC-MS RIC and *m/z* 233 mass chromatograms of the insoluble fraction of sherd 22·6; an example of the distribution obtained from the palm fruit lipid containing vessels. 3-OH $C_{x:0}$ refers to saturated 3-hydroxy acids of carbon length x. C_y diacid refers to α,ω-dicarboxylic acids of carbon length y. The three main peaks in the RIC correspond to even chain saturated fatty acids in the range of $C_{12:0}$ to $C_{16:0}$. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

The insoluble fractions of the remaining 30 sherds are very different in composition from those obtained from the sherds with the characteristic palm kernel lipids. Figure 4.43 shows the GC-MS partial RIC and *m/z* 233 mass chromatogram of a sherd with a fatty acid distribution typical of an animal source. Similar profiles were also seen in sherds displaying free lipid distributions typical of plants. As shown by the *m/z* 233 mass chromatogram, the 3-hydroxy acids are present, but in significantly lower abundances than in the sherd shown in Figure 4.43. In the extract shown in Figure 4.44, $M^{+\cdot}$ ions could not be detected for the 3-hydroxy acids, hence their presence in the insoluble fraction rested on the retention times of the peaks in the *m/z* 233 mass chromatogram. In contrast with the sherds containing absorbed palm kernel

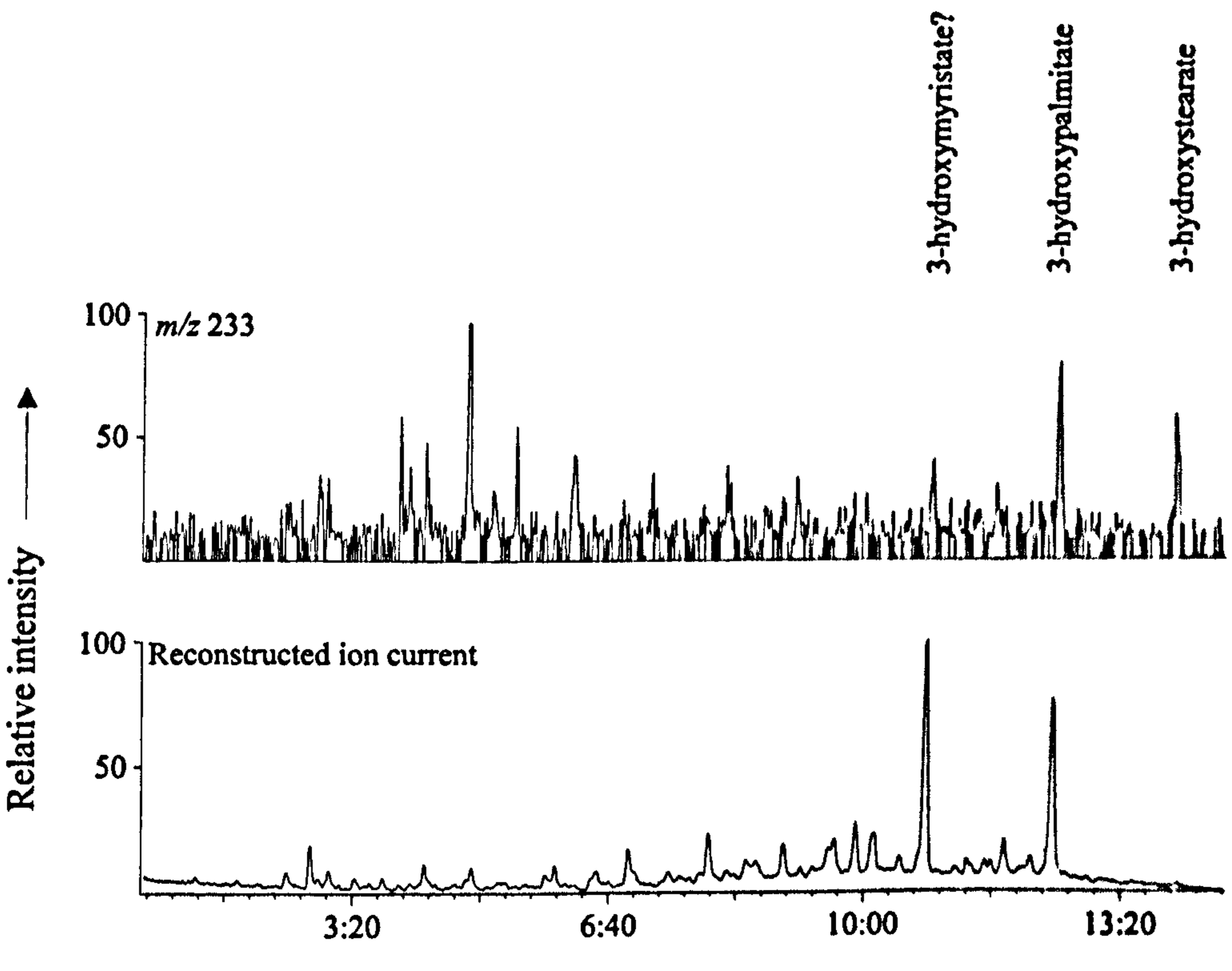

Figure 4.44 Partial GC-MS RIC and *m/z* 233 mass chromatograms of the insoluble fraction of sherd 14·2; an example of the distribution obtained from the vessels that contain typical animal fat distributions. The two main peaks in the RIC correspond to $C_{16:0}$ and $C_{18:0}$ fatty acids. The lipid concentration in the TLE of this sherd was comparable with that from sherd 22·6, shown in Figure 4.43. A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

lipids, no 2- hydroxy acids were detected, although diacids in the C_7 - C_9 carbon number range are present in low abundances. Similar results were also obtained from the GC-MS analysis of the insoluble fraction of sherds that exhibit distinctly plant fatty acid TLE distributions.

Although it would be expected that 4- and 5-hydroxy fatty acids would also be present in the insoluble fraction, they were not detected. γ- and δ-lactones are known to be formed from 4- and 5-hydroxy fatty acids respectively, via the elimination of water (see Appendix 2 [Fig. A2.2] for mechanism), and have been previously detected in mummified human remains from Egypt (Gulaçar, 1989). Neither these lactones, nor any other hydroxy acids were present in any of the insoluble fractions. However, γ- and δ-lactones were detected in the TLEs of four sherds. The carbon chain length of the γ-lactones was determined through GC-MS, confirming that $C_{14:0}$ $C_{15:0}$, $C_{16:0}$ and $C_{18:0}$ γ-lactones were present in the TLEs of the sherds. Due to the low abundance of the $M^{+\cdot}$ ions of the δ-lactones, their carbon length could not be confirmed directly from their mass spectra; although given that the $C_{16:0}$ and $C_{18:0}$ components dominated the γ-lactone distribution, it was assumed that the same homologues would similarly be the dominant δ-lactones.

4.6.3 Origins of β-hydroxy fatty acids

β- or 3-hydroxy fatty acids are known to occur in some bacterial lipids, e.g. *Flavobacterium* sp.(Yano *et al.*, 1976), and particularly in gram negative bacteria (e.g. Saraf *et al.*, 1997) where 3-hydroxytetradecanoic, 3-hydroxyhexadecanoic and 3-hydroxyoctadeacnoic acids have been used as chemical markers for bacterial lipopolysaccharides (e.g. Mielniczuk *et al.*, 1992) and as such have been identified in periodontally diseased human teeth (Lygre *et al.*, 1992).

3-Hydroxy fatty acids have also been previously used as geochemical indicators of microbial activity in lacustrine and other sediments (e.g. Eglinton *et al.*,

1968; Cardoso and Eglinton, 1983; Goossens *et al.*, 1986; Wakeham, 1999; Stefanova and Disnar, 2000). They are recognized products of β-oxidation; a simplified pathway of β-oxidation is shown in Figure 4.45, and involves the sequential loss of two carbon units through the formation of intermediate 3-hydroxy and 3-keto acids. It is therefore possible that the free fatty acids and 3-hydroxy acids in the vessels containing palm kernel lipids are indicative of a direct precursor-product relationship. The 3-hydroxy fatty acids were not detected in the TLE of the date or dom kernels (ancient or modern varieties), thus indicating that they are not components of the living plants, and that microbial inhibitors may prevent their formation in the archaeological propagules. The presence of relatively high abundances of 3-hydroxy acids in the insoluble lipid fraction of the sherds is intriguing, and there may be a few explanations:

(i) Decay was halted after one cycle of β-oxidation. Although intuitively one would expect this to be an unlikely event.

(ii) There was a build up of the polar intermediate during β-oxidation, and this was preferentially absorbed in the vessel wall during use. Although, if this were the case, then one might also expect to find higher abundances of lower, even carbon number 3-hydroxy fatty acids that had been formed through subsequent cycles of β-oxidation.

$$CH_3(CH_2)_nC_\beta H_2C_\alpha H_2CO_2H$$
$$\downarrow$$
$$CH_3(CH_2)_nCH_2{=}CH_2CO_2H$$
$$\downarrow$$
$$CH_3(CH_2)_nC(OH)CH_2CO_2H$$
$$\downarrow$$
$$CH_3(CH_2)_nCOCH_2CO_2H$$
$$\downarrow$$
$$CH_3(CH_2)_nCO_2H + CH_3CO_2H$$
$$\downarrow$$
Further β-oxidation

Figure 4.45 Microbial β-oxidation (after Voet and Voet, 1995:669).

(iii) These hydroxy fatty acids were detected in significantly higher abundances in the sherds that contained palm kernel lipids compared with the sherds that yielded animal/plant fatty lipids. This may arise from specific microbial activity indicative of a particular use of the vessels, for example from the fermentation of the sugars in the palm fruit (see Section 5.1).

4.6.4 Resinous material

In the insoluble fractions of four out of the 50 sherds (8%) abietic and dehydroabietic acid were detected (e.g. Fig. 4.46). Abietic acid is a component of fresh coniferous resins, but does not survive if the resins have been heated to high temperatures. However, dehydroabietic acid is found in fresh cedar resin, and is also known to be present in pyrolysed resin tar (Serpico, 2000:450), and can actually be detected in degraded *Pinacaea* resins that have been

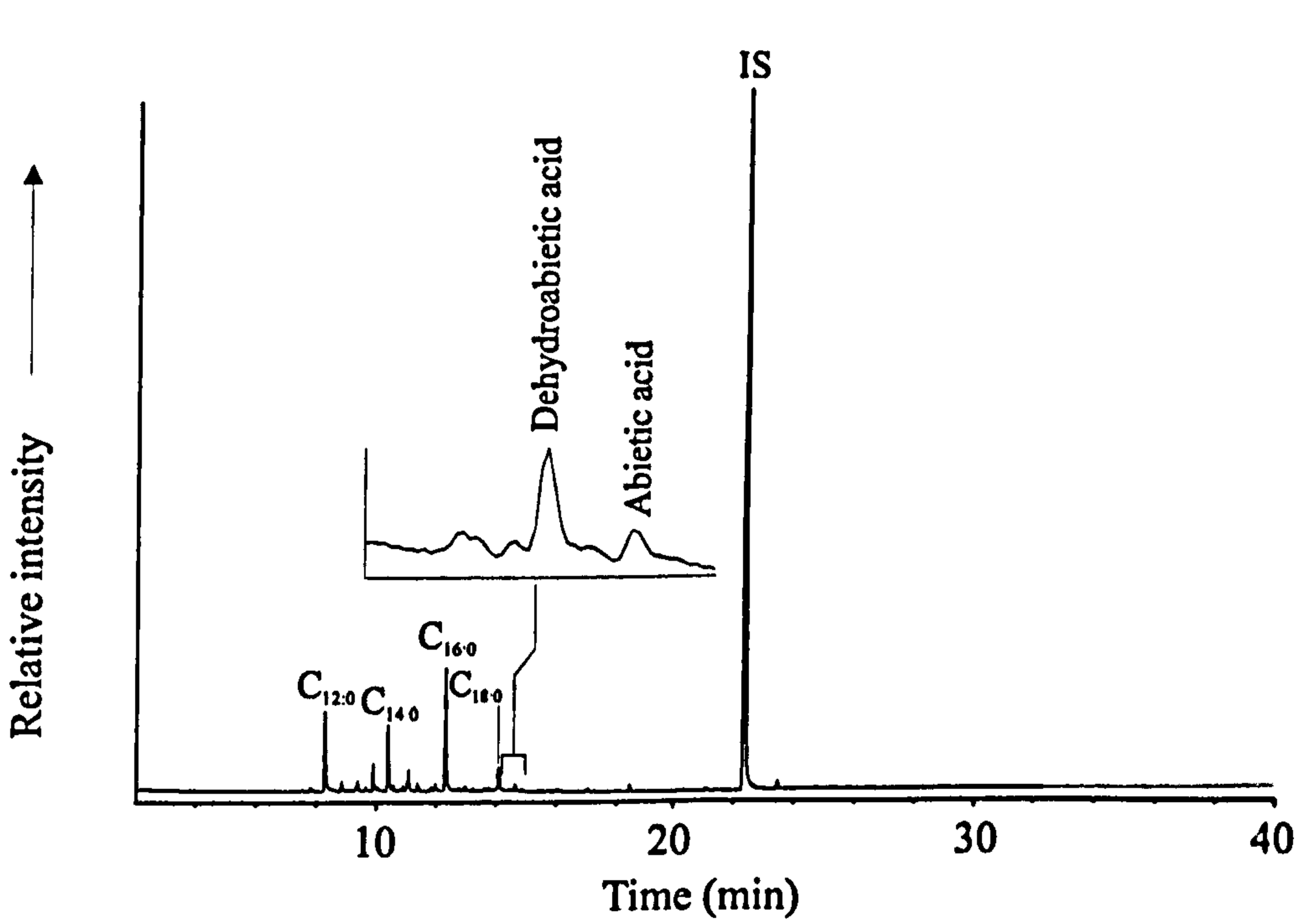

Figure 4.46 Partial gas chromatogram of the insoluble fraction of sherd 22·4. $C_{x:y}$ represents carboxylic acids of carbon chain length x and level of unsaturation y. IS is the internal standard (*n*-tetratriacontane). A DB1 (immobilised dimethyl polysiloxane) fused silica capillary column (15 m x 0.32 mm, 0.1 μm film thickness) was used.

degraded (Serpico, 2000:445). Particularly high abundances of abietic and dehydroabietic acid were detected in one vessel, but generally these compounds are only present in trace concentrations (*c.* 0.5 μg g^{-1}). They are not associated with any specific commodity; two of the sherds contained absorbed palm kernel lipids, another sherd contained predominantly animal fats while the fourth sherd contained fatty acids indicative of a predominantly plant origin.

Resins are known to have had a number of different uses in ancient Egypt. For instance, they were used as an incense, utilised in mummification, and were probably used in the manufacture of artefacts such as amulets (Serpico, 2000). Resins were also used to seal pottery vessels (Bourriau *et al.*, 2000), employed as adhesives or even included as a component of mortar (Newman and Serpico, 2000). In addition, resins may have been used as a flavouring in wine (Serpico, 2000), and although it has been suggested that resinated wine has been detected in Egyptian pottery (McGovern *et al.*, 1997), different analytical techniques will need to be employed in order to provide unequivocal verification. Organic residue analysis has enabled a mixture of coniferous resin and animal fat/plant oil to be detected in pottery from ancient Egypt (Serpico and White, 1996), possibly indicating the production of a scented ointment. In antiquity, there would have been many uses for scented ointments, for instance in religious ceremonies or even for medicinal remedies (Serpico and White, 2000).

However, given the low abundances of the abietic and dehydroabietic acids present in the bound fraction from the sherds analysed herein, and the absence of visual evidence of any resinous deposits on the sherds, it is unlikely resins were being used to seal these vessels. As abietic and dehydroabietic acids were detected in vessels used in the processing of various commodities, this suggests that these vessels were not likely to be used in the production of a resinated wine.

4.6.5 Differentiation of commodities based on the composition of the insoluble fraction

The aim of investigating the composition of the insoluble fraction was to provide a further tool in the classification of the absorbed lipid residues in the sherds. In fact, little difference was seen between the composition of the insoluble fraction from the sherds that contained predominantly animal fats and those that exhibited predominantly plant lipids, thus this fraction was not of particular use in the differentiation of these broad commodity groups. While the pottery vessels that were used to process palm fruit displayed higher abundances of 3-hydroxy acids, this does not actually assist in the differentiation of sherds used to process plant and animals products. For this reason the insoluble fraction was not investigated beyond these initial 50 vessels, although the potential application to other pottery assemblages will be discussed in Section 5.2.

4.7 PALM KERNEL LIPIDS

As was seen from earlier sections of this Chapter, 79 of the vessels from the three archaeological periods exhibited fatty acid distributions characteristic of palm kernel lipids (Fig. 4.47), namely unusually high abundances of $C_{12:0}$ and $C_{14:0}$, and low abundances of $C_{18:0}$. These 79 vessels were assessed separately in order to investigate whether they were used solely to process palm fruit, or whether they were also used to process other commodities.

4.7.1 The botanical reference materials

Modern dates were purchased on four different occasions from British supermarkets in the late summer, autumn and early winter. All of the modern dates were grown in Israel a country with a comparable climate to Nubia. The modern dom palm was purchased in an Egyptian market (in January) by Alan Clapham (the Egypt Exploration Society), and the archaeological date kernels

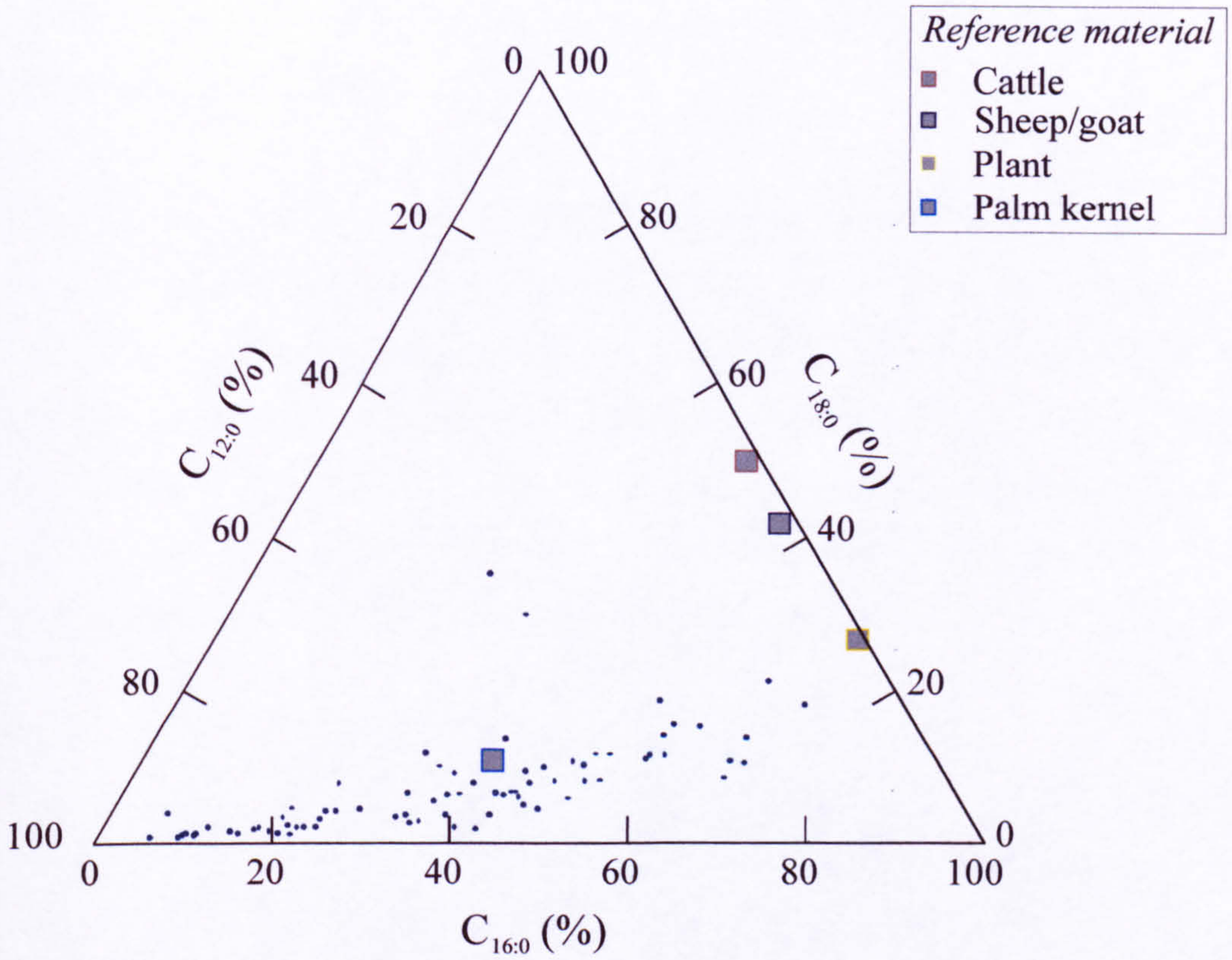

Figure 4.47 Triangular plot showing the fatty acid compositions of all the vessels interpreted to contain palm kernel lipids. The coloured boxes represent the mean values of the three fatty acids for the environmental reference materials as determined in Chapter 3 (Sheep/goat 1.6% $C_{12:0}$, 56.0% $C_{16:0}$ & 42.4% $C_{18:0}$; Cattle 0.6% $C_{12:0}$, 48.6% $C_{16:0}$ & 50.8% $C_{18:0}$; Plant 0.1% $C_{12:0}$, 72.4% $C_{16:0}$ & 27.5% $C_{18:0}$; and Palm kernel 50.7% $C_{12:0}$, 38.1% $C_{16:0}$ & 11.2% $C_{18:0}$).

excavated from Post-Meroitic and Christian contexts and supplied by Peter Rowley-Conwy (Durham University).

Table 4.5 shows the normalised saturated fatty acid compositions for the palm reference materials, and Figure 4.48 shows these values plotted onto a triangular plot. Both the modern dom kernel and the literature values for the palm kernels plot in the far left corner of the diagram, indicating the extremely high abundances of $C_{12:0}$ fatty acid present in these commodities. The modern and archaeological reference dates plot along the x-axis, indicating that there is considerable variation in the abundance of $C_{16:0}$ in the date kernels (although the abundance of $C_{18:0}$ is consistently low). Whilst the palm kernels appear to fall into two distinct groups (Fig. 4.48), i.e. date and other palm kernels, due to the

wide variation in the fatty acid composition of date kernels, these cannot be distinguished statistically.

Palm oil is a commodity distinctly different from the palm fruit, originating from the trunk of the tree, and collected by tapping a hole into the trunk and recovering the oil that oozes out. Since the harvesting of palm oil can actually kill the whole tree, its collection would only have been practiced in certain circumstances in antiquity. The fatty acid distribution of palm oil is dominated by $C_{16:0}$ and $C_{18:1}$ fatty acids (Gunstone *et al.*, 1986), and therefore readily distinguished from the lipids from the kernels of the palm fruit.

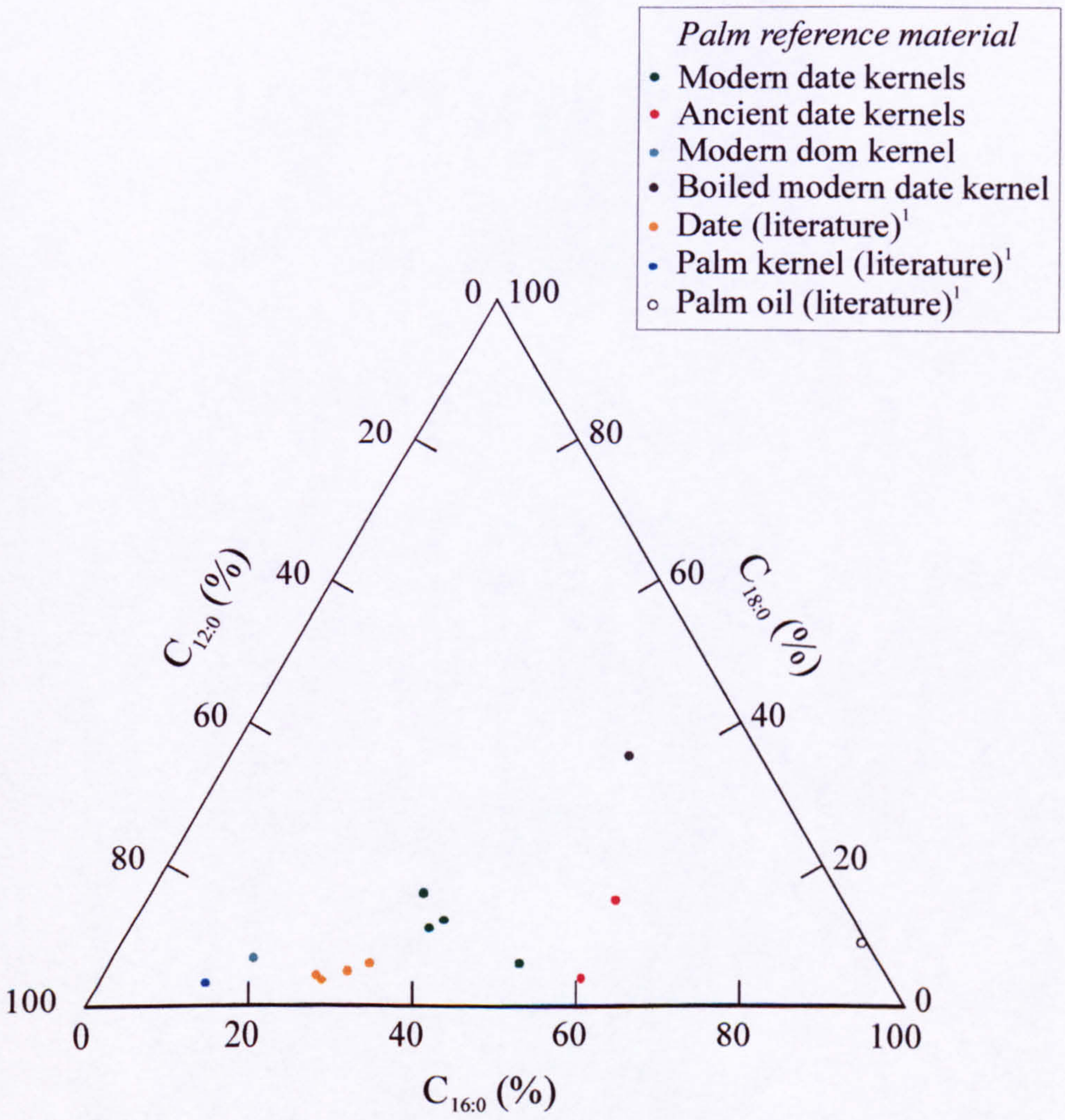

Figure 4.48 Triangular plot showing the fatty acid compositions of all the palm reference materials. [1]Date palms are distinguished here from other varieties of palm (after (Devshony *et al.*, 1992)).

Table 4.5 Fatty acid compositions and $\delta^{13}C$ values of the palm reference materials

Sample name	Archaeological/ Modern?	Normalised fatty acid composition (%)				$\delta^{13}C$ fatty acid values (‰)[1]			
		$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$
Mdate1	Modern	38.7	25.8	27.2	8.3	-33.9	-32.1	-32.4	-31.7
Mdate2	Modern	35.9	27.9	27.4	8.8	-34.0	-32.4	-32.4	-32.8
Mdate3	Modern	37.2	26.1	24.8	11.9	-33.8	-32.3	-32.2	-32.2
Mdate4	Modern	44.5	29.4	20.1	6.0	-33.3	-32.3	-32.5	-33.5
Adate1	Archaeological	24.6	33.8	39.1	2.5	-32.9	-32.1	-32.0	-31.8
Adate2	Archaeological	18.3	33.1	38.5	10.1	-33.4	-31.9	-31.5	-32.1
Bdate	Modern	12.9	18.2	40.0	28.9	-33.2	-32.5	-32.4	-32.5
Mdom	Modern	60.0	20.7	14.0	5.3	-33.6	-33.0	-33.8	-33.1
Ldate1[2]	Modern	53.9	21.6	21.6	2.9	n/a	n/a	n/a	n/a
Ldate2[2]	Modern	44.8	27.6	23.3	4.3	n/a	n/a	n/a	n/a
Ldate3[2]	Modern	52.2	24.6	20.0	3.2	n/a	n/a	n/a	n/a
Ldate4[2]	Modern	48.4	25.7	22.2	3.7	n/a	n/a	n/a	n/a
Lpalm oil[2]	Modern	0.4	2.2	88.6	9.1	n/a	n/a	n/a	n/a

[1]Modern values adjusted for post-industrial fossil fuel burning by adding 1.14‰ to the measured values (Friedli 1986).
[2]Literature values are after (Devshony *et al.*, 1992), and include only fatty acid compositions.

In addition, compositional data were also obtained for a boiled modern date (Section 2.32). As can be seen from Table 4.5, the $\delta^{13}C$ values were not different from the untreated dates (which is not surprising), but the fatty acid composition was different, with some loss of the $C_{12:0}$ and $C_{14:0}$ components (Table 4.5, Fig. 4.48).

4.7.2 The pottery vessels

Figure 4.49 shows the range of abundances of the saturated fatty acids displayed by the pottery vessels and the palm reference materials. The saturated fatty acids from the vessels and palm fruit had the same variance and their means were not significantly different (T test; *df* = 89; $C_{12:0}$ T=0.28, $C_{14:0}$ T=1.01, $C_{16:0}$ T=0.12, $C_{18:0}$ T=0.08), suggesting that the majority of the sherds contained predominantly palm kernel lipids. However, large differences do appear between the $\delta^{13}C$ values of the fatty acids extracted from the archaeological sherds, and those from the palm kernel reference materials (Fig. 4.50). The majority of the $\delta^{13}C$ values of the major fatty acids from the sherds are enriched relative to those obtained from the reference palm kernels. Variations in the $\delta^{13}C$ values of plants can arise from differences in latitude (Beerling *et al.*, 1993), altitude (Vitousek *et al.*, 1990; Beerling *et al.*, 1993), and even season (Lowdon and Dyck, 1973). Furthermore, studies also indicate that natural, intra-species variations also exist within a specific plant species (Ehleringer, 1990; Lockheart *et al.*, 1997).

