Universitat Autònoma de Barcelona

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Al meu germà,

Esta tesis se ha realizado en el marco de los proyectos de investigación:

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ABSTRACT (Catalan)

A partir del Neolític es constaten una sèrie de profunds canvis econòmics i socials que afectaran el mode de vida de les poblacions, especialment a la Mediterrània. En aquest marc espacial, la difusió de la ceràmica y la substitució de les pràctiques caçadores-recol·lectores-pescadores per un model agropecuari esdevindran les causes del canvi en l'alimentació. La possibilitat de fer bullir o barrejar els aliments permetrà obrir un nou ventall de productes alimentaris y unes noves practiques culinàries.

La incorporació de **noves tècniques analítiques en Arqueologia** ofereix el coneixement de noves dades que fins ara ens eren desconegudes sobre com es va desenvolupar el Neolític al nostre territori. Tanmateix, les dades de que disposem de la Península Ibérica pel Neolític en termes d'alimentació és, encara, escàs.

En aquesta tesi doctoral ens hem proposat generar coneixement y aportar noves dades en aquests canvis culinaris a partir de l'anàlisi molecular dels residus orgànics preservats en els recipients ceràmics a l'extrem occidental de la Mediterrània entre el 5600 y el 3800 cal BC.

Un total de 205 vasos ceràmics han estat analitzats y un 62% han preservats lípids arqueològics que provenen de la degradació de productes animals y vegetals que es van processar a l'interior dels vasos. A partir de la caracterització d'aquests compostos s'ha pogut constatar la explotació dels recursos disponibles, com la **cera d'abella més antiga** identificada dins d'un recipient fins a l'actualitat en la Península Ibérica.

Per tal d'analitzar la **composició isotòpica** de compostos específics s'ha dissenyat un **model referencial** de greixos animals procedents de l'àrea geogràfica estudiada amb l'objectiu, no només d'incloure especies potencialment consumibles al Neolític y fins ara no contemplades, sinó també proposar una aproximació més acurada de les diferents especies animals que conformarien les practiques culinàries en un moment on convergeixen especies domèstiques y salvatges.

La caracterització de greixos animals en els recipients ceràmics neolítics ha permès constatar la **evidencia** directa de processat de **productes làctics** des del sud de la Península Ibérica fins els Pirineus. El tractament de les dades des d'un punt de vista pluridisciplinar i transversal ofereix la possibilitat d'anar més enllà en les interpretacions dels resultats i incidir en les pràctiques de subsistència i l'ús dels recipients ceràmics que es donarien en aquest període.

Com a conseqüència d'aquesta recerca, s'obre una nova porta a l'**estudi del recipients ceràmics** més enllà de les decoracions i que ofereix una gran informació sobre l'**economia** i l'**explotació dels recursos** en el moment on s'inicia el model de vida que marcarà el futur de les comunitats mediterrànies.

ABSTRACT (English)

The onset of the Neolithic period is marked by a series of profound economic and social changes which affected the way of life of populations, especially, in the Mediterranean. In this spatial framework, the dissemination of pottery and the replacement of hunting-harvesters-fishermen practices by a farming model became the cause of change in the modes of food consumption. The possibility of boiling or mixing foodstuffs allowed people to access a new range of food products and culinary practices. However, the data that are available on these issues for the Neolithic in the Iberian Peninsula are scarce. In this sense, the incorporation of new analytical techniques in Archeology offers the opportunity to obtain new types of data that, until recently, were unknown about how the Neolithic developed in our territory.

In this doctoral thesis we have aimed to generate new data and knowledge on these culinary changes based on the molecular analysis of organic residues preserved in ceramic vessels in the western part of the Mediterranean between 5600 and 3800 cal BC.

A total of 205 ceramic vessels have been analyzed with organic geochemical techniques, including mass spectrometry and stable isotopic ratio mass spectrometry. About 62% of the vessels studied have yielded archaeological lipids derived from the degradation of animal and plant products that were processed in the vessels. The interpretation of the organic residues is usually quite complex and based on reference models from specific regions of Europe. To tackle this issue a new reference model of animal fats has been assembled for our target study region. The aim is not only to consider animal species potentially consumed during the Neolithic in the Iberian Peninsula that hitherto were not studied; but also, to investigate culinary practices that combined different species of domesticated and wild animals.

Based on the characterization of these compounds, the exploitation of available resources has been established, like the use of beeswax. The identification of this residue

is in fact the oldest ever identified in a container to the present day in the Iberian Peninsula. The characterization of animal fats in Neolithic ceramic containers has also allowed us to confirm the direct evidence of the processing of dairy products from the south of the Iberian Peninsula to the Pyrenees. The treatment of the data from a multidisciplinary perspective offers the possibility to go further in the interpretations of the results, and to dwell deeper on the practices of subsistence and the use of the ceramic containers that were used during the Neolithic.

As a result of this research, a new door opens up to the study of ceramic containers that goes beyond the analysis of decorations and offers great new deal of information about the economy and the exploitation of resources at the onset of new way of life that shaped the future of Mediterranean communities.

INTRODUCTION

The development of agriculture and livestock management are considered one of the key milestones in the History of Humanity, the "Neolithic Revolution", in the words of Childe (1936). The implementation of the peasant way of life, based on agricultural activities, took place in a relatively short period of time in different geographical areas (between 8000 and 6000 cal BC), which shows the expansive potential of this new economic form (Ibáñez et al. 2018). This novel production system spread throughout the European continent and the Mediterranean area in the time interval between 7000 and 5600 cal BC. The earliest evidence of the presence of Neolithic elements in the Iberian Peninsula is documented ca. 5600 cal BC (Bernabeu et al 2014, Rojo et al. 2015). The Iberian Peninsula is a privileged area for analysing the expansion of agricultural and livestock groups (Bernabeu-Auban and Martí-Oliver 2014, Zilhao 2003) due to its geographical location and the persistence of Mesolithic groups (post quem ca. 6000 cal BC). Besides, archaeological research carried out in the last 15 years indicates that the development of new agricultural practices in this region would be explained by a mixed process of colonization and acculturation with a high relative degree of regional variability (Bernabeu Aubán et al. 2018). On the other hand, other researchers point to an interpretative path that visualizes autochthonous social formations as an active part of change (Soto & Alday, 2017).

Pottery has been widely studied and used as a chronological and cultural marker, particularly through the analysis of its forms and decorations. However, the implication of ceramic production goes beyond formal aspects as it involves multiple work processes. Archaeological and ethnographic research in recent years has highlighted all stages of the ceramic manufacturing process chain, from the selection and supply of raw materials to the use of pottery.

There are various approaches from archaeological research to the study of ceramic vessels. These approaches take into account relevant social aspects linked to their production, their economic function, technology and functionality (Rice, 2015; Skibo, 2013).

Today, biomolecular analyses of organic residue preserved in the ceramic matrix are becoming increasingly important in research and they are applied in a systematic manner. These analyses prove the presence of specific productive activities and provide significant data regarding the use of vessels in the past, providing crucial insights into the knowledge of foodways and cooking practices.

For the last thirty years, biomolecular archaeology has focused on the study of the remains of organic substances, especially lipids, through the development of analytical chemistry techniques (Evershed, 1993). Experimental archaeology has enabled a better understanding of the molecular transformations caused by the firing of natural substances during processing, and the degradation processes these substances undergo to identify them in archaeological material (Duce et al. 2015; Isaksson et al. 2010; Lucquin, Gibbs, et al. 2016a; Marchand-Geneste & Carpy, 2003; Pecci et al. 2013; Perruchini et al. 2018; Regert, 2011; Roffet-Salque et al. 2017a; Salque et al. 2012a). The data collected has allowed archaeologists to develop and establish analytical protocols to reliably identify a wide range of products from the detection and interpretation of biomarkers inside the ceramics, the techniques of processing, firing and the dynamics of degradation to which the products have been subjected are evidenced. Among this range of products, we find animal fats (Ethier et al. 2017; Isaksson & Hallgren, 2012; Mukherjee et al. 2007, 2008; Soberl et al. 2014) and shellfish (Craig et al. 2011; Cramp, Jones, et al. 2014; Lucquin, Colonese, et al. 2016) to vegetable oils (Colonese et al. 2017; Dunne et al. 2018; Heron et al. 2016; Steele et al. 2010) and waxy materials of bee or vegetable origin (Evershed et al. 2003; Matlova et al. 2017; Regert et al. 2001). The finger-prints from different food sources (animal fats, plant oils/waxes, beeswax, resins) can be discriminated by mass spectrometry analysis. In addition, through compound specific stable carbon isotope analysis of individual fatty acids, different types of animal fats (e.g. dairy fats, non-ruminants, ruminants) can be separated.

The application of biomolecular analysis techniques to the study of archaeological pottery has provided valuable information on the change in diet that occurs from the Neolithic in prehistoric populations. The study of animal and vegetable fats, in parallel with research in archaeozoology and archaeobotany, has led to a better understanding of the management of domestic and wild resources, particularly for the production and processing of products of animal origin. On the other hand, pottery function analysis (Rice, 2015; Skibo, 2013), together with data obtained from ethnography (although they are still relatively scarce) make it possible to explain the relationship between the form and function of the containers used by prehistoric groups. The analysis of the organic residues preserved in the ceramic matrix offers a complementary approximation to the ceramic use and that constitutes one of the only evidences of what sort of foodstuffs contained the vessel in the past, contributing towards a better understanding of the use of pottery (Fanti et al. 2018; Roffet-Salque et al. 2017b; Vieugué, 2015; Vieugué et al. 2016).

The main objective of this thesis is to generate knowledge and provide new data on the change in diet that occurred with the new production strategies, such as agriculture and livestock, which are developed in the extreme western Mediterranean area at the beginning of the Holocene, covering a time interval between 5600 and 3800 cal BC.

These changes are reflected in the use of ceramic vessels. Given the evidence of food preparation activities and the storage of vegetable and animal products of domestic or wild origin in containers, we consider inferring in the subsistence strategies and management of domestic animals, as well as in the techniques of maintenance, processing and cooking of food products. It is, therefore, necessary to integrate these studies with archaeozoological and archaeobotanical data to provide a higher degree of representativeness of the food contained in pots against consumption strategies and diets defined from other types of study.

Through the identification of the different products processed inside the potsherds, the identification of the different products processed inside the potsherds is intended to contrast whether some containers were designed and produced for specific uses, in which case a correlation between the shape and the function of the container would be documented. The aim is also to characterise the culinary guidelines in greater depth and to assess whether or not significant changes have taken place concerning previous occasions. The aim is also to evaluate the scope and limitations of functional studies on ceramic remains, as the containers often contain different types of natural substances that may respond to the mixture of these products in response to specific culinary guidelines or the repeated use of pots with different food products.

At a methodological level, this doctoral thesis contemplates the design and realisation of a new experimental reference to improve the interpretation of the origin of animal fats by constructing a reference model with samples of current animals from controlled environments and farms in the Iberian Peninsula.

Finally, this work aims to apply this methodology to the study of a significant set of samples from a series of Iberian Peninsula archaeological site located in different geographical areas and chronologies ranging from 5500 to 3800 cal BC. In addition to analysing and obtaining new data on the contents of the vessels, the analysis will also be oriented towards the taphonomic slope. The systematic sampling of different environments will allow the correlation of the food products detected in the containers with the faunal remains consumed in each site, to evaluate the representativeness of the lipids on food consumption.

To achieve these objectives, this work proposes the application of an analysis methodology specific to the field of organic chemistry, such as the identification of organic residues by gas chromatography and the characterisation of their isotopic values, together with the morphological study of the ceramic vessels and the statistical analysis of the results. This methodological strategy is integrated with archaeological, archaeozoological and archaeobotanical data to characterize the culinary and consumption practices of the first peasant societies in the Mediterranean basin of the Iberian Peninsula.

In short, this thesis presents a new state of the question on the problems related to the Neolithisation process of the western end of the Mediterranean, providing new data and proposing new hypotheses and explanatory models around one of the most transcendental historical changes: the origin of peasant and livestock societies.

Chapter I. Food production and consumption at a time of change: the Neolithic as a historical turning point

1.1. Theoretical-conceptual framework

The Neolithic is conceived, from the theoretical perspective of Marxism, as the result of the progressive transformation of hunter-gatherer societies into peasant societies, as a consequence of their own internal conflicts. For Testart (1982), the development of storage, both of wild and domestic products, will be fundamental in the gestation of this conflict, while for Vincent (1990), the loss of inter and intra-group reciprocity in the hunter-gatherer-fisher communities is the reflection of the tensions that existed in the social relations of these groups. This change in the "infrastructure" of society, that is, in the forces and social relations of production, will also generate transformations in the political and ideological "superstructure". In this same context the appearance of ceramics is interpreted, not as an ethnic or cultural marker as conceived from the historical-cultural paradigm, but as a response to the needs of a new economic, social and ideological reality (Vincent 1990). Therefore, for Marxist authors the process of neolitization should not be defined from a supposed typological dichotomy between epipaleolithic-mesolithic and neolithic elements, but as the dismantling of a conflicting social formation and the emergence of a new one.

Thus, **neolitization** is the complex processes that led to the expansion of agriculture and livestock, and the related social, material, ideological, etc. transformations. The different explanations that have been given for this process are determined by several factors: a) the register and the available data; b) the geographical scale used, whether continental, regional or local, and c) the school of thought of each researcher, since the different theoretical frameworks will start from different premises, even in relation to the very meaning of the Neolithic concept. Another of the widely discussed concepts regarding neolitization is the concept of domestication.

Around **animal domestication**, this concept has been treated and characterized as a new production process whose practice has meant important changes in the subsistence practices. From a historical point of view, animal domestication has been approached from different perspectives that coincide in considering the domestication of animals as a complex process in which the interaction between human and animal communities played a fundamental role within the level of subsistence strategies (Saña 2009). Some emphasize the degree of cultural control over natural populations of particular species (Higgs 1969). However, authors such as Ingold emphasize the social order aspects of domestication as well as the social appropriation of successive generations of animals (Ingold 1988). The social appropriation of the resources that entails the animal domestication, until then of collective access, supposes a development in the social forms that legitimate the different use of the resources (Saña 2005). Consequently, the social form that regulates the maintenance, control and use of domestic animals implies a series of work processes. This causes the live animal to constitute a means of production and a product. The difference between these two concepts lies in the type of management: when the animal is a product it is sacrificed, whereas when the objective of animal management is its insertion in the means of production, it is maintained as a permanent resource that requires maintenance and reproduction (Saña 2005, Redman 1990).

With all this, when we talk about livestock, we will refer to the set of production processes aimed at maintaining the animals and obtaining their products (Saña 1999).

We find the same debate when we talk about **agricultural domestication** and plant products. Following Ford's definition of plant food production, this implies a deliberate manipulation of specific species for use or consumption (Ford 1985). This manipulation is established by means of different production methods related to maintenance, in which forage groups or collectors would not participate. However, this implies a gradual process on the part of humans that would always culminate in the cultivation of any type of plant (Antolín, 2013). Many authors have tried to define the intermediate stages between cultivation (of wild plants) and agriculture (with domestic taxa) (Fuller 2007). On the contrary, Ingold asserted that agriculture is not about producing food, but cultivating it (Ingold 1996). Thus, farmers establish the conditions for the growth of those plants that they decide are the most appropriate.

The integration of domestic plants and animals into the economic strategies of Neolithic societies involved the implementation of new work processes linked to their maintenance, breeding and reproduction and, with this, the establishment of new production relations. Within the framework of these, it was essential to provide the availability of seeds for the new agricultural cycles and the feeding and demographic control of the herds of domestic animals.

Farmers needed reliable food supplies throughout the year, which, at least sometimes, involved storage. Storage has been considered a necessary precursor to agriculture (Bender 1978), as a concomitant feature of sedentary life (Testart 1982). This indicator of socio-cultural complexity (Price and Brown 1985, Flannery 1972), promotes an important step in the conceptualization of private property (Bettinger 1999) and social control (Wesson 1999).

The ability to manipulate the availability of food, both wild and domestic, and to regularly exceed the seasonal calendar of availability, in both good and bad years, is a fundamental basis for the emergence of social differentiation in communities. In many geographical and temporal contexts, food storage precedes the domestication of plants, although storage does not automatically lead to food surpluses (Kuijt 2011). According to some researchers, this generates a problem not of economic, but social root: the appropriation of resources (Gamble 1986). The pre-existing organization, based on "generalized reciprocity" both within the group and in inter-group relations, would exclude the possibility of appropriation and lack categories for it (Vincent-García 1991). For these authors, the passage to the peasant way of life would imply the appropriation, both of production and the means of production (Gilman 1984, Testart 1982).

The domestication of animals and plants therefore led to control over the seasonal availability of certain food resources, while at the same time making it possible to include new sources of protein in the diet. Both the storage and processing need of new products have been closely linked to the third element that has been materially characterizing neolitization: ceramics.

Ceramics constitute the category of archaeological materials object of study in this thesis, with the objective of knowing the role it had in the changes that occurred in the strategies of subsistence and feeding during this time interval, in a concrete way for the Iberian Peninsula. In order to do so, it will be essential to know how to use them. Due to their relative abundance, ceramic products constitute a very important part of the material testimonies that have reached us from the human communities of the past. In their role as containers, ceramic containers are tools (Braun 1983) and a subset of a much broader category of utilitarian devices called facilities (Wagner 1960). Ceramic containers are made with the aim of participating in one way or another in a wide variety of work processes within the framework of the different production and reproduction activities carried out by the people who manufacture and/or use them (Clop 2002). Furthermore, these products are not necessarily passive containers; they can increase the usefulness of their contents by extending their useful life or by allowing them to be transformed by different types of energy (Rice, 2015).

For this reason, a distinction needs to be made between the concept of **function** and that of use. In archaeology, various types of indirect evidence can be provided to try to understand the functionality of ceramic vessels. It is important to recognize the difference between direct and indirect evidence, because it will be what marks the use of one or the other concept. Indirect approaches allow us to know the function of vessels, from the context of recovery (Schiffer 1976), ethnography, experimental archaeology (Skibo, 2013), and studies of vessel shape, including physical, technological and mechanical skills (Rice, 2015). As we seek to make more refined inferences, it is imperative that we begin with as specific information as possible on how the package was used throughout its useful life (Skibo, 2013). In contrast, the actual function does not always correspond to the intended function. The pots may be designed to cook stews, but the home may need a storage jar, so the pot is put into service for a function the potter never intended to do. In this way, we will talk about **use**, and more specifically effective use, when we know the content resulting from a certain use of the container or repeated occasions. Biomolecular analyses of the organic residue eventually conserved in its interior will allow us to approach this use.

1.2. Neolithic in the Iberian Peninsula

1.2.1. Explanatory models on the origins of the Neolithic period

The explanatory models on the origins of the Neolithic can be classified between approaches based on migrationist or diffusionist models, in which the movement of the population of Neolithic groups is fundamental to explain the extension of the new way of life throughout Europe, and the autochthonist or indigenist models, a theoretical postulate that explains the process of neolitization based on the development of the "indigenous" mesolithic populations themselves, where the movement of the population has a very secondary, if not existing, role in this process. With the new findings and the extension of dates, a series of intermediate proposals were developed that would justify a foreign beginning of implantation of these new economic practices in certain sites accompanied by an internal development of the autochthonous populations. Among the proposals that give preminence to the movement of the population, we find the model of démica diffusion, which starts from the first available radiocarbon dating (Clark 1965), detecting a pattern that indicated the expansion of the Neolithic in Europe through the Danube from the southeast, and a later influence in the northern European plain and southern Scandinavia. In the 1970s and 1980s, one of the best-known and most debated neolithic models on the European continent (Ammerman and Cavalli-Sforza 1984) would be described and referred to as the "advance wave". This model is based on the inexistence of an autochthonous domestication in Europe, and on the material and chronological evidences that suggested an expansion of the Neolithic through the continent, with a clear gradation in an east-west direction. The term demic diffusion was coined by these authors to define the type of displacement experienced by the first Neolithic communities, which would not be properly a mass migration, as suggested by genetic analysis, nor a colonization. Rather, it would be a progressive infiltration of individuals or small groups (Bogaard, 2004)(Ammerman 2003: 7). The cultural diffusion would be the other responsible for the Neolithic expansion, understood as the transmission of these technological innovations between the local groups, without population geographical displacement (Marchand & Perrin, 2017). Finally, the causes of this diffusion mechanism were specified in three points: the local growth of the population, the local migratory activity and the delay time. All of them are closely related and start from the assumption that the population growth of the first Neolithic communities was high and after a period of delay, demographic pressure makes emigration necessary. The application of the demic diffusion to the reality of the Iberian Peninsula has given rise to the "dual model" (Bernabeu 1997). According to this model, those responsible for peninsular neolitization would be colonizing neolithic communities that would reach the Iberian Peninsula from the western Mediterranean. The discovery and dating of sites such as El Barranquet (Oliva, Valencia) or Mas d'Is (Penàguila, Alicante) (Bernabeu et al. 2009), relates these contexts with different enclaves of the Italian region of Liguria and the French coast, as in Arene Candide (Liguria, Italy) or Abri Pendimoun (Alpes-maritimes, France) (Guilaine & Manen, 2012). Like the démic diffusion, this proposal opts for a mixed model at the time of explaining the neolitization of the territory, in which the knowledge of the population and the information is combined (Bernabeu 2002). This model is argued on the basis of the significant increase in settlements from 5600 cal BC (Bernabeu et al. 2015), the use of demographic proxies and population density trends have been reported in other regions of Europe (Boquet-Appel 2008, Shennan et al. 2013), where the arrival of agriculture and livestock coincides with the rapid increase in settlements with contemporary radiocarbon dating (McClure et al. 2014)(Boquet-Appel 2012).

In the diffusionist interpretative line are the models of Pídola colonization (leapfrog colonization), which defend a neolithic expansion through phenomena of pioneer colonization on a small scale, but discontinuous in space. These proposals arose from the critique of certain aspects of demic diffusion, such as the gradual expansion in the occupation of space and random expansion in its direction, when the data showed a punctuated and directional spatial pattern, centered in areas near water sources with fertile soils for agricultural development (Sherrat 1980: 87, Van Andel and Runnels 1995: 481). Within this group of proposals, a "pioneering maritime colonization model" was proposed to explain the neolitization of the Iberian Peninsula (Zilhão 1997, 2001). Its fundamental idea is that the Ancient Neolithic in the western Mediterranean is characterized both by the presence of ceramics with cardiac decoration and by a population that presents a discontinuous geographical distribution but a synchronic chronology fruit of a fast and long distance maritime colonization.

Finally, an arrhythmic model of neolitization in Europe was proposed that is out of step with time and with a discontinuous speed (Guilaine & Manen, 2012)(Guilaine 2001). These pauses correspond to moments of "cultural mutation". The causes of these interruptions are due to the varied European environments that imply an adaptation and the search of suitable means to be colonized (Guilaine 2001: 269). All these circumstances would provoke a slowing down of the process and would open a period of transformation of these "primary cultures" that would culminate with a new period of expansion, which would follow two great routes of transmission through Europe, one continental and the other Mediterranean. In some areas, after a rapid maritime propagation, a slower diffusion towards the interior would take place, as in the central plateau of the Iberian Peninsula, whose neolitization would take place at the same time as the "cardial navigators" (Guilaine 2001: 272). However, the latest data available for the interior of the Iberian Peninsula make a revision necessary, as they show that neolitization in this area occurred at the same time as the Neolithic Cardial of the Levantine area (Rojo Guerra et al. 2008).

All these models have received numerous criticisms, especially when they have been intended to be applied on a regional or local scale. In the first place, the existence of continuities of the archaeological record in the Iberian Peninsula that question this rupture of material culture in the model of demic diffusion, based on the lithic industry (Barandiarán and Cava 2000), the style of certain ceramics (Alday et al. 2009) or rock art (Vincent 1990), among other aspects. Secondly, the lack of visible population growth in the archaeological record (Guilaine, 2018; Guilaine & Manen, 2012)(Bernabeu et al. 2009). Finally, a common criticism of diffusionist models has been their tendency to underestimate the level of economic and social development of Mesolithic hunter-gatherer-fishers. On the contrary, the archaeological record shows stable, prosperous, often semi-sedentary hunter-gatherer-fishermen communities capable of maintaining relatively high population densities (Soto & Alday, 2017)(Zvelebil 2001: 382) with appreciable levels of socioeconomic complexity (Sassaman 2004).

In the Iberian Peninsula, alternatives to "diffusionist" models can be considered mixed models. In them a certain degree of population mobility is admitted, but a gradual evolution of local groups towards their full neolitization is advocated. One of the most influential proposals in the Iberian Peninsula has been the "capillary diffusion model" defined by Vincent (1990). From a Marxist theoretical framework, the "Neolithic Revolution" is conceived as the process by means of which the "primitive" and "egalitarian" social formations, based on hunting and gathering economies, are transformed into agrarian societies in which processes of growing social differentiation are developed. Faced with the inter-annual and seasonal variability of hunting and gathering, solutions were put into practice to deal with this danger: the investment of work increased, the complexity of resource management increased, and storage developed. In this context, agriculture and livestock would be assumed as guarantors of an important yield for the subsistence of the group, not so much as an "optimization of production", but as one of the multiple "stabilization techniques" that they developed for this purpose (Vincent 1990: 263). The progressive consolidation of agriculture would originate what he calls "agricultural trap" (Vincent 1990: 275), a dependence on the place where important work investments have been made, whose yield is not immediate, but is collected after some time.

Also from the theoretical coordinates of Marxism, a theoretical model has been proposed in recent years in the area of the Bay of Cadiz that proposes the emergence of the autochthonous and independent production economy in that region, as a result of an internal dynamic of the communities of hunter-gatherer-fishermen and that could materialize with the excavations at the site of El Retamar (Puerto Real, Cadiz) (Ramos 2005).

From historical-cultural theoretical perspectives, some researchers have put forward some hypotheses based on the archaeological work carried out in the Ebro Valley. For Alday (1996), continuity is one of the main characteristics of the Neolithic process, which is why he groups the Final Mesolithic and the Ancient Neolithic in the so-called "first cultural cycle". The Mesolithic settlers themselves will be the agents of change, influenced by the circulation of ideas, goods and people (Alday 2012, Alday et al. 2017). Therefore, the economy of production would be progressively adopted.

As a synthesis, we see that, given the different dynamics that are developed in a contemporary way, regional variability in Europe cannot be explained with any single model proposed until now. In recent years a whole series of excellent research projects on the Ancient Neolithic have been carried out in the Iberian Peninsula. From recent data, neolitization in this area seems to have occurred around 5600 cal BC from the French Cardial and spread south along the Mediterranean coast, with sites such as Mas d'Is (5617-5485 cal BC) (Bernabeu Aubán et al. 2018), to the inland areas (Rojo Guerra et al. 2008). Likewise, recent data on bones of fauna identified as domestic in the Cave of Nerja, offer chronologies around 5600 cal BC (Martins et al. 2015) with the presence of boquique, which would indicate entry by different routes, north and south (Bernabeu Aubán et al. 2018)(García-Puchol et al. 2018). According to the dual model, all this would be accompanied by an immediate substitution of subsistence practices in certain sites, given the existence of mesolithic remnants in the northern peninsular zones up to 5300 cal BC (Martínez-de-Lagrán, 2012). This would not imply a massive arrival of population, but rather an acculturation that a posteriori would culminate in a symbiosis with the populations of Mesolithic tradition (Guilaine, 2018).

1.2.2. New productive processes: domestication of plants and animals

The transition from hunter-gatherer societies to agricultural-livestock societies is considered to be one of the most far-reaching socio-economic changes in history. The Neolithic period was marked by the domestication of plants and animals, as well as various technological advances, such as the production of ceramic containers. In order to understand the origins of the Neolithic period in the Iberian Peninsula, it is especially important to know how and when these new production processes were carried out.

In the Iberian Peninsula and the central and eastern Mediterranean (Colledge and Conolly 2007), unlike in other European areas, agriculture is characterised by a great diversity of crops. Among the cereals documented are two species of dressed wheat, the spelt (*Triticum monococcum*) and the spelt (*Triticum dioccocum*); two species of bare wheat, durum wheat (*Triticum durum*) and flour wheat (*Triticum aestivum*); two varieties of barley, dressed barley (*Hordeum vulgare*) and naked barley (*Hordeum vulgare* *nudum*), and five legumes, pea (*Pisum sativum*), lentil (*Lens culinaris*), broad bean (*Vicia faba*), (*Vicia ervilia*) and titarro (*Lathyrus sativus*). The presence of linen (*Linum usitatissimum*) and opium poppy (*Papaver somniferum*) is also documented. This evidence contrasts sharply with other areas, such as the LBK fields in Central Europe, where agriculture is limited to the cultivation of two species of dressed wheat (*Triticum monococcum and T. diococcum*), two leguminous plants (*Pisum sativum* - pea - and *Lens culinaris* - lentil -), and flax (*Lunim usitatissimin*) (Kreuz 2007).

The diffusion of agriculture on a Mediterranean scale was a rapid process documented in the Iberian Peninsula from c. 5600-5500 cal BC, even in inland peninsular areas such as the Pyrenees or the Meseta. The remains of seeds cultivated with older radiocarbon dating are located in the eastern peninsular area around 5600 cal BC, in Mas d'Is (5617-5485 cal BC) (Bernabeu et al. 2014), Cova de l'Or and Cova de les Cendres (Bernabeu and Molina 2011). These identified remains include cereals, including dressed wheat, legumes and some wild fruits (Buxó 1993). In Can Sadurní (Buxó 2007) and La Draga (Antolín and Buxó 2010) the presence of bare wheat also stands out in the oldest phases of the Neolithic. Legumes are documented in these early Neolithic phases in the sites of the eastern peninsular (Pérez Jordà 2005) and also in sites in the northeast, such as La Draga, Plansallosa or Cova 120 (Buxó et al. 2000), as well as in the Pyrenean context in Balma Margineda (Marinval 1995).

In Andalusia the data on the beginnings of agriculture are scarce. The presence of bare wheat, bare barley and legumes is documented in Cueva de El Toro, Cueva de Nerja, Cueva de los Murciélagos de Zuheros, Cueva de los Mármoles and Los Castillejos (Rovira Llorens et al. 2008)(Buxó 1997, Peña Chocarro 1999).

The information from the interior of the peninsula is very limited, and the predominance of domestic species varies according to the region. Bare wheat is found in the Cueva de La Vaquera site (Estremera 2003), consistent with the results obtained in the Mediterranean strip, while in La Lámpara, La Revilla and Cascajos bare wheat and barley dominate (Zapata et al. 2004)(Peña Chocarro et al. 2005).

For the northern peninsular sites, barley and spelt are documented in the Pico Ramos, Kobaederra and Lumentxa sites (Zapata 2007), while in El Mirón bare wheat predominates over clothing (Peña Chocarro et al. 2005).

The collection of wild plants is well documented during the Neolithic period of the Iberian Peninsula (Buxó 1997, Zapata 2000), although the relative proportions of these resources in the human diet are very difficult to estimate. The appearance of remains of wild fruits and plants in excavations shows that after the adoption of agriculture, they continue to be an important resource, possibly as a complement to cultivated products and as a solution in the event of bad harvests or periods of famine (Buxó, 2007). With these data, it seems that at the beginning of the Ancient Neolithic, cereal cultivation was fully developed in some sites of the Iberian Peninsula (Buxó 1997).

With regard to livestock, archaeozoological studies face the challenge of recognising domestic forms. Archaeological work carried out in recent years and the increase in radiocarbon dating of domestic fauna remains at sites in the Iberian Peninsula have documented the presence of the four main domestic animal species at the beginning of the Neolithic period (5600 cal BC): *Ovis aries, Capra hircus, Bos taurus* and *Sus domesticus* (Saña 2013). The practice of hunting activities is reduced with the introduction of domestic species, representing a relative frequency of 30% at peninsular level (Saña 2013). However, the sites dated in this period have exceptions in the north of the peninsula. There are numerous sites located on the northern coast of the Iberian Peninsula that have a low percentage of domestic species among the remains of fauna, such as Los Husos (37.8%), Arenaza (21%) (Altuna 1980), Marizulo (1%) (Altuna 1972) and Herriko Barra (1%) (Mariezkurrena and Altuna 1995). The wild species represented in the faunal groups of these sites are mainly deer, roe deer and wild boar.

When we move to the Ebro valley, a significant variability is documented. While in Chaves and La Puyascada ovicaprinos predominate (62%) (Sierra et al. 2019)(Castaños 2004), in La Renke bovids are the predominant species of a group practically represented by domestic species (99%) (Altuna 2001).

In the northeast of the Iberian Peninsula, the percentage of recovered domestic fauna does not fall in the majority of sites of 90%. In the La Draga site, the group is characterised by a certain balance between ovicaprine, suidae and bovine animals (Navarrete and Saña 2014), while in Cova del Frare ovicaprine animals predominate (Saña et al. 2015). Among the wild species, deer and wild boar stand out. In the plain area of Barcelona, ovicaprinos predominate in the Caserna de Sant Pau faunal complexes (Colominas et al. 2008), while bovids are predominant in the Reina Amàlia site (Saña and Navarrete 2016).

In eastern Valencia, sheep are the predominant domestic species above goats, as in Cova de l'Or and Cova de la Sarsa (Pérez-Ripoll 1977). On the other hand, sites are still documented with a fairly high percentage of wild animals represented, such as in Cova de les Cendres (55%) (Iborra and Martínez 2009) and Cova de les Bruixes (70%) (Mesado 2005).

For the south of the Iberian Peninsula the available data are relatively scarce, we find one of the oldest dates for this region in domestic animal bone from the Cave of Nerja (Martins et al. 2015), or the large number of ovicaprine remains recorded in the Cueva de El Toro faunal complex (Martin-Socas et al. 2004). In general, the data obtained for this region show an oscillation between 60% and 90% of the wild fauna over the domestic fauna (Saña 2013).

The representative variability of the different domestic species and the remnants of hunting activities in the study region are the result of different management strategies for domestic animals in the Iberian Peninsula. Although there are differences between the different territories, there is generally a rapid and not always gradual adoption of domestic species (Saña 2013).

Animal domestication also led to the exploitation of live animals, such as labour force, dairy farming, excrement or animal fibres. Milk production has been extensively documented indirectly from evidence of mortality profiles of domestic ruminants (Halstead 1998), representing about 80% of the sites studied, such as in Ftelia (Greece), Araguina-Sennola (Corsica), Grotte Lombard or Baume de Fontbrégoua (France) (Spiteri et al.

2016). In the Iberian Peninsula this exploitation is documented during the Ancient Neolithic in sites such as La Draga (Saña 2011, Gillis et al. 2014), Chaves (Sierra et al. 2019), La Puyascada (Sierra et al. 2019), Cueva de El Trocs (Rojo Guerra et al. 2013), Coro Trasito (Viñerta 2015, Antolín et al. 2017), Caserna de Sant Pau (Saña and Navarrete 2016), Reina Amàlia (Saña and Navarrete 2016), Can Sadurní (Saña et al. 2015) and Cueva de El Toro (Martin-Socas et al. 2004). These data highlight the importance of this exploitation in the communities of the Ancient Neolithic throughout the region of the Iberian Peninsula.

1.2.3. New technology changes: pottery

In the Mediterranean, the diffusion of ceramics in the initial Neolithic period is markedly coastal. In the discussion on the provenance of Neolithic influences in the Iberian Peninsula, studies on ceramics found in the peninsular sites have enriched the debate in relation to the two routes of provenance, that of the Gulf of Lion and that of North Africa (Bernabeu Aubán et al. 2018)(Carvalho 2010, Cortés et al. 2012).

In a chronological sense, groups ascribed to ceramics from the beginnings of neolithization up to 4900 cal BC can be grouped in the Ligurian printed horizon, the cardial horizon, the bouquique horizon and the almagra horizon.

The group of ceramics ascribed to the Ligurian printed horizon develops between 5800 and 5500 cal BC (Bernabeu et al. 2009, Binder and Sénépart 2010) and presents very heterogeneous characteristics. Its presence is distributed from the Italic Peninsula to the coast of the western Llenguadoc, passing through the Gulf of Lyon (Guilaine & Manen, 2012). Occasionally, they have also been identified in the Iberian Peninsula. Their heterogeneity is marked by the clear divergences that the decorations present and that would imply a varied origin. Therefore, some authors do not give it a fully unitary entity (Manen 2007). Ligurian printed ceramics may present xamota as an added degreaser, as in Peiro Signado (Binder and Maggi 2001), and printed decorations that may be presented linear or digital. In the Iberian Peninsula these ceramics are found in the Barranquet site, dated at 5367 cal BC. Within the ceramic set of this stratigraphic level of Barranquet, printed ceramics, incised ceramics and cardial impressions are found (Bernabeu et al. 2009), as well as the presence of xamota as an added degreaser (García-Atiénzar 2010). Another site with materials from the Ligurian printed group is Mas d'Is at a stratigraphic level dated 5481 cal BC together with traditional cardiac materials (Bernabeu et al. 2011).

The cardial horizon was situated as the first Neolithic phase to the west of the Mediterranean from the stratigraphy defined by Bernabó in 1949 by Arene Candide (Guilaine 2007). This group would develop between 5700 and 4900 cal BC (Oms, 2014)(Clop et al. 1992: 64). In the Iberian Peninsula, apart from Siret, the study of Neolithic printed ceramics began with Bosch Gimpera when in 1920 he defined the "culture of caves with decorated ceramics" (Bosch 1952), although archaeological data from recent years denies this exclusive housing, as in Les Guixeres de Vilobí (Vilobí del Penedès, Barcelona) (Ferrer 1954, Mestres 1981). Printed ceramics present a fundamental typological unit in spite of the local specialisations of the Mediterranean. It is characterised by a good quality paste, well cooked over an irregular fire, resulting in different shades. The ceramics are profusely decorated. This decoration is carried out before firing with a spatula, punch, bone, shells such as the "cardium edule L", animal teeth, etc. The first findings of this type of ceramics are documented under the name of "montserratina ceramics", after the discovery of this type of impressions in Cova Freda and Cova Gran (Colominas 1925). But given that its presence turned out to be more extensive, it became known as "cardial ceramics". In the northeast of the peninsula, cardial ceramics are documented in sites such as Balma Margineda (Oms et al. 2016)(Guilaine and Martzluff 1995), Draga (Tarrús et al. 1994), Can Sadurní cave, cova Bonica (Oms, 2014), the Cova del Frare (Martin et al. 1985), in the open-air settlements of Caserna de Sant Pau del Camp (Molist et al. 2008) and Guixeres de Vilobí (Oms, 2014). In the eastern part of Valencia, cardial materials are documented in sites such as Cova de les Cendres, Cova Ampla, Cova de l'Or, Cova de la Sarsa, Cova d'en Pardo and in open-air sites such as Mas d'Is, Barranquet and Benàmer (García-Atiénzar 2011, García Borja et al. 2012, García Borja 2017). The oldest radiocarbon dating for cardial in the Iberian Peninsula correspond to those of Cova de l'Or (5376 cal BC), Cova de les Cendres (5374 cal BC) and Abric de la Falguera (5324 cal BC) (Bernabeu Aubán et al. 2018)(Bernabeu et al. 2009). For the south peninsula there is little published information, some of the sites where cardial ceramics were documented are Retamar (Ramos and Lazarich 2002), Carigüela (Pellicer 1964), Esperilla, Lebrija, los Pozos o Bustos (Gavilán Ceballos & Escacena Carrasco, 2009)(Garcia Borja et al. 2010). The predominant style, called "cardialoide", is reminiscent of cardial decoration but is made with tools and not with shells. In the lower section of the Tagus River, in Portugal, cardial ceramics were found in sites such as Gruta de Caldeirao and Cisterna between 5300 and 5200 cal BC (Carvalho 2011).

The bouquique-inciso-printed horizon does not have a clear geographical and chronological delimitation (Rojo Guerra et al. 2008). The available radiocarbon dating situates the beginning of this group towards 5400 cal BC. The sites with the presence of material ascribed to this group are distributed throughout the Iberian Peninsula, in the northern sub-plateau area the Cueva del Mirador (Vergès et al. 2008), in the Ebro valley La Lámpara and Revilla del Campo (Rojo Guerra et al. 2008), in the north Los Cascajos (Gacía Gazólaz and García Sesma 2007), in the central system Cueva de la Vaquera (Estremera 2003) and in the southwest peninsular Los Barruecos (Cerrillo et al. 2002). At the level of cultural characterization, the ceramic productions present impressions of diverse matrices, incisions, grooves and plastic motifs, as well as the combination of incised and grooved motifs in the same vessel.

Pellicer defined the almagra-inciso-printed horizon in the lower third of the Iberian Peninsula (Pellicer and Acosta 1997). The almagra technique consists of bathing or small pinzeladas of the reddish ceramic surface, and is combined with other decorative styles, such as cardial, incisions and prints (Rojo Guerra et al. 2008)(García Borja et al. 2010). Among the most outstanding sites are the Cueva de Nerja (Garcia Borja et al. 2010), Cueva de El Toro (Martin-Socas et al. 2004), Castillejos de Montefrío and Cueva de los Murciélagos de Zuheros (Peña Chocarro y Zapata 2010).

Towards 4500 cal BC, there is an overlap of the presence of these cardial decorations together with some decorations called "epicardials" in favour of the printed, incised and grooved decorations in which the use of shells is no longer present, such as the ceramics found in the site of the Cova de l'Avellaner (Les Planes d'Hostoles, Girona). Although other decorative styles are also developed in parallel in some specific geographical areas, such as the Montboló style (Clop et al. 1992, Guilaine 1974) and the Molinot. The Montboló style is characterised by smooth ceramics, very refined and with a reducing firing (Martín 1992). On the other hand, the Molinot style is characterised by the combination of amporoid, globular and subspherical containers with a more oxidising firing, as well as brushed surfaces with the presence of ridges applied in a triangular section (Martín 1992).

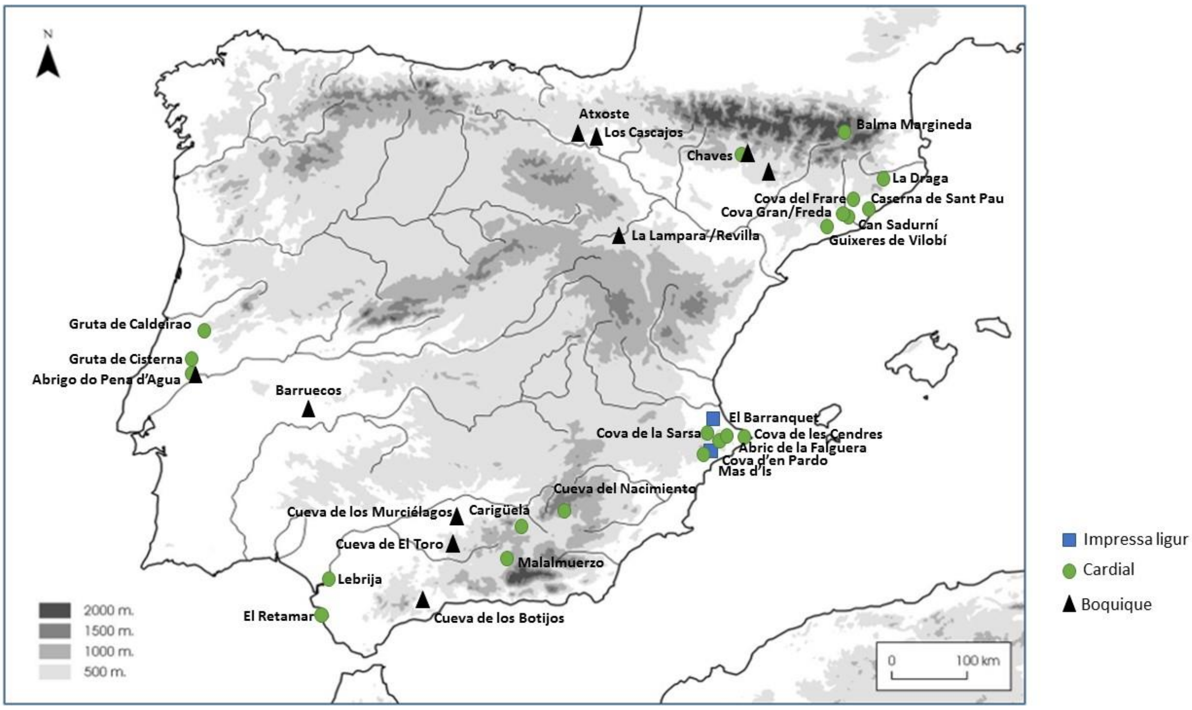


Figure 1.1. Map of the Iberian Peninsula with the sites cited in the text on ceramic horizons.

1.3. New food resources. Food production in the Neolithic period

Food, as a biological necessity, has long been linked to the structuring of social and cultural relations. The Neolithic develops as a decisive period in terms of the transformation of culinary culture. While Childe defined the Neolithic as a period where new food supplies and new substances not found in nature were produced (Childe 1958: 49), Renfrew posed it from a point of view in terms of production and increase of material culture (Renfrew 1998).

The history of food acquires the condition of eclectic and integrating discipline, incorporating conceptual and methodological presuppositions of a wide set of disciplines related to the variety of factors that influence the food/nutrition binomial.

New food products appear as a result of the domestication of plants and animals, such as milk or bread (Fuller & Gonzalez 2018). In fact, it has been suggested that certain cereal species, such as wheat or barley, contain a high concentration of gluten to make bread. This could be one reason why these taxa have become important and cultivated (Fuller and Rowlands 2011).

1.3.1. Isotopic evidence of diet

The issue of livelihoods and diet in the Neolithic has also been addressed more directly from the perspective of biological anthropology. In the past, biological anthropology focused on ancient populations and how to describe and place them in context. On the basis of this description, however, there was no room for anything other than the morphology and typology of human remains. The development of these studies allowed us to go further in the interpretation of human remains, from determining the place of origin of the individual to the reconstruction of his diet and that of the rest of the group. In this way, the application of stable isotope studies in human bones and the analysis of dental calculi, together with osseous pathologies and dental wear.

Recent studies of stable paleodiet isotopes in Iberian Peninsula sites dating from the Ancient Neolithic, such as the Cave of Bats (Valdiosera et al. 2018), the Middle Neolithic (Fontanals Coll, 2015)(Fontanals et al. 2016), until the Final Neolithic, as in the Cova de la Guineu (López-Costas and Alexander 2019), agree that, despite economic development, dietary preferences remain constant from the Early Neolithic to the Late Neolithic in the Iberian Peninsula. It has been indicated that aquatic resources are not abundant in the diet, despite their probable availability in many sites and paleodietary results in mesolithic sites (García-Guixé et al. 2006; Fernández-López de Pablo et al. 2013; Salazar-García et al. 2014), and had preferences for terrestrial foods, with the exception of an individual in Bòbila Madurell who presents evidence of regular marine consumption

(Fontanals Coll, 2015). Fontanals defines the Neolithic diet in a diet based on cultivated plants and the protein contribution of meat and dairy products, although it highlights the predominance of plant consumption over meat, especially in Middle Neolithic sites such as Can Roqueta (Fontanals Coll, 2015).

A diet related to a high protein intake was described based on the analysis of dental calculi in the Final Neolithic site of the Cova del Pantà de Foix. Nevertheless, the high prevalence of carious lesions suggests that the main dietary contribution comes from carbohydrates (Subirà et al. 2014). On the other hand, wild plants consumption, particularly opium compounds, have been identified in human bones and dental calculus among late Neolithic miners in Mines de Gavà (Juan-Tresserras and Villalba 1999).

These studies allow us to approach, through direct and indirect evidence, both the economic aspects of Neolithic populations, such as subsistence practices and the reconstruction of the diet, as well as more social aspects, such as culinary guidelines and resource management.

1.3.2. Food evidence from biomolecular analysis of pottery remains

As we have been commenting, there are different approaches available to study food in the Neolithic. The transition from fishing, hunting and gathering to agriculture and livestock was one of the deepest transitions in human history, with far-reaching consequences for biodiversity, human health and cultural development. One of the most common material evidences of the communities that carried out all these changes are ceramic products. Research into these artefacts now allows us to have a great deal of information, from the formal characteristics of the containers to the specific use of pottery. This is where, based on the determination of the organic residue that may eventually have been preserved and which is the testimony of the different activities (culinary, storage, production, etc.) in which they would have been used (Fanti et al. 2018; Vieugué et al. 2016), we intend to broaden our knowledge of culinary patterns and the consumption of food products during the Neolithic.

The identification of subcutaneous animal fats has been widely documented in ceramics since the beginning of Neolithic in the Near East (Richard P. Evershed et al. 2008a; Gregg & Slater, 2010a; Nieuwenhuyse et al. 2015; Spiteri et al. 2016). From the 7th millennium BC this pattern extends across Europe. Processed animal fats are identified in ceramic containers in the southeast, in Greece (Richard P. Evershed et al. 2008a; Spiteri et al. 2016)(Urem-Kotsou et al. 2014), Bulgaria (Vieugué et al. 2008), Hungary (Richard P. Evershed et al. 2008b) or Romania (Oliver E. Craig et al. 2005)(Craig et al. 2008); in the central Mediterranean they are documented in Italy (Salque et al. 2012a; Spiteri et al. 2016), Croatia (Spiteri et al. 2016), Slovenia (Soberl et al. 2014; Šoberl et al. 2008); and in central Europe, in Germany (Salque et al. 2012a), Czech Republic (Matlova et al. 2017) and Poland (Salque et al. 2013).

The ubiquity of these substances reflects an intense consumption of materials of animal origin in the Neolithic (Regert et al. 1999), particularly in ceramics, either because they have been used to cook meat or to extract bone marrow (Gregg, 2009).

Ruminants (cows, sheep, goats) and non-ruminants (pigs) seem to have been equally exploited in ceramics at the beginning of the Neolithic, in the different regions of the eastern Mediterranean (Oliver E. Craig et al. 2005). From the published results, there appears to be a transformation in processing preference towards ruminant fats, consistent with the predominant ovicaprine remains of faunistic sets (Manning et al. 2013)(Rowlew-Conwy et al. 2013), with some exceptions in Italy and Germany (Salque et al. 2012a). This restricted register of non-ruminant fats is repeated in northern Europe, despite the good documentation available on pig farming from faunal remains (Manning et al. 2013). It is not until the British Neolithic that the presence of pig fat documented in ceramic containers becomes evident (Craig et al. 2015; Mukherjee et al. 2008)(Coopley et al. 2005). This bias in pig representation may be due to the underestimation of this type of fat when mixed (Regert, 2011), which would lead to over-quantification of other species to the detriment of the pig. In this respect, solid reference models are needed to help better understand the interpretation of the fats detected and the possible mixing of these fats.

In the Iberian Peninsula the record is smaller, but the few published papers indicate a predominant processing of ruminant fats (Debono Spiteri, 2012; Tarifa, 2015)(Breu 2019), compared to a consumption of non-ruminants around 35% of identified fats, as in Caserna de Sant Pau (Breu 2019), Guixeres de Vilobí (Breu 2019), La Serreta (Breu 2019), Cova de la Font Major (Breu 2019), Cova de la Guineu (Breu 2019) and Cueva de El Toro (Tarifa-Mateo et al. 2019).

The crossing of biomolecular results with the shape of the container can not only provide information about the use of the pots but can also inform us about how the identified product was processed. Among the few studies that have been able to establish a link between content and function of pottery (Salque et al. 2013; Soberl et al. 2014), ruminant fat was interpreted in a container for storage or transport of liquids as a waterproofing agent (Salque et al. 2013).

Also significant is the percentage of fat from dairy products that is identified throughout Europe in the containers found from the oldest Neolithic strata. Detection of dairy products by biomolecular analysis in ceramics has shed light on our understanding of animal domestication (Oliver E. Craig et al. 2005; Spiteri et al. 2016; Vigne & Helmer, 2007). As mentioned above, this process of neolithization, which accompanied the domestication and exploitation of resources such as dairy products, developed with different intensities in the territory. This is reflected in the presence of milk in the ceramics of the different archaeological sets studied (Oliver E. Craig et al. 2005; Drieu, 2017; Salque et al. 2013, 2012a; Soberl et al. 2014; Spiteri et al. 2016). In the Iberian Peninsula, the presence of dairy products has been verified from the detection of lipids by mass spectrometry (Yañez et al. 2008) and recent studies applying isotopic lipid analysis have confirmed milk production in Can Sadurní (Spiteri et al. 2016), Reina Amàlia (Breu 2019), Sant Pau del Camp (Breu 2019), Guixeres de Vilobí (Breu 2019), Cova del Vidre (Breu 2019) and El Cavet (Breu 2019) and Cueva de El Toro (Tarifa-Mateo et al. 2019).

Ceramic containers would have participated from the acquisition and processing of dairy products to their consumption in the different stages of production. Thus, milk products have been identified in vessels with characteristic elements for the transport of liquids, such as the spout pot of Cueva de El Toro (Tarifa-Mateo et al. 2019), or the cheesemakers of the LBK culture (Salque et al. 2013). These products have also been identified with evidence of processing with fire (Carrer et al. 2016a; Oliver E. Craig et al. 2005; Regert, 2011), related to the production of dairy products such as yoghurt or cheese (Richard P. Evershed et al. 2008a).

Remarkable is the evidence of the persistence of mesolithic fishing practices along with evidence of processing of domestic animal fats and ceramic dairy products from Japan (Craig et al. 2013; Lucquin, Gibbs, et al. 2016b) to northern Europe (Craig, 2004; Craig et al. 2007, 2011; Cramp, Evershed, et al. 2014; Isaksson & Hallgren, 2012; Oras et al. 2017). This biomarker of marine resources at Mesolithic/Neolithic sites led the Craig team to use them as markers to discuss the neolithic transition in northern Europe (Craig et al. 2011). On the other hand, aquatic resources are little processed in Neolithic site from the Mediterranean area (Debono Spiteri, 2012; Drieu, 2017). These results are well correlated with isotopic analyses on human bones showing an absence of these products in diets during the Neolithic (Le Bras-Goude et al. 2006; Lelli et al. 2012; Lightfoot et al. 2011; Richards & Hedges, 1999). In post-Early Neolithic chronologies, there are no significant changes in the fats processed in ceramic containers, but the percentage of fats in domestic animals and the processing of dairy products are consolidated in all regions of the continent.

With respect to vegetable resources, the analysis of organic residues in ceramics presents interpretative and preservation limitations. Even so, vegetable oils and epicuticular waxes have been reported in a large number of European neolithic sites (Craig, 2004; Debono Spiteri, 2012; Ogrinc et al. 2012; Regert et al. 2003; Soberl et al. 2014)(Decavallas 2011, Raemaekers et al. 2013). These products are often found mixed with animal fats and are interpreted as culinary mixtures, such as those interpreted in the Iberian Peninsula (Juan-Tresserras 2004).

Chapter II. Materials

2.1. Temporal and spatial framework

As we commented in the previous chapter, there is a regional variability of neolitization dynamics in the Iberian Peninsula. In the east and few documented cases in the south of the Iberian Peninsula, there are records of mesolithic continuity, unlike the occupational vacuum recorded in the northeast of the peninsula during the time interval between 8500 and 7500 cal BC. The expansion of new farming practices is documented at around 5600 cal BC in the east and consolidated in the northeast 100 years later and in the north from 5400 cal BC. These regional variabilities can respond to the diverse social and economic dynamics presented by the different regions and environments that exist in the Iberian Peninsula.

A description is presented below, focusing on the main aspects and dynamics that characterize each of the areas worked on in this thesis.

2.1.1. Variability registered in the areas under study

2.1.1.1. Dynamics of neolithization in the Pyrenean areas

The Pyrenees constitute a mountain range of alpine orogeny about 450km long. Its orientation is west/north-west-east/southeast, which goes from the Cantabrian Sea to the Mediterranean, which implies the existence of an important climatic gradient between the most Atlantic and the easternmost areas.

The western Pyrenees are characterised by an alpine and subalpine climate depending on the altitude. Rainfall is high due to cooling and condensation. This leads to the existence of hardwoods, conifers and/or sclerophylls. Pyrenean plant landscapes have undergone climatic changes and since the end of the last glaciation, anthropic activity (Gassiot et al. 2012). However, for the present period (5600-3800 cal BC) there is no intense anthropization of the landscape on a regional scale (Rojo Guerra et al. 2013). Pollen analyses show that the forests of the eastern Pyrenees consisted of conifers and deciduous trees, such as the Quercus. Afert the 5600 cal BC, fire peaks have been also detected in the Axial Pyrenees along with an increase in Poaceae. However, in the mountain the arboreal biomass remained at relatively high levels, suggesting that such fires consisted mainly in small opening of the forest (Cunill et al. 2013).

2.1.1.1.1 Early Neolithic (5500-4900 cal BC)

In the Pyrenean contexts between 5700 and 5600 BC, deposits with mesolithic occupations are located, such as in Aizpea and Zatoya, Atxoste (Rojo-Guerra et al. 2010)(García 2015). It will not be between 5400-5300 cal BC that the beginning of the ancient Neolithic is determined in this area. As it happens in the rest of the peninsular territory, the agricultural practices are evident in the majority of ex novo sites (Gassiot et al. 2017). An exceptional example is Atxoste, which starts in the upper Palaeolithic and presents all the phases of the mesolithic until this chronology, where remains of ceramic fragments and remains of domestic fauna from 5300 BC (Ruiz et al. 2012) have been evidenced.

The most explanatory evidences for the Ancient Neolithic in this region are the sites of La Espluga de la Puyascada, El Forcón (Baldellou 1987), Els Trocs (Rojo et al. 2016) and Coro Trasito (Clemente-Conte et al. 2016). These sites present an ex novo settlement from 5400 BC and characterized by a livestock-oriented welfare practice. In them, the percentage of ovicaprines on the total of the remains of fauna documented is of 80% (Rojo et al. 2018). However, caves would not be the only type of settlement during the Ancient Neolithic. Although scarce, in the Madriu-Perafita valley (Andorra) large huts were documented, bordered by a stone fence and dating from 5614 to 5475 BC (Orengo et al. 2014).

Until a few years ago there was a non-explicit consensus that the mountain range, like other mountainous areas of the Iberian Peninsula, remained largely on the fringes of the first expansion of agricultural activities (Bahn 1983, Bertan Petit and Vives 1995, Jiménez 2006, Llovera 1986, Yáñez et al. 2002, Yáñez 2005, Orengo et al. 2014, Walsh et al. 2005). According to this concept, mountain environments are considered to present adversities for the establishment of agriculture and would be at the margin of the first agricultural experiences, being isolated during the pioneering process of neolitization. However, thanks to recent archaeological research, it has been demonstrated that the Pyrenees were not marginal zones and that their neolithic process was faster and more extensive than previously thought (Gassiot et al. 2017). The domain of agriculture is present in the carpological remains of all deposits from ca. 5500 cal BC, with the presence of wheat and barley in Balma Margineda (Guilaine et al. 1995), Coro Trasito or Els Trocs (Antolín 2016). In contrast to Coro Trasito, in Els Trocs no potentially arable area around the site has been identified (Rojo et al. 2018).

Regular archaeofaunistic data has found in the different sites, although with some exceptions. Faunistic groups are dominated by a great representation of ovicaprines, as in Balma Margineda, although it has not been possible to quantify (Geddes 1980), around 85% in Els Trocs (Rojo et al. 2018) and 65% in Coro Trasito (Clemente-Conte et al. 2016). On the other hand, in Aizpea the presence of remains of domestic fauna was not documented, and in Zatoya only dog remains were found (Barandiarán and Cava 1989, 2001). Among the wild species, although not very representative, the deer and wild boar predominate, as well as the rabbit in Coro Trasito (Viñerta 2015) and the Pyrenean goat in Balma Margineda (Martín and Mozota 2018). There is also trout fishing (Salmo trutta) in Balma Margineda, which places the occupation in the autumn period, and birds found in Els Trocs, such as the swallow (Hirundo rustica) or the quail (Coturnix coturnix).

The ceramics recovered from the different sites in the central and eastern Pyrenees are very similar, both decoratively and morphologically. The presence of ceramics in these sites is variable. The ceramic complexes of Aizpea, Zatoya and Cova del Sardo are relatively small in comparison to other sites such as Els Trocs (Rojo et al. 2018). In these sites in the Pyrenees between 7 and 53 ceramic fragments are recorded, respectively, without decoration and reducing firing. This firing offers a blackish tonality that stands out with the engobe or plaster that some external surfaces of vessels found in Zatoya (Barandiarán and Cava 1989, 2001) present. In the cave of El Trocs, on the other hand, ceramics is the most represented and abundant archaeological material, with 30,000 records unevenly located in the space. 60% of the complex belongs to the oldest phases of occupation of the cave and this percentage decreases in the most recent horizons. The decorations are characterized by the presence of ungulations and cardial in the oldest phases of occupation, and impressions with instrument and combination of incision and groove in the most recent phases. Other techniques in a smaller percentage are boquique, comb printing and painting. These decorations have parallels with the ceramics found in Coro Trasito (Clemente et al. 2014) and on the other side of the Pyrenees (Manen et al. 2010), as in the Pre-Pyrenees, such as in Chaves or the Spluga de la Puyascada (Rojo et al. 2018).

The lithic industry is the second most important material evidence in the archaeological record of the high Pyrenean mountain sites. Microliths have been documented in double bevels, such as in Aizpea (Barandiarán and Cava 2001) or trapeziums, triangles and backs, such as the 111 documented in Zatoya (Barandiarán and Cava 1989). In some sites such as Els Trocs, Coro Trasito or La Cova del Sardo, the provenance of the flint has been studied and it was concluded that the flint used came from the plain or piedmont areas (Mazzucco et al. 2014, Rojo et al. 2014, 2018, Clemente et al. 2014, Gassiot et al. 2014). In Els Trocs, 41.1% of the flint documented as allochthonous corresponded to already elaborated supports and already configured cores (Rojo et al. 2018). These data indicate a supply of lithic materials organized on a south-north axis. On the other hand, marine shells (Glycimeris sp.) have also been documented in deposits such as Coro Trasito, used as adornment or for the treatment of the surface of ceramics (Clemente et al. 2014). Finally, the presence of bone tools is also documented with deer punches (Clemente et al. 2014).

2.1.1.1.2. Middle Neolithic (4500-3900 cal BC)

From 4500 cal BC onwards, an evident change in the population and exploitation of the high Pyrenean mountain areas is documented, which can be seen in various spheres. Firstly, there is a clear increase in the number of sites, as well as the continued occupation of Cova del Sardo (Gassiot et al. 2014), Els Trocs (Lancelotti et al. 2014) or Coro Trasito (Clemente et al. 2014). This intensification in population extends to areas where until now no archaeological vestiges had been identified, such as in the valley bottoms at the open-air sites of Juberri (Fortó and Vidal 2016). In this respect, the site with the highest number of silos and post holes is Feixa del Moro (Juberri), which would function as a warehouse and/or landfill (Prats 2018). It is interesting to note the impact on agricultural practices at this site that some authors defend (Fortó et al. 2013). Based on the evidence related to large storage containers with evidence of cultivated seeds, silo type structures, pollen analyses and the scarce faunal records conserved in the sites, a stable economy based on the cultivation of plants, wheat and barley is defended (Antolín, 2013). On the other hand, the occupation of Feixa del Moro was divided into a first phase of occupation of habitat types, contemporaneous with the Carrer Llinàs 28 and Feixa del Moro sites, and a second moment in which funerary structures are documented (Fortó et al. 2013). In the axial Pyrenees, the occupations at the bottom of the valley as in the mines of Sanavastre, located at 1080m a.s.l., stand out. (Mercadal et al. 2009), and the possible cabin of the site of Pleta de Bacives I, at 2500m a.s.l. (Orengo et al. 2014). At a similar altitude (2300m a.s.l.), the ceramic occupations of the Obagues de Ratera were documented in the Parc Nacional d'Aigüestortes-Estany de Sant Maurici, although there is still no radiocarbon dating to confirm their development in these chronologies (Gassiot et al. 2018). All these occupations are related to the practice of intensive stock-breeding, such as the hut funds or the occupation of the Cova del Sardo.

Pottery from this period onwards is little documented in sites with an Ancient Neolithic tradition, such as the Sardinian Cave (Tarifa, 2015). On the other hand, in the three sites of Juberri, Feixa del Moro, Carrer Llinàs 28 and Camp del Colomer, the containers have a great volumetry, between 2 and 8 litres of capacity. The characteristics of these ceramics are located in a horizon of the Middle Neolithic, although elements of the epicardial appear in Carrer Llinàs 28 (Fortó et al. 2018).

Macrolytic materials such as mills and mill hands appear in Carrer Llinàs 28 and Camp del Colomer (Fortó et al. 2018), related to the practice of processing agricultural products (Augé et al. 2016). They also document axes, which explain the specific deforestation practices documented in the pollen records for agricultural and livestock exploitation in the vicinity of some sites, such as Camp del Colomer (Fortó et al. 2018). The lithic industry documented in Sardinian contains 35% allochthonous materials from the Ebro valley (Mazzucco 2014, Gassiot et al. 2018).

2.1.1.2. Neolithic in the northeast of the Iberian Peninsula

The northeast of the Iberian Peninsula has a long coastline to the east and plains inland. It is located in the temperate zone of the northern hemisphere, characterized by the diverse climatic influences of the Mediterranean Sea. The coastal and pre-coastal zone is defined by a Mediterranean climate, while the central depression has a continental climate. Rainfall is higher in the coastal area and lower in the plain area. At the hydrographic level, the most important river is the Ebro, which flows into the Mediterranean Sea. Both the Ebro and the secondary rivers are born in the Pyrenees.

The reconstruction of vegetation from pollen and anthropological analyses between 5700 and 2400 shows a predominance of deciduous forests, characterized by oak groves (Burjachs 2000, Péachs et al. 2007).

2.1.1.2.1. Early Neolithic (5500-4450 cal BC)

In the northeast of the peninsula, a discontinuity in the occupation of this area between 6500-5500 cal BC (Barceló 2008) is documented, accompanied by the scarce presence of settlements with previous chronologies, such as Cova del Vidre (Bosch 2015), Can Sadurní (Edo et al. 2011), Cova del Parco (Moreno-Martínez et al. 2007) and Bauma del Serrat del Pont (Alcalde et al. 2008). This interval of decrease of archaeological sites is located between ca. 6500 cal BC to 5500 cal BC, coinciding with an abrupt climatic change called event 8200 cal BP (López Sáez et al. 2008), a cold stage caused by the increase of the flow of fresh and cold water preceding the melting of the polar ice caps (Wiersma and Renssen 2006). This change is reflected in the vegetation: palynological studies at the Drassanes site, for example, show a greater preponderance of pine to the detriment of oak, and alder and ash disappear as the most significant elements of the riparian forest. These data would support a decrease in humidity, both environmental and edaphic, with greater relevance to vegetation adapted to xeric conditions (Riera

1993, Revelles 2015). It is from this moment on that the first dates are documented for neolithic sites ex novo, such as La Draga (Terrades et al. 2015), Guixeres de Vilobí (Oms et al. 2016) and Sant Pau del Camp (Molist et al. 2008), and sites that presented mesolithic strata are reoccupied (Edo et al. 2011, Alcalde et al. 2002).

The archaeological contexts of the Early Neolithic in the northeast of the peninsula are characterised by the presence of cardial ceramics. From the design of a compilation of radiocarbon dating for the Neolithic levels with the presence of different decorative styles (cardiac, impressions, incisions) in ceramics, a Early Neolithic Cardial chronology was established (5500-4900 cal BC), Early Neolithic Epicardial (4950-4450 cal BC) and a Post-Cardial Neolithic (4650-3900 cal BC), which would give way to the Middle Neolithic (Oms et al. 2016).

Open-air settlements are scarce in the archaeological record during the early stages of the Neolithic and do not allow reliable models of settlement to be proposed. We have data from two huts with homes in Guixeres de Vilobí (Oms et al. 2018) and twelve documented huts in the La Draga lake deposit, as well as in Sant Pau del Camp (Gómez and Molist 2017, Molist et al. 2008). In epicardial chronologies, we could mention the large ellipsoidal structure in Reina Amàlia associated with combustion fires (González et al. 2011).

The first evidence of domestic resources dates back to 5390 cal BC in the Cova de Can Sadurní (Begues, Barcelona) (Edo et al. 2011, Antolín 2016). Simultaneously, in the eastern Prepyrenees with the Bauma de Serrat del Pont deposit, 5380 cal BC (Tortellà, Girona) (Alcalde et al. 2002), Plansallosa, 5250 cal BC (Bosch et al. 1999) and La Draga, 5201 cal BC (Banyoles, Girona) (Terrades et al. 2015), and the northern part of the Catalan coast with the Sant Pau del Camp site, 5360 cal BC (Barcelona) (Molist et al. 2008). Although traditionally attributed an eastern origin to the main domestic species, recent publications highlight the possibility of autochthonous domestication in the case of bovids and suidae (Navarrete 2017). Agricultural practices are also documented in a large number of settlements, with cereal and barley being the most predominant taxa, along with legumes (Buxó 2008, Antolín 2013).

Along with livestock, the cultivation of cereals and domestic legumes is well documented in this region, both in the plain and in mountain areas, and would be a basic axis for the economy of the Neolithic groups. Highlights include naked and dressed wheat (*Triticum sp.*), barley (*Herdeum vulgare*) and legumes (*Pisum sativum* and *Vicia faba*). These data suggest that agriculture was fairly consolidated and varied in the early Neolithic phases in this region. There are few sites that present a carpological study that includes the study of agricultural techniques and seasonality, such as Cova Colomera (Oms et al. 2013), Can Sadurní (Antolín et al. 2013) and La Draga (Antolín and Buxó 2012). The practice of collecting vegetables, although in a percentage lower than agricultural production, is characterized by the presence of remains of apple and wild pear (*Malys sylvestris* and *Pyrus pyraster*), hazelnut (*Corylus avellana*) or acorns (*Quercus sp.*), as in Font del Ros (Pallarès et al. 1997) or La Draga (Antolín and Buxó 2011).

Despite the low dietary impact of the consumption of marine resources (Fontanals et al. 2018), the presence of malacological remains is evident in cave and open-air sites in this region such as Sant Pau del Camp, Cavet, La Draga, Cova Bonica or Cova del Vidre (Bosch et al. 2000, Fontanals et al. 2008). The collection of malacological remains can respond to elements of ornamentation (Oliva 2015). This interpretation is reaffirmed, and not related to food, given the erosion presented by the remains, such as in Sant Pau del Camp (Estrada and Nadal 2008), or the anthropic modifications documented in Can Sadurní or La Draga (Bosch et al. 2011).

In contrast to the Pyrenees, pre-Pyrenean sites such as Cova Colomera and Font del Ros generally document a flint and local quartz (Gibaja et al. 2007). In the plain, the flint outcrops of the Ebro valley or the jasper of Montjuïc (Barcelona) are exploited by nearby sites (Terradas et al. 2011). The presence of lithic tools is predominantly in the form of slabs, raederas, scrapers, escataduras or pieces with marginal retouches, the asclas with predominantly denticulated as in les Guixeres de Vilobí (Oms 2014) stand out. Numerous macrolytic materials are also documented in this area, such as mills and mill hands related to the processing of agricultural products, such as in Sant Pau del Camp (Bofill et al. 2008) or La Draga (Bosch et al. 2000); or axes related to logging activities, such as in La Draga (Palomo 2000). The bone industry includes punches, as well as spoons and rings, such as those found in La Draga (Legrand-Pineau 2011).

2.1.1.2.2. Middle Neolithic (4500-3900 cal BC)

During the Middle Neolithic, changes in settlement patterns are documented. There was an increase in the number of deposits during this period, which allowed different strategies to be proposed in the settlement pattern. Occupations of the Can Sadurní and Cova del Frare sites continue, with clear signs of the use of space related to stabling (Edo et al. 2011). On the contrary, the predominant typology in this period are the open-air habitats. Some of these occupations are interpreted as seasonal from the construction of domestic spaces with perishable materials, of which only post holes are identified, such as in Ca n'Isach (Tarrús et al. 2017) or Serra del Mas Bonet (Rosillo et al. 2012).

With regard to agricultural and livestock practices, the data available for this period are scarce. Generally, agriculture is observed with the presence of mills (Bosch 2005) and the identification of seeds of domestic species in identified deposits and silos, such as in La Dou, Cova 120, Reina Amàlia or Caserna de Sant Pau (Antolín 2013), with which cereals are identified a greater presence of naked barley. Data related to harvesting were also obtained, such as hazelnuts and wild fruits (Antolin 2013). Livestock is defined predominantly by the presence of remains of ovicaprine fauna, followed by *Bos taurus* and *Sus domesticus*, focused on food production. The predominance of ovicaprines increases from the Postcardial Neolithic, as in Sant Pau del Camp or Reina Amàlia (Saña and Navarrete 2016). These data show the continuous decrease in the qualitative importance of wild resources, although the presence of wild boar, deer and roe deer remains was recorded in most of the sites studied (Navarrete 2017).

The characterisation in regional facies for this period is marked by the characteristics of the ceramic sets. The football facies present a continuity of the Ancient Neolithic, with dark tonalities and treatment of the burnished surfaces, being the vertical tubular handles an identifying characteristic for this facie (Molist et al. 2016). For the Molinot facie, the finishes are presented with brushes, as well as crest and applied cords of triangular section (Molist et al. 2016).

Among the material culture, a low presence of lithic industry is documented, which also has a local origin (from the Ebro valley). There are medium and short sheets, projectile tips, slabs and polished fragments (Bosch 2005). The bone industry documented in the post-cardial levels of Can Sadurní is characterised by the presence of punches, needles and spatulas (Edo et al. 2011). The most significant element is the ornamentation, where malacological remains are documented (Bosch 2005) and the first evidences of variscita fruit of the incipient mining that develops in Mines de Gavà (Molist and Gómez 2016). The distribution of necklace beads and variscita tools is recorded throughout Catalonia and southern France (Bosch and Santacana 2009).

2.1.1.3. Neolithic in the south of the Iberian Peninsula

From the pollen analyses carried out in the Cueva de la Carigüela, we know that the landscape of most of the south of the peninsula was made up of deciduous formations, such as the relatively dense Quercus. On the contrary, the edges of the mountains are blurred with birch and oak. For coastal environments, such as the Cave of Nerja, the predominance of wild olive and pine was identified.

2.1.1.3.1. Early Neolithic (5800/5600-4900 cal BC)

There are few published data from systematically studied sites for this region. The initial Early Neolithic is documented in the south of the peninsula between 6000/5800 and 5600 BC with the dating of the neolithic transition levels of La Carigüela. It will not be until the advanced Ancient Neolithic (5600-4900 cal BC) that the extension of Neolithic elements, such as ceramics or the domestication of plants and animals, is documented in other sites in this region, such as the Cueva de los Murciélagos de Zuheros or Cueva de El Toro (Molina et al. 2012).

For many years only cave sites were known, hence the name of cave culture, but as prospecting has intensified, open-air settlements have been noted, such as Cabecicos Negros (Almería) (Camalich Massieu and Martín Socas 1999), Zájara (Almería) (Goñi Quinteiro et al. 2002), La Dehesa (Huelva) (Vera Rodríguez et al. 2010), El Duende (Málaga) (Aguayo Hoyos et al. 1990), El Llano de las Canteras, La Molaina, Las Catorce Fanegas, Cerro de las Ánimas, Peñón de Salobreña, Cíavieja or el conchero de Cañada Honda (Huelva) (Borja Barrera et al. 1994, Martín Gómez y Campos Jara 1997). Sometimes the Neolithic substratum is superimposed on the microlaminar epipaleolithic, as happens in the caves of Ambrosio (Almería) (Jiménez Navarro 1962, Suárez Márquez 1981), La Carigüela (Granada) (Pellicer Catalán 1964), Vega Toscano 1997), Hoyo de la Mina (Málaga) (Such Martín 1996), Nerja (Málaga) (Jordá Pardo 1986, Acosta Martínez and Pellicer Catalán 1997, Pellicer Catalán and Acosta Martínez 1997), Bajondillo (Málaga) (Cortés Sánchez et al. 2007) and Nacimiento (Jaén). In the other known sites, the Neolithic appears ex novo, as the cave of the Bats of Albuñol (Granada).

In the first ceramics, those of high quality stand out, both with cardiac decoration and with almagra technique, whose degree of evolution suggests that they have already been formed, from Levante the cardials and from the southwest of the Iberian Peninsula the almagras. Also identified techniques such as incision, relief and different types of printing, and gestures differentiated in their execution, which is particularly appreciated in the case of the so-called boquique (Alday Ruiz and Moral del Hoyo 2011). The ceramic forms are reduced to bowls with an incoming edge, peralted, hemispherical, globular vessels with gland and ovoid, wavy edges, with grip elements of the mamelon type, perforated or not, straight or raised tongue, ribbon handles, multiperforated, vertical or horizontal tunnel handles. Decorative techniques include plastic with cords, generally with digital or instrumental impressions, incises, grooves, cardials and cardialloids made with toothed matrices, toothed edges, etc. Cardial decoration with the natis of the shell only appears in the cave of Malalmuerzo (Moclín). The decorative motifs, extremely varied, represent parallel broken lines, reticulated, horizontal spikes and large geometric compositions parallel, rectangular, curved and triangular, carried out with the cardial or cardialoid technique. Sometimes, the motifs are inlaid with red paste (Camalich Massieu and Martín Socas 2013).

The carved lithic industry, not very diversified, is of the microlaminar type and of epi-paleolithic tradition, with a high percentage of leaves, leaves and chips retouched and converted into scrapers, chisels, abated edges, denticulates, fractures, ridges and, in a scarce percentage, trapezoidal geometrics. Present, although not very abundant, are the polished, such as axes, axes and azuelas. The milling elements, such as flat and naviform mills, moletas and grinders, are usually stained with ochre and are not used for cereal.

The available data on subsistence practices for the Ancient Neolithic in this region are relatively scarce, we find one of the oldest dates for this region in domestic animal bone from the Cave of Nerja (Martins et al. 2017), or the large number of ovicaprine remains recorded in the set of fauna of Cueva de El Toro (Martin-Socas et al. 2004). In general, the data obtained for this region informs us of an oscillation between 60 and 90% of the wild fauna on the domestic one (Saña 2013), like the one registered in the Cueva de la Carigüela.

The oldest evidence of agriculture in the carpological register is given by the remains documented in the Cave of Bats, Los Castillejos, Cueva de El Tor, Cueva de Nerja (Pérez Jordá et al. 2011; Peña-Chocarro y Zapata, 2012). The radiocarbon dating of the seeds documented in Los Castillejos showed a consolidated agriculture to a greater or lesser extent depending on the areas from 5300 cal BC (Perez-Jordá et al. 2011).

2.1.1.3.2. Middle Neolithic (4900-4300 cal BC)

It is not possible to determine differences between the so-called Ancient Neolithic and the Middle or Full Neolithic from a technical, economic and social perspective (Martín Socas and Camalich Massieu 2014). The definition of the Middle Neolithic is marked by the occupation of sites from the Ancient Neolithic after a hiatus that oscillates generally between 600 and 700 years, between these two periods, as also happens in Los Castillejos de Montefrío (Granada) (Afonso Marrero et al. 1996, Martínez Fernández et al. 2010), the caves of El Toro (Málaga) (Martín Socas et al. 2004) and Los Murciélagos de Zuheros (Córdoba) (Vicent Zara-goza and Muñoz Amilibia 1973, Gavilán Ceballos et al. 2010) and La Loma (Aranda Jiménez et al. 2012). However, it has not been determined in the cave of Nerja, in that of Los Murciélagos de Albuñol or in Cerro Virtud (Delibes de Castro and Montero Ruiz 1997). This could be understood as the result of a differentiated reality between those sites where no occupational vacuum is detected, located in the coastal zone, as opposed to those located in the interior that offer a period of abandonment.

In this period the ceramics present incised, grooved and almagra decorations, with a notable increase in the number of vases with plastic decoration. The ceramic with the almagra has more specific weight towards the western part, decreasing notably the presence of cardial decorations. The ceramic forms follow the formal tradition of the ancient Neolithic, with symptoms of evolution, with deeper, cylindrical, closed and ovoid bowls, with the most indicated edges that prelude the S and Z profiles of the final Neolithic and with beginnings of weak hulls with concave incoming walls, persisting profusely the globular forms with glands. Flat bases, wavy edges and decorated with protrusions and mamelons are present. The gripping elements, continuing the previous types, are diversified with various species of simple mamelons, with horizontal and enhanced reed, with or without perforation for rope, bridge, tunnel and belt handles. It is the apogee of the long multiple or multi-perforated handles, of the so-called spout or spillway handles and of the elbow handles with upper appendage, the spoon making its appearance.

The decorative motifs acquire an unusual richness based on the most varied rectilinear geometrisms of parallel, loose, ramiform, scaliform or rectiform, metopes, reticulates and curvilinear geometrisms such as festoons, filled with other motifs, making their appearance the stelliforms (Nerja, Carigüela, Prado Negro, Carburero, Mujer) and some anthropomorphic with incise or cardialoide technique (Prado Negro). Many decorations start from the handles and, as a characteristic element, the toothed edge is frequent (Martín Socas and Camalich Massieu 2014).

In subsistence practices, a certain preponderance of domestic fauna can be observed over wild fauna, the latter being represented, in decreasing proportion, by bovids, pigs and ovicaprids, as also happens in Los Castillejos de Montefrío. In the caves of La Carigüela, Nacimiento and El Toro, the ovicaprido prevails over the bovine and pig. In general, domestication prevails over hunting. On the coast, marine species, both ichthy-ofauna and malacofauna, form a very substantial part of the diet, with the consumption of "patella" standing out over other species. As far as agriculture is concerned, only the cultivation of wheat in the Toro cave (stratum IV) has been confirmed, while it is still absent in the Nerja cave. The Carigüela is still problematic. The diet also includes acorns and pine nuts. On the other hand, the frequency of ceramics, with smoke black on the outside and burnt remains inside, shows a change in the preparation of food.

By way of synthesis, we see the variability registered in the different areas of study. Although they present clear Neolithic elements, the rhythm and incidence of exploitation of the agricultural practices are different among them. The Pyrenees are characterised by extensive livestock farming and seasonal agricultural practices. This pattern was slightly modified at the beginning of the 5th millennium BC, with the occupation of the valley bottoms in stable open-air deposits and clear signs of agricultural exploitation. In contrast, in the plain we see a change in the pattern of settlement from caves to open-air sites, where the caves would come to have a use related to the stabling of livestock. In the south of the peninsula, the occupation of the sites that we documented in the Ancient Neolithic period continues after a hiatus. This hiatus is not recorded in the open-air coastal sites, which would indicate a change in the settlement pattern and a use of the spaces in the sites occupied since the Ancient Neolithic for different purposes, such as the stabling recorded in phase III of El Toro Cave.

2.2. Analysed sample

For the study of the effective use of ceramics from the Mediterranean basin of the Iberian Peninsula during the Neolithic period, 200 ceramic dated between 5400 and 3800 BC vessels have been selected from 9 sites located from the Pyrenees to the southern of Iberian Peninsula.

The biomolecular study of the content of neolithic ceramics in the Iberian Peninsula was carried out at various levels. On the one hand, it was considered at a historical level in order to influence the strategies of subsistence and exploitation of resources in a period in which great economic changes are taking place. Secondly, the viability of the analysis of lipidic materials in a Mediterranean context, which has been proposed as difficult for this type of analysis given its climatic characteristics and the type of soils. Finally, we wanted to focus on the possible variety of products processed inside the vessels. All these questions are developed and justified below.

2.2.1. Iberian Peninsular sites under study according to their geographical distribution and chronology

The sites studied in this thesis have been selected according to different criteria. As we have commented previously, throughout the archaeological research in different points of the peninsular geography, a great number of sites dated between 5600 and 5400 BC are documented in which the presence of ceramics and evidence of domestic species of animals and plants is recorded, such as in Les Guixeres (Vilobí, Barcelona) (Baldellou and Mestres 1981), la Cova de l'Or (Beniarrés, Alicante) (Martí et al. 1980) or Cueva del Nacimiento (Pontones, Jaén) (Asquerino et al. 1981). In particular, we focus on the Mediterranean strip of the peninsula, a point where some of the evidence of ceramic presence and/or remains of older domestic species converge, such as in Mas d'Is (5600 ca BC), the Cave of the Bats (5600 cal BC) or the cave of Can Sadurní (5500 cal BC). Thus, sites were selected with a chronology between 5400 and 3800 cal BC covering this entire eastern strip, from the Pyrenees to the south of the peninsula.

On the other hand, the location and typology of the settlements have been taken into account. Samples have been selected from five sites in a high mountain context. In this case, the specific objective of the study of these samples is to contribute new data to the knowledge of the practices of exploitation of wild and domestic resources that would be carried out in an area and context for which we still have little data.

For the northeast of the Iberian Peninsula, samples were selected from the La Draga and Mines de Gavà sites, given their relevance and specificity in terms of both rainfall conditions and functionality and type of rainfall. These sites have been studied in profucity but no analysis of organic residues in ceramics had been carried out until then, with the exception of the square mouth vessel analysed by GC-MS recovered in the Minas de Gavà deposit (Juan-Tresserras 2004).

Finally, two sites from the south of the peninsula, Cueva de El Toro and Cabecicos Negros, have been included in this study. The study of the other extreme of the Iberian Peninsula but located in the Mediterranean region allows us to contrast the study with sites from a region with different regional dynamics for the development of the Neolithic.

2.2.2. Peninsular sites under study according to type

Another factor of variability that has been considered for the selection of sites for this study is the type of sites. In this way, three sites have been represented in the context of a cave (Coro Trasito, Cova del Sardo and Cueva de El Toro), four open-air sites (Feixa del Moro, Camp del Colomer, Carrer Llinàs 28 and Cabecicos Negros), a site in a lake context (la Draga) as well as the Minas de Gavà, a mining complex with specific characteristics given that it is not a settlement and the vessels come from inside the mines.

2.2.3. Iberian Peninsular sites under study according to environmental conditions

With regard to lipid preservation, sites located in different soil types have been selected, from soils with neutral pH (pH 6-7) that conserve organic matter worse (DeLaune et al. 1981, Moucawi et al. 1981), such as the sites located in the Pyrenees; even soils with more basic pH (pH 7-8) in which experimentation has shown a greater conservation of lipids (Debono Spiteri 2012: 128-188), as in the eastern coastal zone of the Iberian Peninsula.

It has also been assessed whether the type of settlement, in the open air or in a cave, allows for greater or lesser conservation of organic matter. The degree of humidity and stable temperature offered by the shelters and caves shows a greater conservation of organic matter, while the open-air sites are exposed to changes in humidity and temperature as well as more significant taphonomic processes (Drieu 2017). Exceptionally, the site of La Draga has been selected, as it presents an anaerobic context, optimal for the conservation of organic matter. With this last case, we want to evaluate the effect of this type of context on lipid degradation, given the good results offered by the La Marmotta deposit for this type of analysis (Debono 2012).

Based on this variability, we focused on identifying patterns that relate the use of containers with strategies for articulating the occupied space, subsistence and, at a broader level, population.

2.2.2. Pottery vessels selection

A minimum number of samples considered representative for each case study with reference to the analysis of organic residues in ceramics has not been established so far. Although it is true, in the sites with chronologies ranging from the Neolithic to the Roman Age, only 26 case studies of 52 documented reported more than 10 samples. In the thesis on residues in the Mediterranean area, the frequency of the number of samples per deposit is very variable, ranging from 5 to 30 samples (Debono 2012, Drieu 2017, Breu 2019).

In this case, from the ceramic set of each deposit, between 26 and 50 vessels were selected for sampling. Depending on the number of vessels identified at each site, about 30 vessels were sampled in each case study. Given that 30 is a statistically explanatory and economically feasible number, we have adapted the sampling to each particular case. The high degree of fragmentation in the Cova del Sardo, Coro Trasito and Cabecicos Negros sites did not allow for the restitution of a large part of the vessels that make up each set, which is why around 30 samples were sampled from the identifiable parts of the vessels, lips or bases, which allowed them to be individualised to ensure the sampling of different vessels. In other cases, such as the Feixa del Moro, the four large vessels found during the excavations were sampled. This criterion was conditioned by the availability of the samples and the exceptional category of being four of the six large jars reconstructed at the site.50 samples were considered at Cueva de El Toro, given the large number of restored vessels at the different Neolithic levels of the site. In the sites with the highest volume of ceramic materials, such as La Draga or Mines de Gavà, 30 vessels were selected. This number, not proportional to the number of vessels identified in the sites, was limited due to a question of time and economic resources. Whenever possible, the sampled vessels respond to restored or partially restored forms, in order to know their morphotypology and be able to define their metric aspects (mouth opening, volume, wall thickness, etc.). In this way, the selected vessels are intended to represent the different typologies. In order to do this, all the containers were assessed globally and the morphometric categories were grouped in order to be able to make a representative selection of all of them. In these categories the volume, the opening of the mouth, the presence of handles, the thickness of the walls and the general morphological characteristics (presence of necks, type of bases...) were contemplated. A clear example is the Mines of Gavà, Juberri and Cueva de El Toro, where the restored forms were prioritised given the large number of whole profiles available.

From the vessels selected in each case, the selection of samples is subject to the characteristics presented by each of the containers. The main parameter is the identification of the uses that involved the ceramic containers and the identification of their contents. Therefore, a priori, the aim was to locate visible carbonized remains or traces of use (surface wear, flaming, marks of use, etc.). Secondly, the technological properties of the fired clay of each vessel were taken into account, given that the degree of porosity of the surface has a significant impact on absorption, as well as firing temperature and surface treatments (Correa-Ascencio and Evershed 2013, Evershed 2008, Rice 1987: 350-351, Stern et al. 2000). For this reason, in this work, priority has been given to the internal surfaces of the lips and the bases of the containers.