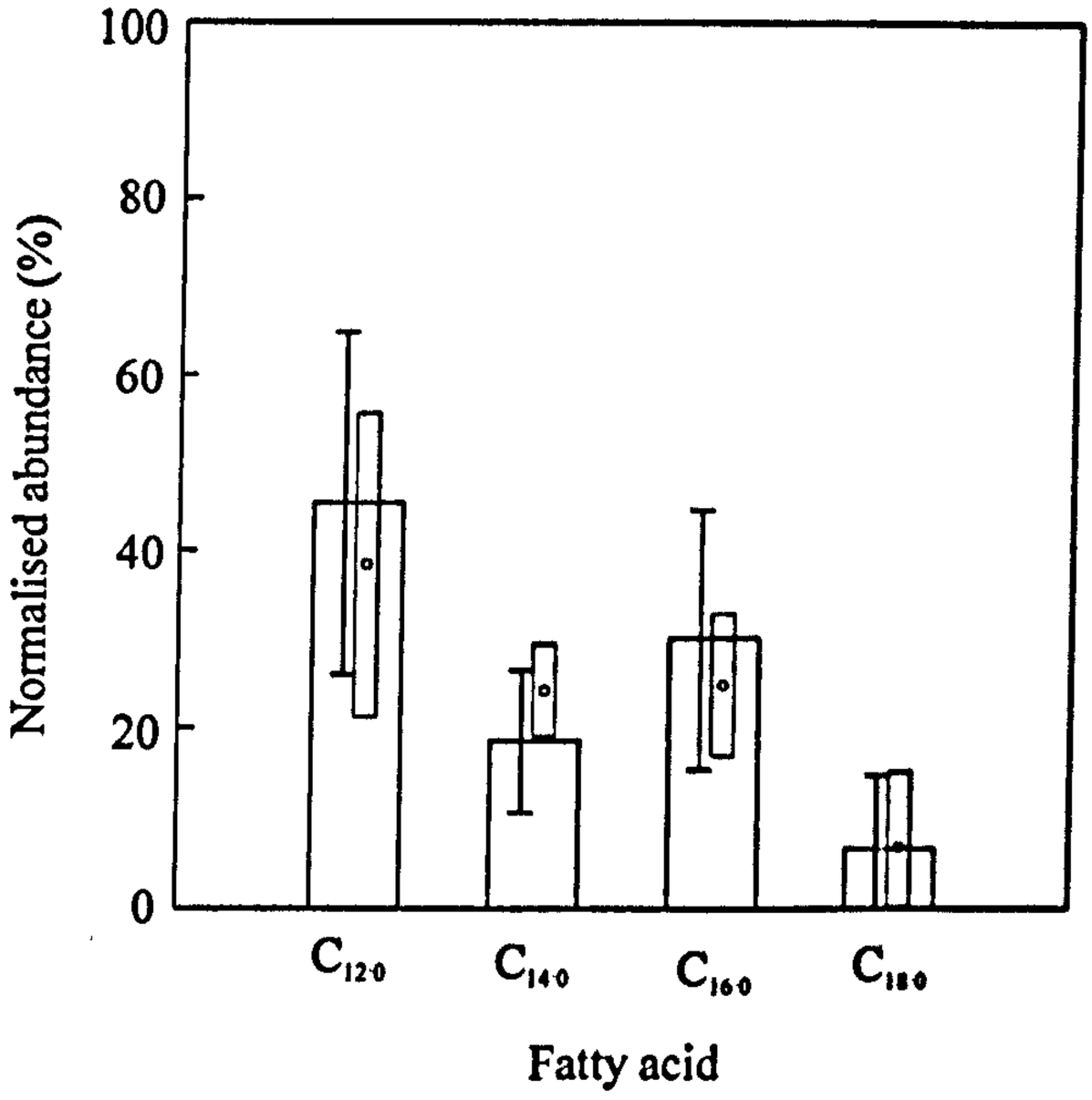

Figure 4.49 Comparison of fatty acid distributions in the pottery vessels and palm kernel reference materials. The black bars are the mean values for the fatty acids extracted from the sherds (with error bars of 1 standard deviation). The orange boxes represent 1 standard deviation error bars from the mean (the circle) for the fatty acids extracted from the palm kernel reference materials.

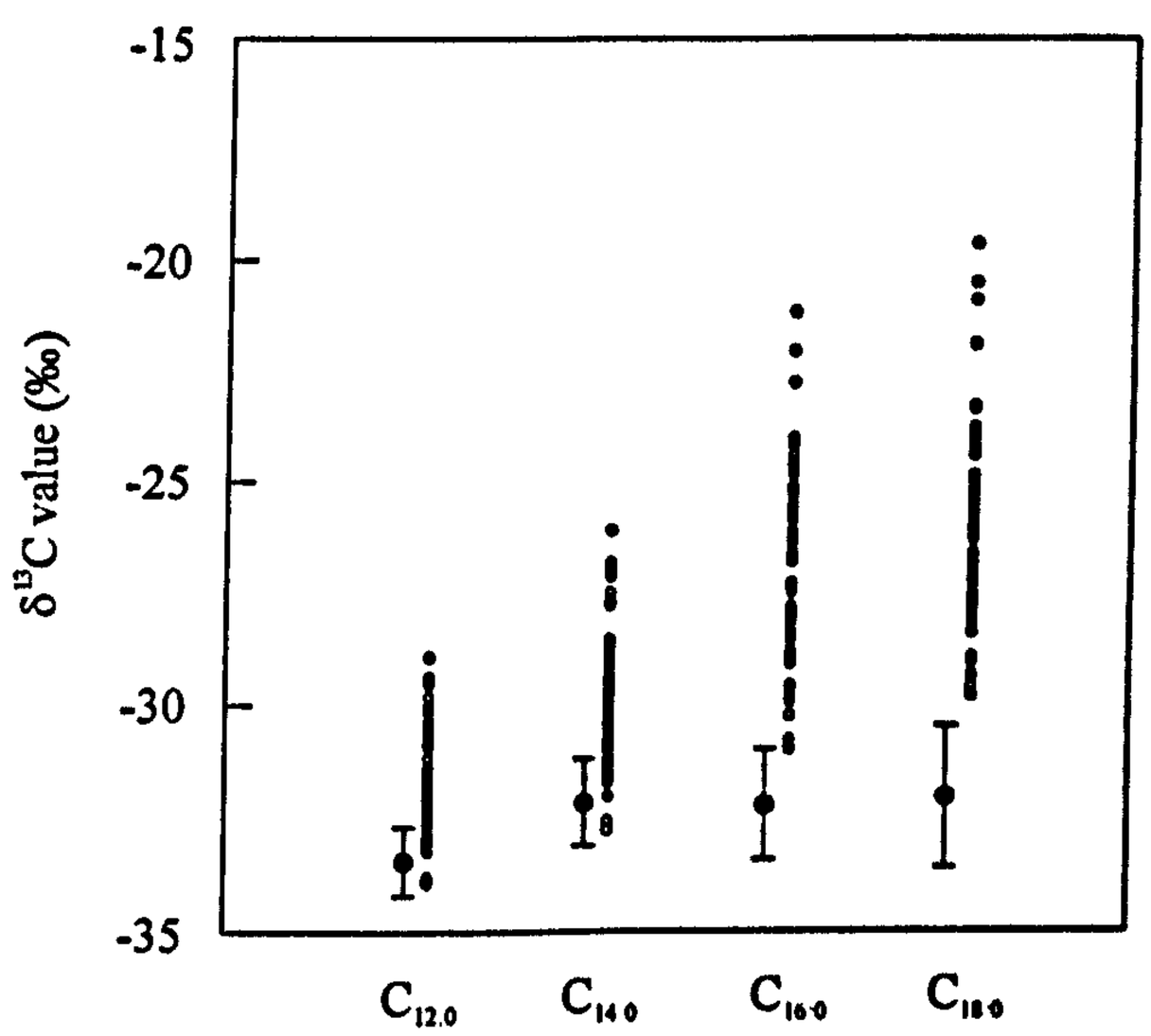

Figure 4.50 Plot of the fatty acid $\delta^{13}C$ values for the major fatty acids contained in the pottery vessels and the palm kernel reference materials. The mean $\delta^{13}C$ values for the reference materials are the filled circles, with 1 standard deviation error bars, while the unfilled circles are the $\delta^{13}C$ values obtained from the sherds.

Since there was no significant variation in the relative abundances of the saturated fatty acids (Fig. 4.49), but the mean $\delta^{13}C$ values were significantly different between the sherds and the palm reference materials (Fig. 4.50), this raises two related questions: Firstly, do the $\delta^{13}C$ values recorded in the pottery vessels reflect variations that can exist within the same species of plant in nature? And secondly, how many of these vessels actually only contain palm kernel lipids? In order to answer these questions, the fatty acid compositions and their $\delta^{13}C$ values were investigated in more detail.

The variability between the $\delta^{13}C$ values of the fatty acids in the pottery vessels and the palm kernel reference materials were compared using F-tests of variance. The results are shown in Table 4.6, and indicate that the sherds do not exhibit a significant difference (at the 95%CI) in the variability of their $\delta^{13}C_{12:0}$ and $\delta^{13}C_{14:0}$ values, nor between the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values. However, significant differences in variability of all of the other paired combinations did exist (e.g. between the $\delta^{13}C_{12:0}$ and $\delta^{13}C_{18:0}$ values). Less variability was observed in the $\delta^{13}C$ values of the palm kernels, but interestingly only the $\delta^{13}C_{12:0}$ and $\delta^{13}C_{18:0}$ values obtained from the reference materials exhibit significantly different variances (Table 4.6).

Importantly, this suggests that some of the variance in the $\delta^{13}C_{18:0}$ values from the palm kernel lipids absorbed in the pottery can be ascribed to natural variance in the $\delta^{13}C$ values of fatty acids of the palm fruit. However, natural variances cannot account for all of the variability in the $\delta^{13}C$ values observed in the sherds, due to the significant variances that were detected between $\delta^{13}C_{12:0}$ & $\delta^{13}C_{14:0}$ and $\delta^{13}C_{12:0}$ & $\delta^{13}C_{16:0}$. Therefore, some of the observed variance may also be due to the mixing of fatty acids from other lipid sources with different $\delta^{13}C$ values, and low abundances of $C_{12:0}$ and $C_{14:0}$.

Table 4.6 Test of variability (F-test) of $\delta^{13}C$ values of the fatty acids in the pottery vessels and palm kernels

			$\delta^{13}C_{12:0}$	$\delta^{13}C_{14:0}$	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$
Pottery vessels		mean (‰)	-32.0	-30.0	-26.9	-26.4
		standard deviation (‰)	1.2	1.4	2.0	2.3
	Significance level (F-test)[1]	$\delta^{13}C_{12:0}$	.	.	.	.
		$\delta^{13}C_{14:0}$	> 0.05	.	.	.
		$\delta^{13}C_{16:0}$	**≤ 0.05**	**≤ 0.05**	.	.
		$\delta^{13}C_{18:0}$	**≤ 0.05**	**≤ 0.05**	> 0.05	.
Palm kernels		mean (‰)	-33.3	-32.0	-32.1	-31.9
		standard deviation (‰)	0.6	0.7	0.9	1.3
	Significance level (F-test)[2]	$\delta^{13}C_{12:0}$	.	.	.	.
		$\delta^{13}C_{14:0}$	> 0.05	.	.	.
		$\delta^{13}C_{16:0}$	> 0.05	> 0.05	.	.
		$\delta^{13}C_{18:0}$	**≤ 0.05**	> 0.05	> 0.05	.

[1] $n = 79$
[2] $n = 10$
F-tests significant at the 95% CI are in bold.

In order to try and account for any natural variance that may occur in the $\delta^{13}C$ values of the absorbed palm kernel lipids, the $\delta^{13}C$ values of the fatty acid components were subtracted from the corresponding $\delta^{13}C_{12:0}$ value in the sherd/palm kernel. Essentially, this standardises the stable isotope values to the $\delta^{13}C_{12:0}$ for each sherd/palm kernel. Following these calculations, the new values were then statistically tested and the results summarised in Table 4.7.

Table 4.7 T-Test of standardised[1] $\delta^{13}C$ values of the fatty acids in the pottery vessels and palm kernels

	Pottery vessels[2]		Palm kernels[2]			
	Mean	Standard deviation	Mean	Standard deviation	Test statistic[3]	Level of significance
$(\delta^{13}C_{12:0} - \delta^{13}C_{14:0})$	-1.85	0.87	-1.27	0.44	1.81	None
$(\delta^{13}C_{12:0} - \delta^{13}C_{16:0})$	-4.67	1.87	-1.21	0.63	5.14	99.9% CI
$(\delta^{13}C_{12:0} - \delta^{13}C_{18:0})$	-5.34	2.43	-1.37	1.05	4.54	99.9% CI

[1] $\delta^{13}C_{X:0}$ is subtracted from the $\delta^{13}C_{12:0}$ value for both the sherds and palm kernels (see text)
[2] All values in ‰
[3] T-Test; *df*=85

There was no significant difference between the mean $\delta^{13}C$ values of the $(\delta^{13}C_{12:0} - \delta^{13}C_{14:0})$ for the pottery vessels and palm kernels. On the other hand, there was a significant difference in the mean values between vessels and the palm kernels for $(\delta^{13}C_{12:0} - \delta^{13}C_{16:0})$ and $(\delta^{13}C_{12:0} - \delta^{13}C_{18:0})$ values. This indicates that mixing of fatty acids of different $\delta^{13}C$ values is likely to have occurred in many of these vessels. This can be stated because not only has some of the natural variation been accounted for by determining the $(\delta^{13}C_{12:0} - \delta^{13}C_{x:0})$ values, but also because the differences that were significant are only associated with the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values. $C_{16:0}$ and $C_{18:0}$ are ubiquitous in animal fats and plant oils, and hence any addition of these commodities would preferentially alter the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values. The exact extent of any mixing of fats and/or oils in the vessels in antiquity can only be assessed by considering both the individual fatty acid compositions and $\delta^{13}C$ values together, two examples of which are given in Figure 4.51.

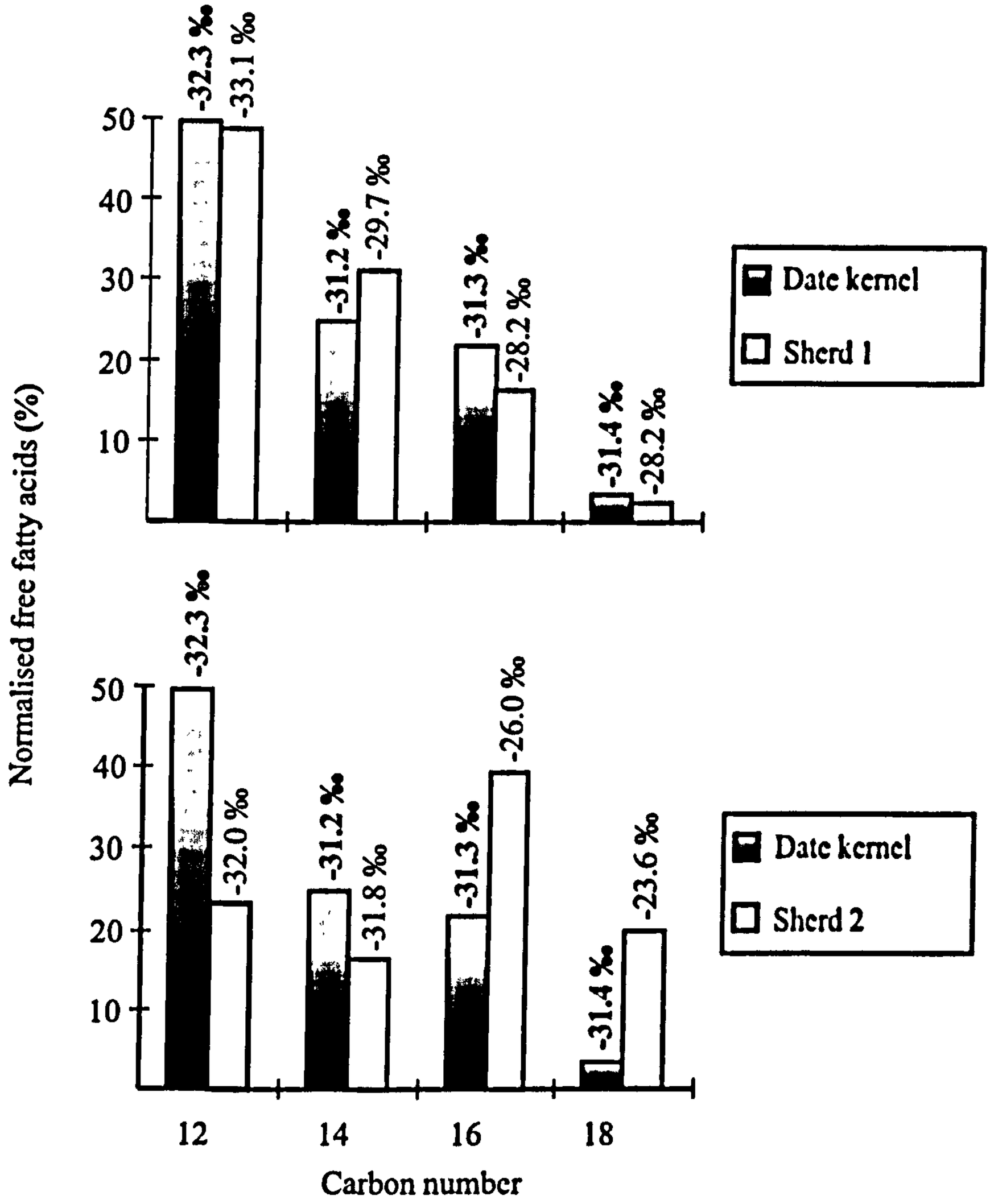

Figure 4.51 Comparison of fatty acid compositions and $\delta^{13}C$ values for two potsherds. Sherd 1 displays a fatty acid distribution and $\delta^{13}C$ values similar to that of date kernels. Sherd 2 contains relatively more $C_{16:0}$ and $C_{18:0}$, and exhibits different $\delta^{13}C$ values for the two components.

Full assessment of the fatty acid distributions of the 79 sherds that contain diagnostic palm kernel lipid appeared to suggest that the sherds could be classified to three groups: (1) Only palm kernel lipids present, with little or no loss in their fatty acid composition during vessel use/burial (i.e. high $C_{12:0}$ and $C_{14:0}$ and low $C_{16:0}$). (2) Only palm kernel lipids are present, with distributions

suggesting the loss some of the shorter chain fatty acids during vessel use/burial. (3) Palm kernel lipids were mixed with another oil/fat, possibly accompanied by a depletion of the shorter chain fatty acids.

Compound specific $\delta^{13}C$ values help to confirm whether or not the mixing of lipids has occurred in the vessels. In the examples shown in Figure 4.51, sherd 1 exhibits a fatty acid composition and $\delta^{13}C$ values similar to those of the reference date kernels. However, the fatty acid composition of sherd 2 contrasts significantly with that of the date reference materials, suggesting it is substantially degraded (resulting in lower abundances of $C_{12:0}$ and $C_{14:0}$), or another fat/oil is also present in the extract (resulting in more $C_{16:0}$ and $C_{18:0}$). If the former were the case, then the stable isotopes would be similar to the reference materials. However, the $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$ are significantly different to those from the reference palm kernels, while the $\delta^{13}C$ values of the $C_{12:0}$ and $C_{14:0}$ are comparable to the reference materials, which together suggests the mixing of palm fruit lipids with another fat or oil (in this case, with less depleted $\delta^{13}C$ values for the $C_{16:0}$ and $C_{18:0}$).

To assess the number of sherds that contain evidence for the mixing of commodities, the $\delta^{13}C$ values and fatty acid compositions are assessed for each individual sherd; Figure 4.52 summarises this graphically. The fatty acid compositions of the sherds have been standardised to range (i.e. $FA_{standardised} = [FA_{sample} - FA_{minimum}] / [FA_{maximum} - FA_{minimum}]$; therefore $FA_{standardised}$ has a range of between 0 and 1). The same was calculated for the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values from the sherds (i.e. $\delta^{13}C_{standardised} = [\delta^{13}C_{sample} - \delta^{13}C_{minimum}] / [\delta^{13}C_{maximum} - \delta^{13}C_{minimum}]$. Hierarchical cluster analysis was performed on the fatty acid distribution (single linkage), producing the dendrogram based on Euclidean distances. The standardised fatty acid compositions and $\delta^{13}C$ values are placed adjacent to the dendrogram for direct comparison of the data.

Seven distinct clusters were observed, and the major ones have been colour coded. These can be seen to correspond to three different fatty acid

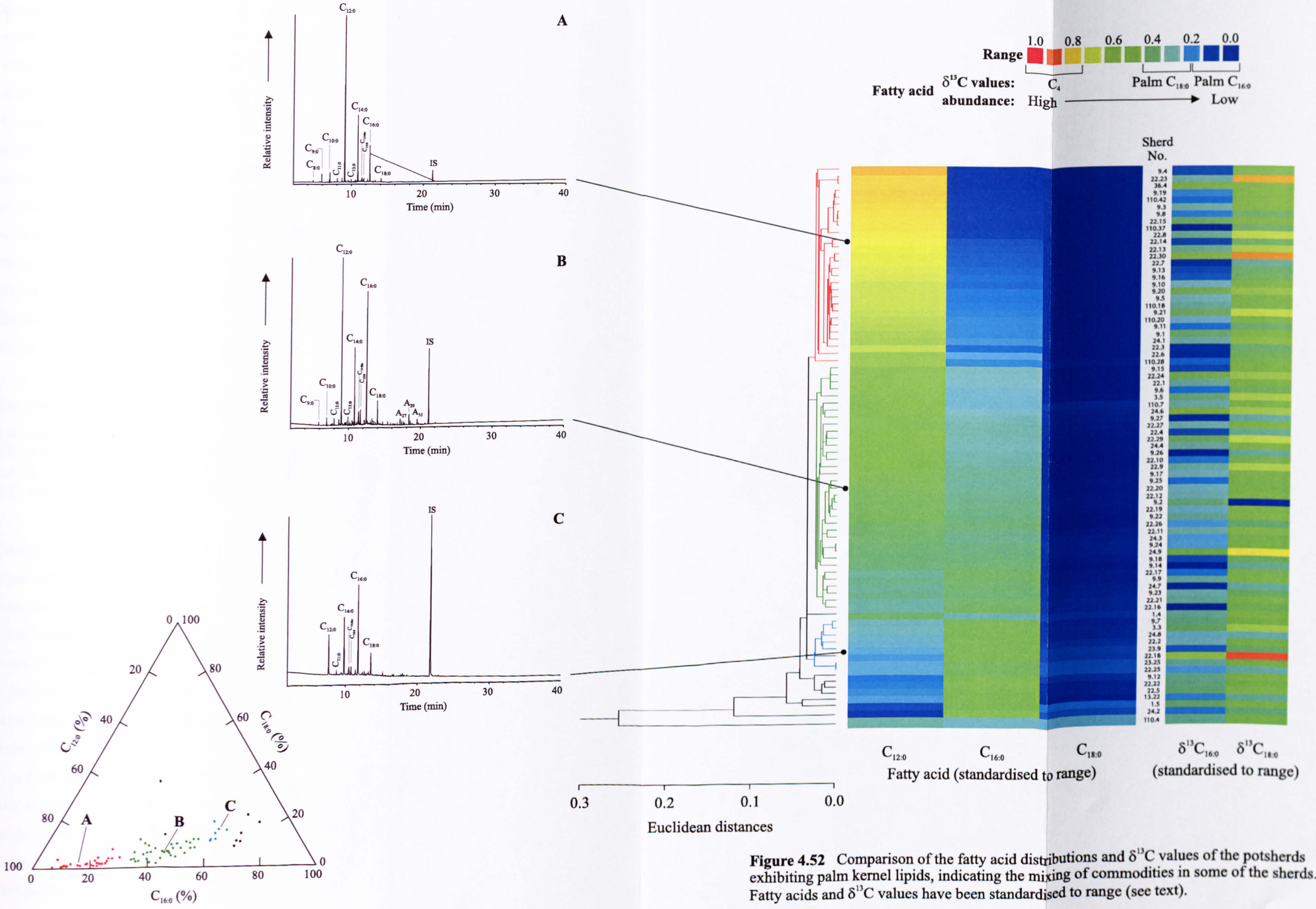

Figure 4.52 Comparison of the fatty acid distributions and $\delta^{13}C$ values of the potsherds exhibiting palm kernel lipids, indicating the mixing of commodities in some of the sherds. Fatty acids and $\delta^{13}C$ values have been standardised to range (see text).

compositions, the red cluster corresponds to typical palm kernel distributions, with $C_{12:0}$ dominating, and an example of which is shown in chromatogram A, and accounts for 29 (37%) of the sherds. The green section of the dendrogram accounts for 35 (44%) of the sherds, and corresponds to high abundances of the $C_{12:0}$ and $C_{16:0}$ fatty acids (as seen in chromatogram B) and includes sherds that have lost a small proportion of the $C_{12:0}$ or also contain lipids from a source other than palm kernel (but the $C_{18:0}$ is always present in relatively low abundances). The blue cluster is similar to the green one, but includes sherds with an even lower abundance of $C_{12:0}$ (e.g. chromatogram C) and significantly more $C_{16:0}$ (and slightly more $C_{18:0}$ in some of the sherds), and accounts for 8 (10%) of the sherds. Finally, the remainder (7 potsherds, 9%) includes sherds that have equal abundances of the three fatty acids (and hence plot towards the centre of the triangular plot), or have relatively low abundances of $C_{12:0}$.

The standardised $\delta^{13}C$ values range from 1.0 to 0.0 such that:
1.0 - 0.8 corresponds to typical C_4 values
0.8 - 0.4 mixed C_3/C_4 *or* less depleted C_3 values
0.4 - 0.2 C_3 values typical for the $\delta^{13}C$ value of $C_{16:0}$ palm kernel lipids
0.2 - 0.0 C_3 values typical for the $\delta^{13}C$ value of $C_{18:0}$ palm kernel lipids

Twenty-four of the 79 sherds (30%) yielded fatty acids with $\delta^{13}C$ values and compositions that are indicative of pure palm kernel lipids, 15 of which exhibit some loss in the $C_{12:0}$ component. Four of the vessels (5%) exhibited $\delta^{13}C$ values indicative of the addition of C_4 fatty acids; the addition of plant/animal material in sherds 24·9 and 22·18 is also accompanied by the significant changes in the fatty acid distributions as well. This can be seen in Figure 4.52, where these latter sherds plot towards the bottom of the diagram (in the green and blue areas of the dendrogram), as such, this is evidence for the addition of $C_{16:0}$ and $C_{18:0}$ from another lipid source. However, with sherds 22·23 and 22·30, the fatty acid distributions are typical of a pure palm kernel source, and yet the stable isotope values (-20.6‰ for the $C_{18:0}$ of 22·30, the least depleted $\delta^{13}C$ value for all of the sherds) are indicative of the addition of substantial proportion of C_4 stearic acids. In all four of these latter sherds, the $\delta^{13}C_{16:0}$ is

significantly more depleted than the $\delta^{13}C_{18:0}$, thus indicating minor addition of fats from animals mainly raised on a C_4 diet.

This detailed assessment illustrates the necessity of investigating the stable isotope values of the individual fatty acids for each sherd. The fatty acid compositions for sherds 22·23 and 22·30 were indicative of a pure palm kernel origin, however only through the determination of the $\delta^{13}C$ values was the mixing of commodities actually been detected.

Based on the variability in the stable isotope data, regardless of their fatty acid distributions, the remainder of the vessels (51; 65%) appear to have other C_3 or small quantities of C_4 fatty acids present (even after the variability of the $\delta^{13}C_{12:0}$ is taken into consideration). This indicates that the majority of the sherds with characteristic palm kernel lipids were either used to process small quantities of other commodities in the vessels at the same time, or that the re-use of vessels at Qasr Ibrim was relatively commonplace.

4.8 SUMMARY

The environmental reference materials analysed in Chapter 3 were used to classify the extracts from the sherds to lipid origin. It is a feature of the arid environment at Qasr Ibrim that large numbers of sherds contained significant concentrations of lipid, thus enabling lipid origin to be assessed.

General

- A large quantity of sherds (209/313; 67%) yielded appreciable concentrations of lipid (>5 $\mu g\ g^{-1}$)
- These are dominated by saturated fatty acids, indicating that extensive oxidation of the double bonds in the unsaturated fatty acids has occurred during vessel use/burial, detected through the presence of α,ω-dicarboxylic acids, 9,10 dihydroxyacids and short chain ($\leq C_9$) fatty acids

- Extensive hydrolysis of the acyl lipids to free fatty acids and glycerol has occurred

The Meroitic Period

- Fifty-five out of 120 (46%) of the sherds yielded significant concentrations of lipid (>5μg g^{-1})
- The sherds are dominated by residues indicative of predominantly animal fats (51%); a further 15% exhibited predominantly plant lipids and 7% contained characteristic palm kernel lipids
- The analysis of both the $\Delta^{13}C$ and $\delta^{13}C$ values has indicated the possibility of the mixing of C_3/C_4 plant lipids and fats from animals fed on C_3/C_4 diets
- Seventeen of the sherds that exhibited fatty acids indicative of predominantly animal fats also contain other diagnostic plant compounds (e.g. sterols, *n*-alkanes, benzoic acid derivatives and levoglucosan), suggesting that plants were also processed in these vessels
- Five sherds from context 18013 contain coprostanol with three of these also yielding other 5β-stanols; this suggests the secondary use of these vessels as chamber pots
- Tentatively, of the sherds that plot in the predominantly animal ellipses more than two thirds came from vessels that were used to process sheep/goat rather than cattle products
- The stable isotope data show that the majority of the extracts are primarily C_3, although a few sherds exhibit $\delta^{13}C$ values that are indicative of the processing of both C_3 and C_4 commodities

The Early Post-Meroitic Period

- Eighty-five out of 94 (90%) of the sherds yielded significant concentrations of lipid (>5μg g^{-1})
- The sherds are dominated by residues characteristic of palm kernel lipids (75%), with sherds containing predominantly other plant lipids accounting for 21% and sherds containing predominantly animal fats accounting for 2% of the total

- The majority (98%) of the potsherds containing palm kernel lipids are wheel-made wares
- The sherds containing lipid from other commodities exhibited $\delta^{13}C$ values indicative of mixtures of C_3 or C_3/C_4 fatty acids

The Post-Meroitic Period

- Sixty-nine out of 99 (70%) of the sherds yielded significant concentrations of lipid (>5μg g^{-1})
  - 45% of the sherds contain predominantly animal fats, whereas 25% contain predominantly plant lipids and 16% yielded palm kernel lipids
  - The majority of the sherds that lack palm kernel lipids exhibit $\delta^{13}C$ values indicative of a predominantly C_3 source, although four of the sherds contained fatty acids with significantly higher $\delta^{13}C$ values than those obtained from the C_3 plant reference materials
  - Six out of 31 (19%) of the sherds that exhibit fatty acid compositions indicative of animal fats also contain diagnostic plant compounds (e.g. derivatives of benzoic acid, *n*-alkanes and sterols)

***n*-alkanes and the insoluble fraction**

- The $\delta^{13}C$ values of the *n*-alkanes become more depleted with increasing chain length
- Overall, the *n*-alkanes exhibited higher $\delta^{13}C$ values in the Post-Meroitic period compared to the Meroitic period
- The insoluble fraction of all of the vessels that contained palm kernel lipids upon saponification yielded a series of 3-hydroxy fatty acids with a similar distribution to that of the free fatty acids in the TLE (i.e. saturated components in the range $C_{12:0}$ to $C_{16:0}$ predominating). Short-chain 3-hydroxyacids are also present (in much lower abundances), always with the C_9 component predominating
- The 3-hydroxy acids are only present in very low relative abundances in the sherds that contain lipids (in the TLE) of predominantly animal or plant (non-palm) origin
- Four out of 50 of the sherds also contain abietic and dehydroabietic acids, characteristic of coniferous resins

The vessels that contained palm kernel lipids

- The mean abundances and variances of the saturated fatty acids from the pottery vessels and the palm reference materials are not significantly different
- The $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values in the pottery vessels have variances that are significantly different from the $\delta^{13}C_{12:0}$ and $\delta^{13}C_{14:0}$ values. After standardising the values (by subtracting the $\delta^{13}C$ value from the corresponding $\delta^{13}C_{12:0}$) it was found that some of the vessels contain mixtures of palm kernel fatty acids and the fatty acids from other commodities
- Even though the fatty acid distributions suggest that the sherds could be categorised into distinct groups: i.e. pure palm kernel, slightly degraded/mixed palm kernel and highly degraded/mixed palm kernel lipids (as seen via the hierarchical cluster analysis), the $\delta^{13}C$ values indicate that the mixing of commodities in vessels (i.e. multiple uses and possibly re-use) was in fact widespread (occurring in *c.* 70% of vessels at Qasr Ibrim)

Chapter 5.