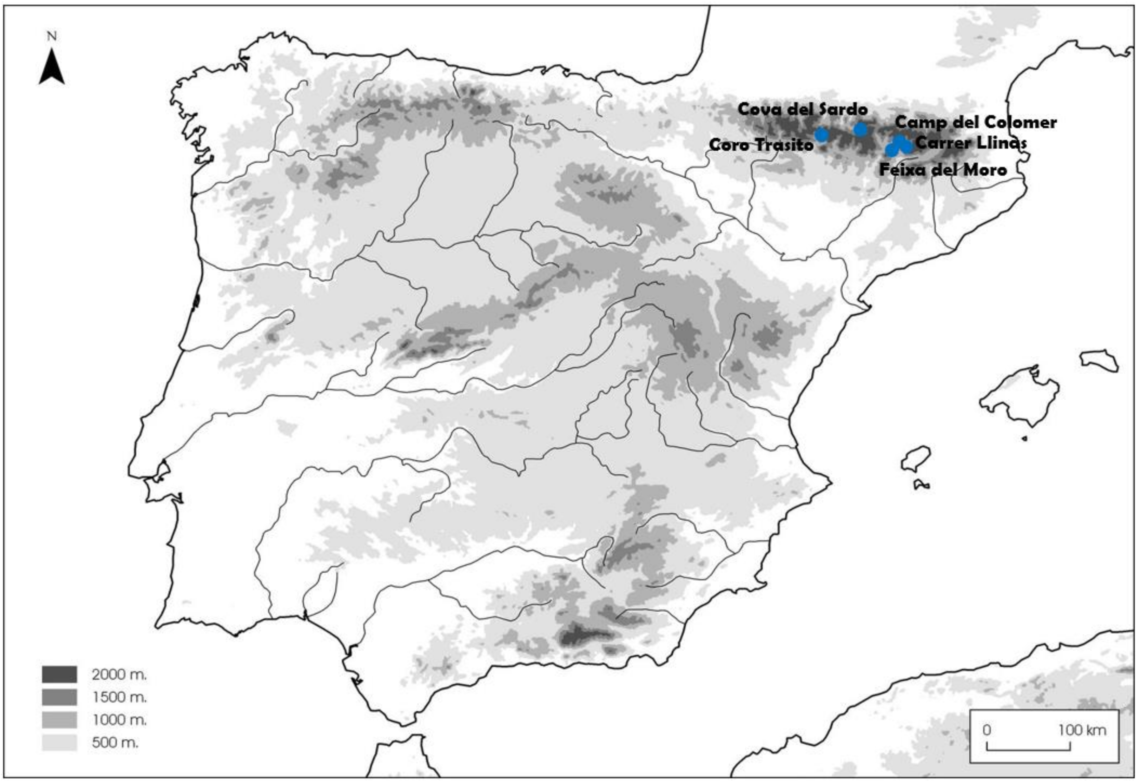
Finally, the preferential accumulation of lipids in certain areas of the vessel was taken into account. Experimental studies show that cooking food by boiling or roasting accumulates the lipids inside the containers in a differential way due to the difference in densities. As a result, the boiling of food products presents a higher concentration of lipids in the upper part of the vessel (Charters et al. 1993, Evershed et al. 1995, Salque 2012), while the roast, due to the production of water and fat from the meat during processing, distributes the concentration of lipids evenly throughout the vessel (Evershed 2008).

2.3. Composition and characteristics of the study sample

The nine sites selected for the analysis of organic residues in ceramics are described below. The section is organized with the classification of the sites in three regions: high mountain, northeast peninsular and south peninsular. In each case, a description of the site, its location and stratigraphy is made with the dates in each of the levels. Next, the interpretations of the use and occupation dynamics of the spaces in each of the sites are explained. The practices of subsistence are described in detail and then retormed in the discussion. Finally, the recovered arcaheological remains are described with emphasis on the description of the ceramic ensemble. Finally, the selected vessels and their procedure are described.

2.3.1. Pyrenees context

From the area of the central and eastern Pyrenees, five sites dated between 5400 and 2500 BC have been selected. These sites present a typology between cave and open-air settlements that develop different practices throughout the periods of occupation. All of them form a set of 64 samples. Each is described below.



SITE	TYPE	REF	LEVEL	SAMPLE	DATE BP	BIBLIOGRAPHY
Camp del Colon	Open-air	Beta-325686	FS29	Seed	5630±40	Oms et al., 2018
Camp del Colon	Open-air	Beta-325684	SJ24	Seed	5350±40	Oms et al., 2018
Camp del Colon	Open-air	Beta-325685	EI11	Seed	5300±30	Oms et al., 2018
Feixa del Moro	Open-air	CNA-2330.1.:	FM2	Human bone	5025±45	Oms et al., 2018
Feixa del Moro	Open-air	CNA-2331.1.:	FM3	Human bone	5095±45	Oms et al., 2018
Cova del Sardo	Cave	KIA-32348	Fireplace	Charcoal	4090±35	Gassiot et al., 2014
Cova del Sardo	Cave	KIA-26251	Test	Charcoal	4210±35	Gassiot et al., 2014
Cova del Sardo	Cave	KIA-40817	Fireplace	Charcoal	5685±35	Gassiot et al., 2014
Cova del Sardo	Cave	KIA-37691	Level	Charcoal	4715±35	Gassiot et al., 2014
Cova del Sardo	Cave	KIA-37689	Fireplace	Charcoal	6525±45	Gassiot et al., 2014
Coro Trasito	Cave	CNA.2520.1.	3002	Charcoal	5830±35	Clemente et al., 2016
Coro Trasito	Cave	Beta-358571	3010	Charcoal	5990±40	Clemente et al., 2016
Coro Trasito	Cave	CNA.2944.1.	3013-3014	Seed	6962±33	Clemente et al., 2016
Coro Trasito	Cave	Beta-366546	3013-3014	Bos taurus	6150±40	Clemente et al., 2016

Figure 2.1. Location and radiocarbon datations of Coro Trasito, Cova del Sardo, Camp del Colomer, Carrer Llinàs and Feixa del Moro.

2.3.1.1. Coro Trasito (Tella-Sin, Huesca) (5300-4600 cal BC)

Coro Trasito is a large cave of more than 300m2 located at 1548m a.s.l. in Tella-Sin (Huesca, Spain). Coro Trasito has ideal orientation for the human habitat since its location prevents the wind from entering at the same time that it lets the light of the sun in and it is quite wide. Being part of a karst system, internal water sources are currently flowing and filtering to re-emerge at lower elevations.

The excavated sediment was composed mostly of fumier, or excrement, and was perforated either by burrows made by animals or by structures made by the ancient inhabitants of the space (Clemente et al. 2014b; 2016).

The site was apparently abandoned in the mid fifth-millennium cal BC and not occupied again until nearly three millennia later, in about 1400 cal BC. Available radiocarbon dates show that the oldest known occupancy phase currently ranges from 5300 to 5100 cal BC.

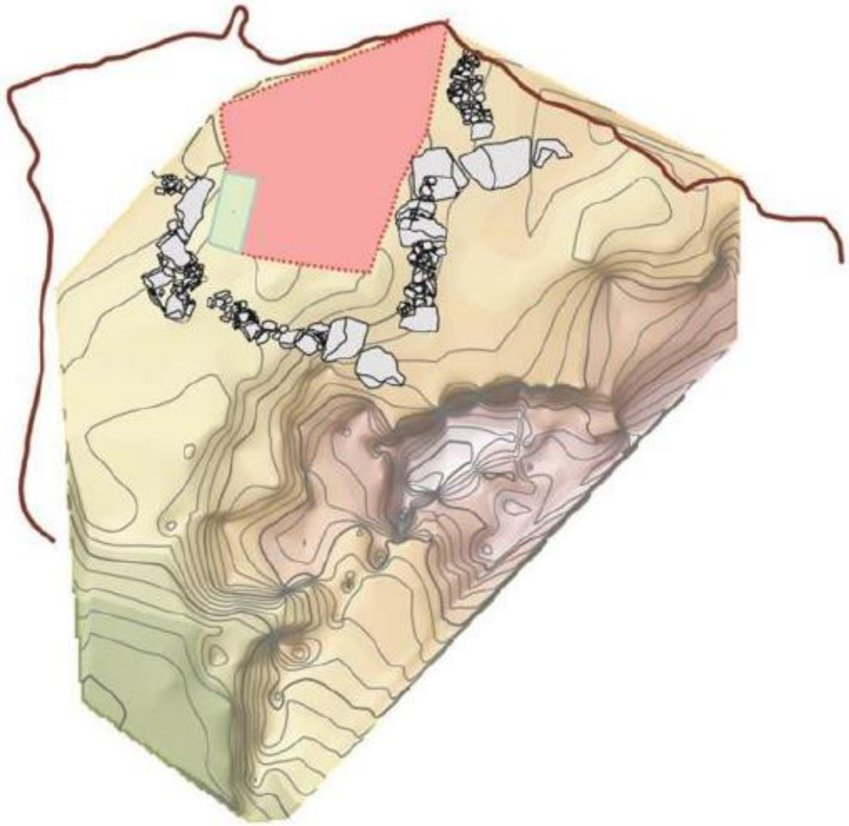


Figure 2.2. Plan of the interior of the cave of Coro Trasito. The area delimited in red marks the grid of the excavation. The green rectangle indicates survey No. 2 for 2011, subsequently extended in 2013 (Clemente et al. 2014).

A test excavation performed in two stages in 2011 and 2013 revealed a thick sequence of foumier sediments (burned layers of animal wastes mixed with soil) dated from 5300 to 4600 cal BC (Clemente et al. 2014a, 2016). The large stratigraphic packages between layers of black earth have defined these UEs, which alternate abundant archaeological material with other completely sterile ones that would correspond to the non-occupation phases in the site (Clemente et al. 2014a and 2014b). Certain UEs and the filling of storage structures and post holes contain soil formed by silts and small clasts of inorganic origin and their presence would respond to anthropic action. In fact, as already indicated, the extensive excavation has revealed a large number of pits. In this time, the cave entrance was used as a stable for livestock, for storage in small silos and as a dwelling.

In different settlement phases, post holes and other negative structures were found, indicating that the cave was multi-functional. Within neolithic stratigraphy, the following phases can be identified:

- Phase III: consisting of ESU 3004, 3002, 3005, 3006 and 3006Base. The EU 3007, with very little material, seems to mark the change with the previous phase. The chronology of this phase is around 4800-4580 cal ANE.

- Phase II: contains ES 3008, 3009, 3010 and 3011. A deposit of gravel and clasts (UE 3012) marks its base. Its chronology is between 5000 and 4800 cal ANE.

- Phase I: consisting of ESU 3013 and 3015. It was possible to excavate in a very reduced area due to the appearance of large blocks of limestone. It is dated between 5300 and 5000 cal ANE.

Both the test and the proper excavation, currently in progress, have recorded a large variety of archaeological remains.

As for the anthracological study, the finding of carbonized woody fuel indicates an intention in its gathering and subsequent use, possibly due to its high calorific potential especially if we take into account the wide availability of manure that could be reused as fuel.

Other tree species identified in the deposit are oak (Quercus caducifolia), present in all stratigraphic units, and red pine (Pinus sylvestris). Both species are characteristic of mid-mountain forests, found between 600 and 1700 m.a.s.l. and their abundance suggests that it could be a resource obtained in the vicinity of Coro Trasito. Hazelnut (Corylus avellana), yew (Taxus baccata), willow (Salix sp.) and maple (Arce sp.) are woody varieties that grow in shady and humid areas -preferably near river courses. If there were water currents in nearby areas, these species could have been collected in an area reasonably close to the site, a hypothesis that has to be confirmed by micromorphological studies (Obea, 2014).

It is interesting to observe how boxwood (Buxus sp.) is represented in the most recent stratigraphic units -II millennium-, while at that time the oak decreases to a minimum level and the pine disappears. This is a species associated with the disappearance of open oak groves, generally due to degradation by human action and pressure on the forests, which would fit with the disappearance of the oak grove by actions of exploitation and massive obtaining of this species.

We have to bear in mind that all the species described above have multiple values: high calorific power as fuel, quality to work with it and make utensils and abundance in the environment (Obea, 2014).

2.3.1.1.1. Subsistence practices

The type of sediment has enabled good conservation of osteological remains, resulting in the identification of 656 faunal remains. 90% of the remains that have been determined taxonomically come from domestic species: sheep, goat, pig and cattle. Not far from the cave, at an altitude between 2100 and 2300m a.s.l. there is a vast area of pastures. This proportion decreases in following Neolithic occupations to 70%. In the second millennium occupation cited above, the percentage of domestic fauna is still smaller; 55% of the identified remains. The wild animals consumed were mostly tortoise, red deer, roe deer and rabbit (Clemente-Conte et al. 2016).

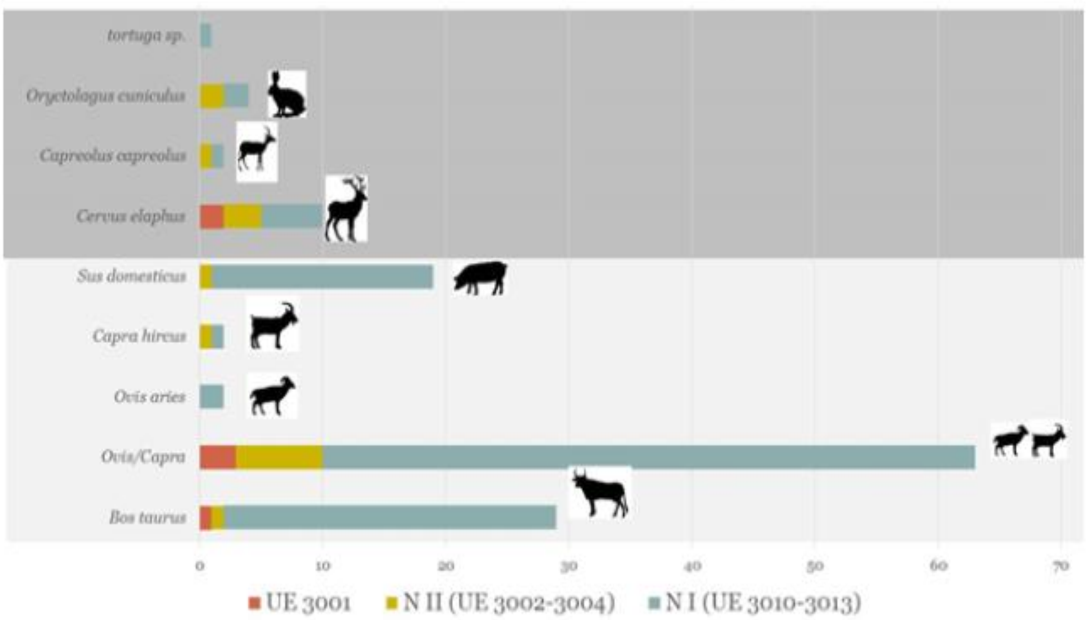


Figure 2.3. Faunal remains from Coro Trasito (Viñerta 2015).

During the test excavation 761 carpological remains has been recovered, following the same trend as the fauna, the largest numbers of cultivated species and seeds came from the oldest occupation phase at the site. Moreover, the closest arable land is found 650 m away, around 1370 m a.s.l. Thus, the carpological study has made it possible to specify that wheat (*Triticum aestivium/durum/turgidum*) and naked barley (*Hordeum vulgare var. Nudum*) are the most recurrent taxa throughout the whole territory, both in terms of representativeness and temporal continuity. Barley (*Hordeum sp.*), minor spelt (*Triticum cf. Diccocum*), wheat (*Triticum sp.*), undetermined cereal and pea (*Pisum sativum*) have also been documented. The cultivated seeds describe three different chronological moments in terms of consumption that corresponds to the three phases observed in the stratigraphy. We even find an abundant quantity of cultivated seeds, one of the oldest levels documented in the survey: the EU 3013 with a chronology of 5300-5000 cal ANE, data that, supported by pollen analyses, denotes the presence of agriculture in nearby areas and in ancient chronologies. In later Neolithic levels, between 5000 and 4600 cal BC, the total number of seeds of domestic cereals decreases, but peas (*Pisum sativum*) have been found in some occupations and, above all, intense consumption of hazelnuts and, in some cases, of blackberries (*Rubus fructicosus*) has been documented in the most recent levels.

The wild fruits collected have great weight in the sample, the most consumed being the hazelnut - from which we have obtained charred samples - the blackberry and the yew (*Taxus baccata*), among others -as a wild rose (*Rosa sp.*), blackberry (*Rubus fructicosus*), red elder (*Sambucus cf. Racernosa*), common juniper (*Juniperus cf. Communis*), pine fruit (*Pinus mugo/sylvestris*), oak acorns (*Quercus sp.*), wild vine (*Vitis vinífera subsp. Sylvestris*), maloidea (*cf. Maloidae*) and malvaceae (*Malvaceae*). Most of them are summer-autumn fruits that are stored in the silos of Coro Trasito, and we have to bear in mind that some of the fruits mentioned above, such as the vine, appear in certain excavation units and do not have recurrence in time like other taxa.

In short, the first occupants of Coro Trasito practised a subsistence economy in which animal husbandry and crops played a significant role in the supply of food. Their material culture is also comparable with that of other “Neolithic” sites of the same period in less mountainous regions.

2.3.1.1.2. Archaeological materials

While lithic objects are not very numerous, abundant micro and macrolytic instruments have been recovered (flint sheets and laminae, axes, azuelas, percutores, hands and fragments of mills, etc.) made of corneal rock, very abundant in this area of the Pyrenees, granite, and rock crystal. The flint used comes from different geographical locations, abounding those coming from the Ebro basin (Mazzuco et al. 2014). Food handling tools have also been documented - as would be the case of a double-edged cutting tool used to harvest and process herbs and/or cereals, while the opposite edge was used to cut meat, cut and perforate skins, elements related to working in wood, thermoaltered boulders possibly used to heat water (Clemente 1995,1997; in Clemente et al. 2014a) and remains of mineral origin suitable for working ceramics (in fact, the hypothesis of using a boulder as a ceramic polisher due to its traces of use on the surface has been raised), something that would be recurrent in some Neolithic sites in the Pyrenees (Mazzuco, 2014).

As far as the bone industry is concerned, remains have been recovered up to the stratum relative to the 4900 cal ANE chronology. A stylised punch made from a highly polished ovicaprid metapod is noteworthy, with evidence of stretch marks and remains of vegetable matter, so that it could have been used in textile or basketwork work. Abraded and pointed splinters have also been documented for sewing or perforating skins.

2.3.1.1.3. Pottery remains

Coro Trasito's ceramic set is represented by containers of different sizes and shapes. The concentration of ceramic material is not very numerous and appears very fragmented. However, it has been possible to restore some profiles. In general, the Coro Trasito containers have large dimensions and a medium wall thickness, which has been related to the storage of products. The diameters are small, whereas the maximum diameters indicate bulky containers. As far as surface finishes are concerned, there are differences between the chronological phases. Ceramics have few decorations and tend to have smooth or polished finishes on both surfaces. As for the typology of vases and decorative techniques, rounded lips predominate at most levels (we do not have data for phase III, as no morphologically representative fragment has been documented). It is remarkable the presence of a fragment (1310.S3.3010.C47) with a type of decoration that we can assimilate with the previously explained boquique. This type of decoration has been documented in numerous peninsular sites in Neolithic chronologies, although it is necessary to specify that it is necessary to break with the historical-cultural idea of isolated entity that gives the presence of certain decorative features. In the case of the boquique, although a certain recurrence is documented in certain areas more than in others, it does not form homogeneous or closed units since its frontiers are very diffuse.

2.3.1.1.4. Analysed samples

For this study, a total of 26 potsherds were sampled from the Neolithic cave of Coro Trasito. Due to the high degree of fragmentation of the Coro Trasito ceramic ensemble, it was impossible to restore any ceramic form. The samples corresponding to the Coro Trasito study correspond to 12 lips from phase I (4992-4786 cal BC) and 12 lips and two base fragments from phase II (4785-4585 cal BC) of the Neolithic in the site.

2.3.1.2. Cova del Sardo (4800-2500 cal BC)

The Cova del Sardo is a mountain rock-shelter located in central Pyrenees, at 1790 meters above sea level. The cavity is 9m wide, 3m deep and 1.3 m high, with an inner surface of about 20 m², which has been progressively filled by ravine sediments (Gassiot, 2010).

It is next to Sant Nicolau River in the valley with the same name. Sant Nicolau valley is one of the wettest valleys in Catalonia at present, with an average annual rainfail of more than 1100mm (Gassiot et al. 2012). The geology of the surrounding area is of granitic nature (Celma et al. 2008).

The systematic archaeological excavation work carried out between 2006 and 2008 documented a long sequence with eight occupations phases (Gassiot 2010). The first archaeological evidences of an anthropic occupation of the cave belong to phase 9, dated to 5600-5400 cal BC, without pottery evidences. After a hiatus of over five centuries, human presence is testified by a number of levels dated between 4800 and 4350 cal BC (phase 8), between 3900 and 3500 cal BC (phase 7), around 3300-3100 cal BC (phase 6) and the last prehistoric occupation of the cave (phase 5), dated between 2900 and 2500 cal BC (Gassiot et al. 2014).

Between 5600 and 2500 cal BC, the cavity was used repeatedly for human settlement, although the patterns of use of space were modified significantly during the different phases. Most of the occupations of the site took place in the area under the rock shelter, although in phase 6 a wooden roof was attached to its entrance. The remains of Coprophilia and microalgae spores have also shown how in their interior moments of human presence alternated with others of absence, indicating that occupations would probably be limited only to certain periods of the year (Gassiot et al. 2010), which would correspond to warm seasons because high temperatures and snow accumulation prevent access to the cave in winter seasons. In each phase, fireplaces acted as a centre of productive activities.

The site we are dealing with seems to be a stable and continuous point from the 6th millennium onwards in the subalpine areas. Pollen analyses show a dense woodland cover of pine and birch with evergreen and deciduous oaks at lower altitudes. Ferns are abundant and riverside forests would be found in the river valley. Grasslands, together with pine woods, are better represented in phase 8. It is considered that human modification of the landscape becomes more intense in this phase, when birch and hazel expand significantly. The anthracological results show a wide predominance of pine wood (Pinus nigra) and some riparian taxa (Obea et al. 2011).

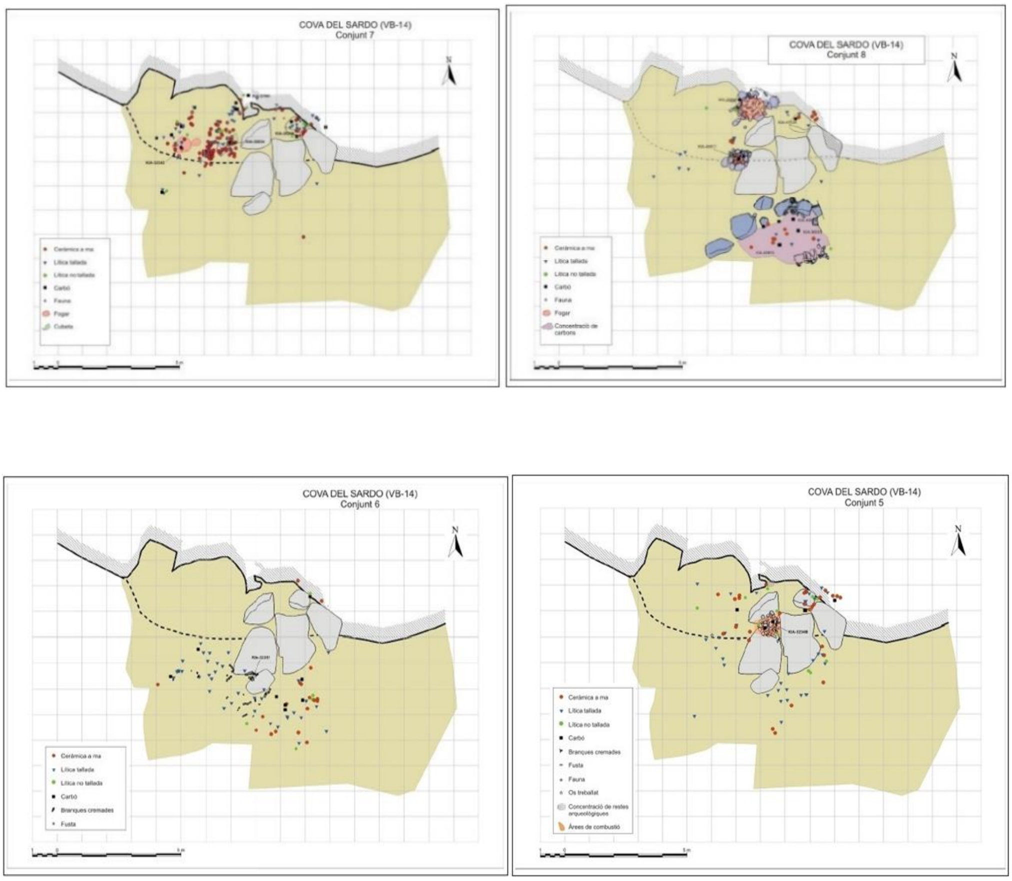


Figure 2.4. Plant drawing from Cova del Sardo.

2.3.1.2.1. Subsistence practices

Pollen and carpological studies show that Hordeum vulgare has been used for agriculture and livestock, and that other plant resources in the area have been exploited. This population dynamics will be complemented by a less frequenting of small shelters in headwaters, in alpine areas (Gassiot et al. 2014). Subsistence strategies are characterized by livestock, with the main exploitation of ovicaprids. The fauna remains of the Cova del Sardo site are made up of 3100 remains distributed in the different levels of occupation of the shelter. The set of remains presents high levels of fragmentation (85% of the average remains less than 2 cm) and oxidation (99% of the remains were burned). As a result, only 32 remains could be classified as ovicaprine, since poor preservation did not allow differentiation between sheep and goat species (Gassiot et al. 2015). Use-wear analyses of flint tools seem to indicate that the most common activity in the cave would be the processing of animal skins and flesh. Hunting could equaly be practiced by inhabitants of the site, especially considering the finding of several arrowheads (Celma et al. 2008).

All these characteristics have made it possible to hypothesize that a pattern of use of elevated pastures is illustrated, probably in summer, from more stable central settlements in the lower areas, such as the Cova del Sardo site, which perhaps did not have such accentuated seasonality or in which jobs were not carried out in the same periods of the year (Gassiot, 2010).

2.3.1.2.2. Pottery remains

The interventions carried out in the Sardo Cave made it possible to recover a total of 231 fragments of handmade ceramics. Despite this, the study was carried out on 220 fragments since the other 11 fragments are very small. Of this lot, 39 (17.72%) correspond to significant fragments, while the remaining 181 (82.28%) are reported fragments.

Of the set of documented significant fragments, 33 (84, 61%) correspond to borders. The high degree of fragmentation has not allowed restitution in any entire form. Only in 3 cases it was possible to reconstitute the diameter of the mouth (9% of the total number of edges). Two fragments of bases were documented, corresponding to a convex base and a flattened base of which the diameter is known, and an inflection in which its diameter could also be calculated. A gripping element was identified corresponding to a reed and two decorated fragments: a cord and an incision.

According to the thickness of the walls it was possible to differentiate three groups of measures, from 0 to 5mm, from 6 to 9mm and more than 10mm, existing a differentiation of the predominance of the thickness of the walls according to the studied archaeological level: in the oldest level the predominating thickness is superior to 10mm, with the appearance of the polished treatment in the set the thicknesses are diminishing until in the last archaeological level we found a majority presence of fragments with a thickness between 0 and 5mm.

The treatment of the surfaces shows a change from archaeological phase 7, where the ceramic products register a different treatment from smoothing and levelling; the polish and scraped, which responds to a greater work investment, but we will not find it again until phase 5. Even so, the smoothing treatment predominates in the ensemble. In the study, the treatment on the internal and external surface is differentiated to see if the relationship between the type of treatment and the surface where it is found could define their aesthetic or functional intentionality. The results determined that in phase 7 the scraped predominates on the external surface over the internal surface and, in phase 5, where the scraped reappears, the external surface treatment is mostly smoothed and on the internal surface polished. Unfortunately, due to the taffonomic processes, we were unable to determine the treatment of some fragments.

2.3.1.2.3. Analysed samples

The samples we analysed in the following study did not follow the sampling protocol from the time of excavation, since a residue analysis was not foreseen. The ceramic set was washed in water after excavation and stored in plastic bags for five years.

The sampling strategy in 9 ceramic fragments was focused on selecting the vessels best suited for residue analysis, seven rims and two bases. In addition, sampling represented each of the levels of the cave to explain chronologically uses of potter: two pottery fragments from level 8, three fragments from level 7, two fragments from level 6 and two fragments from level 5.

2.3.1.3. Juberri (Andorra) (4200-3800 cal BC)

The archaeological site of Juberri is located in the Principality of Andorra at 1300m a.s.l. and forms an open-air site documented in 1980 following emergency excavations for an urban transformation in the municipality (Llovera 1984, 1986, 1992).

After several archaeological interventions, three Neolithic sites located nearby are identified, among them: Carrer Linàs 28, Camp del Colomer and Feixa del Moro (Fortó et al. 2010, Martínez et al. 2014, Fortó and Vidal 2016, Prats 2018) dating from between 4900 and 3300 cal BC.

Carrer Llinàs 28 presents deposition levels together with construction structures in which ceramic, lithic and macrolytic materials were collected, superimposed and adapted to the inclined terrain (Fortó and Vidal 2018).

Camp del Colomer was discovered during the realization of some construction works close to Feixa del Moro. The site is radiocarbon dated around 4500-4200 cal BC. Two trenches of 12 x 60 m were excavated. 40 structures were identified: two large dwelling-type pits, 7 silo-type pits and 31 negative structures of unknown function (Martínez et al. 2011).

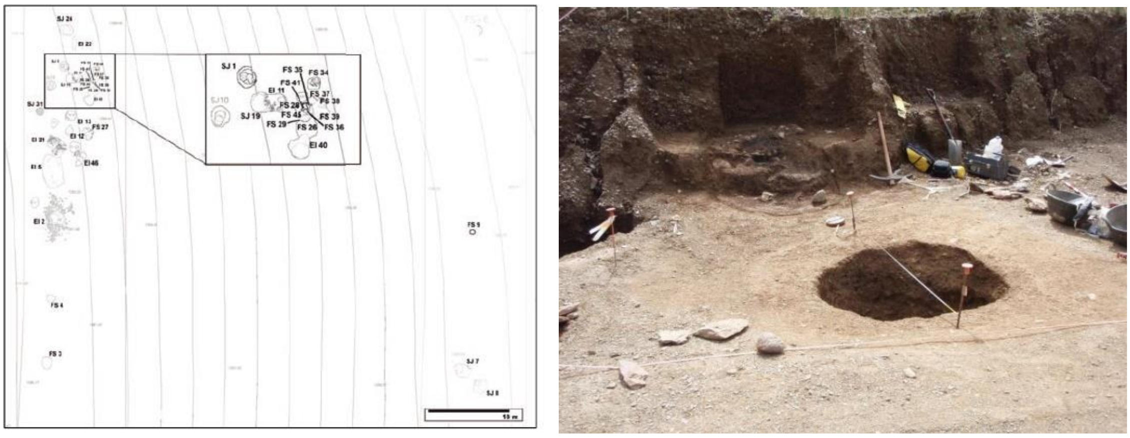


Figure 2.5. Site plan of Camp del Colomer.

Feixa del Moro, a large number of silos, pits and basins interpreted for the storage of products or the processing of plant elements, such as for torrefacturing acorns and hazelnuts, were documented. Combustion structures and stick holes were also identified, which together would define a space for habitat, storage and work (Fortó and Vidal 2018, Prats 2018). Feixa del Moro, on the other hand, also presents funerary structures that would share space with domestic areas.

2.3.1.3.1. Subsistence practices

The large number of polished tools and mills found at the site along with archaeological and botanical analyses have documented the importance of agriculture for subsistence practices at the site. At Camp del Colomer, the presence of domestic plants documented from archeobotanical analyses show a trend towards barley cultivation, particularly naked barley (*Hordeum vulgare var. nudum*), with naked wheat (*Triticum aestivum/durum/turgidum*) not very representative of the species cultivated at the site (Antolín 2016).

Cultivated legumes were also documented, such as the pea (*Pisum sativum*), and several poppy seeds (*Papaver somniferum*), presumably cultivated since the wild variety of this species does not grow above 900 m a.s.l. currently in our region (Antolín et al. 2017).

Finally, a large number of fruits and seeds of wild species such as hazelnut (*Corylus avellana*), acorn (*Quercus sp.*), wild strawberry (*Fragaria sp.*), wild pear (*Pyrus malus*) and blackberry (*Rubus sp.*) were documented (Antolín et al. 2017). In addition, an important concentration of hazelnuts and carbonized acorns was found in different silos (Antolín and Jacomet 2015), which also highlights an important and deferred consumption of species resulting from the harvest at this site.

The documentation of meadows and grasslands also confirms livestock capacity, although no direct evidence has been identified due to the absence and poor conservation of fauna (Antolín 2016, Piqué 2016, Fortó and Vidal 2018).

2.3.1.3.2. Pottery remains

The typological stile of the potsherds recovered was classified as belonging to Late Epicardial Culture, which is belived to develop between 4750-4500 cal BC (Martínez et al. 2011).

Samples from 29 sherds were selected for the biochemical analysis of the organic residues that may have been preserved. The aim of the sampling was to have a large number of samples from the three excavated areas that make up the archaeological site and which are interpreted with different dynamics (Fortó and Vidal, 2016): 4 sherds from La Feixa del Moro, 14 sherds from Camp d'en Colomer and 11 sherds from Carrer Llinàs 28.

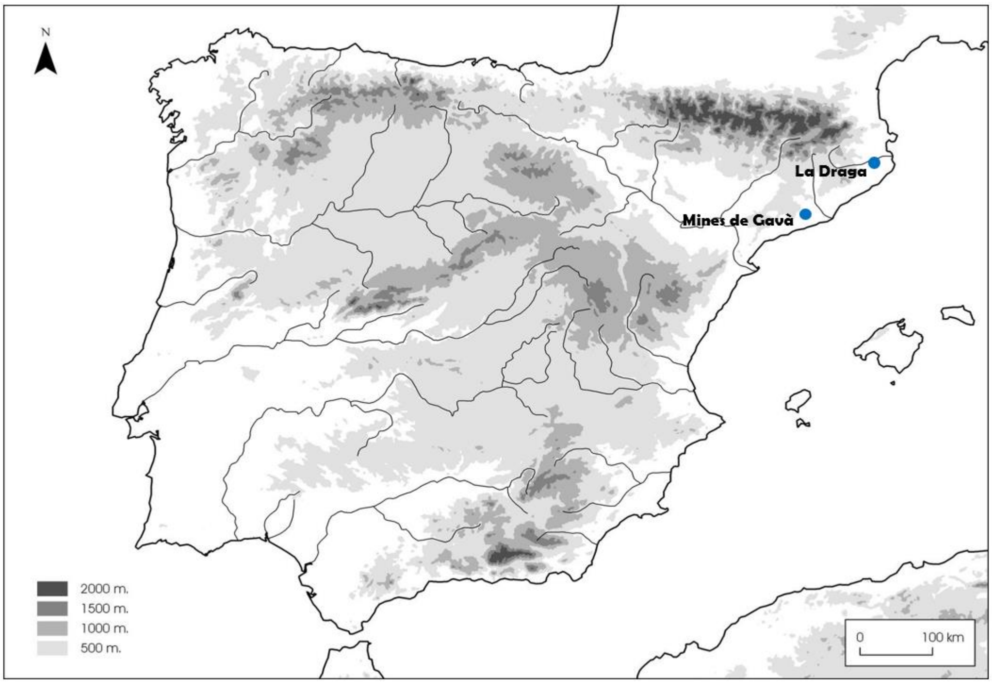
In feixa del Moro were selected: two large globular vessels, with incoming orientation and two simple handles on each side. An open globular vase with no decorations or gripping elements. A container with an exvased neck, of great size and that presents two simple handles.

They were selected from Camp del Colomer: Two fragments with applied cords, one simple and the other with incisions. A medium sized container of globular shape and open with tongues. A lip coming from a vessel with incoming orientation. Three containers with open and hemispherical shape. A container with a straight edge, with an undetermined volume. A small bowl with an open shape. A container with a marked, open-shaped rim that has no decorations. Two containers with medium capacity exvased neck that have handles and cords applied in the form of striae. One container with open shape and concave base with simple handles. A large jar with a very marked neck, handles and cords in applied striae.

Coming from Carrer Llinàs 28 were selected: A fragment of a simple handle. A fragment of cord applied with incisions. A small spherical bowl without decorations. An edge fragment corresponding to a protruding rim. A rim fragment of an exfoliated neck. Three border fragments of incoming shaped vessels. An edge corresponding to a hemispherical bowl with applied moustache and simple handles. Two border fragments corresponding to truncated conical vases with incised decorations on the border

2.3.2. Analysed sets from the northeast of the Iberian Peninsula

Coming from the Northeast area of the Iberian Peninsula, two sites dated between 5200 and 3900 cal BC have been selected. These sites are characterized by being a lake settlement, such as La Draga, and a site dedicated to mining, such as Mines de Gavà. All of them form a set of 54 samples. Each is described below.



SITE	TYPE	REF	LEVEL	SAMPLE	DATE BP	BIBLIOGRAPHY
La Draga	Open-air	HD-15451	E3-hogar	Charcoal	6060±40	Bosch et al., 2011
La Draga	Open-air	UBAR313	E56-hogar	Seed	6010±70	Bosch et al., 2011
La Draga	Open-air	Beta137197	Sector B	Wood	6290±70	Bosch et al., 2011
La Draga	Open-air	Beta137198	Sector B	Wood	6270±70	Bosch et al., 2011
La Draga	Open-air	beta	Sector B	Bos taurus	6184±27	Bosch et al., 2011
La Draga	Open-air	OxA20231	Sector B	Seed	6163±33	Bosch et al., 2011
La Draga	Open-air	OxA20232	Sector B	Seed	6121±33	Bosch et al., 2011
La Draga	Open-air	OxA20233	Sector A	Seed	6179±33	Bosch et al., 2011
La Draga	Open-air	OxA20234	E-5 hogar	Seed	6127±33	Bosch et al., 2011
La Draga	Open-air	OxA20235	E21 hogar	Seed	6143±33	Bosch et al., 2011
La Draga	Open-air	Beta278255	Sector C	Ovis aries	6270±40	Bosch et al., 2011
La Draga	Open-air	Beta278256	Sector C	Ovis aries	6170±40	Bosch et al., 2011
Mines de Gavà	Mine	I –11.786	Mina 6	Charcoal	5070±100	Villalba et al., 1986
Mines de Gavà	Mine	CSIC-488	Mina 7-pozo	Charcoal	4710±50	Villalba et al., 1986
Mines de Gavà	Mine	I–12.158	Mina 8	Charcoal	4880±110	Villalba et al., 1992
Mines de Gavà	Mine	Beta 268.778	Mina 16-nive	Charcoal	5090±40	Bosch 2010
Mines de Gavà	Mine	UBAR -42	Mina 41	Charcoal	4820±100	Bosch 1992
Mines de Gavà	Mine	Beta 72551	Mina 70-2	Charcoal	4930±70	Bosch & Estrada 1994
Mines de Gavà	Mine	Beta 250.403	Mina 84 - ga	Charcoal	4980±40	Bosch & Borrel 2009
Mines de Gavà	Mine	Beta 250.405	Mina 84 - se	Human bone	4880±40	Bosch & Borrel 2009

Figure 2.6. Location and radiocarbon datations of La Draga and Mines de Gavà.

2.3.2.1. La Draga (Banyoles, Girona) (5200-4700 cal BC)

La Draga is an open-air settlement located on the shore of the Estany de Banyoles at 170m a.s.l. Systematic excavations and tests have been carried out in the extension of the site since 1991 and up to the present day. The site is divided into different zones that make up the Neolithic settlement: Zone A, terrestrial; Zone B and D, phreatic, and Zone C, underwater (Bosch et al. 2000, 2011). It should be noted that a large part of the site is in a phreatic environment, which has allowed for excellent conservation of organic remains.

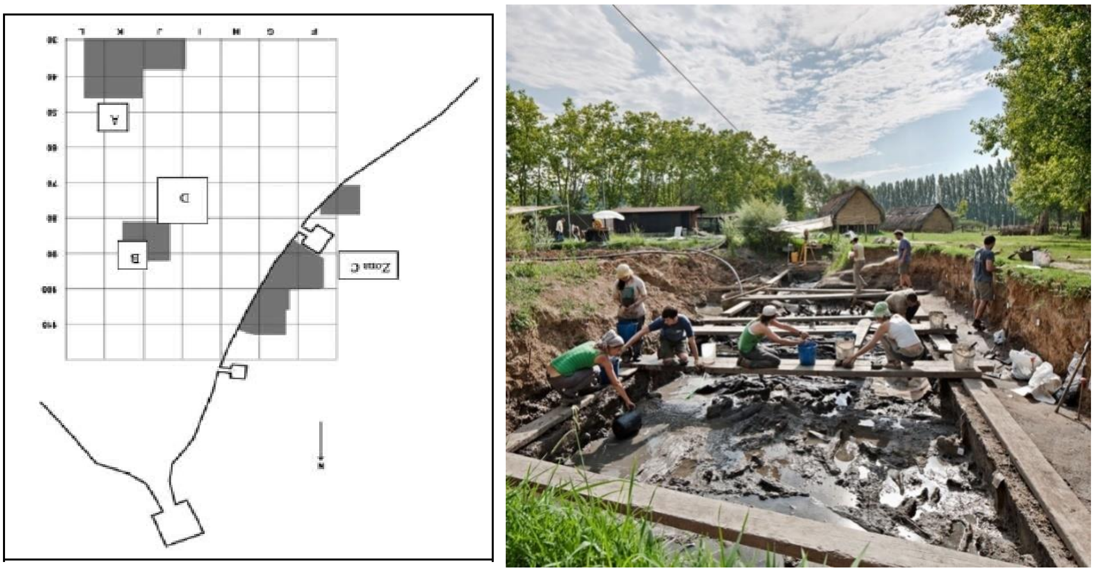


Figure 2.7. Location of the archaeological sectors of the La Draga site

Recent dates chronologically place the settlement between 5201-4721 cal BC (Palomo et al. 2014, Terradas et al. 2015). The settlement would have an area of about 15000 m2, of which 3000m2 have been excavated so far (Bosch et al. 2018). The settlement is characterised by a presence of elevated rectangular constructions in the area near the lake, and oval constructions in the interior area next to structures interpreted as granaries (Bosch et al. 2011). Two main settlement phases were identified in the open-air site of La Draga. The earliest occupation is a real pile dwelling site. Wooden huts were built right on top of the lake marl. This phase could last from c. 5300 to 5200 cal BC (Bogdanovic & Piqué 2012). Immediately after the collapse of the dwelling structures, a rather large accumulation of several layers of clay of terrigenous origin (Balbo & Antolín 2012) covered them and a new settlement was established, mostly using large travertine stones in order to produce an artificial floor. This second phase of occupation could have lasted until 5000 cal BC.

Zones B and D are characterised by the presence of two clear archaeological levels. This separation has been carried out from the architectural structures (Bosch et al. 2011). In zones A and C, on the other hand, the separation of two levels is delimited by a slight sedimentary level. The first archaeological level (5114-4911 cal BC) is characterised by the construction of huts or granaries of travertine forming oval paving. The second level (5394-5096 cal BC) would be characterised by the construction of rectangular wooden huts.

Palynological analyses revealed a beach of carbonate sand, the result of the regression of water in Lake Banyoles, which allowed the establishment of the Neolithic settlement on the shore of the lake. Deciduous forests were the predominant vegetation, especially oak and hazel (Burjachs 2000, Caruso-Fermé and Piqué 2014, Revelles et al. 2016). The data obtained reflect soil erosion processes related to deforestation initiated in the first phase and accentuated during the occupation of the site (Revelles et al. 2015).

2.3.2.1.1. Subsistence practices

Nearly 98% of the carpological remains of La Draga correspond to cultivated plants, both cereals and legumes. These charred remains represent agricultural practices dominated by barley (*Hordeum sp.*), wheat (*Triticum sp.*) and legumes, such as beans (*Vicia faba*) and peas (*Pisum sativum*) (Buxó 2007, Antolín and Buxó 2012, Antolín et al. 2014). Of these, bare wheat (*Triticum durum/turgidum*) accounts for 90% of cultivated spices, followed by dressed barley (*Hordeum vulgare*) (Antolin et al. 2014). Pollen analyses determined that crop fields would have been in higher ground, in an area close enough for intensive management (Antolín et al. 2014). Similarly, the over-representation of other herbaceous and shrub taxa (e.g. *Vitis, Asteraceae, Apiaceae, Plantago*) is the result of anthropogenic contributions to the settlement (Revelles 2017).

Among the wild taxa, the presence of cultivated opium poppy (*Papaver somniferum*) and other ruderal and adventitious species stand out. Others are related to lake or aquatic vegetation, such as Vitis vinífera sylvestris (Antolin and Buxó 2011). Fruits have also been documented, such as plums (*Prunus espinosa*), acorns (*Quercus sp.*), hazelnuts (*Corylus avellana*) and pine nuts (*Pinus pinea*) (Antolín 2013). These results, although representing a very small percentage with respect to the carpological remains of the whole complex, emphasize the importance of harvesting for the human diet in this settlement (Antolín et al. 2014).

The lacustrine site of La Draga constitutes a key settlement to explain the dynamics of integration and exploitation of animal resources by the first Neolithic communities in the western Mediterranean, given the good state of preservation it presents, which allows us to evaluate the weight and importance of animal production during the initial moments of the Neolithic. Among the animal categories represented in the site - amphibians, birds, reptiles, fish, malacofauna and mammals - the presence of macro-mammals stands out (97%). Wild animals (3%) and domestic animals (97%) (Saña 2011) were identified from among the fauna documented at the La Draga site.

Ovicaprids are the most represented species in the site (43%), among which we find *Capra hircus* (20%), *Ovis aries* (60%) and a remaining category with ovicaprines that could not be classified (Saña 2011). Both goats and sheep present cutting marks and anthropic thermoalterations in around 15% of the remains. Likewise, we see a differentiation in the slaughter age trend, the goats were destined for consumption before the end of the growth stage (<2 years), while the sheep were slaughtered between 2 and 4 years of age. From the mortality patterns, described from tooth wear and animal size, it was concluded that the main exploitation of goats would be related to meat and probably milk exploitation, while sheep would be exploited for meat without discarding animal fibers (Antolín et al. 2014).

The remains of bovids identified in La Draga are mainly represented by the domestic taxon (*Bos taurus*) (98%), while Bos primigenius is present among the remains of this species but in a residual form (2%) (Saña 2011). The slaughter age of cattle is grouped between individuals slaughtered before 3 months of age and others around two years of age (Antolín et al. 2014). About 30% of the *Bos taurus* remains show traces of decarnation and disarticulation of the extremities for meat exploitation and evidence of cutting for extraction of the medulla (Saña 2011). A relatively low percentage of individuals present pathologies derived from the systematic exploitation of cattle for agricultural work, transport or loading (Tarrus et al. 2006, Bosch et al. 2008).

The 901 remains of Suidae recovered at the site correspond to *Sus domesticus* (92%), *Sus scrofa* (1%) and a remaining category of *Sus sp.* for those individuals that do not present clear criteria for differentiation between wild and domestic form, since they were sacrificed within the first weeks or months of life. About 10% show traces of processing and thermal alterations that indicate the preparation of the meat food of suidae for consumption (Saña 2011).

Among the wild fauna represented, the wild boar (*Sus scrofa*), the wild goat (*Capra pyrenaica*), the deer (*Cervus elaphus*), the roe deer (*Capreolus capreolus*), as well as small carnivores (badger, wild cat, etc), rabbits (*Oryctolagus cunniculus*) and 112 bird remains (partridge, woodcock, etc) are documented. Some of the wild species, such as the wild boar, deer and goat, are represented by the presence of extremities, which appear thermoaltered, or the use of antlers or bones with marks of wear for use as tools (Saña 2011).

Complementarily to hunting, in La Draga also practiced fishing. The remains of ichthyofauna and malacologicos are not numerous, which shows that fishing and the collection of aquatic resources was not an activity practiced in a systematic way. Among the species of recovered fish are *Cyprinus barbus*, *Anguilla anguilla*, *Ratilus ratilus* and *Bagre bagre* (Bosch 2011). The collection of turtles and terrestrial and marine mollusks was the last strategy practiced at the site. Highlights the 42 remains of turtle pond (*Emys* *orbicularis*), which shows signs of exploitation for consumption. Among the seashells found, the following stand out G*lycimeris sp., Cerastoderma sp., Dentalium sp., Cymanthium vulgatum* and *Columbella rustica*, among which D*entalium, Nassarius mutabilis* and *Columbella rustica* are documented with anthropic modifications (Bosch et al. 2011)

2.3.2.1.2. Pottery remains

The reconstructed vessels from the different excavation campaigns at La Draga make up 40% of the more than 2500 fragments found at the site. The presence of ceramics is unevenly distributed in the different excavated areas, with sectors B and C being the ones that house the greatest presence of ceramic materials (2.44 fragments per m2).

In zones B and C there are frequent fragments measuring more than 10 cm, which can also be joined with fragments located in the vicinity. These are areas with room structures, in which the broken vessels had to be thrown directly to the mud or to the vegetal bottom that formed the ground under the houses, without being repeatedly stepped on. The small number of fragments in zone C, currently located inside the lake, may be due to a taphonomic problem related to the movement of water in a nearby area. To the sector A, the fragments, besides being less numerous, are of smaller dimensions and, often, have the eroded surface. It is a completely emerged area, with homes in buckets on the ground, and where the fragments should have been affected by traffic and the various activities that were done in the area.

The ceramic forms are little varied and standardized, as in other sites of the Ancient Neolithic. Globular profiles with a rounded base and semi-spherical ones with open shapes predominate. A simple form, with a continuous profile, but deeper and more closed, is the subespheric. Large jars with thick walls (>10mm) and cylindrical shape were also found, which are related to storage. Neck vessels are the only composite form, formed by a globular body and a neck. They are very closed and deep vessels, in which two different types can be distinguished: the format for vessels with a sinuous profile, with a neck that follows the profile of the vessel; and the format for vessels with a segmented profile, in which the cylindrical neck is juxtaposed directly on the globular body.

These vessels are intended for the preservation and transport of food, from which we have been able to reconstruct medium and large shapes (Bosch 2011).

In decoration, the most commonly used technique is cardial printing, which is made with the two Mediterranean species (Radicardium and Cerastoderma). It presents, as in all the sites of this period, a great variety of printing gestures: simple (with the edge perpendicular to the surface, or with several degrees of inclination), pivoting, printing with dragged continuation, or directly a dragging or combing of the surface with the edge of the mollusk (Bosch 2011).

2.3.2.1.3. Samples selected

27 fragments of vessels from different levels were selected for this study. In general, the whole from the excavation of sector D in 2012 of La Draga appears very fragmented. We selected 1 fragment of vessel from the level III (5218-4721 cal BC). This level corresponds to a level of tavertine block pavement. 9 fragments of vessels were selected from level VIa (5487-4960 cal BC), which responds to a level of occupation with associated material. 15 fragments of vessels come from level VII (5487-4960 cal BC). This level corresponds to the collapse of wooden structures and Level VIII to chalk with the wooden posts fixed, where 2 samples were selected. The only samples relating to a restored form are Level VIa, 2996, and Level VII, 3525, which correspond to the same vessel, with an ovoid shape and a closed mouth with ribbon handles and incised decoration.

2.3.2.2. Mines de Gavà (Gavà, Barcelona) (4500-3300 cal BC)

The Prehistoric Mines of Gavà are a mining centre for the production and distribution of variscita. They are located in Gavà, in the northeast of the Iberian Peninsula. For the time being, 110 mines have been documented for mining in an area of about 245 m2 with various altitudes, ranging between 33 and 94m a.s.l. Although today the coastline is more distant, probably during the Neolithic the sea would be located two kilometers from the site.

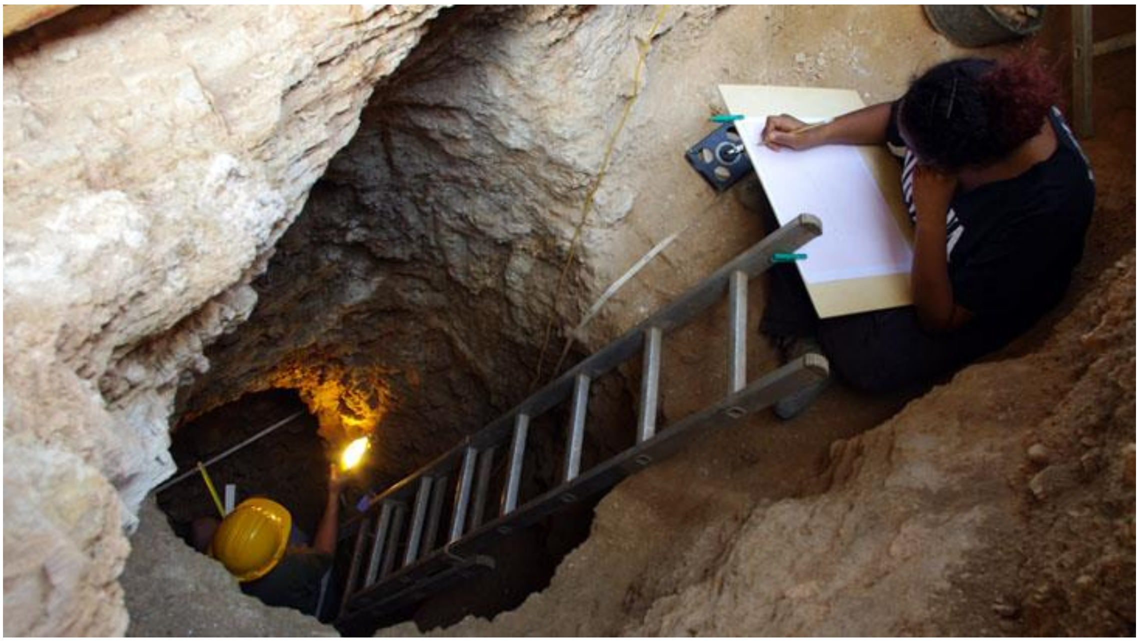


Figure 2.8. Excavation in mine 108 at Mines de Gavà site.

The dating of coal and bone remains from different mines shows that the exploitation phases of the Gavà Mines would begin in the Postcardial Neolithic and end in the Middle Neolithic (4500-3500 cal BC).

The mines present different morphologies of the mining structures. Wells with isolated galleries have been documented, such as mines 4, 9 and 70, and networks of galleries and chambers with higher density and different levels of depth (Borrel and Bosch 2009). Geological studies show that underground mining activities required a complex knowledge of exploitation strategies (Borrel and Bosch 2012).

In the mines dated from the Middle Neolithic onwards, fillings from previous mines already exhausted with shale and waste from mining work have been documented.

With respect to the knowledge of the settlements of the site, until now, all hypotheses speak of settlements located in the immediate surroundings of the mining exploitation, of one or more population groups, established in ephemeral constructions (Bosch and

Santacana 2009). Although no evidence of settlement has been documented, constructive elements related to these domestic spaces have been found, such as adobe materials (García 2009), which together with the available evidence propose a stable settlement.

2.3.2.2.1. Subsistence practices

A possible specialisation of the Neolithic communities of Gavà in mining has been considered. On the other hand, the evidence suggests other economic practices.

There is little evidence of farming during the early stages of mining. We must bear in mind that, given their specificity, remains of consumption are more present in the archaeological record than evidence of production.

The hypotheses generated for agricultural activities are based on the possible secondary use of abandoned mines as a food storage site, where carpological remains have been documented in clay containers (Bosch 1994). Another indication for these moments of the activities directed to the subsistence of the group could be found in the anthracological studies (Ros 1994; Bosch and Estrada 1994d). According to these, the configuration of the local landscape of the post-cardial mines of Gavà was constituted by weeds and bushes, and they are interpreted as the anthropic degradation of the forests for the intensification of the agricultural and/or cattle practices (Bosch and Estrada 1994d).

In the period of exploitation corresponding to the Middle Neolithic, the agricultural exploitation is intensified with the evidence of domestic species. These species are represented by cereals, among which barley has an important percentage, while the proportion of wheat grain is lower (*Triticum dicoccum* and *Triticum aestivum*), and to a lesser extent leguminous plants appear (*Vicia sp.*) (Buxó et al. 1991).

With regard to livestock, the fauna documented in mines 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 83, 84, 85 and 90 in Gavà responds to anthropogenic depositions once the mine is no longer functioning or natural, such as rabbits or amphibians. The anthropically diposited fauna characterizes the presence of the four main domestic taxa, cattle (*Bos taurus*) (45%), sheep (*Ovis aries*) and goats (*Capra hircus*) (28%), and pigs (*Sus domesticus*) (27%) (Saña 2009). Some of the documented remains of these species show traces of decarnation and thermoalterations, related to the herbidity and roasting of meat for food consumption. Some of the bones, especially bovine bones, would be used to make tools, such as miners' beaks (Estevez 1986).

Fauna remains of wild species, such as wild boar, rabbit or deer, have also been documented, although hunting is of minor importance (Bosch and Estrada 1994d).

The studies that have been carried out show a well-established livestock with a slaughter pattern towards young animals oriented to meat exploitation (Estévez 1986; Saña 2009), although dairy exploitation is not ruled out. Some cattle have a high slaughter age, which is interpreted as the use for the transport of heavy loads or activities related to agriculture.

An important number of malacological remains of salt water species have also been documented. Fish characteristic of estuary areas and sandy bottoms have been identified, such as *Sparus aurata* and *Myliobatis aquila*) (Estevez 1989), the presence of a large number of malacological species, including *Glycymeris sp.*, and remains of phocids (Bosch et al. 1999). These evidences indicate the practice of collecting marine resources from land.

2.3.2.2.2. Material culture

Some tools related to the practice of mining work have been recovered, such as axes, azuelas, picks and maces (Donoso 1998). These tools are made with exogenous corneas (Álvarez and Clop 1994, 1998).

Lithic industry has been identified, such as flint and white quartz plates and perforators, as well as quartz firing pins with stigmas of having been used to strike (Bosch 1994). In later stages, the raw material of the carved lithic industry is characterized by having an allochthonous origin, with honeyed flint from Provence, axes from the Alps, obsidian from Sardinia (Borrell, Bosch 2009; Borrell 2009).

The bone industry is not very representative, but there are punches or enmangues for axes made from animal bone (Estrada 1994b). As in the case of the bone industry, in post-cardiac mines the set of ornamental elements recovered is very scarce, but very representative. Two types of elements are found: on the one hand, in hard animal matter, shells; and on the other, stone beads: slates and variscita (Estrada 1994a). Ornamental elements were generally made of variscite, although they also appear on red coral, shells or tusks of wild boar (Borrell and Estrada 2009a).

2.3.2.2.3. Pottery remains

The ceramic remains recovered from the different mines generally appear as fillings of exhausted mines, although vessels associated with the trousseau of some of the graves identified inside the mines have also been documented. Most of the vessels are fragmented, with some exceptions. From the reconstruction of the profiles of the different ceramic studies carried out in some of the mines studied, vases were identified with gripping and suspension elements, with incised and printed decorations (Villalba et al. 1986, Bosch and Estrada 1993; Bosch, Estrada and Segura 1994). However, from the Middle Neolithic onwards we find little decorated vases with a polished surface treatment. In this way, we find that the ceramic ensemble of Mines de Gavà is related to other ensembles of the Middle Neolithic in Catalonia, and an evolution of the types of the facies Montboló and Molinot. The typology and dimensions of these ceramics are very variable. With high quality finishes, the colours tend to be dark as a result of firing in a reducing environment without many decorations. Simple profiles predominate, with globular and hemispherical trends, open and closed, as well as more or less marked fairings (Calvo 2019).

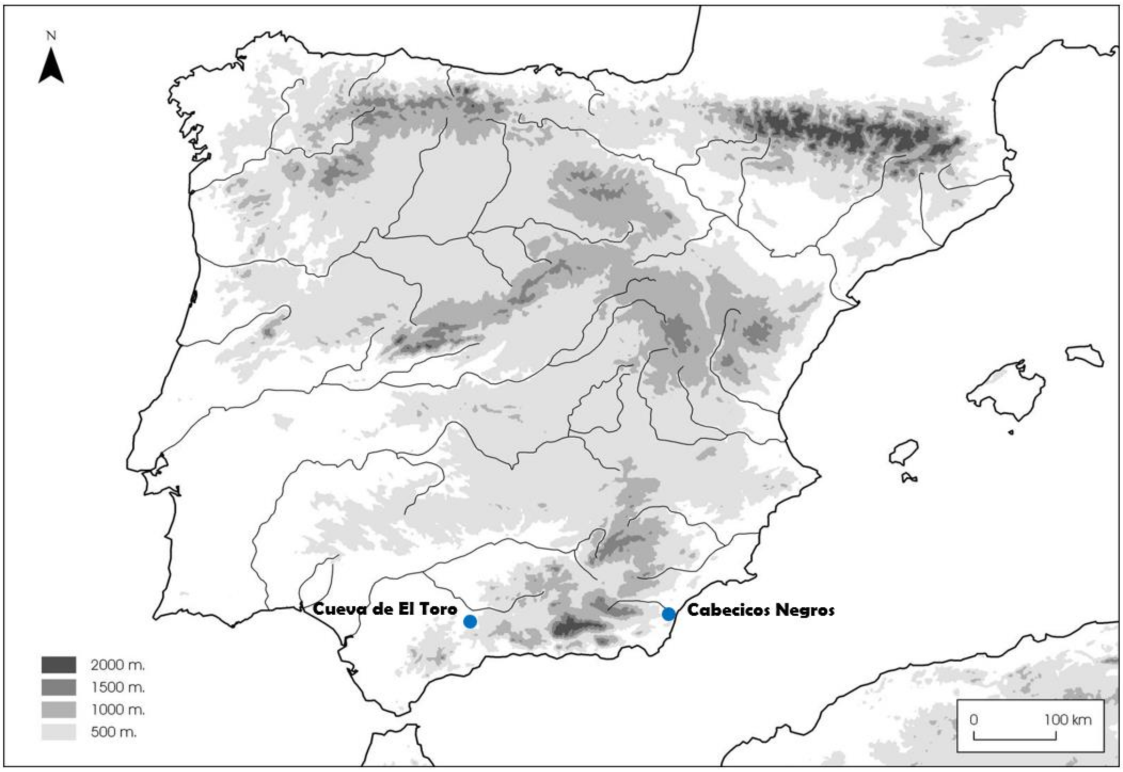
2.3.2.2.4. Samples selected

28 vessels have been selected from mines 6, 7, 8, 41, 68, 83, 84, 85, 90 and 108. The composition of most of the mines are wells dug with the intention of extracting variscita, in some of them appear small cavities for lighting (mine 84). In some cases, the mines were reused as graves once the mineral vein was exhausted (mine 83).

The sampled ceramic set presents practically all the reconstructed forms, among which are represented small bowls of different morphologies, plates, pots and large containers.

2.3.3. Analysed sites from south of the Iberian Peninsula

Coming from the southern area of the Iberian Peninsula, two sites have been selected, dated between 5500 and 3900 cal BC. There are two different types of settlement, Cueva de El Toro is a cave and Cabecicos Negros is an open-air site. All of them form a set of 79 samples. Each of them is described below.



SITE	TYPE	REF	LEVEL	SAMPLE	DATE BP	BIBLIOGRAPHY
Cueva del Toro	Cave	GrN-15443	IV	Charcoal	6320±70	Martín-Socas et al., 2004
Cueva del Toro	Cave	Beta-174308	IV	Charcoal	6160±40	Martín-Socas et al., 2004
Cueva del Toro	Cave	GrN-15444	IV	Charcoal	6030±70	Martín-Socas et al., 2004
Cueva del Toro	Cave	Beta-347630	IV	Seed	6150±30	Camalich & Martín 2013
Cueva del Toro	Cave	Beta-347627	IV	Seed	6110±30	Camalich & Martín 2013
Cabecicos Negr	Open-air	Beta-336255	I	Shell	7300±50	Camalich & Martín 2013
Cabecicos Negr	Open-air	Beta-336258	I	Shell	6550±50	Camalich & Martín 2013
Cabecicos Negr	Open-air	Beta-341132	I	Shell	6490±50	Camalich & Martín 2013
Cabecicos Negr	Open-air	Beta-341131	I	Shell	6360±50	Camalich & Martín 2013

Figure 2.9. Location and radiocarbon datations of Cabecicos Negros and Cueva de El Toro.

2.3.3.1. Cabecicos Negros (Vera, Almería) (5500-4900 cal BC)

Cabecicos Negros (Vera, Almería) is an open-air Neolithic settlement located in the southeast of the peninsula, facing the sea and close to the mouth of the river Antas. This site forms a structured whole with the site of El Pajarraco. Both spaces are currently divided by the layout of a road, which distorts the common cultural identity they possessed in earlier times. This site was the object of a first archaeological intervention in 1991, where a collapse of stones was identified, mixed with remains of mud, some of which had vegetal imprints in Corte 14 and Corte 18 (Goñi et al. 2001). In Corte 14, evidence was documented of stones and mud on the hilltop, which is arranged through a series of natural steps. The distribution of the remains and the special topography were interpreted as the use of the hill for the conditioning of structures as shelters, built on the basis of walls of stones and mud of scarce size and vegetal roof. These evidences are related to the existence of "cabin bottoms" associated with an occupation dated between 5220-4760 cal BC, constructions without great investment of work that derive from a model of settlement defined as semi-nomadic (Cámalich et al. 1999).

Corte 15 was excavated in the southernmost hill of the site and has a surface area of 100m2. The stratigraphic sequence defined four phases of occupation corresponding to different periods. Phase I, corresponding to the Neolithic period, is represented by a stone filling inside which a set of materials can be observed, such as printed ceramics and stone industry, with the presence of perforators on laminites.

2.3.3.1.1. Subsistence practices

Faunistic and botanical remains are scarce at the site, although remains of domestic species such as bovids, ovicaprids and suids, together with the occasional presence of cervids and leporidae, stand out. The presence of malacofauna, preceded by Cerastoderma sp. and Glycimeris sp., explains the exploitation of the estuarine zones by the human groups that inhabited the site. Biometric data, on the other hand, suggest that this shellfish activity should have had little importance in the diet, but in relation to bromatological activities, given the large number of ornamental elements made from shells of molluscs (García. A., pers. comm.).

2.3.3.1.2. Archaeological material

Among the materials found in the site, the elements of personal adornment stand out, such as bracelets made of slate, marble or discoidal beads elaborated on shells; lithic industry carved on flint, with a great presence of laminites and perforators (Rodríguez 1999).

2.3.3.1.3. Pottery remains

The ceramic sets, decorated with a wide variety of techniques, including the presence of almagra fillings and the presence of cardial pottery. In Corte 15, ceramics printed from dots, with matrices or combined with grooved lines were documented. Other containers appear plain or with a ribbon handle. As regards the shape and size of these ceramic vessels, the analysis is limited due to the high rate of fragmentation and erosion of the materials. Although where possible it has resulted in vessels with closed edges and necks indicated with varying dimensions (Goñi et al. 2001).

2.3.3.1.4. Analysed samples

For the chemical analysis of the residues in Cabecicos Negros (Vera, Almería), samples of 29 ceramic potsherds have been selected. Due to the high degree of fragmentation of the ceramic set of Cabecicos Negros, it has not been possible to restore the shape of the vessels analysed. From the 29 fragments selected, samples were taken from 15 rims from Corte 15 and six base fragments together with eight rims from Corte 18.

2.3.3.2. Cueva de El Toro (El Torcal, Málaga) (5300-3900 cal BC)

Cueva de El Toro is located in the south of the Iberian Peninsula, specifically in the karst range of Sierra de El Torcal (Málaga, Spain) at 1190 meters above sea level. The occupation of the internal space is carried out on large limestone blocks detached from the upper part of the cave (Martín-Socas et al. 2004). However, during the first quarter of the 4th millennium BC there was a structural change (Martín-Socas et al. 2018; Égüez et al. 2016), that blocked the entrance until then and configuring a new access and a pit of 17 metres depth. This event coincides with a change in the intensity of the occupation of the cavity.

The Torcal de Antequera mountain range looks like an elongated massif and its morphology responds to a karstic modelling process. Due to the strong karstification of the terrain, the edaphology of El Torcal is included in the forest limestone-brown variety. In this way, in the areas where the clay is deposited, a flora of shrub types grows, represented by hawthorn, roses and brambles, arboreal, dominated by oaks, and herbaceous. In this region the climate is Mediterranean mountain, with an annual rainfall of 1000mm, but with marked features of aridity due to filtration by the rock. Temperatures are low in winter and moderate in summer, although frosts are rare.

The stratigraphy of the cave of El Toro is structured in four phases of occupation with a density of 2.40m. The oldest phase corresponds to phase IV (Early Neolithic, 5280 - 4780 cal BC), is located at the base of the cave and is formed by a large number of collapse slabs. Taking into account the characteristics of the different sedimentary units identified, and the results derived from the functional analysis of the material sets, exploitation practices aimed at generating meat surpluses have been interpreted. The evidence of successive levels of coal and ash associated with large pits of small depth has been interpreted with the practice of smoking through the use of ways of slow combustion and with great calorific potential. With the exception of large pits, no structures associated with the bedroom of a space are documented, except for a small home of final moments of occupation of this phase.

The next phase is organized in two periods, the subphase IIIB (Late Neolithic, 4250-3950 cal BC) and the subphase IIIA (3950-3500 cal BC). Soil regularization is documented by the application of clay soil from outside the cave. The differentiation of the phases is given by a differentiated use of the cave and in the organization of the space. Sub-phase IIIb begins with a reconditioning of the living space and six combustion structures are articulated on top of it at the entrance to the cave. The development of this subphase is determined by a use of the space further inland for the stabling of livestock. In subphase IIIA, there is a new conditioning of the cave and an expansion of the occupation space towards the interior of the cave, characterized by the presence of 24 combustion structures.

After a hiatus of occupation, the cave is inhabited again (phase II) between 2050 and 1100 cal BC.

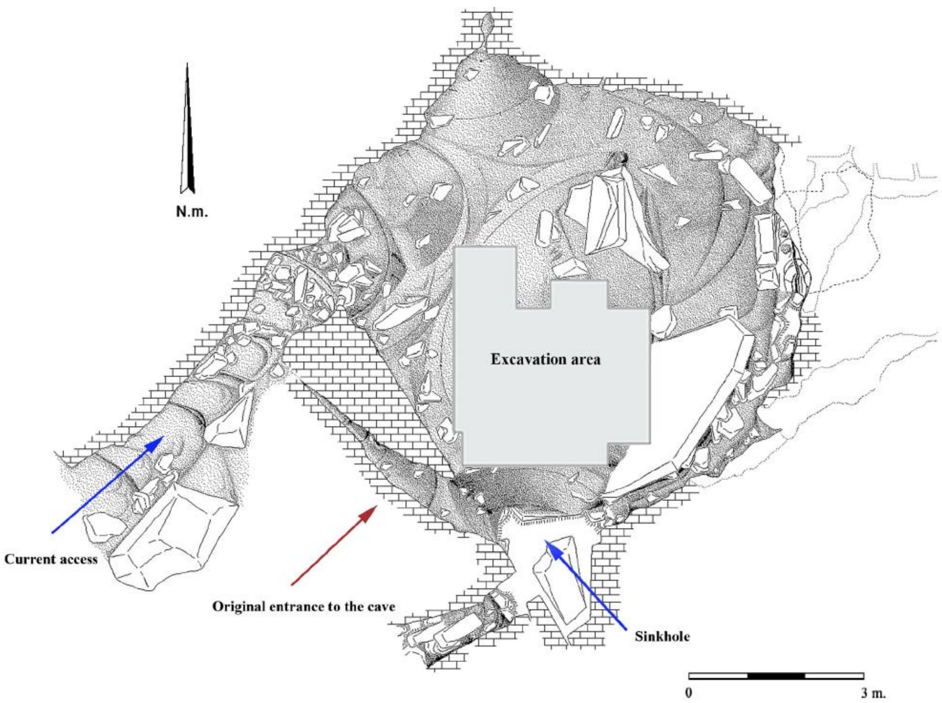


Figure 2.10. Plan drawing of Cueva de El Toro galleries showing the location of the excavated area.

2.3.3.2.1. Subsistence practices

The archaeological evidences of Cueva de El Toro show subsistence strategies with some differences between phase IV and sub-phase IIIB. In general, the pattern of representation of the fauna is similar between the different phases, with the preponderance of ovicaprins (62% and 71%), followed of suids (17% and 5%) and lagomorphs (14% and 10%) (Watson et al. 2004) among other species. In the Early Neolithic the percentage of representation of the domestic fauna is higher than 75%, while in the most recent period it reaches values above 85%. It should be noted that there is a prominent presence of wild boar during phase IV. Spatial and micromorphological analyses reveal that phase IV is characterised by its seasonality and its pastoral orientation, while, sub-phase IIIB, is characterised for the duality in the use of the space, the result of the cohabitation of people and animals (Martín-Socas et al. 2004, Camalich-Massieu and Martín-Socas 2013, Égüez et al. 2016, Martín-Socas et al. 2018). Based on the micromorphological analysis of the archaeological sequence it has been possible to determine a separation between the space used for livestock stabling and that linked to domestic activities.

In summary, livestock subsistence practices during phase IV of Cueva de El Toro would be focused on goat management, followed by pigs and cattle. The contribution of hunting to the meat diet would be small and probably made up of lagomorphs. The type of mortality pattern suggests that goat management would focus on meat production at this stage, slaughtering the animals by three years of age. On the other hand, in phase III the cave would change its functionality and the presence of animals inside the cave would gain presence. The mortality of sheeps and goats in this phase suggests a dairy productivity, as the slaughter age is documented at an earlier age than in phase IV (Martín-Socas et al. 2004: 256-257).

In Cueva de El Toro, the organic remains show an excellent state of preservation. The number of vegetal remains of phase IV is low, highlighting the presence of wild vegetables such as the acorn. Meanwhile, in sub-phase IIIB -in which several hearths or combustion structures were documented-, there is an exponential increase in the number of domestic species across the acorn. The most represented are Vicia faba minor, Tricicum aestivum/durum and Hordeum vulgare nudum (Buxó 2004). In addition, the anthracological analysis of the recovered charcoals shows that Quercus ilex-coccifera and Phillyrea angustifolia were the most commonly used woods in the hearths (Rodríguez-Oliva 2004), without the presence of pine.

2.3.3.2.2. Archaeological materials

On the other hand, functional analysis of flint tools indicates a predominance of butchery and hide treatment activities, with little presence of pieces used in harvesting plants/cereals during the Ancient Neolithic. While the Late Neolithic almost disappear the tools of butchery and those related to harvesting plants/cereals are still of little importance (Rodríguez-Rodríguez et al. 1996, 2013).

2.3.3.2.3. Pottery remains

The ceramic production from the Cueva de El Toro phase IV is characterised by a high quality, with very fine degreaser, cohesive paste and a burnished or spatulated treatment of the surfaces. Approximately 82% of the vessels have decorations on the external surface, either by using the almagra or printed decorations, incised, engraved or with plastic decoration. On a morphological level, its formal development is characterised by containers with incoming walls, of simple or compound forms, with the presence of necks, and with cylindrical or troncoconic tendencies. The simple dominant forms are spherical, ellipsoidal and ovoid. The volumetry of the ensemble is classified into two groups, medium containers with capacities between 1 and 2 litres, and large containers around four and six litres, which have double ribbon handles.

Phase III presents a large volume of ceramic materials, characterised by a decrease in the quality of manufacture, a very reduced presence of decorated containers and a significant increase in the general volumetry of the containers. Vessels with low hulls and an intense spatulate are distinguished and as the occupation progresses, the decorations are reduced. In fact, there is a clear differentiation between the decorated vessels, which have high walls, entrances or with necks, and the undecorated ones, with a domain of open ovoid and ellipsoidal shapes.

2.3.3.2.4. Analysed samples

The study of determination of the organic residues in Cueva de El Toro is based on the analysis of 50 samples of Neolithic pottery, 26 of which correspond to vessels of phase IV (5280 - 4780 cal BC) and 24 to vessels of sub-phase IIIB (4250-3950 cal BC).

The 26 samples of phase IV include vessels such as:

- Vessels with ovoid shapes, incoming walls and an estimated capacity between one and three litres. From this group samples of vessels decorated with almagra and impressions filled by almagra (CTM12), in one case impressions on rim are provided (CTM35), and incised marks (CTM45), some of them filled by almagra (CTM21 and CTM48) were selected. Likewise, grooved vessels filled by almagra (CTM41 and CTM43), with double handle grip elements and cordon applied with impressions (CTM14), or non-decorated (CTM03) were selected.

- Vessels with ellipsoidal shapes, straight walls and an estimated capacity of between one and three litres. From this group samples incised vessels (CTM15), decorated with boquique filled by almagra (CTM42), incised and impressed vessels with ribbon handle (CTM20), vessels with lumps and slips (CTM33) and with printed laces (CTM24 and CTM32) were selected. Further, an ellipsoidal vessel with an estimated capacity of 0,2 l. with two tubular handles and a vertical punch-hole was selected (CTM10).

- Vessels with hemispherical shapes, straight walls and an estimated capacity between one and three litres. One of them is non-decorated (CTM07), and the other one has an estimated capacity upper than four litres, impressed decoration on the rim and a ribbon handle with appendix and horizontal punch-hole (CTM16).

- Vessels with ovoid shapes with narrow neck, and an estimated capacity upper than four litres. A non-decorated with divergent neck vessel (CTM39) and a convergent neck vessel with almagra surface and ribbon handle with double punch-hole (CTM11) were selected.

- Vessels with ovoid or ellipsoidal shapes, with convergent neck and an estimated capacity upper than six litres. A printed lace vessel (CTM44) and an incised and impressed vessel with tubular handle (CTM29) were selected.

- Lastly, some particular vessels were selected. From among them, two vessels have a spout (CTM19 and CTM51) and two fragments present incised decorations (CTM37) and barbotina (CTM30).

The 24 samples from sub-phase IIIB correspond to:

- Vessels with ellipsoidal shapes and straight or convergent rim, and an estimated capacity of between one and three litres (CTM18, CTM23, CTM26, CTM34, CTM36 and CTM38) were selected. One of them has an impressed decoration on the rim (CTM36).

- Vessels with ovoid shapes with divergent rim and convergent neck, and an estimated capacity of between one and three litres. From this group, a vessel with handle grip (CTM17), a convergent neck and ribbon handle with horizontal punch-hole (CTM04) and two vessels without handles (CTM02 and CTM50) were selected.

- Vessels with ellipsoidal or ovoid shapes, straight divergent walls, straight rim or slightly divergent, and an estimated capacity of between three and five litres. Two vessels with handles were selected (CTM31 and CTM05).

- Vessels with hemispherical shapes and straight and divergent rims. A one litre capacity vessel with a ribbon handle with horizontal punch-hole (CTM27) and two vessels with an estimated capacity between four and six litres, with double nippled shapes and vertical punch-hole (CTM08) and a handle horizontal punch-hole (CTM09) were selected.

- Vessels with ellipsoidal or ovoid shapes, straight neck and slightly divergent, and an estimated capacity between twelve and fourteen litres. Non-decorated vessels (CTM22 and CTM46) and a vessel with a handle with horizontal punch-hole (CTM01) were selected.

- Lastly, some particular vessels were selected. From among them, a fragment with a U-shaped cordon (CTM49), an incised vessel (CTM13) and painted decoration fragment (CTM47) were selected.

As a balance, the sample is varied. We observe sites that occupy a large geographical area and span different cultural horizons, from cardiac to postcardial/medium Neolithic (5400-3900 cal BC). In some cases, it has been possible to sample reconstructed vessels, such as in Mines de Gavà, Carrer Llinàs 28, Feixa del Moro, Camp del Colomer and Cueva de El Toro, although in other cases only fragments were sampled that could have identified the part of the vessel to which they correspond (bases and edges). As we have commented in the selection criteria, this sampling will also allow us to evaluate the preservation fat and the taffonomic aspects of each of the sites, given that each sample is ascribed to a dated occupancy level of each of the sites.

Chapter III. Methods

The analysis of organic residues in pottery sherds has emerged in the last 30 years as a tool to evaluate the occurrence of remains of food and non-food products in the original pottery containers. Renfrew already proposed to go beyond typology and ceramic dating to enhance the knowledge of the uses of containers more than three decades ago (Renfrew, 1977), but it has not been until the last 20 years that this field has undergone sufficient technical developments (Heron & Evershed, 1992). The inclusion of techniques specific to the field of organic chemistry has required to overcome, chiefly, technological and interpretation challenges derived from the effects of diagenesis of organic matter in the environment (Evershed et al. 1992, Eglinton and Logan 1991). The evolution of these studies and the increase in the number of studies has allowed the documentation of a wide range of organic substances in ceramic vessels (Regert, 2011, Evershed, 2008, 1999, 2001), which has required the multidisciplinary integration of interpretations.

Research into organic residue analysis has primarily focused on the identification of biomarkers and, since the extracted residues are essentially decayed products of the original lipid compounds. A range of studies have also been carried out to investigate the degradation patterns of various products extracted from archaeological artefacts (Spiteri-Debono, 2012), as well as better extractive methods to optimize the results (Correa-Ascensio et al. 2014). In this way, it has also been possible to know the functional use of archaeological containers and to propose different processing and consumption practices based on their morphological characteristics (Rice, 2015, Vieugué et al. 2016, Fanti et al. 2018).

This chapter reviews the strengths and limitations of organic residue analysis and describe the extraction methods applied in this work for the study of the products contained into the sherds from sites in the Iberian Peninsula. It has also been considered necessary to carry out an experimental study of regional animal fats potentially consumed during the Neolithic, which allows to refine the interpretation of the results. Finally, a statistical mixing model analysis proposal is described to understand the mixture of fats resulting from the food preparation or the reuse of vessels.

3.1 Analytical assumptions

3.1.1. Preliminary considerations about preservation

The concentration of lipid residue absorbed within ceramic vessels and their preservation over archaeological timescales depends on several factors. These include the intensity and mode of use, the permeability of the vessel, the initial deposition, and alterations to the lipid composition and concentration due to diagenetic processes during burial (Charters *et al.* 1993; Copley *et al.* 2005b). Experimental investigations on lipid yields extracted from pottery have shown that the estimated lipid capacity of a potsherd is around 10 mg g^{-1} of lipid, of which only around 100 µg g^{-1} of lipid will survive in archaeological contexts (Evershed, 2008a). Hence, although lipids may not decompose completely (Sherriff *et al.* 1995:109), they are still susceptible to diagenetic alteration prior to and during burial.

Biomolecular preservation is dependent on molecular structure, the depositional environment and diagenetic history (Eglinton and Logan, 1991). Lipid preservation is further enhanced by their incorporation into the ceramic matrix (Evershed, 1993) and charring (Oudemans and Boon, 1991; Oudemans and Erhardt, 1996). The physico-chemical properties of the burial environment will also influence the rate and extent of degradation (Evershed, 1993; Aillaud, 2001), but only during the first year of burial, after that the degradation does not appear to advance significantly (Stacey, 2009, Dudd et al. 1998). The local geology influences the pH of soil, soil aeration, degree of light exposure, and water percolation, while the local climate determines the burial temperature and humidity. These factors will in turn influence the level of microbial activity in the burial environment, which will influence lipid preservation (Eerkens 2007; Eglinton and Logan, 1991; Evershed *et al.* 1991, 1992; Shimoyama *et al.* 1995; Oudemans and Erhardt, 1996; Aillaud, 2001).

Biomolecular analyses of contents have shown that in cold climate contexts organic matter is better preserved, as low temperatures limit hydrolysis and microbial activity (Aillaud, 2001, p. 145; Eglinton *et al.* 1991). The same happens in arid contexts, where due to low humidity, hydrolysis reactions and bacterial activity are limited (Copley *et al.* 2005e, Eerkens 2005, Eglinton *et al.* 1991, Evershed, 2008, 1997). In addition, anaerobic contexts present a good conservation of organic matter due to the low availability of oxygen (Den Dooren De Jong et al. 1961; Eglinton et al. 1991). Although it does not prevent the proliferation of anaerobic bacteria, which cause hydrolysis and β-oxidation (Killops & Killops, 2009, Regert et al. 1999, Berstan et al. 2004, Evershed et al. 1991). Finally, cave sites appear to offer a much more favourable context for the conservation of organic matter, regardless of the type of substrate and soil (Decavallas, 2011). Arguably, cave site is less exposed to seasonal climatic variations and sherds in these contexts are arguably less prone to undergo leaching processes (Debono Spiteri, 2012).

It has thus been shown that burial in a warm, dry climate that alternates with periods of heavy rainfall, such as the Mediterranean, is very unfavourable for the conservation of organic matter, as it favours both hydrolysis and oxidation mechanisms (Cramp, 2008; Evershed, 2008a). In addition, in the Mediterranean basin soil pH range is between 6 and 7 (Drieu, 2017), being a neutral pH soil that favours degradation mechanisms (Reber & Evershed, 2004b, Copley et al. 2005b; Gregg & Slater, 2010; Matlova et al. 2017 ; Smyth & Evershed, 2015, DeLaune et al. 1981).

3.1.2. Biomarkers in archaeology

3.1.2.1. Fatty acids

Fatty acids from natural substances generally consist of an even number of carbon atoms (between 14 and 22) arranged linearly with a group of carboxylic acid at the end of the hydrogen-carbon chain (Christie 1989).

Fatty acids are the most common compounds in archaeological ceramics. Palmitic acid ($C_{16:0}$) and stearic acid ($C_{18:0}$) are the most common, followed by lauric acid ($C_{12:0}$) and myristic acid ($C_{14:0}$). These acids may be the result of the degradation of the triaciglicerides found in animal fats, although they may also be the result of microbial endogenous or exogenous postdepositional contamination.

Unsaturated fatty acids are also frequentely detected: mono- and di-unsaturated acids with 18 carbon atoms ($C_{18:1}$ and $C_{18:2}$), as well as to a lesser extent $C_{16:1}$. $C_{16:1}$ appears in large quantities when it comes from both marine and freshwater aquatic resources (Aillaud 2001, Malainey et al. 1999b).

Odd-named fatty acids of carbon atoms ($C_{15:0}$ y $C_{17:0}$) appear in archaeological vessels as linear or branched isomers. These compounds are present in ruminant fats (Dudd et al. 1999).

The occurrence of long chain fatty acids (from $C_{20:0}$ to $C_{30:0}$) can be attributed to inputs of lipids with an aquatic origin (Craig et al. 2011, 2013, Heron et al. 2015) or waxes of higher plant or honey from bees (Charters et al. 1995, Evershed et al. 1997, Regert et al. 2001a). Long chain unsaturated fatty acids, such as $C_{20:1}$ can also be related to fats of aquatic origin (Craig et al. 2011, 2013, Heron et al. 2015).

3.1.2.2. TriacylglYcerols

Triacylglicerols are the main components of natural fats and oils. They are composed of a glycerol base, which includes each hydroxy group is linked to a fatty acid through an ester connection (Christie 1989). The position of each of the fatty acids that make up the TAG, as well as their nature (chain length, number and position of insaturations) result from the mode of synthesis of TAGs by enzymatic mechanism within living beings and are highly variable according to the natural origin of the fat. In order to identify TAGs and trace the natural origin of a fat, it is necessary to know the composition of the fatty acids, but also their distribution in the glycerin skeleton.

The distribution and concentration of triglycerides allows us to get closer to the origin of the fat. Thus, classical ruminant fat profiles are distributed between T_{46} and T_{54}, with T_{50} and T_{52} predominant (Dudd et al. 1999, Mukherjee et al. 2007, Regert et al. 1999). Dairy products are characterised by a wider distribution, between T_{40} and T_{54}, with special concentrations of T_{50} and T_{52} (Dudd and Evershed 1998, Mirabaud et al. 2007). Finally, non-ruminant fat profiles are distributed between T_{46} and T_{54}, with T_{52} being the most predominant (Dudd et al. 1999).

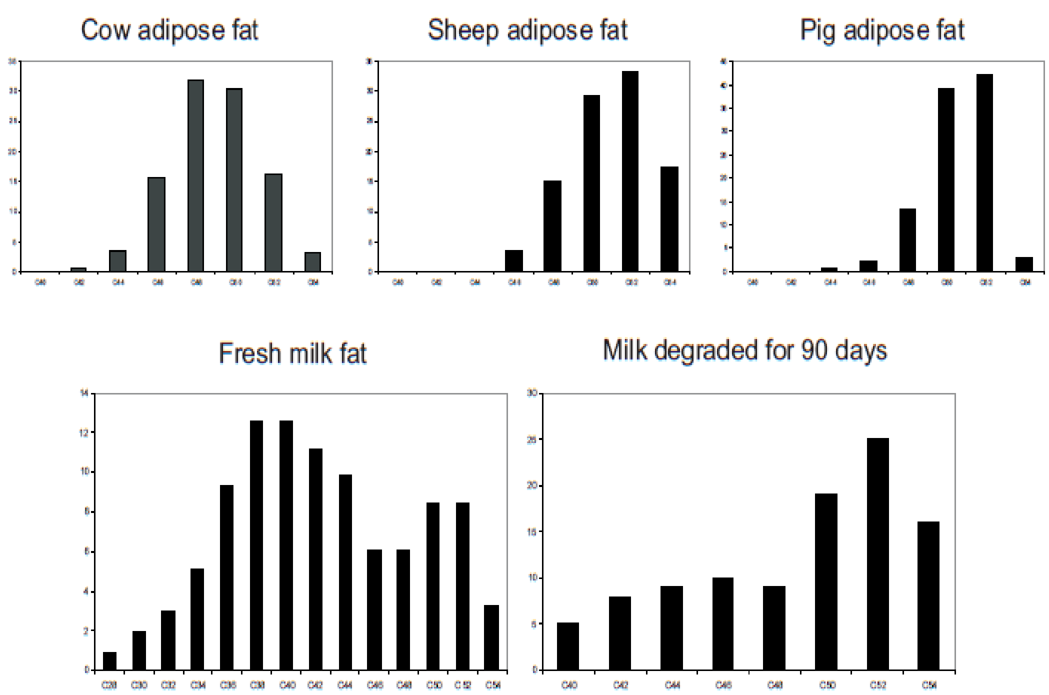


Figure 3.1. Histograms of characteristic triacylglycerols distributions of various reference contemporary animal fats (taken from Mukjerjee et al. 2007).

However, the identification of unsaturated TAGs, often dominated by T_{54}, corresponds to a classical distribution of vegetable oils (Copley et al. 2005e, Drieu 2017).

3.1.2.3. Alkanes

Alkanes are molecular compounds characterized by a saturated hydrogen-carbon chain and the absence of a functional group (Killops and Killops 2009: 30).

Alkane profiles are often presented in analyses of archaeological ceramic vessels and may have different origins. Odd alkane profiles dominated by C_{27} are characteristic of beeswax (Charters et al. 1995, Evershed et al. 1997c, Regert et al. 2001a), whereas when the concentration falls on C_{29} and C_{31} alkanes, they are interpreted as epicuticular waxes (Eglinton and Hamilton 1967). The thermal or natural degradation of beeswax can volatilize the lighter alkanes and present profiles dominated by C_{29}, so its presence must be interpreted with caution (Regert et al. 2001a).

On the contrary, the presence of alkanes with carbon atom pair name chains is related to a petrogenic origin (Killops and Killops 2009).

3.1.2.4. Alcohols

Linear alcohols, sometimes called fatty alcohols, have a hydroxy group at the end of their hydrogen-carbon chain. Like fatty acids, they rarely exist in free form, but rather in combination with other molecules.

Long-chain linear alcohols called carbon atom pairs ($C_{20}OH$-$C_{32}OH$) often appear in archaeological ceramics, dominated by $C_{26}OH$ or $C_{28}OH$, which are identified in vegetal waxes (Egliton and Hamilton 1967), or by $C_{30}OH$ and $C_{32}OH$, characteristic of degraded beewaxes (Regert et al. 2001a, Evershed et al. 1997c, Charters et al. 1995).