The Pottery Vessels: Discussion

5.1 Evidence for the Processing of Palm Fruit

As discussed previously (Section 1.4), three species of palm are reported to have been utilised in Nubian antiquity: the date palm (*Phoenix dactylifera* L.), the dom palm (*Hyphaene thebaica* (L.) Mart.) and the argun palm (*Medemia argun* Württemb.). Although it has been stated that the argun was exported from Nubia to Egypt (Brewer *et al.*, 1994:51) and that the fruit had to be buried in the ground until they gained a sweet taste (Täckholm *et al.*, 1956:298-299), palaeobotanical evidence for the argun palm has rarely been reported from archaeological sites in the region, and it may even be that these palm fruit samples were misidentified in the past (Clapham, *pers. comm.*).

These palms, especially the date palm, can survive in dry climates provided that there is sufficient water through irrigation or a high enough water table (Brown, 1924:7-8; Nixon, 1951), and are therefore well suited to the Egyptian and Nubian environment (Fig. 5.1). In modern Nubia, it has been recorded that palms have been: offered as collateral for loans; given as gifts; used in the manufacture of baskets, ropes, rugs, furniture, and fencing; and used for animal feed and fuel (Treloar, 1884; Beadnell, 1909:218; Adams, 1977:53; Gale *et al.*, 2000:347-348). For this reason, in modern times, the date palm has been

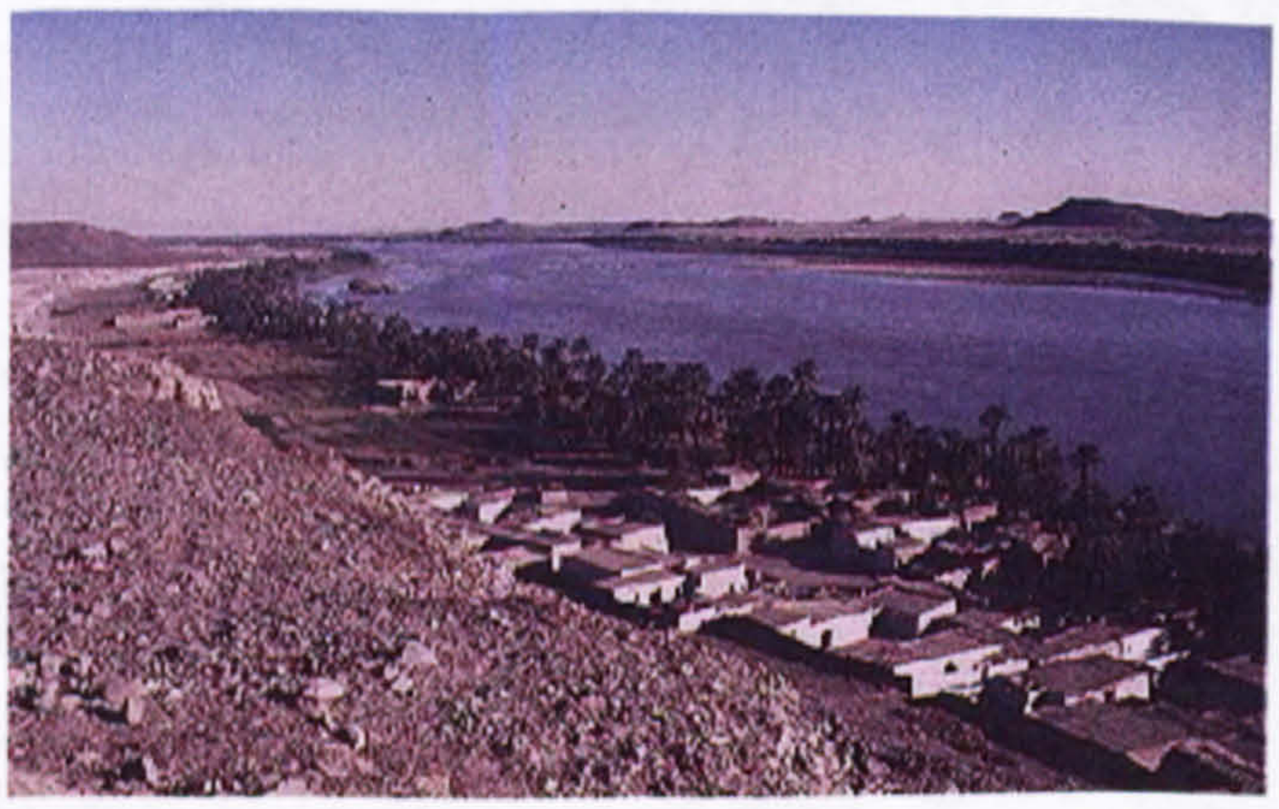

Figure 5.1 Palm trees lining the Nile at Jebel Sesibi, situated North of Kerdurma and the Third Cataract.

economically important, with large-scale commercial cultivation occurring in Egypt (e.g. Nixon, 1951) and has even been described as the 'tree of life for the Arabs' (Dahlgren, 1944:24).

The importance of the date to Nubian economy and society would be expected in the past as it is today. However, direct evidence for the processing of dates has not been proven to any great extent through the archaeology of the region (Welsby, 1996:160). Indeed, although Strabo says that the palm was found "in abundance" in Aithiopia [Nubia] (Strabo and Jones, XVII:2.2), the primary archaeological evidence for this has been through the excavation of goods manufactured from the palm tree and the desiccated remains of the fruit (e.g. Murray, 2000b:618-619).

Due to the fact that the timber from the dom palm is stronger than that of the date palm, it has been preferred in Upper Egypt for the construction of furniture and buildings (Brewer *et al.*, 1994:50; Gale *et al.*, 2000:347-348). However, it is the fruit that provides one of the main economic uses of the palm tree. The date fruit is a berry, and comprises a pericarp and seed. The fleshy pericarp has a high carbohydrate content (*c.* 75-80% dry weight), containing glucose and fructose (Samarawira, 1983), with the remainder of the organic portion of the fruit primarily being proteins and lipids. Economically, the fruit of the dom palm is less useful than that of the date palm due to the fact that the plant is more sensitive to changes in environment, producing fruit of varying sizes (Brewer *et al.*, 1994:50), but of a distinctive taste (Beadnell, 1909). Plates VII to IX show pictures of archaeological date fruit and a modern dom fruit.

In Ancient Egypt, from the Early Dynastic period onwards (*c.* 3000-2670BC), there are depictions of palms in tombs (Täckholm *et al.*, 1956:211) as well as linguistic evidence for the word for "date" (Murray *et al.*, 2000:619). The first evidence for the presence of date and dom palm fruit from archaeological sites appears in the Predynastic period (*c.* 4800-3050BC) and Late Palaeolithic, respectively (Vartavan and Asensi Amorós, 1997:134; Murray, 2000b:621), although the cultivated fruit tend to appear more commonly in the Middle

Plate VII. An archaeological date. Within the desiccated flesh, the date kernel is still in place.

Plate VIII. Archaeological date kernels.

Plate IX. Modern dom fruit.

The scale in all of these Plates is 5 cm.

Kingdom (*c.* 2040-1650BC) onwards (Täckholm *et al.*, 1956:219, 284; Vartavan and Asensi Amorós, 1997:193).

It has been noted, primarily from ethnographic work conducted in the recent past, that there are many ways that the fruit could have been utilised in antiquity, several of which would have required the use of pottery vessels. For instance, the fruit can be eaten by itself or mixed with another foodstuff, e.g. barley (Treloar, 1884:18). It has been reported that people in some parts of the Sahara mixed the pulverised dom fruit with an infusion of dates to produce a cooling drink used to reduce fever (Osborn, 1968). In ancient Egypt there is also evidence for cakes made from the dom fruit (Brewer *et al.*, 1994:50). Furthermore, the sugars can be extracted by boiling to produce a date syrup that could then be either added to food or to beer in order to sweeten them (e.g. Brewer *et al.*, 1994:48), although there is some debate as to whether dates were used in this latter way in New Kingdom beer in Egypt (Samuel, 2000:566-567). In modern Sudan (also attested to in the past) sorghum is also often used to produce a beer, but dates do not appear to have a role in its production (Dirar, 1993:242-248). Alternatively, dates or date syrup were fermented to produce a date wine (Dirar, 1993:280-290) which could then be easily fermented again into a liquor (Beadnell, 1909:218; Murray *et al.*, 2000:592).

The date kernels can also be soaked and fed to animals or, in times of famine, ground up to make a date flour (Beadnell, 1909:218). However, given the large quantity of fodder/forage such as barley and sorghum available at Qasr Ibrim, it is unlikely that palm kernels would have been fed to animals. Similarly, the archaeological evidence does not indicate that the people at Ibrim had any need to resort to grinding the date kernels to make a flour. It is also known that in antiquity, dom kernels have been worked into artefacts such as buttons (Brewer *et al.*, 1994:50), and although there are large quantities of the outer casings of the dom fruit scattered all over the site, the kernel itself is rarely found. However, no artefacts or waste material from the fashioning of the palm kernels have been recovered from excavations, and given the level of organic preservation at the site, the remains of this sort of activity would be expected to be found if it had occurred at all (*cf.* Rowley-Conwy, 1994).

The production of date wines in the Sudan involves the fermentation of the actual fruit in a process that may take up to a fortnight to complete. It has already been stated (Section 1.2) that the people of ancient Nubia had some economic and cultural links with Egypt, but much of their culture has to be explained within a Sudanese context (e.g. Edwards, 1996, Edwards, 1998b). Following from this, there is little literary evidence for the drinking of date wine in Ancient Egypt, and even in modern times, the date wines produced in Sudan are not seen in Egypt (Darby *et al.*, 1977b:614).

Dirar (1993, 1994) describes in detail the processes utilised by modern Sudanese people in the small-scale production of date wine, and the following is from these references. There are three main types of modern date wines: *dakkai*, *sherbot* and *nebit*. *Dakkai* can be fermented further to produce the more alcoholic drink, *araki*, which is essentially a liquor. Figure 5.2 is a flow diagram summarising the stages in the production of date wine. In the case of *sherbot* and *nebit*, this includes the gentle boiling and continuous stirring of the dates, and in the case of *dakkai*, boiling water is simply added to the dates and then the vessel sealed. The length of fermentation varies for each type of date wine, but typically 3 - 6 % ethanol (v/v) is achieved within a few days.

(Dirar, 1993:282) states that:

"Date wines are best prepared in locally made earthenware pots that are impervious. The jars are specially kept for this purpose and other jars, particularly those used for sorghum fermentations, are avoided. The wine jar is given a special treatment, involving thorough washing and the sun drying of the container. Then, one or two dom fruits are burned until they give a heavy smoke; they are then dropped into the jar, which is then sealed until the fire has completely consumed the dom fruit including the inner kernel."

This raises several points worthy of highlight or explanation. Firstly, "impervious" earthenware pots are used (i.e. relatively non-porous), which would suggest that wheel-made would be preferred over hand-made wares. As was shown in Chapter 4, only 2/79 of the vessels that contained characteristic

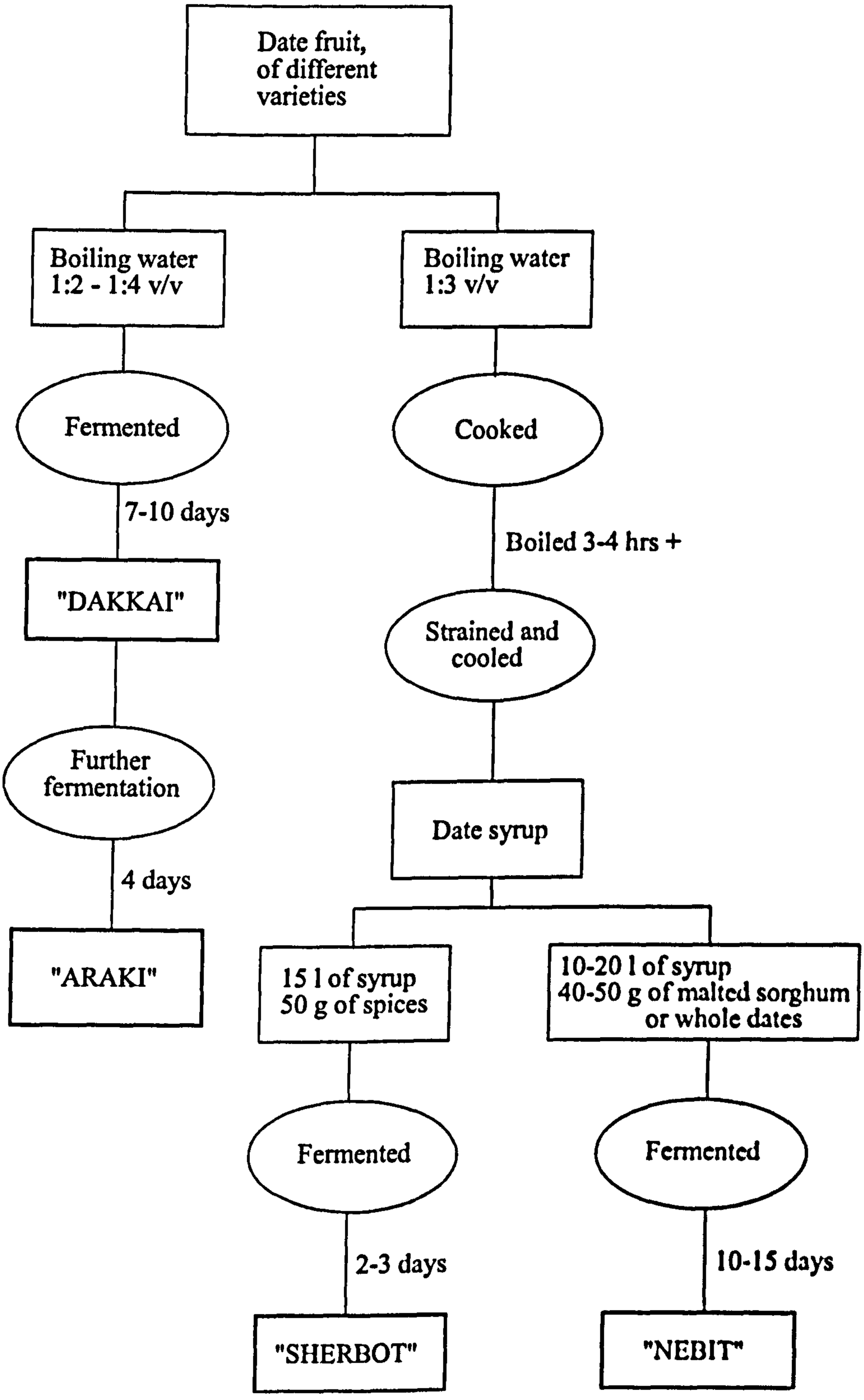

Figure 5.2 The process of making Sudanese date wines (after Dirar, 1993; Dirar, 1994).

palm kernel lipids were of the hand-made wares. Secondly, jars "used for sorghum fermentations are avoided". This indicates that the vessels had not been previously used in the production of grain beers. As was shown in Section 4.7, there is little evidence for the extensive mixing (either contemporaneously or through re-use of the vessel) of fatty acids from C_4 cultivars and palm kernels, with only four sherds out of 79 exhibiting $\delta^{13}C$ values indicative of mixing in the manner described. Thirdly, "one or two dom kernels are burned" inside the vessels, the exact reason for this is not clear. However, the burning of these kernels in this manner would not contribute to the absorbed lipid residues extracted from the archaeological pottery.

During the production of date wines, modern families typically use between 18-30 kg of dates in one large jar. The R25 pots and jars that were analysed in this study were typically 20-40 cm in height and do not have a sufficiently large volume to process such quantities of dates (Adams, 1986b; Adams, 1986a). Whilst it is possible that the ancient Nubians at Ibrim could have made date wine on a smaller scale than their modern counterparts, given that it can take 4 to 16 days to produce, intuitively it seems unlikely that so much effort would be invested in producing such small quantities of date wine.

A further important use of the date palm fruit would have been as a source of sugar. Dates are a very sweet tasting fruit, and have been revered in antiquity for their sugar content. Sugar cane was not introduced into Egypt until the 7th century AD, and even then only some sections of society actually had access to the sugar cane (Darby *et al.*, 1977a:428-429). Hence, in economies that did not have sugar, fruit such as dates and figs were of a high economic value. The extraction of date syrup would proceed using methods utilised in the production of *sherbot* and *nebit* (it is the date syrup that is fermented to produce these wines), i.e. the dates are boiled gently until the fruit disintegrates completely (sometimes needing two 'boiling sessions'), and then strained through a cheesecloth and cooled.

The pottery vessels used in the processing of date syrup would need to have been as non-porous as possible so that little of the syrup was lost. As with the production of date wines, wheel-made wares would have been preferred for this reason. Furthermore, this process could have been completed in much smaller vessels (compared to date wine production) since the syrup could then be utilised straight away. During the processing of the palm fruit for wine production or sugar extraction, it would be expected that both lipids and sugars from the fruit would be absorbed in the vessel wall.

The pottery vessels shown to contain fatty acids characteristic of palm kernels exhibited high concentrations of absorbed lipid, typically between 0.5 to 1.0 mg g^{-1} of potsherd. Such lipid concentrations are similar to those detected in the vessels that contained lipid distributions indicative of animal fats, but were almost a factor of 10 greater than the lipid concentrations from the sherds exhibiting plant-like fatty acid distributions (see Fig. 4.1). This indicates that the pottery vessels in which the palm fruit were processed had been used intensively for this activity. This is unusual, as the dedicated use of vessels in the processing of specific plants in British/European archaeological pottery is rarely, if ever, observed. Although plants such as cabbage have been detected from a wide range of British archaeological pottery vessels through the identification of the components of their epicuticular leaf waxes (e.g. Evershed *et al.*, 1991; Evershed *et al.*, 1992; Evershed *et al.*, 1994), these lipids are usually detected in association with animal fats. However, some animal products, such as milk and bovine/porcine fats, have been detected in prehistoric British pottery, indicating that the continued use of specific vessels in the processing a range of commodities has occurred (e.g. Dudd and Evershed, 1998; Dudd *et al.*, 1999).

After considering the archaeological, ethnographic and biomolecular evidence, it would appear that the pottery vessels displaying palm kernel fatty acid distributions would most likely have been utilised for the extraction of a syrup. Syrup made from the palm fruit would have been of extremely high economic and social value in the region. Archaeologically, there is growing evidence for Qasr Ibrim being a cult centre during the periods investigated herein. It is not

known whether this would have had an influence on the production and distribution of this syrup, for example, through control exercised by the local elite. Regardless of whether the syrup was produced on a relatively small scale (for local consumption by a few people) or on a relatively larger scale possibly involving trade/exchange, this would have been their main method of sweetening foodstuffs and date syrup would have constituted a very important commodity to the people of the region.

5.2 Changes in Vessel Use

In Chapter 4 the extracts from the sherds were classified as to being either predominantly animal fats, plant lipids or palm kernel lipids. Furthermore, it was possible to tentatively distinguish between sheep/goat and cattle fats extracted from the archaeological bone, principally through the $\Delta^{13}C$ (defined as $\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) values and their $C_{16:0}/C_{18:0}$ abundance ratios. The pottery vessels yielded lipids that were indicative of the processing of a wide range of commodities throughout the Meroitic to Post-Meroitic periods, summarised in the graph of the $C_{16:0}/C_{18:0}$ abundance ratios plotted against the $\Delta^{13}C$ values of the lipid components, shown in Figure 5.3. The following sections discuss the changes in vessel use over time based on these classifications, differences in the use of ware types, and finally, changes in stable isotope values over time.

5.2.1 Temporal changes

By removing from the data set the potsherds that contained characteristic palm kernel lipid compositions, and performing data analysis on the remaining sherds, it is possible to determine whether the unusually large proportions of sherds containing palm kernel lipids have any statistical affect on the over all data set. It was revealed in Chapter 3 that the $C_{16:0}/C_{18:0}$ abundance ratios of lipid extracts of the animal and plant reference materials were significantly different. Therefore, by testing the significance of any temporal differences in

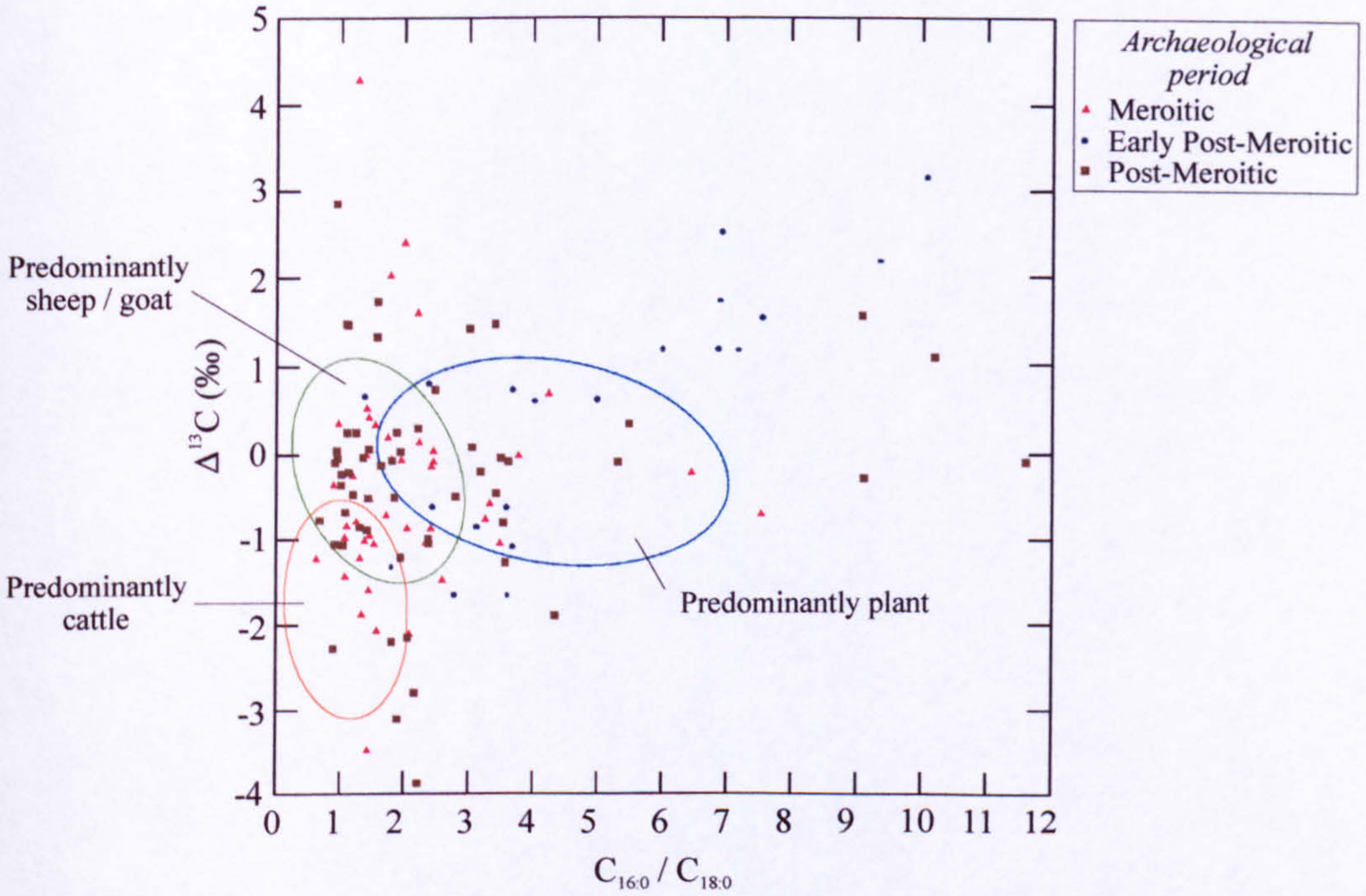

Figure 5.3 $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) of the all of the sherds plotted against their $C_{16:0}/C_{18:0}$ abundance ratios, distinguished by archaeological period. The orange ellipse is generated from the values obtained from the bovine reference materials, the green ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3). The labels indicate the portions of the graph discussed in this section.

these fatty acid ratios, the relative importance of the two broad commodities can be revealed statistically. Figure 5.4 shows the change in the $C_{16:0}/C_{18:0}$ ratio of sherd lipid extracts over time (not including those with characteristic palm kernel lipids). From this figure it can be seen that the Meroitic pottery exhibits the lowest $C_{16:0}/C_{18:0}$ mean value at 2.18, and that this increases to 5.58 in the Early Post-Meroitic ("EPM") period and then decreases to 2.49 in the Post-Meroitic period (Fig. 5.4). This shift is highly significant (ANOVA; $df = 2$; $F\ ratio = 13.4$; $p<0.0005$), and when Bonferroni protection for multiple tests is applied, the variation between the Meroitic and EPM periods is highly significant ($p < 0.0005$), as is the difference between the EPM and Post-Meroitic periods ($p < 0.0005$). However, there is no difference between the Meroitic and the Post-Meroitic periods. Furthermore, when all of the sherds are included in the data analysis (Fig. 5.5), the mean value for the $C_{16:0}/C_{18:0}$ abundance ratios increase slightly in the Meroitic and Post-Meroitic periods (to

2.31 and 2.84 respectively) and the mean value during the EPM period increases to 7.23. This too is highly significant (ANOVA; $df = 2$; $F\ ratio = 48.7$; $p<0.0005$), with the differences in the mean $C_{16:0}/C_{18:0}$ values for the three periods being of the same level of significance as above.

These changes in the $C_{16:0}/C_{18:0}$ ratios are consistent with the processing in the vessels of either an increased quantity of plant material or of a decreased quantity of animal products, during the Early Post-Meroitic period compared with the Meroitic and Post-Meroitic periods. In fact, the higher $C_{16:0}/C_{18:0}$ abundance ratios in the EPM period (compared to the other periods) is due to the absence of large numbers of sherds that contain animal fat residues. This can be seen in Figure 5.5 and in Table 5.1, which shows the variation in the relative proportions of the major classes of lipid residue with archaeological period. Over time, an increase then a decrease in the prevalence of sherds with characteristic palm kernel fatty acids was observed across the assemblage. In order to test whether or not these temporal changes in vessel use are statistically significant, a chi-squared test of association was employed on the data summarised in Table 5.1. A statistical association was discovered between archaeological period and the commodity groups into which the sherds have been classified (Chi-squared; $df = 4$; $\chi^2 = 90.2$; $p < 0.1\%$; Cramér's V = 0.49).

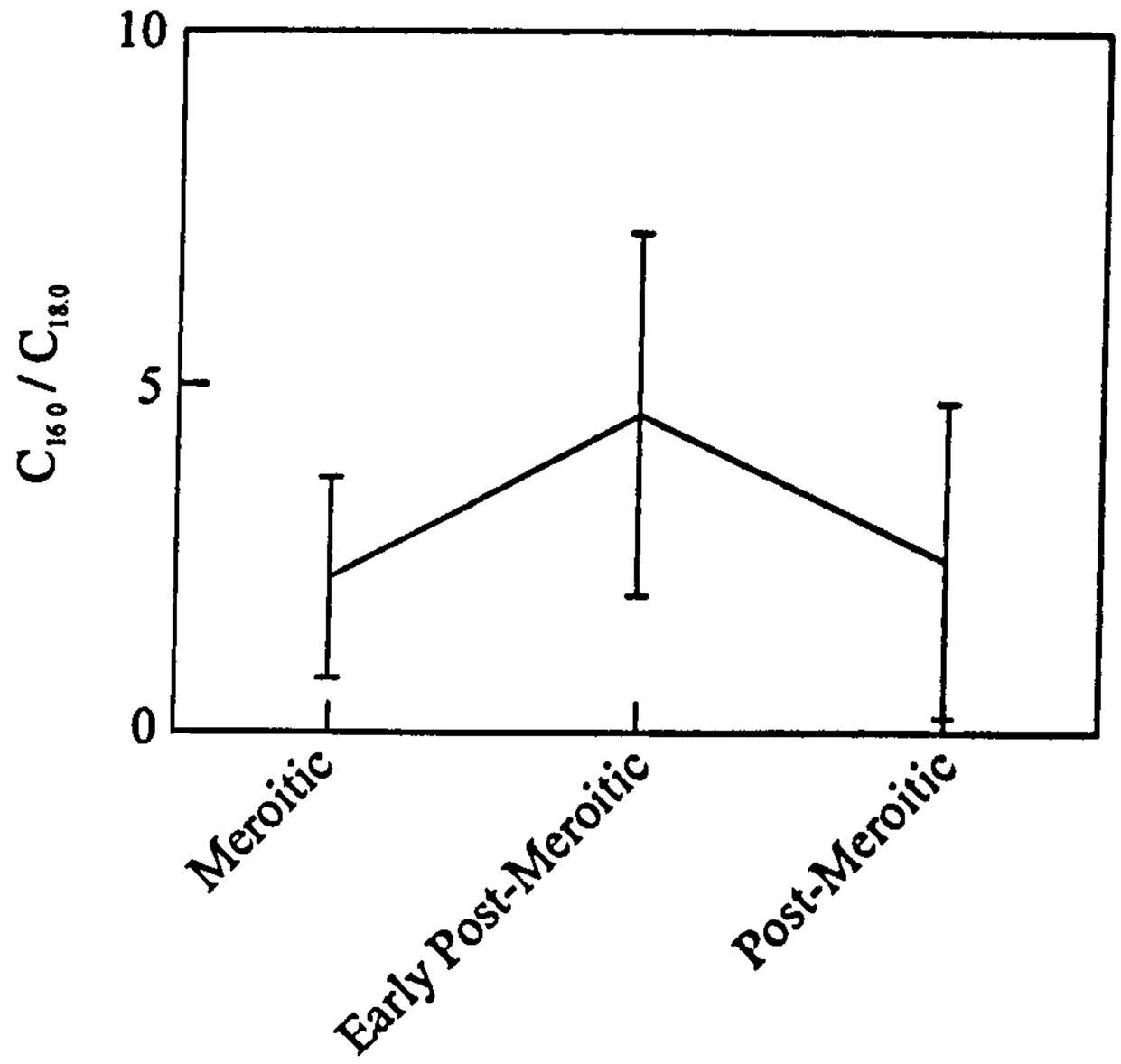

Figure 5.4 The $C_{16:0}/C_{18:0}$ abundance ratio of the extracts from the sherds by period (not including the sherds that contain palm kernel lipids). The vertical bars represent the range and mean values for each period.

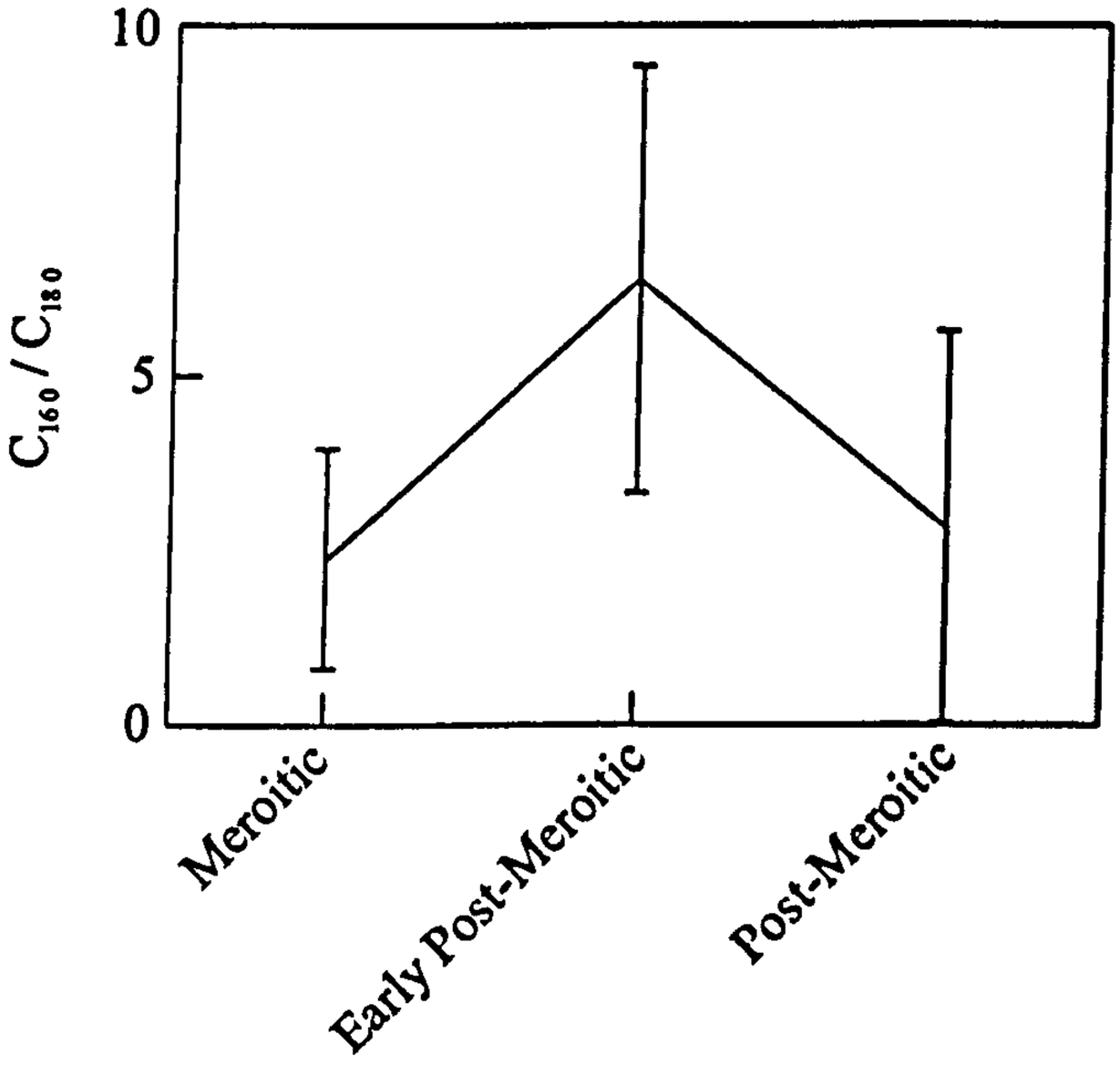

Figure 5.5 The $C_{16:0}/C_{18:0}$ abundance ratio of the extracts from the sherds by period (including the sherds that contain palm kernel lipids). The vertical bars represent the range and mean values for each period.

Table 5.1 Quantity and relative proportions of the major classes of absorbed lipid residues, by archaeological period

Period	Commodity[1]		
	Predominantly animal	Predominantly plant	Predominantly palm kernel
Meroitic	30 (51%)	9 (15%)	4 (7%)
Early Post-Meroitic	2 (2%)	16 (21%)	64 (75%)
Post-Meroitic	31 (45%)	17 (25%)	11 (16%)

[1]Not included in this table are the sherds that are likely to have a mixture of plants/animals and those that cannot be classified as being either animal fats or plant lipids (see Section 3.3). Values in brackets refer to the percentage of all of the residues (including the sherds with 'mixed' commodities).

The reason for the high prevalence of sherds containing palm kernel lipids in the EPM period is intriguing. Qasr Ibrim was situated on top of a craggy outcrop on the edge of the River Nile, and due to its height from the river bed, the immediate vicinity around the site was probably not ideal for agriculture. The opposite was the case for the Western bank of the Nile, and the Nile Valley itself where agriculture would have been possible. Palm trees grown from seed or cuttings require large quantities of water in order for them to grow (Brown, 1924:7). The mature palm trees are very hardy, and can survive periods of flooding and drought (Nixon, 1951); nonetheless, when fully grown they require a water table within a few feet of the ground surface (*ibid.*), and in areas where a low water table exists, irrigation is required. The most effective irrigation method involves flooding the whole surface of the ground, which naturally requires an abundant supply of water (Brown, 1924:8).

In the Meroitic period the only irrigation method available was the *shaduf*, a crude pulley system that was extensively used throughout the region. This device was a counterweighted device that could raise water by *c.* 3 m, thus allowing rather limited irrigation (Welsby, 1996:156). It was not until the Early Post-Meroitic period that an animal driven waterwheel, the *saqia* (Fig. 1.3), was used at Qasr Ibrim (Rose, 1995). The *saqia* could raise water by *c.* 3 to 8 m in height, enabling significantly more efficient irrigation to be achieved, and thus allowing higher yields and greater variety of crops to be grown (Welsby, 1996:156).

If the increase in the prevalence of vessels with palm kernel lipids in the EPM period was directly associated with the introduction of the *saqia*, then it would be expected that a prevalence of these lipids in the Post-Meroitic pottery would also be seen. However this is not the case; only 16% of the sherds from the Post-Meroitic period contained palm fruit lipids, a figure still higher than that found in the Meroitic pottery, yet much lower than in the EPM pottery.

Unless something catastrophic occurred to the palm trees, it is very unlikely that the numbers of the trees would suddenly decline in approximately 100 years. It is more likely that the people at Qasr Ibrim simply did not process dates in pottery vessels in such large quantities, or this practice was undertaken elsewhere on the site. There is archaeological evidence for the cultivation of date fruit in the Post-Meroitic period at Qasr Ibrim, with large quantities of date kernels being found in many Post-Meroitic contexts (Clapham, 1998). Therefore, date fruit were still of importance at Qasr Ibrim, and as such were probably still harvested on a scale similar to that witnessed in the EPM period.

A 'normal' level of use of the vessels, for the processing of palm fruit, can be established through-out the Meroitic to Post-Meroitic periods by considering in detail the individual contexts from the EPM period. The majority of the EPM vessels (95%) from contexts 18009, 18022 and 18024 (all of which are from room 1 in structure 265) displayed fatty acid distributions indicative of palm kernel lipids. However, contexts 18001 and 18003 (also from room 1, but are likely to be of a later date), 18042 and 18047 (from elsewhere in structure 265) have fewer vessels containing characteristic palm kernel lipids (13%). This value is similar to that found in the Post-Meroitic contexts (16%), and appears to indicate that the 13-16% level was typical for the majority of the EPM and Post-Meroitic contexts, and that the high prevalence of palm kernel residues in the sherds from the EPM contexts 18009, 18022 and 18024 is very unusual. There are two possible explanations for this. Firstly, Room 1 of Structure 265 could have been closely associated with the processing of palm fruit during part of the EPM period, or secondly, these sherds have been deposited in Room 1 following 'cleaning' of an initial deposit elsewhere on the site. Further analysis of other EPM contexts would help to elucidate this.

Also apparent from both Table 5.1 and Figure 5.5 is that animal fats are found in large numbers of vessels from both the Meroitic and Post-Meroitic periods. It is very unusual for lipid residues from a pottery assemblage not to be dominated by animal fats, as is often the case with prehistoric vessels from Britain and Europe (e.g. Evershed *et al.*, 1999). The fatty acids extracted from the cattle and sheep/goat bones were statistically differentiated through their $\Delta^{13}C$ values (Section 3.3), and although a certain level of caution has to be attributed to such figures, estimates of the relative proportions of bovid : ovi-caprid fat residues extracted from the pottery vessels can be made. Interestingly, it does appear that more sheep/goat products were processed in the vessels than cattle. This can be seen by considering the number of sherds that plot in the portion of the ellipses from the $\Delta^{13}C$ -v- $C_{16:0}/C_{18:0}$ graph designated **A** and **C** in Figure 5.6, as shown in Figure 5.3 and tabulated in Table 5.2.

The ratio of the numbers of predominantly ovi-caprine : predominantly bovine fat residues extracted from the Meroitic vessels was *c.* 2.3 : 1, while in the Post-Meroitic period it was *c.* 6.0 : 1. These ratios are in accordance with the zooarchaeological evidence from various Nubian sites, indicating that sheep/goat dominated the Nubian economy (e.g. Hewes, 1964; Rowley-Conwy, *pers. comm.*). The limited zooarchaeological work that has been completed at Qasr Ibrim, has indicated that in one area of the site cattle remains overwhelmingly predominate the assemblage (Rowley-Conwy, *pers. comm.*). These bones were from a Meroitic temple precinct that yielded a large quantity of juvenile cattle mandibles. It has been suggested that this may indicate that the 'priests' selectively removed some of the young cattle from the valley economy (*ibid.*), which would be in line with other sites that also had a religious function (e.g. Carter and Foley, 1980; Chaix and Grant, 1993).

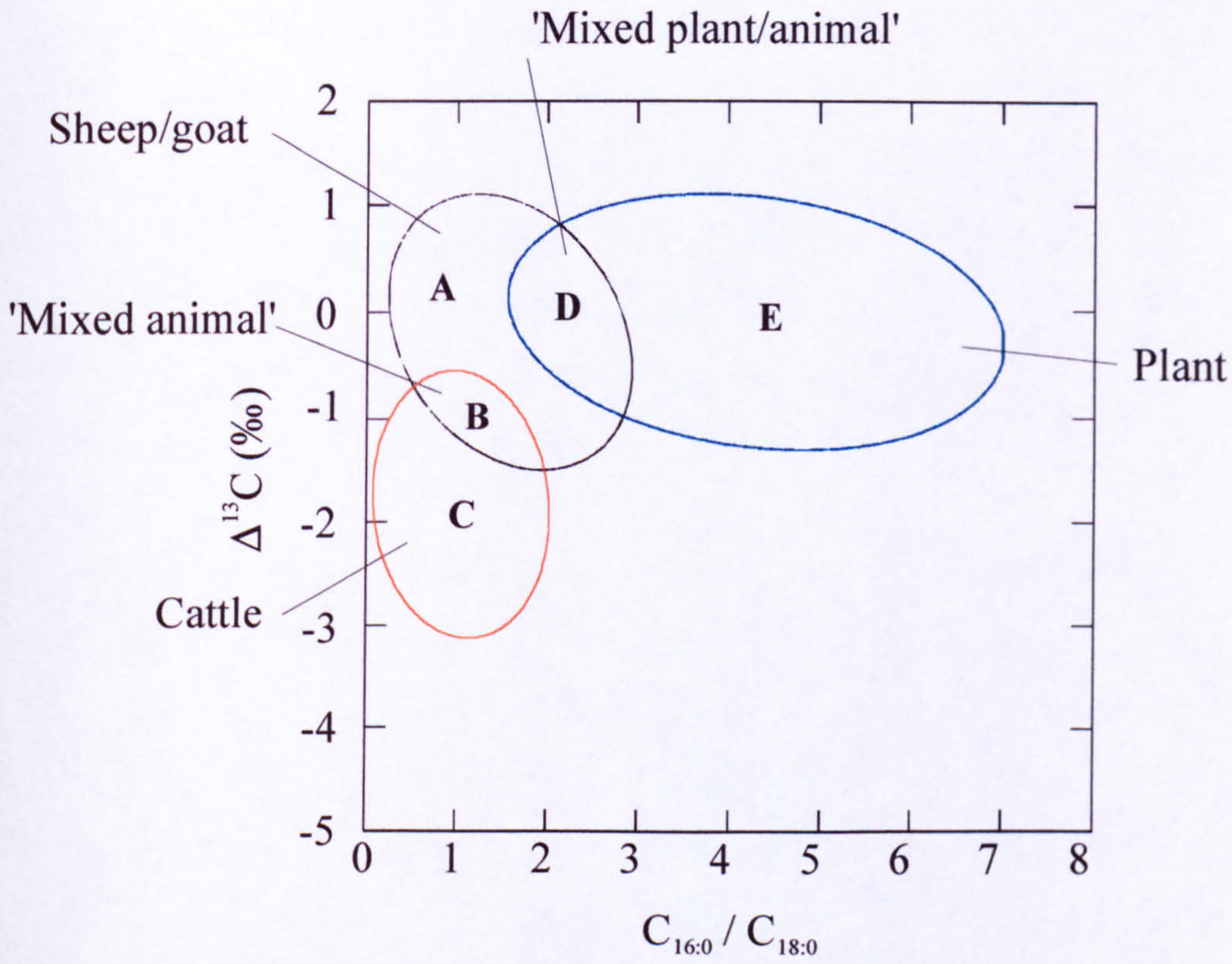

Figure 5.6 The primary fields in the graph of the $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) against $C_{16:0}/C_{18:0}$ abundance ratios. The orange ellipse is generated from the values obtained from the bovine reference materials, the black ellipse from the ovi-caprine reference materials and the blue ellipse from the plant reference materials (Section 3.3).

Table 5.2 Quantity and relative proportions of animal fat residues detected in the pottery vessels, by archaeological period[1]

Period	Animal fat[1]	
	Predominantly ovi-caprine	Predominantly bovine
Meroitic	14 (70%)	6 (30%)
Early Post-Meroitic	1 (100%)	0
Post-Meroitic	18 (86%)	3 (14%)

[1]Sherds within the intersections B and D in Figure 5.6 have been removed from the data set. The % only relate to the values presented in the table and refer to incidence of animal fat residue in sherds from each period.

Using the values in Table 5.2, no association was found between the incidence of fat residues of the two species of animal contained in the sherds and archaeological period (Chi-squared; $df = 1$; $\chi^2 = 1.65$ [with Yates' continuity correction]; $p > 0.1\%$ [not significant]; EPM period not used). Therefore, although it appears that more sheep/goat than cattle products were processed at Qasr Ibrim during the Meroitic and Post-Meroitic periods, there is no statistical difference between the two periods.

The importance of plants to the economy of Qasr Ibrim is illustrated through: (i) the number of vessels with characteristic plant components (e.g. β-sitosterol, *n*-alkanes, derivatives of benzoic acid and levoglucosan), and (ii) the number of vessels with fatty acid distributions indicative of a plant origin. Eleven of the Meroitic vessels (19%) that contained predominantly animal fats also exhibited other diagnostic plant components, whereas none of the EPM vessels and only six of the Post-Meroitic vessels (9%) contained such components (as seen in Chapter 4). This provides direct evidence for the use of the vessels in the processing of both plant and animal products, either contemporaneously or through subsequent re-use of the vessels.

Since both the $C_{16:0}/C_{18:0}$ ratio and the $\Delta^{13}C$ values for the plant reference materials vary considerably between species, it is not possible to assign an origin for such residues to specific plant species (e.g. hyacinth bean / barley). Of course palm kernels have a characteristic fatty acid distribution and hence can be identified, but beyond this, potentially only sorghum and millet (C_4 cultivars) may be identifiable through the $\delta^{13}C$ values of their individual fatty acids. Figure 5.7 shows the range of the $\delta^{13}C$ values of the fatty acids from the sherds that exhibited predominantly plant lipid compositions, overlaid with the $\delta^{13}C$ values for the botanical reference propagules. Mixtures of C_3 and C_4 plant fatty acids in the archaeological vessels are indicated by the fact that the majority of the extracts of the sherds plot between the C_3 and C_4 reference materials. None of the sherds plot within the C_4 region of the graph (top right), indicating that dedicated sorghum or millet processing vessels were not present,

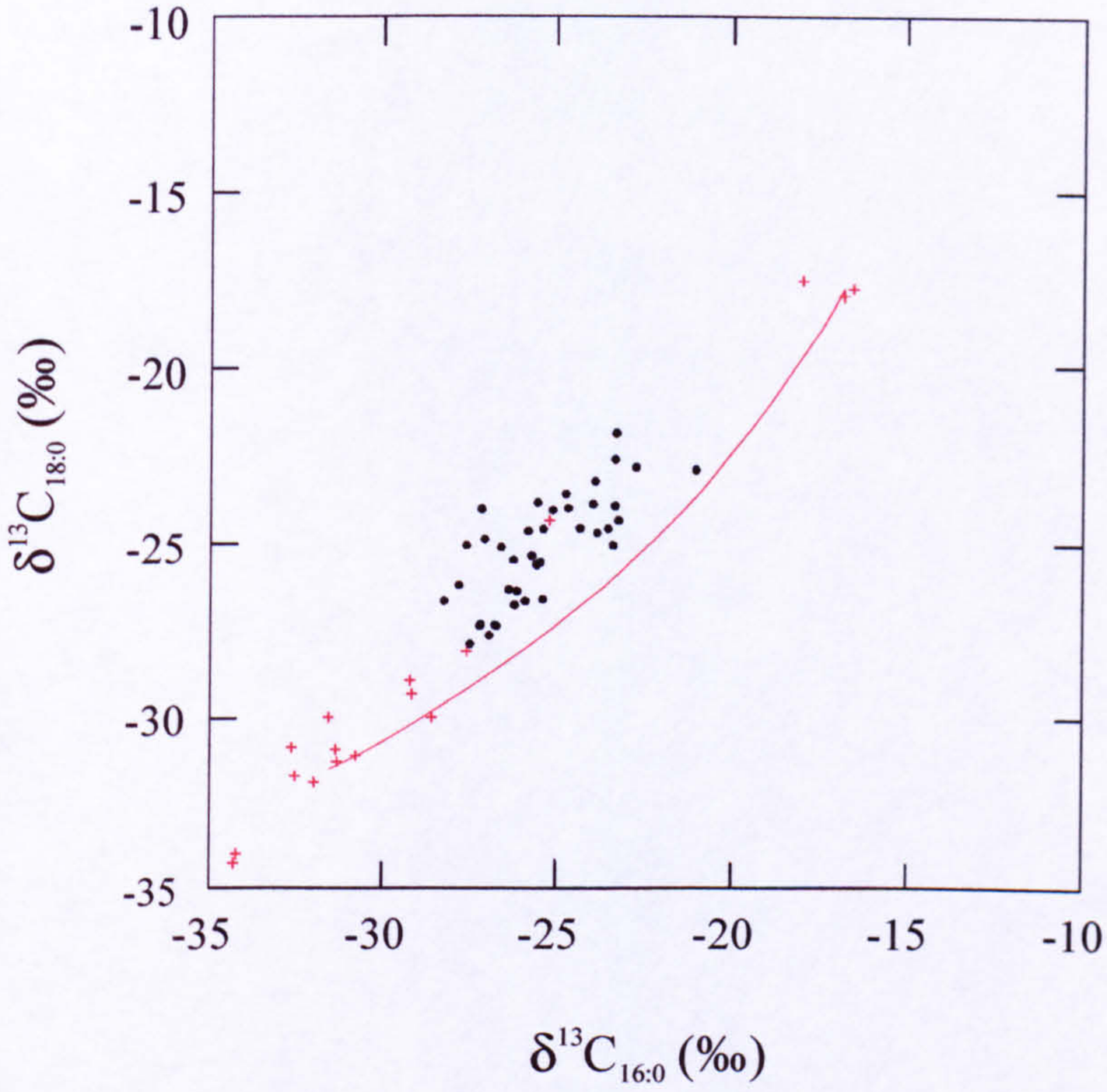

Figure 5.7 Graph of the $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values for the sherds that exhibit predominantly plant fatty acid distributions. The black circles are the sherds, and the red crosses are the botanical reference materials. The red line represents the theoretical mixing of C_3 and C_4 plants (calculated from the mean $\delta^{13}C$ values for the C_3 and C_4 plants, using the formula: $\delta^{13}C_{16:0} = [\delta^{13}C^{3}_{16:0} \times (C^{3}_{16:0}\% \times C^{3}\%) / ((C^{3}_{16:0}\% \times C^{3}\%) + (C^{4}_{16:0}\% \times C^{4}\%))] + [\delta^{13}C^{4}_{16:0} \times (C^{4}_{16:0}\% \times C^{4}\%) / ((C^{3}_{16:0}\% \times C^{3}\%) + (C^{4}_{16:0}\% \times C^{4}\%))]$, where $\delta^{13}C^{3}_{16:0}$ = mean $\delta^{13}C_{16:0}$ value for the C_3 plants, $C^{3}_{16:0}\%$ = the relative abundance of the $C_{16:0}$ in the C_3 plant, $C^{3}\%$ = concentration of C_3 fatty acids in the mixture, $\delta^{13}C^{4}_{16:0}$ = mean $\delta^{13}C_{16:0}$ value for the C_4 plants, $C^{4}_{16:0}\%$ = the relative abundance of the $C_{16:0}$ in the C_4 plant, $C^{4}\%$ = concentration of C_4 fatty acids in the mixture, and the equivalent for the $C_{18:0}$ components). 1.1‰ has been added to the $\delta^{13}C$ values of the modern specimens, in order to account for the post-industrial fossil fuel effect on the $\delta^{13}C$ value of atmospheric carbon dioxide (Friedli *et al.* 1986).

although the mixing of sorghum/millet with other C_3 plants is indicated by the spread of the sherds along the theoretical mixing curve in Figure 5.7.

There are other commodities that can be distinguished through their $\delta^{13}C$ values. Milk is likely to have been an important commodity to Man in prehistory. Due to the different biosynthetic pathways of the fatty acids comprising milk, the $\delta^{13}C_{18:0}$ is *c.* 2.5‰ more depleted compared to the $\delta^{13}C_{18:0}$ in the adipose fat from the same cow. These criteria have led to the successful detection of dairy products residues in pottery vessels from British prehistoric pottery (e.g. Dudd and Evershed, 1998). Furthermore, the $\delta^{13}C_{18:0}$ is also more depleted with respect to the $\delta^{13}C_{16:0}$, such that the $\Delta^{13}C$ value varies between *c.* 3.9 to 5.1‰, thus enabling the detection of ruminant milk fat originating from an animal that had consumed a mixed C_3/C_4 diet.

Only one sherd from Qasr Ibrim yielded fatty acids that had $\Delta^{13}C$ values that fall within the range indicative of milk (Section 4.4.2). Strabo reported that the ancient Nubians ate milk and cheese (Eide *et al.*, 1994:815), and it is known that modern Nubians do the same (Abdelgadir *et al.*, 1998). Nevertheless, based on the stable isotope evidence presented above, dairy products are not represented in the absorbed lipid residues to any extent in the pottery from Qasr Ibrim analysed herein. This is unexpected, given that cattle are known to have played an important role in the Nubian economy during these periods. This could indicate that milk was not collected and stored in pots and jars, or that it was commonly mixed with other commodities (such as barley) and hence the $\Delta^{13}C$ value for the mixture of fatty acids would be different from the expected value for milk.

The insoluble fraction of the sherds was investigated by sodium hydroxide treatment of the extracted sherds in order to see whether it could be used as an additional source of information to distinguish between the commodities that were processed in the vessels. No difference was observed between the insoluble fraction of the vessels that exhibited predominantly plant and those that contained predominantly animal residues, although the vessels containing

palm kernel lipids yielded high abundances of 3-hydroxy fatty acids, whose distributions mirrored those of the free fatty acids (i.e. saturated compounds ranging from $C_{12:0}$ to $C_{18:0}$, with $C_{12:0}$ predominating). The origin of these 3-hydroxy fatty acids is unclear, since if they were the products of β-oxidation as discussed above (Section 4.6), then the vessels that contained predominantly animal fat or plant lipid would also be expected to have more of these hydroxy acids. Although the insoluble fraction did not aid in the differentiation of specific plant oils/animal fats, the 3-hydroxy acid distributions have been shown to provide a further useful means of identifying the processing of palm fruit in these vessels. At sites where the preservation is inferior to that of Qasr Ibrim, the shorter-chain fatty acids ($C_{12:0}$ and $C_{14:0}$) will be partially, if not wholly, lost in the burial environment due to ground water leaching. However, because the hydroxy acids appear to be 'bound' to the clay fabric, they will be more likely to survive a waterlogged burial environment (Regert *et al.*, 1998). Hence, investigation of the 3-hydroxy fatty acids in the insoluble fraction would enable the detection of palm kernel fatty acids in pottery from a wide range of burial environments.

A previous study of archaeological lamps from Qasr Ibrim detected vicinal dihydroxy fatty acids in the insoluble fraction, following base treatment of the TLE (Bland, 1999). These dihydroxy acids are formed through the dihydroxylation of the double bond, such that the position of the hydroxyl groups is directly related to the original position of the double bond. For example, 9,10-dihydroxyoctadecanoic acid is a degradation product of Δ^9 octadecenoic acid. Therefore, through the detection of characteristic dihydroxy acids it was possible to conclude that some lamps had mixtures of oils burned in them, such as castor oil (detected from 9,12 dihydroxy $C_{18:0}$ and 12-hydroxy $C_{18:1}$), and radish oil (detected from 11,12 dihydroxy $C_{20:0}$ and 12,13 dihydroxy $C_{22:0}$). Thirty-three of the sherds (11%) contained 9,10 dihydroxy $C_{18:0}$ in the TLE, and this was the only dihydroxy acid detected in the sherds. Although this is indicative of the dihydroxylation of the Δ^9 double bond in oleic acid, because oleic acid is ubiquitous, little can be deduced with regard to the presence of specific plant oils or animal fats.

5.2.2 Differential use of pottery ware types

As was discussed above (Section 1.3.2), the pottery can be classified into two broad ware types, namely the hand-made and the wheel-made wares (Adams, 1986a; Adams, 1986b). Through ethnographic work in the region, it is believed that hand-made wares were manufactured by family groups, essentially on a small scale, thus enabling them to meet their needs, as and when required. In ancient Nubia, such vessels tended to be roughly fashioned, rarely had decoration on them, and were principally utility wares (e.g. pots) associated with domestic usage (Adams, 1986b:39). Conversely, the wheel-made vessels were well made, robust wares manufactured by 'professional potters'. In the Meroitic period, the wheel-made wares were often decorated and of a very fine quality. However, in the post-Meroitic periods, decorated vessels are not seen as frequently, and it is widely accepted that these vessels are not of the same quality as those used in the Meroitic period.

It is therefore of interest to determine whether:

(i) A particular ware type was preferred in the processing of particular commodities.

(ii) There was any change in the use of particular wares-types over time.

Table 5.3 shows the number and relative proportions of sherds of different ware-types that contained residues of the major commodity groups as determined from Chapter 4. The assemblage at Ibrim during these periods is dominated by wheel-made wares, and the sampling strategy reflects this.

Table 5.3 Number and proportion of the major commodities associated with different wares-types[1]

Commodity	Hand-made wares	Wheel-made wares[2]
Predominantly animal	10 (16%)	53 (84%)
Predominantly plant	11 (26%)	31 (74%)
Palm fruit	2 (3%)	77 (97%)

[1]Percentages are given for each commodity
[2]Includes ware types R32, R25, R1 and R25/32

Interestingly, the vessels that contain palm kernel lipids are almost all wheel-made wares. Furthermore, a higher proportion of the sherds exhibiting characteristic plant fatty acids were processed in hand-made wares compared with the proportion of sherds containing animal products. Indeed, there is a significant association between the two ware types and the incidence of the residues as classified in Table 5.3 (Chi-squared; $df = 2$; $\chi^2 = 14.66$; $p < 0.1\%$). Here, Cramér's V is 0.20, indicating that there is less of an association between commodity detected in the sherds and ware type than between the same commodity group and archaeological period, where Cramér's V was 0.49 (Section 5.2.1).