3.1.2.5. Wax sters

Wax esters consist of fatty acids (mainly linear and saturated but sometimes branched or unsaturated), bound to long chain linear alcohols by an ester bond.

Wax esters are identified in archaeological materials with a distribution between W_{40} and W_{52}, which are interpreted as beeswax (Charters et al. 1995, Evershed et al. 1997c, Garnier et al. 2002, Heron et al. 1994, Regert et al. 2001a). The wax esters correspond to acid fragments from palmitic acid, detected by *m/z* 257 ions (Regert et al. 2001a). The presence of wax esters of different acids is interpreted as plant esters (Ribechini et al. 2008).

3.1.2.6. Sterols

Sterols are molecules formed by steranes and functional groups, such as cholesterol, formed by 27 carbon atoms, which is found in much of animal fat (Evershed et al. 1993, Heron and Evershed 1993). Classic plant sterols, called phytosterols, are composed of 29 carbon atoms, such as stigmasterol, ß-sitosterol, campesterol (Evershed 1993).

3.1.2.7. Terpenes

Terpenes are compounds derived from isoprene, a hydrocarbon of 5 carbon atoms. Terpenes originate from the enzymatic polymerization of two or more isoprene units, so most terpenes have polycyclic structures, which differ from each other not only in functional group but also in their basic carbon skeleton.

The diterpenes commonly identified in archaeological samples come from conifer resin, such as pimaric acid, isopimaric acid and abietic acid. The latter is characteristic of pine resins. All of them degrade and oxidize easily, by exposure or thermal alteration (Colombini et al. 2005b, Regert and Rolando 2002).

The presence of triterpenes, formed by 30 carbons, is usually found in the form of lupeol, betulin and hopans. These are the evidence of plant exudates, such as birch bark (Heron 1998, Binder et al. 1990, Rageot 2015, Regert 2004).

3.1.2.8. Exogenous lipids

Since many of the compounds identified through organic residue analysis are of a common nature, the presence of lipids that do not come from anthropic activities should be taken into account so as not to bias archaeological information.

These contaminants can have two sources of origin: anthropic or taphonomic. Contaminants of anthropogenic origin, i.e. the result of the manipulation of the containers by archaeologists, can have different sources. The presence of squalene and cholesterol, characteristic molecules of fat secreted by human epithelial cells, can be transferred during the manipulation of ceramic vessels. Squalene has a polyunsaturated structure that degrades easily and is not usually preserved in archaeological contexts. In short, when squalene is detected in the analysed sample, it is automatically assumed that the detected cholesterol is not archaeological. Another modern source of anthropic contaminants are phthalate plasticisers, which are easily introduced by contact with plastic bags (Pollard et al. 2007, Stacey, 2009). To avoid this, the use of nitrile gloves during the manipulation of the vessels, as well as the storage of the ceramics in aluminium foil to avoid the migration of compounds such as phthalates, can optimize the analysis of organic residues.

Taphonomic or environmental contaminants are more difficult to control (Condamin et al. 1976). Although the migration of lipids from the sediment is almost insignificant (Rottländer, 1990, Heron et al. 1991), some samples show evenly distributed alkanes of carbon atoms indicating their petrogenic origin (Steele et al. 2008).

Due to underlying problems with contamination, Evershed (2008) suggested that the minimum amount of total lipid extract (TLE) recovered, which can be used for a reliable identification, should not be less than 5µg of lipid per gram of sherd.

3.2. Applied methods

3.2.1. Archaeological samples

3.2.1.1. Sample preparation

The main objective of the analysis of organic residues is the characterization of lipids eventually preserved in the ceramic matrix and which have survived degradation. This technique is destructive for the archaeological material, since the ground of pottery is extracted.

As described in Chapter 2, the samples were taken from the lip or base of the containers, since the edge is more susceptible to contamination (Regert 1999). The samples taken are representative of the diversity of shapes of each of the ceramic sets studied, which implies a previous ceramic analysis.

In order to avoid modern contamination, sample preparation was done in the cleanest environment posible: using nitrile gloves, safety vessels and samples were stored in aluminium foil in a cold environment.

In this work, 200 samples from 9 sites in the Iberian Peninsula have been analysed: Coro Trasito, Cova del Sardo, Camp del Colomer, Carrer Llinàs 28, Feixa del Moro, La Draga, Mines de Gavà, Cabecicos Negros and Cueva de El Toro.

3.2.1.2. Sample extraction

3.2.1.2.1. Extraction strategy

A part of the lipids is easily extractable from the use of organic solvents. However, another part of these lipids remains insoluble due to strong bonds or polymerization between the organic molecules and the ceramic wall (Aillaud 2001, Correa-Ascencio & Evershed, 2014, Craig et al. 2014, Regert et al 2001b).

This raised the need to apply new extractive methods, alternatives to the conventional extraction of diclormethane or chloroform/methanol, in order to increase the efficiency in the extraction of organic molecules from archaeological ceramics, such as extraction by hydrolysis (Craig et al. 2004), extraction by alkaline hydrolysis (Aillaud 2001, Copley et al. 2005e, Craig et al. 2004, Regert et al. 1998, 2001b) or by TMTFTH extraction (Stern et al. 2010). These complementary methods have favored the recovery of identifiable lipids in contexts where acidic soil conditions and atmospheric conditions most affect the degradation of organic compounds (Correa-Ascencio & Evershed, 2013; Goldenberg et al. 2014; Papakosta et al. 2015).

In this thesis, microwave-assisted extraction has been used (Gregg et al. 2009, Gregg and Slater 2010). The use of microwaves favours the mixing of organic solvents with ceramic powder, applying temperature and magnetic stirrers, in order to recover organic compounds in contexts where they are affected by high degradation.

Furthermore, in cases where the lipid concentration is lower than 5µg, samples are reextracted by acid extraction (Correa-Ascencio and Evershed, 2013; Goldenberg et al. 2014; Heron et al. 2015; Papakosta et al. 2015; Stern et al. 2000). Lipid extractions with methanol acid have a significant effect on some of the lipid residues found in archeological ceramics that must be kept in mind. The main residues identified are fatty acids, which are esterified in fatty acid methyl esters (FAMEs), whereas compound lipids, such as acylglycerols or wax esters, are hydrolyzed in their constituent acids - converted into methyl esters - and in alcohols (Correa-Ascencio & Evershed 2014). Other residues, such as alkanes or ketones, remain unaltered, however, derivatization with BSTFA is necessary to protect alkanes as TMS esters in total lipid extracts.

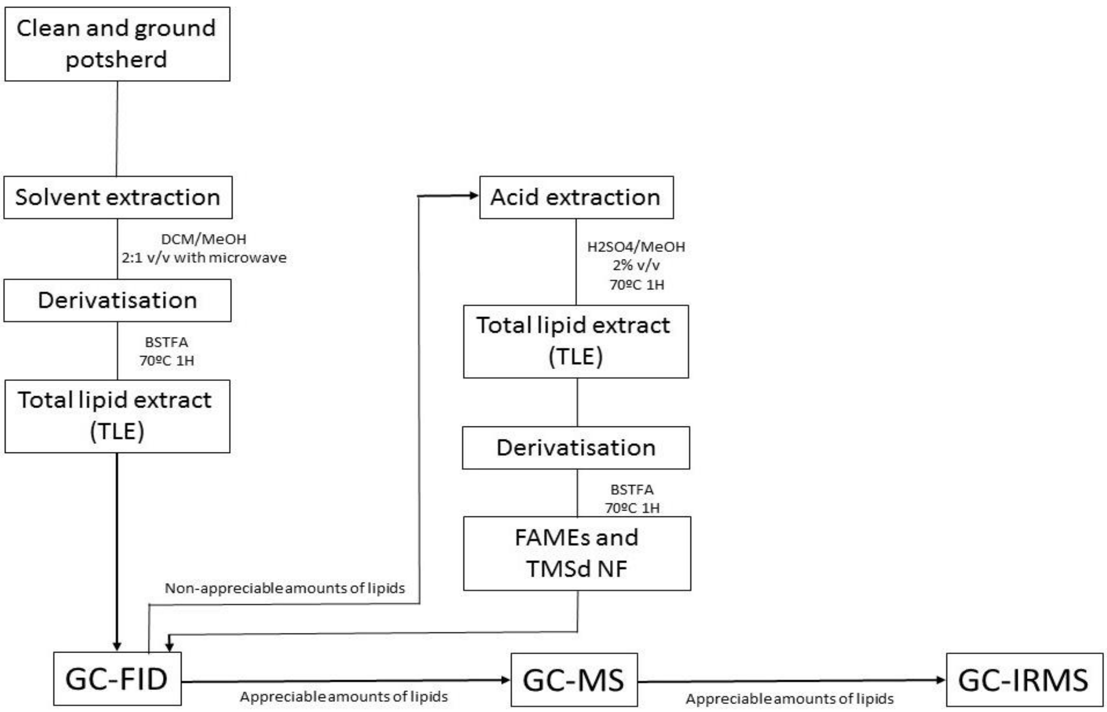


Figure 3.2. Extraction protocol followed in this thesis.

In order to increase the efficiency in the extraction of lipids and the interpretation of the results, a methodological strategy has been followed that starts from the storage of ceramic fragments in the refrigerator wrapped in aluminium foil, to avoid degradation (Figure 3.2). Once the sample is prepared, it is extracted following the microwave-assisted organic solvent extraction protocol and analysed by chromatographic gas analyses (GC-FID). In cases where the samples present interpretable organic residues, they are analysed by gas chromatography-mass spectrometry analyses (GC-MS) to identify the compounds. Finally, samples with sufficient concentrations of $C_{16:0}$ and $C_{18:0}$ are analysed for the isotopic value of $\delta^{13}C$ of both by analysis in a chromatographic gas stable isotope mass spectrometer (GC-IRMS).

In case sufficient lipid concentrations cannot be identified for interpretation in GC-FID, the ceramic powder is re-extracted by acid extraction, cases of Coro Trasito, Camp del Colomer, Carrer Llinàs 28, Feixa del Moro, Cueva de El Toro and Cabecicos Negros. The sample is re-analysed in GC-FID and, if sufficient lipid concentrations are identified, it is rederivatised with BSTFA in order to detect alcohols and alkanes and then analysed by GC-IRMS. This method is used in case extraction by organic solvents fails, since this technique involves a loss of information on the conservation and nature of the heavy compounds, such as esters or triacilglycerides.

3.2.1.2.2. Organic solvent extraction

For lipid extraction of potsherds at the ICTA-UAB (Barcelona, Spain), the surface of a sub-sample of the archaeological potsherds was cleaned with manual modelling drill (Dremel 4000) to remove exogenous lipids (Heron et al. 1993, Stacey 2009); *ca*. 2 g of ground from the inner surface was taken with the cleaned drill and then stored in a vessel tube previously muffled. The powdered samples were extracted using a mixture of 10mL dichloromethane and methanol (3:1 v/v) after addition of 50 µL of internal standard (IS) (n-tetratriacontane). In addition to the archaeological samples, four blanks were analysed, two with the same internal standard (IS) and mixture of solvents and two only with the mixture of DCM and MeOH. Lipids were extracted using a microwave (MarsX, CEM) at 70 °C for 10 min. The resulting extract was decanted, in each case, into vessel tubes and dried by exposure to a gentle nitrogen stream. Next, each sample was dried by eluting them through vessel columns filled in with anhydrous sodium sulphate. After removing the solvent, the dry extracts were redissolved in 50 µL of DCM and derivatised with 50 µL of BSTFA (N,O-bis(trimethylsilyl)trifluoroacetamide) and the mixture heated at 70 °C for 60 min. After removing the derivatisation mixture and dry it with a gentle nitrogen stream, the sample was redissolved with 50 µL of isooctane prior to their analysis by gas chromatography.

For lipid extraction at the CEPAM (Nice, France), a 2g of the inner surface was removed by manual modelling drill (Dremel 4000) and the surface was discarded to avoid exogenous lipids. The powdered samples were extracted using a mixture of 10mL DCM/MeOH (2:1 v/v). After 15 minutes sonication and a 15 minutes centrifugation at 3000 rpm/minute the supernatant was extracted. This procedure was repeated three times and the samples were dried under a gentle nitrogen stream and heated at 40ºC. Next, they were redissolved with 500 µL of DMC/MeOH and derivatised with 100 µL of BSTFA at 70ºC for 1 hour. After cooling, samples were dried under gentle nitrogen stream and redissolved with 20 µL of cyclohexane prior to their analysis by gas chromatography.

3.2.1.2.3. Acidified methanol extraction

1-2g of ground pottery is mixed with 4 mL of MeOH and 50 µL of IS and sonicated in an ultrasonic bath for 15 min. Then, 200 µL of sulphuric acid (H_2SO_4) was added, and the mixture was heated at 70 °C for 4 h. After cooling, lipids were extracted three times by adding 2 mL of hexane and vortexing. The hexane phase was removed with the help of a Pasteur pipette, taken to dryness under a nitrogen stream. Finally, samples were redissolved in isooctane prior to gas chromatographic analysis. After screening for the presence of interpretable lipids, selected samples were trimehtylsilylated and submitted to GC-MS.

3.2.2. Modern samples

3.2.2.1. Sample preparation

Ca. 5g of modern animal fats (Sample selection described in chapter IV), were taken with a scalpel from the thoracic fat of the selected animals using nitrile gloves. The sample was stored in a previously muffled vessel pot and frozen. Once the fat is frozen, it is freeze-dried for 36 hours under vacuum in order to remove all moisture and traces of H_2O. Once dry, a sample of ca. 0.1g is extracted and the rest is stored in a drying chamber together with silicon.

3.2.2.2. Sample extraction

For the extraction of free fatty acids from modern animal fat, an extraction protocol proposed by other researchers has been followed (Evershed et al. 2002, Craig et al. 2012).

3.2.2.2.1. Saponification

After adding 3ml of sodium hydroxide (NaOH) (5%, 2M in methanol), the mixture is heated in a heating plate to 70ºC and after 1 hour it is left to cool. Next, a liquid-liquid extraction is made, shaking it with the help of a vortex and discarding the hexane phase with a Pasteur pipette to eliminate contaminating compounds and traces of H_2O. To acidify the lipids, hydrochloric acid (HCl) (0,5M, 32%) is added to obtain a pH3 and then 3ml of hexane. The mixture is stirred in the vortex and transferred with a Pasteur pipette the first phase from hexane to another tube, this process is repeated three times to ensure all lipids are extracted.

A 2ml of a mixture of 40ml of MilliQ water and 500µl of sodium chloride (NaCl) is added. Then, the hexane phase is extracted. The solvent is evaporated under a soft ray of nitrogen and, once dry, the acidified fraction is derivatised with 500µl of a boron trifluoride complex with methanol (BF3-methanol) and heated in a heating plate to 75ºC for 1 hour.

Once cooled, the reaction is stopped by adding 3ml of MilliQ water and the lipids are extracted adding 3ml of hexane. The hexane transferred to vial is evaporated under nitrogen and we add 50µl of isooctane to inject the sample in the GC-IRMS to know the value $\delta^{13}C$ of the released fatty acids.

3.3. Analytical techniques

3.3.1. GC-FID

At the ICTA-UAB lab, the gas chromatographic analyses were performed on an Agilent 7820A Gas Chromatograph fitted with a Flame Ionisation Detector (FID) using a DB-5 MS column (30 m length × 0.25 mm internal diameter × 0.25 µm stationary phase thickness). The splitless injector temperature was set at 300 °C and helium was used as the carrier gas. The temperature of the flame ionisation detector (FID) was 340 °C. The oven temperature was initially held at 50 °C for 2 min, then the temperature increased at 15 °C/min to 170 °C, and finally to 320 °C at 6 °C/min, and held for a further 46 min.

At the CEPAM lab, the gas chromatographic analyses were performed on an Agilent 7890A Gas Chromatograph fitted with a Flame Ionisation Detector (FID). 1µL was injected via an on-column injector, in order to maximize the amount of material injected into the chromatograph and to avoid contamination by of septum compounds during high temperature analysis. The molecular compunds were separated into a DB-5 MS apolar capillary column (15 m length × 0.32 mm internal diameter × 0.1 µm stationary phase thickness). Hydrogen was used as the carrier gas. The temperature of the flame ionisation detector (FID) was 375 °C. The oven temperature was initially held at 50 °C for 2 min, then the temperature increased at 15 °C/min to 100 °C, and finally to 375 °C at 10 °C/min, and held for a further 46 min.

3.3.2. GC-MS

At the ICTA-UAB lab, the gas chromatography-mass spectrometry analyses were carried out using an Agilent 7890A Gas Chromatograph (GC) coupled to an Agilent 5975C Mass Spectrometer (MS). The GC was fitted with a DB-5 MS column (30 m length × 0.25 mm internal diameter × 0.25 µm stationary phase thickness). The GC injector was operated in splitless mode and helium was used as the carrier gas. The temperature of the flame ionisation detector (FID) was 320 °C. The oven temperature was initially held at 50 °C for 2 min, then the temperature increased at 15 °C/min to 170 °C, and finally to 320 °C at 6 °C/min, and held for a further 46 min. The Mass Spectrometer was run in electron impact mode and masses were acquired in full scan mode between *m/z* 50 to *m/z* 800.

At the CEPAM lab, the gas chromatography-mass spectrometry analyses were carried out using a Shimadzu GC2010PLUS Gas Chromatograph (GC) coupled to an Shimadzu QP2010ULTRA Mass Spectrometer (MS), equipped with a quadrupole analyser and an electronic impact source (EI, Electron ionisation) at 70 eV. The GC was fitted with a DB-5HT column (15 m length × 0.32 mm internal diameter × 0.1 µm stationary phase thickness). The GC injector was operated in splitless mode and helium was used as the carrier gas. The temperature of the flame ionisation detector (FID) was 280 °C. The oven temperature was initially held at 50 °C for 2 min, then the temperature increased at 15 °C/min to 100 °C, then to 240 °C at 10 °C/min, and finally to 380 °C at 20 °C/min. The Mass Spectrometer was run in electron impact mode and masses were acquired in full scan mode between *m/z* 50 to *m/z* 950.

3.3.3. GC-C-IRMS

In order to determine the compound-specific stable isotopic determination ($C_{18:0}$ and $C_{16:0}$), a third analysis is performed using a Delta V Thermo Fisher isotope ratio mass spectrometer (IRMS) hyphenated to a Trace GC Thermo Fischer Scientific gas chromatograph via a combustion interface (GC). The GC is fitted with a DB-5 MS-UI (60 m × 0.25 mm × 0.25 µm) column. The injector temperature is set at 310 °C. The oven is initially held at 80 °C for 1 min, then ramp at 30 °C/min to 120 °C, and finally increased to 320 °C at 6 °C/min and held for 21 min. Helium is used as the carrier gas. The combustion reactor is set at 940 °C.

Data is acquired and analysed using ISODAT 3.0 software. Analytical accuracy is confirmed by running fatty acid methyl ester (FAME) and alkane standards of known isotopic values prior to each batch of analysis. During each run, three pulses of carbon dioxide of known isotopic composition are fed into the ion source from the reference gas injector. These measures ensured that the instrument and combustion furnace are functioning correctly. Instrument precision is ±0.3‰.

The carbon isotope ratios are relative to the standard reference material vPDB, $\delta^{13}C$ ‰ = $[R_{sample} - R_{standard}]/R_{standard}$. The $\delta^{13}C$ values were corrected for the carbon atoms added during methylation using the following equation: $\delta^{13}C_{FA} = ((nC_{FAME}) \times \delta^{13}C_{FAME}) - \delta^{13}C_{MeOH})/n$, where $\delta^{13}C_{FA}$ is the corrected value for the fatty acid, n is the carbon chain length, nC_{FAME} is the total number of carbon atoms in the FAME ($n + 3$ for TMS ester and $n + 1$ for methyl ester), $\delta^{13}C_{FAME}$ is the value measured for the fatty acid methyl ester of carbon chain length n, and $\delta^{13}C_{MeOH}$ is the correction factor for the derivatising agent. Both correction factors were obtained by derivatising a known $\delta^{13}C_{FA}$ value with each of the derivatising agents (BSTFA and H_2SO_4-MeOH).

3.3.4. TLE quantification

To quantify the total lipid extraction of each of the peaks appearing in the GC-FID generated chromatogram, the area of each peak was calculated and quantified from the internal standard (IS) using this formula: [sample area / IS area * weight IS/weight power ceramic sample], omitting contaminant peaks such as plasticizers.

3.4. Pottery analysis

The study of vessel morphology and morphometry, together with biochemical analyses of contents, can provide information about use habits, i.e. the amount of food prepared and stored, the selection of certain types of sherds for specific purposes. The interpretation of the results points to economic and social behaviours related to food consumption and conservation (Arthur, 2002; Skibo, 1992, 2013; Vieugué et al. 2008).

In the macroscopic analysis of the ceramic fragments studied, the different morphologies of vessels (hemispherical, ovoidal, ellipsoidal) and the mouth opening (divergent, straight, incoming), as well as the metric characteristics (thickness of the walls, opening of the vessel and type of lip) and the presence of handles were taken into account. These characteristics form key elements for understanding vessel function. However, not all the assemblies had sufficient conservation of the ceramic profiles to be able to categorise this function.

Of the 9 archaeological sites studied, a total of 62 ceramic containers from Camp del Colomer, Carrer Llinàs 28, Feixa del Moro, Mines de Gavà and Cueva de El Toro were classified into 6 categories according to their presumed function. Following the proposal of functional classification of Rice (2015) and Skibo (2012) from morphological and morphometrical data.

The morphological and morphometric study of vessels was governed by the following criteria already proposed in previous similar studies (Fanti et al. 2018):

Type of profile: simple, carinated, inflected, necked.

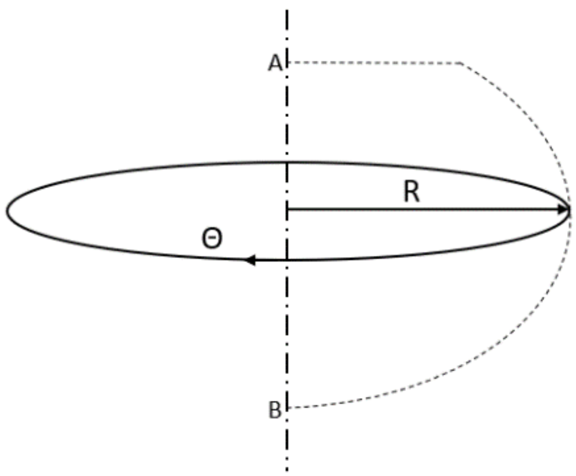
- Openess ratio (rim diameter/tangential diameter). >1: open, =1: restricted, 0.50-0.99: closed, <0.5: very closed.

- Depth (maximum diameter/height). >1.5: shallow, 0.76-1.5: medium deep, 0.51-0.75: deep, 0.25-0.5: very deep.

- Volume.

- Presence of handles.

Measuring the capacity of a container is not always possible, especially because whole archaeological vases are quite rare, and the fragments found do not always make it possible to account for them. In order to calculate the volume of vessels of different shapes and sizes, the following process was followed: initially n-measurements were taken (according to the size of the vessel) of the distance between the axis of symmetry of the vessel and its outer wall (R); then the height was also taken (distance from point A to point B). With these points the curve of the vessel profile is made; using a polynomial of degree p (p=3 to 6) approximates the function: f(r); the degree of the polynomial can vary to get a better approximation (R2≈1). Once the function f(r) is obtained, by means of the complete revolution (Θ=2π rad) of the area between the f(r) and the axis of symmetry, the value of the volume of the vessel is obtained (cylindrical coordinates were used for this calculation) (Figure 3.3).



$$V = \int_0^{2\pi} \int_a^b f(r) dr d\theta$$

Figure 3.3. Variables drawing (left) and volume calculation formula (right).

3.4. Statistical analysis

Interpreting the isotopic $\delta^{13}C$ values of the lipids preserved in ceramic vessels was made possible by the scatter plots generated with R Studio, which allow the results to be compared with the confidence ranges of the reference $\delta^{13}C$ values, generated from the calculation of the mean and standard deviation of a set of $\delta^{13}C$ values extracted from the analysis of modern animal fats.

The descriptive statistical analysis of the variability of the results was based on the unit of median of the obtained values and the data were compared by average of box-plot and t-test analysis generated by the PAST software (Hammer et al. 2001). The comparative analysis of the reference values of modern adipose fats has been carried out with a calculation of the normality (p-normal) of the quantitative data and its subsequent comparison by t-test for parametric analyses and Kruskal-Wallis in non-parametric cases.

For the classification of ceramic vessels according to their relationship between shape and their specific use, multivariate statistical analyses were followed, such as cluster analysis, which groups individuals according to their similarities, and correspondence analysis, which indicates whether the qualitative variables of the groups of ceramic forms and their use are dependent. In order to carry out these statistical analyses, the free software PAST has been used (Hammer et al. 2001).

Chapter IV. Reference model of modern animal fats in the Iberian Peninsula

Fatty acids recovered from archaeological pottery via solvent organic can be analysed for their stable carbon isotope ratio to obtain more accurate resource identification. The most common and abundant are the methyl ester of the palmitic acid (C16:0) and stearic acid ($C_{18:0}$) fatty acids, resulting from laboratory induced or naturally degraded triacyl-glycerides. The stable isotope carbon values of these fatty acids ($\delta^{13}C$) can be measured using gas chromatography-isotope ratio mass spectrometry (GC-C-IRMS) technique.

In order to assess the molecular origin of lipids in archaeological ceramic artefacts, analyses were carried out on modern animal fats from United Kingdom, which offered reference values for distinguishing the origin of fats found in pottery (Evershed et al. 1994, 1997; Dudd y Evershed, 1998; Dudd, 1999; Aillaud, 2001; Copley et al. 2003, 2005; Craig et al. 2005; Mukherjee et al. 2007, 2008; Evershed, 2008). Gradually the database has been extended into northern Europe (Craig et al. 2007, 2011), in central Europe (Craig et al. 2005; Spangenberg et al. 2006), the Near East (Evershed et al. 2008; Gregg et al. 2009; 2010), the Far East (Ottram, 2009), Japan (Craig et al. 2015), as well as in the centre-east Mediterranean (Debono, 2012).

Until then isotopic values from modern British animal species have generally been used as reference data points for comparison against isotopic measurements obtained from archaeological residues. However, the validity of using this dataset as a universal reference collection has been challenged (Debono 2012). Previous work on $\delta^{13}C$ values from animal fats from different regions (Spangenberg et al. 2006, Gregg et al. 2009, Debono 2012, Lucquin et al. 2016), has also reported changes from the proposed reference values with fats from the UK (Dudd 1999, Copley et al. 2003, Craig et al. 2005). These values range from 1.7‰ to 3.5‰ in $\delta^{13}C$ isotope enrichment across regions.

In the following chapter, we develop a new model of reference for the Iberian Peninsula that allows to compare the GC-C-IRMS values obtained from $C_{16:0}$ and $C_{18:0}$ fatty acids of local animals to all the data published from the different regions in order to evaluate any variation in isotopic values, as well as to include new species that do not appear in the bibliography but that we find habitually in the prehistoric sites of this region of the west of the Mediterranean. Before doing so, it is necessary to review all the factors that influence the isotope value of modern fats and that would justify the sampling performed in this study.

4.1. Where and how are the fats analysed produced?

4.1.1. Lipid digestion

In monogastric animals, such as pigs and wild boars, digestion and absorption begin when fats from the diet reach the small intestine. However, in ruminants, such as cows and sheep, food products are broken down in the digestive tract by the growth and development of microorganisms (Noble 1981). While in ruminants' herbivores the enzymatic digestion that occurs after microbial decomposition also digests microbial bodies, in monogastric herbivores the digestion of fermentation follows enzymatic digestion, so that only the products of fermentation, and not the bacterial bodies, are absorbed (Frandson and Spurgeon 1992). Microbial digestion is of little importance in carnivores because digestive processes are virtually complete in the small intestine (Swenson and Reece 1993).

In ruminants, considerable chemical degradative and synthetic changes due to microbial action take place in the rumen (Garton et al. 1961, Katz and Keeney 1966). The result is the hydrogenation of the resulting free unsaturated fatty acids and the production of fatty acids, such as stearic acid, which usually constitutes a very small proportion of dietary lipid intake (Masson and Philipson 1951, Garton 1960). Studies have shown that when digestion leaves the abomasum and enters the small intestine, concentrations of short-chain fatty acids are very low (Huber and Moore 1964), as 70% of short-chain fatty acids produced in the rumen are absorbed into the bloodstream (Noble 1981). However, the remaining lipid components of the digestion remain unchanged (Noble 1981).

In non-ruminants, dietary triglycerides enter the small intestine from the stomach and mix with secretions of bile and pancreatic juice in the duodenum. Bile salts allow emulsification of lipids, while pancreatic lipase hydrolyzes triglycerides to free fatty acids (Harrison and Leat, 1975). The mycelium that is formed allows hydrolyzed compounds to dissolve in intestinal contents, as well as other water-insoluble compounds such as fat-soluble vitamins and cholesterol. These can then be absorbed by the mucosal cells, where the fatty acids are re-synthesized into triglycerides before passing to the lymph (Harrison and Leat, 1975).

Although the route of resynthesis is different in ruminants and non-ruminants, there is no evidence of any morphological difference in lipid absorption between the two (Harrison and Leat, 1975).

Considering that different fat deposits may have different isotopic values in the same animal (Dudd, 1999), the adipose tissue from the animal's torso has been selected, or the epithelial tissue from the thigh in the case of *Leporidae*.

4.1.2. Mammary gland

Dairy products present a characteristic and identifying isotopic signal, this happens by the processes of absorption of lipids by the mammary gland through the blood and its subsequent incorporation into the milk fat (Moore and Christie 1981). During absorption, the triglycerides are hydrolysed, and the released fatty acids are balanced with the fraction of plasma free fatty acids during passage through the mammary gland (Bickerstaffe et al. 1972). Acetate and β-hydroxybutyrate are the two most important substrates and the only carbon sources that allow the synthesis of de novo fatty acids in the mammary gland (Moore and Christie 1981). About 50% of $C_{16:0}$ is biosynthesized in the mammary gland and the remaining 50% results from the direct or indirect incorporation of the diet (Moate et al. 2008). However, the $C_{18:0}$ that is absorbed by the mammary gland is incorporated from dietary fatty acids (Dudd 1998). Triglyceride absorption travels from the intestines through the bloodstream to the udder and adipose tissue in approximately equal proportions. However, in the early stages of lactation (up to week 10), lactating animals cannot consume enough food to supply them with the energy they need, so triglycerides stored in adipose tissue are mobilised and moved to the udder to provide additional $C_{18:0}$. The proportion of $C_{18:0}$ milk derived directly from the diet of a modern cow will therefore vary throughout lactation (Dudd 1998).

Regarding the mobilisation of $C_{18:0}$ from the stored adipose, this is of particular concern for the feeding experiment, as it could introduce a C4 signal into milk samples. However, this was avoided by selecting only animals that had been lactating for more than ten weeks.

4.2. Aspects potentially influencing the stable isotopic composition of carbon in animal fats

Fatty acid stable carbon isotope composition in animal fat may be affected by exogenous and endogenous factors to the animal's body. Given the relation between diet and animal tissues (Ambrose and Krigbaum 2003; Schoeninger and Deniro 1983; DeNiro and Epstein 1978) and the fact that fatty acids synthesis is to a variable extent based on glucose (DeNiro and Epstein 1977), external factors affecting animal diet d13C values may be isotopically transferred to animal fatty acids (Colonese et al. 2017; Colonese et al. 2015; Salque et al. 2017).

Exogenous processes may involve for example the isotope composition of diet, which can be controlled by local or large-scale climate and cultural processes. Large scale processes involve general atmospheric carbon isotope pool that are ultimately assimilated by plants and animals. Local processes, instead, may involve human (through management and indirect impact) and nature-induced (climate) changes in resources type and distribution. Below I will discuss them in relation to potential changes through time and life of animals. Endogenous processes are related to physiology, and in particular difference between monogastric and ruminant animals.

4.2.1. Exogenous factors

4.2.1.1. Ecological variability

The $\delta^{13}C$ value of atmospheric CO_2 ultimately determines the tissue values of heterotrophic organisms through assimilation of plants and the carbon isotope values that undergo photosynthesis. Although this is relatively homogenous at global scale, there has been dramatic changes since the Industrial Revolution through the burning of fossil fuels (Farmer and Baxter 1974, Mook et al. 1983). Anthropogenic CO_2 is derived from the destruction of biomass, including the combustion of fossil fuels, deforestation and improved soil respiration due to increased agriculture (Tans, 1981). Calculations have been made to determine an average trend in variations in CO_2 measurements, indicating that the atmospheric value of CO_2 has decreased by approximately 1.14% from the late 18th century to 1980 and continues to decrease (Friedli et al. 1986). Consequently, archaeological and modern animal fatty acids $\delta^{13}C$ values will be inherently distinct. Regional to local ecological conditions might also account for variability in δ13C values in animal fat. This can be induced by people but could also be the case for regions where C4 plants are widespread (Salque et al. 2017).

4.2.1.2. Altitude

Intraspecific changes in δ13C have been observed in altitudinal gradients (Vitousek et al. 1990). Factors reported by ecologists relate it to soil moisture (Beerling et al. 1996), air temperature (Panek et al. 1995), atmospheric CO2 concentrations (Ehleringer et al. 1995), and leaf physiological traits. These studies have been able to corroborate that at higher altitudes, foliar mass increases and foliar nutrient concentrations decrease, reflecting an enrichment of $\delta^{13}C$.

Some studies carried out on pines at different altitudes have reported an enrichment of +5.3‰ the $\delta^{13}C$ value of the leaves. This could be due to an increase in the length of the CO2 diffusion pathway from the atmosphere to the carboxylating site and a potential decrease in discrimination at higher altitudes (Vitousek et al. 1990). This variability is in the collagen but might also affect fatty acids.

4.2.1.3. Seasonality and latitude

Climate differences cause regional patterns throughout Europe in plants (Van Klinken et al. 1994). The climatic effect is produced by the influence of temperature and/or relative humidity on the photosynthetic process of plants. Thus, the carbon fixation in the plant in the enzymatic step is affected by the temperature and partial pressure of the gases. In this way we see that the environmental factors of temperature, relative humidity or hydric stress can affect the isotopic value $\delta^{13}C$ of the plants that later will be consumed by the animals. For example, low rates of photosynthesis and high humidity lead to an impoverishment plants of $\delta^{13}C$ (Leavitt and Danzer 1994). On the other hand, periods of high temperatures and low humidity, stomata are closed to conserve water and CO_2 concentrations are lower, which presents an enrichment of the $\delta^{13}C$ isotopic value (Van Klinken et al. 1994). Studies on plant ecology have observed that the enrichment trend from northwest to south Europe correlates very strongly with the climatic pattern throughout Europe; the climatic isotopic variability is in the order of 2 to 4‰ (Van Klinken et al. 1994). This variation was reflected by comparing reported data on animal products bred under different climatic conditions, where a change of 3.5‰ is shown between the values obtained for charcoal between the United Kingdom (-27.5‰) and Spain (-24‰) (Van Klinken et al. 1994). This variability is in the colagen but might also affect fatty acids.

Similarly, we can observe that the existence of an isotopic differentiation between northern and southern Europe is replicable to seasonality. In countries where temperatures and humidity levels are not very variable, as in the UK, this variation should be minimal. On the other hand, in the south of the peninsula, where there are climatic influences from the Mediterranean and the Atlantic Ocean, temperatures and humidity are very variable depending on the season.

4.2.1.4. Feeding

The influence of diet on the isotopic composition of fats has been extensively investigated. The influence of dietary fat on the composition of animal fats varies by species, for example, rabbits incorporate a higher amount of $C_{18:3}$ than sheep and deer, although they have an identical diet (Shorland et al. 1952).

In the case of wild ruminants, there is less deposition of neutral glycerides and a higher proportion of phospholipids compared to ruminants fed concentrate (Ledger 1968). Changes due to a forage diet are considered as the result of altered ruminal fermentation. Diets high in forage have been documented to increase deposition of saturated acids, mainly $C_{16:0}$ (McDonald et al. 1988) and increase the proportions of branched and transunsaturated acids, while the proportions of $C_{18:2}$ are reduced (Marmer et al. 1984).

Diet may have a much more significant effect on the composition of omnivores, such as pigs, than on ruminants. Since saturated and unsaturated fatty acids are directly absorbed from the diet in the intestine, they have no structural changes (Mills et al. 1976) and can digest much higher proportions of dietary fat than ruminants (Busboom et al. 1991).

From here on, isotope fractionation is consistent between and within different species (DeNiro and Epstein, 1978). One study determined that there is a -3% deviation in $\delta^{13}C$ in fatty tissue relative to diet (Tieszen et al. 1983, DeNiro and Epstein, 1977). Fat tissue was also found to have a relatively short half-life of 15.6 days, indicating that carbon rotation was relatively fast compared to other tissues, requiring 208 days in total for complete carbon rotation.

Since forage makes a significant contribution to the diet of ruminants, the effect of dietary contributions of different types of plants has been studied in relation to the values of $\delta^{13}C$. Previous studies have focused on differences in $\delta^{13}C$ values of C3 and C4 plants and their effect on fat composition (Minson et al. 1975). Most of terrestrial plant species in temperate countries are C3 (Osmond et al. 1982) and have values around -27‰, while C4-type plants are found in salt marshes or in products that did not exist during the

Neolithic period in our study area, such as maize, and have values around -13% (Debono 2012). Therefore, the stable isotope composition of an animal's diet will influence that of its biosynthesized tissue, as the carbon isotope values of the food ingested are not substantially altered (DeNiro and Epstein 1978).

4.2.2. Endogenous factors

4.2.2.1. Age

The age of animals affects the fat composition, for example, C18:0 decreases over time (Enser, 1991) or subcutaneous samples show an increase of C18:1 versus a decrease of C18:0 in older animals (Leat 1975, 1977, Pyle et al. 1977). In young cattle, before rumen development, dietary fatty acids are deposited directly in their adipose tissues. Apart from that, if the animal is breastfeeding, the milk fats go directly to its adipose tissues. The effects of race and sex on fatty acid composition are relatively negligible (Sumida et al. 1972; Gillis et a., 1973).

4.2.2.2. Fat deposit

It has been shown that the composition of fatty acids varies according to the location of the fat deposit, with marked differences between internal and subcutaneous fats. Ruminants have higher ratios of $C_{18:0}$ and lower than $C_{18:1}$ in perirenal fat, while subcutaneous chest and rump fats contain a higher proportion of unsaturated fatty acids (Christie and Moore 1971, Leat 1975). In contrast, pig fat shows less variation (Whitehead and Turrel 1988).

4.3. Sampling

In the selection of the samples that make up this study, the following aspects have been considered in this experiment.

Given that the main objective of this work is to establish a comparative model with the values published so far in different regions, with special attention to the UK reference models, a total of 76 samples were selected. They represent 9 different species or products that can be found consumed in prehistoric sites in the Iberian Peninsula.

Organically certified producers were contacted from various parts of the Iberian Peninsula which, moreover, did not include maize or other C4 plants to the animal diet.

An example as characteristic as maize, a product originating in the American continent that appeared in Europe from the sixteenth century, is often used to supply livestock during the winter or as a complement to pasture feed. Instead, this type of plants was not part of the feeding of the cattle in the European Neolithic, but it was only of C3 type vegetation (Hunt et al. 2008).

The fact of repeating in several samples from the same species and that they come from different regions allowed us to replicate the obtained data, as well as to corroborate variations in the same region to study. Domestic animals come from organic farms where there is no input of C4 plants in the dietary cycle of the animals. Concerning wild animals, feeding is not controlled but comes from natural areas. In both cases, the most analogous situation to the prehistoric context that we could find in the peninsular region was sought (Figure 4.1).

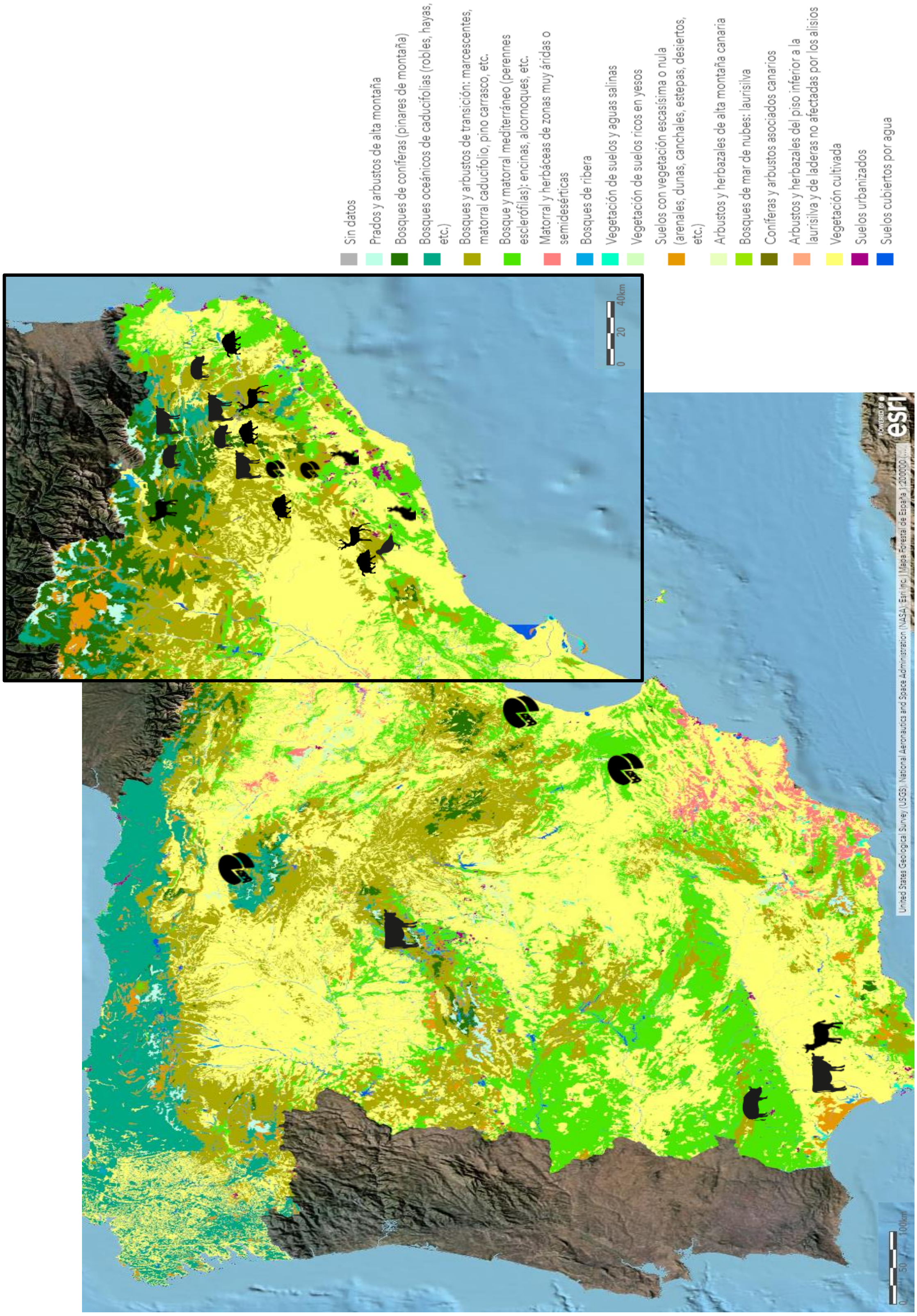


Figure 4.1. Vegetation map of the Iberian Peninsula showing the location of the sites in which modern reference samples analysed were carried out.

The difference between the $\delta^{13}C$ values of the fatty acids varies depending on the fat deposit from which it is extracted. As mentioned above, the synthesis of the components is different parts of the body, adipose fat, liver, mammary gland, etc., which can result in different degrees of isotopic discrimination in fat synthesis. The adipose fat of the loin has been selected in all cases, since the fat route is more direct and more representative of the animal diet (Emery, 1980).

The age of the animals, which we have already seen does not have such a direct affectation with the $\delta^{13}C$ values, has been selected according to the times of cattle slaughter (when the animals achieve their optimum meat).

LAB CODE	SPECIES	AGE	SACRIFICE	TYPE	FEEDING	ORIGIN	δ13C		
							C16:0	**C18:0**	**Δ13C**
V01	Bos taurus	12 months	May	Adipose tissue	Fodder	Vic, Catalunya	-23,46	-24,88	-1,42
V02	Bos taurus	6-10 months	October	Adipose tissue	Fodder	Pirinat (Campdevanol, Catalunya)	-22,70	-24,48	-1,78
V03	Bos taurus		Mars	Adipose tissue	Fodder	Campos (Sevilla)	-26,17	-27,22	-1,05
V04	Bos taurus		Mars	Adipose tissue	Fodder	Braman (La Losa, Segovia)	-29,37	-31,30	-1,93
V05	Bos taurus		November	Adipose tissue	Grass	Mas Espinau (Beuda, Catalunya	-24,66	-26,27	-1,61
V06	Bos taurus		November	Adipose tissue	Grass	Mas Espinau (Beuda, Catalunya	-24,16	-26,66	-2,50
V07	Bos taurus		October	Adipose tissue	Fodder	Pirinat (Campdevanol, Catalunya)	-24,21	-24,16	0,05
MEAN							**-24,96**	**-26,42**	**-1,46**
X01	Ovis aries		October	Adipose tissue	Fodder	Pirinat (Campdevanol)	-26,14	-27,13	-0,99
X02	Ovis aries		Mars	Adipose tissue	Fodder	Campos (Sevilla)	-29,42	-30,37	-0,95
X03	Ovis aries		October	Adipose tissue	Fodder	Pirinat (Campdevanol)	-29,49	-29,80	-0,31
X04	Ovis aries		January	Adipose tissue	Fodder	Tambor del Llano (Cádiz)	-28,28	-31,19	-2,91
X05	Ovis aries		May	Adipose tissue	Fodder	Vic, Catalunya	-26,47	-26,48	-0,01
MEAN							**-27,96**	**-28,99**	**-1,03**
P01	Sus domesticus	5 weeks	May	Adipose tissue	Fodder	Vic	-19,78	-19,36	0,42
P02	Sus domesticus	5 weeks	October	Adipose tissue	Fodder	Pirinat (Granja Romanich, Vall d'en Bas)	-22,99	-20,45	2,54
P03	Sus domesticus		Mars	Adipose tissue	Fodder	Moià	-19,78	-19,36	0,42
P04	Sus domesticus		Mars	Adipose tissue	Fodder	Moià	-22,99	-20,45	2,54
P05	Sus domesticus		January	Adipose tissue	Fodder + acorns	Dehesa Maladua (Jabugo, Extremadura)	-25,65	-25,00	0,65
P06	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,47	-21,81	2,66
PG01	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-25,74	-23,43	2,31
PG02	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-22,02	-20,45	1,57
PG03	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,85	-22,71	2,14
PG04	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-26,05	-24,91	1,14
PG05	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,11	-23,34	0,77
PG06	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,38	-23,32	1,06
PG07	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,73	-23,49	1,24
PG08	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-26,53	-24,18	2,35
PG09	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,37	-24,73	-0,36
PG10	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,92	-23,88	1,04
PG11	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-26,55	-24,34	2,21
PG12	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-25,11	-22,91	2,20
PG13	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,25	-22,30	1,95
PG14	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-21,33	-19,31	2,02
PG15	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,80	-24,24	0,56
PG16	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,28	-23,07	1,21
PG17	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-26,25	-24,50	1,75
PG18	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-24,72	-22,17	2,55
PG19	Sus domesticus		May	Adipose tissue	Fodder + acorns	Garrotxa, Catalunya	-19,78	-19,61	0,17
MEAN							**-24,02**	**-22,53**	**1,48**
S01	Sus scrofa	12 months	December	Adipose tissue	Wild food	Talamanca, Catalunya	-25,98	-24,19	1,79
S02	Sus scrofa	36 months	November	Adipose tissue	Wild food	Vic, Catlunya	-26,16	-24,49	1,67
S03	Sus scrofa	36 months	November	Adipose tissue	Wild food	Vallès, Catalunya	-26,32	-25,51	0,81
S04	Sus scrofa	18 months	November	Adipose tissue	Wild food	Vallès, Catalunya	-26,86	-25,69	1,17
S05	Sus scrofa	36 months	November	Adipose tissue	Wild food	Vallès, Catalunya	-26,28	-25,42	0,86
S06	Sus scrofa	26 months	November	Adipose tissue	Wild food	Vallès, Catalunya	-26,75	-24,57	2,18
S07	Sus scrofa	18 months	November	Adipose tissue	Wild food	Vallès, Catalunya	-30,47	-31,01	-0,54
SG01	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-26,81	-26,50	0,31
SG02	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-23,61	-23,46	0,15
SG03	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-26,57	-25,20	1,37
SG04	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-28,89	-29,65	-0,76
SG05	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-27,60	-26,82	0,78
SG06	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-19,40	-20,23	-0,83
SG07	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-27,22	-27,26	-0,04
SG08	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-27,33	-27,47	-0,14
SG09	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-29,50	-29,24	0,26
SG10	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-26,19	-25,71	0,48
SG11	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-27,58	-26,08	1,50
SG12	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-28,90	-28,54	0,36
SG13	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-27,60	-27,04	0,56
SG14	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-22,38	-20,83	1,55
SG15	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-22,87	-24,75	-1,88
SG16	Sus scrofa		May	Adipose tissue	Wild food	Girona, Catalunya	-28,02	-31,30	-3,28
SG17	Sus scrofa		May	Adipose tissue	Wild food	Prades, Catalunya	-23,96	-26,76	-2,80
SG18	Sus scrofa		May	Adipose tissue	Wild food	Prades, Catalunya	-25,03	-25,43	-0,40
SG19	Sus scrofa		May	Adipose tissue	Wild food	Prades, Catalunya	-32,85	-34,25	-1,40
MEAN							**-26,58**	**-26,44**	**0,14**
LL01	Bos taurus			Milk		Raphel Lladó (Besalú, Catalunya)	-28,00	-34,04	-6,04
LL02	Capra hircus			Milk		Cantero de Letur (Letur, Albacete)	-24,24	-28,30	-4,06
LL03	Bos taurus			Milk		La Torre (Sallent, Catalunya)	-26,60	-30,43	-3,83
FR01	Capra hircus			Cheese		Vall de Catí (Catí, Castellón)	-27,00	-28,97	-1,97
FR02	Capra hircus			Cheese		Betara (St Boi de Lluçanes, Catalunya)	-23,01	-25,32	-2,31
FR03	Bos taurus			Cheese		Betara (St Boi de Lluçanes, Catalunya)	-25,24	-27,70	-2,46
FR04	Ovis aries			Cheese		Betara (St Boi de Lluçanes, Catalunya)	-26,57	-29,30	-2,73
LL04	Bos taurus			Milk		Cantero de Letur (Letur, Albacete)	-22,68	-26,77	-4,09
LL05	Ovis aries			Milk		Cantero de Letur (Letur, Albacete)	-27,24	-32,45	-5,21
FR05	Ovis aries			Iogurt		Can Mateu (Surp, Catalunya)	-23,32	-26,32	-3,00
FR06	Ovis aries			Cheese		Cantero de Letur (Letur, Albacete)	-23,85	-27,07	-3,22
FR07	Capra hircus			Cheese		Santa Gadea (Rioseco, Burgos)	-24,99	-28,21	-3,22
MEAN							**-25,23**	**-28,74**	**-3,51**
CB01	Capreolus capreolus		January	Adipose tissue	Wild food	Vic, Catalunya	-30,47	-30,61	-0,14
CB02	Capreolus capreolus		June	Adipose tissue	Wild food	Prades, Catalunya	-30,47	-31,01	-0,54
CB03	Capreolus capreolus		June	Adipose tissue	Wild food	Prades, Catalunya	-29,76	-31,15	-1,39
MEAN							**-30,23**	**-30,92**	**-0,69**
CN01	Oryctolagus cuniculus		October	Adipose tissue	Wild food	Vallès, Catalunya	-30,97	-30,10	0,87
CN02	Oryctolagus cuniculus		October	Adipose tissue	Wild food	Vallès, Catalunya	-31,61	-30,97	0,64
CN03	Oryctolagus cuniculus		February	Adipose tissue	Fodder + aromatic plants	Moncada (Reus, Catalunya)	-30,55	-28,42	2,13
CN04	Oryctolagus cuniculus		February	Adipose tissue	Fodder + aromatic plants	Moncada (Reus, Catalunya)	-30,85	-28,55	2,30
MEAN							**-31,00**	**-29,51**	**1,49**
BC01	Scolopax rusticola		June	Adipose tissue	Wild food	Prades, Catalunya	-25,76	-24,41	1,35
BC02	Scolopax rusticola		June	Adipose tissue	Wild food	Prades, Catalunya	-24,68	-23,08	1,60
MEAN							**-25,22**	**-23,75**	**1,48**

Table 4.1. Description of samples and results of modern animal fats analysis from Iberian Peninsula.

4.4. Methods

For the extraction of free fatty acids from modern animal fat, an extraction protocol proposed by other researchers has been followed (Evershed et al. 2002, Craig et al. 2012) (described in Chapter III).

The $\delta^{13}C$ measurements for $C_{16:0}$ and $C_{18:0}$ of the modern samples were also corrected for the post-Industrial Revolution effects of fossil fuel burning, which were found to have decreased the $\delta^{13}C$ of atmospheric CO_2 by 1.8‰ (Hellevang and Aagaard 2015). To correct for PIC, 1.14‰ was added to the $\delta^{13}C$ values of the samples measured.

4.5. Results

76 samples of different products (domesticated species adipose, wild species and dairy products) were analysed by GC-IRMS. The stable carbon isotope ($\delta^{13}C$) values of the n-hexadecanoic acid (16:0) and n-octadecanoic acid (18:0) extracted by saponification were measured. All values were plotted to distinguish different product ranges (Figure 4.2).

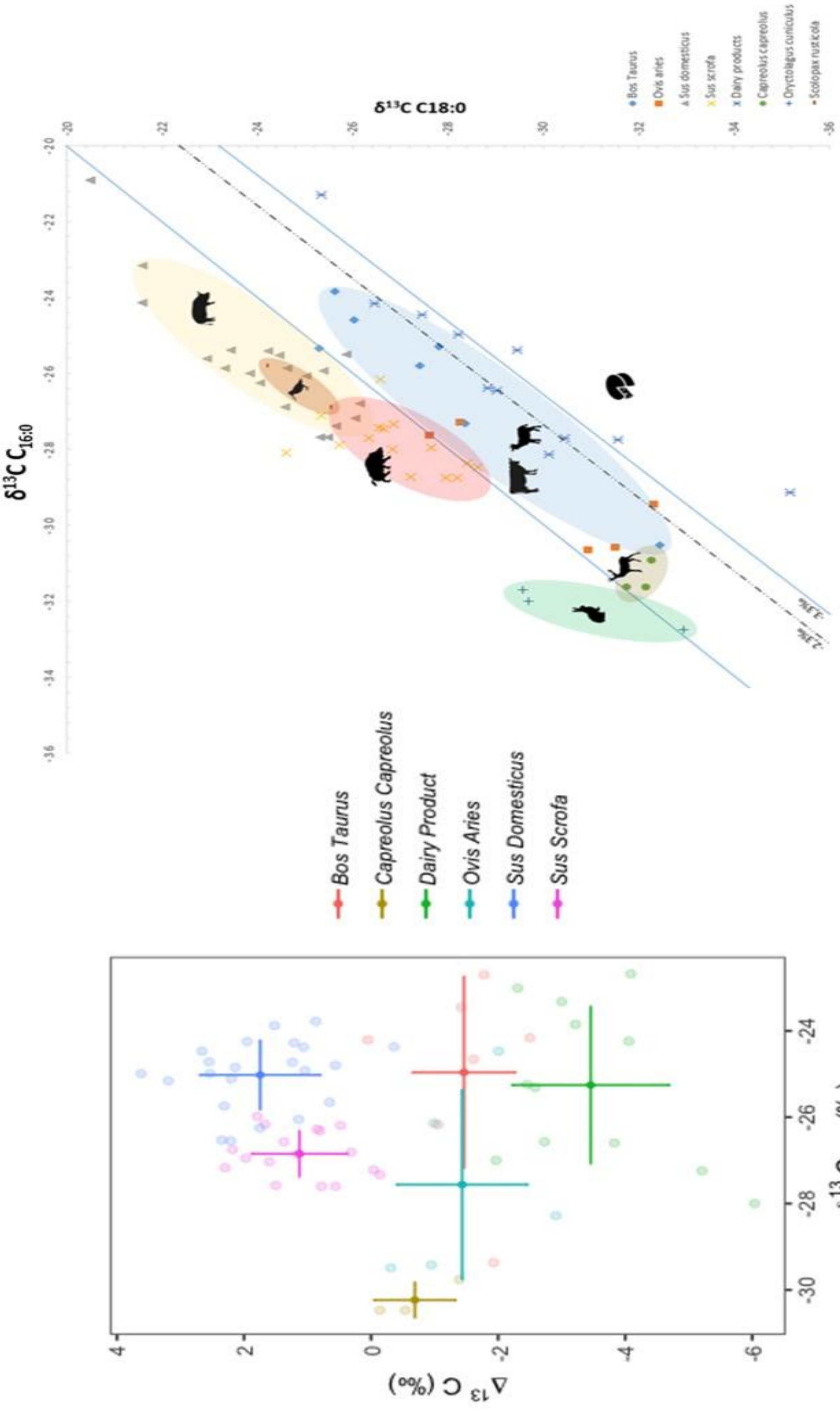


Figure 4.2. Plot showing the Δ13C values (left) and δ13C values of the C16:0 and C18:0 fatty acids (right) obtained from Iberian Peninsula modern animal and dairy fats.

SPECIES	N	AVERAGE δ13C			STDEV δ13C			MAX δ13C			MIN δ13C		
		C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C
Bos taurus	7	-24,96	-26,42	-1,46	2,06	2,26	0,74	-22,70	-24,16	0,05	-29,37	-31,30	-2,50
Ovis aries	5	-27,96	-28,99	-1,03	1,42	1,85	1,01	-26,14	-26,48	-0,01	-29,49	-31,19	-2,91
Sus domesticus	25	-24,02	-22,53	1,48	2,00	1,87	0,86	-19,78	-19,36	2,66	-26,55	-25,00	-0,36
Sus scrofa	26	-26,58	-26,44	0,14	2,64	2,98	1,34	-19,40	-20,23	2,18	-32,85	-34,25	-3,28
Capreolus capreolus	3	-30,23	-30,92	-0,69	0,33	0,23	0,52	-29,76	-30,61	-0,14	-30,47	-31,15	-1,39
Oryctolagus cuniculus	4	-31,00	-29,51	1,49	0,39	1,07	0,74	-30,55	-28,42	2,30	-31,61	-30,97	0,64
Scolopax rusticola	2	-25,22	-23,75	1,48	0,54	0,67	0,13	-24,68	-23,08	1,60	-25,76	-24,41	1,35
Dairy product	12	-25,23	-28,74	-3,51	1,75	2,43	1,15	-22,68	-25,32	-1,97	-28,00	-34,04	-6,04

Table 4.2. Statistical description of δ13C results per species.

From the results obtained, it can immediately be observed that we can discriminate ruminates (*Ovis aries, Bos taurus* and *Capreolus capreolus*) from non-ruminates (*Sus domesticus, Sus scrofa*) and rabbits (*Oryctolagus cuniculus*) and birds (*Scolopax rusticola*), and in turn from the remaining dairy products from the Δ^{13}C index.

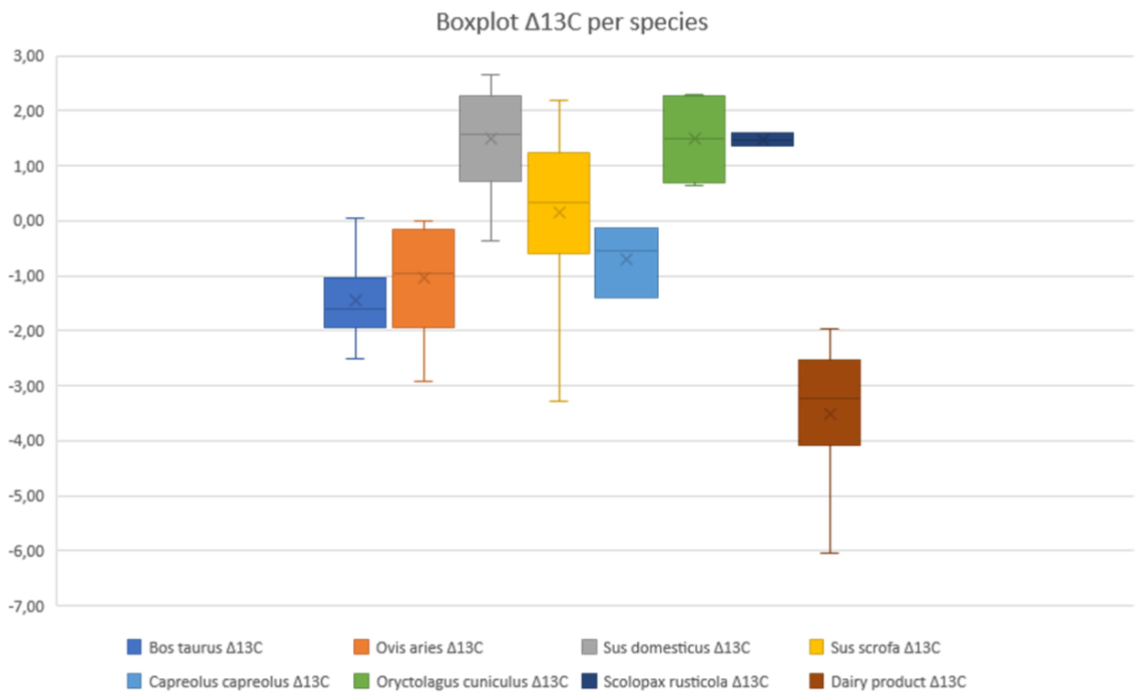


Figure 4.3. Boxplot showing Δ13C mean and SD per species.

Within each range we see differences between the values δ^{13}C of $C_{16:0}$ and $C_{18:0}$. Within ruminants, *Bos taurus* was considerably enriched in δ^{13}C compared to *Ovis aries* and *Capreolus capreolus*. If we observe the high standard deviation (σ) of *Bos taurus*, it exceeds 2‰, since the minimum and maximum values are very separate. If we look at the boxplot graph (Figure 4.3), we can see that this prevents discrimination between *Ovis aries* and *Bos taurus*, since they can be fed with the same type of plants. For that reason, we will treat them as domestic ruminants. On the other hand, Capreolus capreolus can be more easily discriminated given the impoverished $\delta^{13}C$ value of $C_{16:0}$. This may be due, as discussed above, to a wild and forested diet based on plants other than open grassland. Even so, we must keep in mind that, in this case, the number of samples observed is low and these interpretations may vary slightly with larger sampling.

Among the average values higher than 0.0‰ in the $\Delta^{13}C$ index, we find rabbits, which are easily discriminated by their low values of $C_{16:0}$ and $C_{18:0}$. The fat of the analyzed wild birds (*Scolopax rusticola*) presents similar values to the pigs (*Sus domesticus*), in addition to presenting only two samples, which makes it impossible to discriminate between these two species from the relationship $\delta^{13}C$. On the other hand, from the large number of suidae analyzed we can distinguish the domestic taxon (*Sus domesticus*) from the wild taxon (*Sus scrofa*) from the isotopic value $\delta^{13}C$ of the palmitic and stearic acids, which appear more enriched in *Sus domesticus*. This factor may be influenced by diet, since the diet of *Sus domesticus* is usually enriched with animal protein and with a vegetable diet very different from the acorns and wooded vegetables *Sus scrofa* feed on. It is necessary to comment on the two outliers presented in the *Sus scrofa* category, which cause an increase in the standard deviation, although they do not present a significant variation in the mean of the values obtained. In addition to having a large number of samples, these values are presented at each end of the ranges, which does not cause significant variations.

Among dairy products, we observe that isotopic values move in a wide range (t-test $\delta^{13}C$ $C_{16:0}$ p=3.9792E-14; $\delta^{13}C$ $C_{18:0}$ p= 3.85E-13; $\Delta^{13}C$ p= 6.7469E-07). To understand this variation, we have grouped the values according to species. The t-test results show that there are no differences between species (t-test p=>0.05). On the other hand, if we separate the values according to whether they are fresh milk or fermented product, some similarities are registered in the palmitic acid (t-test p=>0.05). When applying the statistical analysis in stearic acid the heterogeneous variance, Kruskal-Wallis was used (p=0.08). The discriminant factor between fresh milk and fermented milk product appears in the index $\Delta^{13}C$ (t-test p=0.0008) and can be seen in the boxplot in figure 4.4. Thus, we see that the values $\Delta^{13}C$ <-3.0‰ correspond to fresh milk, while the higher values correspond to fermented products.

SPECIES	N	AVERAGE δ13C			STDEV δ13C		
		C16:0	**C18:0**	**Δ13C**	**C16:0**	**C18:0**	**Δ13C**
Bos taurus	4	-25,63	-29,73	-4,10	1,963243	2,826221	1,2764
Capra hircus	4	-24,81	-27,70	-2,89	1,448568	1,405116	0,815567
Ovis aries	4	-25,25	-28,79	-3,54	1,687254	2,383008	0,979668
Milk	5	-25,75	-30,40	-4,65	1,985714	2,646916	0,845342
Fermented	7	-24,85	-27,56	-2,70	1,438232	1,322989	0,443087

Table 4.3. Statistical description of δ13C dairy product results per species and type of product.

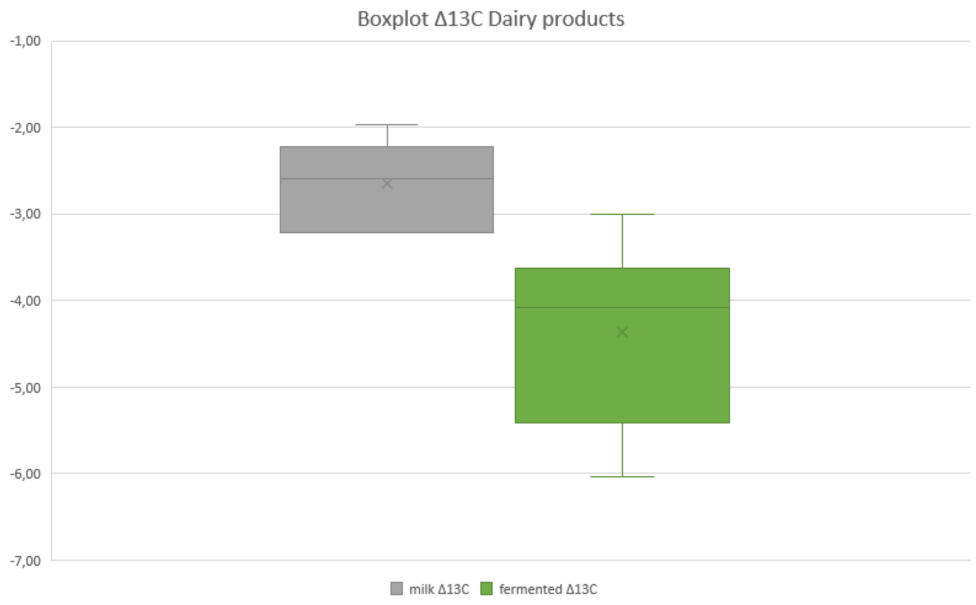


Figure 4.4. Boxplot δ13C means of fresh milk and fermented products.

Another of the factors that we have wanted to represent is the spatial interspecific differentiation, that is to say, if the species are differentiated according to the region of the Iberian Peninsula where they come from. We have selected domestic ruminants (*Ovis aries and Bos taurus*) and compared their $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$ according to their origin. We can see that there are no significant differences between the northeast of the peninsula and the south of the Iberian Peninsula (Kruskal Wallis p=>0.05; t-test $\Delta^{13}C$ p=>0.05). We could not make this observation in the case of the domestic pig, given that we only had one observable for the southern peninsular region. We wanted to observe what happened to wild boar, which has a wild diet, between three areas in the northeast of the Iberian Peninsula; the north (Girona), the center (Vallès) and the south (Prades). The results were also not significant (Kruskal-Wallis p=>0.05). Therefore, with the available data, no significant variations of the isotopic value 13C are observed according to the region within the Iberian Peninsula.

		AVERAGE δ13C			STDEV δ13C		
		C16:0	**C18:0**	**Δ13C**	**C16:0**	**C18:0**	**Δ13C**
Domestic rumiant	NW	-25,16	-26,23	-1,07	2,02	1,69	0,86
	South	-27,96	-29,59	-1,64	1,35	1,71	0,90
Sus domesticus	NW	-23,95	-22,43	1,52	2,01	1,84	0,86
	South	-25,65	-25,00	0,65	N.A.	N.A.	N.A.
Sus scrofa	Girona	-26,28	-26,26	0,02	2,69	2,87	1,21
	Vallès	-27,34	-26,44	0,90	1,58	2,32	0,87
	Prades	-27,28	-28,81	-1,53	3,96	3,88	0,98

Table 4.4. Statistical description of δ13C animal fats results per species and origin.

In this experiment, samples were collected at different times of the year from domestic ruminants, pigs and wild boar in order to observe a variability between whether the last feeding period was in a warm or cold season. Samples of ruminants (*Ovis aries and Bos taurus*) were collected between October and January, corresponding to the cold months before winter; and between March and May, corresponding to the breeding months in which the animals are already one year old and have achieved optimum meat. In this case, with the available data, no significant differences between the two groups were observed in the values of $\delta^{13}C$ of $C_{16:0}$, $C_{18:0}$ and $\Delta^{13}C$ (t-test p=>0.05). The same applies to the values of $\delta^{13}C$ for $C_{16:0}$, $C_{18:0}$ and $\Delta^{13}C$ for domestic pigs (t-test p=>0.05). Wild boar, on the other hand, do not show significant differences in the values of $\delta^{13}C$ for palmitic and stearic acids, but in the values $\Delta^{13}C$ (t-test p=0.02). This would indicate that the values $\Delta^{13}C$ can help to discriminate the seasonality of wild boar, presenting more impoverished values in the coldest months. Even so, we must take this interpretation with caution, given that this variability has not been observed in other cases, which may respond to a sum of factors.

		AVERAGE			STDEV		
			δ13C			δ13C	
		C16:0	**C18:0**	**Δ13C**	**C16:0**	**C18:0**	**Δ13C**
Domestic rumiant	Oct -Jan	-25,66	-27,10	-1,44	2,26	2,40	1,01
	Mar-May	-26,98	-28,05	-1,07	2,23	2,41	0,63
Sus domesticus	Oct -Jan	-24,32	-22,73	1,60	1,33	2,28	0,95
	Mar-May	-23,99	-22,52	1,47	2,04	1,83	0,85
Sus scrofa	Nov-Dec	-26,97	-25,84	1,13	1,46	2,18	0,83
	May	-26,44	-26,66	-0,22	2,95	3,19	1,31

Table 4.5. Statistical description of δ13C animal fats results per species and sacrifice month.

Finally, we wanted to value the feeding of domestic animals. Faced with the low variability of ruminants that ate fodder and those fed natural pastures (t-test p=>0.05), we designed a model among domestic pigs fed on organic farms with animal protein and pigs for the production of acorn-fed serrano ham, fed with acorns as well as organic fodder. The result is significant, the values $\delta^{13}C$ $C_{16:0}$ and $C_{18:0}$ present significant differences according to the feeding (t-test p=0.002; p=0.001, respectively), but there are no differences in the index $\Delta^{13}C$ (t-test p=0.99), which places the pigs in the same group if we only take as reference the index $\Delta^{13}C$ to interpret the archaeological results (Figure 4.5).

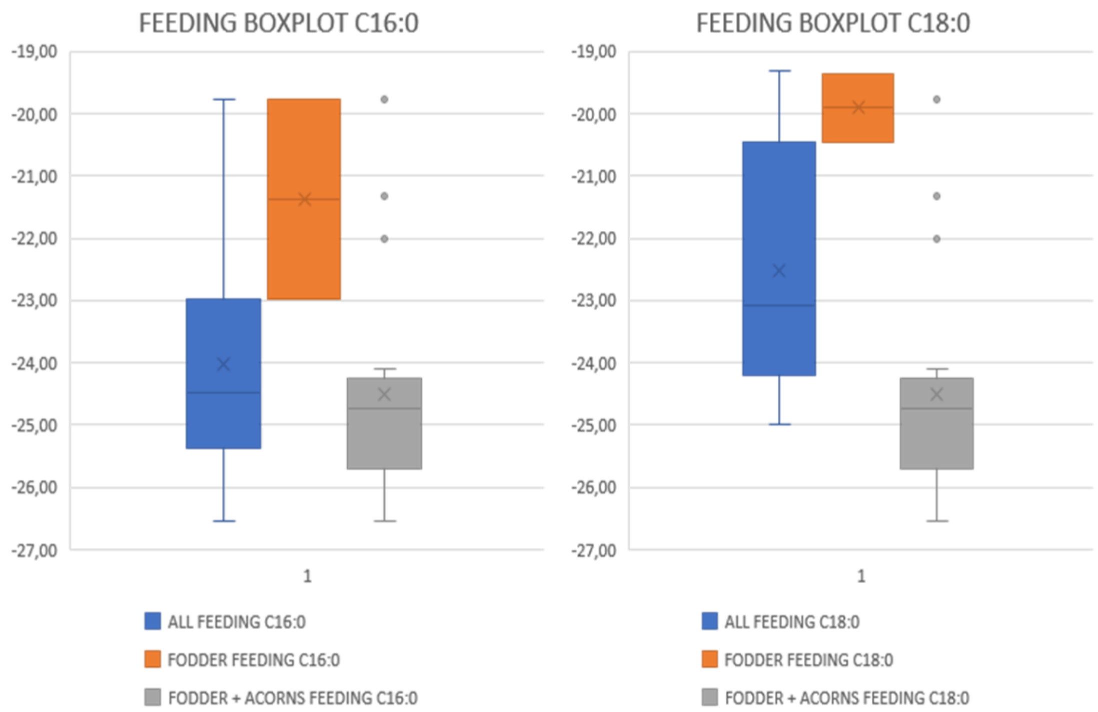


Figure 4.5. Boxplot with δ13C means and SD of C16:0 and C18:0 from pig adipose fats related to their feeding.

		AVERAGE δ13C			STDEV δ13C		
		C16:0	**C18:0**	**Δ13C**	**C16:0**	**C18:0**	**Δ13C**
Domestic rumiant	Fodder	-26,57	-27,70	-1,13	2,41	2,64	0,88
	Grass	-24,41	-26,47	-2,06	0,25	0,20	0,45
Sus domesticus	Acorn	-24,52	-23,03	1,49	1,64	1,59	0,81
	Fodder	-21,39	-19,91	1,48	1,61	0,55	1,06

Table 4.6. Statistical description of $\delta^{13}C$ animal fats results per species and feeding method.

4.6. Discussion

If we compare these averages with the data obtained from the literature, from UK (Craig et al. 2011, 2015, Dudd 1999) and Mediterranean (Spiteri-Debono 2012), we observe some differences.

Firstly, if we compare the $\delta^{13}C$ isotope values obtained in the Iberian Peninsula with those obtained in the Italian Peninsula and Malta (Debono 2012), we can see some similarities. In the experimental study carried out in the central Mediterranean, 9 blood samples were obtained from ruminants (sheep, cows and goats) with slightly enriched values compared to those obtained from ruminant fats in the Iberian Peninsula (~2‰). By means of the comparative study of t-test, we can observe that the values $\delta^{13}C$ of $C_{16:0}$, $C_{18:0}$ and $\Delta^{13}C$ do not present significant differences (p=>0.05). In the case of domestic pigs, the values $\delta^{13}C$ obtained from the blood of this species are unfeasible for statistical analysis, given that only two samples are presented in the bibliography. Even so, we can comment that the values $\delta^{13}C$ $C_{16:0}$ and $C_{18:0}$ are similar to those obtained from fats from the Iberian Peninsula. On the other hand, the index $\Delta^{13}C$ is significantly more enriched than the adipose fats of domestic pigs of the Iberian Peninsula. The lack of observables for the isotopic values of wild boar and wild ruminants makes a comparative study of these categories impossible. Finally, the $\delta^{13}C$ isotopic values obtained from different dairy products in the central Mediterranean compared to those obtained in the Iberian Peninsula show significant differences in palmitic acid (p=0.045). Stearic acid, on the other hand, did not show significant differences (t-test p=>0.05). The same applies to the index $\Delta^{13}C$ (t-test p=>0.05). This leads us to conclude that, in general, the Mediterranean values are similar to those obtained in this study. Although the enrichment of many of the values discussed makes us take caution when mixing the data.

If we compare the data obtained with the $\delta^{13}C$ isotopic values of the different products from the UK, we see that the differences are, in general, very significant. The $\delta^{13}C$ values of domestic ruminant adipose fat present an enrichment in the UK of around ~4‰, a fact that is corroborated by the statistical analysis of these two populations (t-test $C_{16:0}$ p=0.0001, $C_{18:0}$ p=0.0001, $\Delta^{13}C$ p=0.0036). In the case of domestic pigs, we see that these differences are significant for the $\delta^{13}C$ values of palmitic and stearic acid (t-test $C_{16:0}$ p=0.0001, $C_{18:0}$ p=0.0001). On the other hand, the index $\Delta^{13}C$ does not present significant differences (p=>0.05). We found an inverse result in the comparison of the $\delta^{13}C$ values of wild ruminant adipose fats, since they present a difference in the values for palmitic and stearic acid with little significance (p=>0.05), although an impoverishment of the isotopic value resulting from the index $\Delta^{13}C$ of around 2‰ (t-test p=0.044). In reference to dairy products, the $\delta^{13}C$ values present an enrichment in those coming from the Iberian Peninsula, with significant differences (t-test $C_{16:0}$ p=0.0001, $C_{18:0}$ p=0.0001, $\Delta^{13}C$ p=0.0001). This variability, as we have seen above, may be due to the mixture of fresh milk and fermented products in the same category for both regions. Given the clear absence of the type of dairy product analysed in the literature, we cannot extract any more conjectures.

No $\delta^{13}C$ data have been obtained for the fat values of wild boar in the UK, the most consistent data published so far come from Japan (Lucquin et al. 2016). The $\delta^{13}C$ adipose fat samples of wild boar from Japan have been compared with the results obtained from this species for the Iberian Peninsula. Statistical results by t-test show significant differences for palmitic acid (p= 0.032), but not for stearic acid or the index $\Delta^{13}C$.

Iberian Peninsula

SPECIES	N	AVERAGE δ13C			STDEV δ13C		
		C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C
Domestic ruminants	7	-26,21	-27,50	-1,28	2,34	2,45	0,89
Sus domesticus	25	-24,02	-22,53	1,48	2,00	1,87	0,86
Sus scrofa	26	-26,58	-26,44	0,14	2,64	2,98	1,34
Wild ruminants	3	-30,23	-30,92	-0,69	0,33	0,23	0,52
Dairy product	12	-25,23	-28,74	-3,51	1,75	2,43	1,15

UK

SPECIES	N	AVERAGE δ13C			STDEV δ13C		
		C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C
Domestic ruminants	21	-29,71	-31,82	-2,11	0,69	0,79	0,47
Sus domesticus	15	-26,67	-25,44	1,22	0,94	1,06	0,38
Sus scrofa	0						
Wild ruminants	10	-30,22	-32,62	-2,57	1,41	1,64	1,37
Dairy product	23	-29,15	-34,10	-4,94	0,95	0,96	0,77

Mediterranea

SPECIES	N	AVERAGE δ13C			STDEV δ13C		
		C16:0	C18:0	Δ13C	C16:0	C18:0	Δ13C
Domestic ruminants	9	-24,12	-25,10	-0,98	2,39	2,65	0,81
Sus domesticus	2	-24,85	-24,75	0,10	0,15	0,25	0,10
Sus scrofa	1	-28,9	-28,8	0,1			
Wild ruminants	0						
Dairy product	10	-23,68	-27,94	-4,26	1,63	1,02	1,32

Table 4.7. Means of $\delta^{13}C$ values of animal fats from Iberian Peninsula compared to data from the bibliography (Craig et al. 2012, 2015, Copley et al. 2003, Dudd 1999, Dunne et al. 2012, Evershed et al. 1997, 1999, Lucquin et al. 2016, Spiteri-Debono 2012).

4.7. Conclusions

Biomarker detection and carbon isotope measurements of a single compound have allowed greater opportunities to address issues of resource introduction and use at specific times and locations in the past. Lipid residue analysis is not simply a record of technical advances in analytical instrumentation. Overall, the number of case studies and the number of observables in each case has increased. Similarly, the sampling strategy, related to the identification of explicit biomarkers and their integration with broader archaeological research, is ensuring that the results of such work are integrated and considered in relation to other sources. Multidisciplinary work offers the possibility of profiling interpretation and contributing data to the study of past societies that cannot be obtained through other ways of study. However, biomolecular analyses are still incipient and are in a phase of constant development.

That is why we believe in the need to provide new comparative reference data for the $\delta^{13}C$ values, in order to help the consolidation of referentials suitable for comparison with archaeological data.

In this way, we see how from this experimental study, we have been able to contribute new species to the reference collection, such as the wild boar and the rabbit, for the Mediterranean.

On the other hand, we have been able to observe a variability in the data that allows us to discriminate between fresh milk and fermented dairy products. Even so, it is necessary to extend this register in order to be able to confirm this hypothesis with more grounds.

In the case of pigs, it has been possible to discriminate between domestic pigs and wild boar from the large number of adipose fats analysed and has been able to emphasize the importance in the food that receives the pig, as it can be discriminatory.

Finally, we have been able to prove the caution to be followed when comparing the $\delta^{13}C$ values obtained from archaeological sites with reference values from different regions. In this way, we stress the need to be able to compare the data with local reference values. Therefore, the main objective of this work is to be able to offer reference data for the sites of the Iberian Peninsula.

We emphasize the need for the expansion of this collection, either in number for each category, as the variability of the factors that can be represented in the interspecies differentiation.

In conclusion, this chapter has outlined the development of organic residue analysis, both in terms of sample preparation and the instrumentation used and has proposed interpretative improvements that reinforce this type of analysis as a powerful tool for a robust and dynamic field of research.

Chapter V. Natural substances identification in neolithic pottery from the Iberian Peninsula

5.1. Pyrenean context

5.1.1. Coro Trasito

The concentrations of organic components in the ceramic vessel of Coro Trasito, obtained by DCM/MeOH extraction, were relatively low. Only in 19.2% were recovered trace amounts of palmitic and stearic acid, which included samples CTT05, CTT09, CTT17, CTT33 and CTT34, in average, their TLE was 47.20 $\mu g \cdot g^{-1}$. To evaluate the potential presence of other bound lipids, an acidified methanol extraction was also practiced in those samples that presented negligible amount of lipids (<5 $\mu g \cdot g^{-1}$ of lipids) with DCM/MeOH extraction. This led to increasing the number of vessels with organic residues to 73%, so in 19 out of the 26 vessels analysed from Coro Trasito. Their average TLE content was 212.1 $\mu g \cdot g^{-1}$., so 4.5 times higher than when DCM/MeOH extraction was used. The substances identified in Coro Trasito can originate from degraded fats of animal and plant origin, epicuticular waxes and pine resin.

Sample	TLE (µg·g-1)	Lipids detected	$\delta^{13}C$ C16:0 ±0,3 (‰)	$\delta^{13}C$ C18:0 ±0,3 (‰)	Pedominant commodity type
CTT01	167,7	FA (12, 14, 16>18, 18:1, 20, 22, 24, 26, 28); PAH: phenanthrene	-26,34	-30,11	Dairy fat
CTT03	9053,9	FA (8, 9, 10, 11, 12, 14, 15, 16<18, 17, 18:1, 20, 21, 22, 23, 24, 25, 26, 28); Alkanes: C25-C34; Alcohol: C22OH-C32OH	-27,73	-31,01	Ruminant adipose fat Epicuticular wax
CTT05	90,2	FA (12, 14, 16<18)	-25,83	-26,86	Ruminant adipose fat
CTT06	498,3	FA (9, 10, 12, 14, 16<18, 18:1, 20, 22, 24, 26, 28); Diterpenoids: dehidroabietic acid, dehidroabietic acid methyl	-21,75	-23,11	Ruminant adipose fat Pine resin
CTT07	29,4	FA (14, 16>18, 18:1)	-26,53	-25,60	Pig adipose fat
CTT09	60,02	FA (12, 14, 16<18)	-27,76	-28,91	Ruminant adipose fat
CTT10	523,3	FA (9, 10, 12, 14, 16>18, 17, 18:1, 20, 22, 24, 26, 28, 30); Diterpenoids: dehidroabietic acid, dehidroabietic acid methyl	-22,88	-23,95	Ruminant adipose fat Pine resin
CTT13	212,1	FA (16>18, 18:1); Diterpenoids: dehidroabietic acid, dehidroabietic acid methyl; Alkanes: C24-C34	-27,01	-28,23	Ruminant adipose fat Pine resin
CTT15	287,4	FA (16>18, 18:1); Diterpenoids: dehidroabietic acid, dehidroabietic acid methyl; Alkanes: C24-C34	-28,92	-30,83	Ruminant adipose fat Pine resin
CTT17	23,1	FA (14, 16:1, 16>18, 18:1)	-27,18	-26,98	Wild boar adipose fat
CTT20	39,3	FA (12, 14, 16>18, 18:1, 20)	-26,93	-32,29	Dairy fat
CTT33	41,3	FA (16>18)	-26,03	-25,71	Pig adipose fat
CTT34	52,01	FA (14, 16>18, 17)	-25,87	-21,46	Pig adipose fat
CTT35	545,3	FA (14, 15, 16<18, 17, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30)	-26,70	-27,48	Ruminant adipose fat
CTT36	95,5	FA (12, 14, 16>18, 17, 18:1); Alcanes: C24-C35	-24,56	-23,33	Pig adipose fat
CTT37	1786,6	FA (14, 15, 16<18, 17, 20, 22, 24, 26, 28, 30); Alkanes: C24-C35; Alcoholes: C22OH-C34OH	-25,81	-28,49	Dairy fat; epicuticular wax
CTT38	694,9	FA (14, 16>18, 17, 18:1); Alkanes: C24-C35	-24,91	-21,07	Pig adipose fat
CTT39	569,3	FA (14, 15, 16<18, 18:1, 17, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32); Alkanes: C24-C35 ; Alcoholes: C22OH-C34OH	-28,61	-27,47	Wild boar adipose fat Epicuticular wax
CTT40	476,03	FA (14, 15, 16<18, 17, 20, 22, 24, 26, 28, 30); Alkanes: C24-C35; Alcoholes: C22OH-C34OH	-28,11	-31,74	Dairy fat; epicuticular wax

Table 5.1. Organic residues analysis from Coro Trasito results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.1.1.1. Animal fats

Analysis by gas chromatography mass spectrometry (GC-MS) revealed a range of saturated mid-chain length n-alkanoic acids (fatty acids) with even numbers of carbon atoms on 19 of vessels from Coro Trasito, particularly dominated by $C_{16:0}$ (palmitic acid) and $C_{18:0}$ (stearic acid). Considering the palmitic/stearic ratio (P/S), the very high abundance of $C_{18:0}$ in some samples (CTT05, CTT09, CTT35) can be attributed to an animal origin for the lipids. Conversely, some other sherds contained high concentration of $C_{16:0}$ compared to $C_{18:0}$ and oleic acid ($C_{18:1}$) (CTT07, CTT17, CTT36, CTT38), which could be indicative of the presence of plant lipids. However, the absence of other characteristic plant biomarkers, such as plant sterols, prevent us from proposing the presence of plant oils.

The lipid profiles encountered are in fact typical of degraded animal fats, whereas the presence of branched-chain fatty acids ($C_{15:0}$ and $C_{17:0}$) in 7 samples are often attributed a ruminant origin, as they are compounds that are often synthesized by bacterial activity in the rumen of these animals (Dudd et al. 1999; Evershed, 1993). However, its presence may also indicate the occurrence of postdepositional microbial activity, within the vessel or in soils where the pottery was buried or laid (Dudd et al. 1999; Dudd and Evershed, 1998; Muckherjee et al. 2008).

To increase the confidence on the origin of the fatty acids, compound specific $\delta^{13}C$ analyses were carried out on 19 samples that yielded sufficient amounts of fatty acids. The fatty acids $\delta^{13}C$ values for Coro Trasito reveal a significant amount of variability along the $\Delta^{13}C$ index (Figure 5.1).

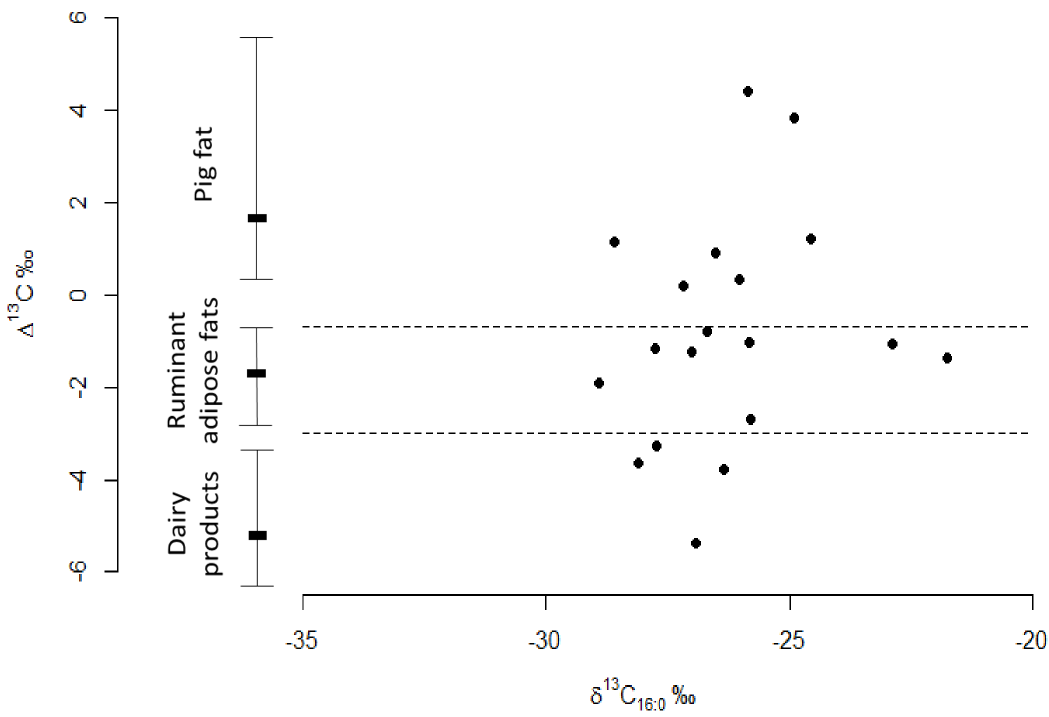


Figure 5.1. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ and $\Delta^{13}C$ values from Coro Trasito samples.