Using the same data presented in Table 5.3, Figure 5.8 illustrates the change in the use of specific ware types over time. If no differences in the use of a ware type existed, then the bar charts in Figure 5.8 would all look similar to that seen for the Post-Meroitic period. During this latter period, the relative proportions

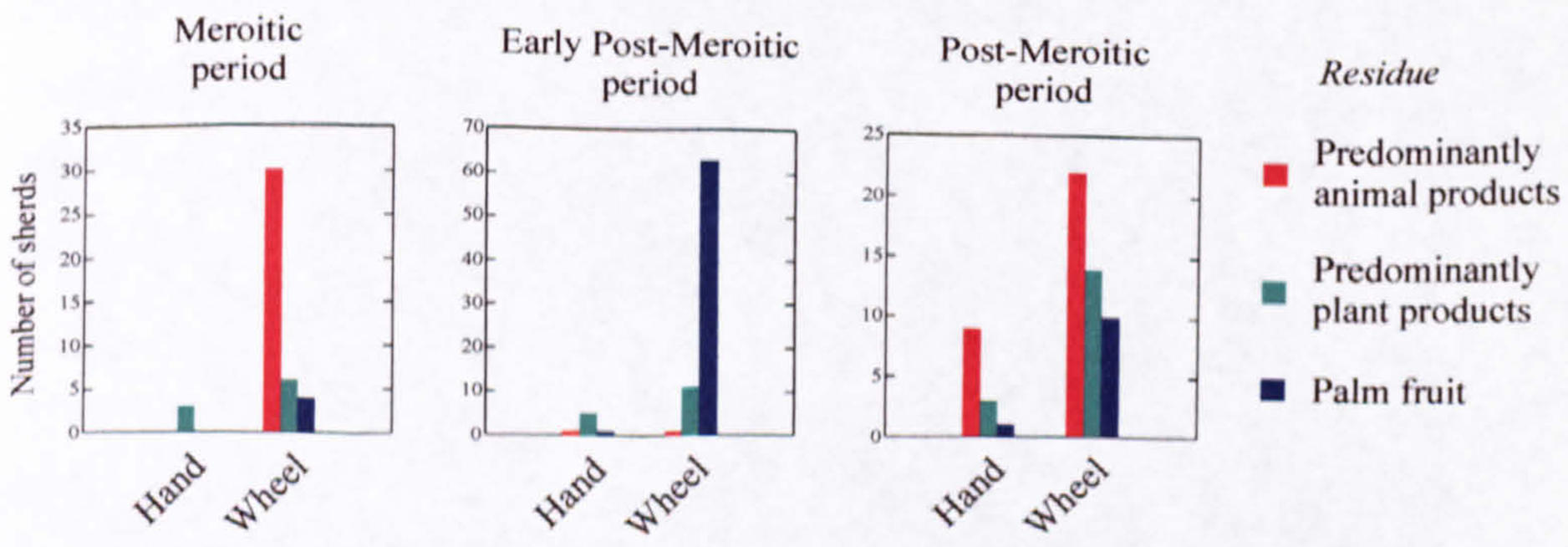

Figure 5.8 Change in use of ware-types over time. 'Hand' refers to hand-made wares, and 'Wheel' to wheel-made wares.

of the vessels used to process particular commodities is similar in the hand-made and the wheel-made wares, suggesting that there was no preferential use of either ware type. This is not the case for the other two periods. The relative abundances of the sherds containing predominantly animal or predominantly plant residues during the EPM period are very similar. It is only the presence of vessels containing palm kernel lipids that distort the bar chart, and the majority of these residues were present in wheel-made wares. Furthermore, the absence of Meroitic hand-made wares containing predominantly animal fats is intriguing. This may reflect the low abundance of hand-made wares from this period that were recovered from the site during excavation. However, it is also possible that a distinct difference in vessel use during the Meroitic may have been employed by the Nubians at Qasr Ibrim and as such was a conscious decision to use the higher quality, higher status wheel-made vessels to process animal products.

5.2.3 Dietary assessments based on stable isotope analyses of lipids in potsherds

Recent work (White and Schwarcz, 1994) discovered a slight, but significant, difference in the δ^{13} values of bone collagen from Nubian mummies from various cemeteries from the Wadi Halfa region in Sudan (which is just North of the Second Cataract, see map in Fig. 1.2). White and Schwarcz found that the mean $\delta^{13}C_{COLLAGEN}$ values changed from -18.1‰ in the Meroitic period to -17.0‰ in the Post-Meroitic period (which is the equivalent of the EPM and the Post-Meroitic periods at Qasr Ibrim) and then to -18.7‰ in the Christian period. Bone collagen in humans is approximately 5‰ less depleted than diet and, importantly, reflects the protein component of the diet (Ambrose and Norr, 1993), thus indicating that these Nubians' diets consisted *mainly* of C_3 plants and animals that were reared primarily on C_3 plants. From White and Schwarcz's study, it was found that there was a significant difference in $\delta^{13}C$ values between the Meroitic and Christian periods, but they did not assess when this change occurred (i.e. between the Meroitic and Post-Meroitic periods, the Meroitic and Christian periods or the Post-Meroitic and Christian periods). They also detected a 1‰ difference in the $\delta^{13}C$ values of male and female

collagen, and suggested that this indicates that the male diet consisted of less C_4 foodstuffs. Finally, through analysis of segments taken along the length of hair from these mummies, temporal differences were detected in the relative consumption of C_3 and C_4 crops/animals raised on C_3 and C_4 diets, and this was attributed to seasonal variations in food consumption.

The lipids extracted from the pottery vessels studied herein directly reflect the actual range of commodities that were processed within the pots and jars. Although the actual use/function of the vessels at Qasr Ibrim has to be considered, the lipid extracts are likely to (at least partially) reflect the diet of the Nubians, therefore legitimising comparison with the results obtained by White and Schwarcz (1994) as long as it is remembered that these human bones were not from Qasr Ibrim. Table 5.4 lists the $\delta^{13}C$ values for the fatty acids from the sherds taken from each of the three periods. In order to account for the high abundance of sherds containing palm kernel lipids, data analysis has been performed both including and excluding these sherds, thereby attempting to avoid any bias these particular sherds might have introduced into the statistical analysis. Compared with the other environmental reference materials, the palm kernel lipids have relatively more depleted $\delta^{13}C$ values for their $C_{16:0}$ and $C_{18:0}$ fatty acids (e.g. the date kernels are *c.* -31‰); therefore their inclusion would result in more depleted mean values, thus biasing the statistical analysis.

Table 5.4 Variation over time of $\delta^{13}C$ values of the major fatty acid components of the residues

		$\delta^{13}C_{16:0}$		$\delta^{13}C_{18:0}$	
	Period	Mean	S.D.	Mean	S.D.
Sherds that contain characteristic palm kernel lipids	Meroitic	-25.8	2.7	-26.6	2.4
	EPM	-26.4	2.1	-25.9	2.3
	Post-Meroitic	-25.3	2.4	-25.6	2.3
Sherds that do not contain characteristic palm kernel lipids	Meroitic	-25.7	2.7	-26.4	2.5
	EPM	-25.3	1.8	-24.8	1.4
	Post-Meroitic	-25.0	2.3	-25.4	2.4

In order to test for any change in the $\delta^{13}C$ values over time, ANOVA was applied to the data set, with the Bonferroni protection for multiple tests. The results are shown in Tables 5.5 and 5.6. From this it can be concluded that a statistical difference was only observed between the EPM and Post-Meroitic periods for the $\delta^{13}C_{16:0}$ values when all of the sherds are included in the data analysis. No such statistical significance was obtained when the sherds with palm kernel lipids were removed from the data set and the remaining sherds analysed separately. As expected, this suggests that the variation seen in the $\delta^{13}C$ values between the EPM and Post-Meroitic periods is due to the more depleted $\delta^{13}C_{16:0}$ values in the palm kernel lipids.

Since a significant difference between the periods was not observed for the $\delta^{13}C$ values of $C_{16:0}$ and the $C_{18:0}$, the results do not conclusively demonstrate changes in the relative proportions of C_3 and C_4 plants or animals fed on C_3/C_4 plants over time. Therefore, whilst the introduction of the *saqia* during the EPM period allowed the cultivation of a larger quantity and variety of crops,

Table 5.5 Significance levels from the ANOVA of the $\delta^{13}C$ values[1] from all of the vessels

	Meroitic	EPM	Post-Meroitic
Meroitic	x	x	x
EPM	0.295 0.255	x	x
Post-Meroitic	0.825 0.059	**0.009** 1.000	x

[1]The figures in green are for the $\delta^{13}C_{16:0}$ and those in blue are for the $\delta^{13}C_{18:0}$. Figures in bold are significant at the 99% CI.

Table 5.6 Significance levels from the ANOVA of the $\delta^{13}C$ values[1] from the vessels that do not contain characteristic palm fruit lipids

	Meroitic	EPM	Post-Meroitic
Meroitic	x	x	x
EPM	1.000 1.000	x	x
Post-Meroitic	1.000 1.000	1.000 1.000	x

[1]The figures in green are for the $\delta^{13}C_{16:0}$ and those in blue are for the $\delta^{13}C_{18:0}$.

based on the values in Tables 5.5 and 5.6, it does not appear to have led to either a relative increase in C_3 cultivars or a decrease in the quantities of the C_4 plants being processed at Qasr Ibrim. The absolute quantities and variety of these plants may have increased, as evidenced in the palaeobotanical record (Rowley-Conwy, 1989; Rowley-Conwy, 1991; Clapham, 1998), with the introduction of new crops from the EPM period onwards. These new plants, which are all C_3 plants, included the wheats *Triticum durum* and *T. aestivium*, the termis bean (*Lupinus albus*) pea (*Pisum sativum*), sesame (*Sesamum indicum*), and pearl millet (*Pennisetum typhoides*) chickpea (*Cicer arientinum*) hyacinth bean (*Lablab purpureus*). However, the $\delta^{13}C$ values of the fatty acids from the sherds do not appear to show any change over time (Tables 5.5 & 5.6).

White and Schwarcz (1994) found a significant difference between the $\delta^{13}C$ value of the bone collagen between the Meroitic, Post-Meroitic and Christian periods. The collagen from the bones of the Post-Meroitic mummies was enriched relative to those from the other two periods. Bone collagen is a long-term indicator of the protein consumption, and it is likely that the major proportion of protein would have been derived from animal products. It is therefore of interest to see whether there is a difference in the $\delta^{13}C$ values of the fatty acids from vessels that have been used to process predominantly animal products and from those that have been used to process predominantly plant products. Table 5.7 lists the $\delta^{13}C$ mean values and the standard deviations for the $C_{16:0}$ and $C_{18:0}$ lipid components from the vessels from the Meroitic and Post-Meroitic periods (due to the high abundances of palm kernel lipids in the sherds from the EPM period, there was insufficient data to include any EPM vessels in this data analysis). To establish whether any significant differences in the stable isotope values existed over time, Students' T-tests were performed on the data set.

The results from the T-test are very clear. Both the $\delta^{13}C_{16:0}$ and the $\delta^{13}C_{18:0}$ values from the extracts that have distributions characteristic of animal products are significantly more depleted in the Meroitic period compared to the Post-Meroitic period. The mean difference between the stable isotope values for the

$C_{16:0}$ was -1.6‰ and for the $C_{18:0}$ it was -1.8‰ between the two periods. Furthermore, the stable isotope values for the fatty acid components derived from the sherds that exhibited distributions characteristic of plant material do not show any significant difference over time (Table 5.7).

This latter finding is contrary to the $\delta^{13}C$ values obtained from the *n*-alkanes in Section 4.5. The isotope values for the C_{29} *n*-alkane indicated that there was a 4‰ difference between vessels from the Meroitic and Post-Meroitic periods, with relatively enriched $\delta^{13}C$ values for the *n*-alkanes in the sherds from the Meroitic compared to those from the Post-Meroitic period. Since no such variation over time was detected in $\delta^{13}C$ values of the fatty acids from the sherds that contained predominantly plant fatty acids, this suggests that in vessels where the mixing of commodities has occurred (i.e. from plants and animal products), the *n*-alkanes are a more reliable indicator as to the actual $\delta^{13}C$ value of the contribution of the plant component. Therefore, although the $\delta^{13}C$ values of the fatty acids from these sherds are more likely to reflect the $\delta^{13}C$ values of the fatty acids from the animal fat residues (*c.f.* the mixing curves in Section 3.3), the $\delta^{13}C$ values of the waxy plant component indicate that in the sherds that contained predominantly animal fats, either more C_3 plants with relatively less depleted $\delta^{13}C$ values, or more C_4 plants were processed in the Meroitic compared with the Post-Meroitic period. However, in vessels where only plants were processed, based on the $\delta^{13}C$ values of the fatty acids, it appears that no such change in use over time occurred.

Table 5.7 Changes in the $\delta^{13}C$ values of the fatty acids of the major commodity groups between the Meroitic and Post-Meroitic periods, determined through Student's T-tests

Fatty acid		Extracts from sherds that contain: Predominantly animal products	Predominantly plant products
$C_{16:0}$	Meroitic period	$\delta^{13}C$ = -26.4‰; s.d. = 2.8‰	$\delta^{13}C$ = -25.4‰; s.d. = 1.7‰
	Post-Meroitic period	$\delta^{13}C$ = -24.8‰; s.d. = 2.1‰	$\delta^{13}C$ = -25.8‰; s.d. = 1.8‰
	T-test	T = -2.30; df = 49; p = 0.025**	T = -0.08; df = 22; p = 0.938
$C_{18:0}$	Meroitic period	$\delta^{13}C$ = -27.2‰; s.d. = 2.8‰	$\delta^{13}C$ = -25.8‰; s.d. = 1.9‰
	Post-Meroitic period	$\delta^{13}C$ = -25.4‰; s.d. = 1.7‰	$\delta^{13}C$ = -25.2‰; s.d. = 1.7‰
	T-test	T = -2.67; df = 49; p = 0.010***	T = -0.04; df = 22; p = 0.532

**Significant at 95% CI
***Significant at 99% CI

Since the majority of the protein in the Nubian diet would have originated from animal products, the values shown in Table 5.7 are in accordance with the collagen data reported by White and Schwarcz (1994). The $\delta^{13}C$ values for the fatty acids from the pottery vessels that contain predominantly animal fats and the $\delta^{13}C$ values for the collagen from the Nubian mummies are significantly enriched in the Post-Meroitic period.

The $\delta^{13}C$ values of the fatty acids from the sherds that contain bovine and ovi-caprine fats also vary over time. This is detectable since those sherds that contain predominantly cattle fats can be tentatively differentiated from those containing predominantly sheep/goat fats (Fig. 5.9; see also Section 3.2).

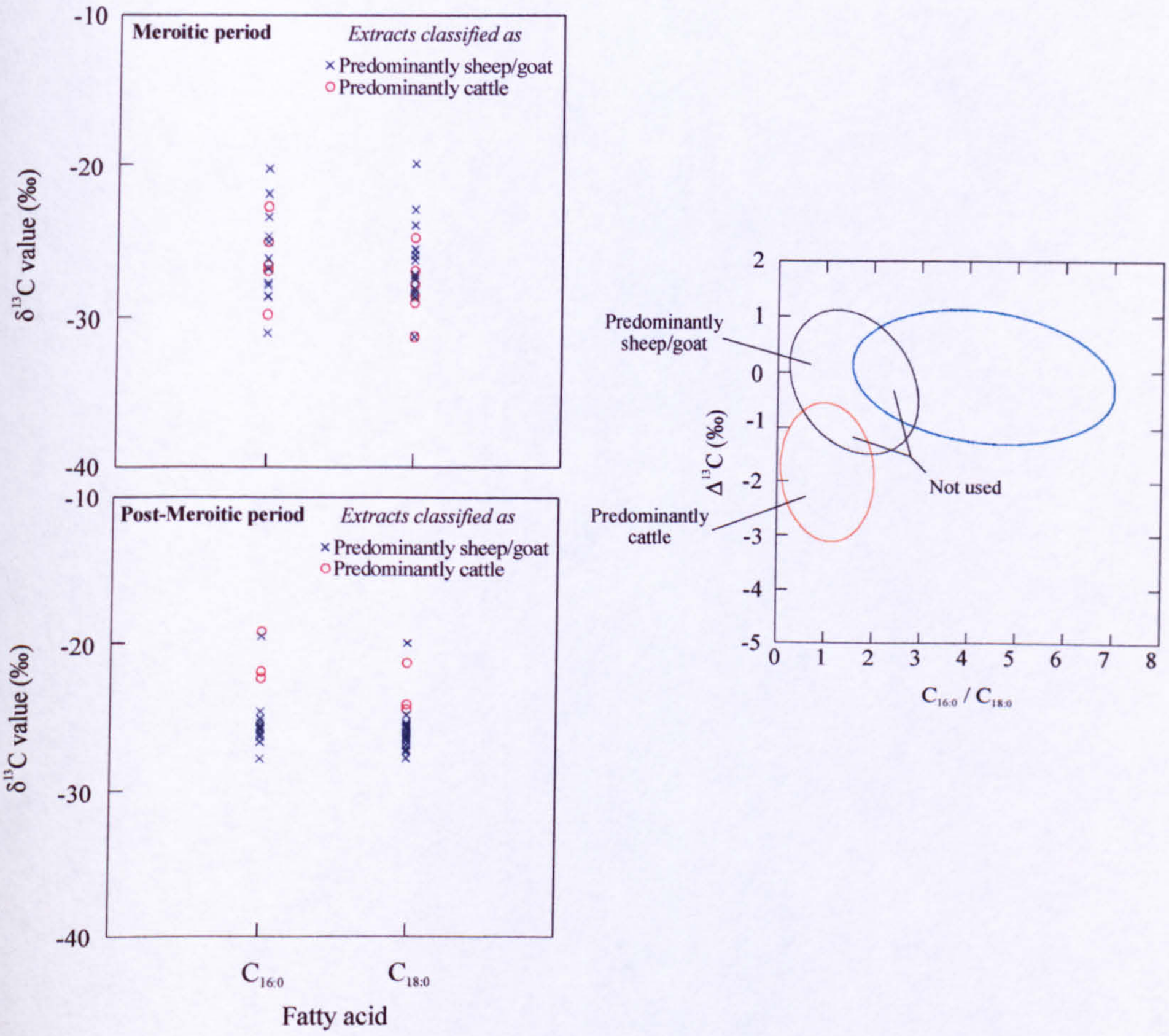

Figure 5.9 The $\delta^{13}C$ values of the fatty acids from the vessels that contain predominantly cattle and predominantly sheep/goat fats. The graph on the right shows the $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$) values v $C_{16:0}/C_{18:0}$ abundance ratios for the reference materials, indicating the region of the graph from which the sherds were selected for the plot on the left.

During the Meroitic period, the sherds that contain predominantly ovi-caprine or predominantly bovine fats show relatively wide variation in the $\delta^{13}C$ values (-19 to -32‰), for their fatty acid components. Later in the Post-Meroitic period, whilst the majority of the sherds that contain predominantly ovi-caprine fats exhibit higher $\delta^{13}C$ values, i.e. *c.* -25 to -27‰, the sherds containing predominantly cattle fats display $\delta^{13}C$ values in the range *c.* -19 to -24‰. This is strongly indicative of a higher contribution of C_4 plants to the cattle's diet, when compared to that of the sheep/goats in the Post-Meroitic period. In contrast, in the Meroitic period the animals were raised on plants with much more varied $\delta^{13}C$ values. Furthermore, whilst in the Meroitic period, all of the

sherds that contain predominantly sheep/goat fats exhibit a wide range of $\delta^{13}C$ values, in the Post-Meroitic period they are tightly clustered at around -26‰, and are indicative of a predominantly C_3 diet. Interestingly, the stable isotope values of the fatty acids from the extracts that contain lipids characteristic of plant lipids, as shown in Table 5.7, indicate there is no difference over time, with the mean $\delta^{13}C$ values of these fatty acids being -25.2 and -25.8‰. Importantly, these latter values are more depleted than those from the sherds that contain predominantly cattle fats. Therefore, it appears that with the onset of the Post-Meroitic period, C_4 cultivars were becoming of specific importance to the local economy, in as much as they were being fed as part of a mixed diet to cattle. However, analysis of the sherds that contained predominantly plant lipid showed that there was no difference in the $\delta^{13}C$ values over time, suggesting that although C_4 crops were being processed in the pottery vessels, the proportions did not change significantly over time.

A level of caution has to be applied here, because only three sherds plotted in the portion of the ellipse representative of predominantly cattle fats for the Post-Meroitic period, and only six are represented in the Meroitic period (hence no statistical analysis has been attempted on these data). However, this may indicate the beginning of preferential feeding of C_4 fodder/forages by the cattle during the Post-Meroitic period.

Significantly, similar results were obtained from the analysis of the fatty acids from the bovine reference bones dated to the Christian period when compared with the Napatan and EPM periods (as discussed in Section 3.1.4), and may represent a continuation of this trend. The palaeobotanical evidence suggests that cultivated sorghum was only introduced to Qasr Ibrim during the Meroitic period, and that quantities of this crop increased over time, such that in the later Christian period, it is one of the most important crops at Qasr Ibrim (Rowley-Conwy, 1989; Rowley-Conwy, 1991). Millet has also been recovered from Napatan contexts onwards, and although this crop was present in later periods as well, following its domestication, sorghum tends to be found in much greater abundances. From the analysis of the pottery vessels, it can be stated that these

C_4 plants were not processed in dedicated pottery vessels, but on occasion were mixed with other plants or animal products. Furthermore, sorghum/millet seems to have been preferentially used as a bovine fodder/forage in the Post-Meroitic period.

The Meroitic to Post-Meroitic periods encompassed widespread changes in Nubia (e.g. Edwards, 1996). With the collapse of the Meroitic State in the fourth century AD, Nubia witnessed political, economic and religious changes. For example, in Lower Nubia few substantial buildings were constructed (Edwards, 1989:178), old funerary traditions re-emerge in Upper Nubia (Welsby, 1996:202), the level of imports represented in the archaeology of Upper Nubia dwindles in this period, contrary to the experience of Lower Nubia (Edwards, 1989:181), and the pottery that is produced in Lower Nubia is of a lower quality compared to the earlier Meroitic wares (e.g. Adams, 1986a; Adams, 1986b). At present, the actual effect of the decline of the Meroitic State is less clear in the North than in the South of the region.

The introduction of the animal-driven water wheel, the *saqia*, in the Early Post-Meroitic period would have allowed not only a greater volume of crops to be cultivated, but also a greater variety of species of plants to be exploited. Certainly the pea, sesame, pearl millet, lentil and beans such as the hyacinth have all been recovered from Post-Meroitic contexts at Qasr Ibrim (Rowley-Conwy, 1989; Rowley-Conwy, 1991; Clapham, 1998).

The importance of plant products to the Nubian people has been proven herein; the sherds that contain predominantly plant lipids comprise 14% of the total number of vessels (39% if the vessels used to process the palm fruit are included as well). There was evidence of the mixing of plants and animal products in 7% of the sherds, indicating that a total of 21% of the sherds were used to processes plants in (not including the palm fruit). In such a harsh environment that existed in Lower Nubia, domesticated animals would have been a highly regarded commodity. This may account for the fact that a significant quantity of the sherds contained only plant lipids; something that is not seen in

British/European prehistoric pottery, where a dominance of animal products is suggested by the lipid extracts (e.g. Evershed *et al.*, 1999).

There is evidence for the date and dom palm fruit from the Napatan period onwards at Qasr Ibrim. From the residue analysis of the pottery vessels discussed above, extensive chemical evidence for the processing of palm fruit in the pottery vessels is detected from all periods, and especially from the Early Post-Meroitic period. It is likely that these lipids are residues of sugar extraction, although whether this was done for the benefit of the elite at Ibrim, or whether this represents the processing of the fruit on a fairly large scale for personal use is not known. However, the proportion of vessels that contained these characteristic lipids, at 75%, is particularly high in the Early Post Meroitic period, and the survival of the characteristic fatty acids is a testament to the unique preservation that exists at Qasr Ibrim.

Chapter 6.
Overview

6.1 BACKGROUND

Qasr Ibrim is situated in Southern Egypt, in what was Lower Nubia, and occupied between *c.* 1000BC to 1800AD. The site is of high archaeological interest principally for the following reasons:

(i) Compared to Upper Nubia, generally less is known about the archaeology of Lower Nubia.

(ii) Following the flooding of the Aswan High Dam in the 1960's, a significant proportion of the archaeology of Lower Nubia is now under water. However, because Qasr Ibrim was situated on top of a cliff top promontory, much of the site has survived the rising water levels of Lake Nasser.

(iii) Qasr Ibrim was a fortified citadel that gained religious, administrative, commercial and strategic importance during its history (Horton, 1991:264), and as such is of intrinsic regional archaeological interest.

(iv) The arid environment that exists at the site (there is negligible rainfall in the region) has led to excellent preservation of organic material, including basketry, leather, textiles, papyrus documents as well as bone and plant remains (e.g. Alexander and Driskell, 1985; Driskell *et al.*, 1989; Rowley-Conwy, 1991; Rowley-Conwy, 1994; Rose and Edwards, 1998). This degree of preservation has also been shown to exist at the molecular level (e.g. O'Donoghue *et al.*, 1996; Evershed *et al.*, 1997b).

The end of the Meroitic period (*c.* 50 to 300AD), associated with the demise of the City of Meroë in the South, led to political, religious and economic changes in Nubia. During the Early Post-Meroitic period (*c.* 300 to 400AD) the *saqia* was introduced to Lower Nubia (Rose, 1996). The *saqia* was an animal driven water-wheel that allowed more efficient irrigation of the dry lands along the River Nile. Therefore, the *saqia* potentially allowed larger quantities and more varieties of crops to be cultivated. The remains of barley (*Hordeum vulgare*), sorghum (*Sorghum bicolor bicolor*), emmer wheat (*Triticum dicoccum*), common millet (*Panicum miliaceum*), lentil (*Lens culinaris*), date palm (*Phoenix dactylifera*) and the dom palm (*Hyphaene thebaica*) have been

recovered from Meroitic contexts (Rowley-Conwy, 1989; Rowley-Conwy, 1991; Clapham, 1998). By the end of the Post-Meroitic period (*c.* 400 to 550AD), other plants were also being utilized, for instance the termis bean (*Lupinus albus*), pea (*Pisum satium*), pearl millet (*Pennisetum typhoides*) and hyacinth bean (*Lablab purpureus*) to name a few (*ibid.*).

To date, the zooarchaeological evidence from Qasr Ibrim has not been studied in as much detail as the archaeobotanical evidence. It is known that from a context within one of the Temple precincts, large quantities of juvenile cattle bones were found; this has been interpreted to represent the specific selection of juvenile cattle from the Valley economy by the 'priests' (Rowley-Conwy, *pers. comm.*). However, for Nubia as a whole, sheep/goats predominate the faunal assemblage (e.g. Hewes, 1964; Rowley-Conwy, *pers. comm.*).

6.2 AIMS

During the preparation and cooking of food in unglazed pottery vessels, organic molecules from animal and plant products (e.g. animal fats and plant oils) can be absorbed into the clay matrix of the fired pot. Through the large-scale chemical analysis of carefully selected pottery vessels, and the use of plant and animal remains as references, it is possible to detect the commodities that were being processed in the vessels, and hence the following questions can be answered:

(i) Is there a change in vessel use over time? And does this correspond to changes in the Meroitic and Post-Meroitic economies?

(ii) Is there a distinction in use of vessels of different ware types (especially hand-made and wheel-made wares)?

(iii) Do the palaeoenvironmental reference materials show any trends over time?

Analyses of modern and archaeological faunal and botanical materials, were initially undertaken, through gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) of the total lipid extract (TLE), and through GC and gas chromatography-combustion isotope ratio mass spectrometry (GC-C-IRMS) of the fatty acid methyl esters (FAMEs). Therefore enabling the state of molecular preservation to be assessed and the identification of lipid characteristics that could be used in the statistical separation of the commodity groups. Thus facilitating the use of these criteria in the classification of the data obtained from the analyses of the sherds.

A point raised in the introduction (Section 1.15) highlighted the fact that very few large-scale studies of absorbed lipids in pottery have been accomplished. A total of 313 sherds from Qasr Ibrim were analysed herein, and to date, this represents the largest analysis of its kind from a single site.

6.3 The Environmental Reference Materials

Lipids were extracted from 45 animal bones that had been excavated from Qasr Ibrim. The total lipid extracts were dominated by saturated fatty acids (principally $C_{16:0}$ and $C_{18:0}$) indicating that extensive hydrolysis of the triacylglycerols (TAGs) has occurred during burial. Oxidative degradation was also evidenced by the detection of vicinal dihydroxy fatty acids (which were present in 33 of the bones; 72%). These dihydroxy fatty acids are believed to be formed by oxidation of the carbon-carbon double bond by dihydroxylation (Regert *et al.*, 1998). Similar evidence for oxidation of the unsaturated fatty acids during burial is exhibited by the presence of α,ϖ-dicarboxylic acids (typically in the range C_6 to C_9, with C_9 predominating), and a group of hydroxyoctadecenoic acids that are unresolvable by GC.

The $C_{16:0}/C_{18:0}$ relative abundance ratios were calculated for the sheep/goat bones (mean = 1.54, s.d. = 0.82), and the cattle bones (mean = 1.08, s.d. = 0.56).

The $\delta^{13}C$ values of the principal fatty acids were determined through GC-C-IRMS of the FAMEs, and it was observed that for the ovi-caprids, the mean $\delta^{13}C_{16:0}$ was -23.9‰ (s.d. = 2.4‰), and the mean $\delta^{13}C_{18:0}$ was -24.5‰ (s.d. = 2.1‰). Whereas for the bovine bones the mean $\delta^{13}C_{16:0}$ was -18.7‰ (s.d. = 3.9‰), and the mean $\delta^{13}C_{18:0}$ was -20.4‰ (s.d. = 3.9‰). The $\delta^{13}C$ values are indicators of the relative proportion of C_3 and C_4 plants consumed by the animals. Furthermore, by examining the periods from which the bones originated, it appears that there is an increasing incidence of C_4 cultivars being used as fodder/forage for cattle from the Christian period onwards. In nine of the bones (20%) cholesterol or cholesteryl fatty acyl esters were detected. The $\delta^{13}C_{CHOLESTEROL}$ values were all less depleted than the $\delta^{13}C_{FATTY\ ACID}$, but no linear relationship was observed between the two (the $\Delta^{13}C_{CHOLESTEROL-C16:0}$ ranged from 0.6 to 12.7‰), perhaps reflecting differences in the turnover rates of the respective compounds in the animals and hence indicating different seasonal feeding strategies.

The botanical reference materials include twelve samples (of 7 species) previously studied by Bland (1999), a further 13 samples (of 5 species) were analysed by the author. HT-GC analysis of modern date and dom kernels indicated that they contained unusual TAG distributions, with acyl carbon numbers ranging from C_{36} to C_{46}, with particularly high abundances of C_{40} to C_{46}. This is in sharp contrast to the TAG distributions detected in the other modern reference materials that typically yielded TAGs in the range of C_{48} to C_{54}. The archaeological reference materials generally only contained low abundances of TAGs, but yielded high abundances of free fatty acids. A striking difference was observed between the fatty acid compositions (as determined through their FAMEs derivatives) of the palm kernels and those obtained from the other botanical reference materials: i.e. the palm kernels exhibited high abundances of $C_{12:0}$, $C_{14:0}$ and $C_{18:1}$ and low abundances of $C_{18:0}$, whereas in the other botanical reference materials, the $C_{16:0}$ predominates, indeed, the fatty acid distribution of the palm kernel is unique in Nature (Gunstone *et al.*, 1986). The $C_{16:0}/C_{18:0}$ relative abundance ratios of the botanical remains is 4.3 (s.d. = 1.7), which is significantly different to the

$C_{16:0}/C_{18:0}$ abundance ratio obtained from both the ovi-caprine and bovine bones.