As we can see from figure 5.1, in 8 vessels the $\Delta^{13}C$ index is consistent with the presence of ruminant adipose fats. Nevertheless, in other 7 samples a significantly more isotopically enrichment is compatible with a source from a non-ruminant adipose fats. The vessels corresponding to the CTT01, CTT20, CTT37 and CTT40 have low $\Delta^{13}C$ values, which is consistent with the range of values that correspond to dairy products obtained from modern reference fats (see chapter IV).

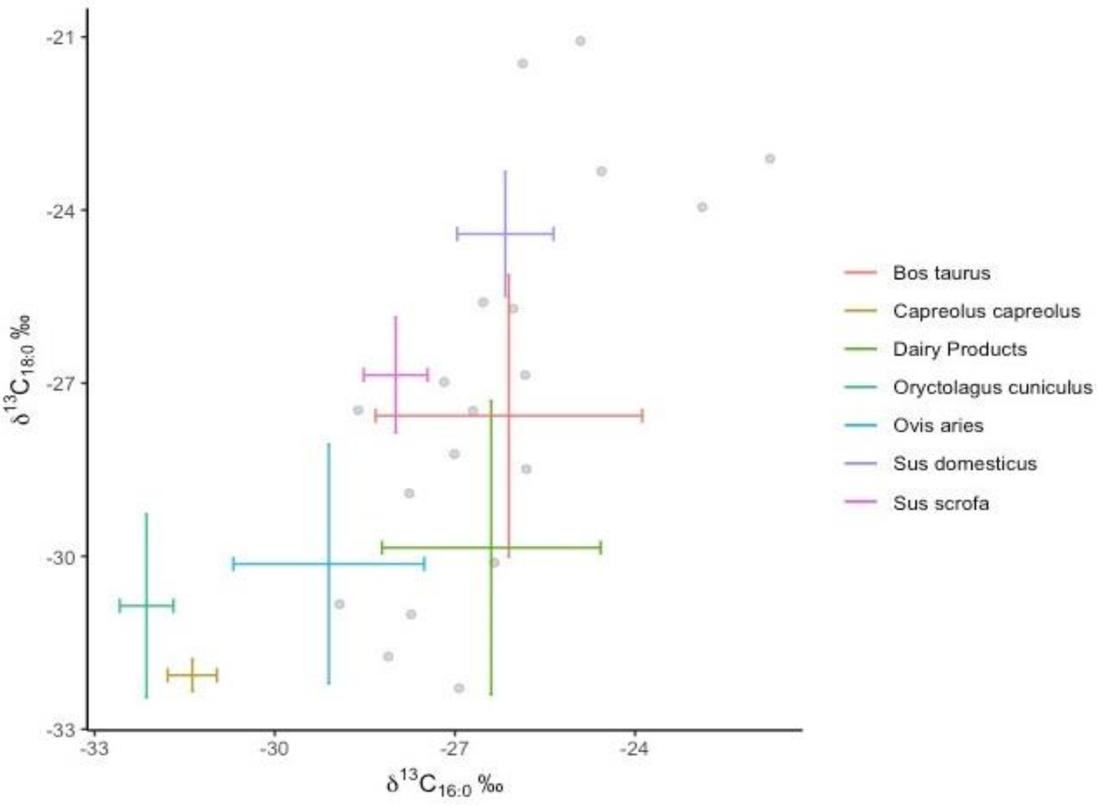


Figure 5.2. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ (X) and $\delta^{13}C$ $C_{18:0}$ (Y) average and standar desviation values from Coro Trasito samples.

By comparing the $\delta^{13}C$ values obtained in Coro Trasito pottery with the different modern fats from the Iberian Peninsula (Figure 5.2), we be more precise on the source attribution based on carbon isotopic values. Thus, we see that ruminant adipose fats consistent to domestic ruminants, while among non-ruminants we find the presence of a d13C value that is consistent with the isotope values of the analysed wild boar adipose fats (CTT39).

5.1.1.2. Epicuticular waxes

Four sherds analysed in Coro Trasito (CTT03, CTT37, CTT39, CTT40) contained i) long-chain fatty acids, with carbon lengths up to $C_{30:0}$, ii) long-chain linear alcohols, with chain lengths between $C_{24}OH$ and $C_{30}OH$, and iii) long-chain odd-numbered n-alkanes, with chain lengths between between C_{25} and C_{35}.

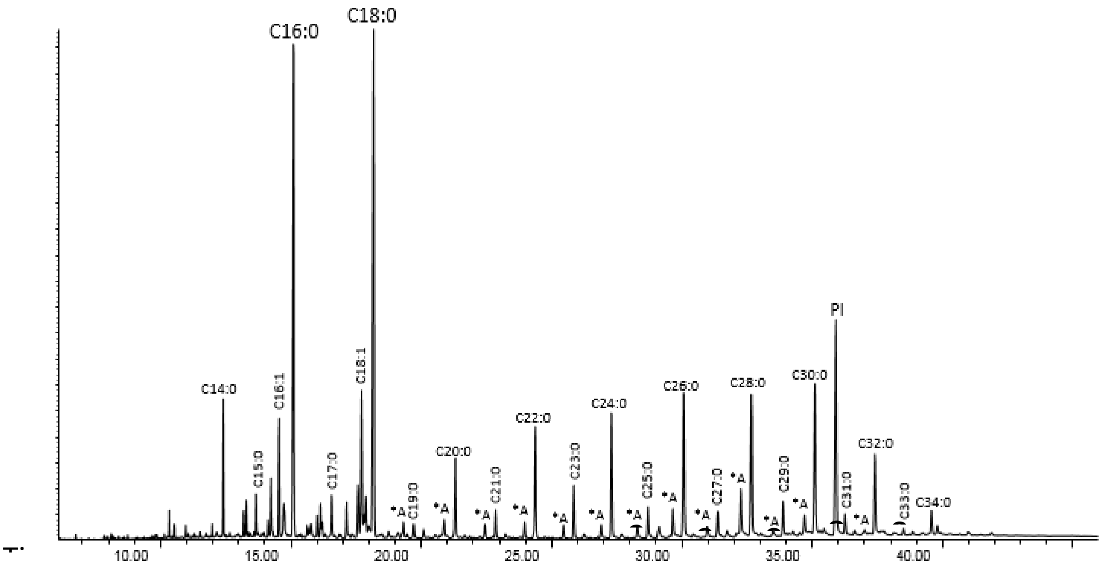


Figure 5.3. Partial gas chromatogram from CTT40 pottery sample showing the various biomarkers detected: fatty acids (FA*x:y* are fatty acids where *x* is the carbon chain length and *y* is the degree of unsaturation); *n*-alkanes, *A; *n*-alcohols, O. Internal Standar, PI.

These compounds present in the samples are found in both epicuticular and bees waxes. Beeswax derived from *A. mellifera* comb wax is a lipid-based complex mixture of more than 300 constituent fractions; it consists mainly of esters (67%), hydrocarbons (14%), free acids (12%), free alcohols (1%), and other (unidentified) constituents (6%).

The major compound families in beeswax are defined as those exceeding 5% (w/w) of the total beeswax composition (alkanes, alkenes, free fatty acids, monoesters, diesters and hydroxymonoesters); according to Tulloch (1980), only several individual constituent fractions constitute more than 5% of the total beeswax composition, namely C40 monoester (6%), C46 monoester (8%), C48 monoester (6%) and C24 (lignoceric/tetracosanoic) acid (6%).

In order to be able to discriminate between them, first of all, it is worth noting that vegetable waxes contain fatty acids with carbon lengths between $C_{8:0}$ and $C_{30:0}$, such as those presented by samples from Coro Trasito (Figure 5.3), while in fresh beeswax only lignoceric acid appears ($C_{24:0}$). Secondly, vegetable and bee waxes have long chains of odd linear alkanes, but with a predominance of C_{31} and C_{33} in epicuticular wax and C_{27} in bee wax (Eglinton and Hamilton, 1967; Evershed et al. 1994; Evershed & Lockheart, 2007; Gilz, 1994; Kolattukudy, 1970; Tulloch & Hoffman, 1973). Several Coro Trasito sherds also contained mid and long-chain length odd-numbered n-alkanes between C_{25} and C_{35}, specially C_{31} and C_{33} (Figure 5.4).

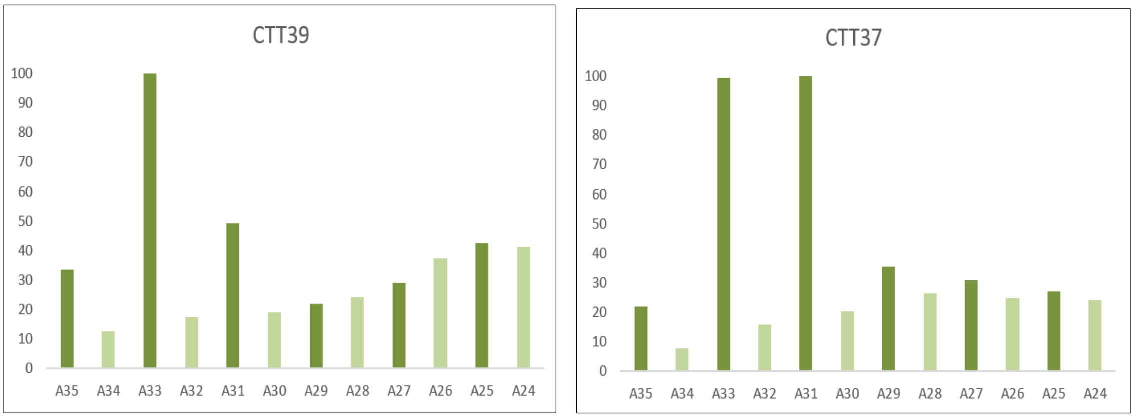


Figure 5.4. Normalized relative abundance of *n*-alkanes from samples CTT37 and CTT39.

Along with alkanes, we find long-chain linear alcohols, with chain lengths between C_{24} and *n*-C_{32}OH, with *n*-C_{26}OH as the most abundant compound. This distribution is common in epicuticular waxes, while bee waxes are dominated by *n*-C_{32}OH (Charters et al. 1995; Evershed et al. 1994; 1997; 2003; Heron et al. 1994; Regert et al. 2001; Salque et al. 2015) (Figure 5.5).

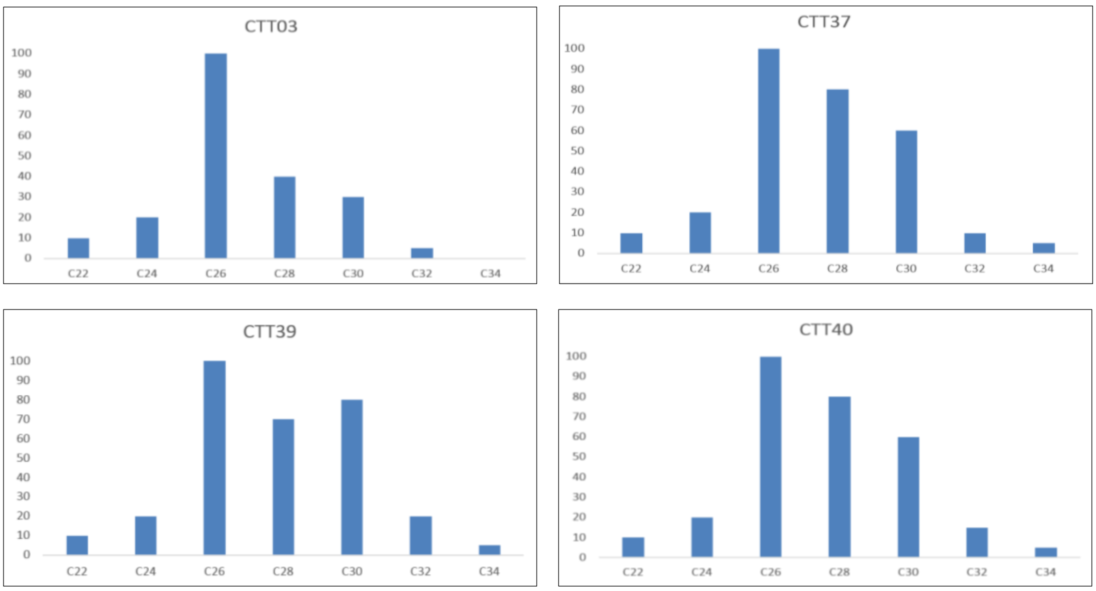


Figure 5.5. Alcohol profile of samples CTT03, CTT37, CTT39 and CTT40.

It should be noted that the method of extraction by sulphuric acid methylation breaks down esterifiedmolecules such as wax esters, which are key to discriminating the origin of wax evidenced inside archaeological vessels (Correa-Asensio et al. 2014).

In order to confirm the origin of vegetable waxes, given the absence of wax esters, an isotopic analysis of the alkanes C_{27}, C_{29} and C_{31} was carried out, comparing the results with the reference bibliographic values (Figure 5.6).

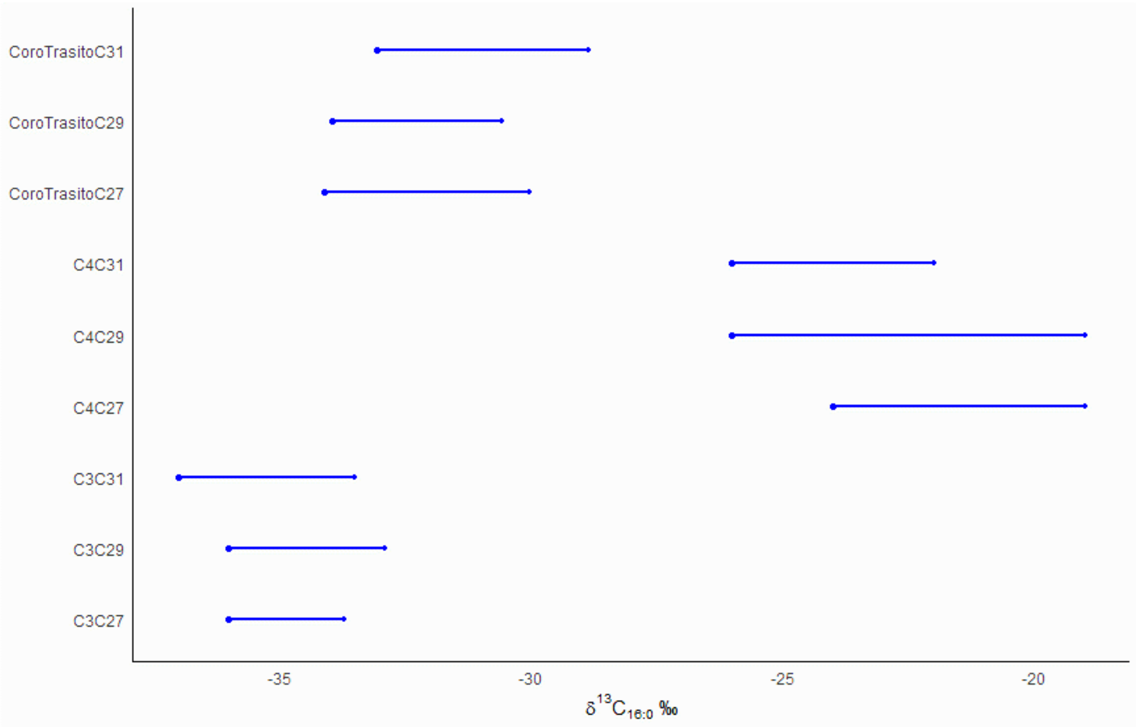


Figure 5.6. Isotopic values of C_{27}, C_{29} and C_{31} alkanes from Coro Trasito compared with modern epicuticular waxes of natural origin (DeNiro & Epstein, 1978, Evershed et al. 1999, Dunne et al. 2016, Jambrina-Enríquez et al. 2018).

In conclusion, the occurrence of relative abundance of alkanes, alcohols and fatty acids with carbon lengths in CTT03, CTT37, CTT39 and CTT40 samples is consistent with epi-cuticular waxes.

5.1.1.3. Resins

Diterpenes are also documented in 4 samples from Coro Trasito. Among these compounds, the products of the degradation of abietic acid have been mainly detected, such as dehydroabietic acid, methylated dehydroabietic acid and 7-oxo-dehydroabietic acid. Their occurrence is indicative of substances containing coniferous exudates, such as pine (Colombini et al. 2005; Regert & Rolando, 2002; Bailly et al. 2016). However, pimaric and isopamiric acids were not detected in the samples, and thus the occurrence of pine exudates cannot be confirmed. The absence of these biomarkers might be related to their high degree of degradation.

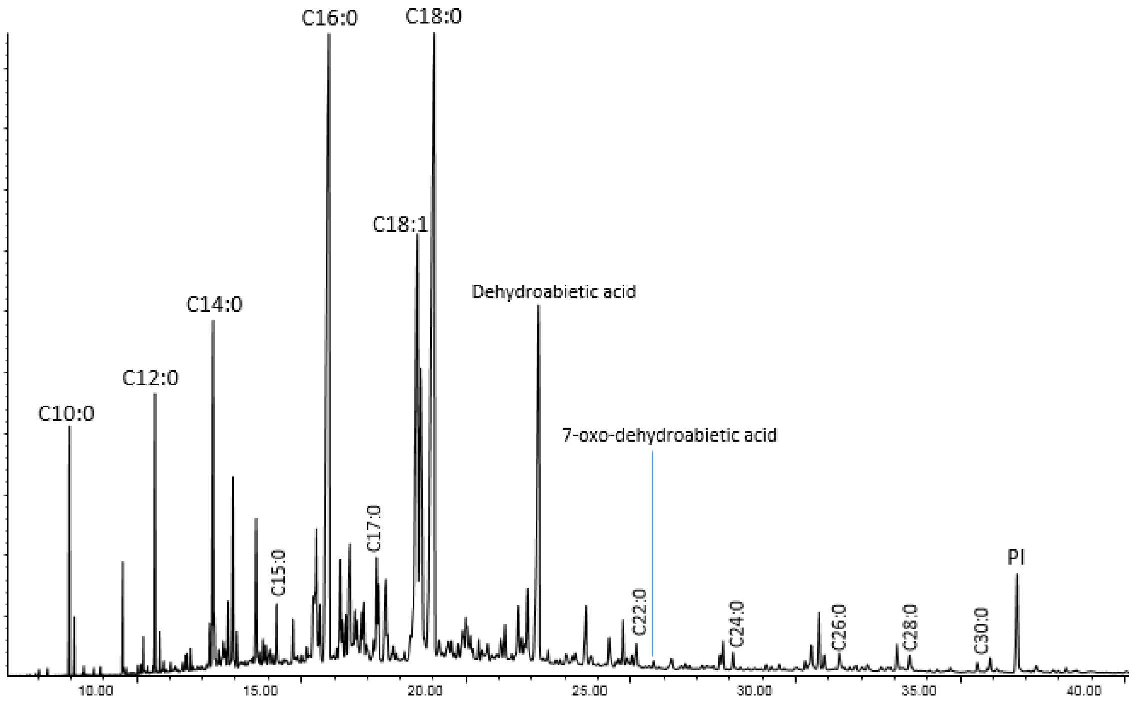


Figure 5.7. Mass chromatogram (GC-MS) from CTT06 sample.

5.1.2. Cova del Sardo

Nine vessels from levels 8, 7, 6 and 5 of Cova del Sardo were analysed. Based on the results obtained using the GC-FID and GC-MS techniques, 4 samples (44.4%) yielded a significant amount of lipids by extracting them with DCM/MeOH (Table 5.2). The good preservation of organic matter in this site is reflected in the mean of the TLE extracted: 672.15 $\mu g \cdot g^{-1}$.

Sample	TLE (µg·g-1)	Lipids detected	$\delta^{13}C$ C16:0 ±0,3 (‰)	$\delta^{13}C$ C18:0 ±0,3 (‰)	Pedominant commodity type
SAR02	1794,27	C14:0-C18:0, MAG, DAG, TAG	-25,26	-27,22	Ruminant adipose fat
SAR03	447,7	C16:0-C18:0, MAG, PAH, Contaminants	-26,36	-28,91	Ruminant adipose fat
SAR04	899,23	C14:0-C18:0, MAG, DAG, TAG	-24,96	-26,13	Ruminant adipose fat
SAR11	2640,96	C14:0-C18:0, MAG, DAG, TAG	-24,77	-27,03	Ruminant adipose fat

Table 5.2. Organic residues analysis from Cova del Sardo results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.1.2.1. Animal fats

Fatty acids of putative animal origin were identified in all vessels investigated (n=4, 44.4% of the samples). In this sense, the occurrence of triacylglycerides in three samples (SAR02, SAR04 and SAR11) is particularly significant. In cases where triacylglycerols degraded from the original substances of the animal fats have been preserved, it can be discerned whether they are fat of lactic origin or fat of subcutaneous animal origin.

The analyses of contemporary reference fats by GC–MS has shown that ruminant and non-ruminant adipose fats can be distinguished based on their TAG distributions. Ruminant adipose fats contain TAGs of total acyl carbon numbers that range between T_{42} for bovine or T_{44} for ovine and T_{54} for porcine (Dudd *et al.* 1999; Mukherjee *et al.* 2007). The adipose fat of non-ruminant species presents a narrower distribution of TAGs that range from T_{44} to T_{54}, with very low abundances of T_{44}, T_{46}, and T_{48} (Dudd *et al.* 1999; Mukherjee *et al.* 2007). In milk fat products, the presence of short-chain fatty acids is responsible for a broad distribution of TAGs ranging from T_{28} to T_{54} (Dudd *et al.* 1998; Copley *et al.* 2005).

Regert (2011) proposed two parameters that show a differential relationship depending on the type of fat from which they come: it is a calculation of the mean of the number of carbons (M) present in the triacylglycerides and their dispersion factor (DF). To obtain the necessary data, the author calculated M and DF from the following calculation: M = (∑(PiCi)/∑Pi); DF = (√∑[(Ci - M)2x CiPi])/∑Pi. When Ci is the number of carbon atoms and Pi the relative percentage of each triacylglycerol (Figure 5.8).

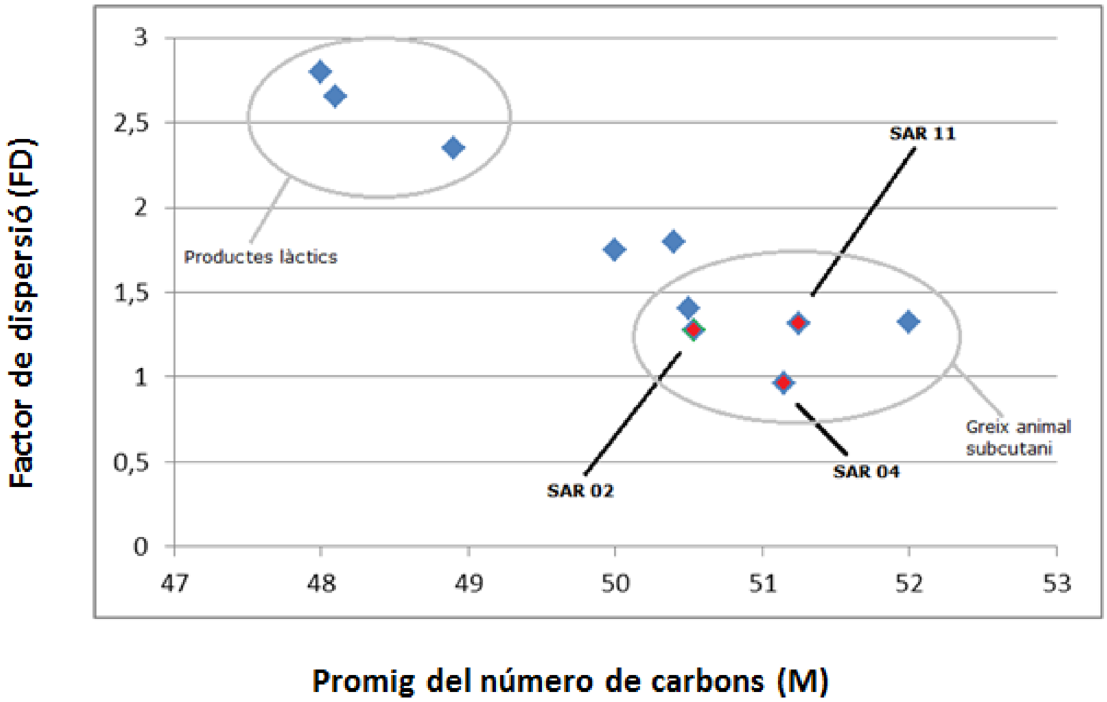


Figure 5.8. Dispersion factor *versus* the average carbon number from Cova del Sardo TAGs in comparision between the TAGs distributions from Clairvaux XIV samples (Regert, 2011).

The scatter diagram (Figure 5.8) shows that the triacylglyceride values of three samples from Cova del Sardo and the experimental data presented by M. Regert (Regert 2011). The results show that all three samples correspond to subcutaneous fatty animal.

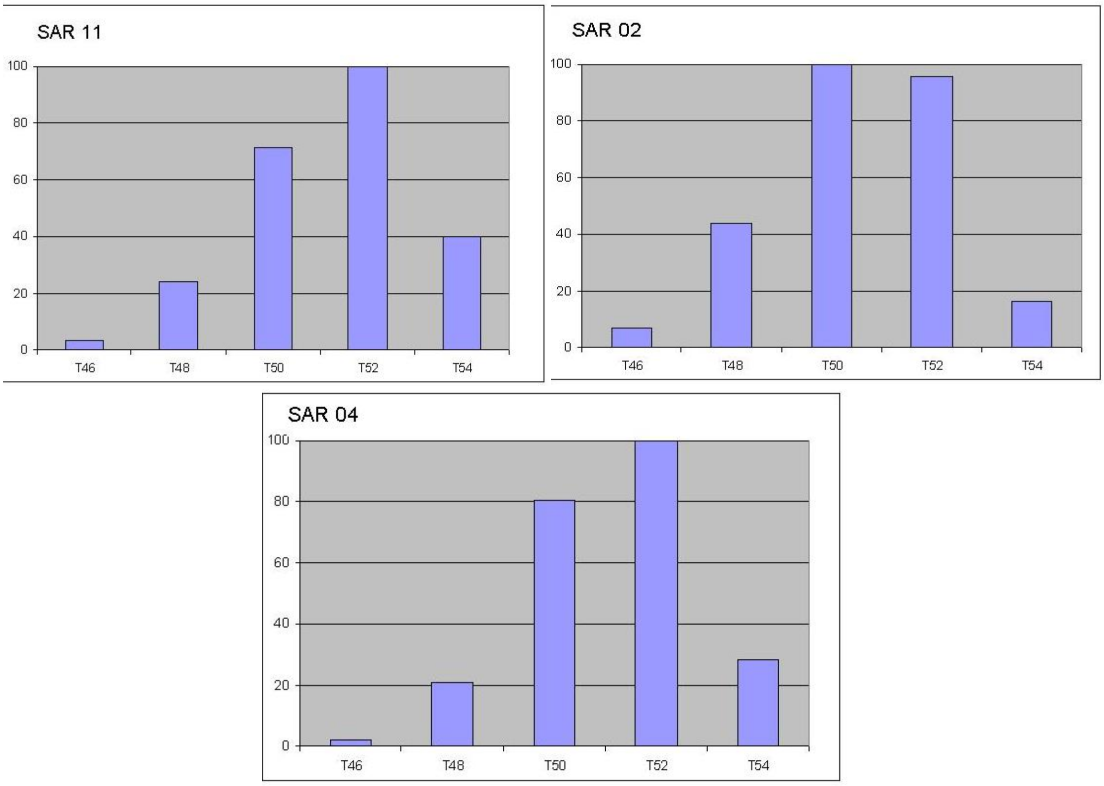
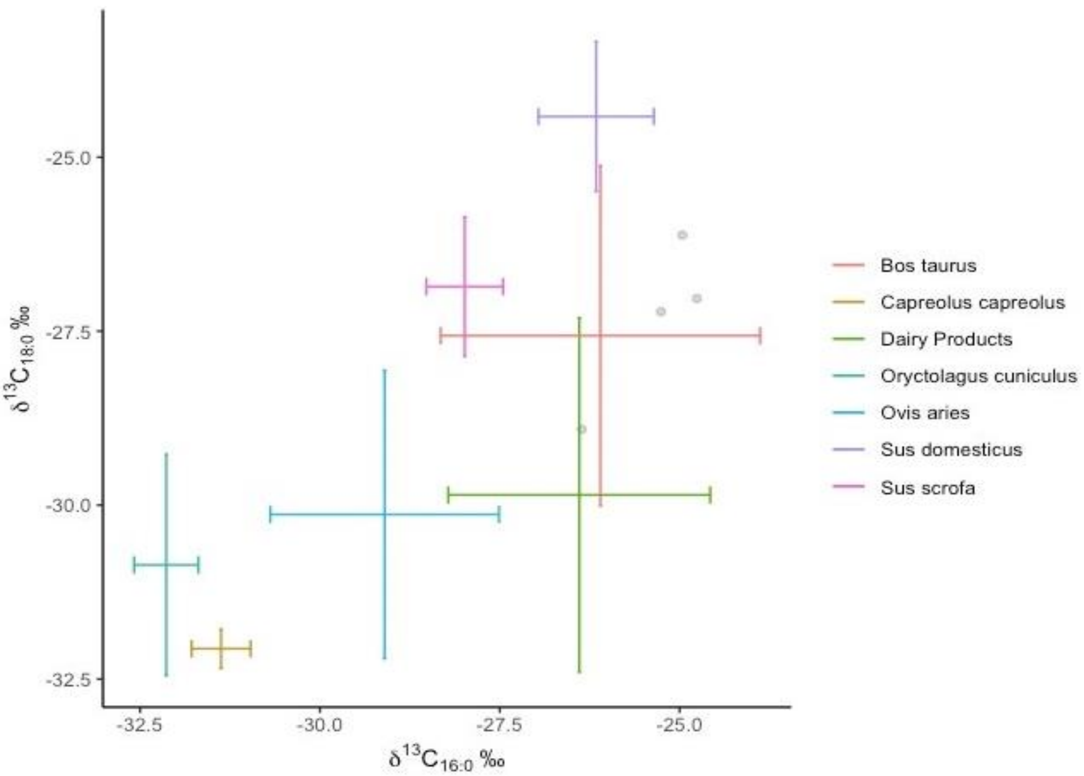


Figura 5.9. Distributions of intact triacylglycerols in total lipid extracts from absorbed and carbonized residues from Cova del Sardo sherds. The relative abundance of each component was calculated by measuring the peak area in the GC-MS profile.

The three samples from Cova del Sardo containing triacylglycerols (Figure 5.9), SAR02 presents a distribution between T_{46} and T_{54} with predominance of T_{50} and T_{52}, triglyceride characteristics of ruminant origin. The SAR11 and SAR04 samples have homogeneous distribution of T_{50} and T_{52}, from ruminant animal fat, but the percentage is quite high and T_{54} can establish the presence of porcine in these two samples.

The carbon isotope composition of the organic residues in vessels from Cova del Sardo reflect the average $\delta^{13}C$ values of the degraded fats during vessel use. The $\delta^{13}C$ values of the major fatty acids ($C_{16:0}$ and $C_{18:0}$) were determined for the four samples that yielded extracts by the GC-C-IRMS technique.



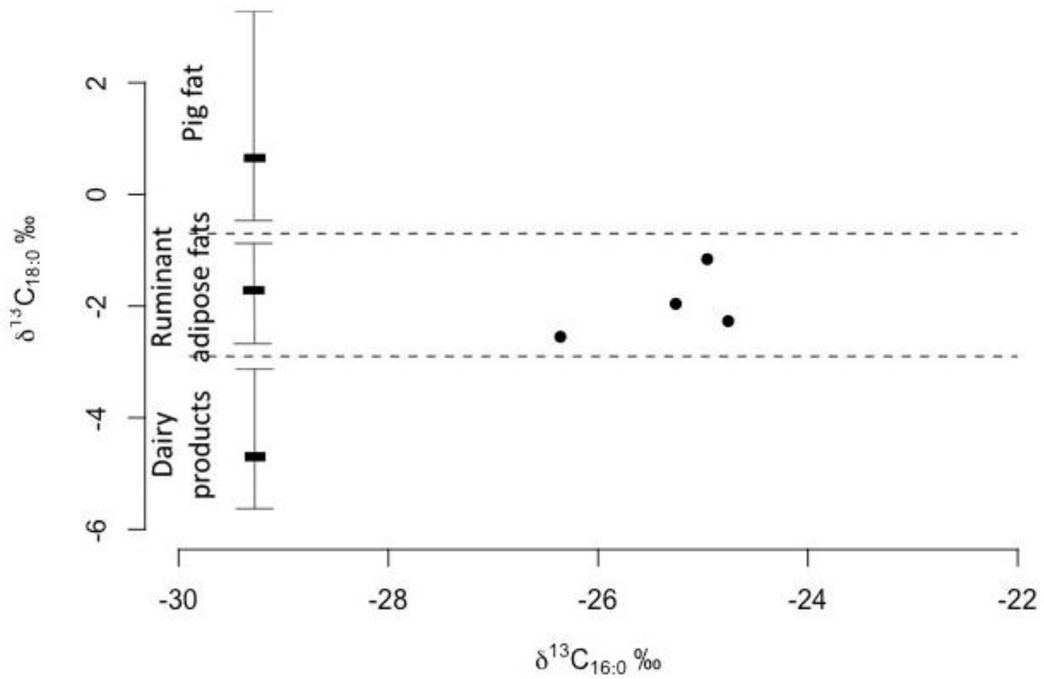


Figure 5.10. Scatter plot showing δ^{13}C $C_{16:0}$ (X) and δ^{13}C $C_{18:0}$ (Y) values and δ^{13}C $C_{16:0}$ and Δ^{13}C values from Cova del Sardo samples.

As we can see from figure 5.10, in 3 vessels analysed from Cova del Sardo by isotopic compounds $\delta^{13}C$, values $\delta^{13}C$ $C_{16:0}$ and $\delta^{13}C$ $C_{18:0}$ (Figure 5.10 left), as well as the $\Delta^{13}C$ index (Figure 5.10 right), are consistent with the presence of ruminant adipose fats.

5.1.3. Juberri

A total of 29 potsherds have been analysed at the three sites from Juberri: 'Camp del Colomer', 'Carrer Llinàs 28' and 'Feixa del Moro'. By means of a first extraction with organic solvents, organic compounds were detected in 8 of the analysed potsherds, with an average TLE of 22.3 $\mu g \cdot g^{-1}$. In order to increase the number of results with sufficient lipid concentrations to perform an interpretation, the samples were re-extracted with negligible amounts of lipids by acidified methanol extraction. This increased the number of interpretable samples to 19 (65.5%), with an average TLE of 214.07 $\mu g \cdot g^{-1}$.

Site	Sample	TLE (µg·g-1)	Lipids detected	$\delta^{13}C$ C16:0 ±0,3 (‰)	$\delta^{13}C$ C18:0 ±0,3 (‰)	Pedominant commodity type
CAMP DEL COLOMER	JBA01	25,30	C16:0 - C18:0	-28,28	-29,29	Ruminant adipose fat
	JBA02	10,60	C16:0 - C18:0	-23,96	-22,85	Pig adipose fat
	JBA04	28,70	C16:0 - C20:0, Alcohols, DAGs	-28,73	-32,62	Dairy product
	JBA10	49,74	C16:0 - C20:0, β-sitosterol, DAGs	-32,34	-35,18	Dairy product
	JBA11	63,19	C16:0 - C18:0, Alcohols	-29,58	-31,84	Wild ruminant adipose fat
	JBA13	24,50	C16:0 - C18:0	-24,16	-24,07	Pig adipose fat
	JBA14	7,80	C16:0 - C18:0	-21,78	-22,22	Pig adipose fat
	JBA28	73,20	C16:0 - C18:0	-27,17	-25,94	Wild boar adipose fat
CARRER LLINÀS 28	JBA12	33,80	C16:0 - C18:0	-30,75	-31,75	Ruminant adipose fat
	JBA19	21,30	C16:0 - C18:0	-25,29	-24,45	Pig adipose fat
	JBA21	10,69	C16:0 - C18:0	-29,31	-29,99	Ruminant adipose fat
	JBA23	32,15	C16:0 - C18:0	-28,50	-30,61	Ruminant adipose fat
	JBA26	78,90	C16:0 - C18:0	-24,08	-26,82	Dairy product
	JBA27	45,60	C16:0 - C18:0	-23,13	-22,74	Pig adipose fat
	JBA29	20,71	C16:0 - C18:0	-28,46	-30,22	Ruminant adipose fat
FEIXA DEL MORO	JBA18	64,50	C16:0 - C18:0, C18:1	-21,03	-22,83	Ruminant adipose fat
	JBA24	66,50	C16:0 - C18:0	-22,09	-21,76	Pig adipose fat
	JBA25	53,20	C16:0 - C18:0	-26,66	-27,17	Ruminant adipose fat

Table 5.3. Organic residues analysis from Juberri results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.1.3.1. Camp del Colomer

Interpretable organic compounds have been identified in 8 of the 14 vessels analysed (57%) from the archaeological intervention carried out in Camp del Colomer.

In all these samples the saturated mid-chain length *n*-alkanoic acids (fatty acids) were particularly dominated by $C_{16:0}$ (palmitic acid) and $C_{18:0}$ (stearic acid). The recovery rate of organic compounds in Camp del Colomer was low and DAGs and MAGs were degraded. In the JBA04 sample there were alcohols that, in the absence of other biomarkers, did not allow any interpretation. The same happens with the ß-sitosterol identified in the JBA10 sample, since no other biomarkers have been detected that reinforce the input of plant lipids hypothesis.

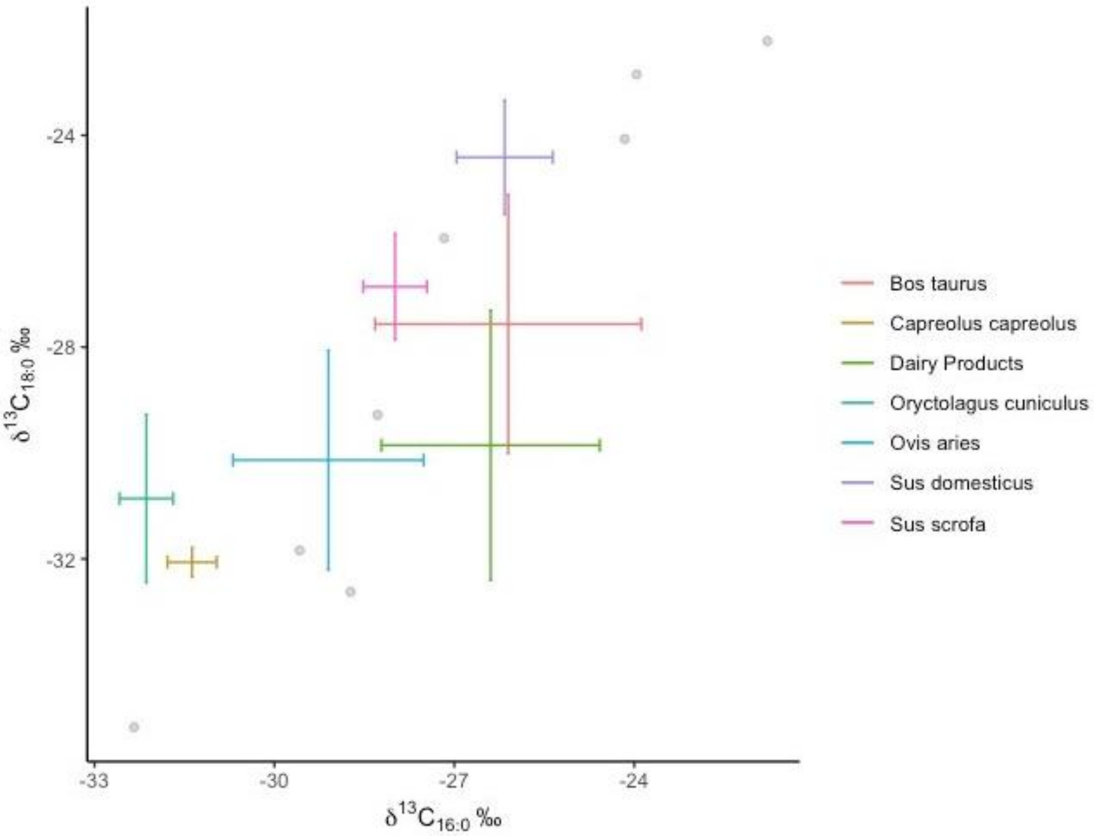


Figure 5.11. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ (X) and $\delta^{13}C$ $C_{18:0}$ (Y) values and $\delta^{13}C$ $C_{16:0}$ from Camp del Colomer samples.

From the identification of the isotopic compound of stearic and palmitic acid detected in 8 of the vessels coming from Camp del Colomer, domestic pig adipose fats were detected (JBA02, JBA13 and JBA14), in the JBA01 sample of domestic ruminant, in the JBA11 sample of wild ruminant, in the JBA28 sample of wild boar and the JBA04 and JBA10 samples dairy products were identified (Figura 5.11).

5.1.3.2. Carrer Llinàs 28

Organic compounds have been recovered in 7 of the 11 vessels analysed (55%) from the archaeological intervention carried out in Carrer Llinàs 28. Trace amounts of palmitic and stearic acid were detected in 7 vessels in amounts greater than 5 $\mu g \cdot g^{-1}$.

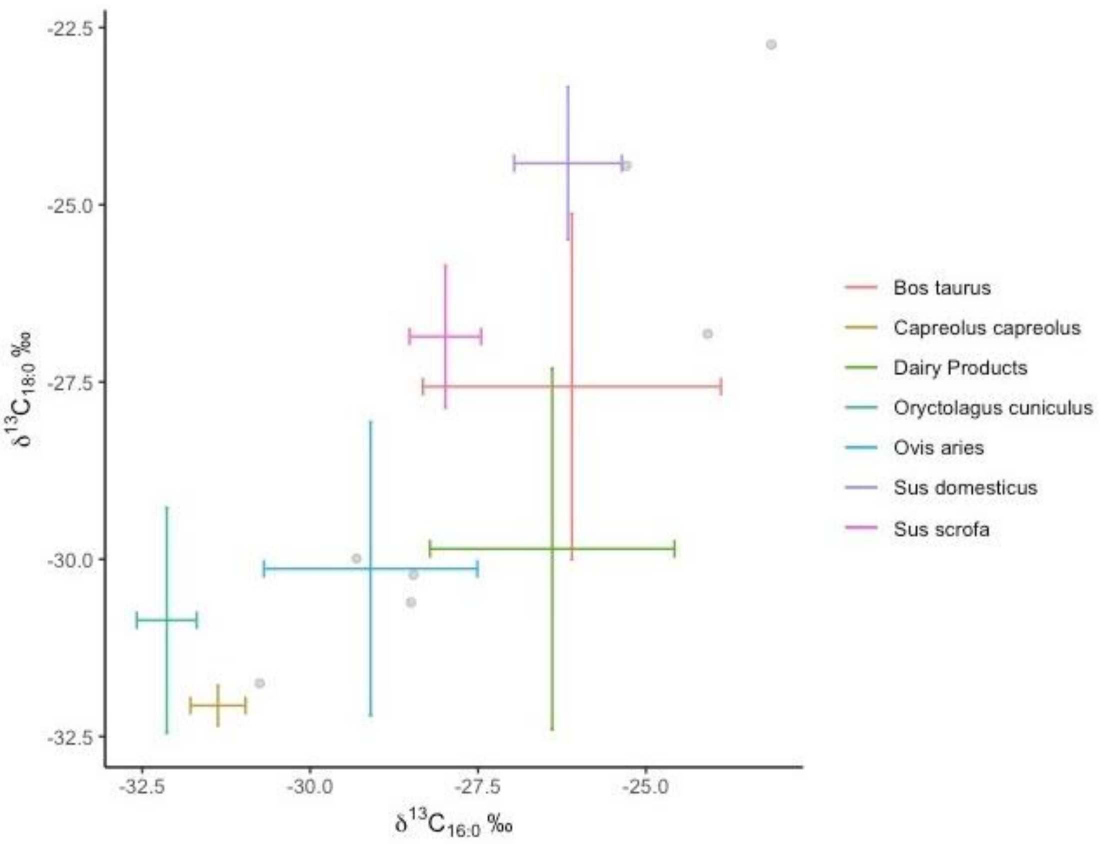


Figure 5.12. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ (X) and $\delta^{13}C$ $C_{18:0}$ (Y) values and $\delta^{13}C$ $C_{16:0}$ from Carrer Llinàs 28 samples.

Analysis by GC-IRMS has made it possible to determine fats from domestic ruminants in samples JBA12, JBA21, JBA23 and JBA29, from domestic pigs in samples JBA19 and JBA27 and from dairy products in sample JBA26 (Figure 5.12).

5.1.3.3. Feixa del Moro

Organic compounds have been identified in 3 of the 4 vessels analysed (75%) from the archaeological intervention carried out in Feixa del Moro.

In the 3 ceramic vessels from Feixa del Moro we identified ranges of saturated mid-chain length fatty acids, between $C_{14:0}$ to $C_{18:0}$, particularly dominated by $C_{16:0}$ (palmitic acid) and $C_{18:0}$ (stearic acid). In the JBA18 sample, large amounts of oleic acid ($C_{18:1}$) were identified without other biomarkers that reinforce a vegetable origin. However, in the rumen of ruminants there is a process of biohydrogenation of dietary fats leading to the formation of various $C_{18:1}$ isomer (Evershed et al. 1997; Regert, 2011). This fact has led to the proposal that the occurrence of the $C_{18:1}$ isomer may be indicative of the presence of ruminant adipose fat in the original ceramic vessel represented by sample JBA18. (Figure 5.13). The P/S ratio is also consistent with an interpretation of the presence of animal fats, given the predominance of stearic acid over palmitic acid.

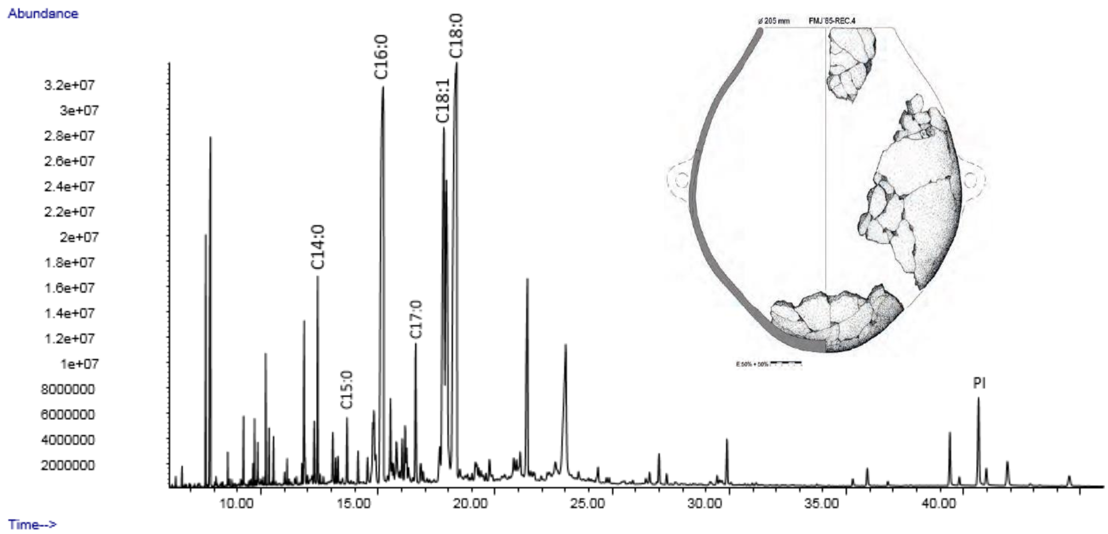


Figura 5.13. Cromatograma de la muestra JBA18, dónde se identifican ácidos grasos de origen animal.

The analyses by by GC-C-IRMS (Figure 5.14) further confirm the presence of domestic pig adipose fat in the JBA24 sample and the presence of domestic ruminant adipose fat in the JBA18 and JBA25 samples.

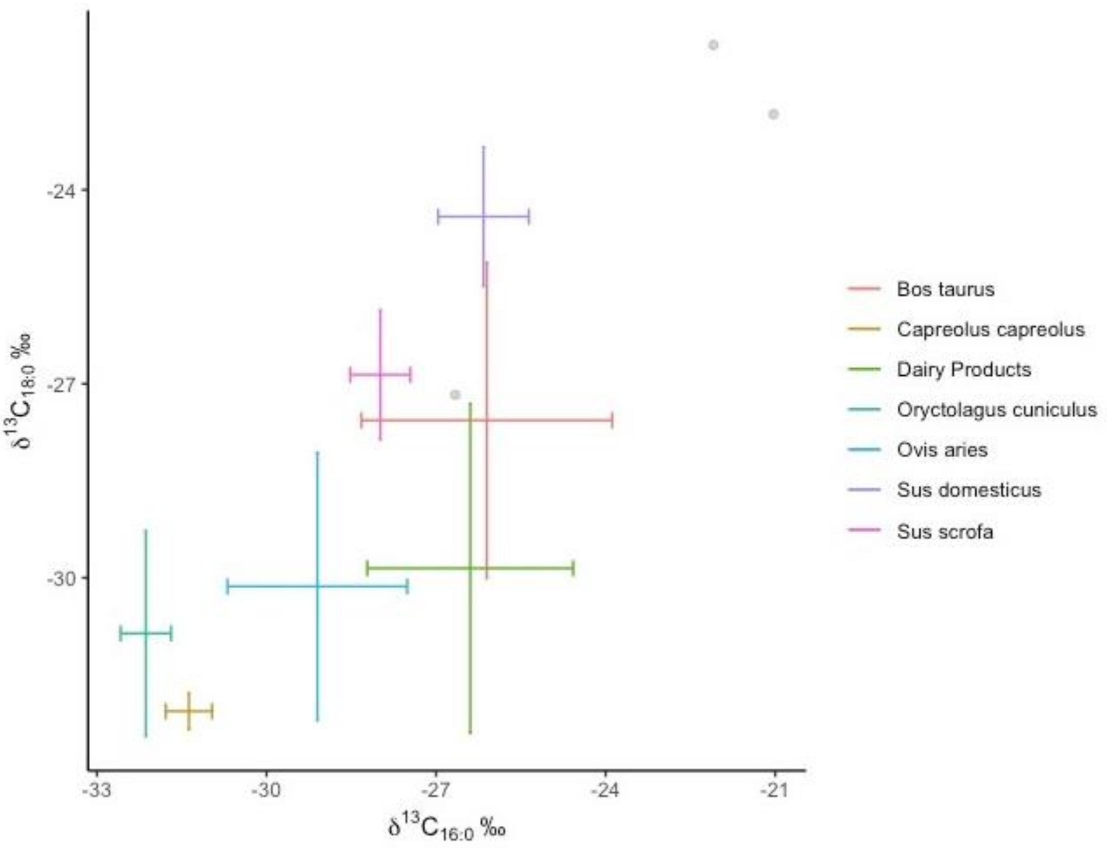


Figure 5.14. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ (X) and $\delta^{13}C$ $C_{18:0}$ (Y) values and $\delta^{13}C$ $C_{16:0}$ from Feixa del Moro samples.

The evaluation of the results obtained in the analysis of containers from Feixa Moro shows the presence of animal fats inside three of the large containers analysed. The absence of ketones and the volume of the containers leads us to think that they would be fats that would cover the internal surface of the container, or fresh meat of domestic animals.

The use of large ceramic containers to contain meat resources could be related to the need to conserve and store them for an indeterminate period of time.

5.2. Northwest of Iberian Peninsula

5.2.1. La Draga

Twenty-six vessels from levels III, VIa, VII and VIII of the La Draga site were analysed. The vessel from level III did not present alipid concentration. On the contrary, in the vessels coming from the rest of the anaerobic context levels, fatty acids from animal and plant origin were identified (n=27), which also presented tracylglycerides in ten samples (Table 5.4).

Sample	TLE (µg·g-1)	Lipids detected	δ^{13}C C16:0 ±0,3 (‰)	δ^{13}C C18:0 ±0,3 (‰)	Pedominant commodity type
D1		C10:0-C26:0, C16:1, C18:1, TAGs	-	-	Subcutaneous animal fat
D2		C9:0-C26:0, TAGs	-26,52	-30,45	Dairy products
D3		C9:0-C26:0, C16:1, C18:1, TAGs	-	-	Dairy products
D4		C12:0-C26:0, C16:1, C18:1	-	-	Subcutaneous animal fat
D5		C14:0-C26:0	-	-	Subcutaneous animal fat
D6		C8:0-C24:0, C16:1, C18:1, TAGs	-25,87	-26,48	Domestic ruminant fat
D7		C12:0-C26:0, C16:1, C18:1, TAGs	-	-	Subcutaneous animal fat
D8		C12:0-C18:0, TAGs	-	-	Subcutaneous animal fat
D9		C10:0-C30:0, C16:1, C18:1	-	-	Subcutaneous animal fat
D10		C9:0-C28:0, C16:1, C18:1	-	-	Subcutaneous animal fat
D11		C14:0-C26:0, C16:1, C18:1, TAGs	-	-	Subcutaneous animal fat
D12		C10:0-C30:0, C16:1, C18:1	-	-	Subcutaneous animal fat
D13		C12:0-C28:0, C16:1, C18:1, TAGs	-	-	Dairy products
D14		C12:0-C26:0, C16:1, C18:1, TAGs	-	-	Subcutaneous animal fat
D15		C9:0-C26:0, TAGs	-25,63	-31,29	Dairy products
D16		-	-	-	-
D17		C14:0-C24:0	-	-	Subcutaneous animal fat
D18		C14:0-C30:0	-	-	Subcutaneous animal fat
D19		C12:0-C18:0	-	-	Subcutaneous animal fat
D20		C12:0-C26:0	-	-	Subcutaneous animal fat
D21		C9:0-C18:0, C16:1, C18:1	-	-	Subcutaneous animal fat
D22		C16:0-C18:0	-	-	Subcutaneous animal fat
D23		C14:0-C18:0	-	-	Subcutaneous animal fat
D24		C9:0-C30:0	-	-	Subcutaneous animal fat
D25		C16:0-C30:0	-	-	Subcutaneous animal fat
D26		C12:0-C26:0	-	-	Subcutaneous animal fat
D27		C12:0-C26:0	-	-	Subcutaneous animal fat
D28		C9:0-C30:0	-	-	Subcutaneous animal fat

Table 5.4. Organic residues analysis from La Draga results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.2.1.1. Animal fats

Triacyglycerols were detected in 10 samples from La Draga (38.4%). Their presence indicates the occurrence of remains of animal fats in the original pottery. By means of the dispersion indexes and the average of carbons number presented by the triacylglycerides of these samples, we were able to get further evidence regarding the type of animal fats (Regert 2011) (Figure 6.3).

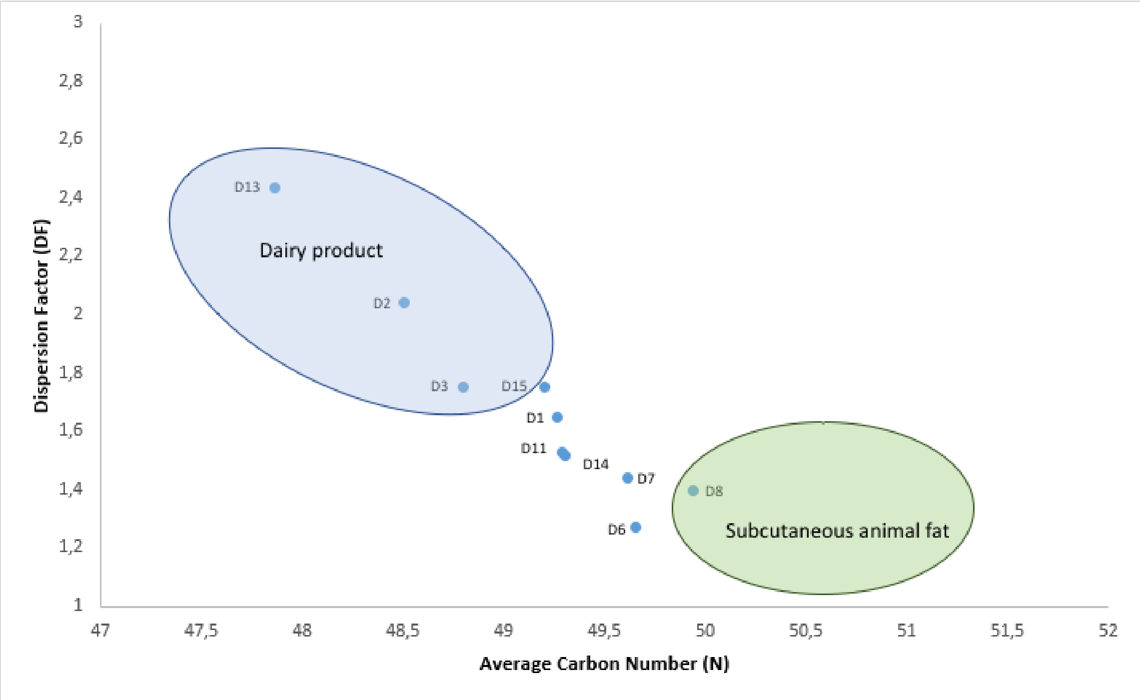


Figure 5.15. Dispersion chart of the classification of animal fats from La Draga, with reference ranges extracted from Mirabaud et al. 2007, Regert 2011.

The scatter plot (Figure 5.16) displays the values of the triacylglycerides of the 10 samples of La Draga analysed. The results show that at least 4 samples have triacylglycerides with profiles consistent with an origin from dairy products (D2, D3, D13 and D15), whereas one corresponds to subcutaneous animal fats (D8). The rest of samples yield TAG profiles that are ambiguous.

The stable carbon isotope compositions ($\delta^{13}C$ values) of the dominant fatty acids ($C_{16:0}$ and $C_{18:0}$) in La Draga pottery were determined to allow classification to commodity group (e.g. non-ruminant fat, ruminant adipose fat and ruminant dairy fat). Only 3 samples had fatty acids in enough concentrations to be analysed by GC-IRMS (>5 µg of lipids per gram). $\delta^{13}C$ values were compared to a modern reference animal fat database assembled from animals from Iberian Peninsula (Figure 5.16).

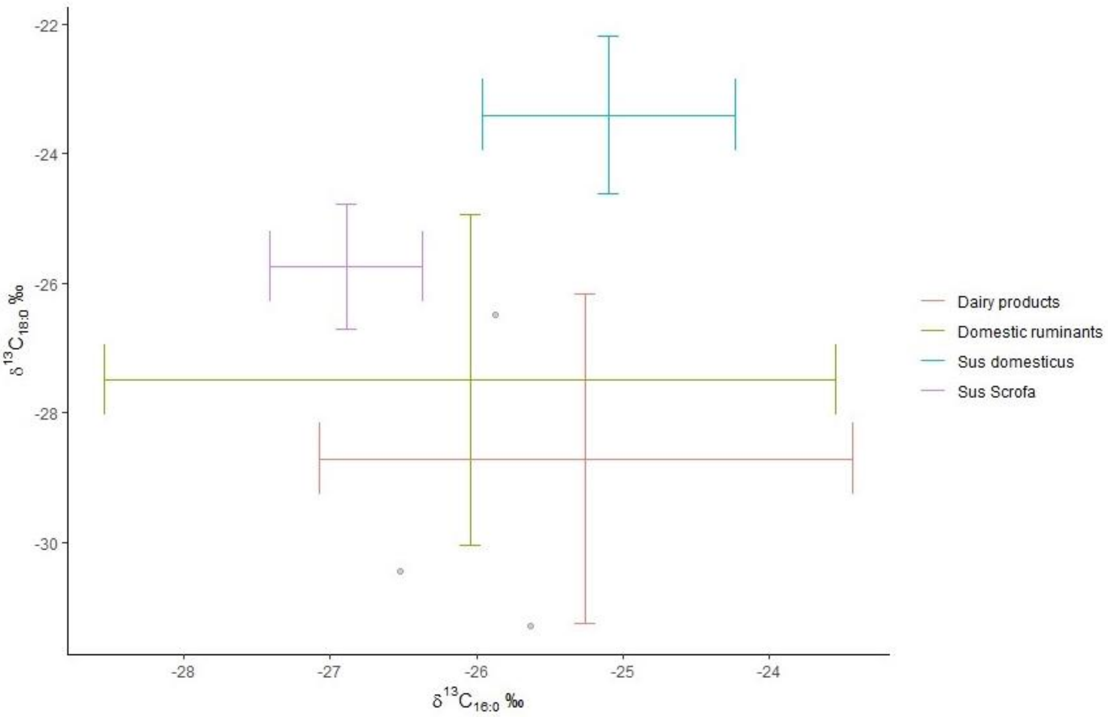


Figure 5.16. Scatter plot with $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ fatty acids from La Draga site compared with $\delta^{13}C$ values from modern reference animal fat.

Compound isotopic values of D2 and D15 are consistent with dairy products range, while D6 is consistent with ruminant fats. Hence, ca. 75% of all the animal fat residues detected were determined to be dairy fats.

5.2.1.2. Plant lipids

Higher concentrations of palmitic acid versus stearic acid (P/S) have been detected in all samples with interpretative amounts of organic compounds from La Draga, except in sample D8. This index may be indicative of the presence of plant lipids (Malainey et al. 1999b). In addition, the good preservation of organic matter in some of the La Draga samples has allowed the detection of unsaturated acids, such as $C_{16:1}$ and $C_{18:1}$, which helps us to interpretate the mixing of animal fats together with an input of plant fats (Evershed et al. 1999; Steele et al. 2010). In the absence of other biomarkers, such as plant sterols, the occurrence of lipids from higher plants in La Draga samples should be taken with caution.

5.2.2. Mines de Gavà

Fifteen of the twenty-eight sherds from Mines de Gavà (54%) contained significant amounts of lipids (i.e. higher than 5 µg·g^{-1}; Evershed 2008) (Table 5.5). Chromatograms are dominated by saturated fatty acids, mainly $C_{16:0}$ and $C_{18:0}$, sometimes with mono- and diacylglicerides, and animal or plant sterols.

Sample	TLE (µg·g-1)	Lipids detected	$\delta^{13}C$ C16:0 ±0,3 (‰)	C18:0 ±0,3 (‰)	Pedominant commodity type
MIG09	103,2	FA (16<18)	-25,10	-22,99	Non-ruminant adipose fat
MIG10	99,89	FA (16<18)	-22,94	-23,27	Non-ruminant adipose fat
MIG11	224,35	FA (16>18, 18:1); Sterols: β-sitosterol	-27,20	-26,83	Non-ruminant adipose fat
MIG12	115,91	FA (16>18), MAG, DAG	-28,93	-31,17	Ruminant adipose fat
MIG13	202,2	FA (16>18, 18:1, 20); Sterols: cholesterol, stigmasterol, β-sitosterol	-25,11	-25,27	Non-ruminant adipose fat Plants
MIG14	104,03	FA (16<18)	-24,50	-23,42	Non-ruminant adipose fat
MIG16	225,69	FA (14, 16>18, 20); Sterols: stigmasterol, β-sitosterol	-25,57	-25,57	Non-ruminant adipose fat
MIG17	89,71	FA (16>18)	-26,91	-29,23	Ruminant adipose fat
MIG18	89,01	Wax esters (42-50); Alcohols (28-32)	x	x	Beeswax
MIG19	232,65	FA (16>18)	-27,40	-29,88	Ruminant adipose fat
MIG20	187,4	FA (16<18)	-24,97	-24,60	Non-ruminant adipose fat
MIG21	408,36	FA (16>18), MAG, DAG. TAG; Sterols: β-sitosterol	-25,53	-25,67	Non-ruminant adipose fat
MIG22	545,1	FA (16<18), MAG, DAG. TAG	-24,85	-25,43	Non-ruminant adipose fat
MIG25	76,69	FA (16<18), MAG, DAG; Wax esters (40-52); Alcohols (26-32)	x	x	Beeswax Animal fat
MIG28	80,2	FA (16>18)	-27,50	-27,54	Ruminant adipose fat

Table 5.5. Organic residues analysis from Mines de Gavà results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.2.2.1. Animal fats

In Mines de Gavà, fatty acids dominated by C16:0 and C18:0 have been identified in 14 potsherds (50%). The good preservation of organic matter has allowed the conservation of mono- (MAGs), di- (DAGs) and in some cases triacyglycerides (TAGs) (MIG12 and MIG22), although their degradation does not allow the interpretation of their distribution. These compounds make it possible to interpret free fatty acids with an animal origin.

Fatty acid extracted from the vessels were analysed by GC-IRMS. The situation in the scatter plot of the $\delta^{13}C$ values obtained in the archaeological samples in relation to the modern values of animal fat from the Iberian Peninsula, allows to determine the origin of the identified lipids (Figure 5.17).

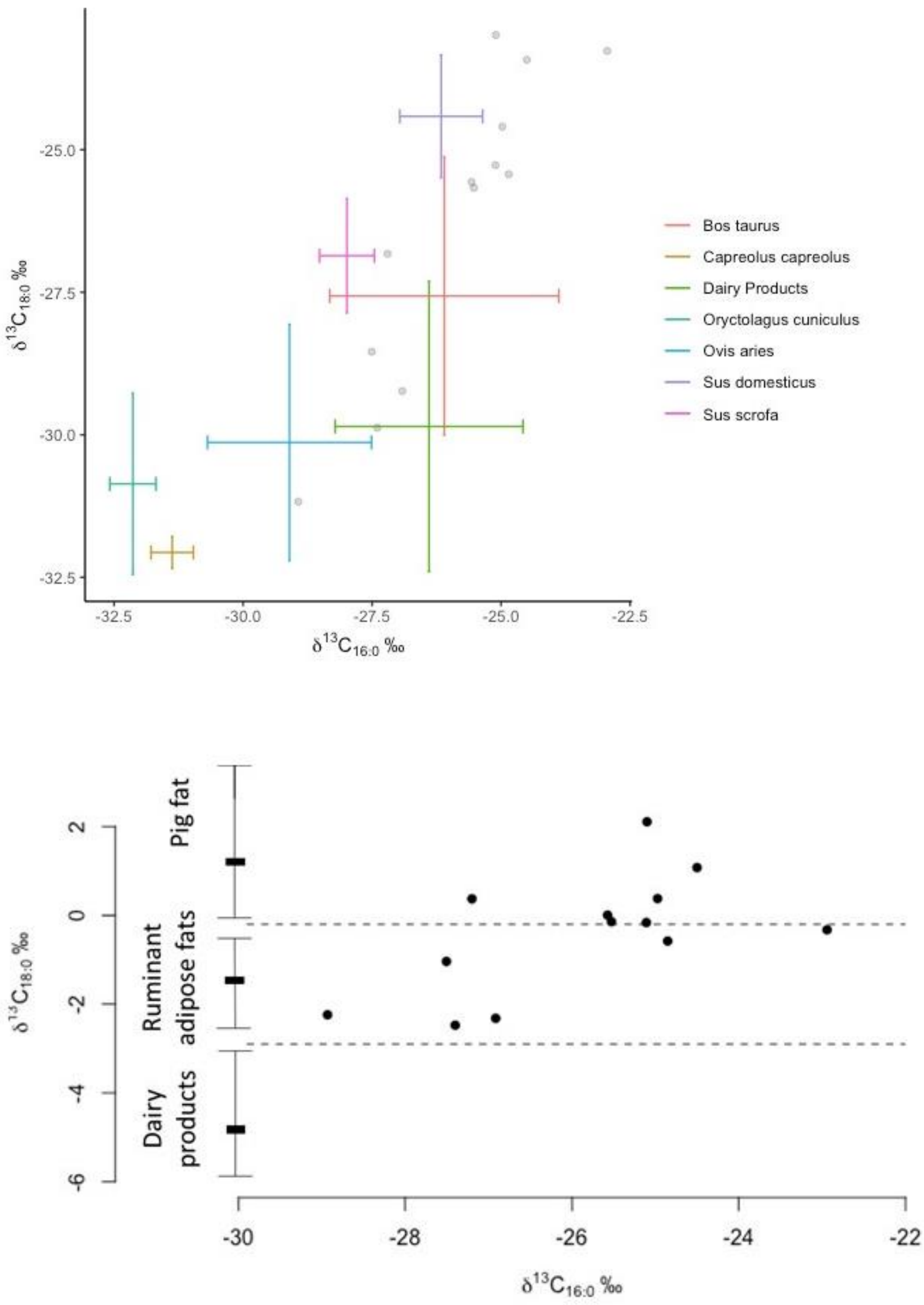


Figure 5.17. Scatter plot showing $\delta^{13}C$ $C_{16:0}$ (X) and $\delta^{13}C$ $C_{18:0}$ (Y) values and $\delta^{13}C$ $C_{16:0}$ and $\Delta^{13}C$ values from Mines de Gavà samples.

From the $\Delta^{13}C$ index we can see how the isotopic values of the fats identified in the MIG09, MIG10, MIG11, MIG13, MIG14, MIG16, MIG20 and MIG21 samples are consistent with modern non-ruminant reference values. The MIG22 sample is coherent with the non-ruminant and ruminant adipose fat ranges, which could be the result of a mixture. If we obtain the scatter plot with the differentiated animal fat values, we can see that the MIG11 sample is consistent with wild boar and the rest of the non-ruminants have values very close to pig adipose fats. In the case of MIG12, MIG17, MIG19 and MIG28 samples, the $\delta^{13}C$ values are consistent with ruminant adipose fats (Figure 5.17 top). Given the difficulty posed by the distinction between the adipose fats of sheep, goats and calves, the interpretation of the ruminant fats of the Mines de Gavà can be clarified as the fat of domestic ruminants (Figure 5.17 bottom).

5.2.2.2. Plant lipids

Considering the palmitic/stearic ratio (P/S), the very high abundance of $C_{18:0}$ in some samples suggests an animal origin for the lipids. Conversely, some other sherds contained high concentration of $C_{16:0}$ compared to $C_{18:0}$, they also presented unsaturated fatty acid ($C_{18:1}$) and plant sterols (mainly β-sitosterol and stigmasterol) (MIG11, MIG13, MIG16 and MIG21), which indicate the presence of plant products or the mixture of meat and plants inside the sherds (Dunne et al. 2016).

5.2.2.3. Beeswax

The MIG18 and MIG25 samples contain putative beeswax biomarkers. Beeswax can be characterised by the presence of even-numbered long-chain monoesters (W_{40}-W_{52}) derived from palmitic fatty acid ($C_{16:0}$), long-chain n-alkanes with odd-numbered carbon atoms ranging from C_{21} to C_{33} and long-chain alcohols (from AL_{24} to AL_{34}) (Garnier et al. 2002, Regert et al. 2001) (Figure 5.18).

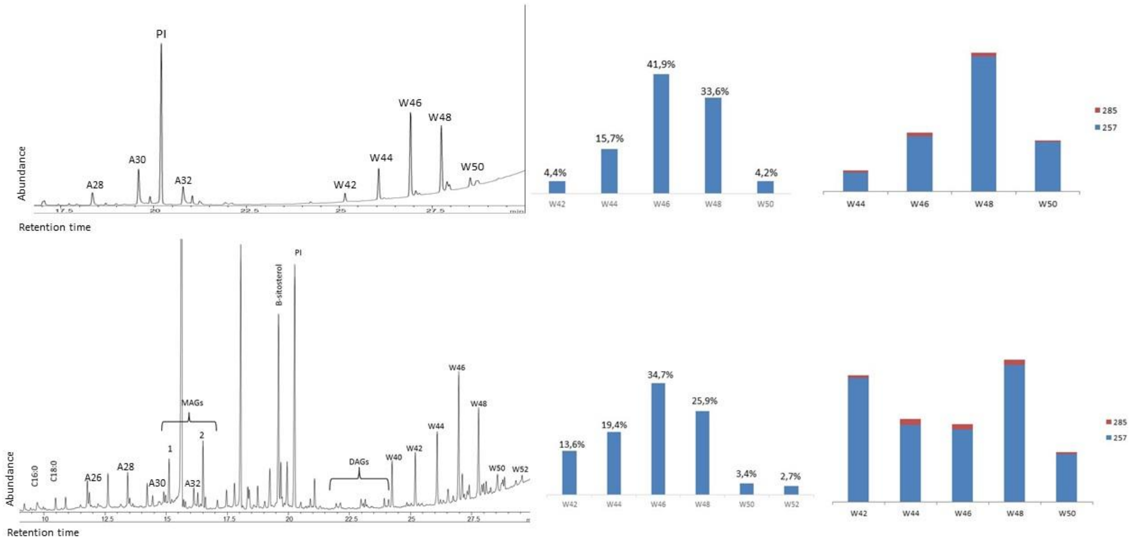


Figure 5.18. Chromatograms with percentage of wax esters identified and the proportion of the original acid (m/z 285 from $C_{18:0}$ and m/z 257 from $C_{16:0}$).

Some *n*-alcohols are present in modern beeswax at low levels (parts per million) but become major components in archaeological beeswax exposed to hydrolytic degradations (Garnier et al. 2002). Hydrolysis of wax esters to even-numbered long-chain alcohols, AL_{24}-AL_{34}, under burial conditions possibly occurs during diagenetic degradation of waxes (Evershed et al. 1997, Namdar et al. 2009). The degree of hydrolysis of wax esters however, increases with heating, but aging or heating process could also affects reversibly on the amount of n-alcohols produced by hydrolysis (Namdar et al. 2009).

5.3. Southeast of Iberian Peninsula

5.3.1. Cabecicos Negros

In this site, in the 29 ceramic fragments selected the first extraction performed by DCM/MeOH only yielded significant amount of lipids in one sample (CNP12). To evaluate the potential presence of other bound lipids, an acidified methanol extraction was also practiced, and lipids were obtained in 19 of the 29 potsherds analysed (65.5%). Fatty acids with animal origin were identified.

5.3.1.1. Animal fats

Cabecicos Negros potsherds revealed a range of saturated and unsaturated mid-chain length *n*-alkanoic acids (fatty acids) with even numbers of carbon atoms from $C_{12:0}$ to $C_{20:0}$, particularly dominated by $C_{16:0}$ and $C_{18:0}$ (Figure 5.19). The greater presence of palmitic acid versus stearic acid in some of the samples raised the possibility of the presence of plant lipids. However, the absence of oleic acid and plant sterols does not allow us to interpret a plant input. In fact, the interpretation of plan lipids from the P/S ratio alone can lead to confusion, since experimental tests have shown that product mixing and degradation can alter the value of the ratio (Dudd 1999: 233, Heron and Evershed 1993).

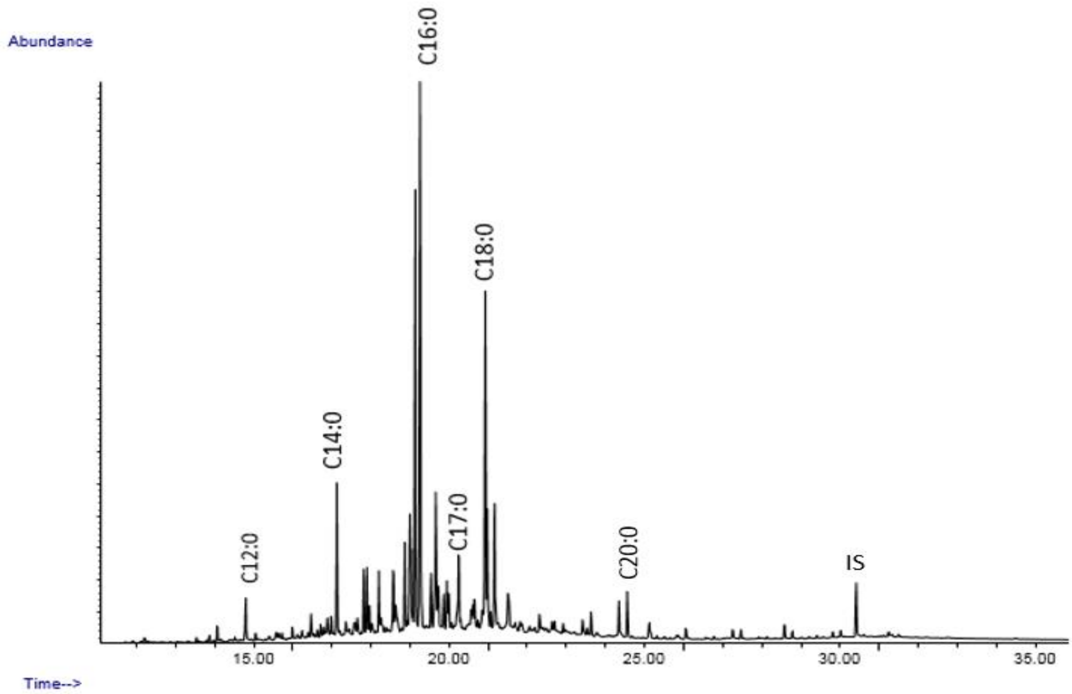


Figure 5.19. GC-MS chromatogram of the CNP21 sample extracted by H_2SO_4: MeOH.

Identification of the degraded animal fats recovered from the pottery took place through the determination of the $\delta^{13}C$ values of $C_{16:0}$ and $C_{18:0}$. Results were compared with fatty acid $\delta^{13}C$ values from modern reference adipose tissue (Dudd 1999, Copley *et al*. 2003, Gregg *et al*. 2009, Debono-Spiteri 2012) (Figure 5.20).

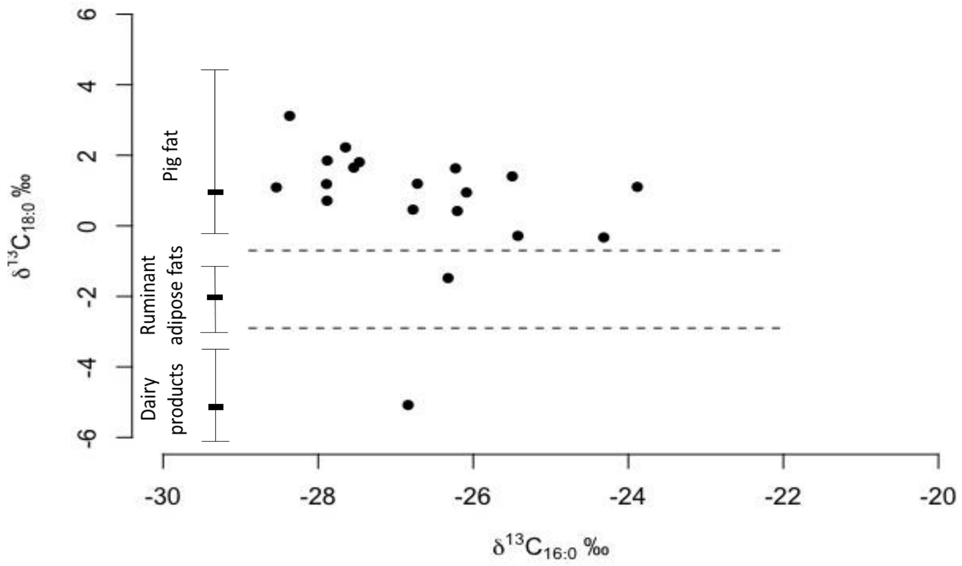


Figure 5.20. Scatter plot showing $\delta^{13}C_{16:0}$ (*X*) and $\Delta^{13}C$ values (*Y*) of fatty acids extracted from Cabecicos Negros site compared with $\delta^{13}C$ values from modern reference animal fat.

As we can see in figure 5.20, about 60% of the vessels analysed have a $\Delta^{13}C$ index consistent with non-ruminant adipose fat. Although values corresponding to ruminant adipose fat (CNP18) and dairy product fat (CNP12) have also been identified.

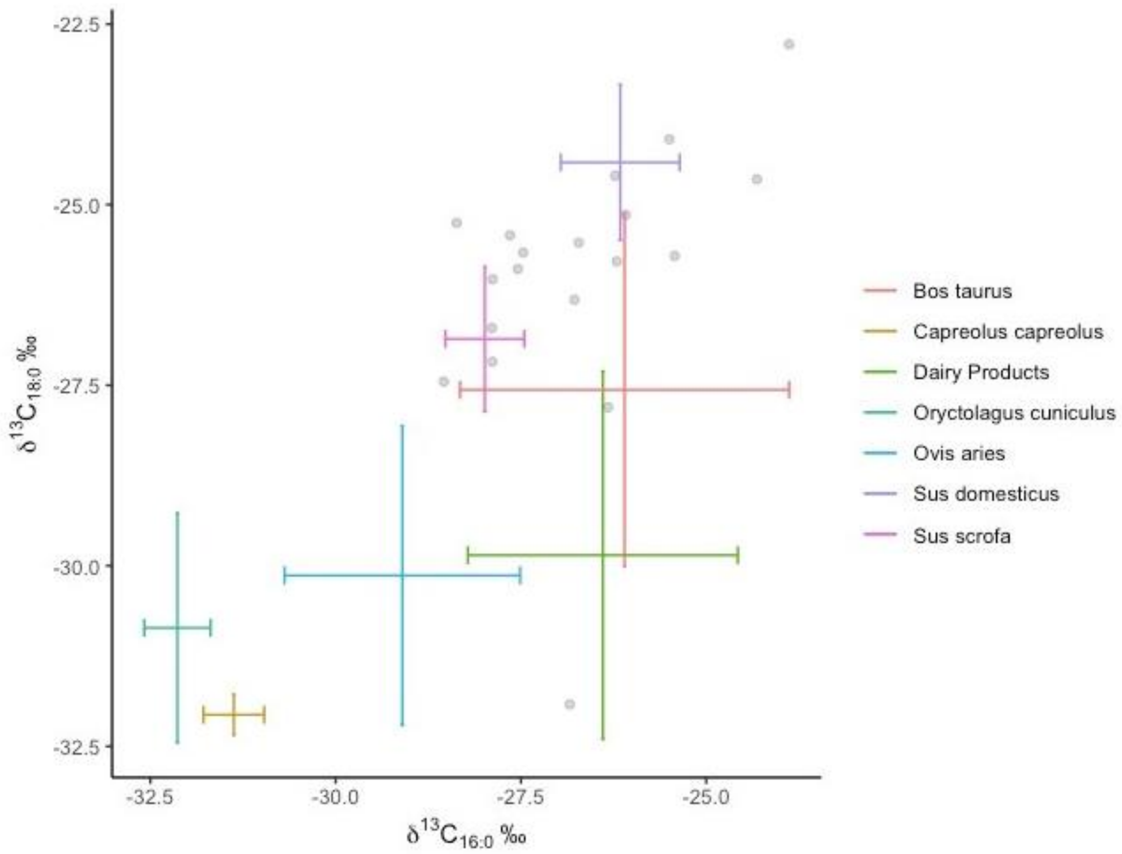


Figure 5.21. Scatter plot with $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ fatty acids from Cabecicos Negros site compared with $\delta^{13}C$ values from modern reference animal fat.

If we put the Cabecicos Negros values in the scatter plot $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ with the ranges of modern animal fats from the Iberian Peninsula (Figure 5.21), we observe that the CNP18 sample corresponds to domestic ruminant fat, while non-ruminants are distributed among the reference values of domestic pig and wild boar.

5.3.2. Cueva de El Toro

Fifty vessels from phase IV (5280-4780 2σ cal BC) and sub-phase IIIb (4250-3950 2σ cal BC) from the cave of Cueva de El Toro were analysed. Of the 24 sherds were animal fats were detected (48%), 14 come from phase IV and 10 from sub-phase IIIB. A total of 18 samples (11 samples from phase IV and 7 samples from sub-phase IIIB) were extracted by solvent extraction (TLE mean = 27.87 $\mu g \cdot g^{-1}$), while 6 samples (3 samples from phase IV and 3 samples from sub-phase IIIB) were re-extracted by using acidified methanol

(TLE mean = 542.53 $\mu g \cdot g^{-1}$). Among the biomarkers identified, the presence of animal fats, vegetables and resins has been detected inside the containers.

In samples CTM01 and CTM04, polycyclic polyaromatic hydrocarbons (PAHs), such as *Anthracene* and *Phenanthrene*, were identified. These volatile compounds are produced during the combustion of woody products over 300ºC (Kilops and Kilops 2005). In the case of Cueva de El Toro, the traces amount of PAHs could be related to the exposure of the container and its contents to a heat source, but in the absence of ketones that confirm the heat treatment of fats, we believe that PAH can come from exogenous contamination with the sediment.

Phase	Sample	TLE (μg·g-1)	Lipids detected	δ13C C16:0 ±0,3 (‰)	δ13C C18:0 ±0,3 (‰)	Pedominant commodity type
IV (5280-4780 2σ cal BC)	CTM03	11,36	FA (16<18); PAHs: phenanthrene, anthracene	-31,28	-32,94	Ruminant adipose fat
	CTM07	9,13	FA (16<18, 10, 12, 14)	-27,65	-25,18	Non-ruminant adipose fat
	CTM11	12,83	FA (16<18, 8, 10, 12, 14)	-24,07	-24,69	Non-ruminant adipose fat
	CTM12	32,84	FA (16>18, 12, 14, 18:1)	-23,82	-22,31	Non-ruminant adipose fat
	CTM16	22,4	FA (16>18)	-26,46	-26,61	Non-ruminant adipose fat
	CTM24	35,97	FA (16>18)	-31,65	-27,14	Non-ruminant adipose fat
	CTM32	78,4	FA (16<18, 10, 12, 14, 20); MAGs, DAGs, TAGs	-26,75	-30,13	Non-ruminant adipose fat
	CTM33	14,93	FA (16<18)	-27,58	-24,39	Non-ruminant adipose fat
	CTM35	58,41	FA (16<18)	-24,76	-23,88	Non-ruminant adipose fat
	CTM41	43,69	FA (16<18)	-27,14	-26,01	Non-ruminant adipose fat
	CTM42	15,3	FA (16<18)	-27,76	-27,54	Non-ruminant adipose fat
	CTM43	834,48	FA (8, 10, 12, 14, 16>18, 16:1, 18:1); Diterpens: dehydroabietic acid, abietic acid; Sterols: cholesterol	-24,42	-24,05	Non-ruminant adipose fat Pine resin
	CTM44	325,3	FA (8, 10, 12, 14, 16>18, 16:1, 18:1); Diterpens: dehydroabietic acid, abietic acid	-30,56	-32,43	Ruminant adipose fat Pine Resin
	CTM51	722,11	FA (14, 16>18, 20)	-27,55	-30,44	Dairy fat
IIIB (4250-3950 2σ cal BC)	CTM01	40,07	FA (16<18)	-28,61	-30,03	Ruminant adipose fat
	CTM04	374,25	FA (12, 14, 16>18, 16:1, 18:1, 20, 22, 24); PAHs: phenanthrene, anthracene; Sterols: ergosta-5,22-dien-3-ol, stigmastan-3,5-dien, cholesterol, stigmasterol, β-sitosterol, retinoic acid	-25,83	-26,22	Non-ruminant adipose fat Plants
	CTM05	461,85	FA (12, 14, 16>18, 16:1, 18:1, 20, 22, 24, 26); Sterols: ergosta-5,22-dien-3-ol, stigmastan-3,5-dien, cholesterol, stigmasterol, β-sitosterol, retinoic acid	-27,11	-28,38	Ruminant adipose fat Plants
	CTM09	46,46	FA (16<18)	-25,95	-25,73	Non-ruminant adipose fat
	CTM13	21,31	FA (16>18, 12, 14, 18:1)	-28,18	-30,20	Ruminant adipose fat
	CTM38	537,2	FA (12, 14, 16>18, 18:1, 20, 22, 24, 26, 28); Sterols: ergosta-5,22-dien-3-ol, stigmastan-3,5-dien,cholesterol, stigmasterol, β-sitosterol, retinoic acid	-28,40	-29,11	Ruminant adipose fat Plants
	CTM40	40,65	FA (16<18)	-25,82	-23,88	Non-ruminant adipose fat
	CTM46	53,22	FA (16<18)	-27,82	-28,33	Ruminant adipose fat
	CTM49	33,84	FA (16<18)	-27,56	-28,84	Ruminant adipose fat
	CTM50	78,36	FA (16<18)	-29,31	-30,37	Ruminant adipose fat

Table 5.6. Organic residues analysis from Cueva de El Toro results. Abbreviations stand for: FA, fatty acids; TLE, total lipid extract; AMS, accelerator mass spectrometer.

5.3.2.1. Animal fats

In 14 samples from phase IV the presence of high concentrations of palmitic ($C_{16:0}$) and stearic acid ($C_{18:0}$) were detected. These fatty acids are usually in the greatest abundance in archaeological lipid extracts with an even carbon number preference. Lipid profiles dominated by these fatty acids have been often observed in degraded animal fats (Copley et al. 2001). The presence of TAGs in the CTM32 sample, although in very low concentrations, allows the animal origin of the fats to be assured. In general, the P/S ratio is very balanced, with the exception of samples CTM12, CTM43, CTM44, CTM04, CTM13 and CTM38, although they also present biomarkers of vegetable origin.

To provide more specific information, 24 sherds with the most abundant $C_{16:0}$ and $C_{18:0}$ acids were selected for GC-C-IRMS analyses with the aim of distinguishing the origins of these compounds based on their stable carbon isotope value ($\delta^{13}C$).

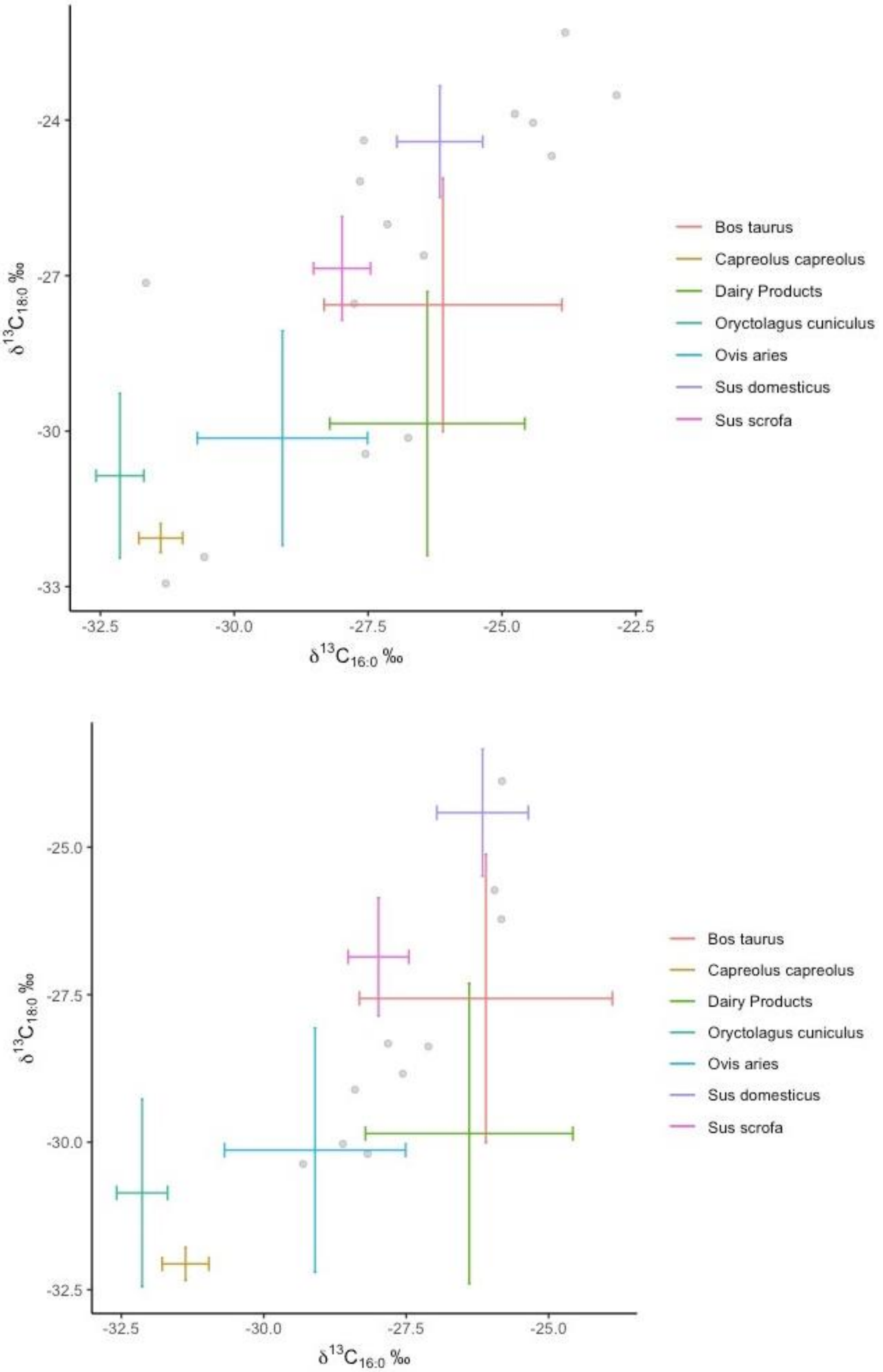


Figure 5.22. Scatter plot with $\delta^{13}C$ values of the $C_{16:0}$ and $C_{18:0}$ fatty acids from Cueva de El Toro site compared with $\delta^{13}C$ values from modern reference animal fat. Top figure corresponds to Phase IV, and bottom figure corresponds to sub-phase IIIb.

The situation in the scatter plot of the obtained values of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ is compared with the modern animal fats from Iberian Peninsula. The situation in the scatter plot of the values obtained in the archaeological samples in relation to the modern values of lipids, allows to determine the origin of the identified lipids. The results of the $\delta^{13}C$ values (Figure 5.22 top) reveal that sherds from phase IV dominates the consumption of pig adipose fats (samples CTM07, CTM11, CTM12, CTM16, CTM24, CTM33, CTM35, CTM41, CTM42 and CTM43), followed by the consumption of domestic ruminants adipose fats (samples CTM03 and CTM44) and dairy product fats (samples CTM32 and CTM51).

In 10 sherds from sub-phase IIIb animal fatty acids (predominated by $C_{16:0}$ and $C_{18:0}$) were detected. From the determination of the isotopic value of the carbon from fatty acids ($\delta^{13}C$) (Figure 5.22 bottom), it can be seen that in sub-phase IIIB, predominate those that have fatty fats of ruminant adipose fats (samples CTM01, CTM05, CTM13, CTM38, CTM46, CTM49 and CTM50), being lower the presence of sherds containing non-ruminant adipose fats (samples CTM04, CTM09 and CTM40).

5.3.2.2. Plant lipids

Plant biomarkers have also been identified in samples CTM04, CTM05 and CTM38 from phase IIIb. The characterisation of plant products has been possible thanks to the high concentration of these samples of palmitic acid ($C_{16:0}$) and oleic acid ($C_{18:1}$), as well as the vegetal sterols *Ergosta-5,22-dien-3-ol, Stigmastam-3,5-dien, Stigmasterol* y *β-sitosterol* (Evershed et al. 1991; Cert et al. 1994).

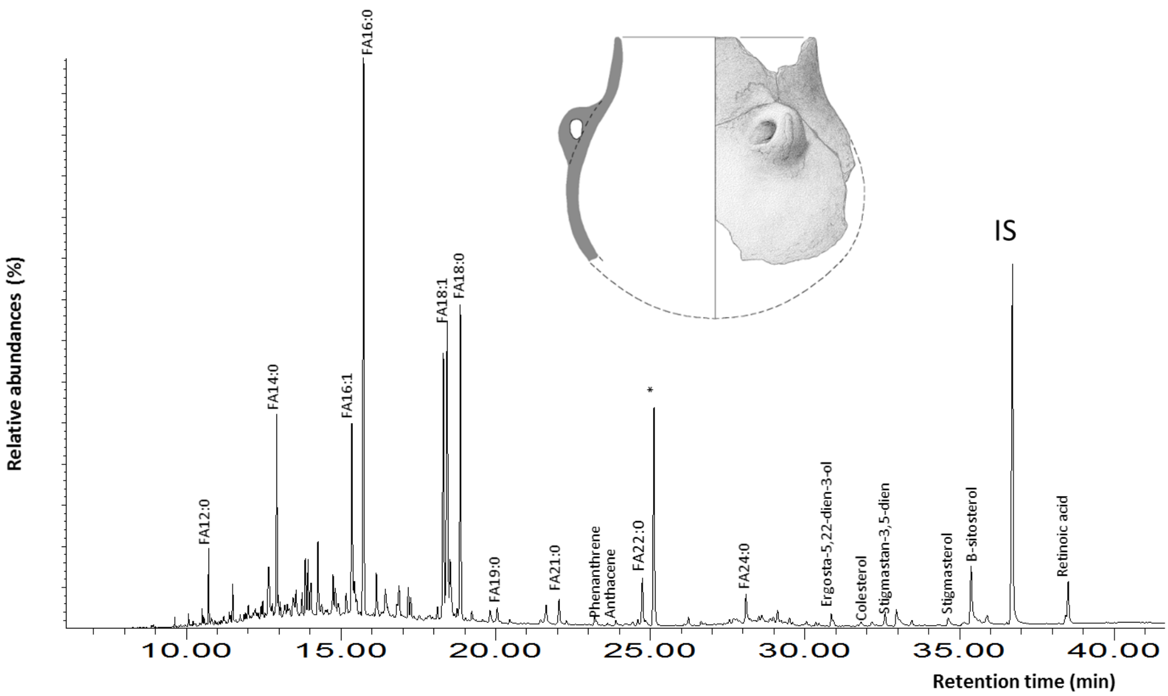


Figure 5.23. GC-MS chromatogram of the CTM04 sample extracted by H_2SO_4: MeOH.

Vegetable oils are present in large quantities in certain seeds or fruits (Marinval 2005; Evershed and Lockheart 2007). In sub-phase IIIB of Cueva de El Toro a large number of carbonised cereal seeds and legumes were found, as well as wild fruits, such as olives, acorns and myrtle and poppies (*Papaver somniferum* ssp. *Somniferum*), which, in addition to its use as a synanthropic plant, the possibility of using its seeds to obtain oil is also pointed out (Buxó 2004; Guerra-Doce and López-Sáez 2006; Rovira-Buendía 2007; Martín-Socas et al. 2018). The biomarkers of vegetable oils preserved in the vessels of Cueva de El Toro could be evidence of the processing of some of these species, which could have been roasted, boiled or bathed in water (Hally 1986; Amouretti 2005; Mason and Nesbitt 2009). These procedures allow eliminating the toxic tannins of some fruits, making them suitable for human consumption (Saul et al. 2012).

In the samples of Cueva de El Toro in which substances with vegetable origin have been identified, the presence of animal fats has also been detected. This makes possible to raise the possibility that the mixture of these products could have been intentional with the purpose to enhance the taste of food, some containers were used in the processing of various products at different times.

5.3.2.3. Resin

In samples CTM43 and CTM44, where non-ruminant and ruminant fats have been detected respectively, biomarkers of vegetable resin have also been identified, such as dehydroabietic acid and abietic acid (figure 5.24). The natural resins are exudates from the tree that, in certain cases and when are exposed to the light and air for a long period of time, undergo an oxidation process, as happens for example with pine resin (Azemard et al. 2016). The same oxidation effect can also occur when the resins are exposed to a heat source (Marchand-Geneste 2003). The oxidation process causes the apparition of specific compounds (diterpenes and dehydroabietic acid) which serve as biomarkers of vegetal resins.

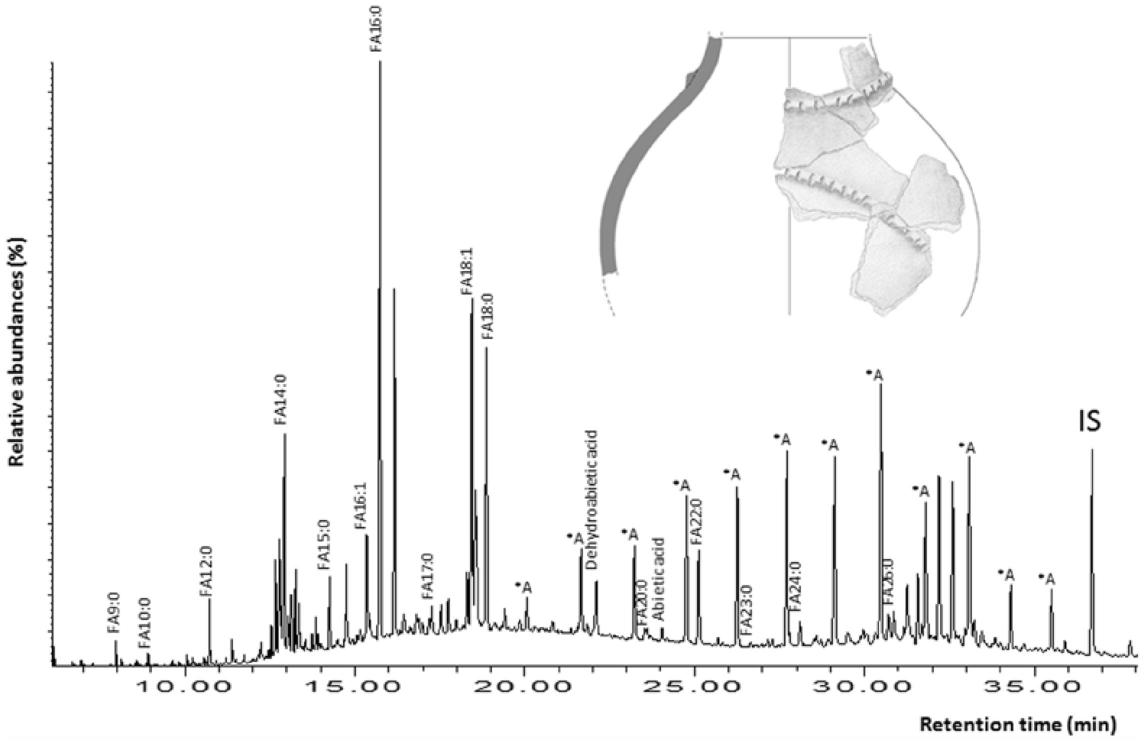


Figure 5.24. Partial gas chromatogram of the trimethylsilylated TLEs from CTM44 pottery sample showing the various biomarkers detected: animal fats (FA*x:y* are fatty acids where *x* is the carbon chain length and *y* is the degree of unsaturation), pine resin (dehydroabietic and abietic acid) and contamination from plants and handling (unsaturated fatty acids and **A* are a long chain of alkanes, with odd-numbered carbon dominance).

The characterization of the abietic acid, dehydroabietic acid and 7-oxo-dehydroabietic acid in the potsherds analysed indicate that the dehydrogenation of the compounds explains the application of more than 110ºC to melt the resin and to be able to apply it on the surface as a post-cooking treatment in order to waterproof the walls of the sherds (Charters et al. 1995, Pietrzak, 2012) in order to develop an effective use probably related to the storage or processing of animal fats in liquid or semiliquid state (Correa-Ascencio et al. 2014).

Chapter VI. Subsistence and product acquisition strategies in the Early Neolithic and their representation in organic residues in ceramics

In the first centuries of the Neolithic, there was a change in the relationship between society and the environment, characterized by a transformation in the way humans managed and exploited natural resources. Linked to the Neolithic origin, the appropriation and direct intervention on the reproductive rhythms of animal and plants show the scope of the new socio-economic system (Saña 1999). In this framework, the domestication process allowed for the exploitation of natural resources as means of production, while the relations of production changed according to the new forms of appropriation of these resources (Ingold 1984, 1988, Saña 1999, 2005).

The chronological variability documented in the Iberian Peninsula on the adoption of different domestic species may have been influenced by the variability of different factors, such as climate or orography, which may interfere with the adaptation of domestic species in the territory, as well as the type of livestock farming. These types of livestock could be mixed or specialized in the exploitation of specific products, or in the degree of incidence, with intensive or extensive livestock practices. Traditionally, it has been pointed out that in mountain areas hunting would predominate unlike in the plains or river valleys (Llovera 1986, Yáñez et al. 2002, Yáñez 2005, Orengo et al. 2014, Walsh et al. 2005). A greater or lesser importance of hunting and livestock has also been linked to the types of site (cave versus open-air). However, assessing the importance of certain domestic species at the beginning of the Neolithic is sometimes difficult due to the difficulty of separating between domestic and wild. The most commonly used criterion is biometrics, although it often does not have sufficient resolution to become interpretative.

Studies related to the identification and classification of the fauna documented in the archaeological record have been fundamental to understanding the implementation of livestock practices in Neolithic communities. However, the representativeness of the sets and the subsequent differentiation between domestic species is affected by the degree of preservation of the sets. Thus, in some studies taxa are classified as *Sp.* due to the impossibility of identifying whether the species is domestic or wild.

In the Iberian Peninsula the dynamics of animal domestication are not developed in the same way throughout the territory. In general, the implementation of livestock practices reduces the evidence of hunting activities. Around 70% of faunal remains were identified as domestic animal species. They datedfrom 5700 cal BC, with predominance of sheep and goats in the early Neolithic period (Saña 2013, Sierra et al. 2019). While the northern peninsular area presents a clear exception, where more diverse strategies are evident with the simultaneous and exclusive exploitation of wild resources (Altuna 1972, 1980). The different management strategies of domestic animals in the Mediterranean coastcan be separated between the northeast of the peninsula, the east and the south of the peninsula. In the northeast of the peninsula, a percentage of domestic fauna of around 90% is documented (Navarrete and Saña 2014, Saña et al. 2015), based on the remains of ovicaprines (Colominas et al. 2008, Bordas 2013, Saña et al. 2015) and bovines (Navarrete and Saña 2014, Saña and Navarrete 2016). In most of these sites, the wild species documented are wild boar, roe deer and deer. In the eastern zone, sheep predominate over goats (Pérez-Ripoll 1977, 1980), with the percentage of domestic animals at around 50% of the total number of documented species. Among the wild animals present in the sites of this region are the deer and the mountain goat (Navarrete 2015). Finally, few data are available on livestock exploitation in the south of the peninsula. Among the sites studied, the percentage of domestic animals ranges between 60 and 69%, with the predominant presence of ovicaprines (Saña 2013). Wild species include rabbits, wild goats and deer.

From the Middle Neolithic (4500-2500 cal BC) onwards, ovicaprines take a back seat to the increase in the presence of domestic species, with the ox and pig being the animals that increase their relative importance (Saña 2013). This leads to a decrease in hunting, although in the faunal groups of the sites dated between these chronologies there is evidence of mixed exploitation between domestic and wild, such as wild boar, deer and roe deer (Navarrete 2014).

As mentioned above, the documented variability in the importance of hunting and stockbreeding has sometimes also been attributed to orographic characteristics or to climate differentiating between mountainous regions and plains or river valleys. The works carried out in the Ebro valley document a percentage of domestication between 60 and 90%, with a strong presence of ox and sheep (Castaños 2004, Altuna 2001), as well as the recent data obtained from the studies of the faunal remains in the Cueva de Chaves (Sierra et al. 2019). However, in mountainous regions, the presence of domestic herds has tended to be related to seasonal movements or displacements linked to the search for food (transhumance). The greater presence of wild animal remains in the caves has also been related to population models that contemplate the simultaneous use by a community of several complementary settlements, some of a more permanent nature, outdoors, and others of a more specific or seasonal nature, which would correspond to the caves (Bosch 1991, Molist et al. 2003). Although it is true that cave sites such as Cova del Frare (Martín 2011) have been related to the practice of seasonal transhumance and hunting, a high degree of domestication and sedentarisation has been observed in other cave sites such as El Toro (Martín-Socas et al. 2004) or Coro Trasito (Gassiot et al. 2016).

Subsistence strategies have been widely approached from archeobotany and archaeozoology in the Iberian Peninsula (Saña 2013, Antolín et al. 2015, Navarrete et al. 2018). However, the analysis of organic residues eventually preserved in ceramic containers can complement the information we have. By identifying and characterising the origin of the animal fats and vegetable resources that were processed inside the ceramic containers, we aim to provide data on acquisition and subsistence strategies in sites where we have little archaeozoological data, such as the south of the peninsula or the Pyrenees. Because of the acidity of some of the peninsular soils, the inclemency of the weather or the anthropogenic processes of adaptation of the spaces, we have little archaeozoological data for these regions, which makes it difficult to evaluate livestock in the initial stages of the Neolithic.

Such is the case of Cabecicos Negros, a site for which we have very little information due to problems of material fragmentation or organic matter preservation. The same goes for Coro Trasito, located in the high mountain areas of the Pyrenees, of which few places of occupation have been excavated and faunal remains present problems of fragmentation preventing in many cases its biometric analysis to determine differences between the domestic and wild form for some species.

In contexts where faunal registration is scarce or affected by taphonomic processes, the biomolecular analysis of lipids in pottery offers a complementary route to archaeobotanical and archaeozoological studies in order to understand the management of resources and contribute new data to the economic exploitation strategies and diet of Neolithic communities in these contexts.

6.1. Livestock management in Cabecicos Negros

Cabecicos Negros represents an example of a site with a faunal record with a low degree of preservation, linked to an area where little is known of the general importance of hunting at the beginning of the Neolithic.

Cabecicos Negros is an open-air Neolithic village located in the southeast of the peninsula, facing the sea and near the mouth of the river Antas. Due to the changes in temperature throughout the seasons along with the taphonomic processes of which the open-air sites tend to be victims, the registration of organic material in the site is scarce. Faunal and botanical remains are under-represented, making it difficult to know about animal management at this site, in addition to the few data available in the Andalusian Neolithic. Under these premises, the analysis of organic residues in the ceramics documented in the site of Cabecicos Negros focuses on contrasting the presence or absence of livestock practices, given the little knowledge about the subsistence strategies available at the moment.

For this purpose, 29 ceramic fragments were sampled and analysed by gas chromatography, mass spectrometry (GC-MS) and mass spectrometry of isotopic relations (GC-IRMS). Appreciable amounts of absorbed lipids were extracted from 18 of the 29 analysed potsherds (62%) by acid extraction (see chapter V). A range of saturated and unsaturated mid-chain length n-alkanoic acids (fatty acids) was detected, particularly dominated by $C_{16:0}$ and $C_{18:0}$, and isotopic values resulted in the predominant presence of suido-fats in 89.4% of the identified fats (Figure 6.1).

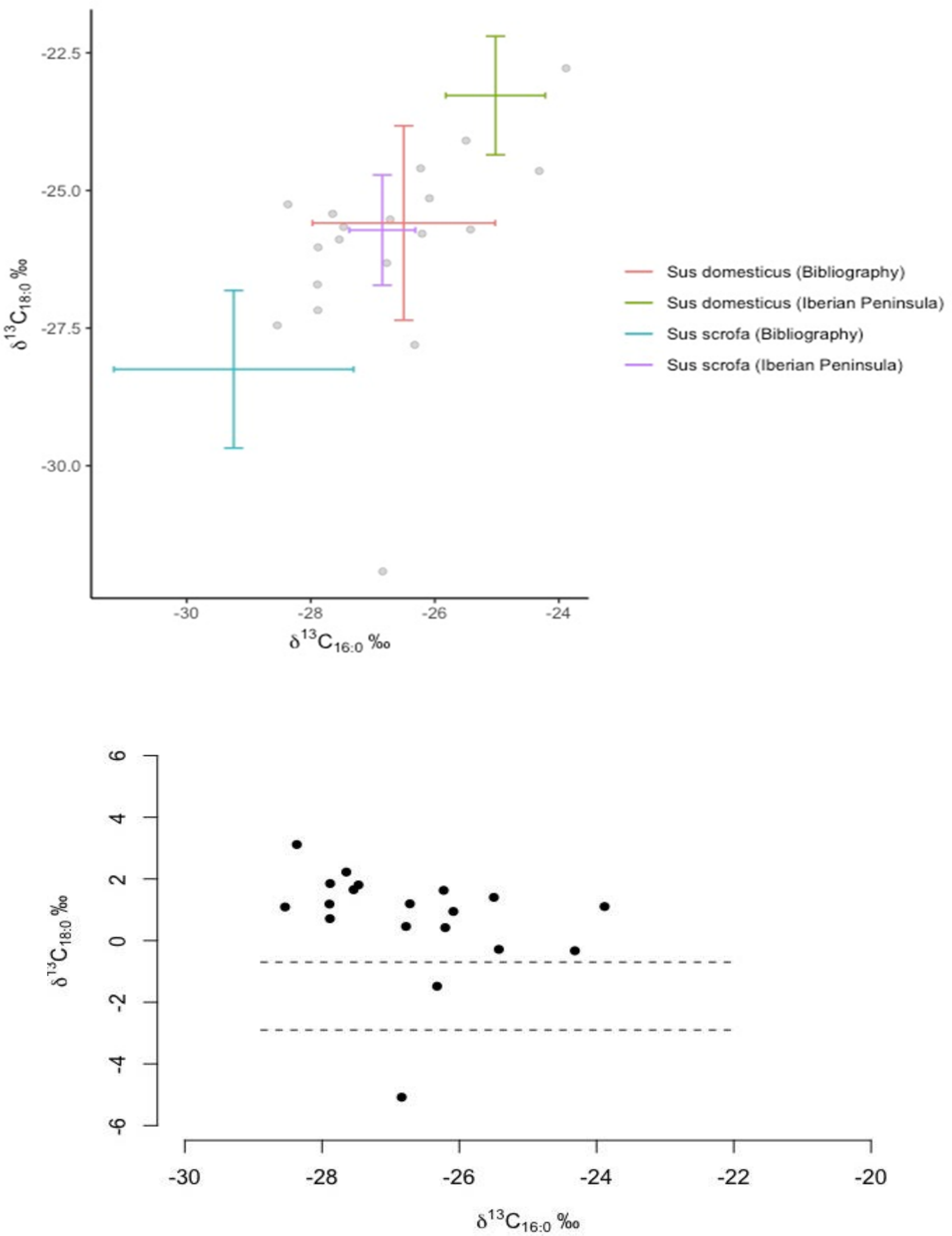


Figure 6.1. Scatter plot showing $\delta^{13}C_{16:0}$ (X) and $\delta^{13}C_{18:0}$ values (Y) (up) and $\delta^{13}C_{18:0}$ (X) and $\Delta^{13}C$ values (Y) (down) of fatty acids extracted from Cabecicos Negros site pottery. The ranges for the modern reference fats are obtained from the literature (Copley et al. 2003; Evershed et al. 1997; Mottram et al. 1999) and the reference model (see chapter V).

In the comparison of the isotopic results obtained from Cabecicos Negros with the published reference values from the UK, we see that these values are consistent with the ellipse of modern pig adipose, although with a wide range of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values. The remaining potsherds have fatty acid $\delta^{13}C$ values that fall within the reference ranges for fat from ruminant adipose (5.3%) and ruminant milk (5.3%).

The few faunal remains studied in the southern peninsular sites highlight the presence of ovicaprines documented in the Ancient Neolithic levels (Saña 2013, Martín-Socas et al. 2004). In the present study, it has been possible to document the processing in ceramic containers of domestic ruminant fats, as well as the dairy exploitation of these, and an important processing of porcine fat.

These results allow us to demonstrate the practice of livestock activities related to the management of domestic ruminants and pigs. This mixed type of livestock shows the exploitation of live animals in the site, from the presence of secondary products.

Also noteworthy is the representation of the processing of porcine fats in the potsherds from Cabecicos Negros. Among domestic animals, pigs (*Sus domesticus*) provided meat during the establishment of farms in the Iberian Peninsula (Saña 1998, 2013). Their presence in the faunal record is estimated to be around 23.9% and varies considerably between sites and throughout the Neolithic periods, suggesting different scales of livestock regimes (Saña 2013, Saña et al. 2015).

Pigs would have been a valuable commodity to prehistoric people; they have a short reproductive cycle and large litter size, thus, in favourable conditions they have an extraordinary capacity to reproduce (Grigson 1982, Saña 2013). Pigs eat virtually anything, thereby converting inedible organic debris to meat and in doing so providing a means of controlling settlement waste (Gregg 1988).

Previous studies of lipid residues extracted from prehistoric ceramics in the Iberian Peninsula have shown that relatively few vessels contain predominantly porcine lipids

(Spiteri et al. 2016), although this may be due to the lack of residue studies in this region. For the time being, we only have one case study in which the processing of pig fat in the south of the Iberian Peninsula was made visible, as is the case of Cueva de El Toro (Tarifa-Mateo et al. 2019). This may be due to alternative methods of pork cooking, such as roasting (Albarella and Serjeantson 2002), or it may be a dietary bias, whereby beef, lamb and dairy products were cooked and consumed in preference to pork (Mukherjee et al. 2015).

In the isotopic results obtained at the Cabecicos Negros site, the wide range of dispersion of $C_{16:0}$ and $C_{18:0}$ values between non-ruminant samples, whose values are dispersed among domestic pigs (*Sus domesticus*) and wild boar (*Sus scrofa*), leads us to look for evidence the presence or not of *Sus* scrofa. A recent study on feeding management strategies for domestic pigs at Neolithic sites in the northeast of the peninsula showed that there are differences in pig management between sites based on the isotopic ratio of carbon ($^{13}C/^{12}C$) and nitrogen ($^{15}N/^{14}N$) (Navarrete et al. 2017). On the other hand, some individuals from Can Sadurní and Reina Amàlia-Caserna de Sant Pau presented values $\delta^{15}N$ higher than the local herbivores by 2.5 and 1.4‰ respectively, and in the case of Can Sadurní similar to carnivore $\delta^{15}N$ values. This led us to think that the diet could reflect this isotopic differentiation in the fats detected in Cabecicos Negros.

Comparison of compound isotopic ($\delta^{13}C$) results of fatty acids extracted from Cabecicos Negros pottery, which are consistent with wild boar and domestic pig fats with a variety of diets, was raised in order to be able to distinguish the origin of the identified fats. At present, the reference collections of modern fats suggest a differentiation between *Sus domesticus* and *Sus scrofa*, although the literature is still very scarce regarding reference fats of wild animals (Craig et al. 2012; Debono Spiteri, 2012; Gregg et al. 2009; Gregg & Slater, 2010b). If we take into account the values obtained with the 26 samples of wild boar adipose fats, and the 26 samples of pig adipose fat fed on acorns, C3 pasture and forbs from organic farms, the archaeological results seem consistent with the presence of two groups of *suidae* with an herbivorous diet and others with a diet enriched with animal protein.

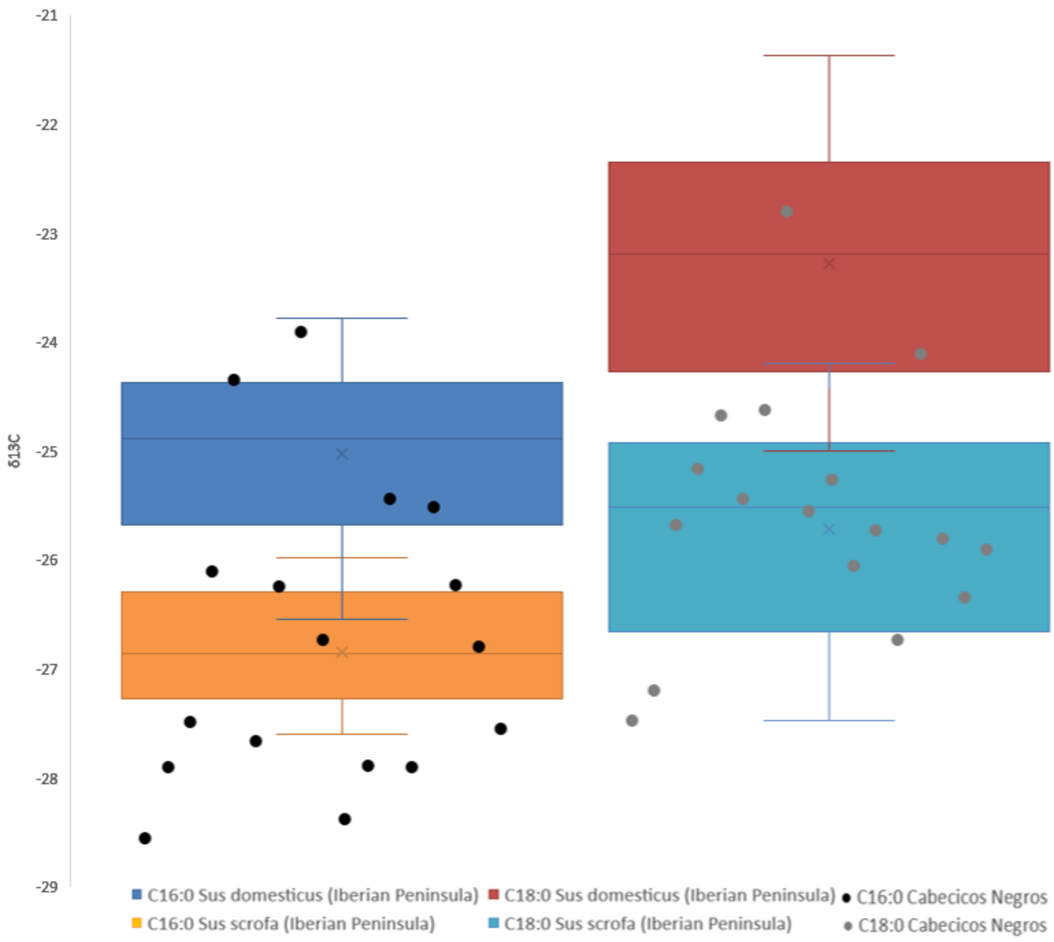


Figure 6.2. Boxplot with the mean values of domestic pig and wild boar fats from the Iberian Peninsula and the isotopic values from Cabecicos Negros fats.

The average of the modern reference values for domestic pig and wild boar fats have a distinctive range for $C_{16:0}$ <-25.5‰ and $C_{18:0}$ <-24.5‰. Following these ranges, samples CNP14, CNP17 and CNP25 correspond to domestic pigs, while the rest of non-ruminant samples ($\Delta^{13}C$<0‰) would be located in the wild boar ranges. This distinction is evidenced by a t-test analysis between samples from Cabecicos Negros: the average values $\delta^{13}C$ of domestic pigs from Cabecicos Negros (-24.56±0.27‰) do not differ significantly from the mean values for modern domestic pig fats (Table 6.1) ($p=0.05$ for $C_{16:0}$ and $p=0.07$ for $C_{18:0}$), and from wild boar ($C_{16:0}$ $p=0.05$, $C_{18:0}$ $p=0.02$).

		δ13C	
		C16:0 ±0,3 (‰)	C18:0 ±0,3 (‰)
Cabecicos Negros	CNP01	-28,54	-27,45
	CNP02	-27,89	-27,18
	CNP06	-27,47	-25,66
	CNP12	-26,84	-31,92
	CNP13	-26,09	-25,14
	CNP14	-24,32	-24,65
	CNP15	-27,65	-25,42
	CNP16	-26,23	-24,60
	CNP17	-23,88	-22,78
	CNP19	-26,72	-25,53
	CNP21	-28,37	-25,25
	CNP22	-27,88	-26,03
	CNP23	-25,42	-25,71
	CNP24	-27,89	-26,71
	CNP25	-25,50	-24,09
	CNP26	-26,21	-25,79
	CNP27	-26,78	-26,32
	CNP28	-27,54	-25,89
Domestic pig (*Sus domesticus*)	***mean***	-25.02±0.8‰	-23.27±1.07‰
Wild boar (*Sus scrofa*)	***mean***	-26.84±0.53‰	-25.71±1.00‰

Table 6.1. Table of results of non-ruminant $\delta^{13}C$ values from Cabecicos Negros and $\delta^{13}C$ average values from domestic pig and wild boar modern fats.

The isotopic differentiation of animal fats can be affected by various factors (see chapter IV). As mentioned above, the isotopic value of animal fats can vary considerably (3%), especially in non-ruminants, since they absorb fatty acids directly from the intestine (Mills et al. 1976).

The wild boar (*Sus scrofa*) presents a mostly herbivorous diet, based on the consumption of acorns, roots of plants and some insects. On the other hand, pigs (*Sus domesticus*), due to their omnivorous character, can feed on vegetable materials (ground cereals and legumes) and human waste, including products of animal origin. This second case would imply a greater degree of human control over pigs. We must bear in mind that isotopic variations between the reference values of domestic pig and ruminant fats do not respond to a differentiation between species but rather a variation in diet. Therefore, a differentiation in the management of *Sus sp.* in Cabecicos Negros should be considered. In the UK, differences have been found between the $\delta^{13}C$ values of Neolithic and Iron Age domestic pig bones (Hamilton et al. 2009). In contrast to the enrichment of the value of carbon in Neolithic pigs, the authors propose that this change may be due to different feeding environments and factors, such as feeding in open environments, feeding linked to human waste, a marine contribution to the diet, or the consumption of mushrooms (Hamilton et al. 2009).

Traditional breeding practices may have involved home-based systems with full or partial housing of herds in the vicinity of settlements, or extensive management of herds in semi-freedom or open-air regimes. Although these management practices are known in modern traditional communities in northern Mediterranean areas (Albarella et al. 2007; Hadjikoumis, 2012), they have also been postulated for prehistoric groups in Europe (Balasse et al. 2016). Oak and riverbank forests, such as those found around Cabecicos Negros, are excellent environments for the development of wild boar or the production of domestic suidae under an extensive management regime (Webbs et al. 2017). A clear example of the difference in the pig diets comes from a recent study in the Northeast of the Iberian Peninsula, where it was found that the pigs of La Draga, Cova del Frare and Serra del Mas Bonet had values consistent with their respective local herbivores and that they could be associated with an open-air husbandry system in forest environments (Navarrete Belda & Saña Seguí, 2017). They also showed a different management regime to the one described above in two specimens of La Draga, which had higher values of 15N whichcould be due to selective feeding practices or to the variability of protein intake (Navarrete et al. 2017).

These results lead us to question the causes of the differentiation in diets within the same site, as seen in Cabecicos Negros:

1. Complementary of livestock and hunting. The comparative study of animal fats identified at Cabecicos Negros could show a complementarity of protein intake from the practice of hunting activities.

2. Diferentiation by sex or age. The changes in the diets of the three pigs in Cabecicos Negros may respond to an intentional practice of greater control over these animals. From the ethnographic data collected, we can see that the enclosure criteria may respond to reproductive reasons (Halstead and Isaakidou 2011). Sows bred could give birth from the first and second year, under optimal feeding conditions. In this way, some herders retain sows always in order to continue breastfeeding and encourage reproduction until 7-8 years of age, while others slaughter sows around 4-5 years, before the meat becomes too hard (Halstead and Isaakidou 2011).

In summary, we see a variability of the values δ13C corresponding to a direct contribution of the differentiated diet. The low levels of δ13C in most non-ruminants of Cabecicos Negros may respond to a diet in forested or riparian environments (De Goene et al. 2018). Plants and trees in dense forests are more depleted in $\delta^{13}C$ than open grasslands, especially plants closer to the ground (Drucker and Bocherens 2009). Thus, the observed low levels of $\delta^{13}C$ in suidae are consistent with a differentiation between extensive diets versus a more omnivorous dietary intake. In this sense, we have discussed the possible causes of pig food differentiation, such as mixed subsistence strategies or a more controlled diet focused on the reproduction of females.

6.2. High mountain environments and exploitation of wild and domestic resources. The cases of Coro Trasito, Cova del Sardo and Juberri sites

In the case of high mountain areas, the lack of archaeological data has generated an underdeveloped image of Neolithic populations that lived there, with an incipient agriculture combined with hunting activities and wild plant gathering (Yañez et al. 2002, Galop et al. 2003, Llovera 1986). In other cases, the persistent idea of an economic model focused on pastoralism and the practice of transhumance is interpreted in some Pyrenean sites to explain the human occupation of high mountain settlements (Lancelotti et al. 2014, Martín et al. 2010, Rojo Guerra et al. 2013, 2014, Tornero et al. 2016). This preconception that links pastoral activities to mountain areas is partly due to our current perception of farming, usually dichotomized into crop husbandry and animal herding, leading some researchers to exclude any possibility of high mountain agriculture and sedentary life. Recent archaeological research in the Pyrenees and the Alps has allowed for the recording of plenty of structures that show the impact that human occupation has had since prehistoric times in these regions (Díaz-Bonilla et al. 2016, Gassiot et al. 2016, Orengo et al. 2014, para el Pirneo, Walsh *et al.* 2005, 2007, Garcia *et al.* 2007, Carrer 2015, para los Alpes). The specific studies of these vestiges allow us to argue that these groups had the same knowledge and technological innovations as the inhabitants of the plains (Gassiot et al. 2014, Hafner and Schwörer, 2017, Antolín et al. 2017).

There are still few faunal sets from sites in a Pyrenean context documented so far and which are representative of animal management in these areas (Antolín et al. 2017, Lancelotti et al. 2014). This is either because of the lack of studies or because of the use of bones as fuel in these contexts, which results in the presence of very fractured sets with a low degree of resolution of the faunal sets that allows inference in the animal management strategies practiced in these contexts. For this purpose, the results obtained from the analysis of organic residues have been taken into account from 66 ceramic containers from the Neolithic site of Coro Trasito (4990-4460 cal BC), the cave of Cova del Sardo (4800-2500 cal BC) and the open-air settlements of Camp del Colomer, Feixa del Moro and Carrer Llinàs 28 (4500-3800 cal BC), located between 1300 and 1800 m a.s.l. in the Eastern Pyrenees.

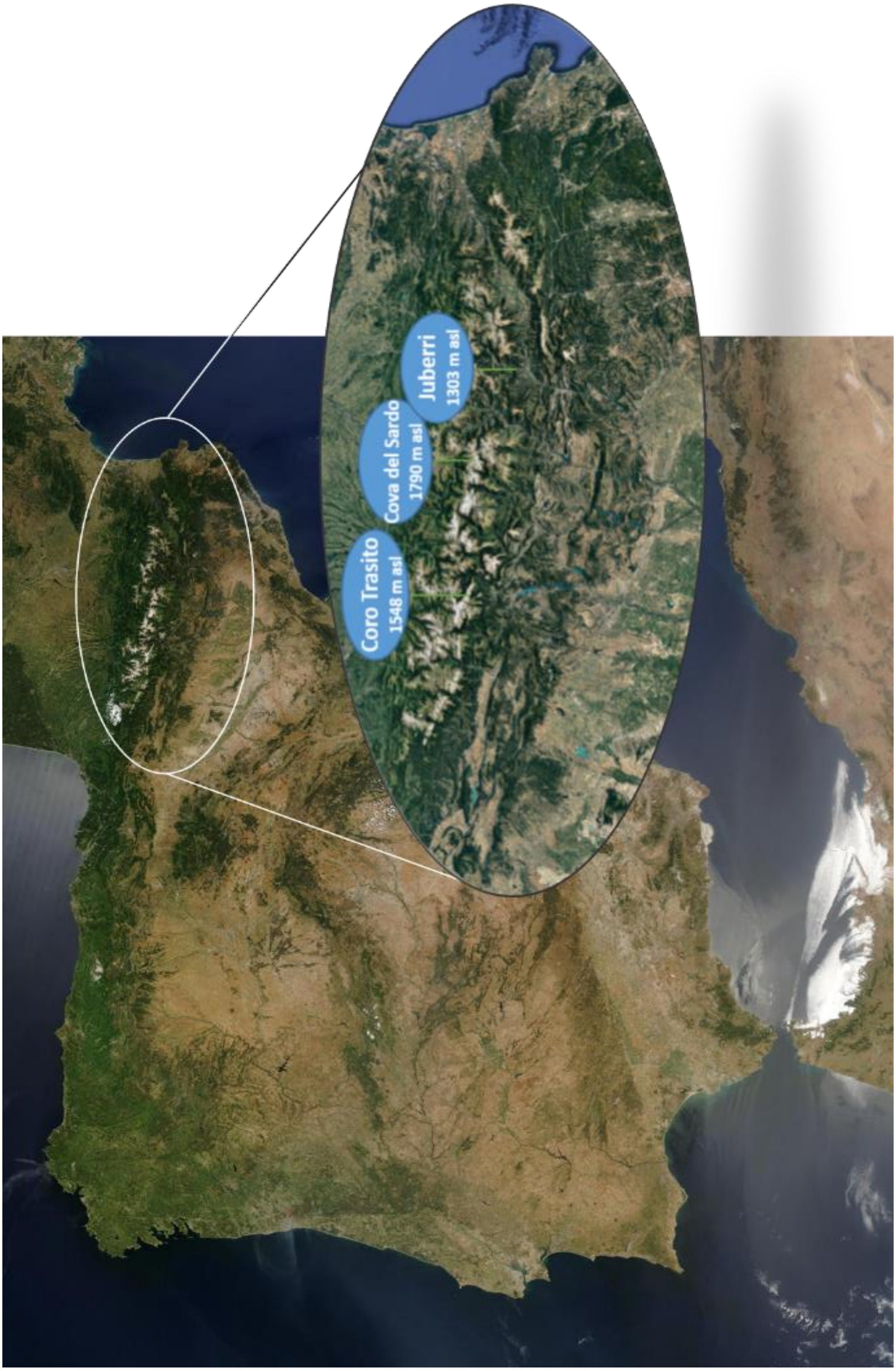


Figure 6.3. Location of the sites studied from high mountain contexts

These settlements have different dynamics that are reflected in the archaeological record, dated between 5300 and 4600 cal BC. Coro Trasito (Tella-Sin, Aragon) is a large cave of more than 300m^2 located at an altitude of 1548 m a.s.l. This settlement is located in the vicinity of an arable area at a lower altitude (1370 m a.s.l.) and a vast pasture area, between 2100 and 2300 m a.s.l. The different archaeological analyses as well as its excavation describe Coro Trasito as a permanent occupation site with a strategy of mixed exploitation focused on domestic resources.

Cova del Sardo, a shelter located at 1790 m a.s.l., presents different phases of prehistoric occupation ranging from 5600 to 2500 cal BC (Gassiot 2010). Throughout the phases, the patterns of use of space change significantly with the presence of homes and evidence of structures, even with periods of non-occupancy. Specific studies in the site showed the cultivation and consumption of barley (Antolín et al. 2017). On the other hand, the high degree of fracturing of the fauna remains and the high frequency of thermoalterations prevented an exhaustive analysis of the hunting and livestock strategies carried out at the site (Navarrete and Saña, 2015). All these characteristics have made it possible to hypothesize that the Cova del Sardo served as a refuge, especially in warm seasons, for the inhabitants of other central settlements in order to exploit the available domestic resources, ovicaprinos and barley (Gassiot, 2010).

The interventions carried out in the municipality of Juberri (Andorra), Camp del Colomer, Feixa del Moro and Carrer Llinàs 28, revealed an open air site dated between 4900 and 3300 BC. No clear evidence has been found to think that the three interventions respond to the same settlement (Fortó and Vidal 2016), as each of the interventions has different dynamics: Carrer Llinàs 28 has levels along with constructive structures, while Camp del Colomer and Feixa del Moro have a large number of silos for the storage of products and evidence of the processing of plant elements (Fortó and Vidal 2018). Archaeobotanical analyses documented the importance of agriculture in the economy of the inhabitants of this site. The poor preservation of the remains of fauna has not allowed us to evaluate the degree of representativeness of the animals nor to influence the livestock practices in Juberri.

The molecular results documented interpretable residues in 41 of the 66 ceramic containers from the Neolithic sites in the Pyrenees studied (62%). Among the results (described in chapter V), animal fats and vegetable residues were identified (Table 6.2).

Site	Sample	$\delta^{13}C$ C16:0 ±0,3 (‰)	$\delta^{13}C$ C18:0 ±0,3 (‰)	Predominant commodity type
Coro Trasito (Tella-Sin, Aragón)	CTT01	-26,34	-30,11	Dairy fat
	CTT03	-27,73	-31,01	Ruminant adipose fat; epicuticular wax
	CTT05	-25,83	-26,86	Ruminant adipose fat
	CTT06	-21,75	-23,11	Ruminant adipose fat; pine resin
	CTT07	-26,53	-25,6	Pig adipose fat
	CTT09	-27,76	-28,91	Ruminant adipose fat
	CTT10	-22,88	-23,95	Ruminant adipose fat; pine resin
	CTT13	-27,01	-28,23	Ruminant adipose fat; pine resin
	CTT15	-28,92	-30,83	Ruminant adipose fat; pine resin
	CTT17	-27,18	-26,98	Wild boar adipose fat
	CTT20	-26,93	-32,29	Dairy fat
	CTT33	-26,03	-25,71	Pig adipose fat
	CTT34	-25,87	-21,46	Pig adipose fat
	CTT35	-26,7	-27,48	Ruminant adipose fat
	CTT36	-24,56	-23,33	Pig adipose fat
	CTT37	-25,81	-28,49	Dairy fat; epicuticular wax
	CTT38	-24,91	-21,07	Pig adipose fat
	CTT39	-28,61	-27,47	Wild boar adipose fat; epicuticular wax
	CTT40	-28,11	-31,74	Dairy fat; epicuticular wax
Cova del Sardo (Boí, Lleida)	SAR01	-24,96	-26,12	Ruminant adipose fat
	SAR02	-24,76	-27,03	Ruminant adipose fat
	SAR03	-25,26	-27,22	Ruminant adipose fat
	SAR04	-26,36	-28,91	Ruminant adipose fat
Camp del Colomer (Juberri, Andorra)	JBA01	-28,28	-29,28	Ruminant adipose fat
	JBA02	-23,96	-22,85	Pig adipose fat
	JBA04	-28,73	-32,62	Dairy fat
	JBA10	-32,34	-35,18	Dairy fat
	JBA11	-29,58	-31,84	Wild ruminant adipose fat
	JBA13	-24,16	-24,07	Pig adipose fat
	JBA14	-21,78	-22,22	Pig adipose fat
	JBA28	-27,17	-25,94	Wild boar adipose fat
Carrer Llinàs 28 (Juberri, Andorra)	JBA12	-30,75	-31,75	Ruminant adipose fat
	JBA19	-25,29	-24,45	Pig adipose fat
	JBA21	-29,31	-29,99	Ruminant adipose fat
	JBA23	-28,5	-30,61	Ruminant adipose fat
	JBA26	-24,08	-26,82	Dairy fat
	JBA27	-23,13	-22,74	Pig adipose fat
	JBA29	-28,46	-30,22	Ruminant adipose fat
Feixa del Moro (Juberri, Andorra)	JBA18	-21,03	-22,83	Ruminant adipose fat
	JBA24	-22,09	-21,76	Pig adipose fat
	JBA25	-26,66	-27,17	Ruminant adipose fat

Table 6.2. Results of δ13C values of sites from high mountain contexts

As can be seen from Figure 4.4, both ruminant and porcine carcass products as well as ruminant dairy products were processed in these mountain sites. Most of these commodities are domestic animals, although the consumption of wild boar is evident in three cases.

About 80% of the fat recovered from the surface of Coro Trasito, Cova del Sardo, Camp del Colomer, Carrer Llinàs 28 and Feixa del Moro sherds is subcutaneous fat from animals. These comprised of 45% ruminant, among which we find wild ruminates fat (3%), and 35% porcine adipose fats, where the wild boar represents 7% (Figure 6.4).

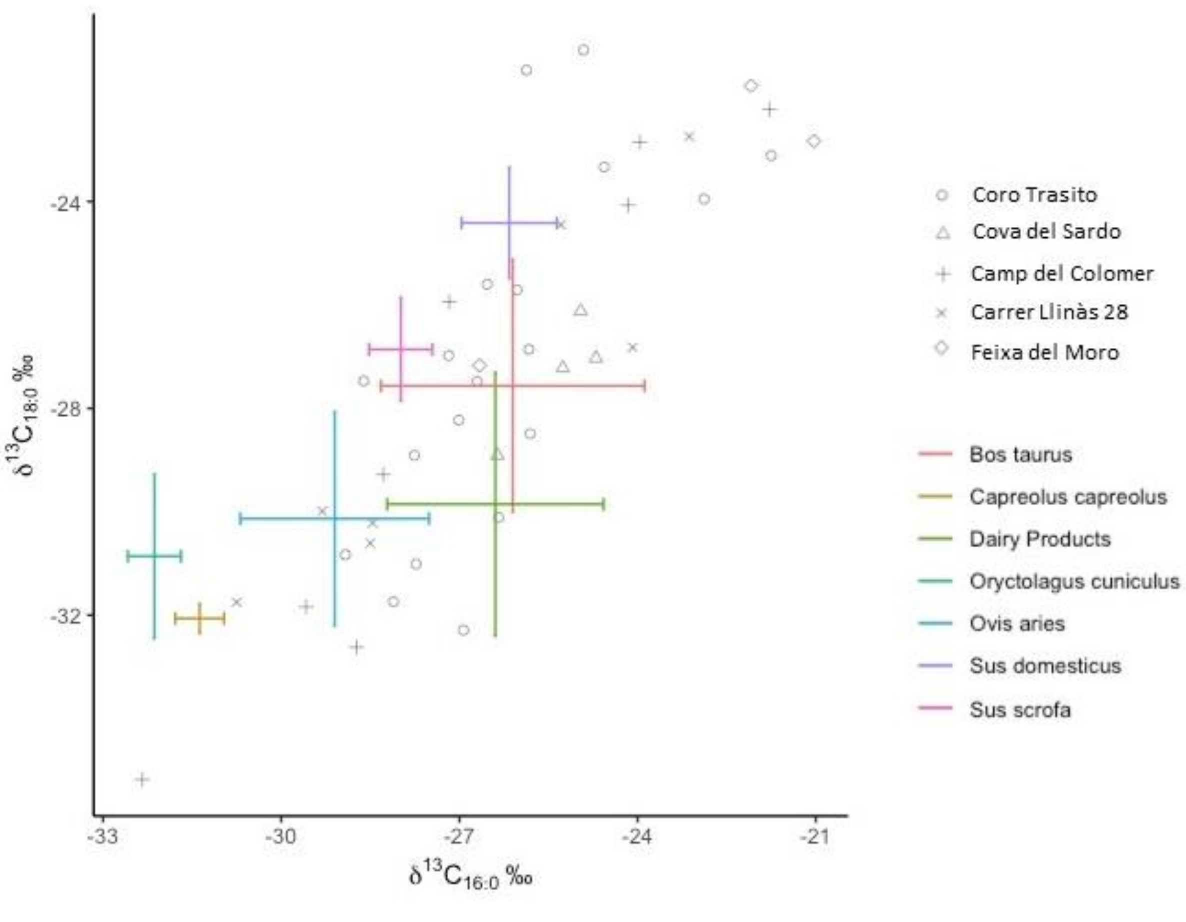


Figure 6.4. Scatter plot showing δ13C values C16:0 (*X*) and C18:0 (*Y*) of fatty acids extracted from Coro Trasito, Cova del Sardo and Juberri sites pottery. The ranges for the modern reference fats obtained in this thesis: violet range indicates domestic pig; red range indicates boar; blue range indicates domestic ruminants; orange range indicates wild ruminants and values less than -3‰ indicates dairy products.

The predominance of the presence of domestic ruminant adipose fats, close to 50% of the identified fats, is reinforced by the remains of fauna studied in these high mountain sites. The results obtained show the important role played by the management of domestic species in these contexts. Despite the different functions played by the sites, the remarkable presence of ovicaprines in high mountain sites, especially sheep, indicates the rapid adoption and incorporation of these species into the economic strategy (Antolín et al. 2017).

Of the 3100 fauna remains recovered at the different levels of Cova del Sardo, only 32 could be classified as ovicaprine due to the high level of fragmentation and oxidation resulting from the taphonomic processes that occurred in the settlement (Antolín et al. 2017, Gassiot et al. 2015). The isotopic results of the animal fats detected in the Cova del Sardo containers confirm the processing of domestic ruminant adipose fats in the three vessels with identifiable lipids, among which we find sheep and goats. The analysis of organic residues from the containers supports the proposed theory of economic practices focused on the exploitation of ovicaprids, although it has not been possible to contribute new data to the exploitation of other resources.

In Camp del Colomer, where the fauna is not representative because of the taphonomic affects that the remains have suffered (Fortó and Vidal 2018), the results of organic waste in the vessels of this settlement provide data on the exploitation of meat resources, which until now has been unknown. As for the biomolecular results, domestic ruminant adipose fats were detected in one of the containers, a second showed wild ruminant fats, another three from domestic pigs and a last one from wild boar fat. Thus, biomolecular analyses have shown that, apart from the farm that was developed in this area and which supports the large number of silos documented, the meat farm is evident and includes different domestic and wild species. A similar pattern occurs in Carrer Llinàs 28 and Feixa del Moro, where one vessel with wild ruminant fat (Carrer Llinàs 28), six vessels with domestic ruminant and three with the presence of domestic pig fat are also documented.

Ceramic organic waste reflects a high percentage of domestic versus wild animal fats, which only represents 10% of the characterized fats. In high mountain contexts it is considered that hunting had a strong presence even in the early stages of the Neolithic. However, in the settlements studied, both the faunal remains (Antolín et al. 2017) and the results of organic residues in sherds show that the consumption of wild species was not as important in the Early Neolithic phases. On the other hand, the evidence of hunting hoofed animal increases in the later Neolithic phases in some sites, such as Coro Trasito (Viñerta 2015). More specifically, the remains of wild fauna documented in the sites of the Pyrenees show the hunting of deer, wild boars, wild goats and rabbits, which represent a maximum percentage around 15.27% (Antolín et al. 2017).

It is difficult to differentiate between domestic and wild taxa, pig (*Sus domesticus*) and wild boar (*Sus scrofa*), and sheep (*Ovis aries*), goat (*Capra hircus*) and wild goat (*Capra pyrenaica*), when the faunal groups present taphonomic problems (fragmentation, oxidation, etc).

In Cova del Sardo it was not possible to make an identification beyond ovicaprinos. From the lithic industry of the studied site, abrasions and microfractures are observed on the edges used that suggest its use as a blade to cut soft animal substances, probably in relation to the slaughtering activities (Mazzucco 2014: 275), while hunting practices in the site are evidenced by the presence of projectiles possibly related to the consumption of chamois (*Rupicapra rupicapra*) (Mazzucco 2014: 295).

On the contrary, the vessels analysed in Cova del Sardo do not account for the processing of meat from wild animals in the containers, on the other hand, it is coherent with the ovicaprine remains studied, so we can conclude that they would be domestic sheep and goats.

The fauna recovered at the Coro Trasito site consists mainly of ovicaprins (sheep and goat), bovids (cattle) and domestic suids (pig), and more residual wild species such as deer, roe deer, rabbit, wild boar and wild goat (Viñerta, 2015). However, some of the faunal remains corresponding to the category of suidae could not be classified as domestic or wild, so they were grouped in the category *Sus sp* (Viñerta 2015). The present study reflects this predominance of processing of domestic species over wild species, related to hunting activities. Nonetheless, the fats of wild animals processed inside the ceramic containers represent 18% of the total fat identified in Coro Trasito. This shows that the protein contribution from hunting would be evident in a site with highly developed subsistence practices focused primarily on the management of domestic resources.

Finally, in Camp del Colomer, Feixa del Moro and Carrer Llinàs 28, where we had no evidence of meat exploitation, the analysis of organic residues in ceramics show that 5% of the animal fats identified come from animals resulting from hunting activities, such as deer in Carrer Llinàs 28 and wild boar in Camp del Colomer.

The presence of rabbit, the target of the hunting practice in Coro Trasito (Viñerta 2015), is absent in the results of the analysis of organic residues in ceramics. This may be due to the fact that these products would be processed outside the scope of ceramics, such as roasted directly over a fire, or rabbit fats present such a weak signal that they would go unnoticed when mixed with fats from other animals.

There are few works focused on the specific use of ceramics in high mountain settlements. Recent biomolecular and isotopic studies on pottery from high mountain contexts in the Swiss Alps region (Carrer et al. 2016b) and the French Eastern Pyrenees (Spiteri et al. 2016) have made it possible to identify the processing of domestic resources inside the containers.

6.2.1. Dairy product exploitation in high mountain context

Archaeological evidence of cattle ranching is represented by the faunal remains (species, ages, skeletal variability, diet, etc.). In addition, the use of animal products (meat, skins, extraction of bone marrow, obtaining fats, etc.) can be directly inferred from butchery marks and bone fragmentation (Driesch and Boessneck 1975, Binford 1978). However, the identification of secondary products has been based on archaeological tradition from less direct approaches, such as bone pathologies related to stress on limb joints (Baker 1984), nutritional stress (Mulville 1993) or animal mortality patterns (Payne 1973). However, until the detection of dairy products from biomolecular and isotopic analysis of organic residues preserved in the clay matrix of ceramic vessels from the 7th millennium cal BC in the Mediterranean (Copley et al. 2005; Craig et al. 2011; O. E. Craig et al. 2005; Richard P. Evershed et al. 2008b; Salque et al. 2012b; Šoberl et al. 2008; Spangenberg et al. 2006), the transformation of ruminant milk, possibly in the form of dairy products to be stored, by neolithic communities had not been directly observed.

From biomolecular and stable isotope analyses it has been possible to identify 7 extracts with milk products (20%) inside the vessels from Coro Trasito, Camp del Colomer and Carrer Llinàs 29. Given the scarcity of archaeofaunistic data available for these sites, due to the affectation of the remains by anthropogenic activities, such as the combustion of bones, or because of the taphonomic processes, we did not have any data to relate dairy exploitation with the Pyrenean sites. The only exception, which presents a good conservation of organic matter, is Coro Trasito. The fauna studied in Coro Trasito presents remains of fetal or neonate ovicaprines, especially in the oldest Neolithic phase, being absent in later chronologies (Antolín et al. 2017). This evidence relates to strategies to maximize resources in specific environments (Llorente et al. 2014) or for milk production (Helmer, 1992).

The organic residue analysis in high mountain contexts is scarce, that is why we find a significant absence of parallels in which the consumption of these products is evidenced. Recent work has made it possible to begin to observe the consumption of dairy products also in mountain contexts, such as in Font-Juvénal (France) (Spiteri et al. 2016). It should also be noted that in the Pyrenean site of Segudet (Andorra), located at 1324 m a.s.l. and with a chronology between 5450 and 4900 cal BC (Yañez et al. 2002), it has been proposed that some ceramic vessel would have contained dairy products, a result that should be taken with some caution since its detection was based on the distribution of fatty acids detected by gas chromatography and not on their isotopic value.

The direct evidence of dairy farming from the early Neolithic stages in the Pyrenees high mountain areas raises a number of questions about the role of dairy farming in the management of herds and the consumption of these products. Dairy products could have played a crucial role in the survival of first farming communities throughout the western Mediterranean, especially in regions where agriculture is not intensively practised, with livestock complementing production (Legge 1981, Rowley-Conwy 2011).

Faunistic studies are increasingly appearing that show mortality profiles with a high relative percentage of sheep, goats and young oxen documented in high altitude sites, such as Coro Trasito (Antolín et al. 2017) and Els Trocs cave (Lancelotti et al. 2014), as well as in sheep and cattle in alpine areas (Legge 1981, Becker 1981) from the 5th millennium BC. However, this is not an exceptional survival practice, but represents the mixed or diversified exploitation of the herd in an area where cereal production is difficult (Oliver E. Craig et al. 2005) (McGovern 1992, Halstead 1998, Mainland and Halstead 2005). In addition, the high nutritional value of ruminant milk could have served as a food supplement during low-productivity seasons, such as winter (Degen 2007).

Some ethnographic studies allow us to learn about herd management practices in mountain areas in order to better understand the dynamics of the subsistence strategies that would be developed in the sites studied. In the villages in the plain, livestock management is reduced to the domestic level with a scarce number of animals, whereas in the areas where pastoralism practices are developed, livestock is more numerous (Hasltead 1998). Some researchers have shown an interest in dairy maximization strategies. In the mountainous areas of northwestern Greece (~1200m a.s.l.) sheep are artificially fed and housed in stone constructions or caves during the winter months, when pastures are covered with snow and temperatures are extreme, in order to be able to exploit the milk of females who have calved between spring and summer (Halstead 1998). With regard to slaughter guidelines, herders allow a period of lactation of the brood necessary to establish lactation safely before slaughtering the brood for meat (Halstead et al 2011). This practice of using breeding to facilitate milk let-down, also documented in artistic representations of the 3rd millennium BC in Mesopotamia (Sherratt 1981), is evidenced by the high number of lactating or post-lactating individuals at archaeological sites. Neither the composition by species nor the proportion of ceramics used to heat milk is a reliable index of the intensity of milk production, but the integration of bio-molecular and faunal data allows us to better understand certain aspects in order to know this production better as a whole.

Another question raised by the identification of the milk farm in the Pyrenees is how this milk would be consumed in the populations of the highlands. Human digestion of milk requires the hydrolysis of its main sugar, lactose, by means of lactase, a specific enzyme produced at birth, but which disappears in most adults (Gerbault et al. 2013), although some populations developed a persistence of this enzyme (Sherratt, 1983). The absence of this adaptation in the early Neolithic communities does not seem to affect the possible consumption of this product, probably because lactose is partly degraded by bacterial activity during fermentation to produce products such as butter or cheese (Pollard and Heron, 2008), which would facilitate its tolerance in humans. Milk processing, given the confirmation of lactose intolerance by DNA in Neolithic Europe (Burger et al. 2007), will improve digestion as well as the shelf life of dairy products. The available evidence indicates that, as in the case of ploughing, processing of dairy products may have been irregular, both regionally (R. P. Evershed et al. 2008) and locally (Urem-Kotsou and Kotsakis 2007). The advantage of applying organic residue analysis has provided unequivocal and direct evidence for the use of dairy products and, therefore, pastoral activities. In addition, the ability to process milk offers the added advantage of storing surplus dairy products, such as cheese, yoghurt or butter, making them available throughout the year (R. P. Evershed et al. 2008).

Of storable dairy products, written sources and genetic studies of bacterial activity suggest that yoghurt is a recent phenomenon in Europe (Kekhagias 2008; van de Guchte et al. 2006), but cheese and butter were known in ancient times (Halstead et al. 2011). The development of the bacterial activity that turns fresh milk into storable cheese may have been discovered by rennet stored in the skin or stomach tissue of a lamb, kid or calf as a container (Halstead et al. 2011). Butter, on the other hand, is produced by letting the milk rest and removing the superficial layer that is created from the fermentation of fats.

In this case, we ask ourselves what role ceramic containers played in the transformation of milk.

Based on the identification of dairy products in the ceramic containers of other prehistoric sites in Europe, some researchers propose a classification of three types of vessels related to the manufacture and/or preservation of dairy products: perforated containers (or "cheese makers"), cooking containers and small containers (Drieu, 2017).

Although it is not possible to return the complete cups in all cases, the JBA10 cup is consistent with the typology of small containers for short-term storage, consumption or manufacture of dairy products such as those documented at various Neolithic sites in Great Britain (O. E. Craig et al. 2005) or at the French Clairvaux XIV site (Mirabaud and Regert, 2016). On the contrary, the vessels corresponding to samples JBA04 and JBA26 could be related to the small containers mentioned above or to cooking containers for manufacturing or food preparation, such as those documented in Neolithic sites in Scotland or the Alps (Carrer et al. 2016b; Copley et al. 2005).

6.2.2. Consumption of vegetal sources

Five containers from the Neolithic mountain site of Coro Trasito (CTT03, CTT35, CTT37, CTT39 and CTT40) present biomarkers of epicuticular waxes (see chapter V). Similar profiles were identified in the Neolithic sites of Mala Triglavca (Slovenia) (Soberl et al. 2014), Gribaia (France) (Drieu, 2017) and Fondo Azzolini (Italy) (Debono Spiteri, 2012). A container from Camp del Colomer presents biomarkers of animal substances together with plants, identified by the presence of oleic acid ($C_{18:1}$) and ß-sitosterol. The distribution of phytosterols, such as ß-sitosterol, together with stigmasterol and campesterol, has been linked to the presence of vegetable oil (Cramp 2008, deMan 1999). Similar biomarkers have recently been detected in containers at Soldartorpet (Sweden) and Syltholm (Denmark) (Papakosta et al. 2019).

It is still difficult to deepen the interpretations related to plant products, since these distributions of fatty acids, sterols, alkanes and linear alcohols are ubiquitous in the plant kingdom. Epicuticular waxes are found in the leaves of plants. The symmetrical distribution of alcohols suggests the leaves of plants of the *Brassicaceae* family, including grasses. Experimental studies relate the presence of epicuticular waxes with the boiling of vegetable leaves (Charters et al. 1997), which identified together with fats of animal origin, can respond to specific culinary patterns, such as stews. Carpological data attest to the exploitation of different types of vegetables in Coro Trasito: cereals, legumes and fruits, but none of these raw materials can be specifically recognised by chemical biomarkers. Even so, both settlements with evidence of vegetable products, Coro Trasito and Camp del Colomer, present naked barley which could have represented an important crop in the Pyrenees since the second half of the sixth millennium BC (Antolín 2016, Antolín et al. 2015).

The cultivation of domestic plants was documented in the high mountain sites (higher than 1200 m a.s.l.) of Coro Trasito, Cova del Sardo and Camp del Colomer (Antolín et al. 2017) with a percentage of 68.5%. Some of these sites document a predominance of clean plants lacking any chaff remains which indicates annual cultivation involving a permanent occupation in the sites of Camp del Colomer and Coro Trasito. This crop would be focused on naked barley, in the cases of Coro Trasito and Camp del Colomer, and on wheat, in Cova del Sardo. The important number of fruits and seeds of wild species documented in the sites of high mountains (ca. 70%), emphasizes the capacity of exploitation of the resources of the environment. Consumption evidence of hazelnut (*Corylus avellana*), acorn (*Quercus sp.*), wild strawberry (*Fragaria sp.*), wild pear (*Pyrus malus*) and blackberry (*Rubus sp*.) are present in Coro Trasito and Camp del Colomer.

In this aspect, the analysis of organic residues in ceramics has been able to show the processing of vegetable resources in the vessels of both sites, although it has not been possible to specify the strategies of subsistence related to the consumption of vegetable and agricultural resources.

In summary, biomolecular analyses of organic waste eventually preserved in ceramic containers are a viable resource for inferring resource acquisition strategies. Firstly, we understand that not all food resources would be be processed in ceramic containers, since tools made of other materials such as wood or basketry could be used, or the food simply does not need containers for processing. Secondly, the physical and economic limitations do not allow for the study of all the ceramic containers found in the site, so we must be aware that we only identify a part of the products. However, the selection criteria were established to minimize the effect of this factor on the interpretation of the results. Finally, the substances identified with the means at our disposal today allow us to provide more data on strategies for acquiring animal resources than plants, which are very difficult to identify.

Based on the results discussed in this chapter on animal management, the analysis of organic residues in ceramics has contributed knowledge on the strategy of pig management in the Cabecicos Negros site in the south of the Iberian Peninsula. In this case, it is hypothesized that there is a variability in the feeding strategies of the first domestic pigs at a local level, reflecting both free and semi-free livestock systems, as well as more specialized feeding practices.

The documentation of different feeding strategies among domestic herds at the regional and local level points towards the existence of an important control over this aspect of management. These strategies allowed, on the one hand to implement dynamics of feeding the herds at the margin of the natural availability of the vegetal alimentary resources in the environment. It develops artificial feeding modes, with the contribution of vegetal resources during the seasonal cycle of independent form to its natural availability. This possibility of modifying the characteristics of animal feeding shows the magnitude and intensity of the changes introduced.

Regarding the Pyrenees, it is common to find publications in which human presence in ranges of medium and high mountains is related to hunting, the collection of wild plants or some kind of incipient agriculture in non-permanent plots related to slash-and-burn practices (Galop et al. 2003, Llovera 1986, Orengo et al. 2014, Yanez 2005, Yañez et al. 2002). The combination of this work together with previous studies that face the same problem from a different materiality emphasizes that there are many complex models of mixed agriculture in the mountain regions where the grazing of animals interact (Ebersbach 2010, Vincze 1980).

The results point to the processing of domestic animals, products derived from them, such as dairy products, and plant resources in a variety of locally produced ceramic containers. These elements reinforce the proposed interpretation of most mountainous sites as more or less permanent mixed farming communities (Navarrete et al. 2017).

The importance of the role of hunting activities in mountain sites is weakened by our results of container analyses in the three sites analysed in this chapter, as has also been indicated from archaeozological data (Saña, 1998). All this leads us to propose that in the contexts of high mountains and abrupt orography, subsistence activities based on a productive economy, such as agriculture and livestock, were developed at the same time as in the plain and with a level of complexity comparable to that found in coastal sites (Navarrete et al. 2017).

In conclusion, we can stress that the analysis of organic waste in ceramics not only provides data on the culinary patterns of the communities of the past, but also allow us to identify new evidence for practices not previously assessed, such as the processing of milk, and influence the strategies of acquisition of food products. It has also allowed us to investigate the consumption of fats from domestic animals in archaeological sites where it has not been possible to determine the degree of livestock management from the remains of fauna.

Chapter VII. Production strategies at the beginning of the Neolithic period and their representation in ceramic wastes

The domestication of plants and animals offers a more direct control of the resources, focused on their maintenance and reproduction, while obtaining the resources of the living animal. When the animal is a product destined for consumption, management requires its death; on the other hand, when the animal is inserted into the community as a means of production, management is materialized in the maintenance and reproduction of live animals (Saña 1997). Live animals are a new form of meat storage, while they can be exploited for milk, as labour force in transport and agricultural practices, and in the production of faeces for the marinating of crops.

From the archaeozoological record point of view, these actions can be inferred from the demographic patterns of the animals, which allow us to know if animals were slaughtered before reaching their optimum meat, so we interpret a farm focused on dairy resources (Vigne and Helmer 2007), or through anthropic fracturing and thermoalterations of faunal remains.

All of this does not represent direct archaeological evidence, as the patterns can be identified but not contrasted from the faunal remains themselves. The analysis of ceramic residues, on the other hand, opens a new way to knowledge of consumption patterns since it is direct evidence of the processing of these resources with a focus on consumption (Vigne and Helmer 2007). In fact, the identification of dairy residues has been a revulsive for the explanations on the Neolithic and raises the need to estimate the importance of milk versus meat resources in Neolithic societies, as well as to know which dairy products and how they were consumed. In spite of this, some previous considerations must be taken into account, as the answer to these questions can be biased by the way in which these new products are obtained and processed.

In this chapter, we evaluate results on the production of food resources obtained by analysing the contents of ceramic containers. The available archaeological data of the studied sites are complemented in order to extract the maximum information in relation to the production strategies practiced by Neolithic communities.

7.1. Production of dairy products in the Neolithic

It is now more than thirty years since Andrew Sherratt (1981) argued that, several millennia after the beginning of the development of animal husbandry, another innovation in animal exploitation occurred, involving the intensive use of secondary products such as milk, blood, wool and traction, which can be repeatedly extracted from an animal throughout its lifetime. Since then there has been much debate about the Sherratt's model (Chapman 1982, Greenfield 2010), especially from the identification of cattle mortality profiles from Brzesc Kujawski's LBK site, as well as the presence of cheesemakers in central Europe in chronologies between the 6th and 5th millennium BC. (Bogucki 1984). Later on, and taking these arguments into account, Sherratt accepted that "milk-drinking, while secondary, may have emerged during the first spread of farming" (Sherratt 1997). Nonetheless, Greenfield argues that Sherratt was always concerned with how and when the scale of exploitation changes and how this ultimately affected human society. He suggested that a distinction should be made between the first origins of dairying practices and the timing of its later intensification (Greenfield 2010).

Today, we are well aware of the importance that the consumption of dairy products may have had for Neolithic populations. Milk is often considered a nutritional advantage because of its high content on fats, proteins, carbohydrates, vitamins and calcium (Wooding 2007). In contrast, about 65% of the world's population cannot digest milk beyond the age of eight, as digestion requires the body to break down the disaccharide of lactose, the sugar in milk. Most babies naturally produce the enzyme lactase-phlorizin-hydrolase (or LPH) so that they can take advantage of nutrients in breast milk, but for most of the world's population, lactase production is interrupted in the post-weaning period. Drinking milk after that leads to a battery of symptoms, including diarrhea, cramps, gas, nausea and vomiting.

Several lines of research show that Neolithic dairy farming created selection pressures that favored the development of the lactose allele in herders (Simoons 1970, Durham 1991, Holden and Mace 1997, Myles et al. 2005, Burger et al. 2007, Leonardi et al. 2012). The majority of Neolithic populations showing LPH are located in northern Europe (Ingram et al. 2009, Itan et al. 2010). That advantage would have occurred in situations where milk was, or could be, an important part of the diet, in which the group was in situations of dietary stress (Gerbault et al. 2011, Bocquet-Appel 2011).

Archaeologically, the consumption of dairy products in northern Europe is observed since the beginning of the Neolithic (Salque et al. 2012, Craig et al 2011) and as these products are incorporated into the diet of populations.

Nevertheless, where community members have not developed this allele or in most communities in southern Europe, they would process the milk to make low lactose products such as cheese, yoghurt or butter. Milk is host to a variety of microorganisms (streptococcus, lactobacillus, bacillus, yeasts and molds) and each plays a role in converting milk into milk products by breaking down lactose into lactic acid, which sours the milk and coagulates the milk protein, allowing yeast and mold to proliferate and reduce the acid.

7.2. Evidence of dairy products in the Iberian Peninsula

On the basis of archaeozoological studies and mortality patterns studied in Neolithic sites in the Mediterranean basin, milk exploitation is observed in 80% of the sites studied (Antolín et al. 2017, Gillis 2012, Gillis et al. 2014, Helmer et al. 2007, Spiteri-Debono et al. 2016, Vigne 2011). In two cases, this hypothesis could be argued with the identification of milk product fats from ceramic organic residues, such as in the Colle Santo Stefano (Italy) and Font-Júvenal (France) sites (Spiteri-Debono et al. 2016).

In the Iberian Peninsula, archaeozoological data show mortality profiles in sub-adult cattle at the El Trocs cave (Rojo-Guerra et al. 2013), Coro Trasito (Viñerta 2015, Antolín et al. 2017), La Draga (Saña 2011, Gillis et al. 2014), Caserna de Sant Pau (Saña and Navarrete 2016), Reina Amàlia (Saña and Navarrete 2016), Can Sadurní (Saña et al. 2015), Cova del Vidre (Saña et al. in press) and Cueva de El Toro (Martín-Socas et al. 2004).

Mortality patterns of sheep and goat remains in the El Trocs cave indicate an age of slaughter below two years of age, i.e. before achieving the optimum meat, around 86% of all the ovicaprine remains studied, which points to the likely existence of a dairy farm (Rojo-Guerra et al. 2013). The presence of animal remains of sheep and goats in perinatal stages in the early stages of development of the Coro Trasito and Els Trocs settlements is interpreted as a practice of risk minimization strategies during lambing periods (Antolín et al. 2017), where pregnant females are separated from the flock (Martín et al. 2016), although the dairy exploitation of ovicaprines is not ruled out (Antolín et al. 2017). In La Draga, age histograms point to the exploitation of a significant number of calves and goats to obtain milk (Saña 2011, Gillis et al. 2014). In the cases of Reina Amàlia and Caserna de Sant Pau, the variable distribution in slaughter age is interpreted as the practice of a multipurpose livestock strategy, combining milk production, the exploitation of meat and the use of animals as a means of work (Saña and Navarrete 2016). For the Can Sadurní site, histograms show high mortality rates before the first year of life for sheep and goat species during the Ancient Neolithic, which are related to dairy farming. These patterns are not clearly reflected for *Bos taurus*, conditioned by the low number of recovered remains (Saña et al. 2015). In the Cova del Vidre, the study of kill-off patterns through age estimation indicates that an elevated number of animals were sacrificed before 6 months and between six and twelve months age. Such early sacrifice ages could be coherent with the slaughter of animals before weaning, so the milk produced by the mother can be kept for human consumption (Saña et al. in press). Finally, in El Toro Cave, the pattern of ovicaprine mortality in the oldest Neolithic phase focuses on clear meat exploitation, whereas in the next phase of occupation the mortality of these species at an early age induces to think about a change in the economic system focusing on milk production (Martín-Socas et al. 2004). In summary, we can appreciate that there is a trend towards a milk-producing species, such as sheep and goats, as opposed to cattle. With the exception of the La Draga site, where there is no evidence of milk exploitation of the sheep but there is evidence of milk exploitation of the cow, although this information could be biased by the absence of studies of specific slaughter patterns in other sites.

From the first analyses of residues in prehistoric sites that confirmed the milk exploitation and its processing in ceramic containers (Copley et al. 2003, 2005a, b, Craig et al. 2003, 2005, Regert et al. 1998, 1999), the evidences of fats of milk origin have been increasing and confirming a generalized exploitation in all Europe of these products, which was already pointed out from the archaeozoology. There is ample evidence of dairy products in Neolithic sites of Southern Europe from the 6th millennium cal BC (Evershed et al. 2008, Perić et al. 2013, Ethier et al. 2017, Soberl et al. 2008, 2014, Debono 2012, McClure et al. 2018, Salque et al. 2012, Martí et al. 2009, Whelton et al. 2018, Ogrinc et al. 2012). Next, we evaluate the exploitation of dairy products in the furthest west of the Mediterranean basin.

In particular for the Iberian Peninsula we have little representation, due to the lack of specific studies. For the Ancient Neolithic, milk processing has been observed in Can Sardurní containers (Spiteri-Debono et al. 2016) in around 15% of vessels with interpretable concentrations of lipids, and in El Cavet, les Guixeres de Vilobí, Cova del Vidre and Reina Amàlia (Breu 2019). The results of five vesseles from the Cova de l'Or site that have been interpreted as goat's milk have also been published from a comparison of the isotopic analysis of the fats identified in the ceramics with current goat's milk (Martí et al. 2009). However, in the review of isotopic values of $\delta^{13}C$ we see that they do not correspond to the fats of milk origin of established standards, so they are questionable data. The same is observed in the Segudet and Cova d'en Pardo sites, where biomarkers of milk presence were identified from the distribution of fatty acids (Yañez et al. 2002) or by the presence of a plant biomarker potentially consumed by milk-producing animals, such as beta-cedrene (Soler and Togores, 2008). For all these reasons, currently, and pending the publication of another doctoral thesis in progress on Neolithic ceramic residues in the northeast of the peninsula, the only reliable evidence of milk processing in ceramic containers is that of Can Sadurní.

In this study, evidence has been obtained of the processing of this type of production in the ceramic vessels of the Neolithic sites studied.

	Total number of vessels analysed	Total number of vessels containing >5 µg g-1 lipid	No. of vessels with animal fats	No. of vessels with dairy products
Coro Trasito	26	19	19	4
Cova del Sardo	10	4	4	0
Camp del Colomer	14	8	8	2
Carrer Llinàs 28	11	7	7	1
Feixa del Moro	4	3	3	0
Mines de Gavà	28	15	14	0
Cabecicos Negros	29	18	18	1
Cueva de El Toro	50	24	24	2
La Draga	28	27	6	4
TOTAL	200	125	103	14

Table 7.1. Results of organic residues preserved in pottery from Iberian Peninsula Neolithic sites.

Of the 200 potsherds analysed, 125 (62.5%) yielded identifiable biomarkers. Dietary sources of these lipids are distinguished by characteristic $\delta^{13}C$ value quadrangles defined by $\delta^{13}C_{18:0}$ versus $\delta^{13}C_{16:0}$ values, as well as the isotopic difference between the two, [$\Delta^{13}C_{18:0}\pm_{16:0}$]. Of these, the $\delta^{13}C$ values show that 11 potsherds can clearly be attributed to a ruminant dairy product origin (10% of the total animal fats identified), plotting within the known range of ruminant dairy products determined by analysis of modern reference dairy fats from cattle and ewes raised on a wholly C3 diet in Iberian Peninsula. These data confirm the exploitation of domesticated animals and their secondary products was taking place at these Iberian Neolithic sites in the sixth and fifth millennium BC.

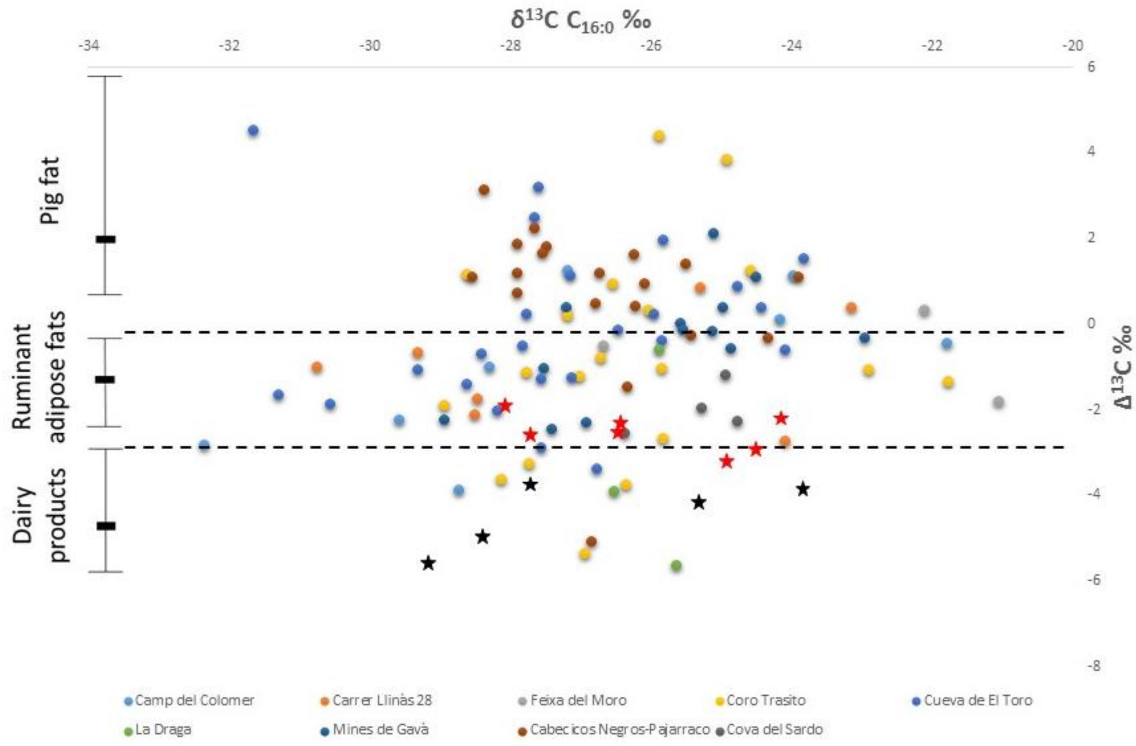


Figure 7.1. $\delta^{13}C_{16:0}$ (X) and $\Delta^{13}C$ values (Y) of fatty acids extracted from Peninsula Iberian Neolithic sites. The ranges for the modern reference fats obtained from own modern reference values (see chapter IV). Black stars represent modern milk and red stars modern fermented dairy products.

Ruminant dairy products are differentiated from ruminant adipose products when they display $\delta^{13}C$ values of less than -3.1‰ (Dunne et al. 2012, Salque 2012). However, some overlapping is observed between the observed $\delta^{13}C$ values of ruminant carcass and dairy fats, hence archaeological fats which plot between -2.4‰ and -3.1‰ boundary should not be firmly interpreted as ruminant dairy or adipose products, rather it is likely that some mixing of these fats occurred in the vessels.

Crossplots of $\delta^{13}C_{16:0}$ versus $\Delta^{13}C_{18:0}\pm_{16:0}$ further show differences among lipid residues from secondary dairy products. Based on previous modern reference studies (Spangenberg et al. 2008, Regert 2011), modern samples from unfermented ruminant dairy fats y fermented ruminant dairy fats (yogurt, cheese, etc.) that we have analysed (Chapter IV), show differences in the δ13C values: milk, $\Delta^{13}C_{18:0}\pm_{16:0}$ < -3.3½; cheese, 0½ > $\Delta^{13}C_{18:0}\pm_{16:0}$ > -3.3½ (Figure 6.1).

7.2.1. Coro Trasito

As mentioned in Chapter V, at the Coro Trasito Pyrenean site, 4 samples with isotopic values were identified that clearly correspond to dairy products (Figure 6.1), dated between 4992 - 4786 2σ cal BC (CTM01 and CTM20) and between 4605 - 4460 cal BC (CTM37 and CTM40). However, one of the samples identified as animal fats is located in the band between milk products and ruminants ($\delta^{13}C$ $C_{16:0}$ = -25.81 ‰, $\delta^{13}C$ $C_{18:0}$ = -28.49 ‰). This could respond to a mixture or use at a different time of these two products inside the container, or the fermentation of milk which, as we have discussed above (Figure 7.1), have values of $\Delta^{13}C$ >-3.1 ‰.

In Coro Trasito there is evidence of economic practices focused on domestic animal husbandry from the first phases of occupation (5300 - 5100 cal BC). Livestock farming focuses on the exploitation of sheep and goats (around 80% of the documented fauna), although we also find the presence of pigs and cattle. Mortality patterns reflect a slaughter of the cattle in adulthood, when they would have obtained their optimum meat. On the other hand, the presence of sheep and goat remains in perinatal stages has been interpreted as a practice of risk minimization strategies during calving periods (Antolín et al. 2017), where pregnant females are separated from the flock (Martín et al. 2016), although the dairy exploitation of ovicaprinos is not ruled out (Antolín et al. 2017).

7.2.2. Camp del Colomer y Carrer Llinàs 28 (Juberri)

Among the samples identified as dairy products, from the isotopic analysis of palmitic and stearic acids, we found a sample from Camp del Colomer with values $\Delta^{13}C$ <-3.1 ‰, whereas another sample from Camp del Colomer and a container from Carrer Llinàs 28 present values very close to the range stipulated for dairy products, although the values $\Delta^{13}C$ are higher than -3.1 ‰ (Figure 7.1), although we cannot affirm with certainty the characterisation of the contents of the containers as dairy products, the reference values analysed in chapter IV make us think that the samples would correspond to fermented dairy products, which have more enriched values than fresh milk around a value of $\Delta^{13}C$ of -3 ‰.

The fauna remains from Camp del Colomer and Carrer Llinàs 28 have been affected by the taphonomic processes, a fact that has not allowed the archaeozoological study of the remains (Fortó and Vidal 2018).

7.2.3. Cabecicos Negros

Of the 29 containers analysed from the Andalusian site of Cabecicos Negros, one of the samples showing sufficient quantities of lipids where animal fats were detected was identified as a dairy product from the isotopic analysis of the values $\delta^{13}C$ of palmitic (-26.84 ‰) and stearic (-31.92 ‰) fatty acids (Figure 7.1). The study of the remains of fauna found at the site, very affected by taphonomy, is in progress. Therefore, it has not been possible to contrast the results with archaeozoological data.

7.2.4. Cueva de El Toro

In the Cave of El Toro, animal fats were identified with an isotopic value $\delta^{13}C$ ($\delta^{13}C$ $C_{16:0}$ = -25.81 ‰, $\delta^{13}C$ $C_{18:0}$ = -28.49 ‰) corresponding to an origin of dairy products in one of the analysed containers. On the other hand, another container had values δ13C for palmitic (-27.55 ‰) and stearic (-30.44 ‰) fatty acids ($\Delta^{13}C$ >-3.1 ‰) which are located in between the ranges for dairy products and adipose fats of ruminant origin (Figure 7.1). Both vessels come from phase IV of occupation of the Cueva de El Toro (5280 - 4780 2σ cal BC), the oldest phase of Neolithic occupation at the site.

If we compare these results with the remains of fauna documented on the site, we see a livestock focused on the management of ovicaprinos, which could not make a differentiation between sheep and goat, which represent 78% of the fauna studied from phase IV of occupation, and 86% in the later phase. Bovids represent around 3% of the domestic fauna represented in the site for both phases (Martín-Socas et al. 2004). Mortality patterns identified from the age of fusion of bones in ovicaprines, from the oldest phase of occupation in the Neolithic period, are identified with an age of slaughter around 3 years of age. This practice indicates a clear meat exploitation focus. On the other hand, in the next phase of occupation, the mortality of these species at an early age induces to think about a change in the economic system focused on milk production (Martín-Socas et al. 2004).

7.2.5. La Draga

In La Draga the processing of dairy products in 4 cup containers (D2, D3, D13 and 15) was identified from the distribution of triacylglycerides (Chapter V). From the identification of the isotopic compound of the palmitic and stearic acids of two containers, the presence of dairy products was observed in samples D15 and D2.

These findings are consistent with the work carried out by archaeozoological analysis pointing at evidence for dairying practices based upon slaughtering profiles (Gillis et al. 2014, Antolín et al. 2014).

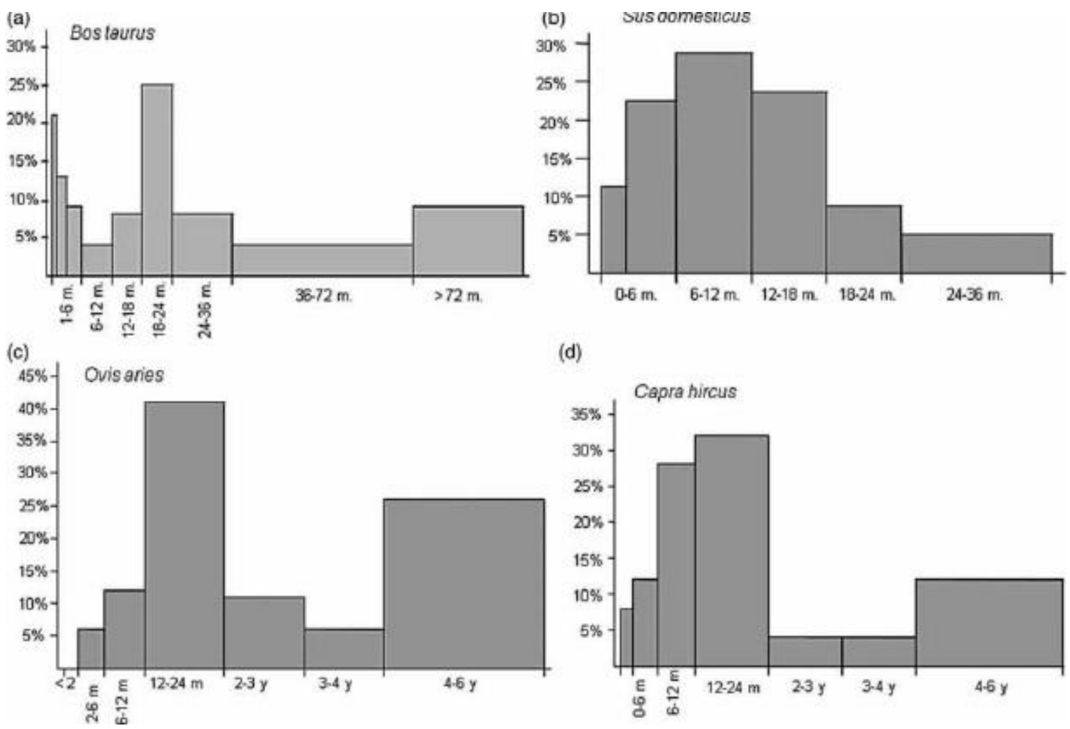


Figure 7.2. Slaughter patterns documented for *Bos taurus* (A), *Sus domesticus* (B), *Ovis aries* (C) and *Capra hircus* (D) in La Draga (Taken from Antolín et al. 2014).