Triangular plots of the relative abundances of $C_{12:0}$, $C_{16:0}$ and $C_{18:0}$ are particularly useful for visually displaying the sherds containing palm kernel lipid residues, due to the high abundances of the $C_{12:0}$ fatty acid. Hence, these triangular plots have been used extensively herein. Furthermore, significantly different $\Delta^{13}C$ (defined as $\delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) values were detected between the fatty acids from the cattle bones & plant material and between the cattle & sheep/goat bones. Therefore a plot of the $\Delta^{13}C$ v $C_{16:0}/C_{18:0}$ is also useful in classifying the extracts to lipid origin.

6.4 Determination of Vessel Use Based on Lipid Content

A total of 313 sherds were analysed; 120 were excavated from Meroitic contexts, 94 from Early Post-Meroitic contexts and 99 from Post-Meroitic contexts. Of these, 209 (67%) sherds yielded appreciable concentrations of lipid (> 5 $\mu g\ g^{-1}$), thus allowing the $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ components and the fatty acid distributions to be determined. Not unexpectedly, saturated fatty acids dominated the lipid distributions, indicating that extensive hydrolysis of the acyl lipids to free fatty acids and glycerol has occurred during burial/vessel use. Furthermore, α,ϖ-dicarboxylic acids, 9,10-dihydroxy acids and short chain ($\leq C_9$) fatty acids were also detected in numerous vessels, these latter compounds being evidence that oxidation of the double bonds in unsaturated fatty acids has occurred during burial/vessel use.

51% of the sherds from the Meroitic period were dominated by residues indicative of predominantly animal fats, and 19% of the sherds also contained other diagnostic plant compounds (e.g. sterols, *n*-alkanes, derivatives of benzoic acid and levoglucosan), suggesting that plants were also processed in these vessels. Fifteen percent of the sherds exhibited predominantly plant lipids, and a further 7% contained characteristic palm kernel lipids. The $\delta^{13}C$ values of the

fatty acids indicated that the majority of these lipids originated primarily from C_3 plants/animals fed on predominantly C_3 plants, although a few sherds exhibited $\delta^{13}C$ values indicative of the processing of significantly quantities of C_4 products in the vessels. Five sherds from context 18013 contained coprostanol; three of which also yielded other 5β-stanols whose relative abundances is indicative of human/porcine faeces. It is suggested that these represent re-use of the vessels as chamber pots.

In sharp contrast to the Meroitic period, the pottery excavated from the Early Post-Meroitic contexts were dominated by sherds that contained palm kernel lipids (75%). Sherds with predominantly plant products accounted for 21% and predominantly animal fats were only detected in 2% of the sherds. The pottery from the Post-Meroitic period was dominated by extracts indicative of predominantly animal fats (45%), whereas 25% contained predominantly plant lipids and 16% of the sherds yielded palm kernel lipids. Diagnostic plant compounds were detected in 19% of the sherds containing predominantly animal fats, indicating the use of these vessels in the processing of both animal and plant products.

The detection of palm kernel lipids in the sherds was intriguing. It is known that there are many ways that the palm fruit could have been utilised in antiquity, several of which would have required the use of pottery vessels. Date wines are manufactured by the fermentation of the sugars within the fruit, modern ethnographic evidence suggests that vessels with very large capacities would be preferred (Dirar, 1993; Dirar, 1994). The vessels from which the palm kernel lipids were extracted did not have such large volumetric capacities (Adams, 1986a; Adams, 1986b). Therefore, it is perhaps more likely that sugars were being extracted from the palm fruit, especially since sugar cane was not introduced to Egypt until the 7thC AD (Darby *et al.*, 1977a:428-9). Interestingly, 98% of the vessels that contained palm kernel lipids were wheel-made wares, these wares are less porous than the hand-made wares, which is likely to aid in the recovery of the extracted sugars during the processing of the palm fruit.

The majority of the sherds (95%) from three specific Early Post-Meroitic contexts contained palm kernel lipids. Of the remaining contexts from the Early Post-Meroitic period, only 13% of the sherds yielded these characteristic palm kernel lipids, a proportion similar to that found in the Post-Meroitic period (16%), but significantly higher than the 7% detected in the Meroitic period. This suggests that the 13-16% level may be typical for all of the Post-Meroitic contexts, and that the unusually high prevalence of these residues in these three specific Early Post-Meroitic contexts may directly or indirectly indicate the intensive processing of palm fruit at the site. Detailed assessment of the $\delta^{13}C$ values of the fatty acids from all of these sherds has indicated that approximately 65% appear to have other C_3 or small quantities of C_4 fatty acids present. In other words, the majority of the sherds containing palm kernel lipids were either used to process small quantities of other commodities in the vessels at the same time, or that re-use of the vessels at Qasr Ibrim was relatively commonplace.

The $\delta^{13}C$ values of the fatty acids extracted from the sherds that displayed lipid distributions indicative of a predominantly plant origin were not significantly different between the Meroitic and Post-Meroitic periods. This indicates that the relative importance of C_3 and C_4 plants to the people and animals at Qasr Ibrim did not alter over time. However, the $\delta^{13}C$ values of the *n*-alkanes did show a significant difference over time, with more depleted values in the Post-Meroitic compared to the Meroitic period (indicating more reliance on C_3 than C_4 waxy plant components). The fact that the majority of the *n*-alkanes were detected in sherds also containing fatty acids indicative of predominantly animal fats suggests that as more C_4 crops were being cultivated at Ibrim (as seen in the palaeobotanical record), these crops were perhaps being preferentially processed in the pottery vessels with animal products. However, in the vessels that were only used to process plant products, a relatively constant proportion of C_3 and C_4 plants were being utilised over time (as detected through the $\delta^{13}C$ values of the fatty acids).

The $\delta^{13}C$ values of the fatty acids from the vessels containing predominantly animal fats did exhibit significant differences over time. The $\delta^{13}C_{16:0}$ and the $\delta^{13}C_{18:0}$ were both significantly less depleted in the Post-Meroitic compared with the Meroitic period, indicating the greater consumption by the animals of C_4 plants in the Post-Meroitic period. Furthermore, lipid origin can be tentatively classified as to being either predominantly sheep/goat or predominantly cattle fats, and it appears that whilst in the Meroitic period both sheep/goat and cattle included in their diet a wide range of C_3 and C_4 plants, in the Post-Meroitic period, there appears to be a polarisation of feeding strategies, with the cattle consuming a predominantly C_4 diet, and the sheep/goat consuming a predominantly C_3 diet. This temporal change in $\delta^{13}C$ values was also detected in the zooarchaeological reference materials (see above).

In conclusion, the arid environment at Qasr Ibrim has contributed to the excellent preservation of organic material. By using archaeological and modern reference materials, a detailed assessment of absorbed lipid residues in pottery has been accomplished, giving insights into changing vessel use and economy during the Meroitic to Post-Meroitic periods.

Chapter 7.
Further Work

Through the large-scale analysis of absorbed lipid residues in pottery from Qasr Ibrim, it has been possible to detect changes in vessels use over time, preferential use of specific ware-types, and the relative importance of C_3 and C_4 plants to the diet of cattle and sheep/goats. There are several aspects of the work described herein that could warrant further analysis, many of which require very large sample sizes.

(i) The processing of palm fruit has been detected at Qasr Ibrim. It would be of interest to know the geographical and temporal extent of this practice, hence analysing the organic residues from pottery not only from Qasr Ibrim, but also from other sites within the region. Significantly fewer sherds contained palm kernel lipids in the Meroitic vessels, compared with sherds from the later periods; this has to be taken into account when the sample size of any such analysis is being calculated. Therefore, in order to determine whether there are any intra-site variations in the prevalence of these extracts, several hundred more sherds would need to be analysed. Similarly, on a regional basis, it is likely that a few thousand sherds would need to be analysed.

(ii) At sites where arid conditions do not prevail, it is possible that the shorter-chain fatty acids (especially $C_{12:0}$) from the palm kernel residues would be preferentially lost through ground water leaching. Therefore analysis of the insoluble fraction would aid in the confirmation of the processing of palm fruit in vessels (in the manner discussed herein). This would be accomplished through the detection of a suite of 3-hydroxy fatty acids whose distributions mirror those of the saturated fatty acids in the TLE.

(iii) The actual origin of these 3-hydroxy fatty acids is a matter of conjecture. It is likely that they are bacterially mediated, and since they were only present in high abundances in the insoluble fraction of the sherds containing palm kernel lipids, it is possible that they are a product of the specific manner in which the palm fruit were processed in the pottery vessels. Further work on the origins of these 3-hydroxy (and 2-hydroxy) fatty acids would be useful.

(iv) A detailed investigation of the $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ extracted from the animal bones recovered from the beginning of the Napatan period through to the end of the Islamic period may enable changes in the diet of the different species of animal to be confirmed. This would necessitate the analysis of a few hundred animal bones, and could be accompanied by cholesterol, collagen and apatite $\delta^{13}C$ and $\delta^{15}N$ analyses in order to distinguish between short-term and long-term changes in diet. The collagen, apatite and amino-acid analysis of the bones investigated herein is already in progress.

(v) The *n*-alkanes obtained from the sherds yielded many different distributions, however at present it is not possible to determine their origin. A data base of *n*-alkane distributions of numerous waxy plant components, and the $\delta^{13}C$ values of the individual *n*-alkane components would help in the identification of the *n*-alkanes detected in the pottery vessels. This would be of use not only at Qasr Ibrim, but also at other archaeological sites.

(vi) Excellent preservation of lipid residues exists at Qasr Ibrim, and it would be likely that potentially similar preservation of amino-acids and sugars would also exist. The analysis of absorbed sugars from archaeological pottery and seeds as their alditol acetates following acid hydrolysis was attempted (data not shown). Whilst the extraction of sugars from the palaeobotanical reference materials was successful, repeated attempts failed to extract sugars from the vessels that had been proven to contain characteristic palm kernel lipids. There are two possible reasons for these findings. Firstly the sugars, being soluble in water, may have been removed from the vessel during use/washing of the vessel after use. Or secondly, the sugars are strongly bound to the vessel fabric such that other extraction methods are needed.

(vii) In several sherds, short-chain ($\leq C_9$) fatty acids were detected. These are assumed to be degradation products of unsaturated fatty acids (as the point of unsaturation in most monounsaturated fatty acids is at the Δ^9 position), however this has not been conclusively determined by laboratory experiments.

(viii) Other types of vessel have been excavated from the site, and include cups, plates and amphorae. During the Meroitic period a large quantity of wine was brought down to Ibrim from the southern Egyptian border. It would be of interest to analyse the residues of these amphorae (e.g. by LC-MS).

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Appendices

Appendix 1
Appendix 2

Table A1.1 The zooarchaeological reference materials

Sample Number	Context	Period[1]	Species	Bone	Notes
B27 pelv	B27	Chr	Cow	Pelvis	
B27 calc	B27	Chr	Cow	Calcaneous	Large adult
B27 phal	B27	Chr	Cow	Phalange	Large – indicative of traction?
B55	B55; rm 277; 6	Chr	Cow	Scapula	
B35	B35; rm 77 bel fl 6	Chr	Cow	Calcaneous	Large adult
NW Bast	NW Bastion	Nap	Unidentified	Radius?	?sheep/goat
B1-12	B1-12 rm 4	Chr	Sheep/goat	Radius; shaft	
B56.1	B56; rm 278;	Chr	Cow	Metacarpal; Prox.	
B56.2	B56; rm 278;	Chr	Cow	Carpal; distal	
B51.1	Ib 82 b51 courtyard	Chr	Pig	Jaw	
B51.2	Ib 82 b51 courtyard	Chr	Pig	Femur; shaft	
IB 82	IB 82 11.7	Chr	Cow	Metatarsal	
10045	10045	Otto	Sheep/goat	Humerus; shaft	
10048.1	10048	Otto	Cow	Humerus; shaft	Young adult
10048.2	10048	Otto	Cow	Metatarsal; distal	Adult
10063	10063	PM	Sheep/goat	Long bone; shaft	Adult
10110.1	10110	PM	Sheep/goat	Pelvis	Butchery marks
10110.2	10110	PM	Sheep/goat	Rib	
10203	10203	Chr	Sheep/goat	Femur; distal	Butchery marks
10216.1	10216	PM	Sheep/goat	Humerus; shaft	Butchery marks
10216.2	10216	PM	Sheep/goat	Metatarsal; proximal	Young adult
10216.3	10216	PM	Sheep/goat	Metacarpal	Juvenile
10377	10377	EPM	Sheep/goat	Humerus; distal	Adult
10384	10384	EPM	Cow	Vertebrae	Young adult
10478	10478	PM	Sheep/goat	Femur; proximal	Adult
12451.1	12451	Nap	Sheep/goat	Rib	
12451.2	12451	Nap	Sheep/goat	Femur; shaft	
12457	12457	Nap	Unidentified		?large sh/gt
12513.1	12513	Nap	Sheep/goat	Long bone; shaft	
12513.2	12513	Nap	Sheep/goat	Tibia; shaft	
14133	14133	Otto	Sheep/goat	Tibia; distal	Young adult
14146	14146	Chr/ Otto	Unidentified		?large cow
17035	17035	Nap	Sheep/goat	Humerus; shaft	
17042.1	17042	Nap	Sheep/goat	Radius; shaft	
17042.2	17042	Nap	Sheep/goat	Tibia; shaft	Juvenile
17048.1	17048	Nap	Cow	Rib	Young adult
17048.2	17048	Nap	Sheep/goat	Radius; distal	
17052.1	17052	Nap	Unidentified	Long bone	
17052.2	17052	Nap	Sheep/goat	Radius; shaft	
18013.1	18013	M	Sheep/goat	Rib	
18013.2	18013	M	Unidentified	Long bone	?cow
18013.3	18013	M	Unidentified	Long bone	?sh/gt
18013.4	18013	M	Unidentified	Long bone	?sh/gt
18013.5	18013	M	Unidentified	Long bone	?sh/gt
18022	18022	EPM	Cow	Rib; distal, large	

[1]M = Meroitic (50-300AD); EPM = Early Post-Meroitic (300-400AD); PM = Post-Meroitic (400-550AD); Chr = Christian (550-1500AD); and Otto = Ottoman (1500-1800AD)

Table A1.2 Normalised fatty acid composition and $\delta^{13}C$ values of the zooarchaeological reference materials

Sample Name	Fatty acid composition (%)				$\delta^{13}C$ value (‰)[1]	
	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{16:0}$	$C_{18:0}$
B27 pelv	3.0	48.4	42.1	6.5	-14.0	-17.0
B27 calc	2.2	46.5	41.4	9.9	-15.2	-18.2
B27 phal	n/d	51.0	45.5	3.5	-13.5	-12.9
B55	n/d	30.8	69.2	n/d	-18.9	-22.0
B35	3.0	40.0	45.8	11.2	-20.2	-21.3
NW Bast	1.5	34.3	57.7	6.5	-21.8	-22.5
B1-12	4.7	50.1	28.8	16.4	-23.6	-24.0
B56.1	n/d	49.9	25.2	24.9	-15.8	-17.6
B51.1	9.6	61.2	29.2	n/d	-23.8	-24.9
B56.2	3.8	54.5	23.2	18.3	-16.1	-17.2
B51.2	13.4	59.6	27.0	n/d	-23.6	-24.9
IB 82	n/d	44.9	25.2	24.9	-19.9	-21.0
10045	6.6	49.8	43.5	n/d	-26.0	-25.9
10048.1	4.6	48.9	35.1	11.4	-21.7	-23.4
10048.2	1.2	28.4	70.4	n/d	-21.7	-22.9
10063	2.9	56.6	20.0	17.5	-21.4	-23.3
10110.1	2.6	28.4	69.0	n/d	-23.6	-24.0
10110.2	2.1	48.7	49.2	n/d	-21.7	-23.2
10203	3.3	52.6	39.0	5.1	-27.0	-26.8
10216.1	1.2	35.4	45.8	17.6	-22.1	-21.6
10216.2	7.6	69.0	23.4	n/d	-21.2	-20.9
10216.3	4.1	46.2	32.9	16.8	-23.0	-22.9
10377	6.9	61.5	31.6	n/d	-27.2	-27.9
10384	2.7	32.6	59.2	5.5	-19.5	-20.6
10478	3.4	42.4	40.9	13.3	-25.0	-24.5
12451.1	4.3	58.4	35.2	2.1	-27.5	-26.3
12451.2	n/d	57.5	42.5	n/d	-22.5	-23.0
12457	3.3	53.1	41.0	2.6	-26.0	-28.0
12513.1	10.7	46.5	36.7	7.1	-25.9	-26.9
12513.2	25.0	45.2	29.8	n/d	-24.2	-24.2
14133	4.1	70.7	21.7	3.5	-20.8	-21.4
14146	n/d	33.6	66.4	n/d	-18.7	-21.1
17035	2.3	48.1	49.6	n/d	-26.2	-25.9
17042.1	n/d	52.1	47.9	n/d	-26.1	-27.5
17042.2	n/d	51.6	49.4	n/d	-24.8	-26.5
17048.1	1.1	32.8	64.2	1.8	-27.2	-28.2
17048.2	n/d	48.3	51.7	n/d	-18.8	-22.9
18013.1	n/d	42.0	58.0	n/d	-25.6	-25.4
18013.2	4.7	40.9	52.3	2.1	-25.4	-27.5
18013.3	22.4	49.7	27.9	n/d	-25.5	-25.5
18013.4	1.3	40.3	58.4	n/d	-23.7	-25.8
18013.5	21.7	50.3	28.0	n/d	-22.4	-25.1
18022	1.6	45.6	49.9	2.9	-21.0	-23.1

[1]Values have been corrected for the derivatisation factor and represent the mean value of 3 runs (more runs were completed if the s.d. > 0.3‰), instrumental precision = ±0.3‰
n/d = not detected

Table A1.3 Normalised fatty acid compositions and abundance ratios of the animal fats from the literature

Animal	Depot site	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{16:0}/C_{18:0}$	Source
Sheep	Perirenal	2.6	28.0	37.1	32.3	0.75	(Duncan and Garton, 1967)
Sheep	Mesenteric	2.2	26.3	36.2.	35.3	0.73	(Duncan and Garton, 1967)
Sheep	Chest	2.5	28.5	22.6	46.4	1.26	(Duncan and Garton, 1967)
Goat	n/a	2.2	27.1	29.9	40.8	0.91	(Gunstone *et al.*, 1986)
Cow	Perirenal	4.1	29.6	27.4	38.9	1.08	(Marmer *et al.*, 1984)
Cow	Subcutaneous	3.8	30.1	14.5	51.6	2.08	(Marmer *et al.*, 1984)
Pig	Perirenal	2.0	33.8	22.3	41.9	1.51	(Enser, 1991)
Pig	Subcutaneous	1.9	30.1	15.5	52.5	1.94	(Enser, 1991)

Table A1.4 Normalised fatty acid composition of the botanical reference materials (After Bland 1999).

Sample name	Species	Latin name	Archaeological/ Modern?	Fatty acid composition (%)							
				$C_{12.0}$	$C_{14:0}$	$C_{16.0}$	$C_{18:0}$	$C_{18\,1}$	$C_{18.2}$	$C_{18:3}$	$C_{20.0}$
Mdate1	Date palm kernel	*Phoenix dactylifera*	Modern	22.7	15.0	10.3	3.1	41.0	6.0	n/d	0.4
Adate1	Date palm kernel	*Phoenix dactylifera*	Archaeological	9.0	16.5	19.1	5.0	47.9	1.1	n/d	0.6
Mrad[1]	Radish	*Raphanus sativus*	Modern	n/d	0.1	10.1	2.6	29.1	29.1	10.1	1.3
Arad[2]	Radish	*Raphanus sativus*	Archaeological	n/d	0.1	8.6	3.4	18.7	18. 7	4.3	2.378
Mcas[3]	Castor bean	*Ricinus communis*	Modern	n/d	n/d	3.3	9.0	13.3	3.3	n/d	n/d
Acas[4]	Castor bean	*Ricinus communis*	Archaeological	n/d	n/d	3.1	2.1	9.1	3.3	n/d	n/d
Mbar	Barley	*Hordeum vulgare.*	Modern	n/d	0.6	33.7	11.0	11.4	38. 6	2.2	1.8
Abar[5]	Barley	*Hordeum vulgare*	Archaeological	n/d	0.8	30.9	13.5	24.6	18.6	n/d	n/d
Mwheat	Wheat	*Triticum dicoccum*	Modern	n/d	n/d	20.4	2.8	13.3	56.5	4.8	0.5
Awheat	Wheat	*Triticum dicoccum*	Archaeological	n/d	0.5	42. 8	10.9	31.4	6.7	n/d	0.6
Mbicol	Sorghum	*Sorghum bicolor*	Modern	n/d	n/d	14.8	2.8	43.0	35.1	1.8	0.5
Abicol	Sorghum	*Sorghum bicolor*	Archaeological	n/d	n/d	28.0	6.6	58.6	4.4	n/d	0.8
Adurra	Sorghum	*Sorghum durra*	Archaeological	n/d	n/d	38.6	9.5	46.4	46.4	2.5	0.7

[1] $C_{20:1}$ = 9.9%; $C_{22.1}$ = 43.7%
[2] $C_{20:1}$ = 10.8%; $C_{22.1}$ =24.1%
[3] $C_{18\,1\,10\text{-}OH}$ = 70.1%
[4] $C_{18\,1\,10\text{-}OH}$ = 82.6%
[5] $C_{20:1}$ = 11.1%
n/d = not detected

Table A1.5 Normalised fatty acid composition of the botanical reference materials extracted by the author

Sample name	Species	Latin name	Archaeological/ Modern?	Fatty acid composition (%)							
				$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$	$C_{20:0}$
Afig	Sycamore fig	*Ficus sycomorus*	Archaeological	n/d	2.7	60.4	18.2	12.0	n/d	n/d	6.6
Mlab	Hyacinth bean	*Lablab purpureus*	Modern	n/d	n/d	22.4	4.3	8.9	53.7	9.5	0.5
Alab	Hyacinth bean	*Lablab purpureus*	Archaeological	n/d	n/d	75.7	11.3	7.5	n/d	n/d	5.5
Acot	Cotton	*Gossypium herbacea*	Archaeological	n/d	0.7	70.8	11.7	12.3	n/d	n/d	0.9
Mdom	Dom palm kernel	*Hyphaene thebaica*	Modern	36.6	12.6	8.5	3.3	32.8	5.8	n/d	0.3

n/d = not detected

Refer to Table 4.5 for the fatty acid composition of the remaining palm kernel reference materials

Table A1.6 Fatty acid $\delta^{13}C$ values of the botanical reference materials (After Bland 1999)

Sample name[1]	Species	Latin name	Archaeological/ Modern[1]	Fatty acid $\delta^{13}C$ value (‰)							
				$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$	$C_{20:0}$
Mdate1	Date palm kernel	*Phoenix dactylifera*	Modern	-33.5	-32.1	-32.0	-31.9	-30.7	-30.9		
Adate1	Date palm kernel	*Phoenix dactylifera*	Archaeological	-32.6	-30.9	-30.9	-28.9	-29.1			
Mrad[2]	Radish	*Raphanus sativus*	Modern			-32.5	-32.1	-30.1	-30.9	-31.9	-33.6
Arad[3]	Radish	*Raphanus sativus*	Archaeological			-29.2	-28.9	-26.9	-26.0		-30.4
Mcas[4]	Castor bean	*Ricinus communis*	Modern			-33.7	-32.9	-30.4	-31.2		
Acas[5]	Castor bean	*Ricinus communis*	Archaeological			-31.3	-31.3	-27.7	-28.0		
Mbar	Barley	*Hordeum vulgare*	Modern			-32.7		-32.6	-34.6		
Abar	Barley	*Hordeum vulgare*	Archaeological			-28.6		-29.6	-28.7		
Mwheat	Wheat	*Triticum dicoccum*	Modern			-33.9		-32.3	-33.0	-31.5	
Awheat	Wheat	*Triticum dicoccum*	Archaeological			-31.4		-29.9	-27.9		
Mbicol	Sorghum	*Sorghum bicolor*	Modern			-18.4	-18.7	-15.9	-16.1	-20.7	
Abicol	Sorghum	*Sorghum bicolor*	Archaeological			-16.8	-18.0	-13.8	12.5		
Adurra	Sorghum	*Sorghum durra*	Archaeological			-16.6	-17.8	-13.6			

[1]Modern values adjusted for post-industrial fossil fuel burning by adding 1.14‰ to the measured values (Friedli 1986)
[2]$C_{20:1}$ = -31.6‰; $C_{22:1}$ = -31.0‰
[3]$C_{20:1}$ = -28.1‰; $C_{22:1}$ = -27.8‰
[4]$C_{18:1\ 10\text{-}OH}$ = -30.8‰
[5]$C_{18:1\ 10\text{-}OH}$ = -27.5‰

Table A1.7 Fatty acid $\delta^{13}C$ values of the botanical reference materials extracted by the author

Sample name	Species	Latin name	Archaeological/ Modern[1]	Fatty acid $\delta^{13}C$ value (‰)							
				$C_{12:0}$	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$	$C_{20:0}$
Afig	Sycamore fig	*Ficus sycomorus*	Archaeological			-25.2	-24.3	-20.3			
Mlab	Hyacinth bean	*Lablab purpureus*	Modern			-34.3	-34.3	-33.0			
Alab	Hyacinth bean	*Lablab purpureus*	Archaeological			-30.8	-31.1	-26.9			
Acot	Cotton	*Gossypium herbacea*	Archaeological			-29.1	-29.3	-33.6			
Mdom	Dom palm kernel	*Hyphaene thebaica*	Modern	-34.4	-34.1	-34.2	-34.1	-31.3			

[1]Modern values adjusted for post-industrial fossil fuel burning by adding 1.14‰ to the measured values (Friedli 1986)
Refer to Table 4.5 for the $\delta^{13}C$ values of the fatty acids from the remaining palm kernel reference materials

Table A1.8 Provenance of the Hinterland pottery

Sample Number (ML +)	Associated/ nearest structure	'P number'
1	20 (south building)	P220/3/6
2	20 (south building)	P220/3/6
3	20 (south building)	P220/3/6
4	20 (north building)	P220/P226
5i	20 (between north and south building)	P220/P224/P225
5ii	20 (between north and south building)	P220/P224/P225
6	20 (between north and south building)	P220/P224/P225
7	19 (northern corner)	P224/P227
8	19 (northern corner)	P224/P227
9	19 (southern corner)	P224/P227
10	84 (northern side)	P228
11	84 (eastern side)	P228
12	21 (south-eastern side)	P217
13i	22 (north of eastern annex)	P235
13ii	22 (north of eastern annex)	P235
14	32	P207
15	12 (south-west corner)	P280
16	338 (northern corner)	P283
17	362 (southern room)	P303
18	360 (southern corner)	P302
19	15 (nr. western wall)	P290
20	Midway between 334 & 333	P306
21	349	P304
22	83 (southern chamber)	P138/9
23	85 (west of)	P139/P140/P141
24	85 (west of)	P139/P140/P141
25	94 (west of)	P133
26	127 (western side)	P101
27	127 (south-west corner)	P101
28	128 (north of)	P114

Table A1.8 cont.

Sample Number (ML +)	Associated/ nearest structure	'P number'
29	127/8 (east of)	P101/114
30	129 (south of)	
31	130 (west of)	P104
32	129 (west of)	
33	131 (north of)	P103
34	133	P98
35	123 (north of)	P105b
36	214 (centre of)	P263
37	213 (east of)	P264
38	172 (western room)	P10
39	166 (main room)	P12
40	165 (southern room)	P13
41	165 (southern room)	P13
42	157 (northern corner)	P17
43	228 (west of)	P37/P54
44	289 (southern corner)	
45	230 (southern corner)	P41/P42
46	257 (western corner)	P67
47	178 (northern corner of north room)	P24/P27
48i	178 (centre of middle room)	P24/P27
48ii	178 (centre of middle room)	P24/P27
49	178 (southern room)	P24/P27

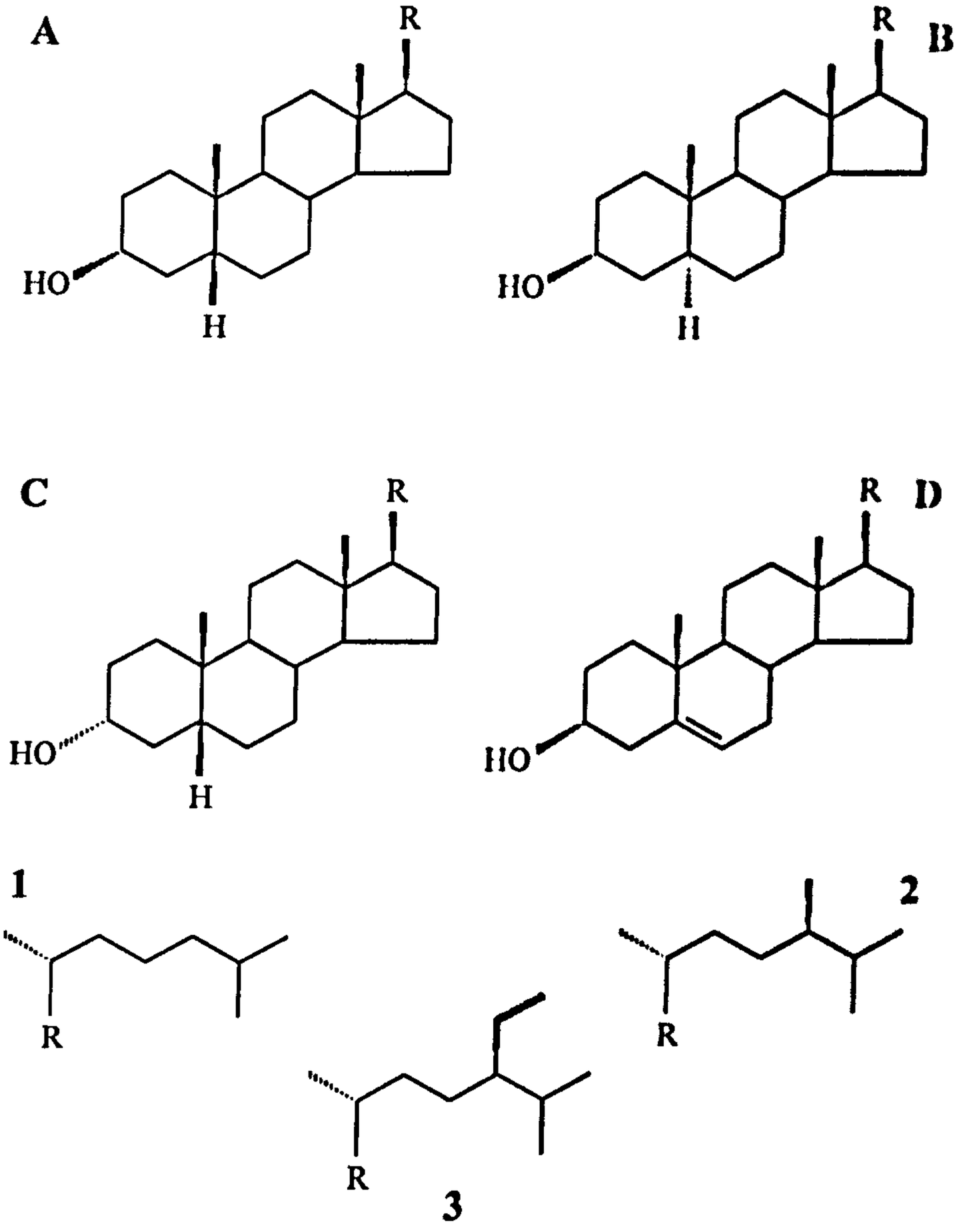
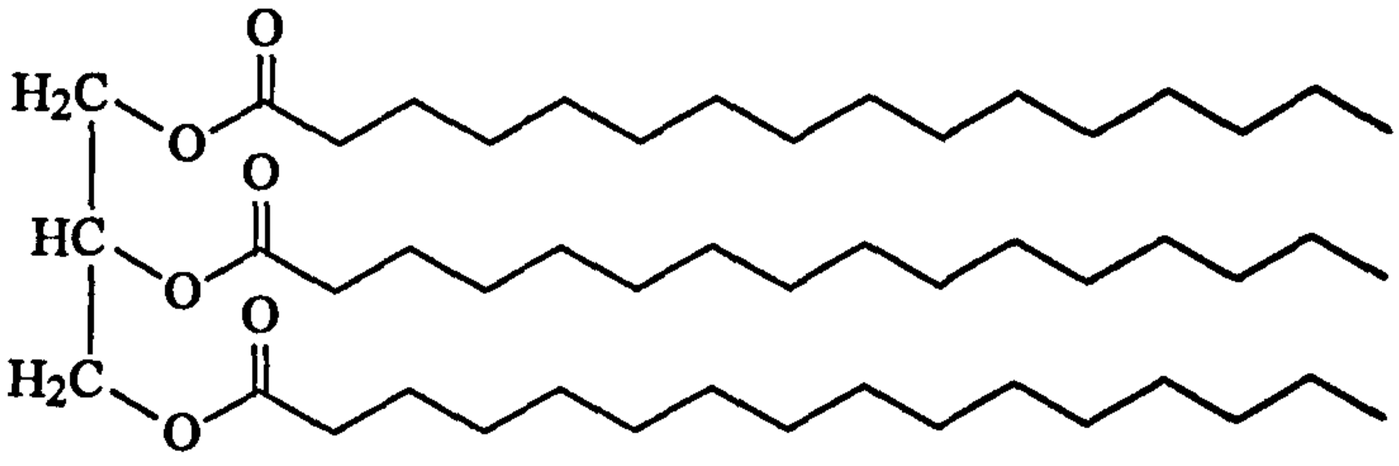


Figure A2.1 Structures of lipids cited.

Cholesterol (cholest-5en-3β-ol) **D1**; β-sitosterol (stigmast-5-en-3β-ol) **D3**; cholestanol (5α cholestan-3β-ol) **B1**; coprostanol (5β cholestan-3β-ol) **A1**; epicoprostanol (5β cholestan-3α-ol) **C1**; 5β-campestanol (5β-campestan-3β-ol) **A2**; epi-5β-campestanol (5β-campestan-3α-ol) **C2**; stigmastanol (5α stigmastan-3β-ol) **B3**; 5β-stigmastanol (5β-stigmast-3β-ol) **A3**; epi-5β-stigmastanol (5β-stigmastan-3α-ol) **C3**.

Tristearin



1,3 Distearin

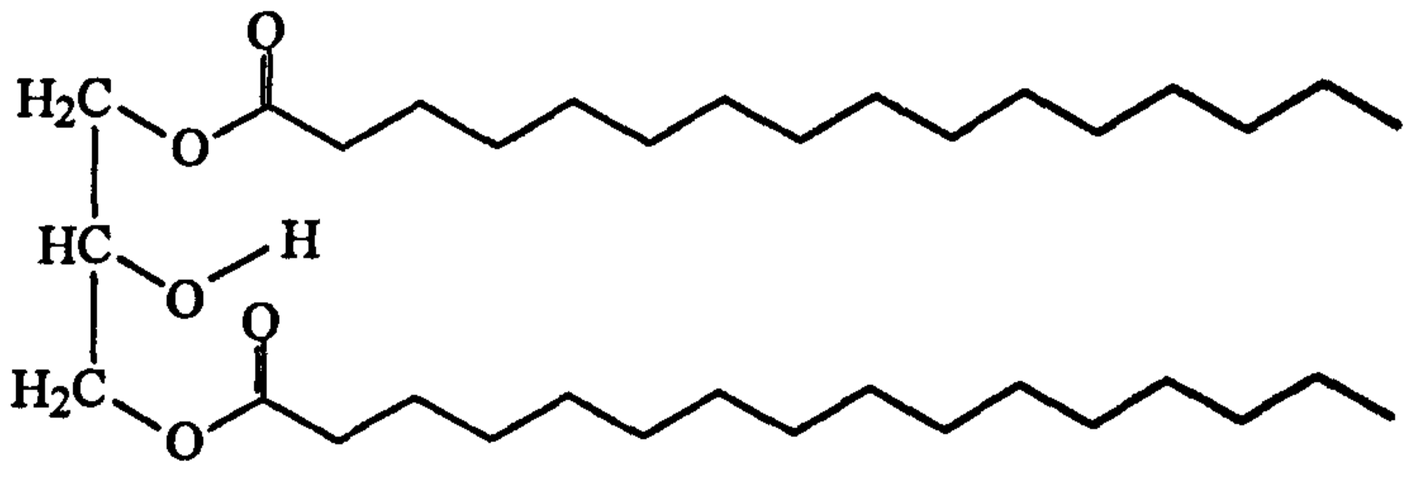
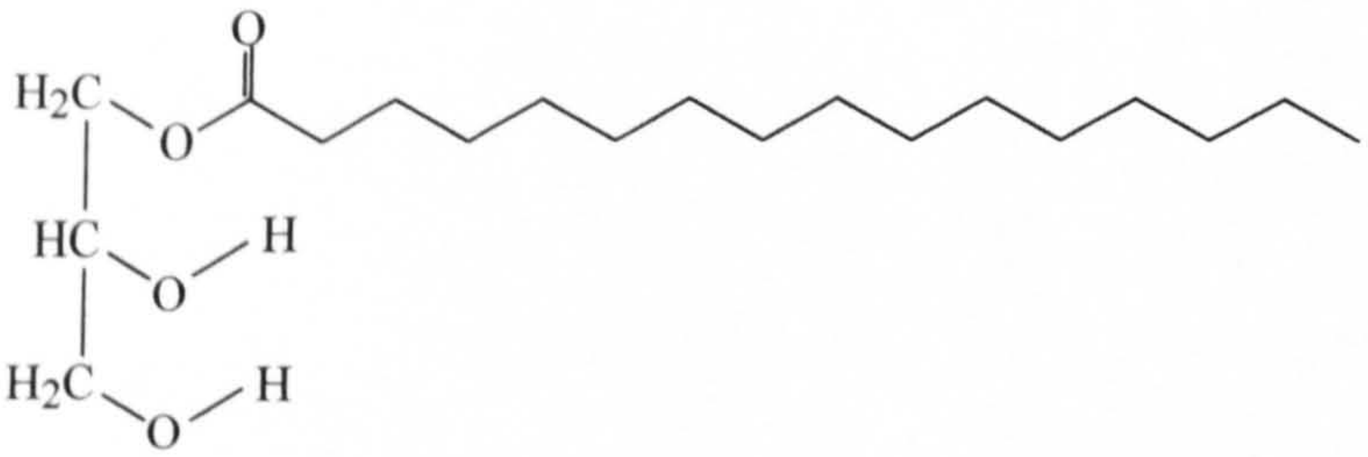
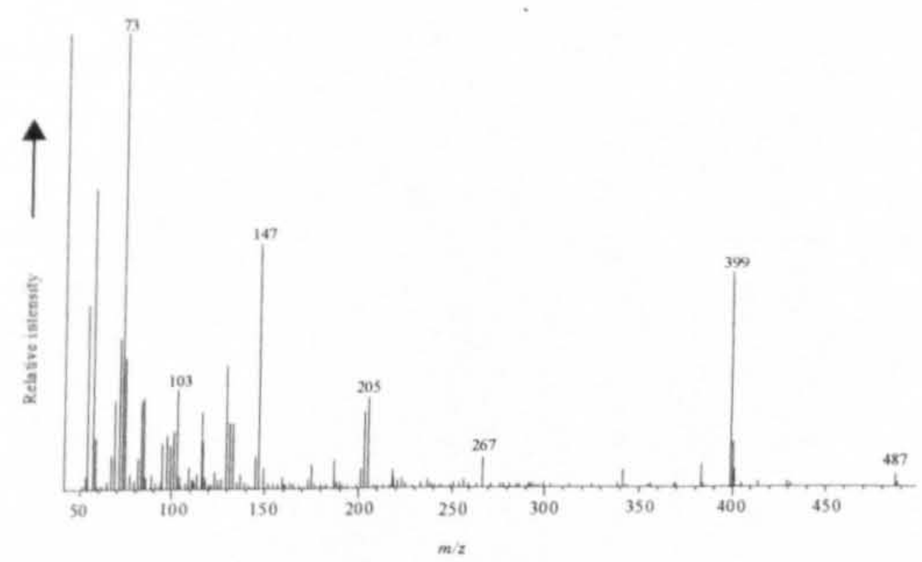


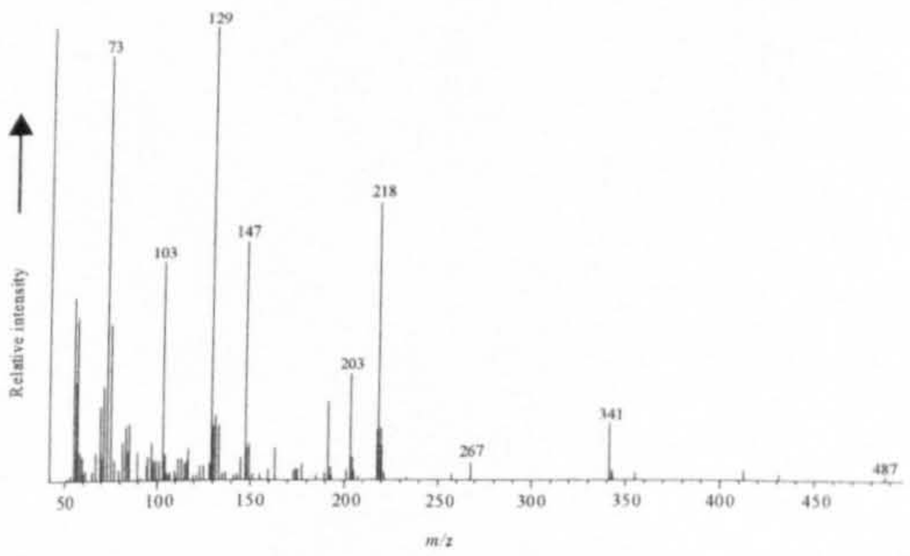
Figure A2.1 cont.

1 Monostearin





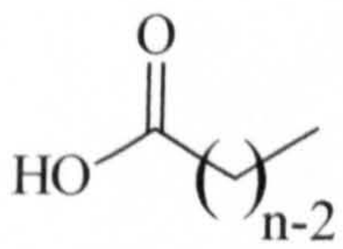
Mass spectrum of TMS derivative of 1-monostearin ($M^{+\cdot}$ 502)



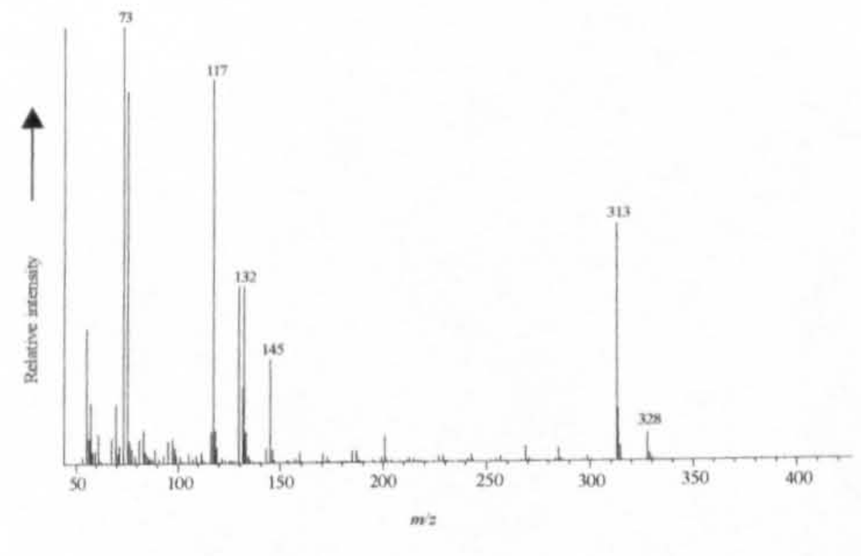
Mass spectrum of TMS derivative of 2-monostearin ($M^{+\cdot}$ 502)

Figure A2.1 cont.

n-Alkanoic acid



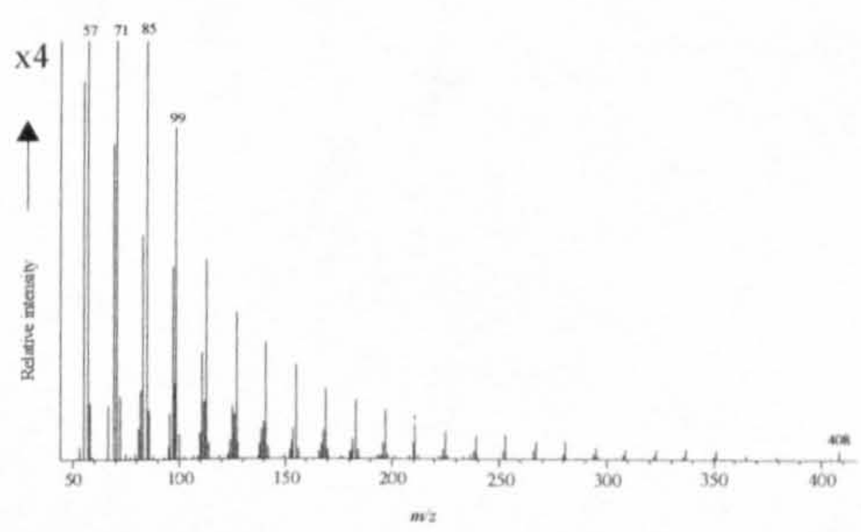
E.g.



Mass spectrum of TMS derivative of $C_{16:0}$ fatty acid ($M^{+\cdot}$ 328)

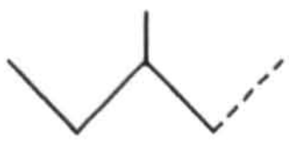
n-Alkane

E.g.



Mass spectrum of C_{29} *n*-alkane ($M^{+\cdot}$408)

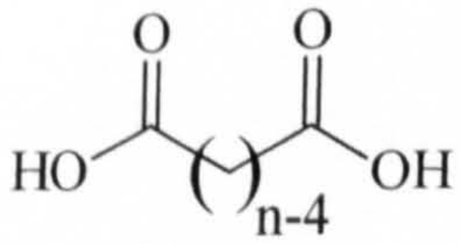
iso-



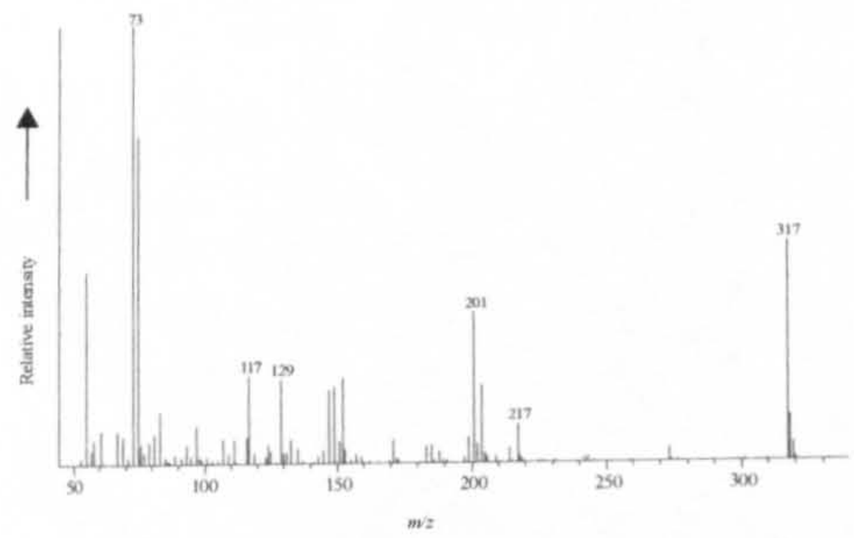
anteiso-

Figure A2.1 cont.

α,ϖ-Dicarboxylic acid

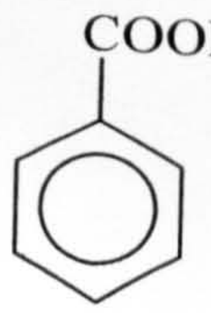


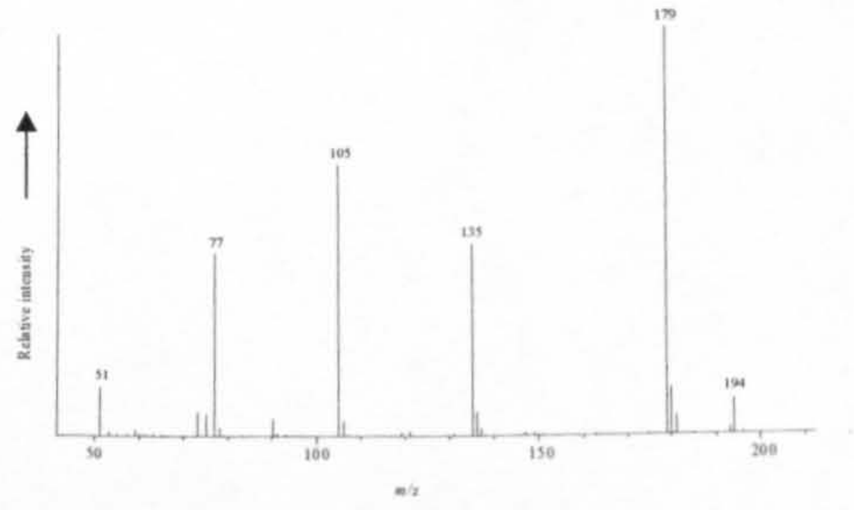
E.g.



Mass spectrum of TMS derivative of C_9 α,ϖ-dicarboxylic acid ($M^{+\cdot}$ 332)

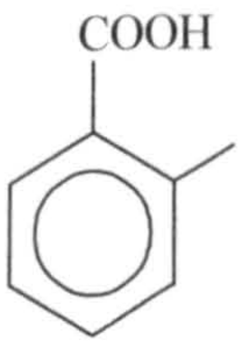
Benzoic acid

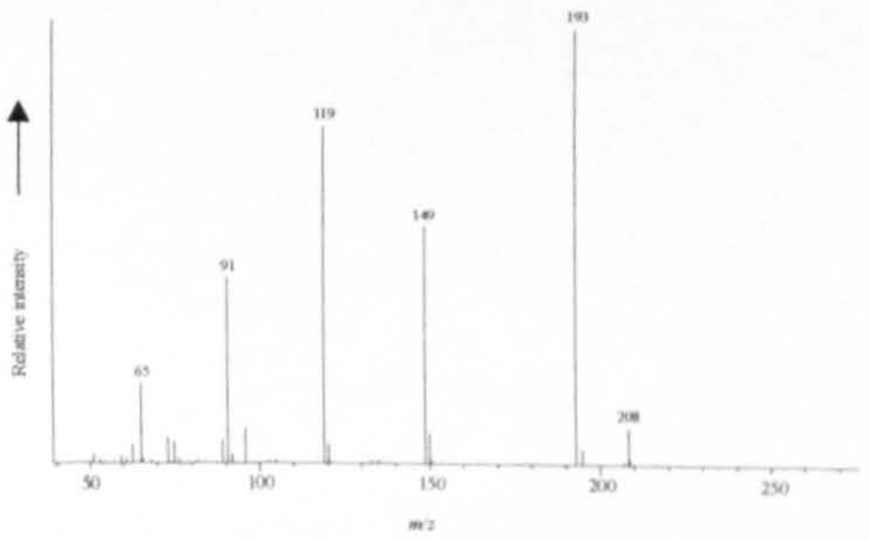




Mass spectrum of TMS derivative of benzoic acid ($M^{+\cdot}$ 194)

2-Methyl benzoic acid

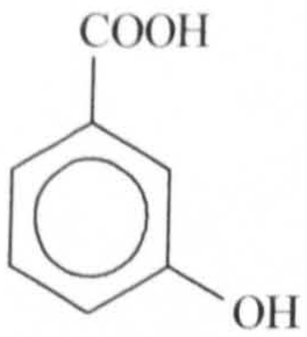


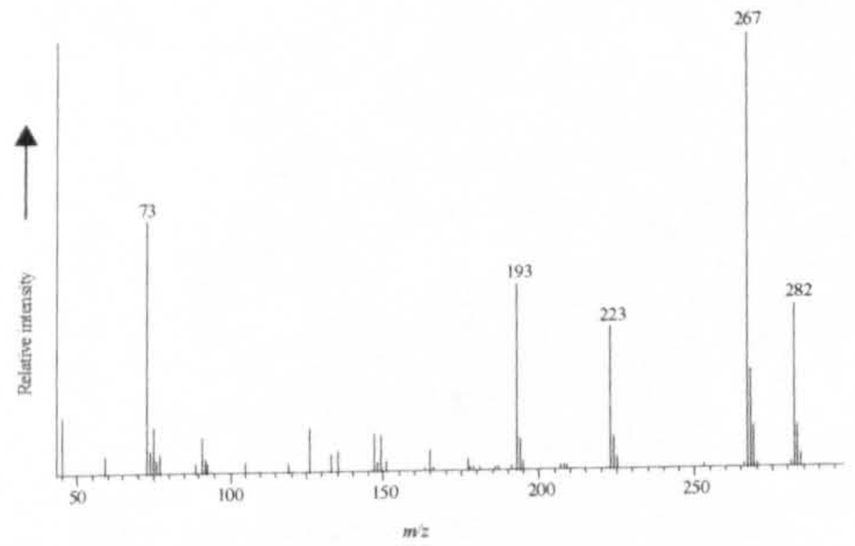


Mass spectrum of TMS derivative of 2-methyl benzoic acid ($M^{+\cdot}$ 208)

Figure A2.1 cont.

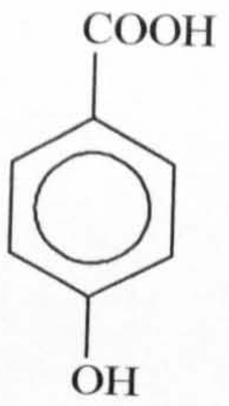
3-Hydroxy benzoic acid

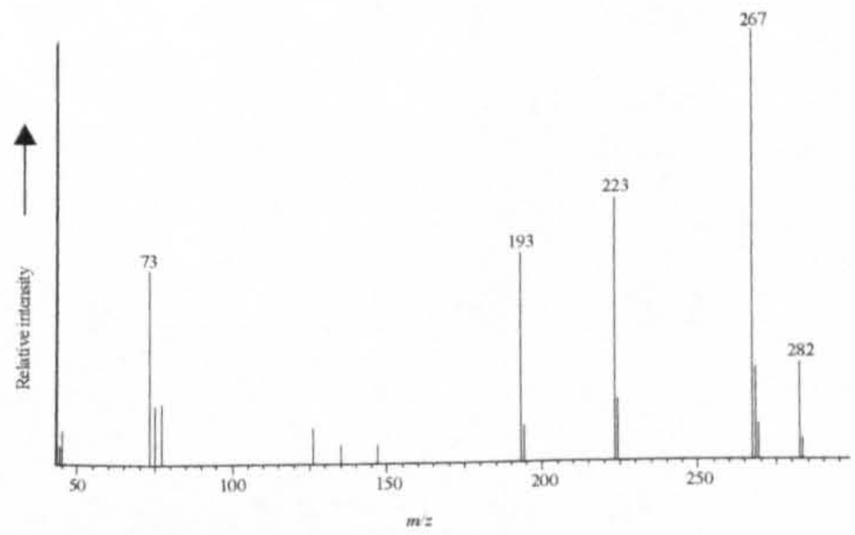




Mass spectrum of TMS derivative of 3-hydroxy benzoic acid ($M^{+\cdot}$ 282)

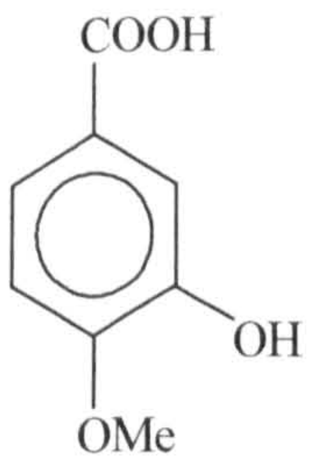
4-Hydroxy benzoic acid

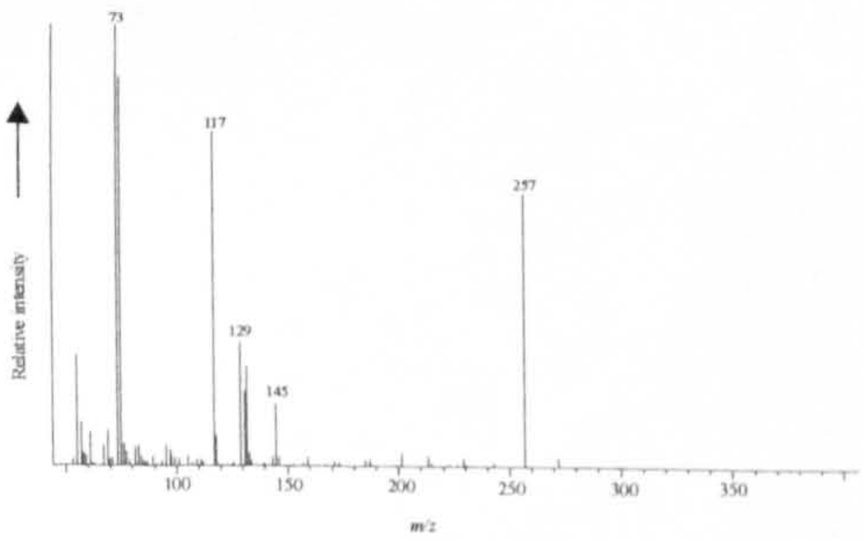




Mass spectrum of TMS derivative of 4-hydroxy benzoic acid ($M^{+\cdot}$ 282)

4-Methoxy, 3-hydroxy benzoic acid

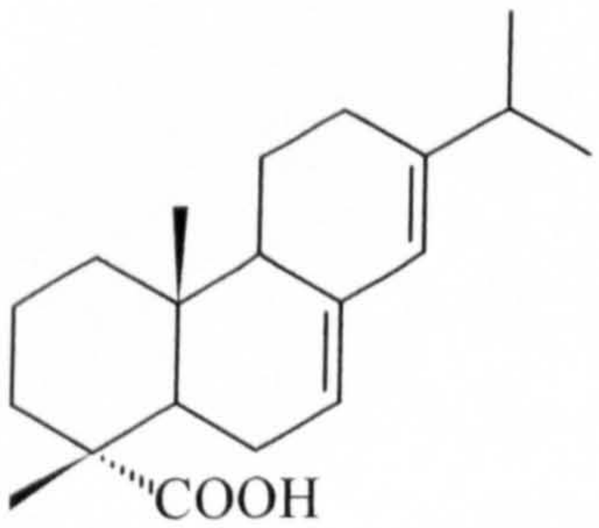


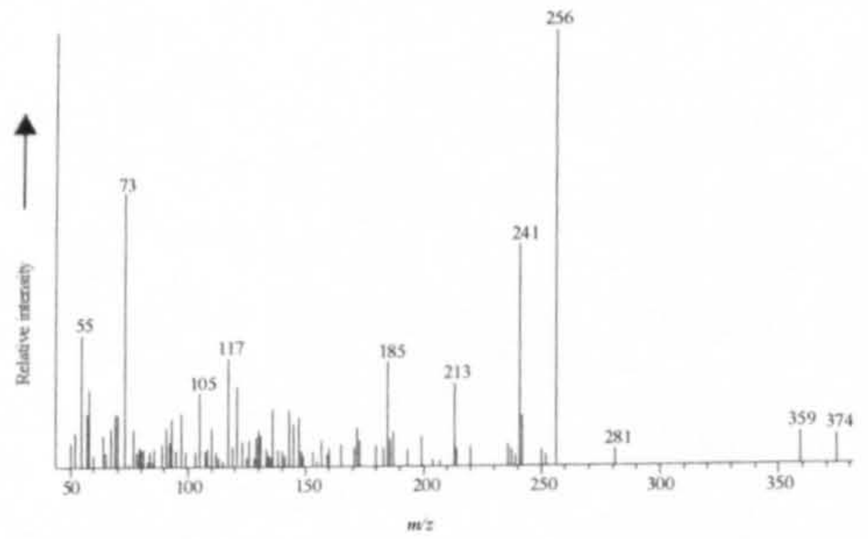


Mass spectrum of TMS derivative of 4-methoxy, 3-hydroxy benzoic acid ($M^{+\cdot}$ 312). On a DB1 high temperature GC column, this compound elutes just before $C_{12:0}$ fatty acid.

Figure A2.1 cont.