The age profiles of the sheep (*Ovis aries*) of La Draga show a mortality of 40% between the first and second year of age, which corresponds to the optimal meat of the animal, while another large group is slaughtered between 4 and 6 years of age (Figure 7.2, Antolín et al. 2014). This last group can respond to a breeding strategy for the maintenance of the herd.

The histograms for goats (*Capra hircus*) identified in La Draga show mortality profiles similar to those of sheep, except for the peak age between 6 and 12 months of life, prior to the development of the optimum meat (Figure 7.2, Antolín et al. 2014). This is interpreted as a practice for dairy farming.

The mortality patterns of calves slaughtered between 6 and 24 months of age during the period they are being weaned from their mothers are indicative of the management of dairy products (Figure 7.2). In addition, a group of retired lactating females with a peak age greater than 4 years was also identified. On the contrary, a sample of bovine slaughter between 2 and 4 years of age, which would be related to the meat farm, is also observed (Gillis et al. 2014).

In summary, ovicaprids were used for a mixed milk and meat production in La Draga site already during the Early Neolithic. As we have seen, the dairy exploitation of goats occurs in other sites, such as Cueva de El Toro (Martín-Socas et al. 2004). Although the absence of dairy exploitation for sheep is remarkable, except for the recent data obtained for Cueva de Chaves (Sierra et al. 2019). This pattern is also documented in Bercy (France), where it was also concluded that the time of slaughter coincided with the beginning of winter (Balasse and Tresset 2002), a difficult period for the acquisition of fodder and feeding of livestock. Therefore, the elimination of unwanted animals, especially males, would have taken place before this seasonal change, for the maintenance of the herd (Gillis et al. 2014).

Archaeozoological studies are indeed a powerful means of unravelling herding practices and milk exploitation at archaeological sites. They allow the contextualization of the foodstuffs present as lipid residues in sherds and, as lipid residue analyses are not species-specific, a tentative identification of animal species represented in the residues.

7.3. Processing and preservation of dairy products

Starting from this premise, we turn to pottery that preserve the profile with evidence of dairy products. In this case, the two pots from Camp del Colomer, a pot from La Draga and two vessels from Cueva de El Toro (Figure 7.2). In the case of Camp del Colomer, the pots present open and shallow shapes that induce one to think of easy access to the content and that it could be in a semi-liquid state, that is to say, that they could have participated in the production process of fermented dairy products. In addition, the isotopic values $\delta^{13}C$ of Camp del Colomer, not less than -3.8‰, could reinforce the hypothesis of the fermentation of dairy products in the potsherds of Camp del Colomer. The pot coming from the open-air site of La Draga, presents a more closed and deep form, although without great volumetric capacity, given the impoverished value of $\delta^{13}C$ that the fats of milk origin present in this vessel, we can propose that it could contain milk, either for collection, storage or transport. However, two samples were taken from this container: the sample taken from the internal base of the beaker presents isotopic values corresponding to milk, while in the sample taken from the upper part of the beaker, they characterized fats from domestic ruminant animals. Thus, we see how the vessel could be reused to contain two different products, which have left different traces.

Finally, the two pots with evidence of milk products from Cueva de El Toro have different shapes to the ones mentioned above: on the one hand, the milk products appear in a closed container, deep and with a medium volumetric capacity, which induces us to think about the storage and/or the first stages of fermentation of the milk in liquid state. On the other hand, fats of milk origin were identified in a spout without restitution of the rest of the container, although the spout becomes a fairly defining element of the transport or processing of milk.

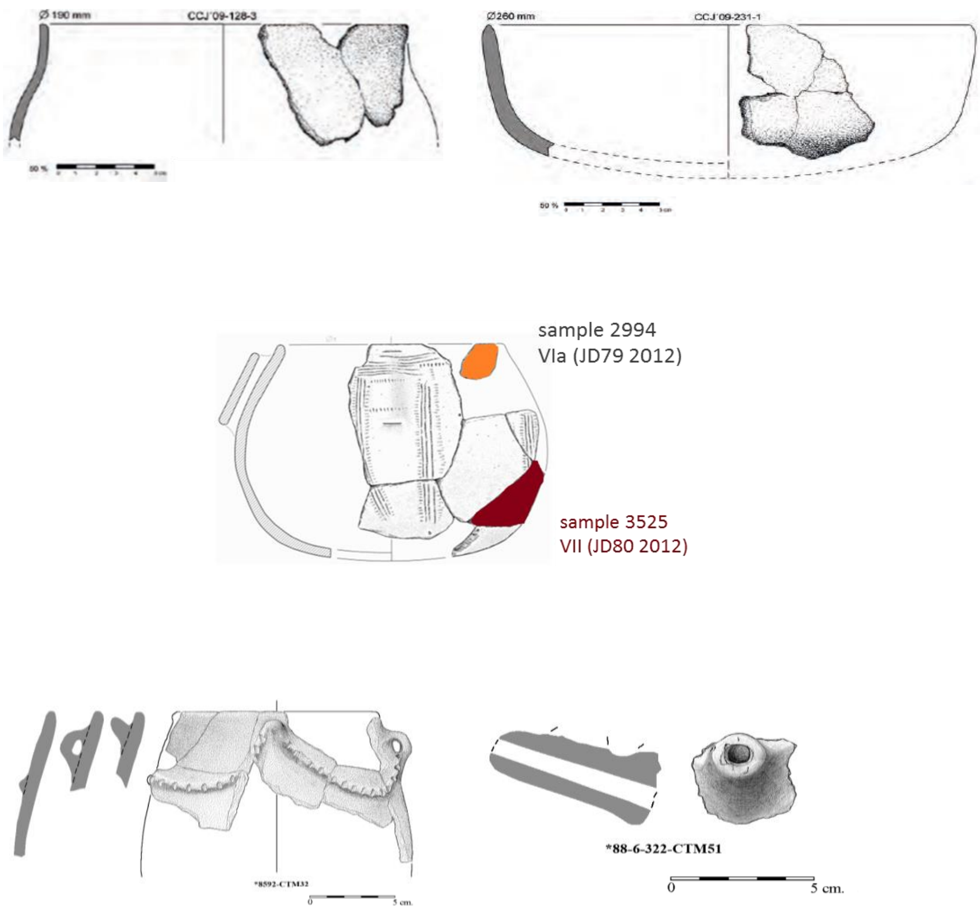


Figure 7.3. Pots containing dairy products from: 1) Camp del Colomer; 2) La Draga; 3) Cueva de El Toro

Milk yields four to five times the amount of energy and protein compared with meat (Salque 2010). However, it becomes complicated when we attempt to evaluate the precise role of milk production in a subsistence economy (Halstead 1996; Balasse and Tresset 2002; Craig 2011) because it requires estimating the amount of milk available for human consumption. This in turn requires knowing both the milk yield of primitive cattle, which itself is difficult to estimate (Gregg 1988; Peske 1994; Tresset 1996), and the amount of milk actually devoted to human as opposed to calf consumption.

Regardless, it now seems clear, from both organic residue analyses of absorbed residues from archaeological pottery and archaeozoological techniques allowing the reconstruction of animal management practices, that the exploitation of secondary products begins with the first Neolithic farmers, supporting claims that milk was one of the main attractions leading to domestication (e.g. Vigne and Helmer, 2007; Evershed et al. 2008;

Conolly et al. 2011). In this line of study, new techniques are being developed to identify direct milk consumption from ß-lactoglobulin protein (BLG), identifiable from the analysis of dental calculi (Charlton et al. 2019, Warinner et al. 2014), as well as the discrimination of the original spice of the dairy residue from the analysis of amino acids, such as that performed in Çatalhöyük (Hendy et al. 2018).

Archaeozoological analyses suggest that both caprines and cattle were managed for milk, with specialized intensive husbandries for the former. Ages at death for caprines from La Draga site group around the postlactation, prime meat, and adult classes, suggesting mixed husbandries, possibly including milk production. Cattle were intensively slaughtered during infancy and postlactation, probably associated with dairying. The integrated study of the analysis of organic residues in ceramics together with the archaeozoological analysis of the fauna remains identified in the site has allowed to verify the milk exploitation in six of the Neolithic sites studied. Mortality patterns suggest that sheep and goats were the predominant producing species, with specialized intensive husbandries. On the other hand, the dredge presents an exception, given that sheep do not present clear evidence of intensive milk exploitation, and goats and cows were in charge of dairy production.

The preference for sheep's milk over goat's or cow's milk is explained by the fact that it does not taste as strong as goat's milk and is more acceptable to the human digestive system compared to cow's milk. This argument could explain the predominance of sheep in Neolithic sites, such as the Cueva de Chaves (Sierra et al. 2019), although it may respond to a particular economic strategy. On the other hand, in the case of La Draga it is contradicted, given that sheep's milk would be the least exploited.

Finally, we have been able to approach the production of dairy products, showing an isotopic differentiation between fresh milk and fermented dairy products, which brings us closer to know the production and consumption strategies in the Neolithic peninsular.

Chapter VIII.1. Pottery technology and natural substances

The analysis of organic residues from pottery has been highly effective in gaining insights into a range of different aspects relating to ceramics, including production, use, repair and technological change and specialisation. Technologies involved in the production of ceramic vessels can be identified using organic residue analysis. The detection of certain non-food substances, such as waxes or resins, has various properties related to the waterproofing and sealing. They could be used as adhesives for the repair of containers, flammable agents for the lighting of spaces, disinfectants and antibacterials, etc. (Binder et al. 1990, Regert et al. 1998, Aveling and Heron 1998, Urem-Kotsou et al. 2002, Mitkidou et al. 2008, Mirabaud et al. 2015, Regert and Mirabaud 2014). For example, unglazed fabrics offer the highest potential for the retention and survival of absorbed residues, yet their surfaces need to be sealed to decrease the permeability of the fabric and make them effective containers for liquids. To date, a range of sealants such as waxes, resins and bituminous materials have been identified on archaeological ceramics, dating back to the Neolithic (Salque et al. 2017).

In the following chapter, the uses of non-food substances detected in potsherds from Neolithic sites in the Iberian Peninsula will be identified and explained. It is also intended to evaluate the properties that these products contribute to pottery technology, which are often identified together with food products.

Beeswax and honey are produced naturally by bees from the nectars of flowers. Although the most obvious reason for exploiting the honeybee would be for honey, a rare source of sweetener for prehistoric people, beeswax would likely have been an equally important material. Beeswax is one of the organic components and the earliest waxy material commonly exploited in the past for different purposes. These include technological purposes, such as a fuel for illumination or a waterproofing agent (Regert et al. 2001, Baeten et al. 2010). These uses however, can be attributed to the fact that beeswax burns very slowly and is characterised by its hydrophobicity, plasticity and therapeutic properties.

In terms of the chemical analysis of organic residues preserved in archaeological sites, biomarkers of honey have rarely been reported in archaeological contexts and this can be attributed to their high susceptibility to degradation under most environmental conditions (Regert et al. 2003). Contrarily, biomarkers of beeswax are more stable and resistant to degradative processes; therefore, they have been detected in many archaeological contexts (Heron et al. 1994, Regert 2004, Regert et al. 2001, 2003, 2005, Bonaduce and Colombini 2004, Garnier et al. 2002 Copley et al. 2005, Evershed et al. 1997, Baeten et al. 2010, Namdar et al. 2009)

Organic residues, such as beeswax, are found amorphous, invisible or absorbed in archaeological ceramic vessels and usually present in low concentration levels; therefore, analytical organic chemical techniques, such as gas chromatography – mass spectrometry (GC-MS), are the most efficient methods for analysing organic residues.

In recent years, the application of these analytical techniques has been favoured for the detection of the use and consumption of beeswax since the beginning of the Neolithic period (Salque et al. 2016). Unfortunately, this product has not been documented in the Iberian Peninsula until the III millennium cal BC. In this context, thirty sherds from the archaeological site of Mines de Gavà were analysed to produce new information on the use and consumption of different products, particularly wax products, inside the sherds during the Neolithic.

8.1. La identificación de cera de abeja en Mines de Gavà

Of the twenty-eight vessels of the Mines of Gavà analysed, biomarkers of beeswax were identified in the MIG18 and MIG25 samples from the presence of even-numbered long-chain monoesters (W40-W52) derived from palmitic fatty acid (C16:0), long-chain n-alkanes with odd-numbered carbon atoms ranging from C21 to C33 and long-chain alcohols (from AL24 to AL34) (Garnier et al. 2002, Regert et al. 2001) (Figure 8.1).

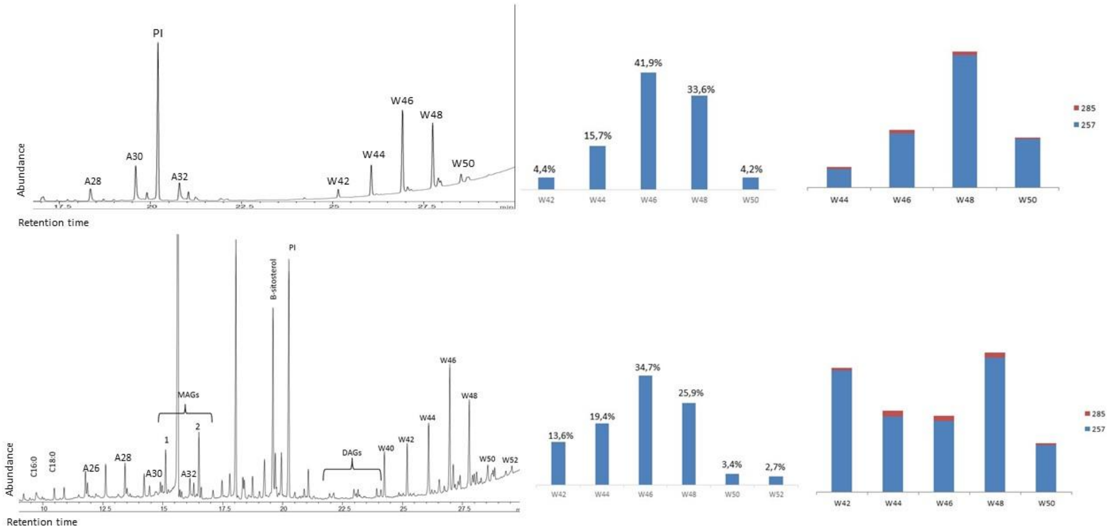


Figure 8.1. Chromatograms with the percentage of wax esters identified and the proportion of the original acid (m/z 285 from C18:0 and m/z 257 from C16:0)

N-alcohols are present in modern beeswax at low levels (parts per million) but become major components in archaeological beeswax exposed to hydrolytic degradations (Garnier et al. 2002). Hydrolysis of wax esters to even-numbered long-chain alcohols, such as AL24-AL34, under burial conditions possibly occurs during diagenetic degradation of waxes (Evershed et al. 1997, Namdar et al. 2009). The degree of hydrolysis of wax esters increases with heating but aging or heating processes could also reversibly affect the amount of n-alcohols produced by hydrolysis (Namdar et al. 2009).

Beeswax has been identified in combination with animal or vegetable fats in other archaeological sites (Heron et al. 1994, Charters et al. 1995, Evershed et al. 1997, Garnier et al. 2002, Regert et al. 2003, Copley et al. 2005, Namdar et al. 2009). The occurrence of beeswax, either alone or mixed with other materials, in archaeological sherds is attributed to the fact that vessels were exploited in the past for different uses, such as cooking, preparing, processing, storage, transport and consumption of natural organic materials.

The preservation of beeswax has been reported in ceramics from different chronological periods in archaeological sites located in different regions in Europe, the Near East and the Far East. The oldest evidence of beeswax in pottery dates back to the 7th millennium BC in sherds from Neolithic sites in Anatolia, such as Çayönu Tepesi and Çatalhöyük (Roffet-Salque et al. 2015). From the 6th millennium BC, beeswax is still foundin Anatolian pottery (Aşaği Pinar and Toptepe), but also in the Balkans, such as Liménaria, Dikili Tash, Paliambela (Greece), Măgura (Romania) and Drenovac (Serbia) (Roffet-Salque et al. 2015). It has also been identified in Central Europe, in Brunn am Gebirge and Niederhummel (Germany) (Salque et al. 2012, Salque et al. 2013, Salque et al. 2015). In southern and western Europe, the first beeswax certificates date back to the second half of the 5th millennium, at the Font-Juvénal, Chassey-le Camp and Bercy (France) and at the Ajdovska Jama and Moverna Vas (Slovenia) (Regert et al. 2001, Salque et al. 2015, Šoberl et al. 2014). Over the following millennia, they were found on sites of Clairvaux-les-Lacs, Chalain 3 (France) (Mirabaud 2007, Mirabaud and Regert 2016, Regert et al. 1999, 2001), Ergolding Fischergasse (Germany) (Heron et al. 2015), Eton rowing lake, Runnymede (Great Britain) (Copley et al. 2005), and Ale and Bjornsholm (Denmark) (Heron et al. 2007).

The presence of organic residues of beeswax in various types of archaeological vessels indicates the diversity of the uses of beeswax. These include as a sealant for vessels or adhesives (Rageot et al. 2015), a fuel for illumination (Decavallas 2011, Evershed et al. 1997, Namdar et al. 2009, Roffet-Salque et al. 2015), an ingredient in the production of medicinal ointments (Regert et al. 2001, Baeten et al. 2010, Crane 2011, Regert et al. 2001, Ribechini et al. 2011), as a preservative due to its antibacterial properties (Crane 2011) and as a foodstuff, either from honey as a sweetening product (Crane 2011, McGovern et al. 2004, Mirabaud 2007, Regert 2007) or for the production of alcoholic beverages (Crane 2011, Heron et al. 1994, McGovern et al. 2004, 2013).

Its hydrophobic properties have made it suitable for use as a material to waterproof pottery (Charters et al. 1995, Heron et al. 1994, 2015, Regert et al. 2001, Roumpou et al. 2003, Salque et al. 2013). The hypothesis of using beeswax as a waterproofing agent for pottery surfaces is more likely when there is no heating trace on the sherds and the beeswax signal is slightly degraded (Mirabaud and Regert 2016). Once sealed with this product, the vessels were probably not used as cooking vessels because the melting point of the wax is quite low at 60ºC (Regert et al. 2001, Drieu 2017) and because wax is soluble, in fact, in the liquid state (Charters et al. 1995, Evershed et al. 1997). However, moderate wax heating can soften it to facilitate its application to surfaces (Heron et al. 1994).

Beeswax has been found in Mines de Gavà either alone and mixed with animal fats. These may be two separate uses of the potsherds or traces of a mixture of honey with other foods (Mirabaud and Regert 2016, Roffet-Salque et al. 2015).

Vessels in which beeswax has been identified are open-shaped, shallow containers (Figure 7.3). In particular, one of them has a unique shape. This type of vase appears for the first time, according to the data currently available, in the context of the so-called "Cultura di vasi a bocca quadrata" (VBQ) that developed in northern Italy during the 5th millennium cal BC (Bagolini and Pedrotti 1998, Bazzanella 1999). Some petrographic analyses of these vessels have determined that the vessels outside the Italic Peninsula, as in the cases of the Iberian Peninsula, are local productions, which would imply that the concept would circulate and not the vessels (Clop 2009).

Figure 8.2. Photographs of beeswax vases from the Mines de Gavà site. Square mouth vase (right).

8.2. Discussing the production and use of resinous materials in ceramic vessels

The study of plant sub-products such as resins and tars can also contribute to a better understanding of plant exploitation. Resins were extremely desirable commodities in the past —and indeed remain so— for their various aromatic, adhesive, antibacterial and aesthetic properties (Salque et al. 2017). However, the study of such remains is less common than that of other botanical evidence because of their low degree of preservation and the need of chemical analyses for their identification.

Conifer resin has been detected in ceramic recipients from the Neolithic sites of Paliambela (Mitkidou et al. 2008), Makriyalos (Urem-kotsou et al. 2002), Dikili Tash (Garnier and Valamoti 2016) (Greece), Drakaina Cave (Croatia) (Debono 2012), Giribaldi (France) (Rageot 2015), Šventoj (Lithuania) (Heron et al. 2015), Könnu, Kääpa, Akali, Riigiküla, Vihasoo, Narva, Kalmaküla (Estonia) (Oras et al. 2017) and in United Kingdom (Mukherjee et al. 2008). In all of them, the presence of resins mixed with animal fats is 87.3% and it is interpreted as a waterproofing product of the container internal walls.

The presence of vegetable resins has been detected in two sherds from Cueva de El Toro and four sherds from Coro Trasito, along with animal fatty acids (Figure 7.4). The characterization of the abietic acid, dehydroabietic acid and 7-oxo-dehydroabietic acid in the potsherds analysed indicate that the dehydrogenation of the compounds is evidence for an application of more than 110ºC to melt the resin to be able to apply it on the surface as a post-cooking treatment in order to waterproof the walls of the sherds (Charters et al. 1995, Pietrzak, 2012), probably related to the storage or processing of animal fats in liquid or semiliquid state (Correa-Ascencio et al. 2014).

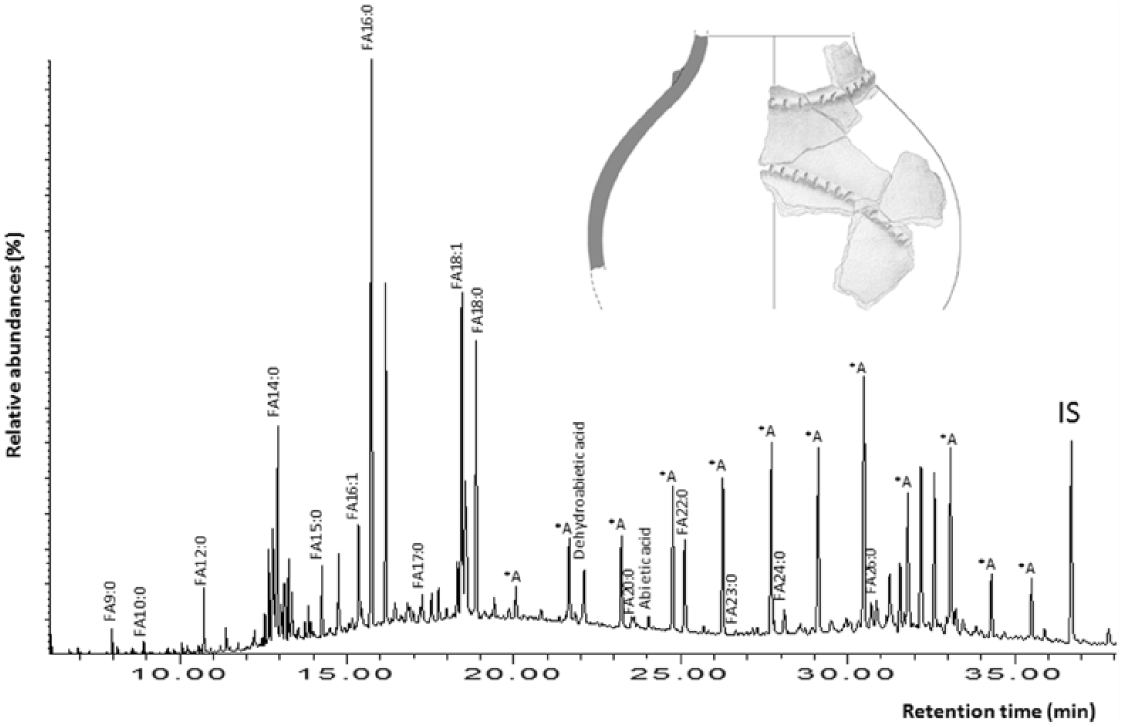


Figure 8.3. Partial gas chromatogram of the trimethylsilylated TLEs from CTM44 pottery sample showing the various biomarkers detected: animal fats (FA*x:y* are fatty acids where *x* is the carbon chain length and *y* is the degree of unsaturation), pine resin (dehydroabietic and abietic acid) and contamination from plants and handling (unsaturated fatty acids and **A* are a long chain of alkanes, with odd-numbered carbon dominance).

However, the presence of methylated dehydroabietic acid and methylated abietate in some Coro Trasito potsherds could show that resinous pine wood was distilled at high temperatures to extract tar, and not by natural pyrolysis of the resin (Hjulström et al. 2006; Izzo et al. 2013). Nevertheless, since the extraction method used methyls organic compounds (Corre-Ascencio et al. 2014), the transformation of abiotic and dehydroabietic acid to methylated compounds could have occurred during extraction in the laboratory. In addition, the absence of polyaromatics, such as retene or phenanthene, which are volatile compounds produced during wood combustion at more than 350ºC, would reinforce the hypothesis that methylated dehydroabietic acid came from laboratory extraction (Bailey et al. 2016).

8.2.1. Resin production

Very little is known about their systems of production, and key questions regarding the choice and harvesting of the raw materials, the manufacturing processes, the organisation of the production and the level of craftsmanship, especially for prehistoric times, still remain unanswered (Rageot et al. 2018).

The anthracological study carried out at the Coro Trasito site highlights the oak (*Quercus sp.*) as the dominant species for fuel, combined with the Scots pine (*Pinus sylvestris*) which, although to a lesser extent, would have functioned as a firebrand to start the fire or increase its luminosity (Obea et al. pers. com.). On the other hand, at the Cueva de El Toro the wood of holm oak (*Quercus ilex*), gall oak (*Quercus faginea*) and olive tree (*Olea europaea*) predominates, with the residual or absent use of pine wood to obtain energy (Martín-Socas et al. 2004). This would show that the exploitation of coniferous resins would imply different wood harvesting activities than for wood to generate heat and light energy in fireplaces.

According to experimental and ethnographic data, ceramic vessels can also be used to manufacture adhesives (Rageot 2015, Mirabaud et al. 2016, Rageot et al. 2016, Regert and Rolando 2002). Ceramics may have been used to heat resins to facilitate their mixing with other products, such as beeswax or animal fats (Rageot 2015, Regert et al. 2003b), or for their application to repair or waterproof containers, or simply to store resins.

The process of obtaining pine resin involves two very different processes: in the first, the bark is removed from the pine and the resin that oozes is collected over the next 2-3 days and poured into a container. For the second process, the distillation of the wood is a practice that implies a greater investment of work and infrastructure. This autothermal system is based on the direct transfer of heat to the wood, which is distilled by exposing it to fire (between 220 and 280ºC) inside a potsherd (Rageot et al. 2018). Finally, the deposited pitch is collected on the bottom of the recipient (Figure 7.5). In both cases, this collection of pine resins would take place during the warm months of the year (Pietrzak 2012).

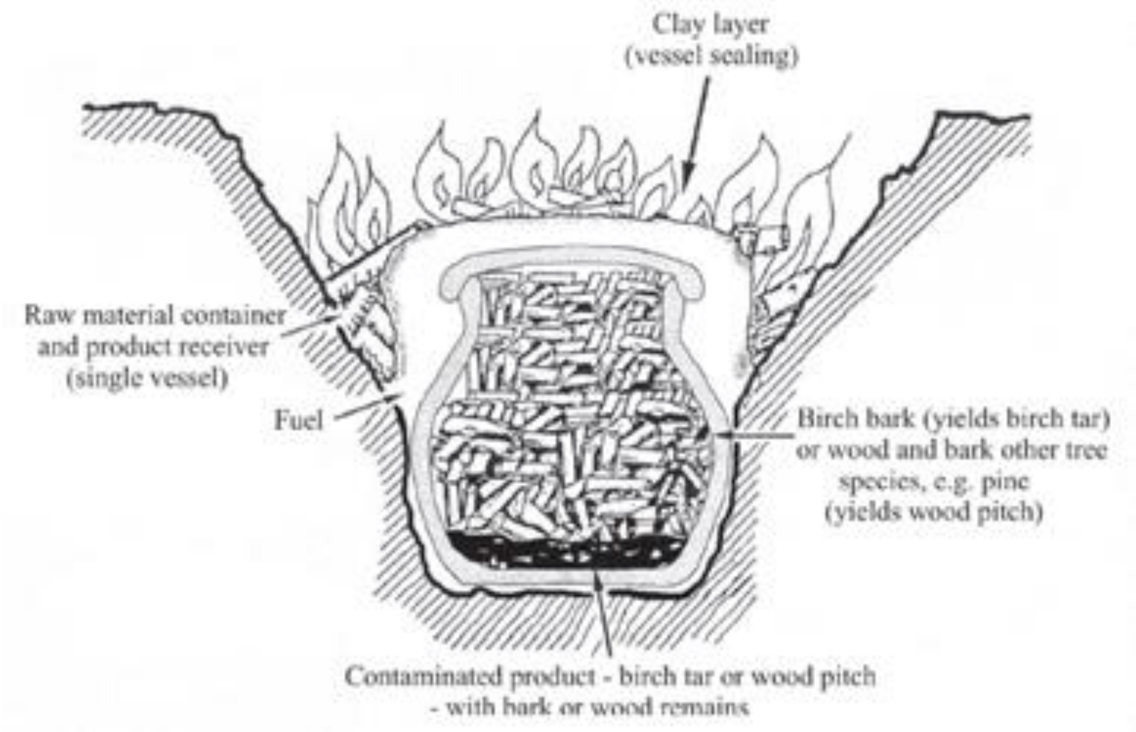


Figure 8.4. Diagram of a single-pot resin extraction process (extracted from Pietrzak 2012).

8.2.2. Resin use

It is difficult to explain with certainty the use of resins in each case. Of the seven potsherds in which the presence of pine resin has been identified, from Coro Trasito and Cueva de El Toro, we only have completed of the vessels from Cueva de El Toro (Figure 7.6). Therefore, it is almost impossible to give an evidence-based explanation to the use of resins in Coro Trasito, since they could have been used for the repair of the pots, for waterproofing or the vessels were used for the extraction of resin and/or storage it. In the case of Cueva de El Toro, it has already been proposed that resin use is linked to the waterproofing of the walls (see Tarifa et al. 2019).

In other cases where vegetable resins have been detected in prehistoric pottery, it has been proposed that their application is intended to waterproof the walls of the vessels, as has been suggested for the Neolithic sites of Paliambela, Makriyalos (Urem-Kutsou et al. 2002; Mitkidou et al. 2008), Neustadt (Saul et al. 2012) and for hundreds of vessels from different sites in the United Kingdom (Mukherjee et al. 2008). In other cases, the resin would have served as an adhesive together with the mixture of triterpenic products, as in the sites studied in France (Regert 2004, Regert and Rolando, 2002) or the medieval West Cotton site (Charters et al. 1993).

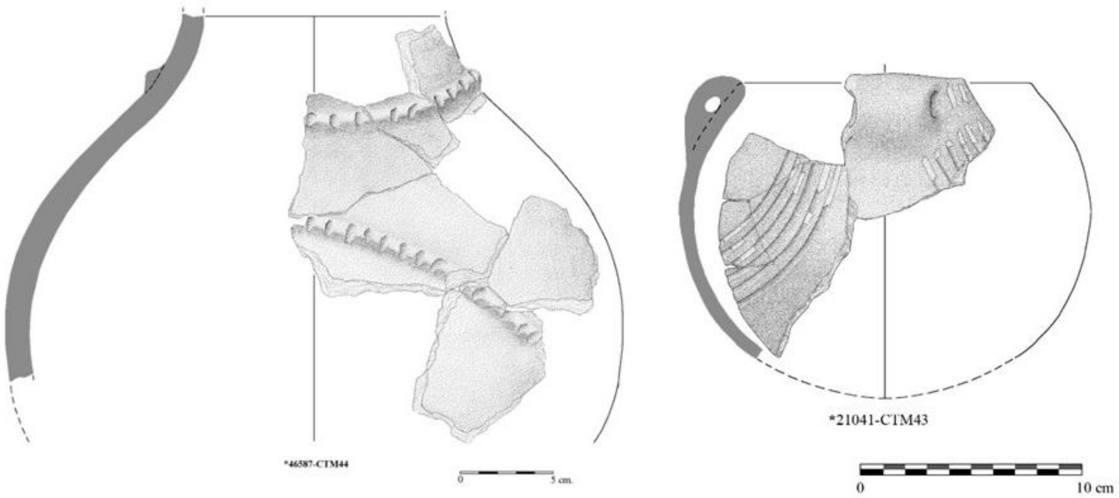


Figure 8.5. Sherds containing pine resin from Cueva de El Toro site.

8.3. Conclusions

The characterisation of waxes and resins in the containers of the sites studied has allowed us to identify products that were known to be exploited but could not be detected previously. The analysis of organic residues in ceramics constitutes a way of knowledge production, not only in the detection of natural substances for consumption, but also in the identification of products that offer technological properties to the containers. In this way, we can know the degree of technological complexity presented by ceramic vessels in the Early Neolithic in the Iberian Peninsula.

Chapter VIII.2. Morphology and use of pottery related to storage and processing of food products

Ceramic vessels are particularly suitable for many functions related to food activities: cooking with water, roasting, baking, mixing various ingredients, grinding, fermenting, storing, etc. (Skibo 2013). Compared with containers of perishable materials, such as baskets, gourds, and skin or net bags, fired clay vessels can accommodate far more varied functions and have longer and far more varied use-lives (Rice 2015). The foods prepared in these vessels can be varied, but some pottery can be specifically dedicated to the preparation of particular dishes (Skibo 2013). Pottery containers come in all shapes and sizes, and each food item or specific social event may have its own dedicated vessel or set of vessels in a well-equipped household. Ethnographically, determining the function of these vessels is a relatively simple matter of asking questions and recording observations. Unfortunately, we cannot go as far as that level of documentation with regard to pot.

The biomolecular and isotopic characterization of organic residues preserved in ceramic vessels provides essential information to approach the use of vessels, especially the relationship between technology, form and function of pottery (Burri 2003, Fanti 2015, Salque et al. 2016, Vieugué 2010). However, this link between residues and the morphotypological characteristics of vessels is rarely investigated in southern Europe (only Debono-Spiteri 2012, Drieu 2017, Fanti et al. 2018, Šoberl et al. 2014, Vieugué et al. 2016, Tarifa et al. 2019).

In the following chapter we explore the function of vases whose profile could be restituted and have been the object of analysis in this thesis. The variability of the shapes in this ceramic assemblage suggests that the vessels responded to different needs, such as, for example, cooking or storage. The identification of the functionality of the containers used and the characterisation of the products contained in them will allow us to approach the culinary activities practiced by the groups studied and will help us to better understand why the ceramic containers have certain morphological characteristics.

8.4. Functional categories

8.4.1. Pots for product processing

Placed directly on the fire, suspended above or near it, the ceramic potsherds allow to vary the intensity of the heating and the amount of water added to boil, simmer, roast or simply heat a content (Skibo 2013: 63). Therefore, they allow for greater dietary diversity compared to leather containers whose contents can only be heated by the addition of hot stones or compared to roasting techniques by direct contact with fire (Skibo 2013: 96). This makes certain foods more digestible (Eerkens 2005, Skibo 2013: 96-97), prepares soups and porridge (Le Mière and Picon 1998) or extracts certain substances from foods. For example, cooking in boiling water can remove toxic tannins from acorns (Pignone and Laghetti 2010, Thissen et al. 2010), extract vegetable or fish oils (Gallay 2012, Hally 1986, Hayden, 2009) or recover ossein and fat from bones (Hayden 2009).

Generally, vessels for boiling have closed shapes; medium wall thicknesses, which allows heat to be conducted better but also the presence of handles which allow their transport and mobility and withstands thermal stress without breaking (Rye 1981: 27). Among the potsherds catalogued for food processing, we find the casseroles, which are best suited for processing food exposed to a heat source. There are no documented traces of soot or ketones in the containers from this study, so we cannot determine if any of them were exposed to a heat source. This may be due to a post-depositional processes or cooking processes at temperatures lower than 250ºC which do not allow for ketones formation (Skibo 2013).

The processing of uncooked food involves a large number of processes, most of which can be used in ceramics, such as grinding, pressing, transfer, fermentation, moulding and separation by filtration and mixing by incorporation. Pottery may also present thick walls, which would be stronger and more resistant to sharp blows during pounding or stirring (Rice 2015: 422). These activities can be reflected in archaeological pots as use-wear traces. For example, some archaeological vessels have significantly worn lower internal surfaces, indicating that their contents have undergone transformations that do not necessarily require heating: mixing, cutting, grinding or sampling, for example (Fanti

2015: 345, Vieugué 2012). Other cups used to transfer products, by pouring liquids or semi-liquids, are identified by their shape: rims with spout, taps, etc. (Vieugué 2012).

Medium sized bowls have been interpreted as uncooked food processing cups, given the opening form they present. The morphology of these vessels, open and shallow, helps food processing and provides easy access to food handling (e.g. cutting, mixing). Among the products identified inside these containers, dairy products stand out, which would explain the type of pots in which milk would be processed to make fermented products (Drieu 2017).

8.4.2. Service/Consumption pots

Consumption or service containers are characterized by small sizes with or without gripping elements, which provide manageability to the containers. Among the cups catalogued for consumption in the studied archaeological sites, we differentiate between those with relatively open shapes and those with necks or spouts. The small bowls, which present open and shallow shapes, make food handling more accessible, but at the same time avoid spillage, and may be related to the consumption of solid or semi-liquid elements, such as stews. The jugs have more closed shapes, with collars and gripping elements, and are related to the transport, pouring and/or consumption of liquid products, such as water or milk, although in the cases studied, they also present substances of animal origin.

8.4.3. Storage pots

Pots for storage may vary in composition and in their primary and secondary formal characteristics depending on the nature of their contents (liquid or solid), on whether they are intended for long- or short-term storage, and on how frequently their contents are accessed (Rice 2005: 422). A survey of ethnographic data suggest that liquid storage vessels may be more variable in shape than dry storage vessels; liquid storage vessels are relatively taller as an aid for pouring, while long-term dry storage vessels may be relatively short and squat. Vessels used for long-term storage are generally large and, when full, too heavy for easy movement. They may be set into the ground or floor for stability (Rice 2005).

The storage pots identified in this study have a higher volumetric capacity. On the one hand, we identified a series of storage cups with highly open shapes, related to the short-term storage of animal and vegetable products. These cups would allow easy access to the contents that could be kept dry.

On the other hand, the closed deep necked jars would be related to the storage of meat products, and possibly other undetected products, either because they were not organic or because they would not leave evidences detectable by lipid organic residue analysis, such as water, grains or pulses. The presence of necks may help prevent spillages or other content losses.

8.5. Mixture of products or successive uses of pots

The combined presence of certain foods in a vessel can be very well identified when dealing with very different materials, for example, animal fats and waxes (Charters et al. 1995, Heron and Evershed 1993). On the other hand, it is considerably more difficult to demonstrate the mixture of materials of presenting a similar molecular composition, such as subcutaneous fat from ruminants and dairy products, especially in the absence of triacylglycerides (Regert & Dudd, 1999). However, isotopic analyses can provide valuable clues. Nevertheless, it is almost impossible to determine whether the mixture is the result of an intentional combination, for example a recipe for cooking, the manufacture of an adhesive, or whether it is the result of successive uses.

Some researchers have suggested that only the first use is recorded in the clay pores (Eerkens, 2005), others, however, suggest that the molecular signal of recent uses has replaced the one (Craig et al. 2004). In fact, molecular signals accumulate on walls during successive uses, which is particularly noticeable when the same container is used several times to cook different foods (Charters et al. 1997, Debono Spiteri 2012: 269-270, Evershed 2008b). Analyses performed on ethnographic containers have confirmed these interpretations: the accumulation of the molecular signal on the wall across multiple uses and fact that the carbonized surface residues reflect the last use of the container (Skibo and Deal, 1995).

Since organic matter is absorbed by the accumulation of successive uses, it is mainly the intensity of the use that will determine the amount of fat found in the ceramics, as well as the amount of lipids naturally present in the food. Containers which are likely to contain only one fat for a very short period of time will, for example, have a very limited lipid uptake (Regert 2007).

8.6. Formal and metric characteristics of vessels

Only vessels whose profile could be fully reconstructed (n=62) were selected for the functional study. These were formally described following formal descriptions based on the general shape (ovoid, hemispherical or ellipsoidal), on the mouth aperture (incoming, straight or divergent) and, in the case of necks, their description (narrow or convergent). As well as the indication of absence and presence of handles, and the description of these (with punch-holes, ribbon handle or simple handle).

Once this parameter has been described, we proceed to describe the metric variables. For each of the samples, the diameter of the mouth, the tangential diameter (body), the height of the vessel, the aperture index (diameter of mouth / tangential diameter) and the depth index (diameter of the mouth / height) were calculated. Finally, using the aforementioned values, the volume was calculated (Table 8.2).

The combination of morphological and metric variables has allowed us to propose a classification for the 62 containers coming from Cueva de El Toro (5280-4780/4250-3950 cal BC) (n=32), Mines de Gavà (4500-3300 cal BC) (n=10) and the three sites of Juberri (4500-3300 cal BC) (n=20). As a result, the containers were classified into 5 functional categories (Table 8.1):

Category	n	Shape	Aperture	Aperture index	Depth index	Volume average (l)
Little bowl	**6**	Hemispherical	Divergent	1,5	>2	0,5
Pitcher	**3**	Ellipsoidal	Incoming (neck)	<0,5	0,3	0,6
Casserole	**22**	Ovoidal	Incoming	0,6-1,5	0,5	0,5-1
Jar	**8**	Ellipsoidal	Incoming (possibly neck)	<0,5	0,3-0,6	5
Deep bowls	**14**	Ovoidal	Straight	1,2	<0,9	2
Shallow bowls	**9**	Hemispherical	Straight	1,5	1,5	1

Table 8.1. Results of functional classification from morphological and metrical combination data.

If we look at the data from the proposed classification (Table 8.1), we see clear differences between the smaller vessels, characterized by bowl or jar type vessels. Medium sized vessels are distinguished between deep open bowls or shallow and casseroles. Finally, the jars have large volumes and very closed shapes. As we can see, metric differences are significant for vessels with a volume greater than 3L, such as jars. While deep bowls, medium-shallow bowls and casseroles have similar volumes, with some outlayer. The smallest volumes fall into the categories of little bowls and pitchers (Table 8.6).

Samples		RIM diameter (mm)	Vessel height (mm)	Volume (Liters)	Opening rate	Deep rate
JUBERRI	JBA01	290,00	238,15	1,85	0,98	1,22
	JBA02	100,00	35,00	0,10	1,42	2,86
	JBA03	140,00	224,00	1,05	0,71	0,63
	JBA04	190,00	NA	0,60	0,84	NA
	JBA07	195,00	NA	0,50	0,97	NA
	JBA08	185,00	85,00	0,50	0,88	2,18
	JBA09	115,00	92,00	0,30	0,86	1,25
	JBA10	260,00	NA	0,50	1,03	NA
	JBA11	110,00	88,00	0,27	0,95	1,25
	JBA13	165,00	NA	0,50	0,97	NA
	JBA14	220,00	632,70	6,52	0,04	0,35
	JBA15	184,00	228,00	2,20	0,50	0,81
	JBA17	440,00	447,30	5,20	0,97	0,98
	JBA18	205,00	392,00	3,77	0,50	0,52
	JBA23	235,00	NA	0,40	1,00	NA
	JBA24	205,00	340,00	2,81	0,60	0,60
	JBA25	255,00	262,50	2,17	0,80	0,97
	JBA26	270,00	NA	0,60	1,06	NA
	JBA27	165,00	NA	0,25	1,20	NA
	CL78	65,00	NA	0,20	0,86	NA
CUEVA DE EL TORO	CTM01	105,00	330,00	2,15	0,39	0,32
	CTM02	120,00	105,00	0,36	0,92	1,14
	CTM03	180,00	145,00	0,80	0,90	1,24
	CTM04	135,00	187,50	0,95	0,68	0,72
	CTM05	195,00	162,50	0,76	1,86	1,20
	CTM07	190,00	120,00	0,63	1,41	1,58
	CTM08	255,00	152,50	0,97	1,76	1,67
	CTM09	285,00	150,00	1,02	1,10	1,90
	CTM10	65,00	62,50	0,10	1,08	1,04
	CTM11	130,00	190,00	1,03	0,50	0,68
	CTM12	140,00	125,00	0,20	0,80	1,12
	CTM16	150,00	NA	1,20	0,94	NA
	CTM17	148,00	NA	0,45	0,88	NA
	CTM18	169,00	NA	0,62	0,93	NA
	CTM23	125,00	132,50	0,65	0,66	0,94
	CTM24	172,00	NA	0,80	0,76	NA
	CTM27	180,00	92,50	0,38	1,16	1,95
	CTM29	215,00	347,50	3,65	0,50	0,62
	CTM31	200,00	NA	0,80	1,00	NA
	CTM32	110,00	175,00	0,75	0,84	0,63
	CTM33	110,00	92,50	0,50	0,72	1,19
	CTM34	140,00	105,00	0,50	0,58	1,33
	CTM35	130,00	NA	0,80	0,65	NA
	CTM38	150,00	130,00	0,80	0,61	1,15
	CTM39	185,00	185,00	1,10	0,83	1,00
	CTM41	130,00	137,50	0,70	0,84	0,95
	CTM42	113,00	NA	0,40	0,85	NA
	CTM43	150,00	137,50	0,60	0,83	1,09
	CTM44	140,00	287,50	2,25	0,43	0,49
	CTM45	146,00	NA	0,70	0,73	NA
	CTM48	130,00	NA	1,10	0,79	NA
	CTM50	156,00	NA	0,60	0,82	NA
MINES DE GAVÀ	MIG09	140,00	78,30	0,20	1,17	1,79
	MIG12	170,00	97,80	0,50	0,54	1,74
	MIG13	171,20	97,80	0,40	1,48	1,75
	MIG16	190,00	100,00	0,45	1,23	1,90
	MIG17	364,00	160,00	1,21	2,94	2,28
	MIG18	340,00	50,00	0,90	1,02	6,80
	MIG19	90,00	180,00	0,70	0,56	0,50
	MIG20	70,00	127,70	0,50	0,41	0,55
	MIG21	144,00	81,80	0,30	1,20	1,76
	MIG25	148,00	73,00	0,30	1,19	2,03

Table 8.2. Metrical parameters for functional analysis

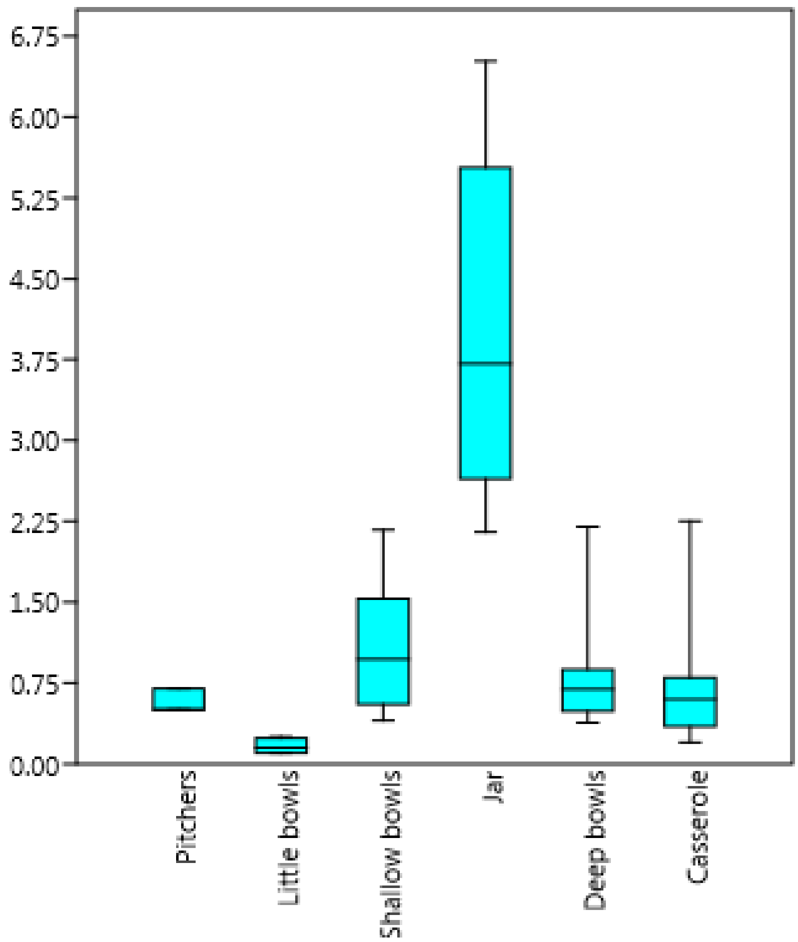


Figure 8.6. Volumes related with each functional category.

8.7. Functional classification

Based on the presented functional classification, we want to see if there is a relationship between the projected function and the specific pottery use. In this way we have elaborated a table of presence-absence of each one of the products identified in each one of the ceramics from the biomolecular analyses (Table 8.3).

	Shallow bowls	Deep bowls	Jar	Closed casserole	Litle bowl	Pitcher
Ruminant	5	2	2	2	1	2
Pig	1	6	2	10	5	1
Dairy product	2	1	0	0	2	0
Plant	0	3	0	3	2	0
Beeswax	2	0	0	0	0	0
Resin	0	0	1	3	0	0
Total	10	12	5	18	10	3

Table 8.3. Table with products associated to vessel forms from Cueva de El Toro, Mines de Gavà, Feixa del Moro, Camp del Colomer and Carrer Llinàs 28.

To identify possible relations between function and use, the morphometric groups have been analysed by means of a correspondance analysis with the results of the analysis of organic residues (Figure 8.7).

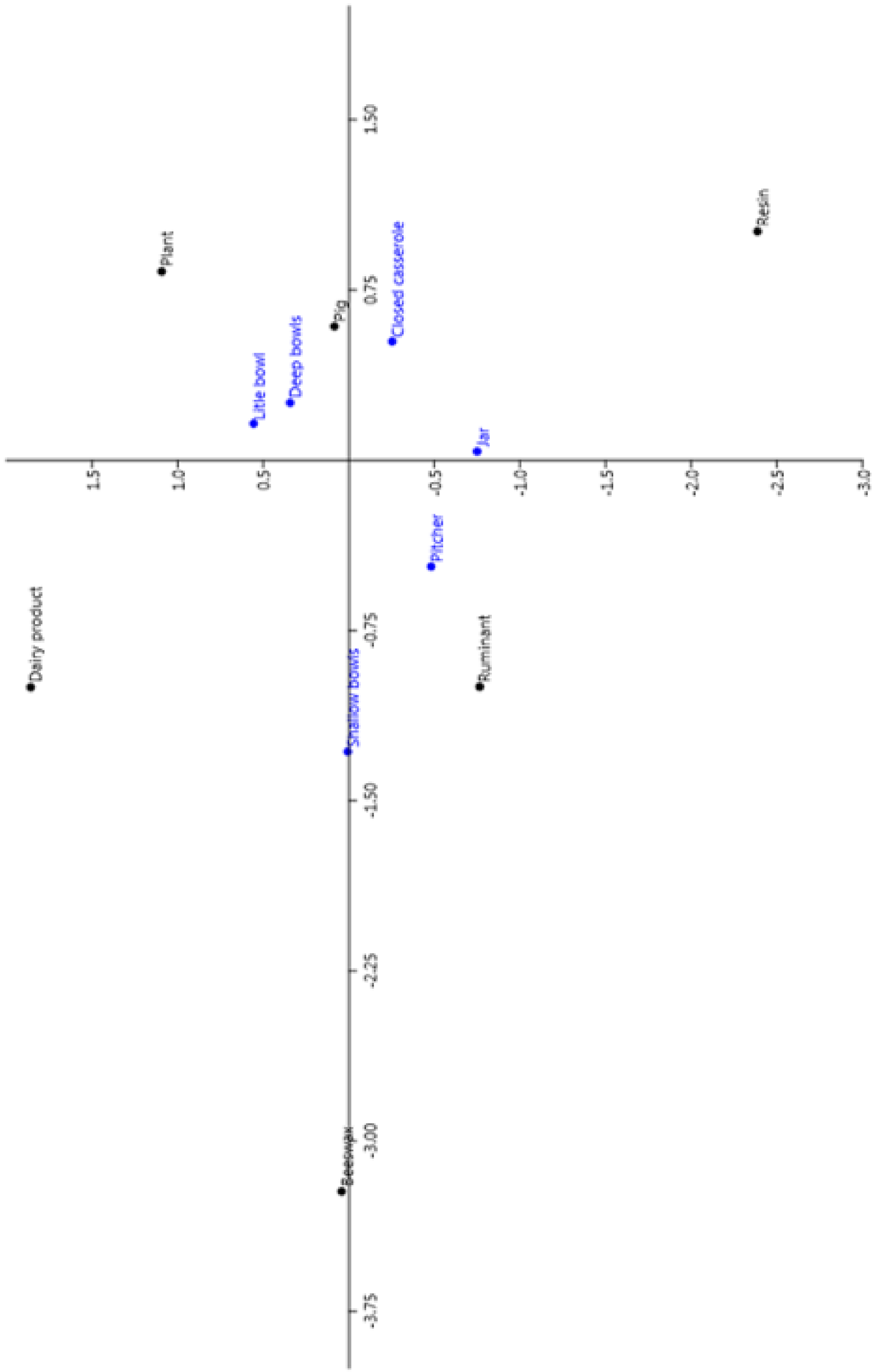


Figure 8.7. Correspondence analysis of morphometrical categories with the substances identified in them.

With 62.2% confidence, the CA presents a list of morphometric categories with the products identified in each of them. No precise correlation between the kind of foodstuffs and vessel morphology is evident, but two opposed behaviours emerge in the relationship between morphometry and the contained products (Figure 8.3), with a strong specialization between storage pots, related to meat and pine resin, and open-shallow bowls, featuring animal fats, dairy products and beeswax, which could be interpreted for food processing. On the other hand, the pots interpreted for processing (casseroles) and consumption (little bowls) present very similar foodstuffs, which could explain the process of food preparation and subsequent consumption.

Combining morphological, morphometric and chemical data from the Cueva de El Toro, Juberri (Feixa del Moro, Carrer Llinàs 28, Camp del Colomer) and Mines de Gavà sites, we identify five groups of ceramic morphologies that can be grouped into three categories of uses of containers, representing the three main functional areas (Rice 1987): food processing, service and storage (Figure 8.8).

From the isotopic characterization of the animal fats detected in each of the functional groups, we can observe a predominance of pork fat in the bowls, while the jars present a greater number of ruminant fats. The shallow vessels show a use focused on ruminant fats, although the presence of beeswax as a possible waterproofing agent has also been observed. Casseroles mainly contain pig fat. Finally, large containers combine animal fats with plants or resins. In general, although the group studied comes from three very different sites (some mines, an open-air site in a high mountain context and a cave site in the south of the Iberian Peninsula) the vessels have a very similar use that could be related to shape.

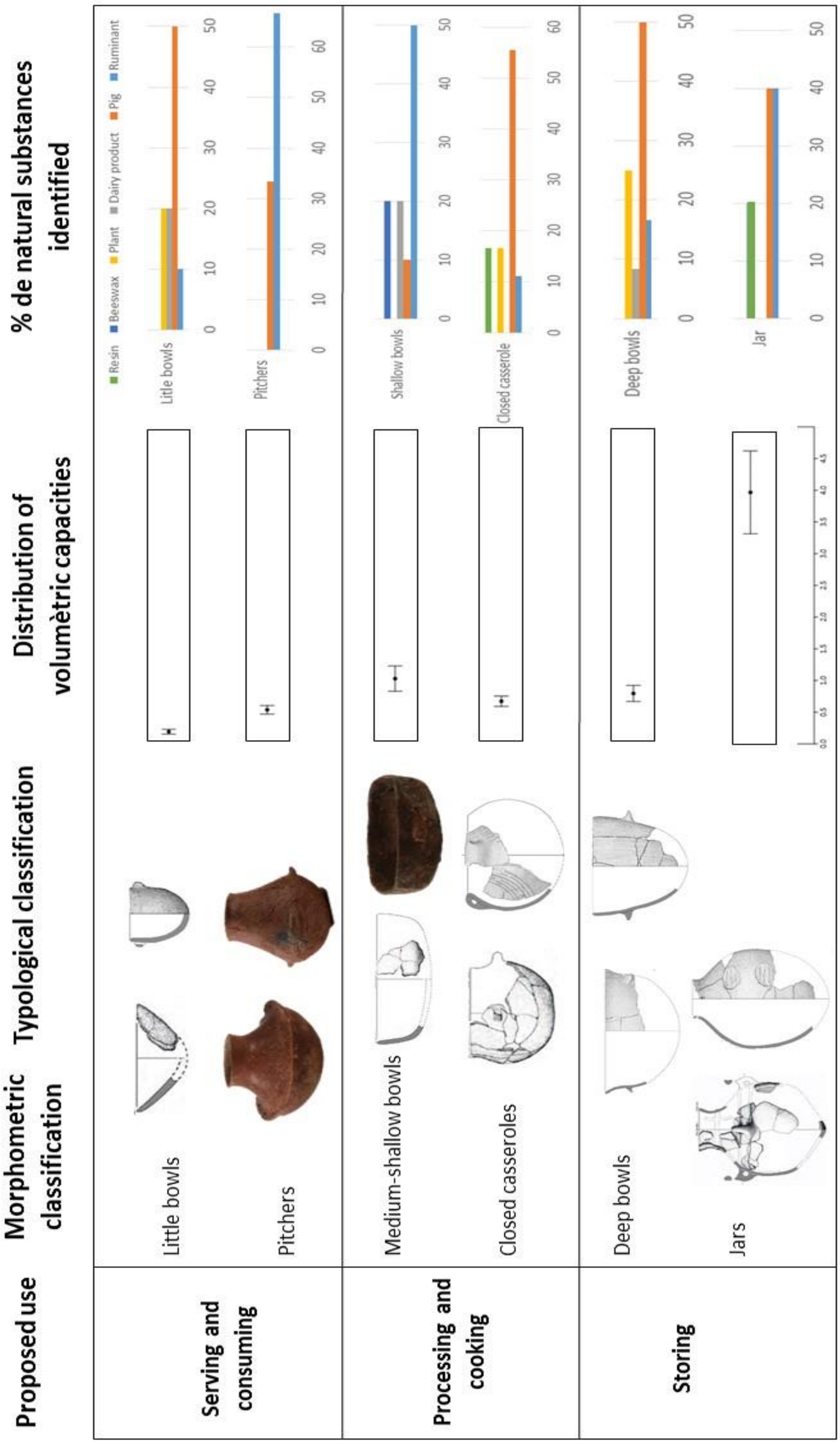


Figure 8.8. Functional classification of pottery from Cueva de El Toro, Feixa del Moro, Carrer Llinàs 28, Camp del Colomer and Mines de Gavà and % of natural substances identified.

8.8. The use of pottery in the western Mediterranean during the Neolithic

These results were compared with the pottery studied from the analysis of organic residues in ceramics in other sites in the Mediterranean. In the review of vessels from Southern European Neolithic sites that combine morphometric and biomolecular studies (Debono-Spiteri 2012, Drieu 2017, Fanti et al. 2018, Soberl et al. 2014), we can see that morphological variability and its effective use is recurrent over time and space.

From the morphological classification of the vessels studied from Cueva de El Toro (5280-4780/4250-3950 cal BC), Juberri (4500-3300 cal BC), Mines de Gavà (4500-3300 cal BC), Clairvaux XIV (4000-3500 cal BC), Grotte Lombard (5230-5125 cal BC), Gribaia (4500-4000 cal BC), Bau Angius (4500-4000 cal BC), Su Molinu Mannu (4500-4200 cal BC), Ajdovska jama (4340-4235 cal BC), Moverna Vas (4945-4135 cal BC) and Grotta San Michele (6100-5600 cal BC) resulted in seven categories of pots: C1. Jars of medium size, closed shape with neck. C2. Medium depth fairing bowls. C3. Plates very open, large and shallow. C4. Medium bowls with medium depth and open semispherical or truncated cone shape. C5. Small open bowls. C6. Very closed-mouth globular pots. C7. Large deep jars with closed shapes or narrow necks.

The contents of these pots were classified into meat, dairy products, vegetables, resin and beeswax (Figure 5). As we can see in the correspondence analysis graph, the jars (C1) contain mostly dairy products, these could be stored or processed in a liquid state (milk). The carinated bowls (C2) are related to animal fats and vegetable products, their open shape and average depth would allow processing and preparation with food mixture. Dishes (C3), medium bowls (C4) and bowls (C5) were used for processing and consumption of animal fats or meat. Finally, the storage jars (C7) also contained animal fat, some together with resin.

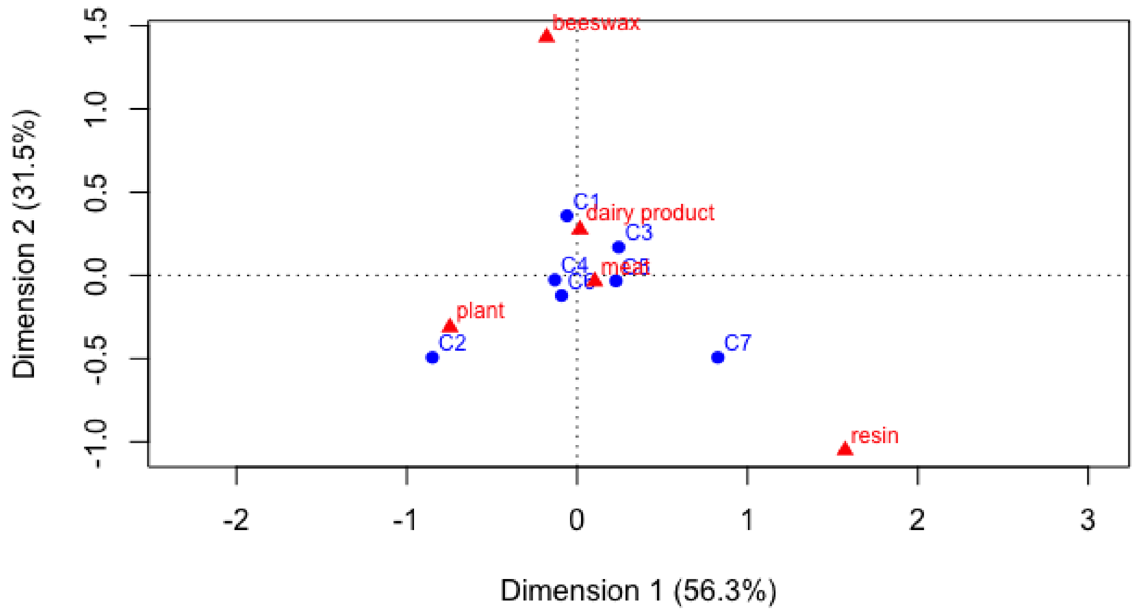


Figure 8.9. Analysis of the correspondence of the seven morphological and morphometric categories of neolithic vessels in southern Europe documented with the biomolecular result of their content.

In order to know more specifically the relationship of these categories with the origin of animal fats, a ternary plot was designed from the isotope values of each vessel and grouped by categories according to color (Figure 8.10). This triplot presents the values for dairy products, porcine fats and ruminant fats either in the mixture of dietary patterns or by the reuse of pots. As a result, we see that the categories C1 to C4 are related to ruminant fats and dairy products, while the categories C5, C6 and C7 contained mainly ruminant and porcine fats.

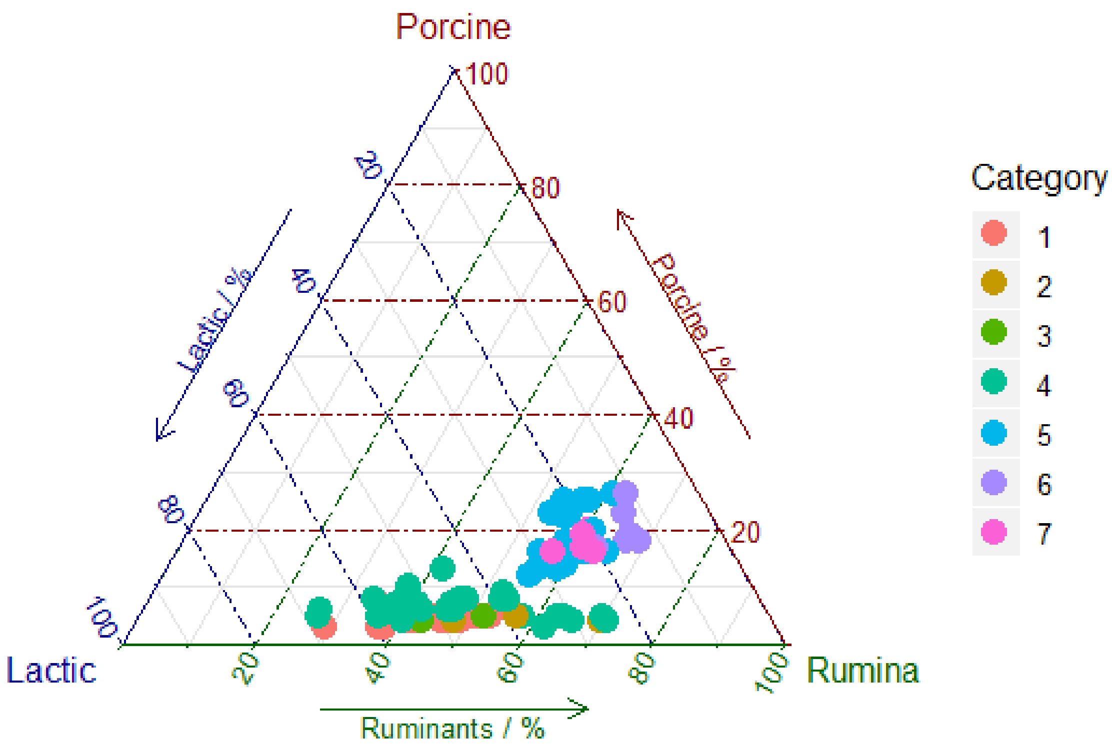


Figure 8.10. Ternary plot of the percentage of contribution of each group of animal fat obtained from isotopic values of neolithic vessels in southern Europe.

8.9. Conclusions

In this chapter we have been able to classify the pots studied in this thesis according to their morphometry and to establish three functional categories for the vessels. In the relationship of form and function with their specific use, no evident correlation has been detected that would lead us to believe that there would be vessels manufactured with the purpose of containing a single product. On the other hand, the application of residue analysis on ceramics has allowed a better understanding of the use of the pottery with the different food products detected, from the storage of meat products to the processing of dairy products.

Chapter IX. General discussion

The analysis of organic waste recovered in ceramic containers has made it possible to determine the content of neolithic containers from different geographical contexts and to overcome a large part of the taphonomic issues tied to the warmest climatic zones, such as the Mediterranean area (Drieu 2017, Evershed et al. 2008b; Gregg et al. 2009; Martí-Oliver et al. 2009, Tarifa et al. 2019). It is indeed necessary to evaluate the degree of preservation of organic waste recovered at studied sites from the Iberian Peninsula to justify the reliability of systematic and extensive future studies from this region.

Biomolecular archaeology applied to ceramic containers has shed light on the mechanisms involved in neolithization, from domestication to milk production and the use of ceramics as a new form of food processing and cooking. This work has made it possible to highlight milk exploitation in areas such as the UK (Copley et al. 2005a) and the Mediterranean area (Debono Spiteri et al. 2016), and to highlight the exploitation of domestic animals in regions where sufficient data were not available (Evershed et al. 2008b; Salque et al. 2013). Other works have focused on the management of domestic animals, such as pigs in the UK (Copley et al. 2005a; 2005b, Muherjee et al. 2007) or dairy farming (Evershed et al. 2008b).

Here, we will discuss the representativeness of the products detected inside the pots that have been analysed. We will first discuss, in a general way for all the results obtained in this thesis, the preservation of organic residues in ceramics retrieved in a Mediterranean context, i.e. where organic matter is more sensitive to degradation. We will also evaluate the representativeness of the products identified in relation to the archaeological, archaeofaunistic and malacological data available in each case. Finally, we will assess the potential of these analyses for the interpretation of the effective use of the containers from the vessels studied. Placed in the context of the European Neolithic, our results will allow us to analyse the divergences and similarities with other regions.

9.1. The preservation of lipids in ceramic containers in the Iberian Peninsula

Soil properties have a significant impact on the degradation of the organic matter (Reber and Evershed 2004b). The conservation of molecules from two containers that absorbed the same product but were buried in different types of sediment can present very different degrees of preservation, demonstrating the need to take into account and evaluate the potential different degradation mechanisms (Debono-Spiteri 2012: 128-188, Reber and Evershed 2004a). This conclusion was reached by several researchers, who evaluated the preservation of lipids according to soil pH, concluding that natural substances absorbed by containers would be better conserved in acid soils under aerobic conditions (Copley et al. 2005b; Gregg and Slater, 2010; Matlova et al. 2017; Smyth and Evershed, 2015). Experiments on the degree of soil degradation showed that soils with neutral pH (pH 6.5) favor degradation (DeLaune et al. 1981; Moucawi et al. 1981), while basic (pH 8.5) and acid (pH 5) soils are less favorable to the development of microorganisms responsible for some of the degradation mechanisms (Moucawi et al. 1981). Some authors also suggested that within basic sediments, such as calcareous soils, acids exist in the form of soluble salts and can therefore be more easily removed by leaching processes (Oudemans and Boon, 2007).

For this reason, lipid preservation in ceramics from Neolithic sites throughout the Mediterranean area tends to be lower than in other regions of Europe (Figure 9.1). The average recovery of natural archaeological substances in containers along the Mediterranean Sea, including the Near East (Evershed et al. 2008b Gregg et al. 2009; Gregg and Slater, 2010; Knappett et al. 2005; Nieuwenhuyse et al. 2015; Thissen et al. 2010), is about 40%.

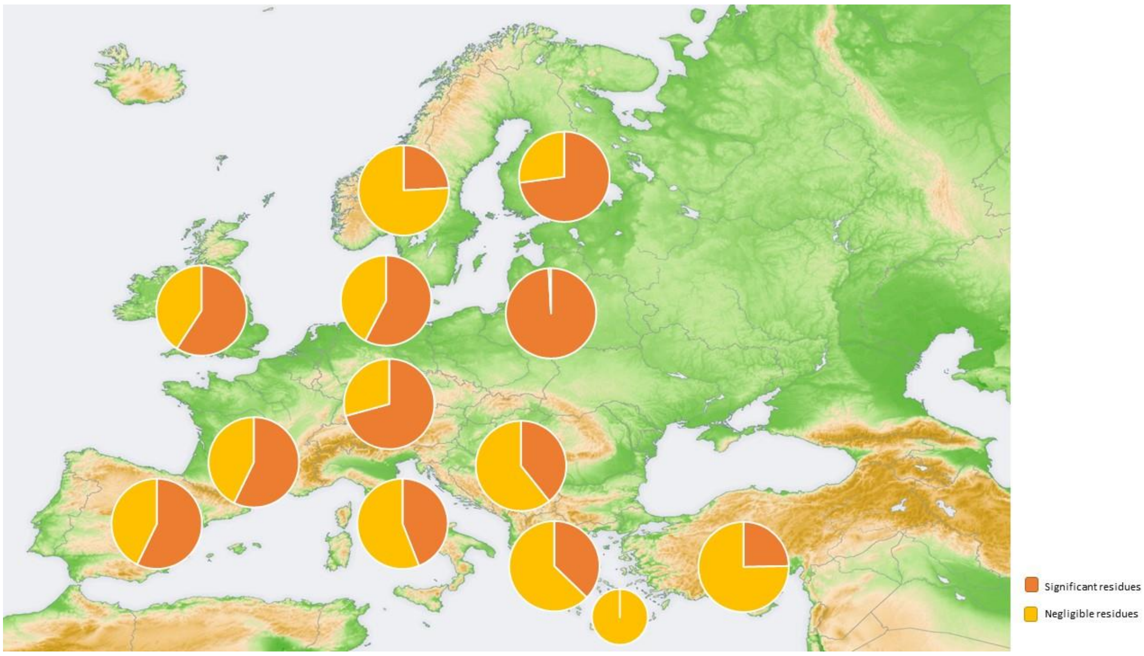


Figure 9.1. Map showing the conservation percentage of organic residues extracted from pottery from different prehistoric sites in Europe. The data were obtained from previous published studies (Regert et al 1999, 2001a, 2001b, 2003, Salque et al 2012, 2013, 2015, Mirabaud 2007, Mirabaud et al 2016, Salque 2012, Spangenberg et al 2006, 2008, Cramp et al 2011, 2014a, 2014b, 2015, Cramp 2008, Drieu 2017, Needham and Evans 1987, Oras et al 2017, Heron et al. 2013, 2015, 2016, Craig et al 2000b, 2003, 2005b, 2007, 2011, 2015, Hayek et al 1991, Bosquet et al 2011, Vieugue et al 2008, Quye and Ritson 1998, Debono 2012, Sanchez et al 1998, Hansson and Foley 2008, Foley et al. 2009, Decavallas 2007, Evershed et al. 1997d, 2008, Urem-Kotsou et al 2002a, 2002b, 2009, Smyth and Evershed et al 2015, Giorgi et al. 2010, Carrer et al 2016, Garnier et al. 2002, Faraco et al. 2016, Ribechini et al 2008a, Colombini et al 2005b, Rompou et al 2003, Raemaekers et al 2013, McGovern et al 2015, Copley et al 2005a, 2005b, 2005c, Mukherjee et al 2008, Campbell et al. 2004, Dudd 1999, Dudd and Evershed 1999, Matlova et al 2017, Soberl et al 2008, 2014, Ogrinc et al 2014).

Another factor that would lead to poor preservation in Mediterranean contexts is the hot and dry climate, alternating with periods of heavy rainfall. Such a climate favours both hydrolysis and oxidation mechanisms (Cramp, 2008; Evershed, 2008a). The most suitable environments for lipid conservation are flooded and dried (Regert et al. 1998, Copley et al. 2005).

On the other hand, the absence of lipids in a container may mean that it has been used to contain non-fat or low-fat materials, such as water or seeds (Copley et al. 2005b, Cramp, 2008: 194-196). This is the argument proposed by Debono-Spiteri for the absence of biomarkers in some of the containers studied in neolithic sites of the Mediterranean area (Debono Spiteri 2012: 268). However, the function of the containers is not the only parameter to take into account. Some works, which have studied ceramics in a domestic context, have shown that pottery related to the consumption of food products also presents low percentages of lipid concentration, as is the case of Kovacevo (Vieugué et al. 2008; Vieugué, 2010).

Another factor to take into account is the porosity of the surface of the vessels. Polishing treatments or clay compaction, which both reduce porosity, prevent the encapsulation of organic substances in the clay matrix. An exemplary case is that of the ancient Neolithic site of La Marmotta (Italy), where no organic matter conserved inside the walls could be identified, whereas the vessels presented carbonized deposits adhered on the surface of the walls (Spiteri-Debono 2012: 239).

In the case of the Iberian Peninsula sites analysed in this thesis, out of the 200 potsherds analysed, 124 (62%) yielded identifiable biomarkers in concentrations greater than 5 $\mu g \cdot g^{-1}$ of lipids per gram of sherd. Overall lipid abundances were higher than those reported for other studies in the Mediterranean area, and we suggest that this was due to the success of the extraction strategy we used in order to improve the results.

The significant percentage of lipid recovery in the Neolithic sites of the Iberian Peninsula has been possible thanks to the methodological strategy we followed (chapter III). In cases where a low concentration of organic matter had first been recovered, such as in the case of Cabecicos Negros, the use of the acid extraction protocol (Correa Ascencio and Evershed 2013) has significantly increased the yield of lipid extraction (Figure 9.2). In some samples where very low lipid concentrations (<5 $\mu g \cdot g^{-1}$) had been re-extracted, the acid lipid extraction has increased the concentration of these lipids and made visible those lipids most sensitive to degradation, such as plant sterols (Evershed et al. 1995b, Budja 2014).

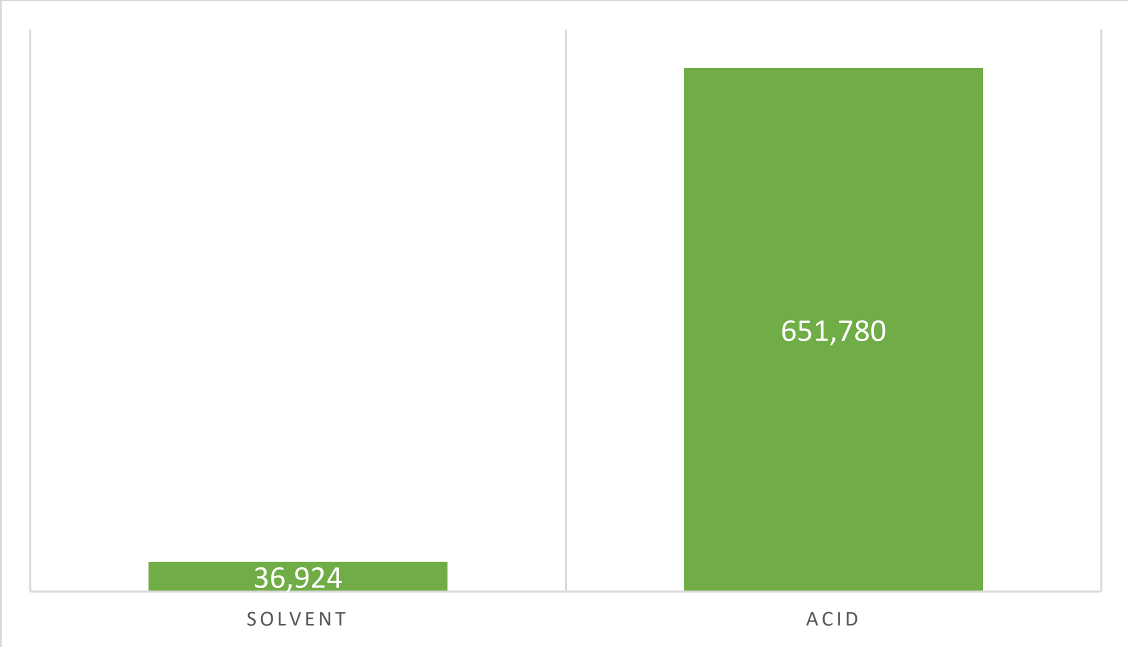


Figure 9.2. Comparative graph of the TLE extracted by solvent versus methanol acidified lipid extraction from the ceramic set analysed for organic residues from Cueva de El Toro.

As we can see in Figure 9.2, at the Cueva de El Toro site, the lipid signal increased considerably, which also allowed the detection of plant sterols and diterpenes.

This is not the first time that these two extractive methods have been applied in order to increase the concentration of lipids in Mediterranean ceramic samples that presented illegible substances. At Abri Pendimoun we observed a 40% increase in the lipid signal, which allowed isotopic analysis of a large number of samples (Drieu 2017).

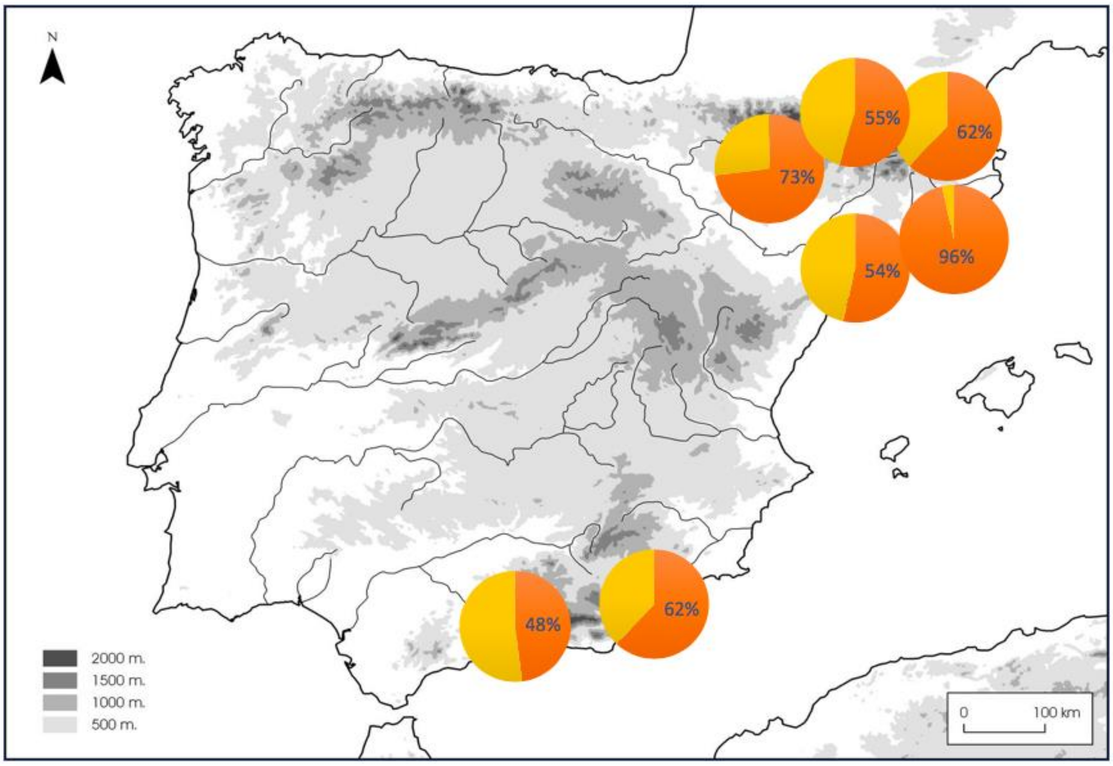


Figure 9.3. Map showing the conservation percentage of Iberian Peninsula archaeological sites analysed in this thesis.

The conservation of organic waste shows substantial variations between our different studied sites (from 48% at Cabecicos Negros to 96% at La Draga) but does not appear here strongly related to the context of each of these archaeological sites (Figure 9.3, Table 9.1), in contrast to the variability documented in the Italic Peninsula between coastal and inland sites (Debono 2012). In this work it was evidenced that the percentage of TLE recovered was 20% higher in coastal sites. The sites in the Pyrenees generally show good conservation of organic matter, as attested by previous studies in high mountain contexts (Debono-Spiteri et al. 2016, Carrer et al. 2016), not far from the conservation percentage of the sites located in the plain (ca. 60%). However, cave sites, such as the three Juberri sites, where conservation is assumed to be greater because they are sheltered from seasonal cycles, present an average percentage of conservation similar to open-air sites (ca. 50%). Finally, the results provided by the site in the anaerobic context of La Draga should be highlighted. In this site, samples were taken from vessels of four different levels: level III, under aerobic conditions, did not offer identifiable residues, whereas in the other three levels, characterized by anaerobic conditions, all the samples were marked by the presence lipids. The fact that sufficient concentrations of lipids (including the presence of TAGs) only occurred in samples extracted from levels VIa and VII, which correspond to levels where the soil presents a constant level of humidity but is not flooded as in the case of level VIII, is singular. This suggests that the preservation of lipids is better when the degree of humidity is relatively high and stable. Six containers from the Neolithic site of La Marmotta, which presents anaerobic conditions comparable to those of the La Draga site, were analysed by means of organic waste analysis. Five of these containers had carbonised waste deposits, testifying here again of the high degree of conservation of organic matter in this type of depositional conditions (Debono 2012).

9.2. Degree of representativeness of consumption of animal and plant species

The study of diet constitutes an important line of research in Archaeology that has been rapidly developed over the last 35 years. The topic has been approached with various methods ranging from archaeozoological and archeobotanical analyses to bioarchaeological methods (Budja 2014, Nigra et al. 2014, Oudemans 2007). In this thesis, we have been able to identify food products from domestic animals, such as pork fat or dairy products, based on the analysis of organic residues.

In order to know the food products processed and consumed at the Early Neolithic sites, we have also crossed the archaeozoological and archaeobotanical data of the sites under study to evaluate the degree of representativeness offered by organic waste analysis.

9.2.1. Animal products

Following the TAGs identification methodology (Mirabaud et al. 2007) and the isotopic analysis of palmitic and stearic fatty acids, lipids derived from porcine fats, ruminant fats and ruminant milk as well as dairy products were distinguished (Figure 9.4).

Some small variations appear in the subsistence practices carried out in the Neolithic sites located in the Iberian Peninsula which we focused on in this study: Coro Trasito, Juberri and Cueva de El Toro sites present a generally equitable exploitation of the available resources, whereas the sites of Cova del Sardo and Mines de Gavà denote a possibly preference for the exploitation of meat. In these cases, we interpret Cova del Sardo as a seasonal occupation shelter related to the exploitation of ovicaprine cattle and Mines de Gavà a site with a clear functionality of exploitation of variscita, where the reflected consumption is a part of domestic consumption, the spaces of which there is still no evidence. Finally, Cabecicos Negros presents a large number of pots with pork adipose fat compared to other resources and La Draga shows a dairy farm, as well as fats, which has been evidenced from mortality profiles (Saña 1999).

We then discuss these interpretations based on archaeological and archaeofaunistic data in each of the case studies.

9.2.1.1. Pyrenean context

Of the three Pyrenean sites studied in this thesis, Cova del Sardo and Juberri present a higher fragmentation of the faunal remains of the recovered complex. However, the analysis of organic residues in ceramics of the sets of these sites has allowed us to provide new data that contribute to a better understanding of the exploitation of these resources from 4800 cal BC. In parallel, the study of the faunal record present in Coro Trasito showed a predominance of domestic animal species (92%), among which we find the four main domesticated species - ovicaprines, bovids and pigs - as opposed to 8% of wild fauna, made up of species such as deer, chamois, rabbit, wild boar and Pyrenean goat (Viñerta 2015).

The consumption of ruminants and domestic pig fats in mountain contexts during the Neolithic period has been shown both from organic residue analyses, such as in Abri Urschai (Val d'Urschai, Switzerland) (Carrer et al. 2016), and from archaeozoological studies (Navarrete et al. 2017). The identification of domestic ruminants and non-ruminant fats in biomolecular analyses in ceramic containers of Coro Trasito and Juberri allows us to consider meat and dairy products derived from these species as exploited and consumed at both these settlements. It is remarkable to highlight the identification of dairy products in these sites in a Pyrenean context between the 6th and 5th millennium cal BC. While it is true that dairy farming appears well documented from the Early Neolithic period around the European continent (Debono-Spiteri et al. 2016), there is little evidence of these resources in high mountain contexts, such as in Font-Juvénal (France) (Debono-Spiteri et al. 2016), in Abri Urschai (Switzerland) (Carrer et al. 2016) or in Segudet (Andorra) (Yañez et al. 2002), although the identification of dairy products in the pots of this last site must be taken with certain caution since its detection was based on the distribution of fatty acids detected by gas chromatography and not on their isotopic value.

As far as the exploitation of wild fauna is concerned, few studies have focused on the complementarity of the protein contribution resulting from hunting activities based on the analysis of organic residues. These works have taken account the comparative analysis with wild animal fats to try to identify this type of products in ceramic containers (Craig et al. 2012, Evershed et al. 2002; Spangenberg et al. 2006; Lucquin et al. 2016). In this work we have been able to identify the exploitation of around 5% of the total fat as corresponding to wild boar fat in Camp del Colomer and Coro Trasito, as well as a vessel with $\delta^{13}C$ values consistent with deer adipose fat in Carrer Llinàs 28.

The bone remains of fauna recovered in some of the pits documented in the archaeological site of Juberri revealed a high level of fragmentation and thermal alterations by the action of fire, which has been interpreted as the final result of the anthropic activity related to the processing of these resources. Because the small size of the faunal remains and their poor preservation prevented their determination, we do not have precise data on the structure and composition of the domestic fauna of these sites (Fortó and Vidal, 2016). The study of the bone industry found in the Feixa del Moro helps us to identify the exploitation of different wild species, such as deer and wild boar (Llovera, 1986; Llovera and Beltran, 1991).

In summary, the data obtained from the analysis of organic residues identified in the ceramic containers have allowed us to identify a large percentage of fats from domestic animals over wild animals (90 vs 10%) in the sites located in the Pyrenean context. These are two settlements that, based on the available data, have been identified as stable with a complex economy based on agriculture and livestock, which would exploit the resources of domestic species, such as meat and milk, and the dietary protein intake would be complemented by the meat of wild species, which could reach the deposits as fresh meat, smoked, salted, and so on. On the other hand, the role that the shelter of the Cova del Sardo would play has been defined as seasonal and focused on the breeding of ovicaprine species, as well as agricultural exploitation.

9.2.1.2. La Draga

The animal management developed in the settlement of La Draga focuses primarily on livestock. It is a mixed livestock, with economic weight in the four main domestic species (see chapter VI). The contribution of domestic species (85%) to meat production is significantly higher than that of wild species (15%), with the fats contribution of bovines being more important than that of domestic pigs, sheep and goats (Saña 2011). In the case of *Suidae*, the mortality pattern occurs in the growth stage (<12 months of age). The cost involved in the breeding and exploitation of this species is lower than that required in cattle breeding, as they reach the optimum meat at an earlier age.

From the analysis of organic residues, it was possible to identify the dairy product origin of the fats in only two samples (D6 and D14), and in the case of the sample D6 it was possible to verify that the fat came from a domestic ruminant (cow, sheep or goat). These results are consistent with the beef farm proposed from the faunal analysis of the site, although the pig fat farm, which could not be identified in the ceramic containers, is under-represented.

On the other hand, a significant proportion of slaughtered cattle and goats do not exceed the first months of age and, therefore, would not have reached their optimum carnage (estimated for cattle between 2 and 4 years) (Saña 2011). This mortality pattern is related to the dairy farm at the specific sites of La Draga.

In the ceramic containers from the La Draga site, the dairy farm is observed from the distribution of TAGs and the isotopic analysis of stearic and palmitic fatty acids, which show that dairy products constitute 67% of the fats identified in the site. This evidence supports a dairy farm since the beginning of the Early Neolithic in the Iberian Peninsula, as it also occurred in Can Sadurní (Debono 2012) and in Cueva de El Toro (Tarifa et al. 2019). The processing of the milk, in which the ceramic containers would participate, could be related to the presence of *Silybum marianum* and *Euphorbia helioscopia* which, among its possible uses, could be used for curdle milk (Antolín 2013).

9.2.1.3. Mines de Gavà

The fauna documented in the mines 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 83, 84, 85 and 90 of Gavà corresponds to anthropic depositions once the mine ceased to be functioning and to natural fauna, such as rabbits or amphibians. The anthropically deposited fauna characterizes the presence of the four main domestic taxa, cattle (*Bos taurus*) (45%), sheep (*ovis aries*) and goats (*capra hircus*) (28%), and pigs (*sus domesticus*) (27%) (Saña 2009). Some of the documented remains of these species show traces of decarnation and thermoalterations, related to the herbidity and roasting of meat for food consumption. Some of the bones, especially bovine bones, would be used to make tools, such as miners' beaks (Estevez 1986).

In the vessels coming from the Mines de Gavà we have characterized adipose fats of ruminant origin (26%) and of pig (60%) through the analysis of organic residues. These results are consistent with the meat consumption of bovines, ovicaprines and pigs evidenced from archaeozoological studies. The absence of dairy products in the Mines of Gavà is remarkable, since they have not been proposed from the studies of the faunistic groups (Saña 2009) nor have they been detected from the molecular archaeology. This absence could be due to a taphonomic factor or to the fact that the exploitation of the animals was focused on domestic species with a purpose of meat feeding. Thus, from the analysis of organic residues preserved in ceramic containers, it has been possible to place the consumption of these meat products in a context where we do not have information on their production.

9.2.1.4. Cabecicos Negros

The study of the fauna documented in Cabecicos Negros is ongoing. The analysis of organic residues contained in the ceramic containers of this site has evidenced the important processing of meat of porcine species, which represent 94% of the fats characterized in the settlement. The analyses of organic residues in ceramics from European Neolithic sites attest to the meat exploitation of *Suidae* in the early stages of the Ancient Neolithic (Craig et al. 2011, Evershed et al. 2008, Mukherjee et al. 2008, Perić et al. 2013), whereas the analyses in Mediterranean contexts do not reveal such a high percentage of processing of these products in ceramic vessels (Debono 2012, Drieu 2017), where the pig does not represent even 10% of the identified animal fats.

Given the significant result of the study, the isotopic data of Cabecicos Negros have been compared with the reference model of current animal fats that has been designed in chapter IV, in order to understand the variability of $\delta^{13}C$ of $C_{16:0}$ that corresponds to the non-ruminant fats in the Cabecicos Negros site. Among the pig fats, pigs were identified with an omnivorous diet, more typical of a management of specimens within the settlement; pigs with an herbivorous diet, based on agricultural fodder and acorns; and wild boar.

The results show a differentiation between pigs with omnivorous diets and wild boar, while pigs fed with vegetable materials exhibit $C_{16:0}$ with $\delta^{13}C$ values in the middle of the ranges of pigs fed with protein and wild boar, suggesting the existence of differentiation of $\delta^{13}C$ values in $C_{16:0}$ in relation with the diet of the animals.

In the case of animal management at the ancient Neolithic site of Cabecicos Negros, we see that the diet of the *suidae* presents clear dietary differences. The differential diet of domestic pigs can potentially reflect the scale of husbandry practice in the past (Balasse et al. 2015, 2016). This appears to be supported by ethnographic studies on traditional husbandry systems in Southern Europe (Italy, Spain, Greece and Corsica) that also reveal some variability in pig diet in relation to management strategies (Albarella et al. 2007, 2011; Hadjikoumis, 2012; Isaakidou, 2011), and by recent isotope studies of $\delta^{15}N$ values of pig bones from sites in the northeast of the Iberian Peninsula which indicate a herbivorous feeding of pigs, generalized in that region, although with evidence of two specimens with a more proteic diet (Navarrete et al. 2017).

9.2.1.5. Cueva de El Toro

The evaluation of the results obtained in the study of the organic residues of pottery from the Neolithic phases of Cueva de El Toro reveals clear differences in the consumption of faunal resources between the two studied phases.

In Phase IV (5280-4780 cal BC), the most represented animal in the analysed samples is the non-ruminant, followed by ruminants. Although the presence of ruminant fats usually predominates in the analyses of organic residues in vessels from contemporary site to phase IV of Cueva de El Toro , they have also been identified in sites such as Grotte Gazel and Font-Juvénal (Spiteri-Debono et al. 2016), in France; Colle Santo Stefano (Spiteri-Debono et al. 2016), in Italy; Apsalos (Dimitrakoudi 2009), in Greece; Motel-Slatina (Perić and Bartkowiak 2013, oral presentation), in Serbia; Mâgura-Buduiasca, Kosylowce, Rehelyi Dülo or Póhalom (Evershed et al. 2008), in Romania; Niederhummel or Eythra (Salque et al. 2012), in Germany. In general, in the analysed datasets of this time interval, it is not usual that the identification of non-ruminants predominates over ruminants, as in the case for the phase IV of Cueva de El Toro. Although exceptional, this pattern has also been identified, in some sites in central and northern Europe, such as Bylany (Matlova et al. 2017), in Czech Republic; Wang and Zwenkau (Salque et al. 2012), in Germany; Åkonge, Åmose, Salpetermossen or Roskilde Fjord (Craig et al. 2011), in Denmark.

Mainly, the results obtained in the analysis of residues from pottery vessels seem to indicate the development of different strategies in the consumption of domestic faunal resources by the first Neolithic communities in Europe. These results suggest, as in the case of Cueva de El Toro, that the consumption of porcine fats could have been more relevant in some areas than it has been generally considered up to now, or that the culinary practices associated to ruminants and pigs have been different. On the other hand, according to archaeofaunistic data, ovicaprines predominate in this phase and the percentage of domestic fauna is already relevant (>75%), although researchers also highlight the presence of wild boar in phase IV (Watson et al. 2004). This leads us to propose that the results obtained in this work would reflect both domestic and wild animal fats contributions. This could indicate a certain degree of hunting exploitation that, at least part of it, would have been used as a protein supplement. It is expected that the expansion of these studies in more sites of similar chronology and with more numerous samples' sets will refine this hypothesis.

On the other hand, in the samples analysed from sub-phase IIIB of Cueva de El Toro (4250-3950 cal BC), the presence of ruminants predominates over that of non-ruminants, which is consistent with the fauna recovered at the site. In the Iberian Peninsula, similar results are found in Minas de Gavà (Barcelona) (Juan-Tresserras 2009) and Cova del Sardo (Lleida) (Tarifa 2015). In the rest of Europe there are also some similar results in sites such as Chalain (Regert et al. 1999), Clairvaux XIV (Regert 2010) and Giribaldi (Drieu 2017), in France; the Sardinian sites of Gribaia, Bau Angius and Su Molinu Mannu (Drieu 2017), in Italy; Moverna Vas and Ajdovska Jama (Soberl et al. 2014), in Slovenia; Neustadt (Saul et al. 2012), in Germany; Swifterbant (Raemaekers et al. 2013), in Holland; Yarnton, Runnymede and Abingdon (Copley et al. 2005), in United Kingdom; Skogmossen (Isaksson and Hallgren 2012), in Sweden; Vantaa Stenkulla or Maarinkunnas (Cramp et al. 2014), in Finland. It is therefore a relatively common result in the datasets analysed in Europe with similar chronology.

The detection of fatty acids from dairy products in samples CTM32 and CTM51, coming from phase IV, constitute the first direct evidence recording the consumption of dairy products in the south of the Iberian Peninsula. So far, this consumption during the early

Neolithic in the southwestern Europe had only been identified in sites of the eastern Peninsula, such as Can Sadurní (Barcelona) (5394-4037 cal BC), the open-air site Caserna de Sant Pau (Barcelona) (5250-5205 cal BC) (Spiteri-Debono et al. 2016) and Cova de l'Or (Alicante (5205-5021 cal BC) (Martí et al. 2009). The data obtained in Cueva de El Toro make it possible, therefore, to expand the geographical evidence of the use of this type of resources in the Iberian Peninsula since the first appearances of agricultural and live-stock groups in this area.

From the *kill-off* patterns of the faunal remains of phase IV of Cueva de El Toro, it has been proposed that ovicaprids would have been slaughtered at an age greater than three years, while in sub-phase IIIB they were sacrificed, in most cases, at less than one-year-old (Martín et al. 2004). In general, the data available so far indicate that the management of ovicaprids during phase IV of Cueva de El Toro would be mainly oriented to meat exploitation, whereas in sub-phase IIIB (4500-3500 cal BC) milk exploitation would be prioritised (Martín-Socas et al. 2004).

Comparing the data obtained in the analysis of residues of pottery from Cueva de El Toro with the data from archaeozoological studies, we notice the presence in phase IV, in a context where livestock management is apparently very focused on meat exploitation, of evidence of the consumption of dairy products in some pottery containers. This highlights once again the importance of these resources already from the first steps of development of the agro-livestock communities.

This situation contrasts with the data from sub-phase IIIB where, while the archaeofaunal data make it possible to propose a livestock production that could have had a clear interest in obtaining dairy products, the presence of these products has not been determined in any of the pottery samples of this phase.

This absence can be explained by different hypotheses. In the first place, it may be due to the low number of samples analysed. A second possibility could be that the use of the same pottery vessels in the processing of different types of products would have combined the lactic residues, which would have made it impossible to detect their presence with the current analysis techniques.

The identification of dairy products leads us to consider the work processes involved in their production, which have been until now not directly evidenced, and especially to consider who was in charge of their production. Numerous genetic studies assure that only few Early Neolithic individuals were lactose tolerant (for example Itan et al 2009). Thus, it is not clear in some cases whether the milk itself was consumed or whether it was transformed into cheese or other readily storable products (O'Brien and Bentley 2015). The fermented milk product can then be safely stored because it is still acidic enough to kill harmful microorganisms. Alternatively, cheese is made by adding the digestive enzyme rennet to acidified milk, coagulating it to the point that solids can be set aside and stored. Cheese making was possibly discovered by accident when milk was stored in a container made from the stomach of an animal, which still contained rennet. The heat from the sun turned the milk sour and the rennet turned the milk into curds and whey (Ridgwell and Ridgway 1986), could have allowed the consumption of these products without physiological repercussions.

Thanks to the analysis of the organic residues, it has been possible to verify the exploitation of the dairy resources from 5400 cal BC in the Iberian Peninsula. The knowledge of this exploitation, of which it has not been possible to have evidence until its identification through the analysis of the organic residues in ceramics (Debono-Spiteri et al. 2016, Copley et al. 2003, Craig et al. 2011, Evershed et al. 2008, Regert et al. 2001, Salque et al. 2012), makes us consider a new work process that had not yet been registered in prehistory.

Some ethnographic works have provided a gender approach to reconceptualise access to resources, considering the multiple spaces and levels at which livestock-related resources are managed.

Pastoral spaces are spaces with a high gender component, and for most pastoral societies, the household is within the sphere of women's control (Talle 1987, Wangui 2014). Pastoral women are responsible for the daily care tasks with livestock within households, including milking and raising young animals (Wangui 2014). This spatial bias obscures the importance of livestock-related activities that take place within the livestock farm and influence livelihood outcomes, including dairy resource management for household economy and food security (Yurco 2018).

In many pastoral societies today, women are in charge of milking and milk processing (Bollig and Schnegg 2013). As "milk managers", women have the responsibility to obtain, store and process milk, as well as allocate it to lactating dairy farmers, the family and children, and decide whether surpluses can be exchanged or sold (Hodgson 2000, Parsons and Lombard 2016). This demonstrates that women are/were respected milk managers in various pastoralist communities, controlling access and consumption.

What are the women's milking strategies and on what factors are their decisions based? Women in some pastoral communities separate the mother from the calf during the night to obtain milk for human consumption (Grandin 1988). In general, during the first four months, obtaining milk for human consumption is about 30% of total milk production in pastoral herds, but it may increase in relation to the development of a functional rumen (Nicholson 1984). The newborn calf feeds only on milk and the rumen, reticulum and omasum are underdeveloped compared to the abomasum (Svensk Mjölk, 2003). When the calf is 2-3 weeks old it can begin to eat forage and the forage particles begin a fermentation process upon entering the rumen, thus initiating rumen development (Sjaastad et al. 2003).

On the other hand, seasonal impact affects milk production and grazing strategies. During the dry season, cows have a higher age at first calving and calving intervals and lactation periods are longer in order to increase milk yield (Lindell 2013).

In conclusion, the ethnographic work of pastoral communities might offer us an analogy about the variable gender behaviours in the past and the opportunity to tentatively identify the producing agents of these new work processes involved in milk production.

9.2.3. Plant inputs

Vegetable processing is still an interpretative challenge with organic residues analyses, given the difficulties in interpreting vegetable origins. Plant remains have been attested in 9 vessels from four of the Neolithic sites of the Iberian Peninsula studied: Coro Trasito, Juberri, Mines de Gavà and Cueva de El Toro. The lipid profiles obtained generally includes low levels of $C_{16:0}$ and $C_{18:0}$ (with a P/S ratio >4), a wide series of alkanes and alcohols, as well as the presence of phytosterols and wax esters.

Plant biomarkers are more susceptible to degradation than animal fats and may be under-represented (Reber and Evershed, 2004b; Evershed, 2008a). Vegetable biomarkers are transferred to the containers when they are cooked or processed (grinding, roasting, etc.) (Skibo 2015). On the other hand, the dry grain stored in the large vessels would not be registered in the biomolecular record. Stored cereals and legumes would survive in the archaeological record mainly if the grains and seeds were charred. Although it is true that the archaeological-botanical record of the Feixa del Moro or Cueva de El Toro potsherds showed the presence of seeds inside the large containers (Antolín 2018), it has not been possible to identify them by means of biomolecular analysis. In other cases, such as La Draga, the grain would be stored naked in basketry containers or in paved structures that have been interpreted as grain storage sites (Tarrús et al. 2008), in which the ceramics would not participate in its storage.

Therefore, plant biomarkers detected in ceramic containers would have been processed for food consumption. We often find plants together with ruminant fats, fruit of the reuse of vessels for processing different products, or for the preparation of stews that would mix different products from different sources of origin, as has been proposed in other cases (Juan-Tresserras 2009). Another noteworthy fact is the presence of plants together with dairy products, as is the case in Juberri and Coro Trasito. The use of plants, such as *Silybum marianum* and *Euphorbia helioscopia*, can be used in the processing of fermented dairy products to curdle milk (Antolin 2013).

The low percentage of plant materials depicted by organic residue analyses in the context of studies on the reconstruction of the human diet from the analysis of isotopes in human bones and data obtained from carpological studies, lead us to think that vegetable processing is under-represented in biomolecular analysis. Carpological and pollen studies of Neolithic sites located in the Mediterranean strip of the Iberian Peninsula attest to the extensive cultivation of cereals and legumes from the early moments of the Ancient Neolithic (5600 cal BC), dominated by bare barley (Hordeum sp.), wheat (Trittícum sp) and peas (Pisum sativum), as well as the consumption of fruits, although to a lesser extent, of hazelnuts, acorns, apples and wild pears (Antolin 2013).

The studies on dietary reconstruction in sites such as Bóbila Madurell or Feixa del Moro suggest a diet rich in proteins and fibers derived from plant material resulting from agricultural activities in the neolithic populations of the Iberian Peninsula (Fontanals-Coll et al. 2015).

9.2.4. Lack of aquatic resources

The witnessed contribution of aquatic resources to the diet of the mesolithic populations of the Iberian Peninsula has been evidenced both in the ichthyological record and in isotopic analyses in human bones (Fontanals-Coll et al. 2014). However, during the transition to economic practices focused on agriculture and livestock, the consumption of aquatic resources is drastically reduced in the Mediterranean area (Le Bras-Gaude et al. 2010), in the Pyrenees (Le Bras-Gaude and Claustre, 2009) and in the Iberian Peninsula (Fontanals 2015). In reference to the evidence of exploitation of these resources in the archaeological record, we see that the number of fish bones or malacological remains is anecdotal in the studied sites, such as the presence of malacological remains in the Black Head site for ornamental purposes. On the other hand, in the Mines of Gavà fish characteristic from estuary areas and sandy bottoms have been identified, such as *Sparus aurata* and *Myliobatis aquila* (Estevez 1989), the presence of a large number of malacological species, among which the *Glycymeris sp*., and the remains of phocids (Bosch et al. 1999). Another example that presents a large number of malacological remains is La Draga, where *Glycymeris sp*., *Acanthocardia tuberculata and Cerastoderma glaucum* have been found, related to estuary areas, with abrasion and percussion marks related to the production of ornamentation elements (Oliva 2015).

Despite the presence, albeit scarce, of these resources, the food reconstructions of the Neolithic populations confirm the absence of contributions of aquatic resources to the diet (Fontanals-Coll et al. 2018), as well as the absence in the register of organic ceramic waste, which extends to the rest of the Mediterranean area (Debono 2012, Drieu 2017). On the other hand, in northern European countries this substitution of practices related to fishing or the collection of aquatic resources does not occur as suddenly as in the Mediterranean. Some Baltic and northern sites evidence a strong consumption of marine resources along with domestic resources (Brown, 2001, Brown and Heron, 2003, Morgan et al. 1992; Patrick et al. 1985, Copley et al. 2004; Hansel et al. 2004, 2011 Craig et al. 2007; Evershed et al. 2008a; Olsson and Isaksson, 2008; Hansel and Evershed, 2009; Heron et al. 2010; Craig et al. 2011; Paakkonen et al. 2018).

It is difficult to perceive the reason why the Neolithic populations of the Mediterranean would turn their backs on resources that had played such an important role during the Mesolithic and were easily accessible, given the proximity of most settlements to water sources or the coast. This leads us to think that the scarce exploited aquatic resources would not be processed in ceramic vessels. In this aspect, we can hypothesize that the scarce marine resources consumed in the Neolithic would be processed directly roasted in the fire or consumed raw. Although the evidence points to a food production in favor of land products.

9.3. Ceramic functionality based on the residues detected in its interior

9.3.1. Production processes and cooking of foodstuffs

Step by step, the recent studies of integrated functional analysis have made it possible to identify, thanks to the analysis of organic residues, the specific use of certain ceramic containers (Salque et al. 2013) and the global functioning of ceramic sets (Craig et al. 2015, Fanti 2015, Šoberl et al. 2014).

In this work, fatty residues of adipose tissues from ruminants and pigs, ruminant milk fats, vegetable products and beeswax have been identified in a variety of container forms, including cooking, such as in pots, and service containers, such as bowls (Chapter VIII). Despite multivariate statistical analyses, there does not appear to be an apparent association between the shape of the containers tested and the type of products processed in them.

The combined presence of animal fats together with vegetables, resins and waxes allows for many functional interpretations, from the use of products, such as resins and waxes, to provide technological properties to the containers, and the mixing of animal and vegetable products for culinary preparation, such as the preparation of stews. Conversely, samples that do not release or only release a small amount of lipids are important to consider when conducting a functional study. Indeed, the absence of lipids can mean not only that organic matter has been completely degraded, but also that the containers have not been used, have been used only on rare occasions or have only contained non-fat substances during their use: water, cereals, fruits, tools, salt, shells, etc. (Drieu 2017). We have compared the proposed function of the vessels and their specific use with other systematic and integrated studies in the Mediterranean area, such as Grotte Lombard (5230-5125 cal BC), Gribaia (4500-4000 cal BC), Bau Angius (4500-4000 cal BC), Su Molinu Mannu (4500-4200 cal BC), Ajdovska jama (4340-4235 cal BC), Moverna Vas (4945-4135 cal BC) y Grotta San Michele (6100-5600 cal BC) (Debono-Spiteri 2012, Drieu 2017, Fanti et al. 2018, Soberl et al. 2014).

Some of the forms have similarities between the sites from the Iberian Peninsula, Corsica, the Iberian Peninsula and Slovenia. In this way we see the presence of animal fats in storage containers, the mixing of animal fats and plants in processing pots and consumption bowls, and the presence of dairy products both in wide open containers and in collared jars.

This integrated study helps us to better understand the function of ceramic containers as well as to observe a similar pattern throughout the Mediterranean. The presence of vessels with similar volumes and variability ranging from small service containers, medium pots focused on food processing and cooking, and the presence of large jars related to storage is detected. In the comparison with the use of the containers, we observe a consumption of vegetable and animal products that presents many similarities.

9.3.2. Storage methods for food supplies

To accommodate the new role played by animals during the Neolithic period, pastoralists needed reliable, year-round food supplies, which would have, at least on occasion, involved storage. Storage has been considered a necessary precursor to agriculture (Bender 1978), as a concomitant characteristic of sedentary life (Testart 1982). This indicator of sociocultural complexity (Price and Brown 1985, Flannery 1972) promotes an important step in the conceptualization of private property (Bettinger 1999) and social control (Wesson 1999).

Food storage is key to understanding the impact of human populations on their ecological and developmental environments (O'Brien and Bentley 2015). For Sherratt's, food storage was key to short- and long-term energy provision. He developed numerous structures related to this practice, from ceramic pots to granaries (Sherratt's 1983). Storage containers are usually large in capacity. This type of packaging, although not cooked, is found throughout the Neolithic (Fanti 2015, Salque et al. 2013, Vieugué 2012) and they are also documented in Iberian Peninsula studied pots.

The analysis of organic waste does not allow us to identify storage practices, but the relationship between the form and function proposed together with the products identified inside it, allows us to propose approximations on the deferred consumption of food and conservation techniques. Unfortunately, the currently available data on other archaeological remains which relate storage installations are still inadequate to enable confirmation or refutation of the hypothesis made on the basis of the function of ceramic vessels.

Livestock and hunting are related to storage and surplus in different ways. Smoked or salted meat and lard may be stored for certain periods of time, depending on the shelf life of each category. About 70% of the containers coming from the Neolithic sites studied in this thesis and interpreted as storage jars, given their large volumetric capacity, present as the main product subcutaneous fats of animal origin, which are often mixed with biomarkers of non-food products, such as pine resin.

The pine resin detected in the Cueva de El Toro and Coro Trasito sites is interpreted as waterproofing the interior walls of ceramic containers (chapter VIII.1). On the other hand, the large jar that presents this product together with animal fats could be related to a sealer as an antibacterial element that would slow down the degradation of the meat inside the large jar (Binder et al. 1990, Bonfield et al. 1997). Another hypothesis for the presence of fats in interpreted storage containers is the use of these fats to preserve other food products, such as meat from other species or vegetables. A clear example of this practice today is the confit, which is made by cooking between 50 º C and 80 º C for a few hours the food product to be preserved with its own fat or an added fat without reaching boiling. By subjecting the food to medium temperatures over a prolonged period of time, pathogens and bacteria are eliminated (Duhart and Macbeth 2018). In this way, the product can be preserved and isolated from the degradation of microorganisms by candied fat. However, this storage technique would coeludiate the food product with the fat produced by the confit and this would only be visible in the analysis of organic residues.

The ability to manipulate the availability of food, both wild and domestic, and to regularly overcome the seasonal calendar of availability, through good and bad years, is a critical basis for the emergence of social differentiation in mid-range communities. In many geographical and temporal contexts, food storage precedes the domestication of plants, although storage does not automatically result in a food surplus (Kuijt 2011).

Conclusions

This research has applied the organic residue analysis extensively on the Mediterranean coast of the Iberian Peninsula.

The design of a current reference model was carried out from $\partial^{13}C$ analysis of modern animal fats and dairy products from the Iberian Peninsula. The model has facilitated the identification and differentiation of the fats that we did not know before, such as rabbit or wild boar. Moreover, it has allowed to extend a model that gathers isotopic values $\partial^{13}C$ of the different products which were potentially consumed during the prehistory; the finding that brings us closer to the interpretation of the organic compounds identified in the clay matrix of the containers. Finally, it offers a comparative reference with the isotopic variables of the Iberian Peninsula.

The preservation of lipids absorbed in the walls of ceramic vessels recovered in archaeological contexts of the Iberian Peninsula is reasonably good, in contrast to previous studies which were carried out in similar areas and comparable climatological conditions in the Mediterranean. The variable sampling that was carried out in different contexts and sites has allowed us to see that the variability in the preservation of organic compounds is not significant. However, it has been necessary to propose a methodological strategy that would combine two extractive methods in order to recover as much information as possible. The strategy showed that the function assigned to the individual vessels also determines how likely the lipids are to be absorbed within the ceramic walls, which results in the ceramics retaining a significant residue along the archaeological time scales.

Finally, 200 lipidic residues have been extracted from ceramic containers from 9 Neolithic sites in the Iberian Peninsula (5400-3500 cal BC). Analyses of GC-FID, GC-MS and GC-C-IRMS confirmed that around 20% of the containers had been used to process ruminant animal fats, while 5% of the fats were identified as dairy products. Domestic pigs accounted for 20% of the fats identified in the containers. Less than 5% of the fats were from the wild animals. High percentage of the fats from domestic animals found in ceramic containers in the peninsular context shows the relevance of the exploitation of livestock resources, Vegetable resources are around 5% and are mixed with animal fats. Of particular interest has been the oldest identification of beeswax in the Mines of Gavà, which is a direct evidence of the exploitation of this product in the Iberian Peninsula related to the technology of ceramic containers. The absence of marine biomarkers in all the extracted residues is consistent with the results obtained in other studies for the Mediterranean, such as on the French and Italian coasts. This suggests a decline in the consumption of marine resources, contrasted with analyses of dietary reconstruction of Neolithic populations in this region, and the decoupling of ceramic containers with the processing of these products.

Possible uses of the containers that were studied have been proposed the analysis of organic residues that were preserved inside the containers was used to infer the possible uses of those containers. Nonetheless, no clear relationship was found between the shape and function of the vessels, which suggests a varied use of the containers to process different products. The identified ceramic forms are coherent with the Neolithic vessels studied in other sites in the area of the Mediterranean basin.

Finally, the identification of milk residues in ceramic containers through the application of organic residue analysis has produced new direct evidence of milk production from the Early Neolithic in the Iberian Peninsula. Dairy products were identified in containers from different geographical contexts, from the south of the peninsula to the high mountain areas, thus providing reliable evidence that the nutritional qualities of dairy products were widely recognised and included in the diet since the Early Neolithic.

Further work

The referential model of animal fats from the Iberian Peninsula offers a series of future explorations that could build on this study. , We believe it is necessary to expand the reference collection for animal fats from the Iberian Peninsula, together with an exhaustive $\partial^{13}C$ isotopic analysis of the plants consumed by the animals in order to know the variability $\partial^{13}C$ in the diet. This extension can collect plants potentially consumed in prehistoric sites, as well as a reference sample of the characteristic aquatic resources of the Mediterranean. The collected data would allow a more robust comparison with the records obtained in other regions, for example the Northern Europe. Taking into account the interpretative limitations in relation to regional factors, the increase in the number of fats from the studied wild ruminant animals, such as: deer, chamois, Hispanic goats, etc., should be considered. To address the limitations from the seasonality, the analysis of animal fats collected by means of isotopic analyses of deuterium (δ^2H) is proposed, which would allow a variability in the amount of hydrogen in fats according to the region and the season of the year in which they were slaughtered. Finally, in order to design fat sampling, it is necessary to include the extraction of collagen from the same bones in order to establish C/N relationships that are comparable with the reference data of the archaeological fauna recovered from the sites. The difficulties that we have encountered in identifying the different varieties of plants processed in ceramic vessels have lead us to under-represent the interpretation of plant residues in relation to the use of ceramic containers analysed through the analysis of organic residues. This justifies the need to extend, the reference collection of lipids from different plants and plant resources potentially consumed in prehistory, as well as the application of more precise extraction and analysis methods. These techniques, such as HT-GC/MS or Pyrolysis-GC/M, allow the identification of protein and carbohydrate fractions, which are the major constituent of plants. In the same way, the complementarity of the phytolith analysis together with the residue analysis of the carbonized deposits detected in the ceramic containers can favour the identification of the different plants processed inside the containers.

Finally, more analysis on organic residues in ceramics from Neolithic sites in the Iberian Peninsula is necessary in order to evaluate the implication of the different products on a peninsular scale and propose new data for the implementation of the new subsistence practices developed during this period on a peninsular scale.

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Table 8.3. Table with products associated to vessel forms from Cueva de El Toro, Mines de Gavà, Feixa del Moro, Camp del Colomer and Carrer Llinàs 28.

Chapter IX. General discussion

Figure 9.1. Map showing the conservation percentage of organic residues extracted from pottery from different prehistoric sites in Europe. The data were obtained from previous published studies (Regert et al 1999, 2001a, 2001b, 2003, Salque et al 2012, 2013, 2015, Mirabaud 2007, Mirabaud et al 2016, Salque 2012, Spangenberg et al 2006, 2008, Cramp et al 2011, 2014a, 2014b, 2015, Cramp 2008, Drieu 2017, Needham and Evans 1987, Oras et al 2017, Heron et al. 2013, 2015, 2016, Craig et al 2000b, 2003, 2005b, 2007, 2011, 2015, Hayek et al 1991, Bosquet et al 2011, Vieugue et al 2008, Quye and Ritson 1998, Debono 2012, Sanchez et al 1998, Hansson and Foley 2008, Foley et al. 2009, Decavallas 2007, Evershed et al. 1997d, 2008, Urem-Kotsou et al 2002a, 2002b, 2009, Smyth and Evershed et al 2015, Giorgi et al. 2010, Carrer et al 2016, Garnier et al. 2002, Faraco et al. 2016, Ribechini et al 2008a, Colombini et al 2005b, Rompou et al 2003, Raemaekers et al 2013, McGovern et al 2015, Copley et al 2005a, 2005b, 2005c, Mukherjee et al 2008, Campbell et al. 2004, Dudd 1999, Dudd and Evershed 1999, Matlova et al 2017, Soberl et al 2008, 2014, Ogrinc et al 2014).

Figure 9.2. Comparative graph of the TLE extracted by solvent versus methanol acidified lipid extraction from the ceramic set analysed for organic residues from Cueva de El Toro.

Figure 9.3. Map showing the conservation percentage of Iberian Peninsula archaeological sites analysed in this thesis.

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ANNEX I. PUBLISHED PAPER

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ORIGINAL PAPER

New insights from Neolithic pottery analyses reveal subsistence practices and pottery use in early farmers from Cueva de El Toro (Málaga, Spain)

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Abstract
Archaeological potsherds have become a valuable source of information about diet and economic practices of past societies. We have studied the organic residues in prehistoric pottery from the Neolithic rock shelter of La Cueva de El Toro (Málaga, Spain) that was continuously occupied from the second quarter of the sixth millennium to the second millennium cal BC. The site of Cueva de El Toro is remarkable because it contains evidence that its inhabitants possessed a high technological level and complex subsistence practices based on the exploitation of livestock and agriculture. By applying gas chromatographic-mass spectrometry (GC-MS) and isotopic analysis (GC-IRMS), the goal was to determine the nature and origin of preserved lipids, and thereby provide new insights into food preparation and pottery function. Detection of fatty acids and traces of diterpene compounds originating from plants suggested a consumption of meat, dairy products and plants, as well as the pine resin utilisation. Furthermore, this work allows extending the data on faunal management and exploitation in Cueva de El Toro.

Keywords Neolithic · Andalusia · GC-IRMS · Pottery · Dairy products · Lipids

Introduction

The application of chemical analysis techniques to accurately determine the origin of the organic residues of prehistoric pottery has allowed to considerably expand our knowledge both on the consumption of different types of food products and on the use of different substances for various purposes. In the last 35 years, an increasing number of studies on pottery residues have shed new light into the daily life of Neolithic farmers in Europe (Regert et al. 1999; Craig 2004; Evershed et al. 2008; Mitkidou et al. 2008; Craig et al. 2011; Ogrinc et al. 2012; Salque et al. 2012; Saul et al. 2012; Raemaekers et al. 2013).

However, there are areas in Europe where the information available is still very limited or absent. This would be the case, for example, in the south of the Iberian Peninsula, where there have been only a few studies published (Juan-Tresserras 2009; Martí et al. 2009; Spiteri-Debono et al. 2016).

The study of determination of the organic residues in pottery vessels of the Cueva de El Toro (Málaga, Spain) is, in this sense, a significant novelty both for the results obtained and for constituting the first wide and systematic study for this area of the Iberian Peninsula.

The sequence of occupation at the site of Cueva de El Toro is broad, being particularly significant to the Neolithic occupation, from Early Neolithic 6200–5980 BP (5280–4780 2σ cal BC) to Late Neolithic 5320–5170 BP (4250–3950 2σ cal BC) (Martín-Socas et al. 2004).

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The goal of this study was to apply organic residue analysis to 50 prehistoric sherds from the Neolithic phases of Cueva de El Toro in order to determine the nature and origin of preserved lipids and thereby provide new insights into food preparation, conservation and consumption of the inhabitants. Likewise, the sampling of a wide sequence of human occupation allows including in the study of characterisation of organic residues a diachronic perspective and, therefore, the appreciation and discussion about the possible permanence and changes in the consumption and use of the different identified products.

Site

Cueva de El Toro is located in the south of the Iberian Peninsula, specifically in the karst range of Sierra de El Torcal (Málaga, Spain) at 1190 m above sea level (Fig. 1). The occupation of the internal space is carried out on large limestone blocks detached from the upper part of the cave (Martín-Socas et al. 2004). However, during the first quarter of the 4th millennium BC, there was a structural change (Martín-Socas et al. 2017; Égüez et al. 2016) that blocked the entrance until then and configuring a new access and a pit of 17 m depth. This event coincides with a change in the intensity of the occupation of the cavity.

The archaeological evidences of Cueva de El Toro show subsistence strategies with some differences between phase IV (Early Neolithic) and sub-phase IIIB (Late Neolithic). In general, the pattern of representation of the fauna is similar between the different phases, with the preponderance of ovicaprids (62% and 71%), followed by suids (17% and 5%) and lagomorphs (14% and 10%) (Watson et al. 2004) among other species. In the Early Neolithic, the percentage of representation of the domestic fauna is higher than 75%, while in the most recent period, it reaches values above 85%. It should be noted that there is a prominent presence of wild boar during phase IV. Spatial and micromorphological analyses reveal that phase IV is characterised by its seasonality and its pastoral orientation, while sub-phase IIIB is characterised for the duality in the use of the space, the result of the cohabitation of people and animals (Martín-Socas et al. 2004; Camalich-Massieu and Martín-Socas 2013; Égüez et al. 2016; Martín-Socas et al. 2017). Based on the micromorphological analysis of the archaeological sequence, it has been possible to determine a separation between the space used for livestock stabling and that linked to domestic activities.

In Cueva de El Toro, the organic remains show an excellent state of preservation. The number of vegetal remains of phase IV is low, highlighting the presence of wild vegetables such as the acorn. Meanwhile, in sub-phase IIIB—in which several hearths or combustion structures were documented—there is

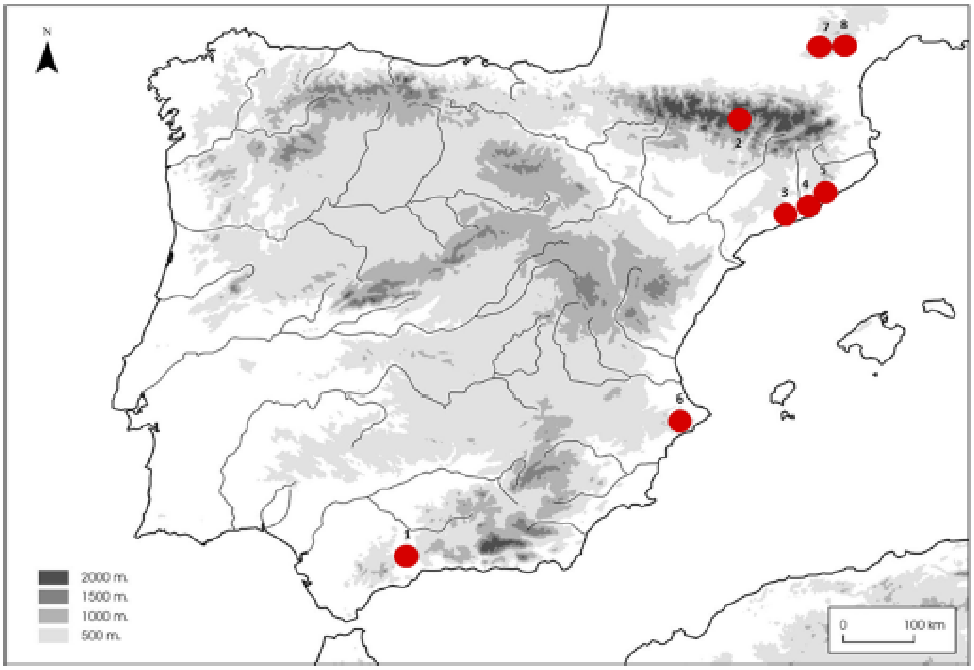

Fig. 1 Geographical situation of the sites mentioned in this work: (1) Cueva de El Toro, (2) Cova del Sardo, (3) Mines de Gavà, (4) Can Sadurní, (5) Caserna de Sant Pau, (6) Cova de l'Or, (7) Grotte Gazel, (8) Font-Juvénal

an exponential increase in the number of domestic species across the acorn. The most represented are *Vicia faba minor*, *Triticum aestivum/durum* and *Hordeum vulgare nudum* (Buxó, 2004). In addition, the anthracological analysis of the recovered charcoals shows that *Quercus ilex-coccifera* and *Phillyrea angustifolia* were the most commonly used woods in the hearths (Rodríguez-Ariza 2004), without the presence of pine.

On the other hand, functional analysis of flint tools indicates a predominance of butchery and hides treatment activities, with little presence of pieces used in harvesting plants/cereals during the Ancient Neolithic. While the Late Neolithic almost disappear, the tools of butchery and those related to harvesting plants/cereals are still of little importance (Rodríguez-Rodríguez et al. 1996, 2013).

Samples and methods

The study of determination of the organic residues is based on the analysis of 50 samples of Neolithic pottery, 26 of which correspond to vessels of phase IV and 24 to vessels of subphase IIIB. These samples correspond to vessels which represent the different morphologies, dimensions and decorative techniques documented in Cueva de El Toro.

For lipid extraction of potsherds, the surface of a subsample of the archaeological potsherds was cleaned with manual modelling drill to remove exogenous lipids (Stacey 2009); a 2 g fine powder was extracted with the cleaned drill and then stored in a glass tube previously muffled.

The powdered samples were extracted using a mixture of dichloromethane and methanol (3:1 *v/v*) after addition of 50 μL of internal standard (*n-tetratriacontane*). In addition to the archaeological samples, four blanks were analysed, two with the same internal standard and mixture of solvents and two only with the mixture of DCM and MeOH. Lipids were extracted using a microwave and heated to 70 °C for 10 min. The resulting extract was decanted, in each case, into glass tubes and dried by exposure to a gentle nitrogen stream. Next, each sample was dried by eluting them through glass columns filled in with anhydrous sodium sulphate. After removing the solvent, the dry extracts were redissolved in 50 μL of dichloromethane and derivatised with 50 μL of BSTFA (N,O-bis(trimethylsilyl)trifluoroacetamide) and the mixture heated at 70 °C for 60 min. After removing the derivatisation mixture, the sample was redissolved with 50 μL of isooctane prior to their analysis by gas chromatography.

The application of this analytical protocol provided appreciable amount of lipids in 48% of the samples studied, although 8 samples did not present enough significant concentration of lipids to be characterised (> 5 μg g^{-1}). To improve the recovery rate of lipids in the potsherds, we applied a second extraction method based on the use of sulphuric acid and methanol to the samples (CTM04, CTM05, CTM18, CTM21, CTM38, CTM43, CTM44 and CTM51) (Correa-Ascencio and Evershed 2014). This potentially offers a better chance in determining the origin of the fats and pottery use. However, the use of acid sulphuric will break down the ester link of heavier compounds, such as wax esters and acyl lipids. The lower resolution in the spectrum of potentially identifiable substances is the reason why this method is often used as an alternative analytical resource to the method of solvent extraction. The pottery powder is mixed with 4 mL of MeOH and sonicated in an ultrasonic bath for 15 min. Then, 200 μL of sulphuric acid was added, and the mixture was heated at 70 °C for 4 h. After cooling, 2 mL of hexane was added to each sample, thus creating two liquid phases. The hexane phase was removed with the help of a Pasteur pipette, taken to dryness under a nitrogen stream and subsequently redissolved in isooctane prior to gas chromatographic analysis.

The gas chromatographic analyses were performed on an Agilent 7820A gas chromatograph using a DB-5 MS column (30 m length × 0.25 mm internal diameter × 0.25 m stationary phase thickness). The splitless injector temperature was set at 300 °C and helium was used as the carrier gas. The temperature of the flame ionisation detector (FID) was 340 °C. The oven was initially held at 50 °C for 2 min, then the temperature increased at 15 °C/min to 170 °C, and finally to 320 °C at 6 °C/min, and held for a further 46 min.

The gas chromatography-mass spectrometry analyses were carried out using the same conditions as described for GC analysis, using an Agilent 7890A coupled to a mass spectrometer (Agilent 5975C).

Finally, and in the case that fatty acids are identified ($C_{18:0}$ and $C_{16:0}$), a third analysis is performed using a stable isotope mass spectrometer (Delta V, Thermo Fisher) hyphenated to a gas chromatograph via a combustion interface (Trace GC, Thermo Fisher Scientific). The GC is fitted with a DB-5 MS (60 m × 0.25 mm × 0.25 μm) column. The injector temperature is set at 310 °C. The oven is initially held at 80 °C for 1 min, then ramp at 30 °C/min to 120 °C, and finally increased to 320 °C at 6 °C/min and held for 21 min. Helium is used as the carrier gas. The combustion reactor is set at 940 °C.

The carbon isotope ratios are relative to the standard reference material vPDB, $\delta^{13}C$ ‰ = $[R_{sample} - R_{standard}]/R_{standard}$. The $\delta^{13}C$ values were corrected for the carbon atoms added during methylation using the following equation: $\delta^{13}C_{FA} = ((nC_{FAME}) \times \delta^{13}C_{FAME}) - \delta^{13}C_{MeOH})/n$, where $\delta^{13}C_{FA}$ is the corrected value for the fatty acid, n is the carbon chain length, nC_{FAME} is the total number of carbon atoms in the FAME ($n + 3$ for TMS ester and $n + 1$ for methyl ester), $\delta^{13}C_{FAME}$ is the value measured for the fatty acid methyl ester of carbon chain length n, and $\delta^{13}C_{MeOH}$ is the correction factor for the derivatising agent. Both correction factors were obtained by derivatising a known $\delta^{13}C_{FA}$ value with each of the derivatising agents (BSTFA and H_2SO_4-MeOH).

Results

Of the 24 sherds, animal fats were detected, 14 come from phase IV and 10 from sub-phase IIIB. A total of 18 samples (11 samples from phase IV and 7 samples from sub-phase IIIB) were extracted by solvent extraction, while 6 samples (3 samples from phase IV and 3 samples from sub-phase IIIB) were re-extracted by using sulphuric acid and methanol.

Phase IV (5280-4780 2σ cal BC)

In 14 samples of this phase, we detected the presence of high concentrations of palmitic ($C_{16:0}$) and stearic acid ($C_{18:0}$) (Table 1). These fatty acids are usually in the greatest abundance in archaeological lipid extracts with an even carbon number preference. Lipid profiles dominated by these fatty acids have been often observed in degraded animal fats (Copley et al. 2001). To provide more specific information, 24 sherds with the most abundant C16:0 and C18:0 acids were selected for GC-combustion-isotope ratio mass spectrometry (GC-C-IRMS) with the aim of distinguishing the origins of these compounds based on their stable carbon isotope value ($\delta^{13}C$).

The situation in the scatter plot of the obtained values of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ is compared with the current known values of lipids of different origins (Dudd et al. 1999; Evershed et al. 1997; Copley et al. 2003; Gregg et al. 2009; Dunne et al. 2012; Spiteri-Debono 2012). The situation in the scatter plot of the values obtained in the archaeological samples in relation to the current values of lipids allows to determine the origin of the identified lipids.

As can be seen in Fig. 2, the results of the $\delta^{13}C$ values reveal that sherds from phase IV dominates the consumption of porcine fats (samples CTM07, CTM11, CTM12, CTM16, CTM24, CTM33, CTM35, CTM41, CTM42 and CTM43), followed by the consumption of domestic ruminants (samples CTM03 and CTM44) and dairy products (samples CTM32 and CTM51).

On the other hand, in samples CTM43 and CTM44, where non-ruminant and ruminant fats have been detected, respectively, biomarkers of vegetable resin have also been identified, such as dehydroabietic acid and abietic acid (Fig. 3). The natural resins are exudates from the tree that, in certain cases and when are exposed to the light and air for a long period of time, undergo an oxidation process, as happens, for example, with pine resin (Azemard et al. 2016). The same oxidation effect can also occur when the resins are exposed to a heat source (Marchand-Geneste and Carpy 2003). The oxidation process causes the apparition of specific compounds (diterpenes and dehydroabietic acid) which serve as biomarkers of vegetal resins.

Sub-phase IIIB (4250-3950 2σ cal BC)

In 10 sherds of this phase, animal fatty acids were detected ($C_{16:0}$, $C_{18:0}$) (Table 2). From the determination of the isotopic value of the carbon from fatty acids ($\delta^{13}C$) (Fig. 4), it can be seen that in sub-phase IIIB, those that have fatty fats of ruminants (samples CTM01, CTM05, CTM13, CTM38, CTM46, CTM49 and CTM50) predominate, being lower in the presence of sherds containing non-ruminant fats (samples CTM04, CTM09 and CTM40).

Plant biomarkers have also been identified in samples CTM04, CTM05 and CTM38 (Fig. 5). The characterisation of plant products has been possible thanks to the high concentration of these samples of palmitic acid ($C_{16:0}$) and oleic acid ($C_{18:1}$), as well as the vegetal sterols *ergosta-5,22-dien-3-ol*, *stigmasta-3,5-dien*, *stigmasterol* and *β-sitosterol* (Christie 1989; Evershed et al. 1991; Cert et al. 1994).

Finally, in samples CTM01 and CTM04, we also identify polycyclic polyaromatic hydrocarbons (PAHs), such as *anthracene* and *phenanthrene*, volatile compounds produced during the combustion of woody products over 300 °C (Killops and Killops 2005) (Fig. 5).

Generally, in the samples from sub-phase IIIB, although predominate those in which only a single substance has been identified, there are three samples (CTM01, CTM05 and CTM38) in which we find two different substances and one sample in which it has been possible to identify the presence of three different substances (CTM04). The mixing of various commodities throughout the life of the vessels can also be seen by $\delta^{13}C$ values plotting close to, or between, the ranges of modern reference fats. For this interpretation, we assume that the contribution of fats from plants is likely to be negligible even if there is evidence of plant biomarkers in the vessels (Evershed et al. 1991; Evershed 2008).

Discussion

Evaluation of extraction methods for recovery of lipids

The solvent extraction has been shown to be a very effective system in the functionalisation of lipids from cave contexts in southern Europe, as is evidenced by the results obtained, for example, in Cueva de Nerja (Málaga, Spain), Los Castillejos (Málaga, Spain), Cova de la Sarsa (Valencia, Spain) (Spiteri-Debono et al. 2016), Cova del Sardo (Lérida, Spain) (Tarifa 2015), Can Sadurní (Barcelona, Spain) (Spiteri-Debono

Table 1 Peaks identified from phase IV pottery. Keys: FA*x* and FA*x*:*y*, free fatty acids containing *x* carbon atoms and *y* double bonds; MAGs, monoacylglycerols containing 16–18 acyl carbon atoms; DAGs, diacylglycerols containing 32–36 acyl carbon atoms; TAGs, triacylglycerols containing 48–54 acyl carbon atoms; and PAHs, polycyclic aromatic hydrocarbons

Phase	Samples ID	TLE (μg g^{-1})		Lipids detected	$\delta^{13}C$		Predominant commodity type (Copley et al. 2003; Gregg et al. 2009; Craig et al. 2012; Spiteri-Debono 2012; Dunne et al. 2012)
		Solvent extraction	Acid extraction		C16:0 ± 0.3 (‰)	C18:0 ± 0.3 (‰)	Ruminant adipose fat
	CTM03 8330	11.36		FA (16 < 18); PAHs: phenanthrene, anthracene	− 31.28	−32.94	Ruminant adipose fat
	CTM07 41905	9.13		FA (16 < 18, 10, 12, 14)	− 27.65	−25.18	Non-ruminant adipose fat
	CTM11 8521	12.83		FA (16 < 18, 8, 10, 12, 14)	− 24.07	−24.69	Non-ruminant adipose fat
	CTM12 9457	32.84		FA (16 > 18, 12, 14, 18; 1)	− 23.82	−22.31	Non-ruminant adipose fat
	CTM16 9862	22.4		FA (16 > 18)	− 26.46	−26.61	Non-ruminant adipose fat
	CTM24 1509	35.97		FA (16 > 18)	− 31.65	−27.14	Non-ruminant adipose fat
	CTM32 8704	78.4		FA (16 < 18, 10, 12, 14, 20), MAGs, DAGs, TAGs	− 26.75	−30.13	Dairy fat
	CTM33 18197	14.93		FA (16 < 18)	− 27.58	−24.99	Non-ruminant adipose fat
IV (5280-4780 2 σ cal BC)	CTM35 6922	58.41		FA (16 < 18)	− 24.76	−23.88	Non-ruminant adipose fat
	CTM41 40938	43.69		FA (16 < 18)	− 27.14	−26.01	Non-ruminant adipose fat
	CTM42 40939	15.3			− 27.76	−27.54	Non-ruminant adipose fat
	CTM43 40887	4.82	834.48	FA (8, 10, 12, 14, 16 > 18, 16, 1, 18; 1) Diterpenes; dehydroabietic acid, abietic acid; sterols:cholesterol	− 24.42	−24.05	Non-ruminant adipose fat
	CTM44 46587	1.54	325.3	FA (8, 10, 12, 14, 16 > 18, 16, 1, 18; 1) Diterpenes; dehydroabietic acid, abietic acid	− 30.56	−32.43	Ruminant adipose fat; pine resin
	CTM51 322	4.07	722.11	FA (14, 16 > 18, 20)	− 27.55	−30.44	Dairy fat

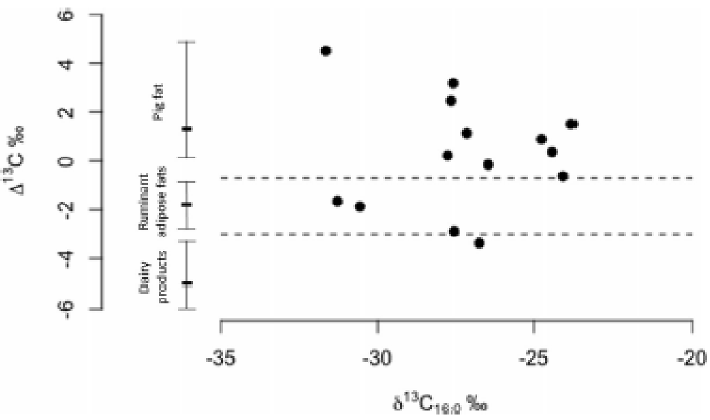

Fig. 2 Scatter plot showing $\delta^{13}C_{16:0}$ (*X*) and $\Delta^{13}C$ values (*Y*) of fatty acids extracted from phase IV pottery from site Cueva de El Toro. The ranges for the modern reference fats obtained from the current values reference (Evershed et al. 1997; Dudd et al. 1999; Copley et al. 2003; Dunne et al. 2012; Gregg et al. 2009; Spiteri-Debono 2012)

2012), Colle Santo Stefano (Italy) (Salque et al. 2012), La Marmotta (Italy), Grotta San Michele (Italy), Skorba (Malta), Nakovana Cave (Croatia), Apsalos (Greece), Toumba Kremastis Koiladas (Greece) and Ritini (Greece) (Spiteri-Debono et al. 2016). These are contexts where environmental conditions (temperature, humidity, redox conditions) are well regulated and contribute to the good preservation of organic residues in pottery vessels (Evershed 2008).

The analysis of samples from archaeological contexts where the conservation of organic residues in pottery vessels is much more deficient has stimulated the search of alternative methods to improve the possibilities of obtaining information, although this does not have the same degree of precision. Thus, an alternative extraction method is currently available from the application of an acidified methanol solution, which allows the extraction of chemisorbed compounds that cannot

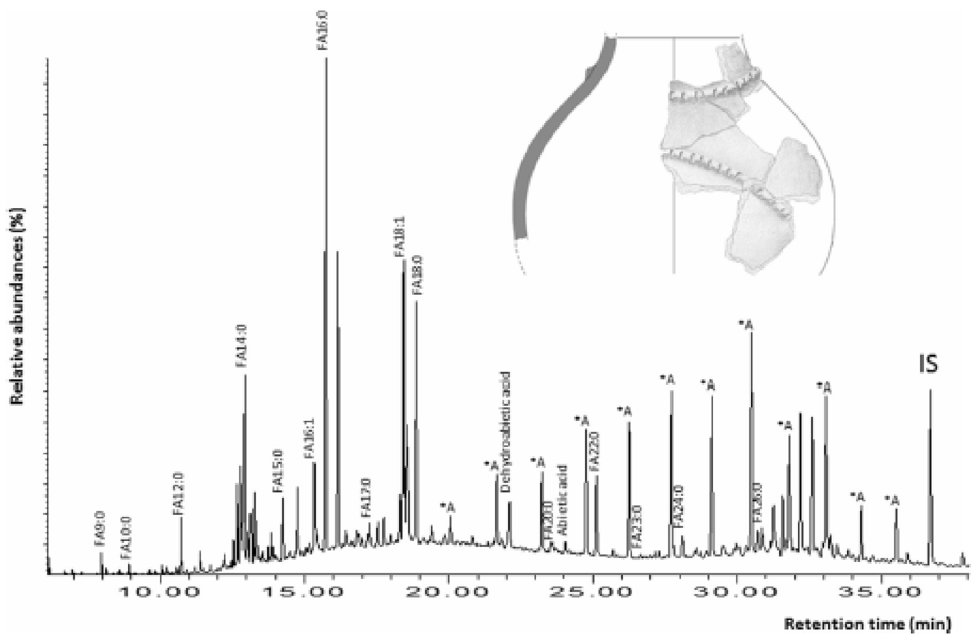

Fig. 3 Partial gas chromatogram of the trimethylsilylated TLEs from CTM44 pottery sample showing the various biomarkers detected: animal fats (FA*x*:*y* are fatty acids where *x* is the carbon chain length and *y* is the degree of unsaturation), pine resin (dehydroabietic and abietic acid) and contamination from plants and handling (unsaturated fatty acids and **A* are a long chain of alkanes, with odd-numbered carbon dominance)

Table 2 Peaks identified from sub-phase IIIB pottery. Keys: FA*x* and FA*x*:*y*, free fatty acids containing *x* carbon atoms and *y* double bonds; and PAHs, polycyclic aromatic hydrocarbons

Phase	Samples	ID	TLE (μg g^{-1})		Lipids detected	δ^{13}C		Predominant commodity type (Copley et al. 2003; Gregg et al. 2009; Craig et al. 2012; Spiteri-Debono 2012; Dunne et al. 2012)
			Solvent extraction	Acid extraction		C16:0 ± 0.3 (‰)	C18:0 ± 0.3 (‰)	Non-ruminant adipose: fat, plants, fire
	CTM01	5307-1	40.07		FA (16 < 18)	−28.61	−30.03	Ruminant adipose fat, plants
	CTM04	1047	2.19	374.25	FA (12, 14, 16 > 18; 16:1, 18; 1, 20, 22, 24); PAHS; phenanthrene, anthracene; sterols:ergosta-5,22-dien 3-ol, stigmasta-3,5-dein, cholesterol, stigmasterol, β-sitosterol, retinoic acid	−25.83	−26.02	Non-ruminant adipose fat
IIIB (4250 3950 2 σ cal BC)	CTM05	40692-6	4.43	461.85	FA (12, 14, 16 > 18, 16:1, 18:1, 20, 22, 24, 26); sterols:ergosta-5,22-dien-3-ol, stigmasta-3,5-dein, cholesterol, stigmasterol, β-sitosterol, retinoic acid	−27.11	−28.38	Ruminant adipose fat, plants
	CTM09	19711-1	46.46		FA (16 < 18)	−25.95	−25.73	Non-ruminant adipose fat
	CTM13	5661	21.31		FA (12, 14, 16 > 18, 12, 14, 18; 1)	−28.18	−30.2	Ruminant adipose fat
	CTM38	40698	2.78	537.2	FA (12, 14, 16 > 18, 16:1, 18:1, 20, 22, 24, 26); sterols:ergosta-5,22-dien-3-ol, stigmasta-3,5-dein, cholesterol, stigmasterol, β-sitosterol, retinoic acid	−28.4	−29.11	Ruminant adipose fat, plants
	CTM40	1215	40.65		FA (16 < 18)	−25.82	−23.88	Non-ruminant adipose fat
	CTM46	5811	53.22		FA (16 < 18)	−27.82	−28.33	Ruminant adipose fat
	CTM49	46716	33.84		FA (16 < 18)	−27.56	−28.84	Ruminant adipose fat
	CTM50	1038	78.36		FA (16 < 18)	−29.31	−30.37	Ruminant adipose fat

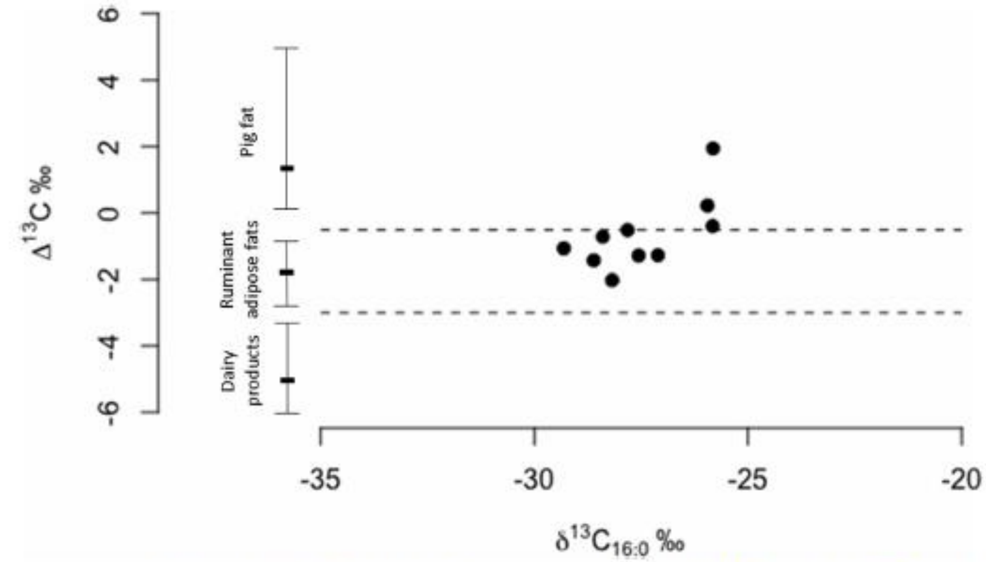

Fig. 4 Scatter plot showing $\delta^{13}C_{16:0}$ (*X*) and $\Delta^{13}C$ values (*Y*) of fatty acids extracted from sub-phase IIIB pottery from site Cueva de El Toro. The ranges for the modern reference fats obtained from the current values reference (Evershed et al. 1997; Dudd et al. 1999; Copley et al. 2003; Dunne et al. 2012; Gregg et al. 2009; Spiteri-Debono 2012)

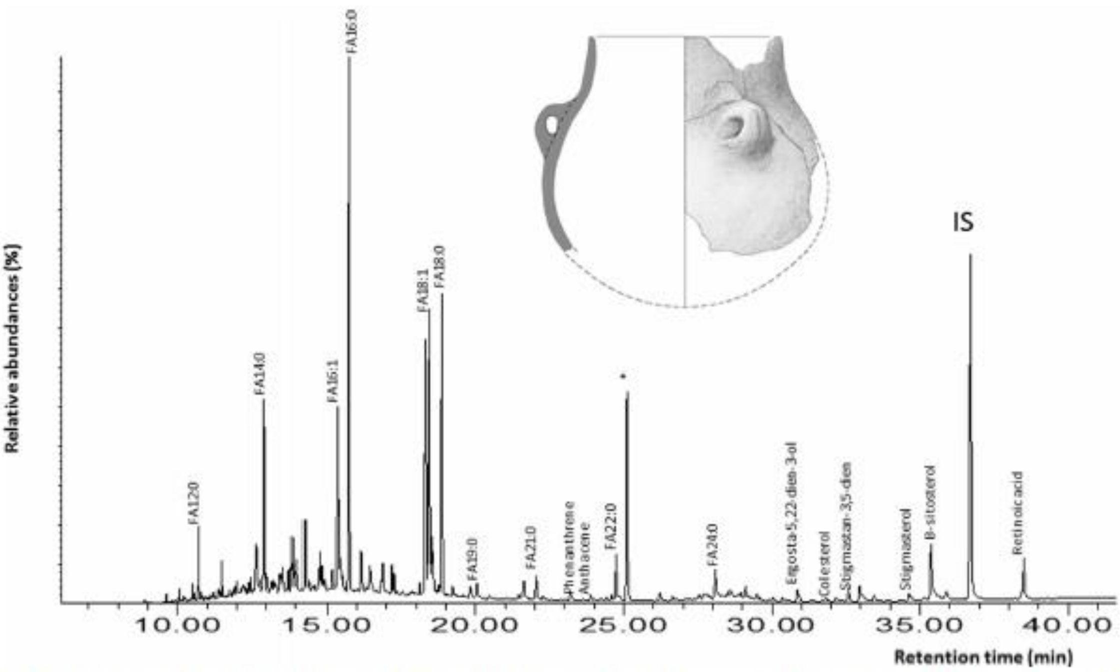

Fig. 5 Partial gas chromatogram of the trimethylsilylated TLEs from CTM04 pottery sample showing the various biomarkers detected: animal fats (FA*x*:*y* are fatty acids where *x* is the carbon chain length and *y* is the degree of unsaturation), wood combustion (PAHs), plant fats (sterols) and contaminants (*)

be extracted by the use of organic solvents. This method has been used to reanalyse some of the samples of pottery from Cueva de El Toro, which in the first instance, they had very low lipid concentrations and made impossible to determine their δ^{13}C value.

The positive results obtained in the analysis of Cueva de El Toro samples by using two different methods of extraction allow for some considerations.

First, it is observed again that in a cave context, as in this case, the application of organic solvents allows obtaining an appreciable amount of lipids, which in this case has been around 50% of the total samples analysed from this site.

Secondly, the application of the acidified methanol extraction method, although it has the disadvantage of restricting the spectrum of identifiable substances, allows obtaining high lipid concentrations in some of the sherds that had been negative in the first instance or, in certain samples, increase the signal of the concentration of lipids and, therefore, allow their identification.

In short, the complementary use of two different extraction methods has allowed obtaining a high number of sherds with interpretable residues and, therefore, improving the use of the archaeological samples analysed, which in turn allows improving the amount of available data and widening both the diversity of organic elements identified as the discussion on the processes of use and consumption of different types of substances and foods of the Neolithic communities.

Consumption of animal fats

The evaluation of the results obtained in the study of the organic residues of pottery from Neolithic phases of Cueva de El Toro allows us to observe clear differences in the consumption of faunal resources between the two phases studied.

In phase IV (5280-4780 2σ cal BC), the most represented animal in the analysed samples is the non-ruminant, followed by ruminants.

Although in the analyses of organic residues in vessels from contemporary deposits of phase IV of Cueva de El Toro usually predominates the presence of ruminant fats, they have also been identified in site like Grotte Gazel and Font-Juvénal (Spiteri-Debono et al. 2016), in France; Colle Santo Stefano (Spiteri-Debono et al. 2016), in Italy; Apsalos (Dimitrakoudi 2009), in Greece; Motel-Slatina (Perić and Bartkowiak 2013, oral presentation), in Serbia; Măgura-Buduiasca, Kosylowce, Rehelyi Dūlo or Póhalom (Evershed et al. 2008), in Romania; Niederhummel or Eythra (Salque et al. 2012), in Germany. In general, in the analysed sets of this chronology, it is not usual that the identification of non-ruminants predominate over ruminants, as in the case of phase IV of Cueva de El Toro. Although exceptional, this pattern has also been identified, in some sites in central and northern Europe, such as Bylany (Matlova et al. 2017), in Czech Republic; Wang and Zwenkau (Salque et al. 2012), in Germany; Åkonge, Åmose, Salpetermossen or Roskilde Fjord (Craig et al. 2011), in Denmark.

Mainly, the results obtained in the analysis of residues from pottery vessels seem to indicate the development of different strategies in the consumption of domestic faunal resources by the first Neolithic communities in Europe. These results suggest, as in the case of Cueva de El Toro, that the consumption of porcine fats could have been more relevant in some areas than has been generally considered up to now or the culinary practices associated to ruminants and pigs are different. On the other hand, according to archaeofaunistic data, ovicaprids predominate in this phase and the percentage of domestic fauna is already relevant (> 75%), although researchers also highlight the presence of wild boar in phase IV (Watson et al. 2004). This leads us to propose that the results obtained in this work would reflect both domestic and wild animal fats contributions. This could indicate a certain degree of hunting exploitation that, at least part of it, would have been used as a protein supplement. It is expected that the expansion of these studies in more sites of similar chronology and with more numerous samples sets allows us to specify this vision.

On the other hand, in the samples analysed from sub-phase IIIB of Cueva de El Toro (4250-3950 2σ cal BC), the presence of ruminants predominates over non-ruminants, data consistent with the fauna recovered at the site. In the Iberian Peninsula, similar results are found in Minas de Gavà (Barcelona) (Juan-Tresserras 2009) and Cova del Sardo (Lleida) (Tarifa 2015). In the rest of Europe, there are also some similar results in sites such as Chalain (Regert et al. 1999), Clairvaux XIV (Regert 2010) and Giribaldi (Drieu 2017), in France; the Sardinian sites of Gribaia, Bau Angius and Su Molinu Mannu (Drieu 2017), in Italy; Moverna Vas and Ajdovska Jama (Šoberl et al. 2014), in Slovenia; Neustadt (Saul et al. 2012), in Germany; Swifterbant (Raemaekers et al. 2013), in Holland; Yarnton, Runnymede and Abingdon (Copley et al. 2005), in the UK; Skogmossen (Isaksson and Hallgren 2012), in Sweden; Vantaa Stenkulla or Maarinkunnas (Cramp et al. 2014), in Finland. It is therefore a relatively common result in the sets analysed in Europe with similar chronology.

Consumption of dairy products

The detection of fatty acids from dairy products in samples CTM32 and CTM51, coming from phase IV, constitutes the first direct evidences recorded of the consumption of dairy products in the south of the Iberian Peninsula. So far, this consumption during the early Neolithic in the southwestern Europe had only been identified in deposits of the eastern Peninsula, such as Can Sadurní (Barcelona) (5394-4037 cal BC), the open-air site Caserna de Sant Pau (Barcelona) (5250-5205 cal BC) (Spiteri-Debono et al. 2016) and Cova de l'Or

(Alicante (5205-5021 cal BC) (Martí et al. 2009). The data obtained in Cueva de El Toro allow, therefore, expanding the geographical evidence of the use of this type of resources in the Iberian Peninsula since the first moments of the presence of agricultural and livestock groups in this area.

In the rest of Europe, there is abundant available evidence of the transformation and consumption of dairy products during the VI millennium cal BC: Grotte Gazel and Font Juvénal (Spiteri-Debono et al. 2016), in France; Arene Candide (Drieu 2017), Colle Santo Stefano (Spiteri-Debono et al. 2016), Fondo Azzolini or Ciccotto (Spiteri-Debono 2012), in Italy; Motel-Slatina and Drenovac (Perić and Bartkowiak 2013, oral presentation), in Serbia; Mâgura-Buduiasca, Kosylowce, Rehelyi Dülo and Póhalom (Evershed et al. 2008b), in Romania; Stare Nakonowo, Wolica Nowa, Smólsk, Miechowice, Brześć Kujawski or Ludwinowo (Salque et al. 2008), in Poland; Niederhummel, Wang and Brodau (Salque et al. 2012), in Germany; Åkonge, Åmose, Salpetermosen, and Roskilde Fjord (Craig et al. 2011), in Denmark.

From the *kill-off* patterns of the faunal remains of phase IV of Cueva de El Toro, it has been proposed that ovicaprids would be slaughtered at an age greater than 3 years, while in sub-phase IIIB, they are sacrificed in a large part of the cases, with less than 1 year of age (Martín-Socas et al. 2004). In general, the data available so far indicates that the management of ovicaprids during phase IV of Cueva de El Toro would be mainly oriented to meat exploitation, whereas in sub-phase IIIB (4500-3500 cal BC), milk exploitation would be prioritised (Martín-Socas et al. 2004).

Comparing the data obtained in the analysis of residues of pottery from Cueva de El Toro with the data from archaeozoological studies, we notice the presence in phase IV, in a context where livestock management apparently is very focused on meat exploitation, evidence of the consumption of dairy products in some pottery containers, which highlights once again the importance of these resources already from the first moments of development of the agro-livestock communities.

This situation contrasts with the data from sub-phase IIIB, where if the archaeofaunal data allow proposing a livestock operation that could have a clear interest in obtaining dairy products, the presence of these products in any of the pottery samples of this phase has not been determined.

This absence can respond to different possibilities. In the first place, it may be due to the low number of samples analysed. A second possibility would be that the use of the same pottery vessels in the processing of different types of products would have combined the lactic residues, which would have made it impossible to detect their presence with the current analysis techniques.

Consumption of vegetal resources

In samples CTM43 and CTM44 of phase IV of Cueva de El Toro, besides porcine and ruminant animal fats, biomarkers of pine resin have been detected.

The presence of diterpenes from pine resin is rarely identified in Neolithic contexts (Regert 2004). In some Neolithic sites, their presence has been interpreted as evidence of the use of adhesives or waterproofing, as has been proposed, for example, in Clairvaux-les-Lacs (Masschelein-Kleiner 1989) and Chalain (Regert 2004), in France; Makriyalos or Paliambela (Mitkidou et al. 2008), in Greece. However, the extension of organic residues analyses studies has allowed expanding the knowledge on the use of these products during recent prehistory. In places like Kuşakli (Steele and Stern 2017), in Cyprus; Neustadt (Saul et al. 2012), in Germany; Runnymede (Copley et al. 2003), Willington Quarry (Graham et al. 2004) or Skara Brae (Mukherjee et al. 2008), in the UK; as well as in the vessels of the Bronze Age of Peñalosa (Manzano et al. 2015), in Spain; Sn ret, Ryssgärdet, Kättsta or Nibble (Isaksson et al. 2010), in Sweden, the presence of vegetable resins has been detected inside of different vessels along with that of fatty acids with animal origin, having been proposed as an explanation that the resins could have been applied as a post-cooking treatment in order to waterproof the walls of the containers (Charters et al. 1995). In this way, the resins could have been applied during the production process of the containers to, later, develop an effective use probably related to the storage or processing of animal fats in liquid or semiliquid state (Correa-Ascencio and Evershed 2014).

On the other hand, in samples CTM04, CTM05 and CTM38 of sub-phase IIIB, plant biomarkers were identified that show the use of vegetable oils. Vegetable oils are present in large quantities in certain seeds or fruits (Marinval 2005; Evershed and Lockheart 2007).

In sub-phase IIIB of Cueva de El Toro, a large number of carbonised cereal seeds and legumes were found, as well as wild fruits, such as olives, acorns and myrtle and poppies (*Papaver somniferum* ssp. *somniferum*), which, in addition to its use as a synanthropic plant, the possibility of using its seeds to obtain oil is also pointed out (Buxó 2004; Guerra-Doce and López-Sáez 2006; Rovira-Buendia 2007; Martín-Socas et al. 2017). The biomarkers of vegetable oils preserved in the vessels of Cueva de El Toro could be evidence of the processing of some of these species, which could have been roasted, boiled or bathed in water (Hally 1986; Amouretti 2005; Mason and Nesbitt 2009). These procedures allow eliminating the toxic tannins of some fruits, making them suitable for human consumption (Saul et al. 2012).

In the samples of Cueva de El Toro in which substances with vegetable origin have been identified, the presence of animal fats has also been detected. This makes possible to raise the possibility that the mixture of these products could have been intentional with the purpose to enhance the taste of food; some containers were used in the processing of various products at different times.

Although the analytical verification of the mixture of these two products is certainly not very common, given the scant preservation that plant biomarkers generally have, it has been possible to identify their simultaneous presence in some cases, such as in the Neolithic sites of Polideportivo de Martos (Sánchez et al. 1998) and Cueva de Can Sadurní (Spiteri-Debono 2012), in Spain; Bercy (Regert et al. 2003) and Clairvaux XIV (Mirabaud and Regert 2016), in France; Maharski Prekop (Ogrinc et al. 2012) and Mala Triglavca (Šoberl et al. 2008), in Slovenia; Oudenaarde (Craig 2004), in Belgium; and Swifterbant (Raemaekers et al. 2013), in Holland.

When a mixture of products is detected, it is practically impossible to determine if the mixture was due to an intentional association (culinary guideline or manufacturing technique to waterproof the vessel) or if it is the result of successive uses of pottery. It has been proposed that it should be considered that the most superficial residue derives from the last use of the vessel (Mukherjee et al. 2008). However, it should be remembered that the surface of the vessel has a much greater risk of contamination of exogenous substances (Stacey 2009).

Conclusions

The chemical analyses of the organic residues of 50 samples of pottery vessels from Cueva de El Toro allows us to have, for the first time in the south of the Iberian Peninsula, relevant data on the effective consumption of different types of food during the Neolithic.

The data obtained indicate that in the oldest phase, Early Neolithic, pottery vessels were used to process animal and plant fats. The determination of the presence of dairy products in two of the analysed samples is very relevant since it constitutes the first direct testimony of the consumption of this type of food products in this area and chronology.

This observation makes it possible to extend this practice within the framework of the systematic use of dairy products by the first Neolithic communities. Finally, we must also highlight the finding of evidence of the use of pine resins in two vessels, for which it is proposed as a hypothesis that it may be a waterproofing technique for containers prior to the processing of animal fats.

Moreover, the study highlights the variability of elements consumed by human groups during the Early Neolithic in this southern area of the Iberian Peninsula, which includes the use of both domestic faunal resources and wildlife faunal resources.

In the Late Neolithic phase, as is generally observed in the occupation of the cave, clear differences with respect to the previous phase can be seen.

The most significant change recorded is the absence of evidence of dairy fats during occupation in phase III. Fats of ruminants and porcine fats have been identified, where the high presence of porcine processed fats in sherds over non-ruminants in phase IV is highlighted.

A second difference is found in the plant resources identified. They have been determined in three of the ten samples studied, which is a significant percentage that points to the extension of this practice at this moment. This time, and unlike what was determined in the samples from phase IV, these are vegetable oils, which could respond to a culinary guideline that mixes animal and plant fats or to the use of the container in the processing of different types of products at different moments.

The hypothesis proposed, derived from a pioneering study in this area, should undoubtedly be contrasted with new studies that expand the sample and the number of settlements in this period in the region.

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ANNEX II. MODERN REFERENCE DATA FROM LITERATURE

(adapted from Drieu, 2017)

Reference	δC16:0	δC18:0	Δ13C	Specie	Geography	Type
Craig et al. 2012	-29,6	-31,7	-2,1	ruminant	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,1	-31,9	-2,8	cow	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,3	-32,15	-2,85	cow	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-30,25	-32	-1,75	cow	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-30,2	-32,7	-2,5	cow	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,8	-32,2	-2,4	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-30,6	-32,7	-2,1	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-28,6	-30,4	-1,8	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,4	-30,8	-1,4	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,8	-31,6	-1,8	sheep	UK	subcutani

Copley et al. 2003 ; Dudd, 1999	-30,9	-32,9	-2	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,2	-30,5	-1,3	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-29,6	-31,5	-1,9	sheep	UK	subcutani
Copley et al. 2003 ; Dudd, 1999	-30,8	-32,6	-1,8	sheep	UK	subcutani
Evershed et al. 1997	-30,4	-32,2	-1,8	sheep	UK	subcutani
Evershed et al. 1999	-29,8	-31,8	-2	sheep	UK	subcutani
Evershed et al. 1999	-29,5	-31,9	-2,4	sheep	UK	subcutani
Evershed et al. 1999	-29,5	-32,3	-2,8	sheep	UK	subcutani
Craig et al. 2005	-30,5	-33	-2,5	ovin		subcutani
Evershed et al. 1997	-28,3	-31,1	-2,8	cow	UK	subcutani
Craig et al. 2005	-28,8	-30,3	-1,5	bovin		subcutani

Spangenberg et al. 2006	-30,25	-30,6	-0,35	cow	Suisse	subcutani
Spangenberg et al. 2006	-31,65	-31,65	0	cow	Suisse	subcutani
Debono-Spiteri, 2012	-25,9	-25,8	0,1	cow	Malte	subcutani
Debono-Spiteri, 2012	-25,9	-26,1	-0,2	cow	Malte	subcutani
Debono-Spiteri, 2012	-25,3	-27,8	-2,5	sheep	Malte	subcutani
Debono-Spiteri, 2012	-25,9	-27,3	-1,4	sheep	Malte	subcutani
Debono-Spiteri, 2012	-26,3	-27,4	-1,1	goat	Malte	subcutani
Debono-Spiteri, 2012	-24,3	-26,3	-2	goat	Malte	subcutani
Debono-Spiteri, 2012	-22,8	-23,1	-0,3	cow	Malte	subcutani
Debono-Spiteri, 2012	-22	-22,8	-0,8	goat	Malte	subcutani
Debono-Spiteri, 2012	-18,7	-19,3	-0,6	goat	Malte	subcutani

Gregg et al. 2009	-25,4	-25,28	0,12	sheep	Israël	subcutani
Gregg et al. 2009	-31,16	-31,72	-0,56	goat	Palestine	subcutani
Gregg et al. 2009	-31,36	-31,6	-0,24	goat	Palestine	subcutani
Evershed et al. 1997	-25,9	-24,6	1,3	pig	UK	subcutani
Copley et al. 2003	-26,9	-25,2	1,7	non-ruminant	UK	subcutani
Copley et al. 2003	-26,8	-25,2	1,6	non-ruminant	UK	subcutani
Copley et al. 2003	-26,1	-24,8	1,3	non-ruminant	UK	subcutani
Copley et al. 2003	-25,8	-24,5	1,3	non-ruminant	UK	subcutani
Copley et al. 2003	-25,5	-24,15	1,35	non-ruminant	UK	subcutani
Copley et al. 2003	-26,4	-25,4	1	non-ruminant	UK	subcutani
Copley et al. 2003	-25,9	-24,8	1,1	non-ruminant	UK	subcutani

Copley et al. 2003	-25,2	-24,4	0,8	non-ruminant	UK	subcutani
Craig et al. 2005	-26,8	-24,6	2,2	pig		subcutani
Debono-Spiteri	-24,7	-24,5	0,2	pig	Malte	subcutani
Debono-Spiteri	-25	-25	0	pig	Malte	subcutani
Spangenberg et al. 2006	-29	-27,8	1,2	pig	Suisse	subcutani
Spangenberg et al. 2006	-26	-26,9	-0,9	pig	Suisse	subcutani
Colonese et al. 2017	-26,8	-25,8	1	pig	UK	subcutani
Colonese et al. 2017	-28,3	-27,2	1,1	pig	UK	subcutani
Colonese et al. 2017	-27,5	-26,6	0,9	pig	UK	subcutani
Colonese et al. 2017	-27,8	-27,2	0,6	pig	UK	subcutani
Colonese et al. 2017	-28,3	-27,2	1,1	pig	UK	subcutani

Dudd, 1999	-28,8	-31,7	-2,9	wild ruminant	Hébrides	subcutani
Copley et al. 2003 ; Dudd, 2009	-29,1	-30,8	-1,7	wild ruminant	UK	subcutani
Copley et al. 2003 ; Dudd, 2009	-28,8	-30,5	-1,7	wild ruminant	UK	subcutani
Debono-Spiteri, 2012	-27,8	-31,6	-3,8	wild ruminant	Allemagne	wild
Debono-Spiteri, 2012	-25,7	-26,9	-1,2	wild ruminant	Allemagne	wild
Carrer et al. 2016	-29,6	-30,7	-1,2	wild ruminant	Suisse, Grisons	wild
Carrer et al. 2016	-28,4	-29,2	-0,8	wild ruminant	Suisse, Grisons	wild
Carrer et al. 2016	-27,8	-30,2	-2,4	wild ruminant	Suisse, Grisons	wild
Carrer et al. 2016	-28,1	-31	-2,8	wild ruminant	Suisse, Grisons	wild
Carrer et al. 2016	-29,7	-34	-4,2	wild ruminant	Suisse, Grisons	wild
Spangenberg et al. 2006	-31,28	-33,45	-4,2	wild ruminant		wild

Lucquin et al. 2016b	-32,5	-34,1	-1,6	wild ruminant	UK	wild
Lucquin et al. 2016b	-32,4	-34,5	-2,1	wild ruminant	UK	wild
Dudd, 1999	-28,7	-30,1	-1,4	wild ruminant	UK	wild
Dudd, 1999	-29,2	-30,9	-1,7	wild ruminant	UK	wild
Dudd, 1999	-29,8	-30,1	-0,3	wild ruminant	UK	wild
Dudd, 1999	-29,2	-32,5	-3,3	wild ruminant	UK	wild
Dudd, 1999	-28,4	-33,6	-5,2	wild ruminant	UK	wild
Dudd, 1999	-31,1	-34,2	-3,1	wild ruminant	UK	wild
Dudd, 1999	-31,2	-34	-2,8	wild ruminant	UK	wild
Craig et al. 2012	-29,7	-32,2	-4,2	wild ruminant	UK	wild
Craig et al. 2012	-28,13	-31,89	-3,76	wild ruminant	Pologne	wild

Craig et al. 2012	-27,77	-31,54	-3,77	wild ruminant	Pologne	wild
Craig et al. 2012	-28,78	-32,98	-4,2	wild ruminant	Pologne	wild
Craig et al. 2012	-30,41	-34,06	-3,65	wild ruminant	Pologne	wild
Craig et al. 2012	-29,49	-33,08	-3,59	wild ruminant	Pologne	wild
Craig et al. 2012	-29,16	-33,43	-4,27	wild ruminant	Pologne	wild
Craig et al. 2012	-30,75	-33,44	-2,69	wild ruminant	Pologne	wild
Craig et al. 2012	-29,88	-33,47	-3,59	wild ruminant	Pologne	wild
Craig et al. 2012	-29,31	-32,69	-3,38	wild ruminant	Pologne	wild
Craig et al. 2012	-29,82	-33,41	-3,59	wild ruminant	Pologne	wild
Horiuchi et al. 2015	-29	-32,4	-3,4	wild ruminant	Japon	wild
Horiuchi et al. 2015	-24,7	-26,6	-1,9	wild ruminant	Japon	wild

Gregg et al. 2009	-30,19	-28,53	1,66	wild boar	Israël	wild
Gregg et al. 2009	-33,19	-31,48	1,71	wild boar	Israël	wild
Gregg et al. 2009	-30,14	-28,03	2,11	wild boar	Israël	wild
Gregg et al. 2009	-31,56	-27,51	4,05	wild boar	Israël	wild
Debono-Spiteri	-28,9	-28,8	0,1	wild boar	Italie	wild
Debono-Spiteri	-25,9	-25,6	0,3	wild boar	Allemagne	wild
Horiuchi et al. 2015	-28,4	-27,1	1,3	wild boar	Japon	wild
Horiuchi et al. 2015	-26,4	-26,9	-0,5	wild boar	Japon	wild
Horiuchi et al. 2015	-28,1	-27,4	0,7	wild boar	Japon	wild
Lucquin et al. 2016b	-28,1	-28,9	-0,8	wild boar	Japon	wild
Lucquin et al. 2016b	-27,8	-27,9	-0,1	wild boar	Japon	wild

Lucquin et al. 2016b	-27,8	-29,2	-1,4	wild boar	Japon	wild
Lucquin et al. 2016b	-29,5	-29,6	-0,1	wild boar	Japon	wild
Lucquin et al. 2016b	-28,9	-29,5	-0,6	wild boar	Japon	wild
Lucquin et al. 2016b	-28,3	-27,4	0,9	wild boar	Japon	wild
Lucquin et al. 2016b	-28,3	-27,2	1,1	wild boar	Japon	wild
Lucquin et al. 2016b	-28,7	-27,7	1	wild boar	Japon	wild
Lucquin et al. 2016b	-28,6	-27,8	0,8	wild boar	Japon	wild
Lucquin et al. 2016b	-28,3	-27,4	0,9	wild boar	Japon	wild
Craig et al. 2012	-29,2	-34	-4,8		UK	Milk
Craig et al. 2005	-30,15	-36,4	-6,25	cow	UK	Milk
Craig et al. 2005	-28,15	-34,74	-6,59	cow	UK	Milk

Copley et al. 2003	-28,3	-33,55	-5,25		UK	Milk
Copley et al. 2003	-27,8	-32,6	-4,8		UK	Milk
Copley et al. 2003	-29,3	-34,1	-4,8		UK	Milk
Copley et al. 2003	-28,25	-32,4	-4,15		UK	Milk
Copley et al. 2003	-28,5	-34,4	-5,9		UK	Milk
Copley et al. 2003	-30	-35,3	-5,3		UK	Milk
Copley et al. 2003	-29,4	-33,6	-4,2		UK	Milk
Copley et al. 2003	-29,8	-34,2	-4,4		UK	Milk
Copley et al. 2003	-31,2	-34,8	-3,6		UK	Milk
Copley et al. 2003	-29	-34,5	-5,5		UK	Milk
Debono-Spiteri	-25,7	-28,9	-3,2	goat	Malte	Milk

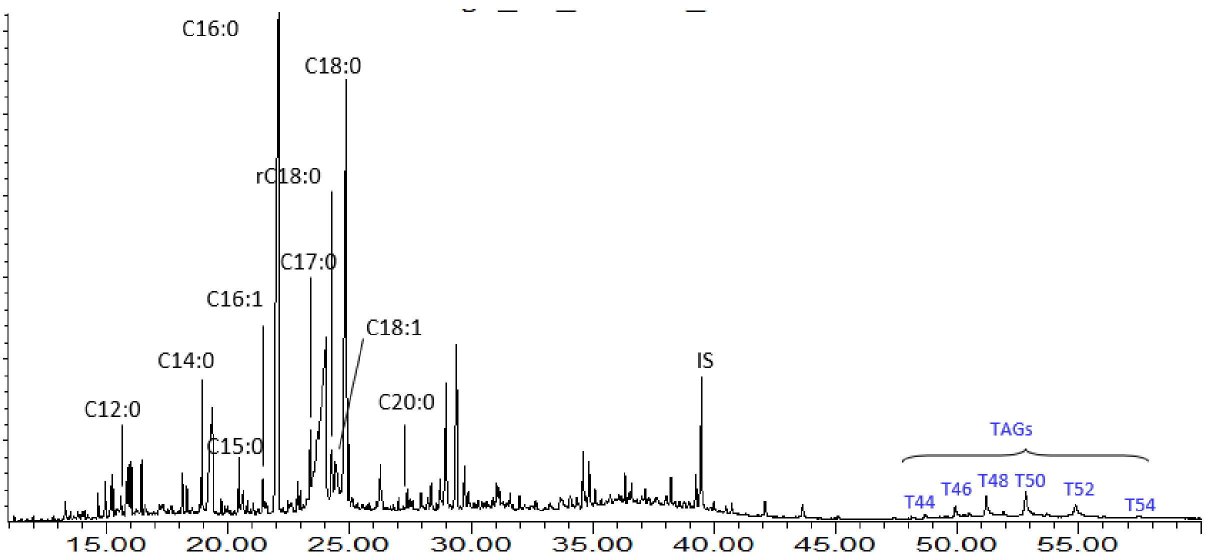
Debono-Spiteri	-24,2	-27,2	-3	goat	Malte	Milk
Debono-Spiteri	-25,2	-29,8	-4,6	goat	Malte	Milk
Debono-Spiteri	-25	-29,3	-4,3	goat	Malte	Milk
Debono-Spiteri	-22,4	-26,7	-4,3	goat	Malte	Milk
Debono-Spiteri	-23,3	-28,4	-5,1		Trani, Italie	Cheese
Debono-Spiteri	-22,1	-27,5	-5,4		Matera, Italie	Cheese
Debono-Spiteri	-20,1	-27,2	-7,1	goat	Malte	Milk
Debono-Spiteri	-24,7	-27,1	-2,4	cow	Malte	Milk
Debono-Spiteri	-24,1	-27,3	-3,2	cow	Malte	Milk
Dudd, 1999	-28,2	-32,4	-4,2	cow	UK	Milk
Dudd, 1999	-31,2	-34,8	-3,6	cow	UK	Milk

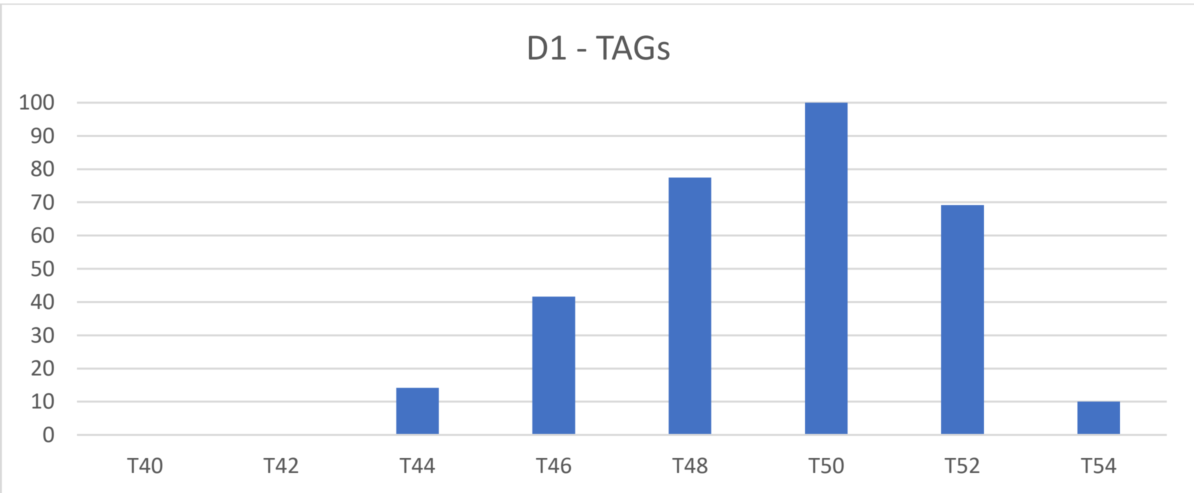
Dudd, 1999	-28,5	-34,4	-5,9	cow	UK	Milk
Dudd, 1999	-27,8	-32,6	-4,8	cow	UK	Milk
Dudd, 1999	-29	-34,5	-5,5	cow	UK	Milk
Dudd, 1999	-30	-35,3	-5,3	cow	UK	Milk
Dudd, 1999	-28,3	-33,5	-5,2	cow	UK	Milk
Dudd, 1999	-29,3	-34,1	-4,8	cow	UK	Milk
Debono-Spiteri	-25,3	-31,3	-6	cow	Allemagne	Milk
Carrer et al. 2016	-28,2	-33,5	-5,2	Fromage de vache	Suisse, Grisons	Cheese
Carrer et al. 2016	-27,6	-32,3	-4,8	fromage de chèvre	Suisse, Grisons	Milk
Carrer et al. 2016	-28,2	-34,1	-5,9		Suisse, Grisons	Milk
Dudd, 1999	-29,4	-33,8	-4,4		UK	Milk