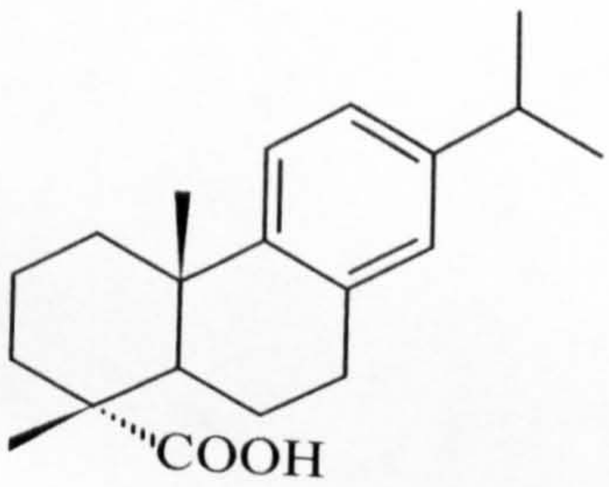
Abietic acid

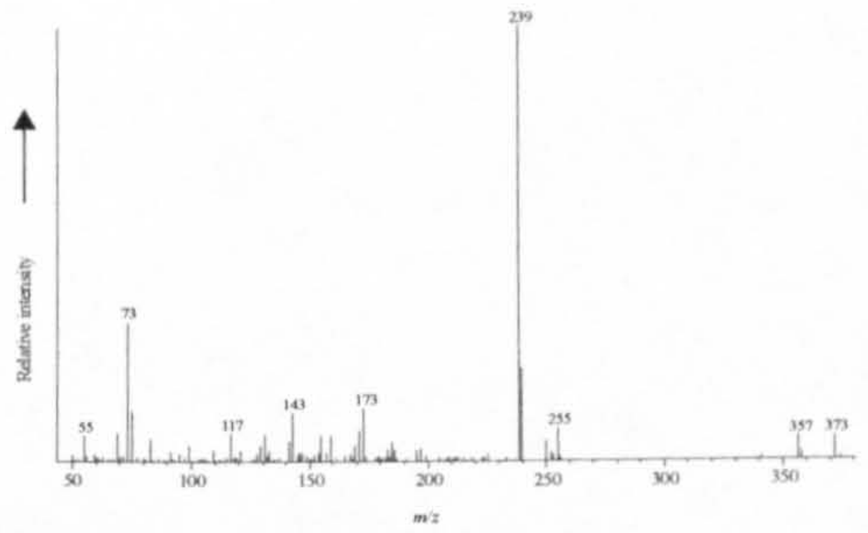




Mass spectrum of TMS derivative of abietic acid ($M^{+\cdot}$ 374)

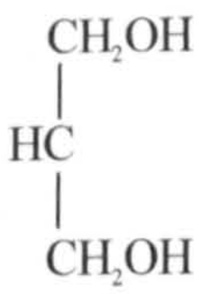
Dehydroabietic acid

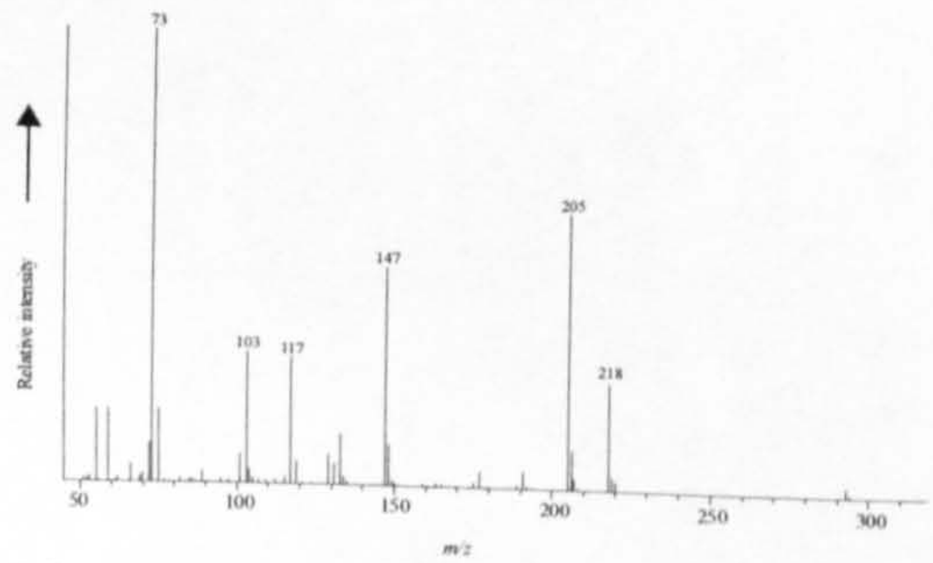




Mass spectrum of TMS derivative of dehydroabietic acid ($M^{+\cdot}$ 372)

Glycerol

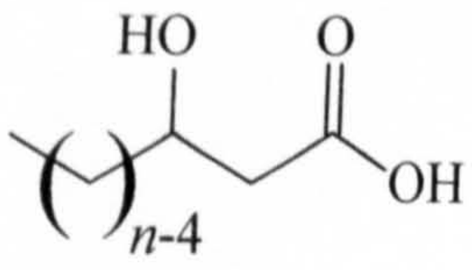




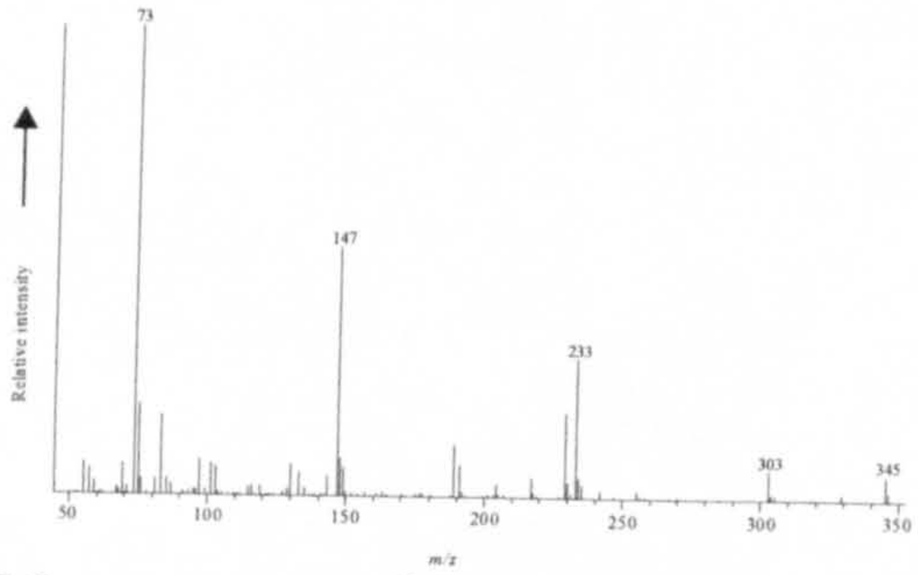
Mass spectrum of TMS derivative of glycerol ($M^{+\cdot}$ 218)

Figure A2.1 cont.

3-Hydroxy fatty acid

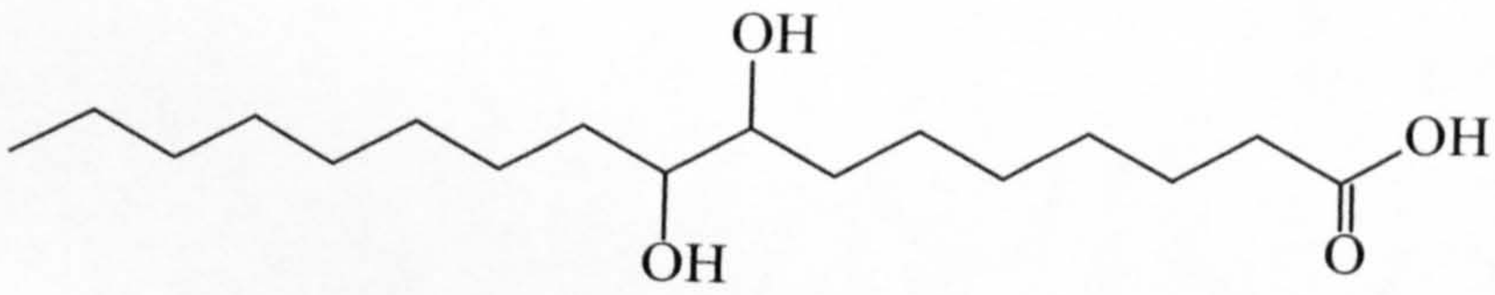


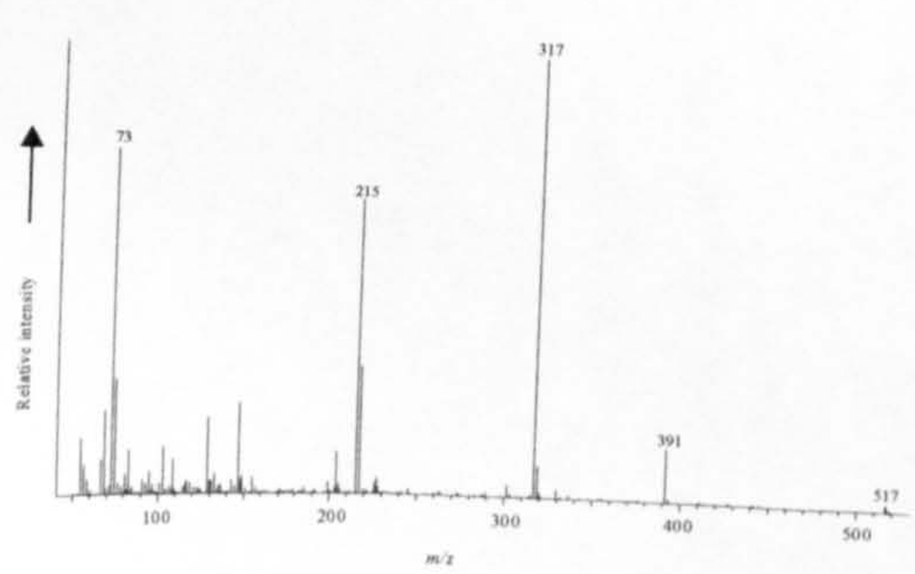
E.g.



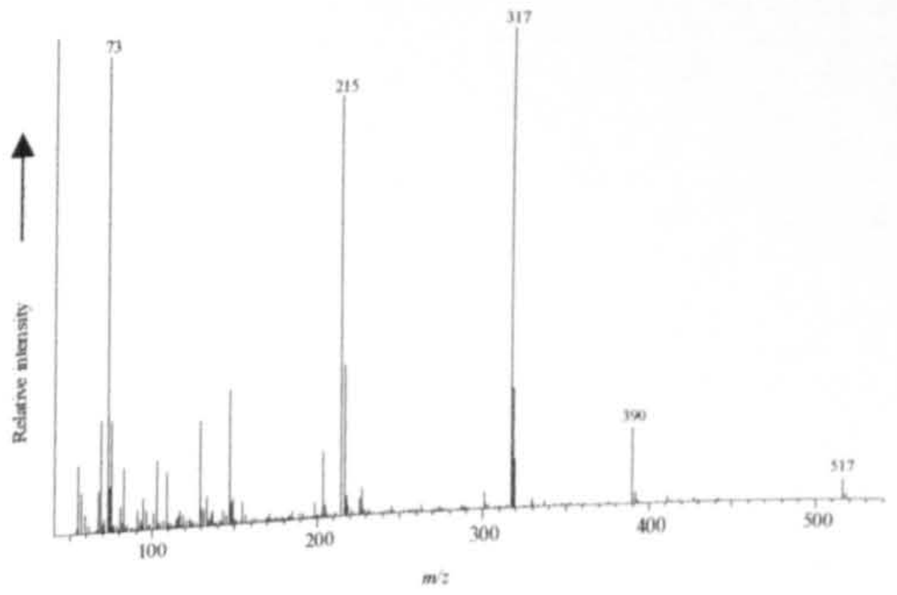
Mass spectrum of TMS derivative of 3-hydroxylauric acid ($M^{+\cdot}$ 416)

Z- 9,10-Dihydroxyoctadecanoic acid





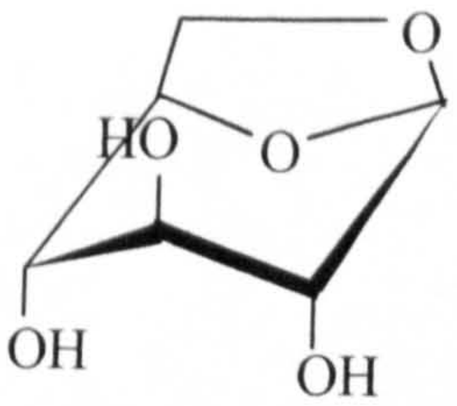
Mass spectrum of TMS derivative of *threo* isomer ($M^{+\cdot}$ 532)

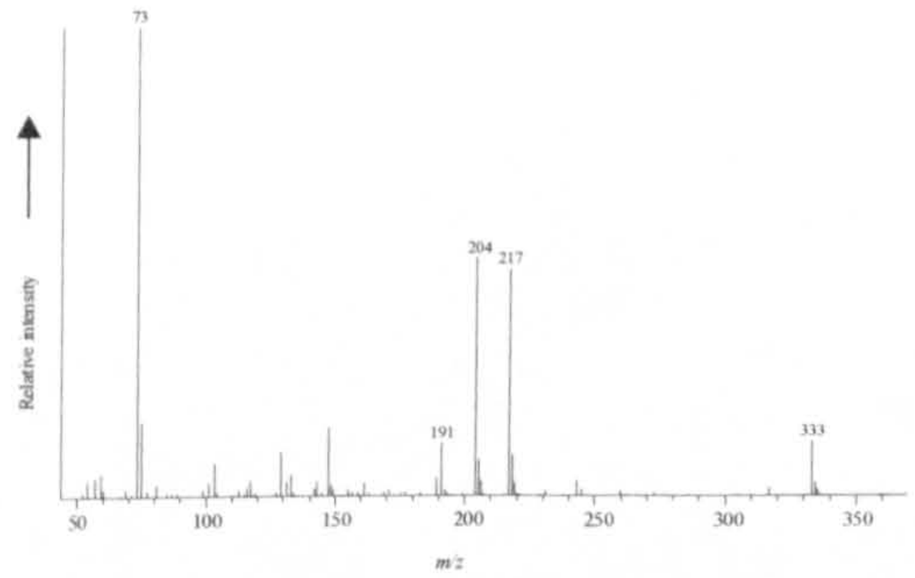


Mass spectrum of TMS derivative of *erythro* isomer ($M^{+\cdot}$ 532)

Figure A2.1 cont.

Levoglucosan (1,6-anhydro-β-D-glucopyranose)





Mass spectrum of TMS derivative of levoglucosan ($M^{+\cdot}$ 378)

Figure A2.1 cont.

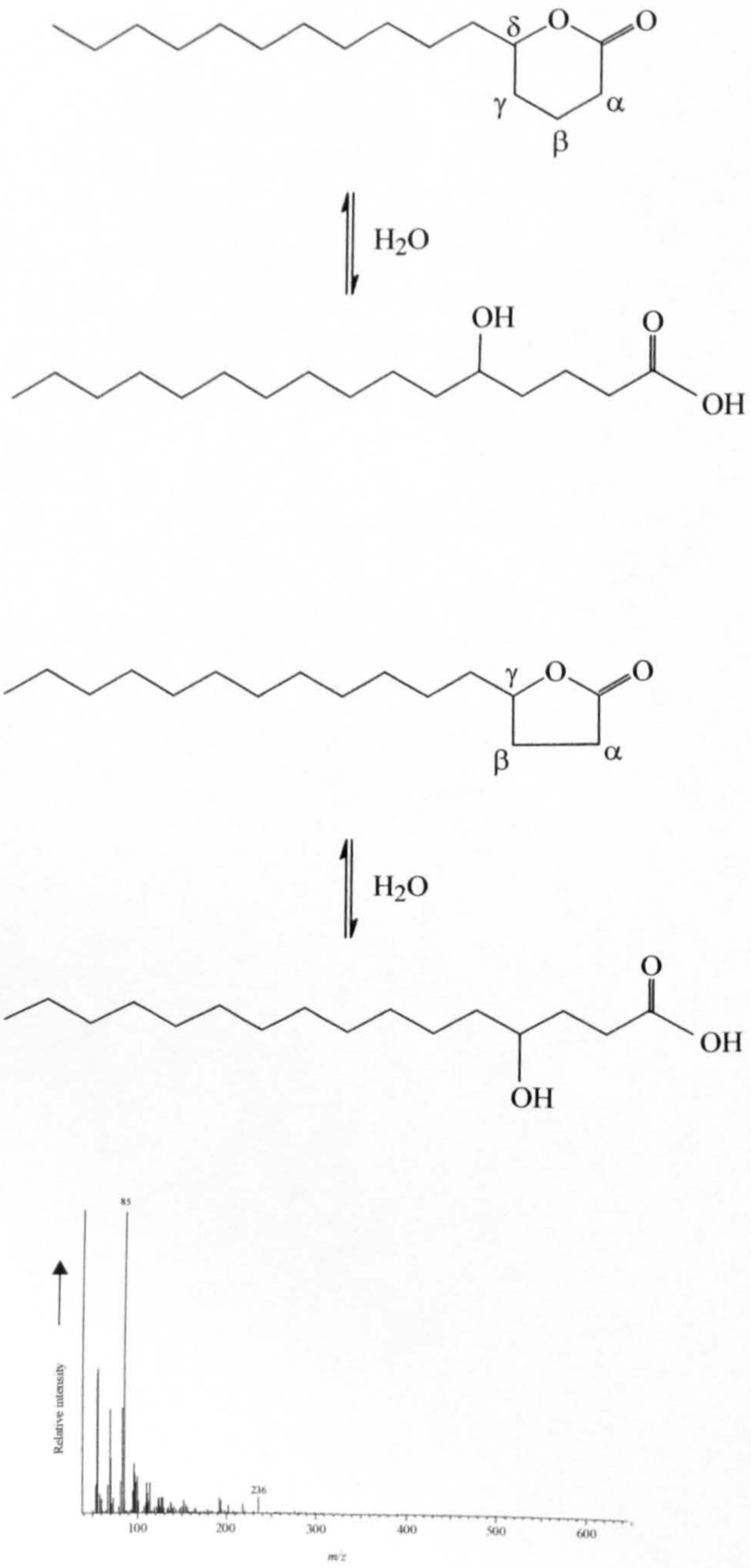


Figure A2.2 The formation of δ- (top) and γ- (middle) lactones through the elimination of water. The $C_{16:0}$ lactones are shown as examples. Mass spectrum of TMS derivative of $C_{16:0}$ γ-lactone ($M^{+\cdot}$ 254). δ-lactones have base peaks of 99, but no other diagnostic fragment ions.

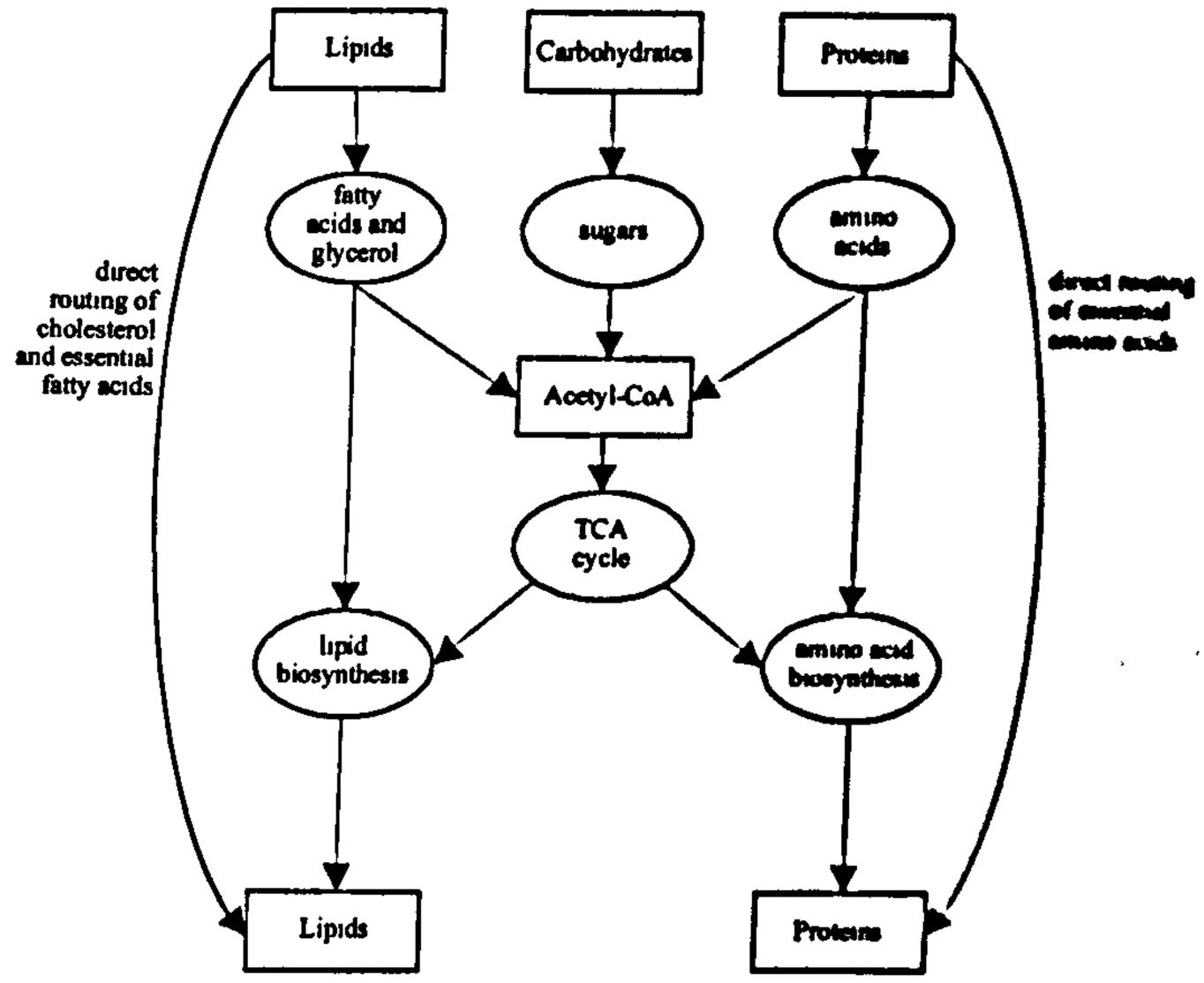


Figure A2.3 The metabolism of dietary lipids, carbohydrates and proteins (after Jim, 2000:36). TCA cycle refers to the Tricarboxylic Acid cycle (refer to Voet and Voet (1995) for more detailed information)

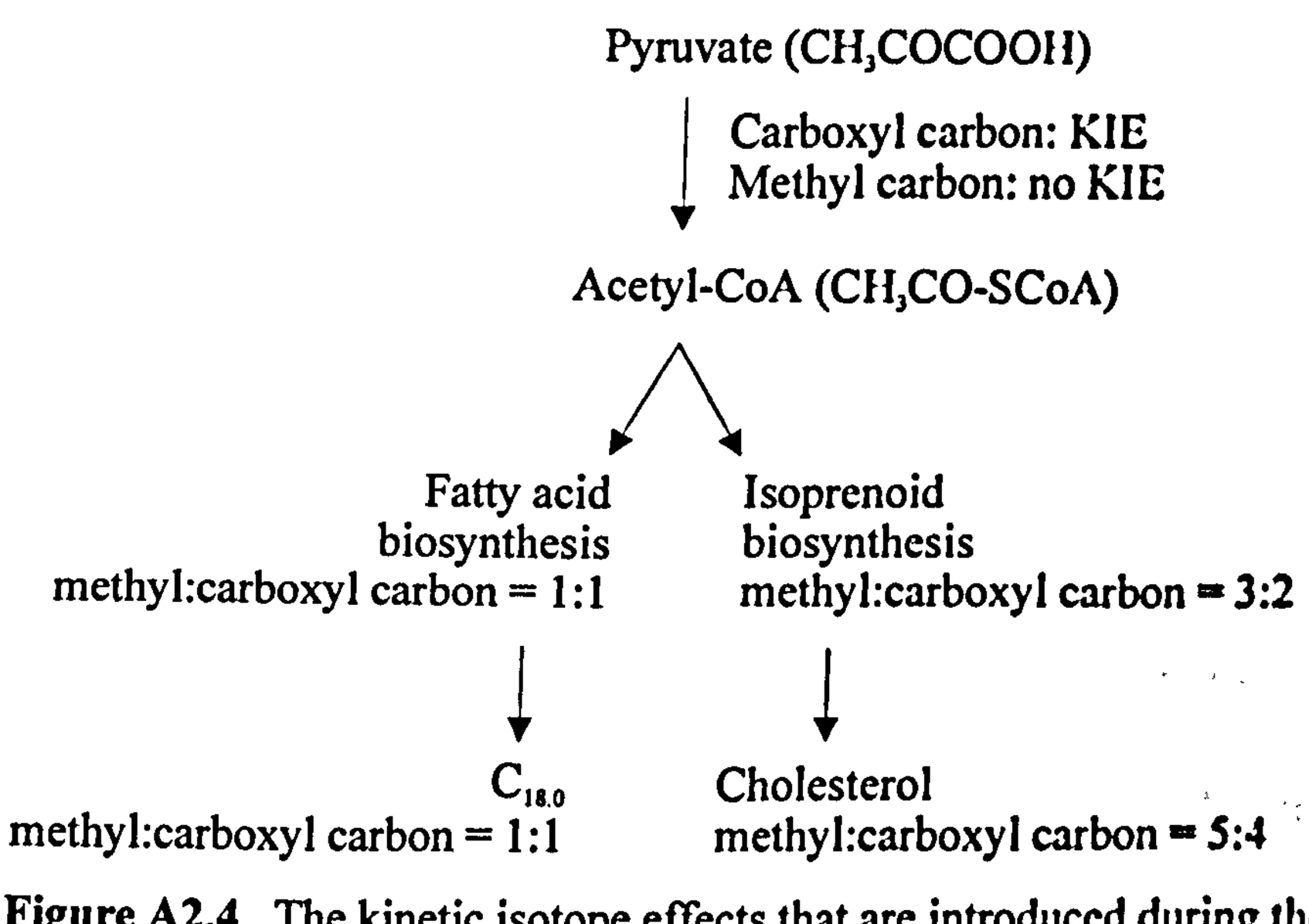


Figure A2.4 The kinetic isotope effects that are introduced during the biosynthesis of cholesterol and fatty acids (after Hayes, 1993). There is a kinetic isotope effect (KIE) during the pyruvate dehydrogenase complex reaction, where the carboxyl carbon in acetyl-CoA is depleted in ^{13}C with respect to pyruvate. However, the methyl carbon is not fractionated against, with respect to the pyruvate. Since cholesterol contains more of the methyl carbons, it will be less depleted in ^{13}C compared to even-chain fatty acids. N.b. this does not take into account of cholesterol that directly routed to the tissue (see Fig. A2.3; Jim (2000)).

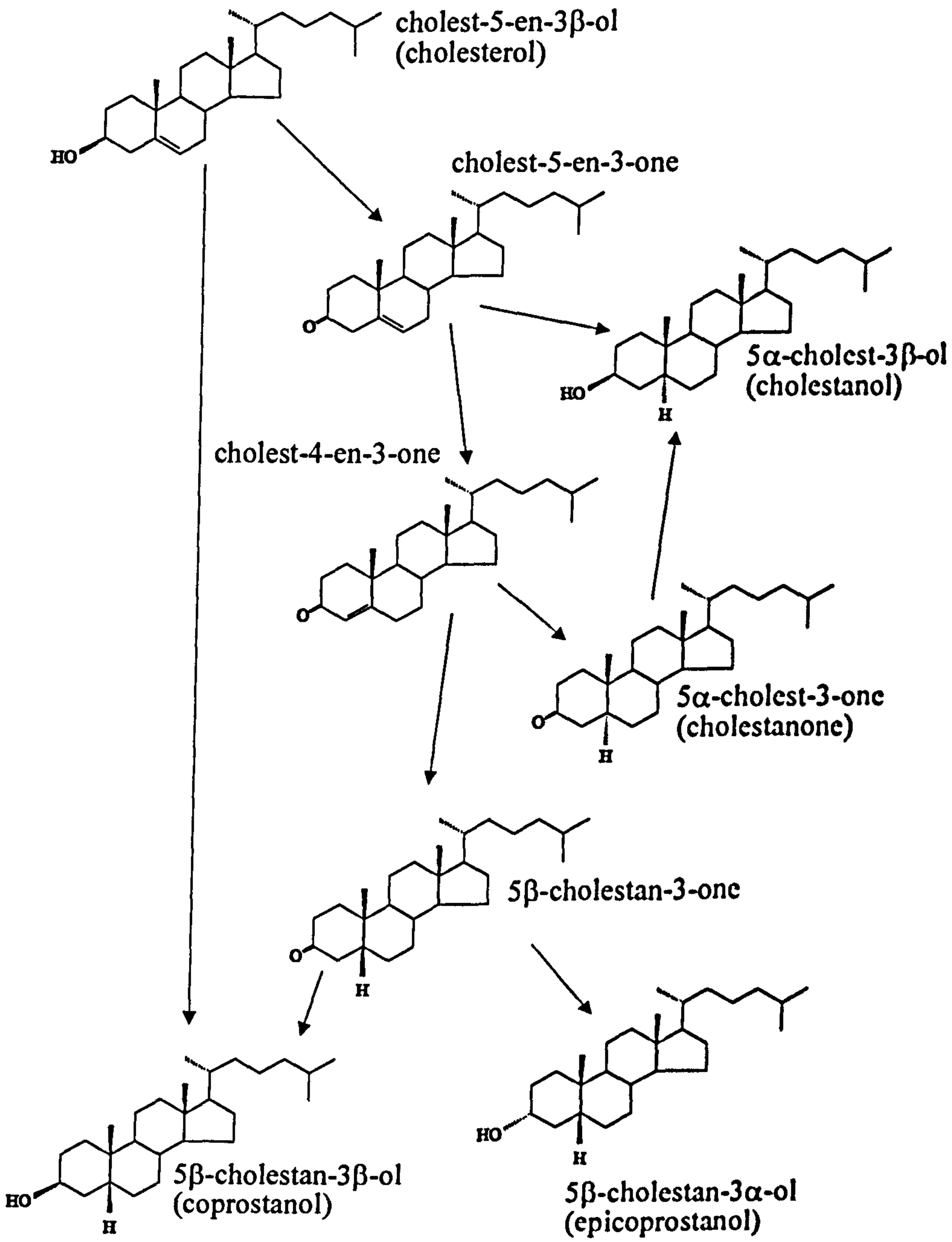


Figure A2.5 The reduction of cholesterol to 5α- and 5β-stanols (after Ren *et al.*, 1996).