Dudd, 1999	-29,8	-34,2	-4,4		UK	Milk
Spangenberg et al. 2006	-31,7	-37,25	-5,55		Suisse	Milk
Spangenberg et al. 2006	-31,6	-38,25	-6,65		Suisse	Milk
Spangenberg et al. 2006	-31,3	-38,55	-7,25		Suisse	Milk
Spangenberg et al. 2006	-32,28	-39,45	-7,17		Suisse	Milk
Spangenberg et al. 2006	-28,35	-33,7	-5,35		Suisse	Cheese
Spangenberg et al. 2006	-29,39	-34,15	-4,76		Suisse	Cheese
Spangenberg et al. 2006	-31,2	-33,4	-2,2		Suisse	Cheese

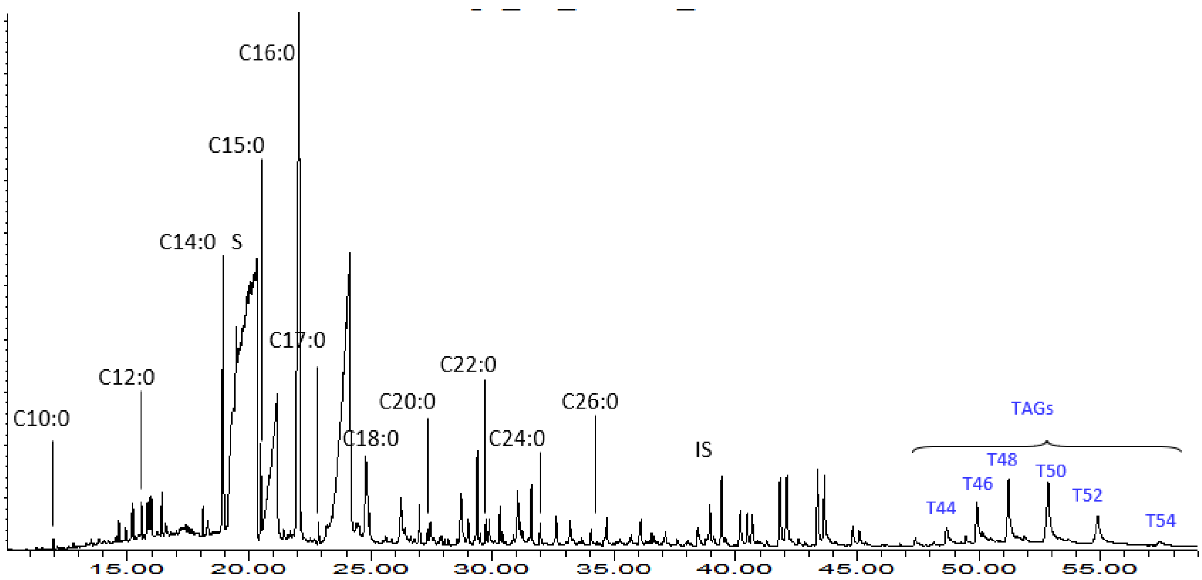
ANNEX III. SAMPLES DATA BASE

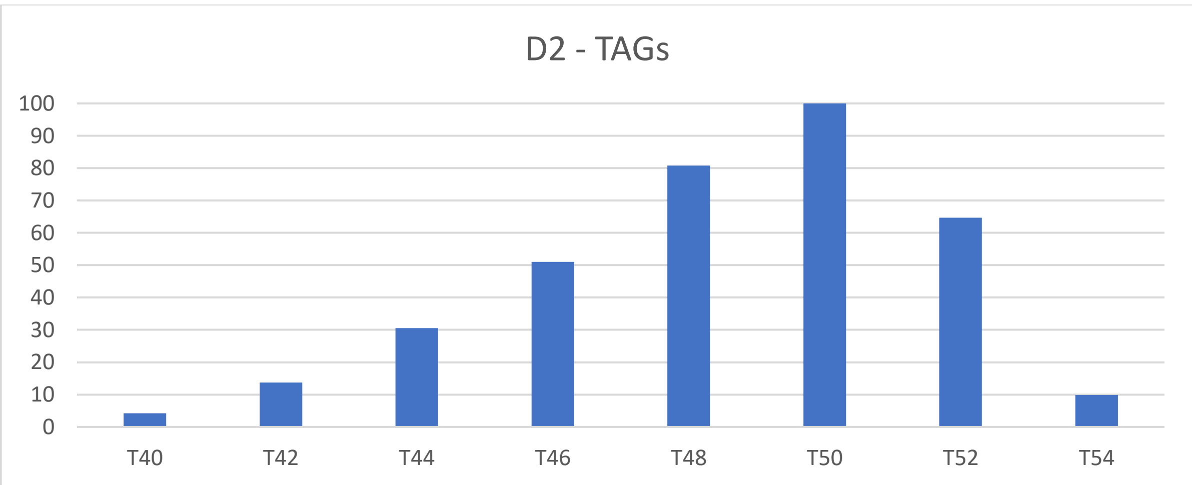
ID Lab	D1		
ID Sample	131	VI a/JA79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	345.4		
Interpretation	Subcutaneous animal fat		



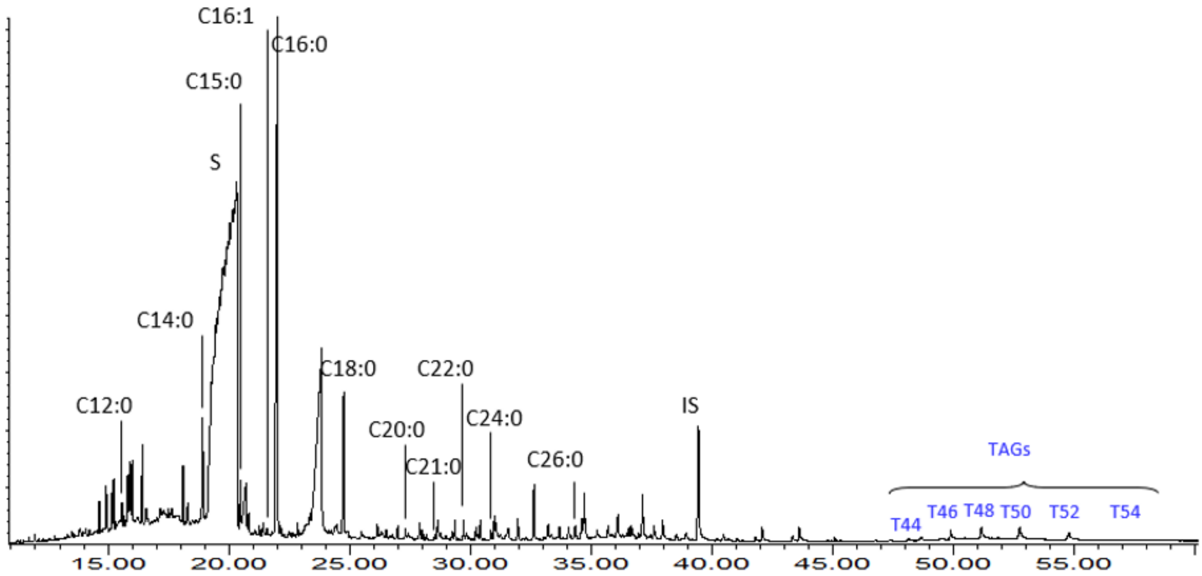


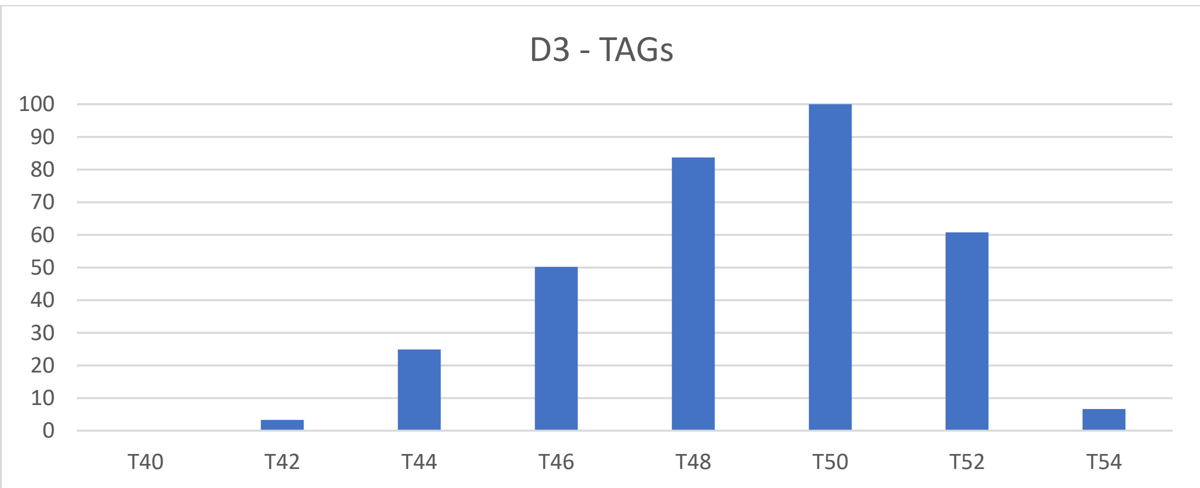
ID Lab	D2	
ID Sample	3525	VII/JD80
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.52	-30.45
TLE (μg·g-1)	402.5	
Interpretation	Dairy products	



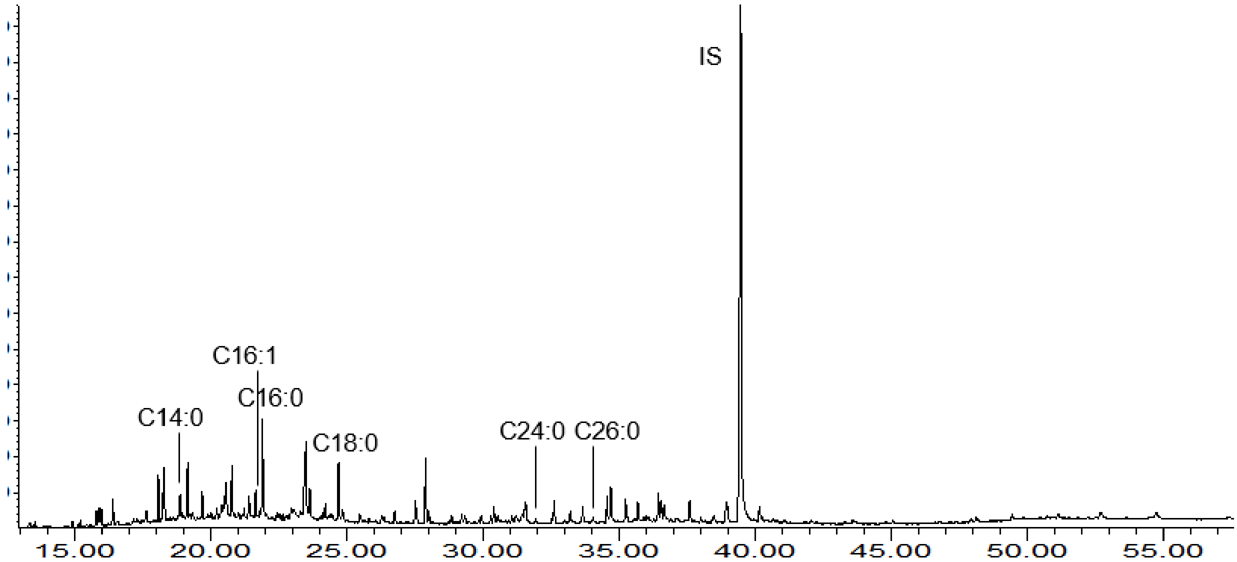


ID Lab	D3	
ID Sample	4480	VII/JC79
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)	344.4	
Interpretation	Dairy products	

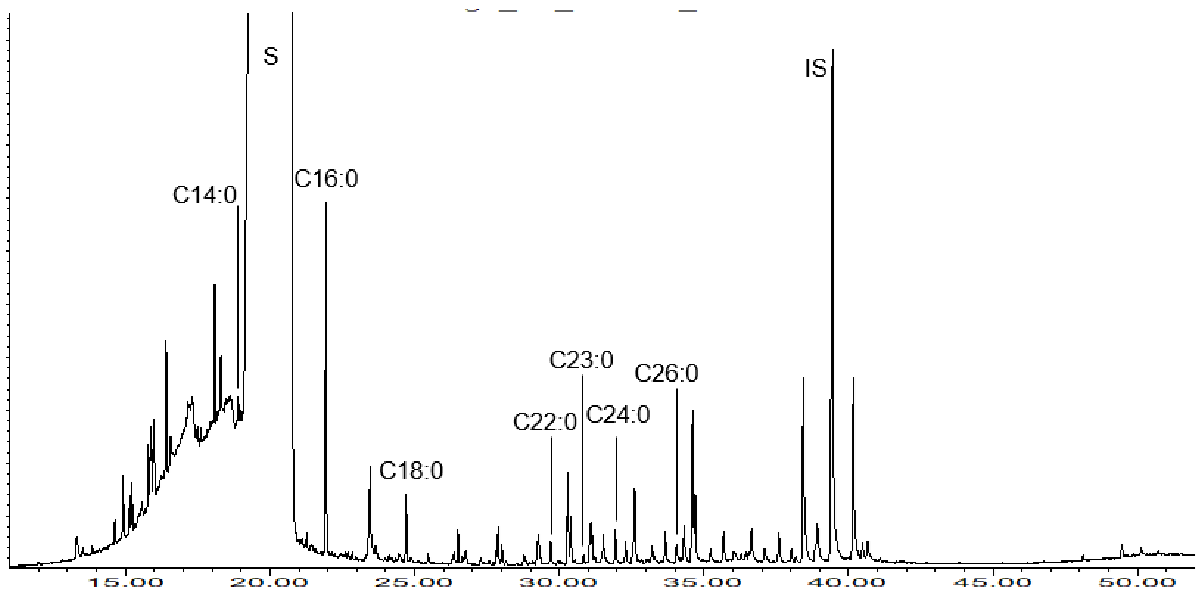




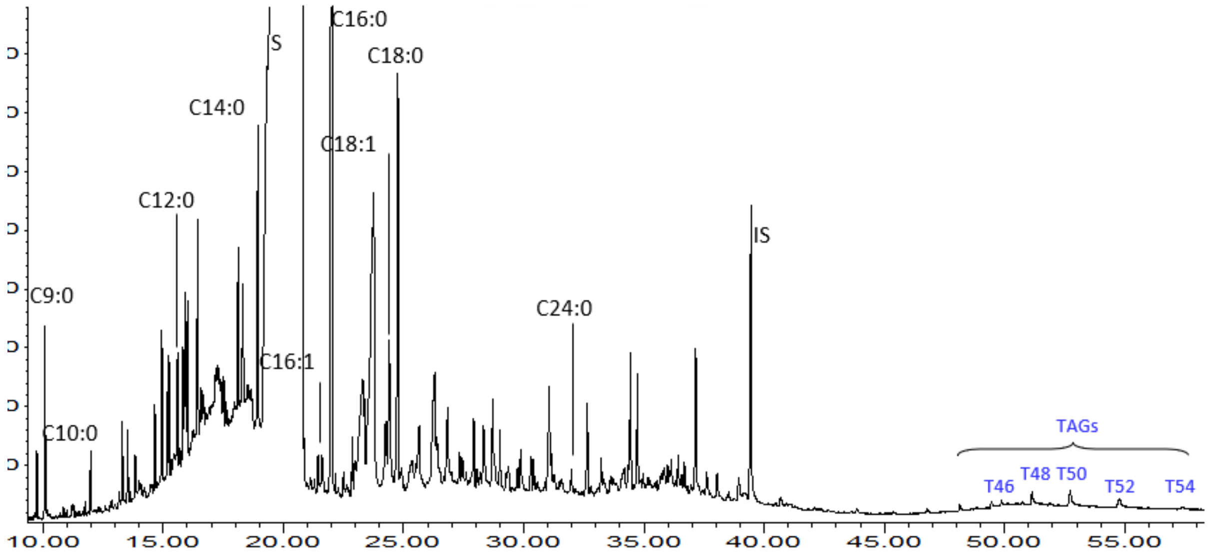
ID Lab	D4		
ID Sample	2933	VIa/IJ81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})	53.4		
Interpretation	Subcutaneous animal fat		

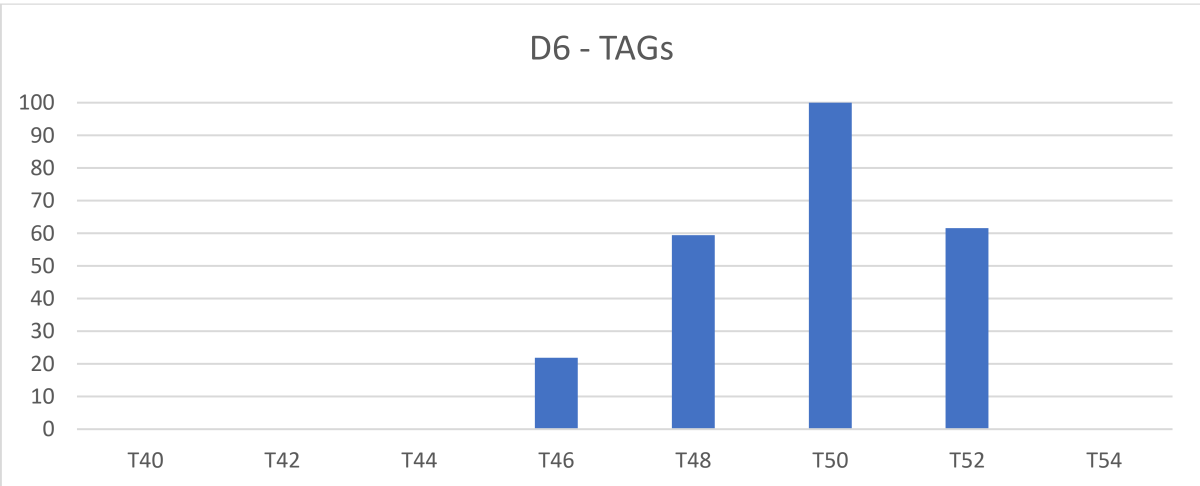


ID Lab	D5		
ID Sample	4111	VII/KA79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	23.4		
Interpretation	Subcutaneous animal fat		

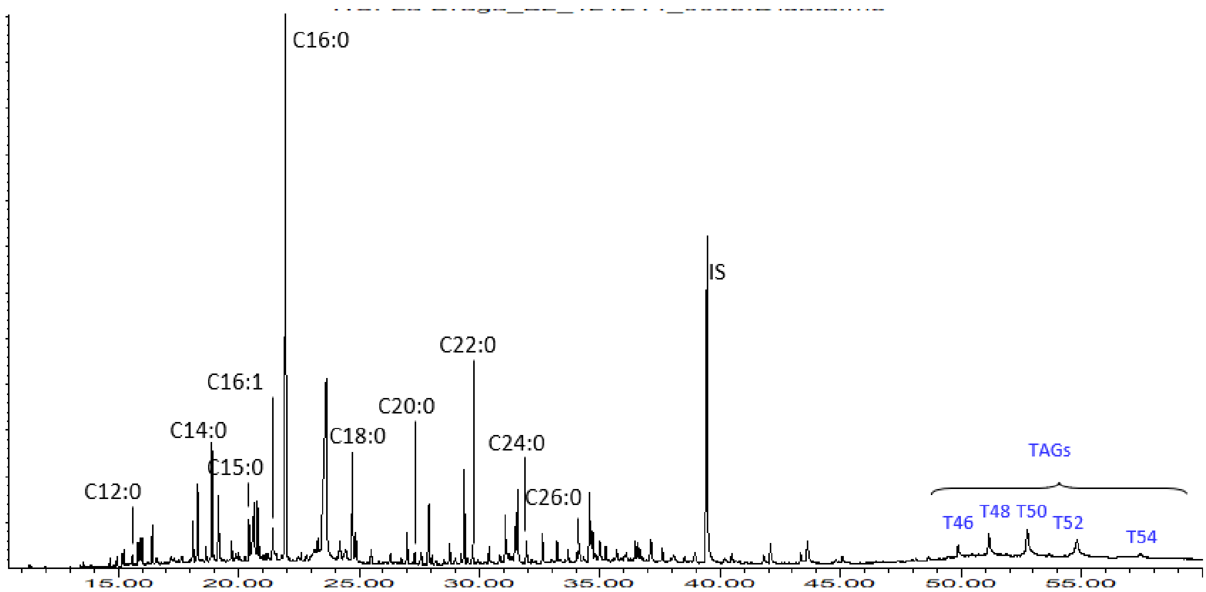


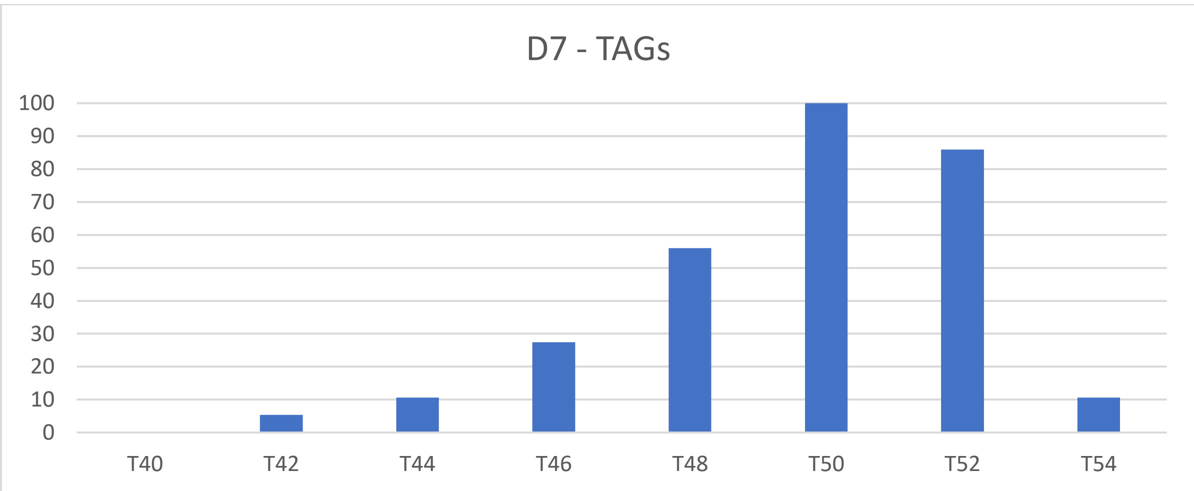
ID Lab	D6	
ID Sample	2595	VII/JH80
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.87	-26.48
TLE (µg·g^{-1})	167.7	
Interpretation	Domestic ruminant fat	



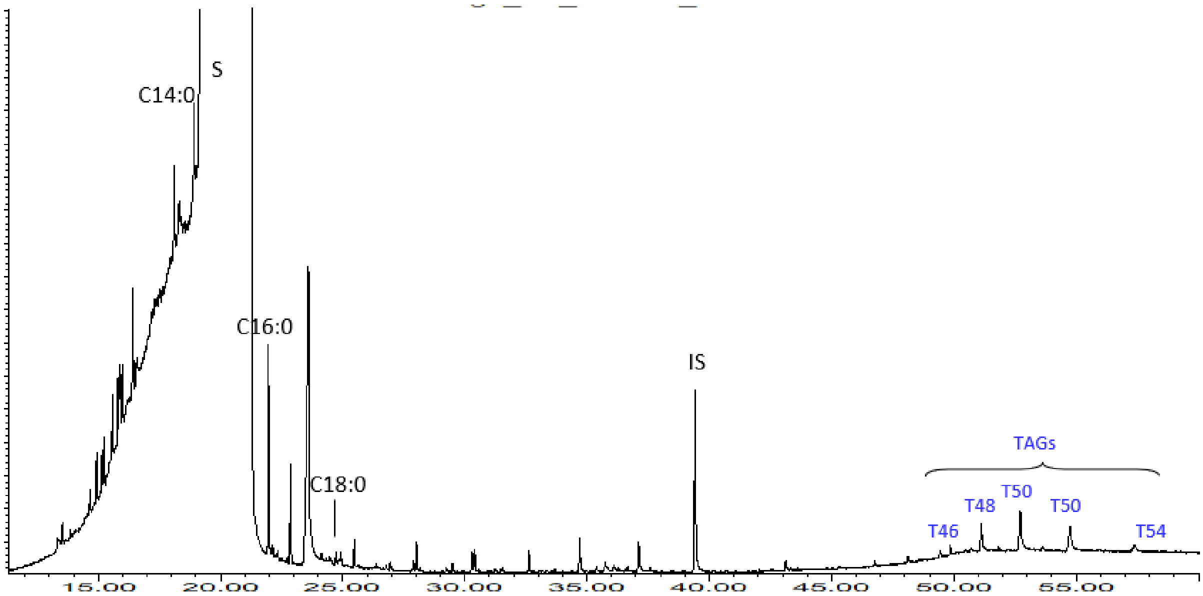


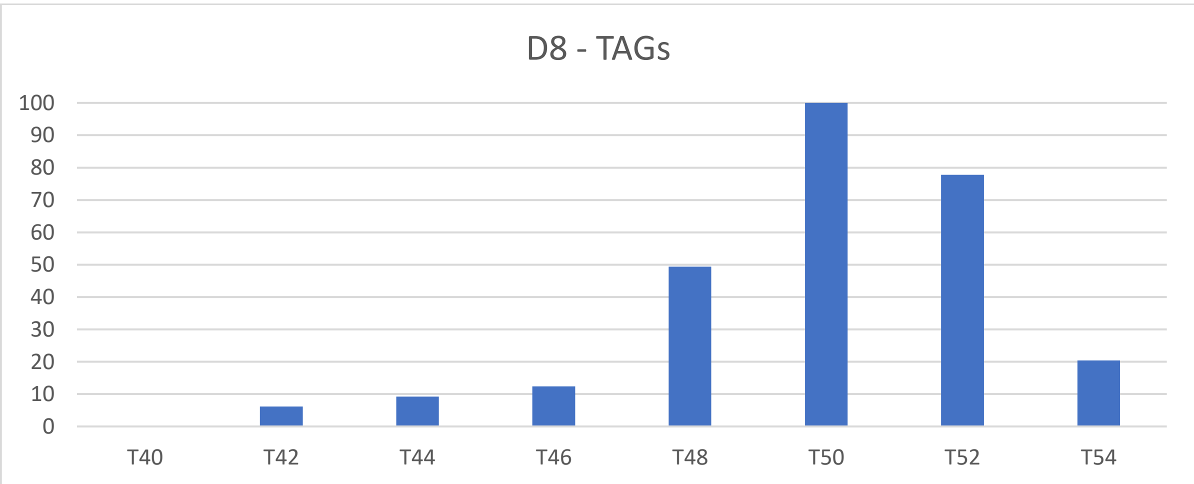
ID Lab	D7		
ID Sample	2994	VIa/JD79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	78.9		
Interpretation	Subcutaneous animal fat		



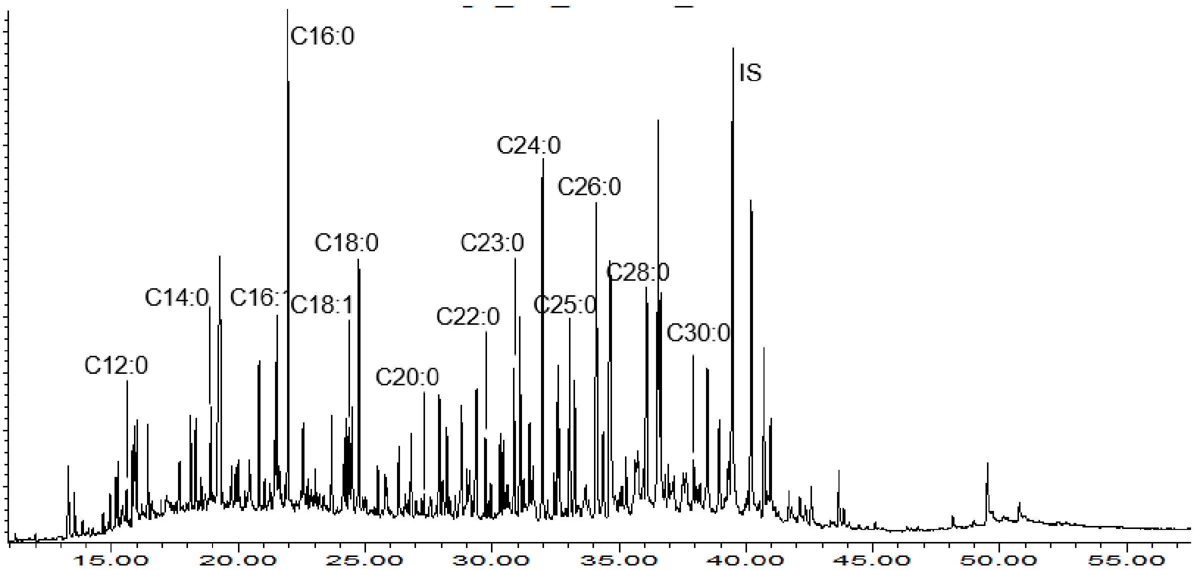


ID Lab	D8	
ID Sample	3677	VI/JD80
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})	58.8	
Interpretation	Subcutaneous animal fat	

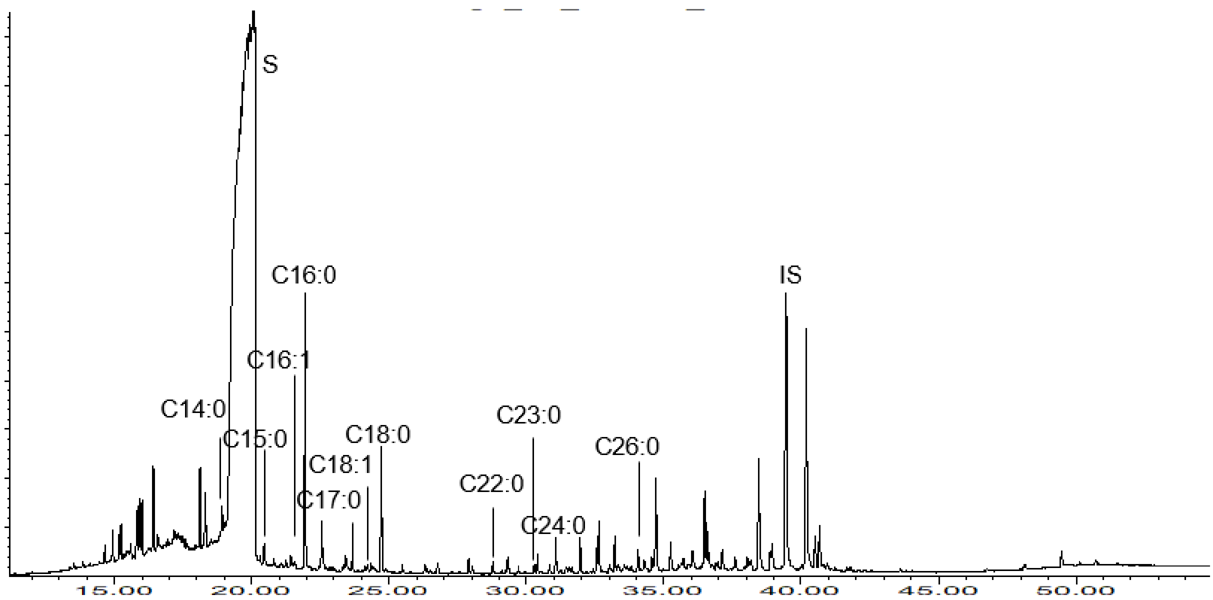




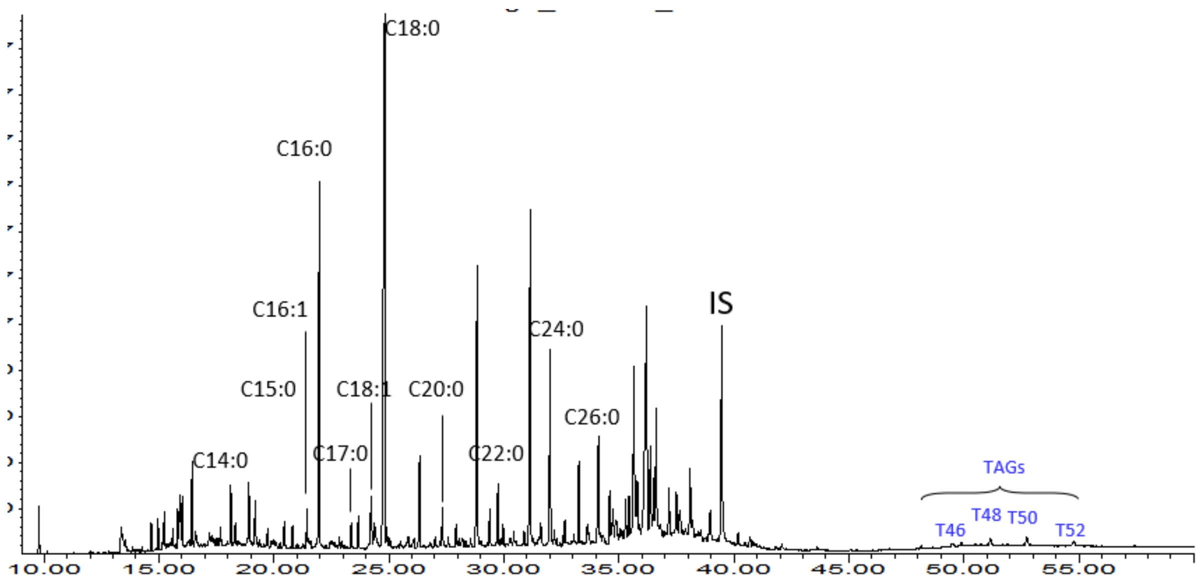
ID Lab	D9		
ID Sample	2845	VIa/JB78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	176.1		
Interpretation	Subcutaneous animal fat		

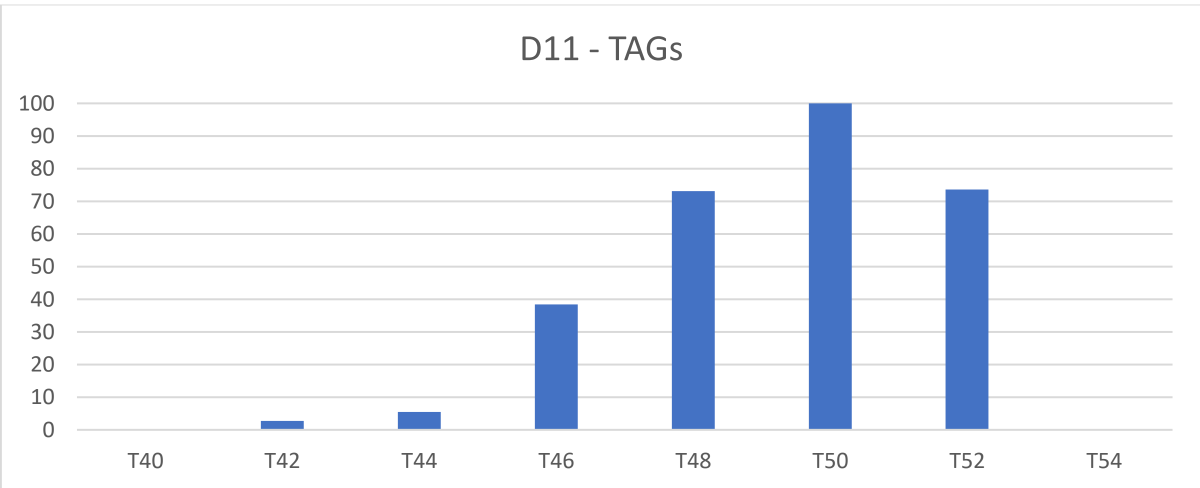


ID Lab	D10		
ID Sample	3378	VIa/JA81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	67.7		
Interpretation	Subcutaneous animal fat		

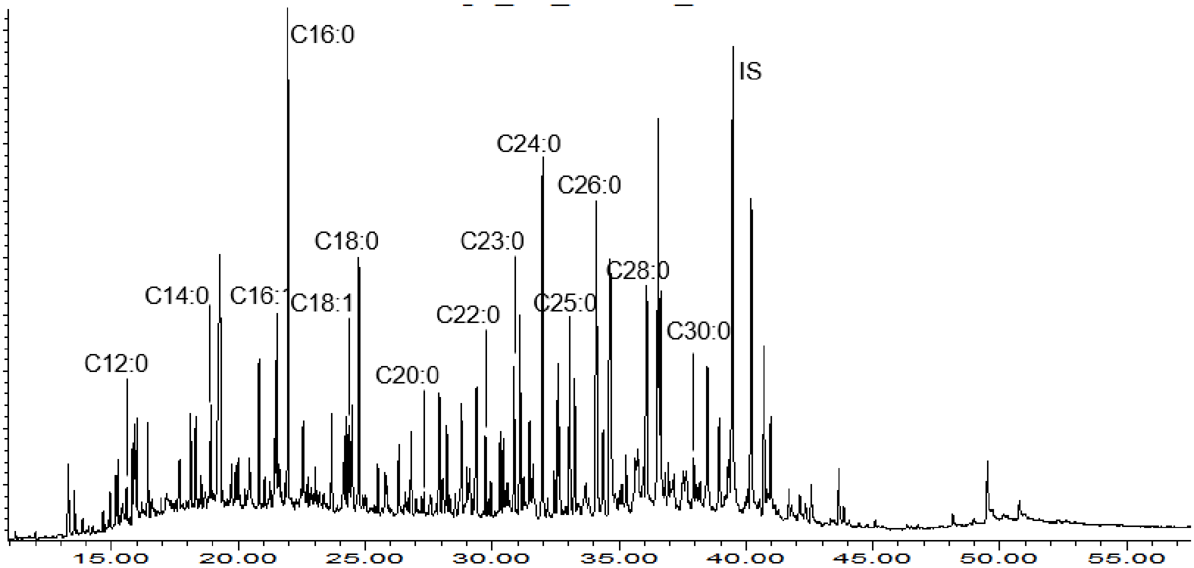


ID Lab	D11		
ID Sample	3929	VII/JB78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	221.5		
Interpretation	Subcutaneous animal fat		

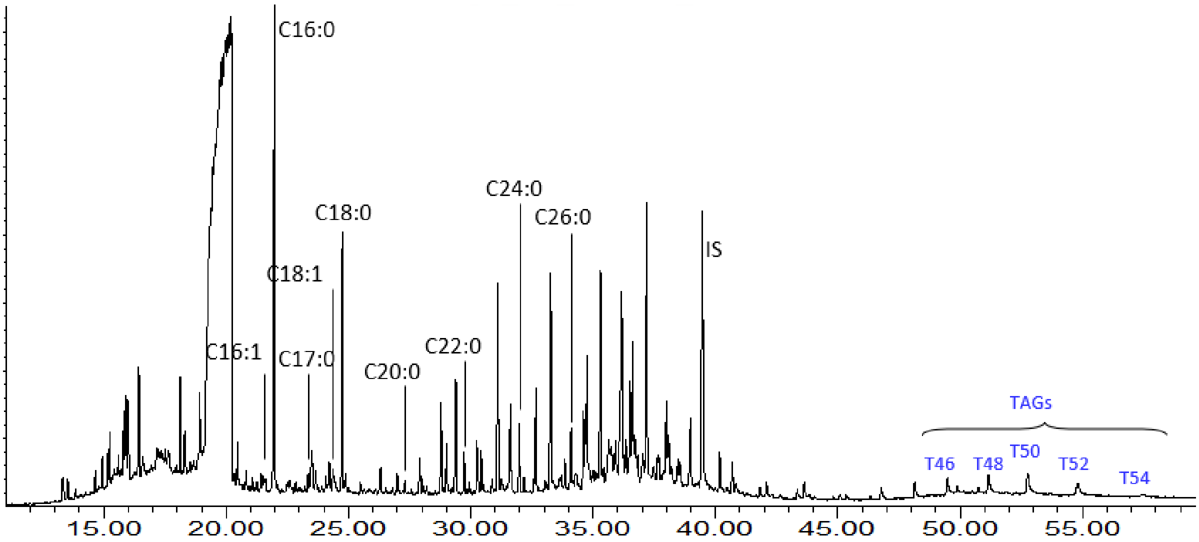


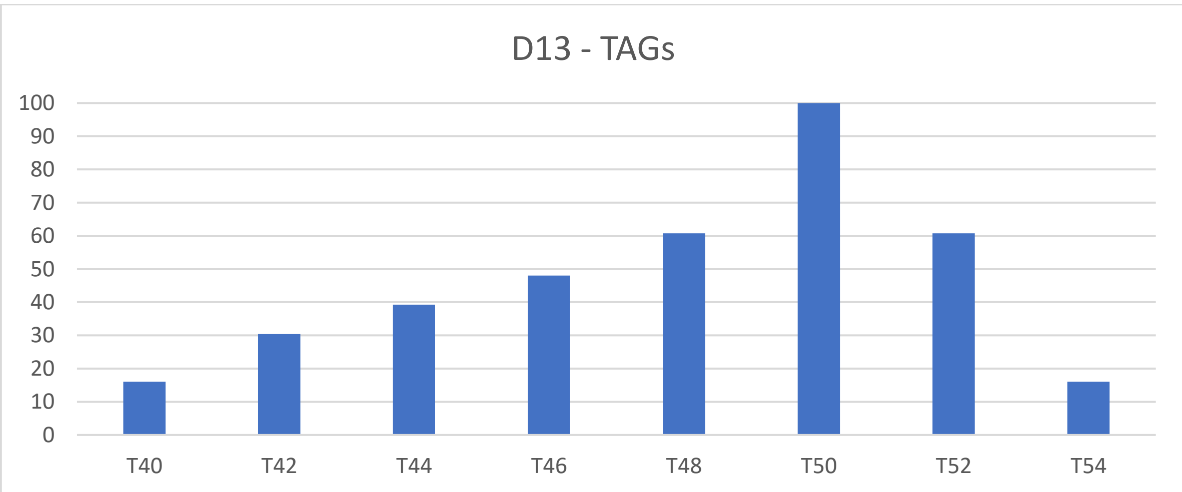


ID Lab	D12		
ID Sample	2845	VIa/JB78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (µg·g^{-1})	220.1		
Interpretation	Subcutaneous animal fat		

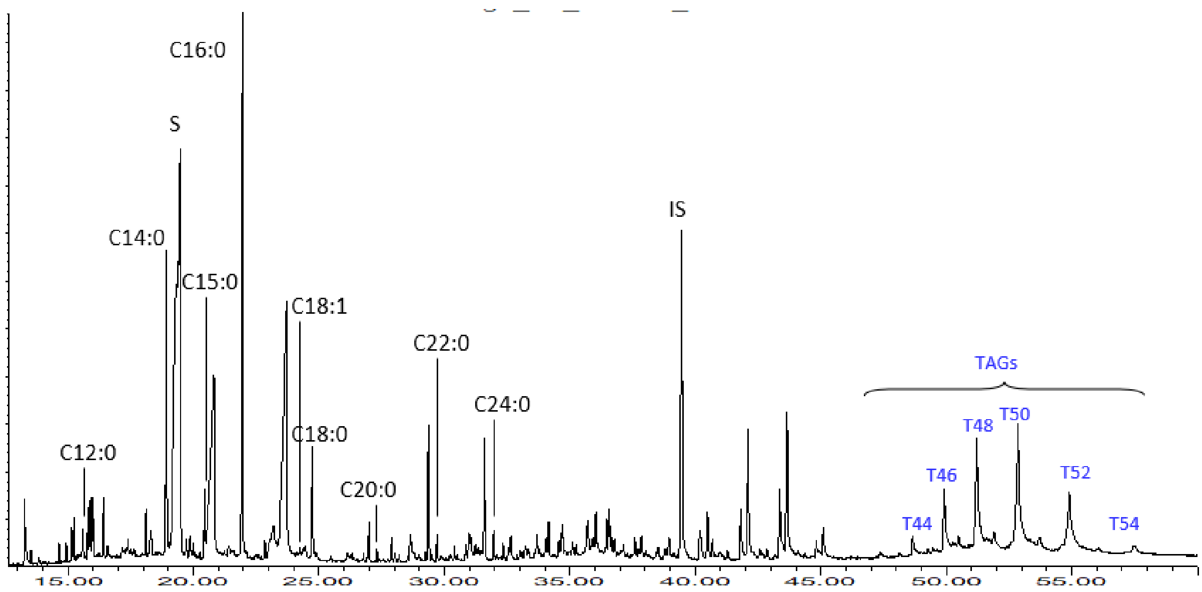


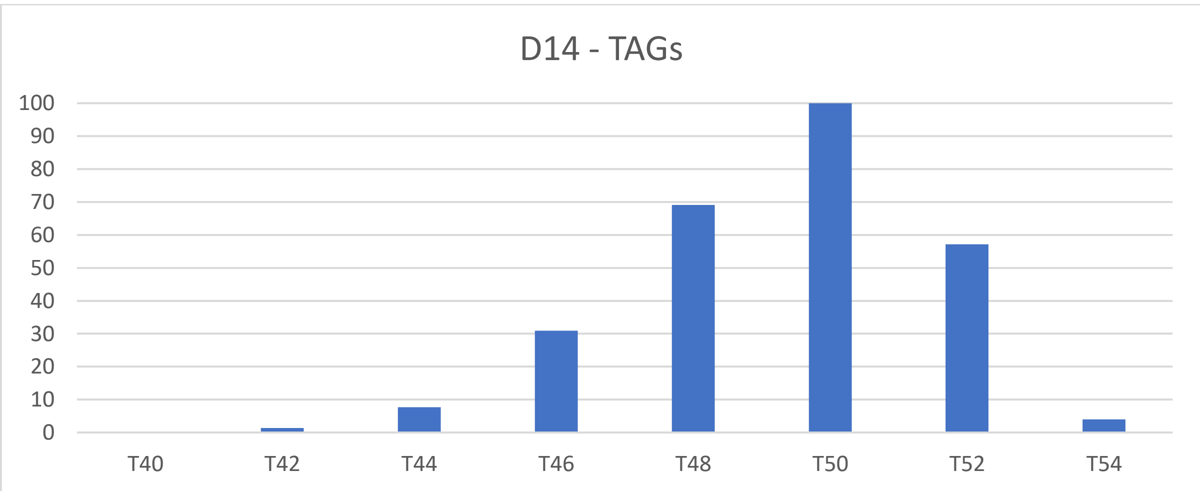
ID Lab	D13		
ID Sample	3503	VII/JC81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (µg·g-1)	67.8		
Interpretation	Dairy products		



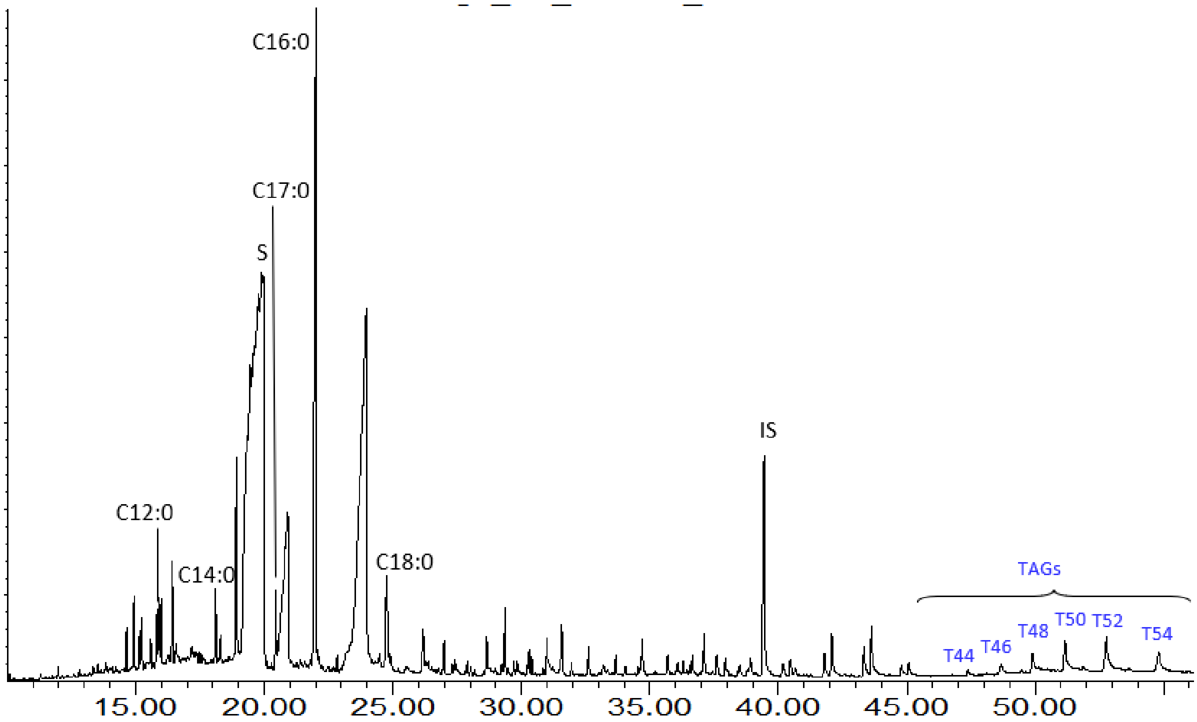


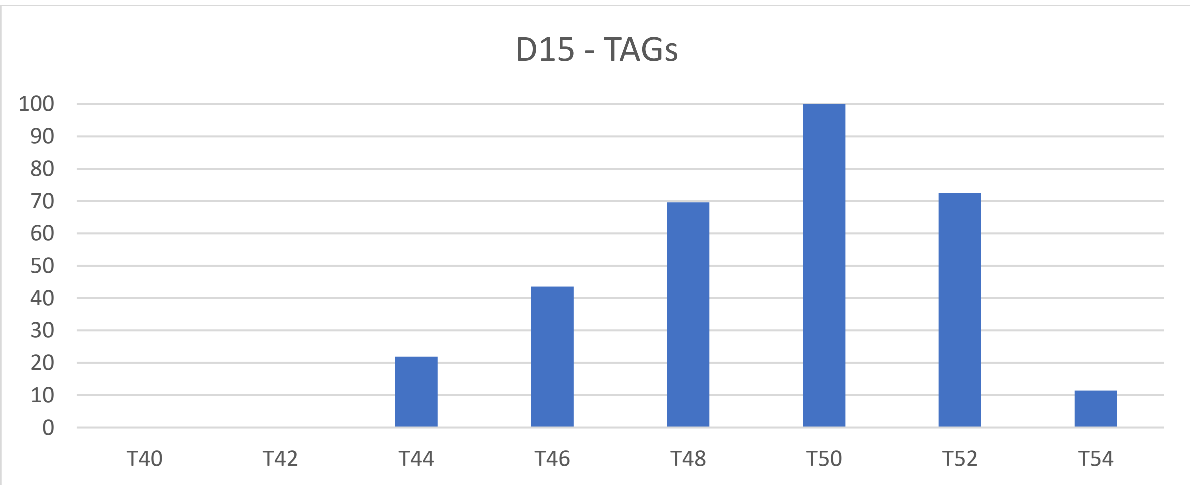
ID Lab	D14	
ID Sample	3990	VIa/JB78
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})	142.2	
Interpretation	Subcutaneous animal fat	



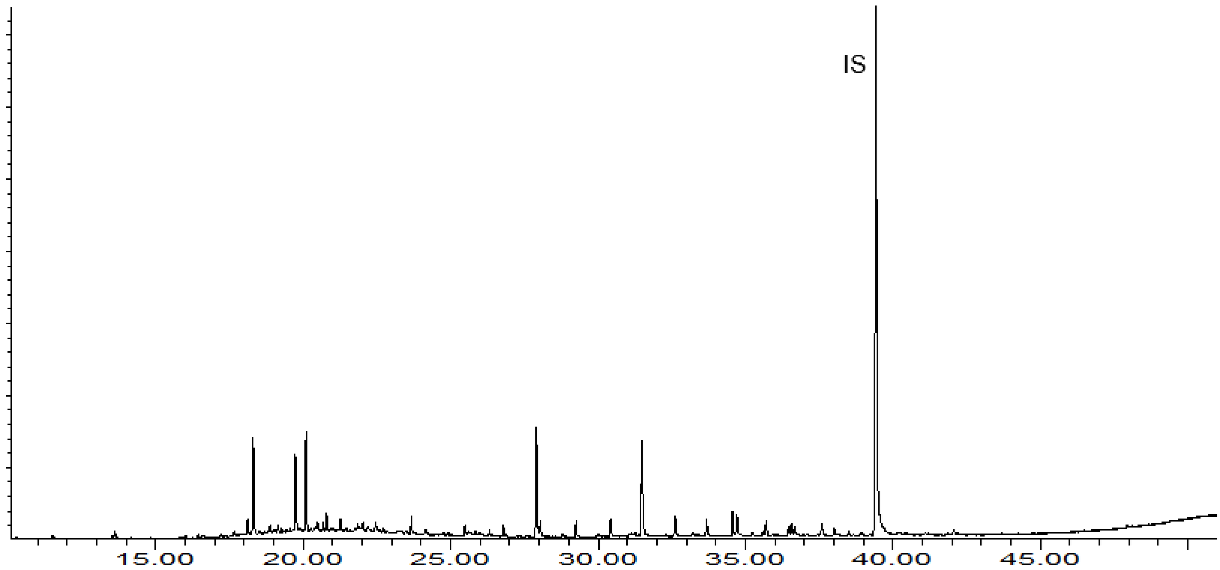


ID Lab	D15	
ID Sample	4447	VII/JD79
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.63	-31.29
TLE (µg·g-1)	220.0	
Interpretation	Dairy products	

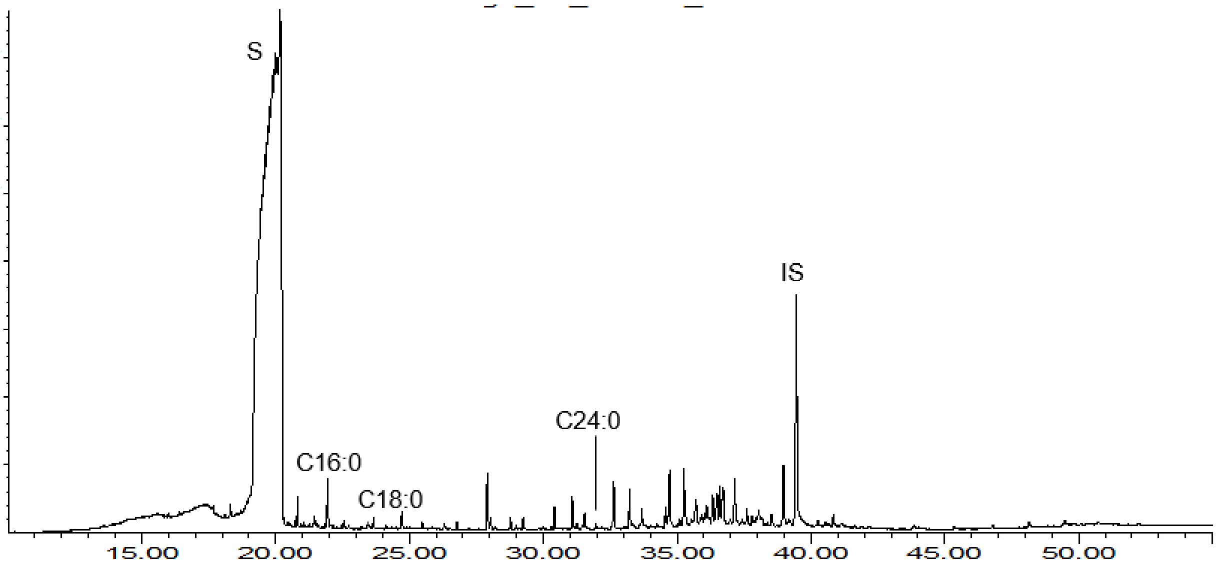




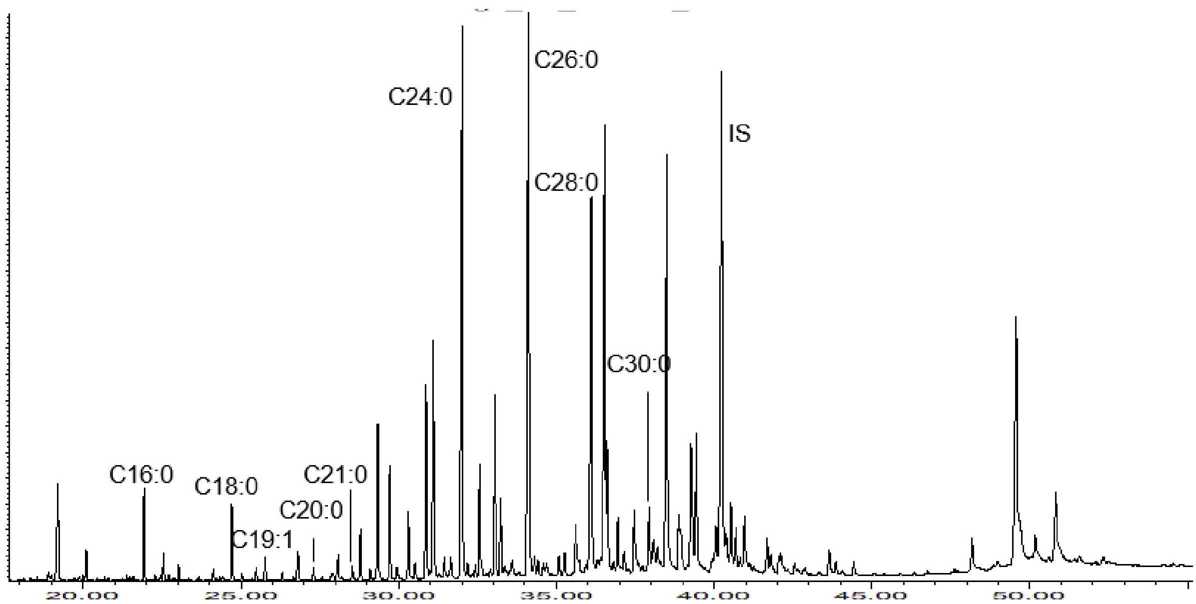
ID Lab	D16		
ID Sample	2394	III/JD81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		



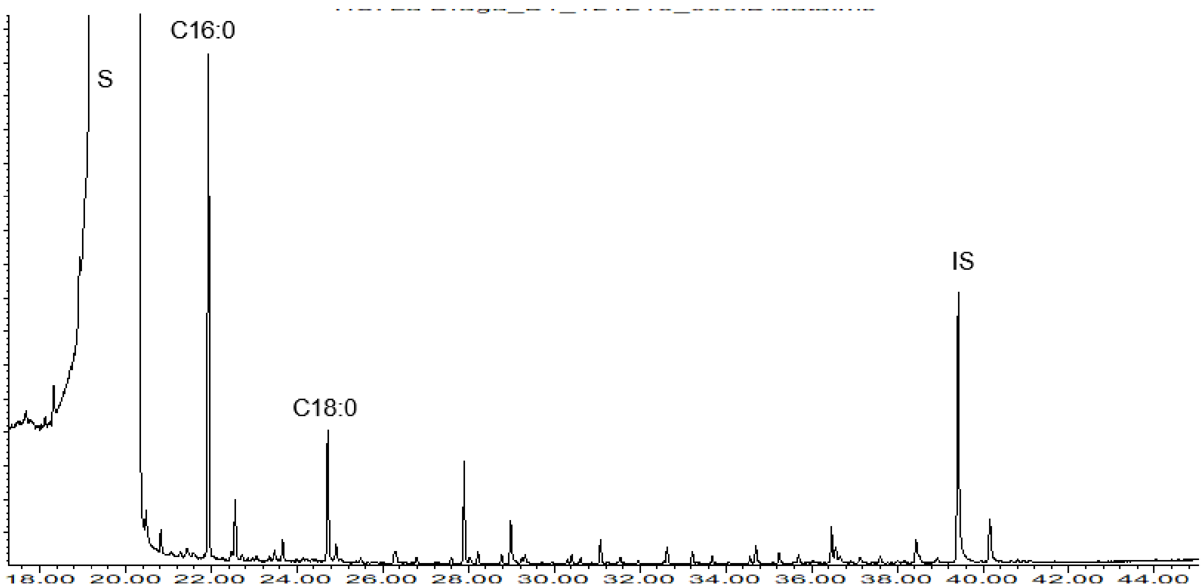
ID Lab	D17		
ID Sample	2200	VIII/JD81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	6.5		
Interpretation	Subcutaneous animal fat		



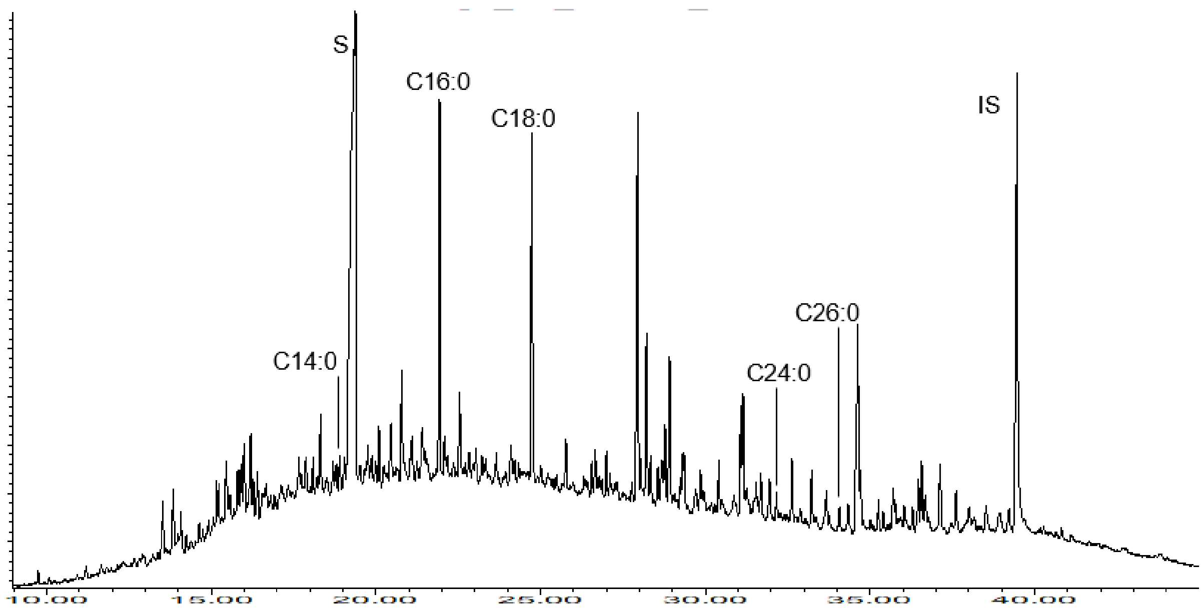
ID Lab	D18		
ID Sample	2469	VII/JG81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	134.5		
Interpretation	Subcutaneous animal fat		



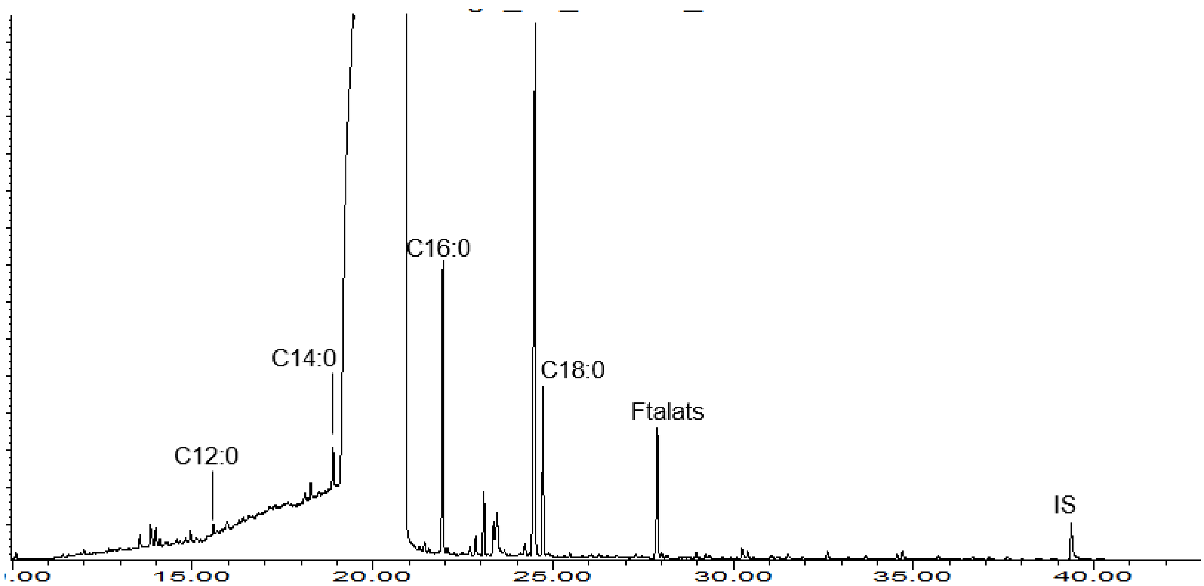
ID Lab	D19		
ID Sample	2192	VII/JE79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	36.2		
Interpretation	Subcutaneous animal fat		



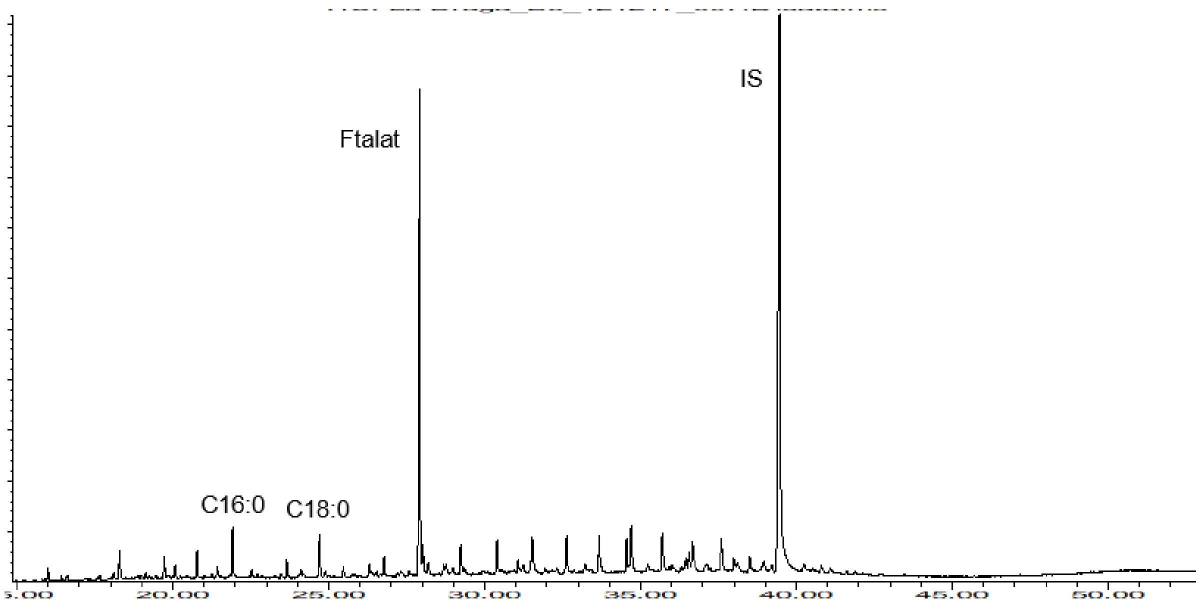
ID Lab	D20		
ID Sample	2102	VIa/IJ79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})	54.3		
Interpretation	Subcutaneous animal fat		



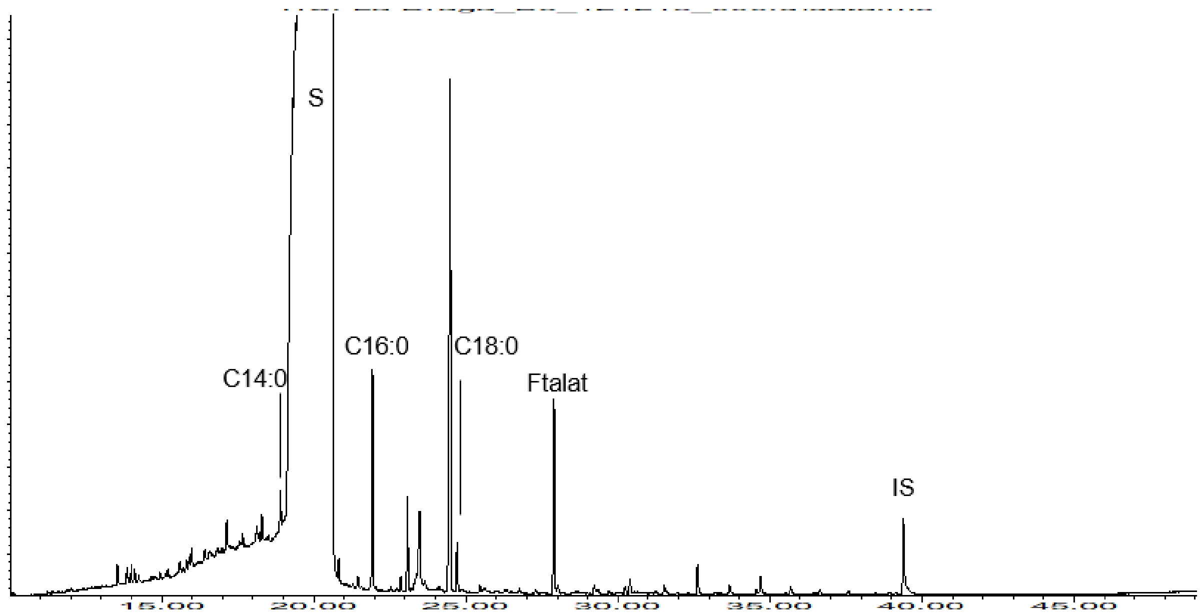
ID Lab	D21		IMAGE NOT AVAILABLE
ID Sample	1698	VIII/JJ78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (µg·g⁻¹)	59.9		
Interpretation	Subcutaneous animal fat		



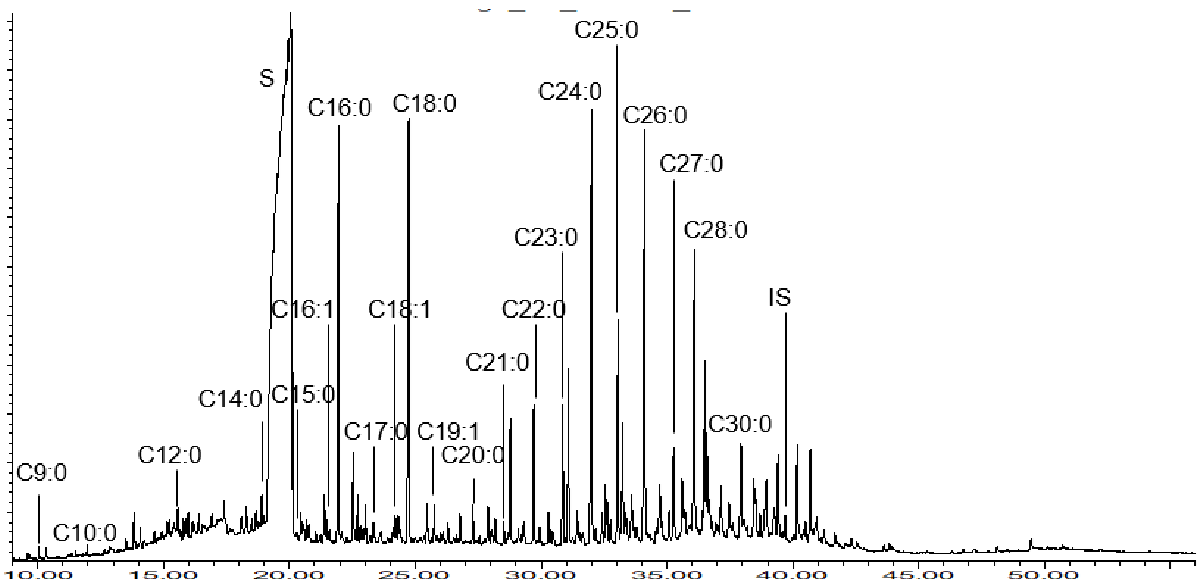
ID Lab	D22		
ID Sample	2208	VII/JD78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (µg·g^{-1})	10.9		
Interpretation	Subcutaneous animal fat		



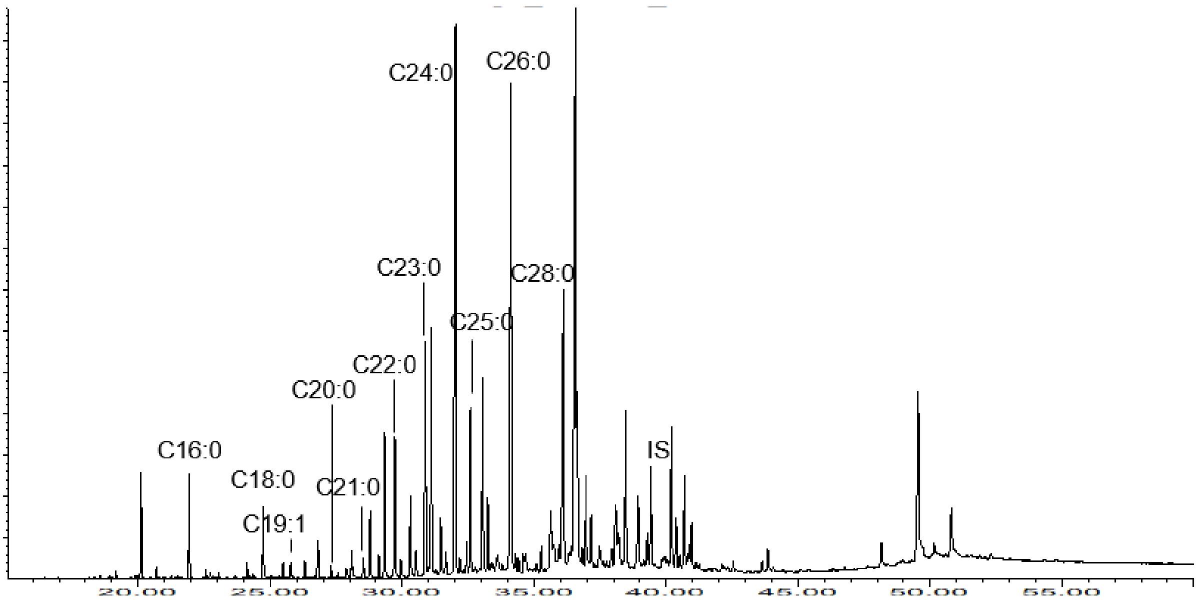
ID Lab	D23		IMAGE NOT AVAILABLE
ID Sample	1699	VII/JJ79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})	64.2		
Interpretation	Subcutaneous animal fat		



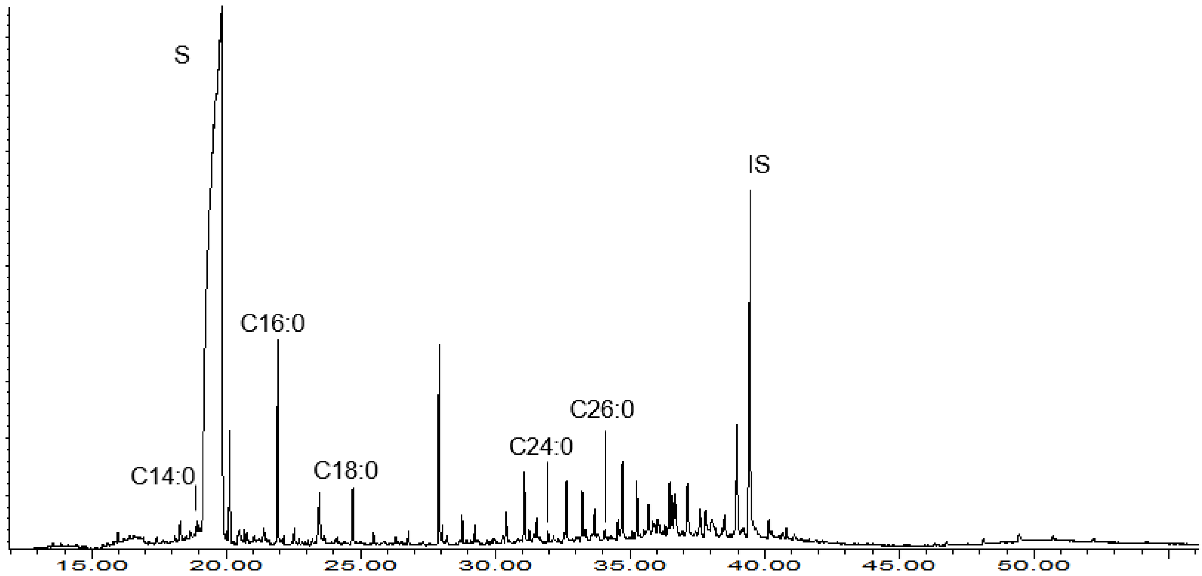
ID Lab	D24		
ID Sample	1963	VII/JE79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	339.9		
Interpretation	Subcutaneous animal fat		



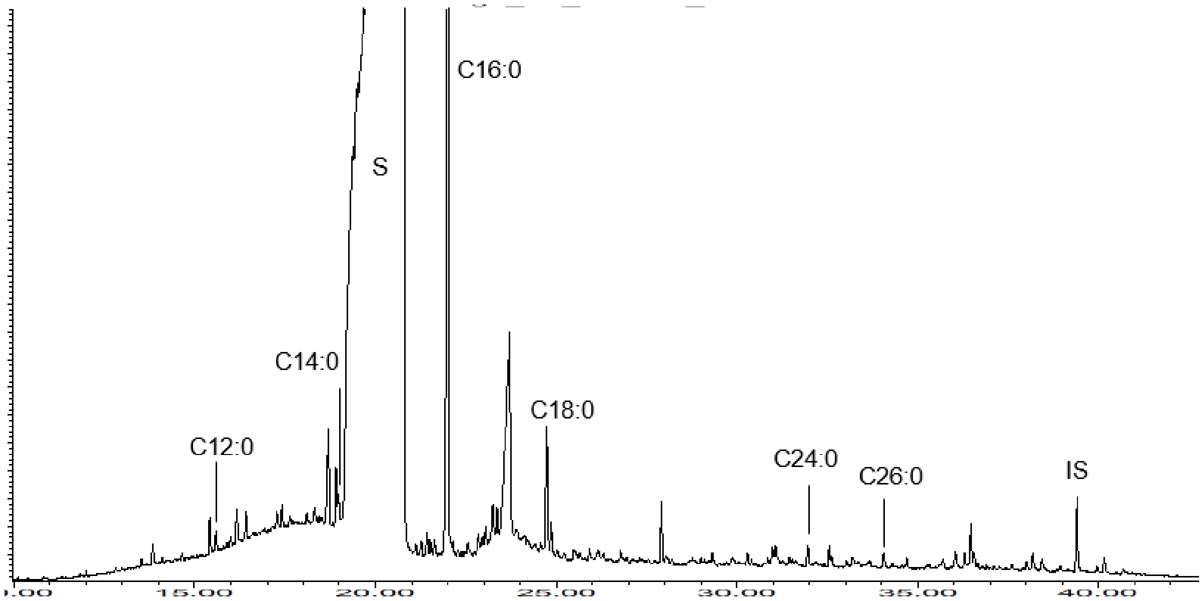
ID Lab	D25		IMAGE NOT AVAILABLE
ID Sample	2472	VII/JG81	
Site	La Draga		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (µg·g^{-1})	329.8		
Interpretation	Subcutaneous animal fat		



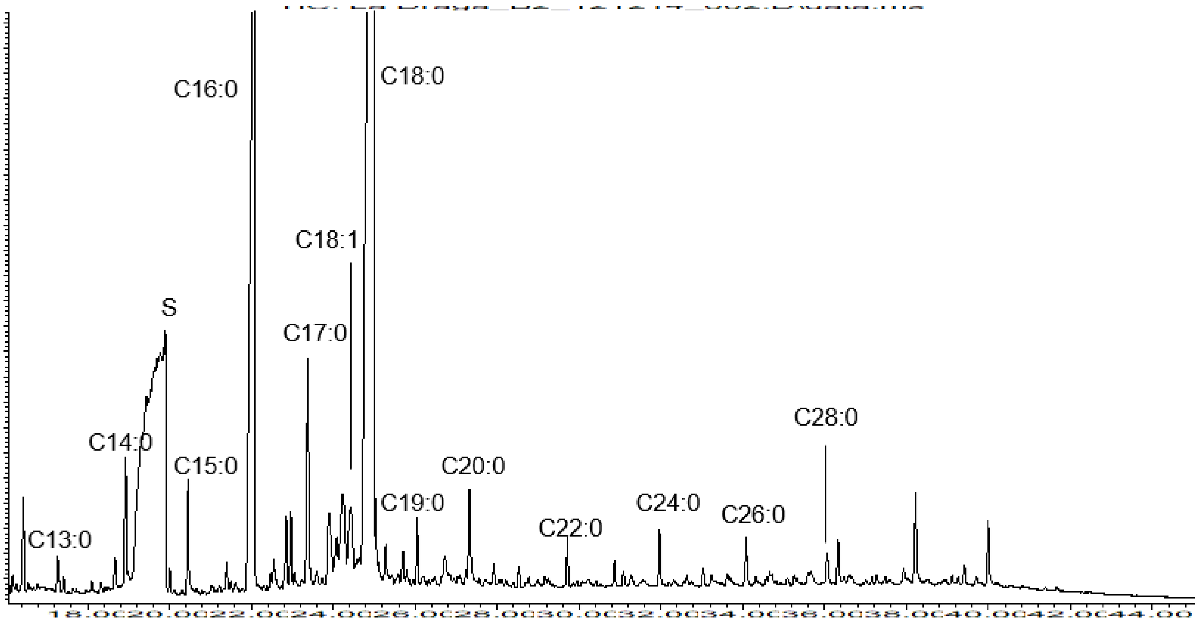
ID Lab	D26		
ID Sample	1955	VII/JE78	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	58.3		
Interpretation	Subcutaneous animal fat		



ID Lab	D27		
ID Sample	2172	VIa/JB79	
Site	La Draga		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)	67.8		
Interpretation	Subcutaneous animal fat		



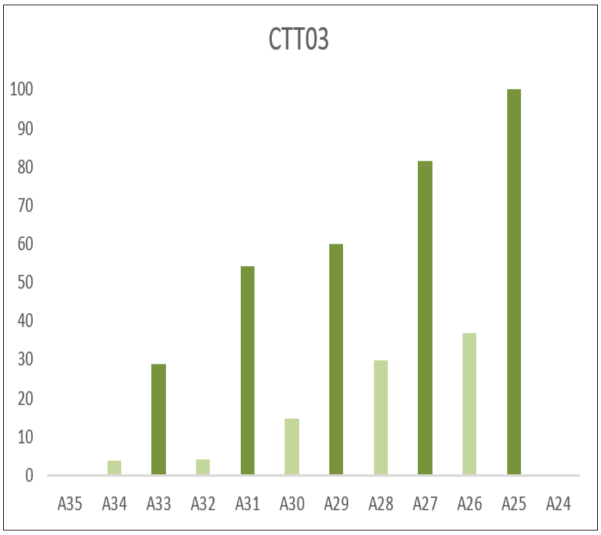
ID Lab	D28	
ID Sample	2107	VII/JE78
Site	La Draga	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)	125.9	
Interpretation	Subcutaneous animal fat	

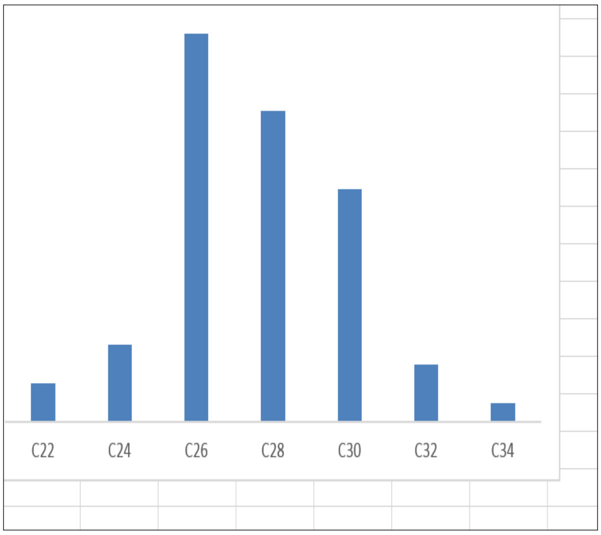


ID Lab	CTT01	
ID Sample	3010	C34
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.34	-30.11
TLE ($\mu g \cdot g^{-1}$)	167.7	
Interpretation	Dairy fat	

ID Lab	CTT02		
ID Sample	3008	C2	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

ID Lab	CTT03	
ID Sample	3008	C1
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
$\delta^{13}C$ values [C16:0 / C18:0]	-27.73	-31.01
TLE ($\mu g \cdot g^{-1}$)	9053.9	
Interpretation	Ruminant adipose fat Epicuticular wax	

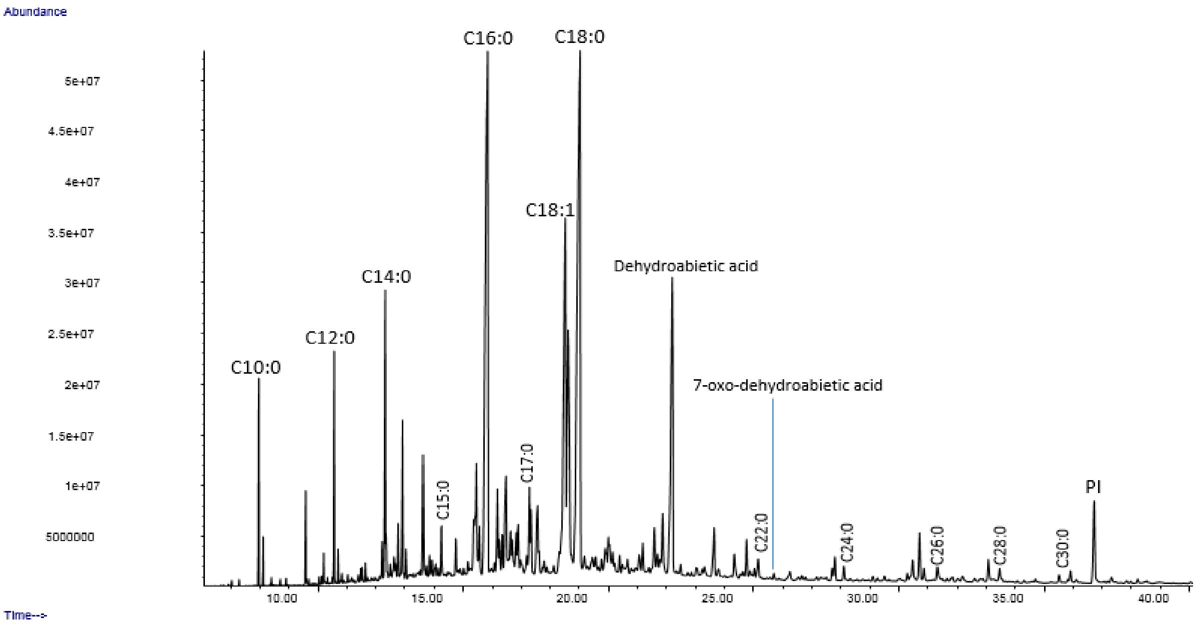




ID Lab	CTT04	
ID Sample	3009	C3-14
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (µg·g^{-1})		
Interpretation	Negligible	

ID Lab	CTT05		
ID Sample	3006	C1	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.83	-26.86	
TLE ($\mu g \cdot g^{-1}$)	90.2		
Interpretation	Ruminant adipose fat		

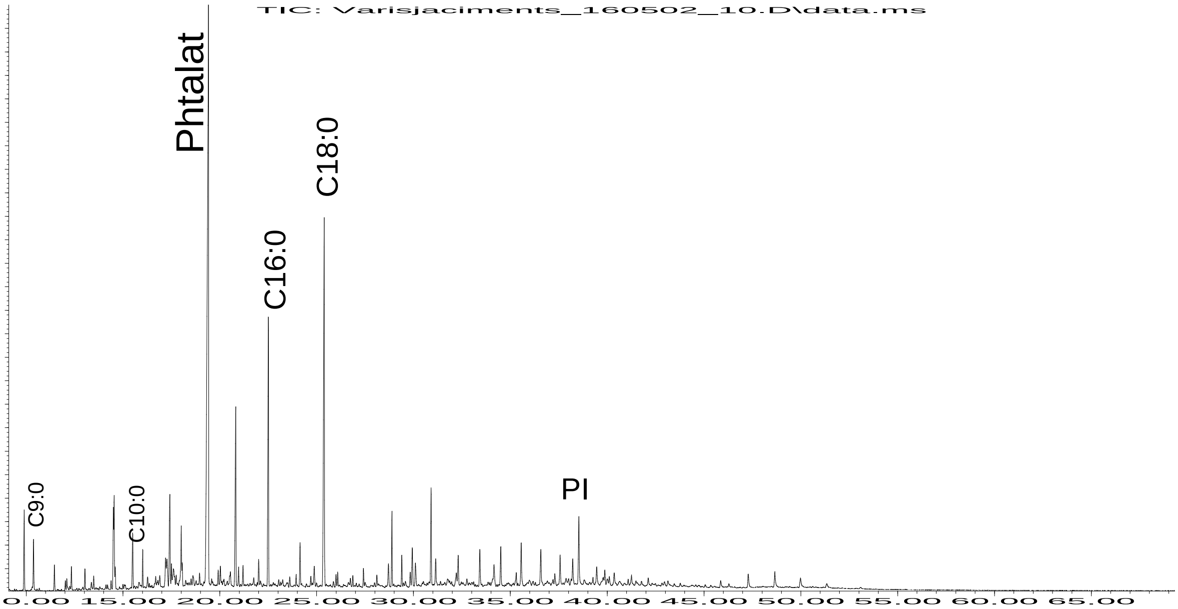
ID Lab	CTT06	
ID Sample	3005	C34
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-21.75	-23.11
TLE (µg·g^{-1})	498.3	
Interpretation	Ruminant adipose fat Pine resin	



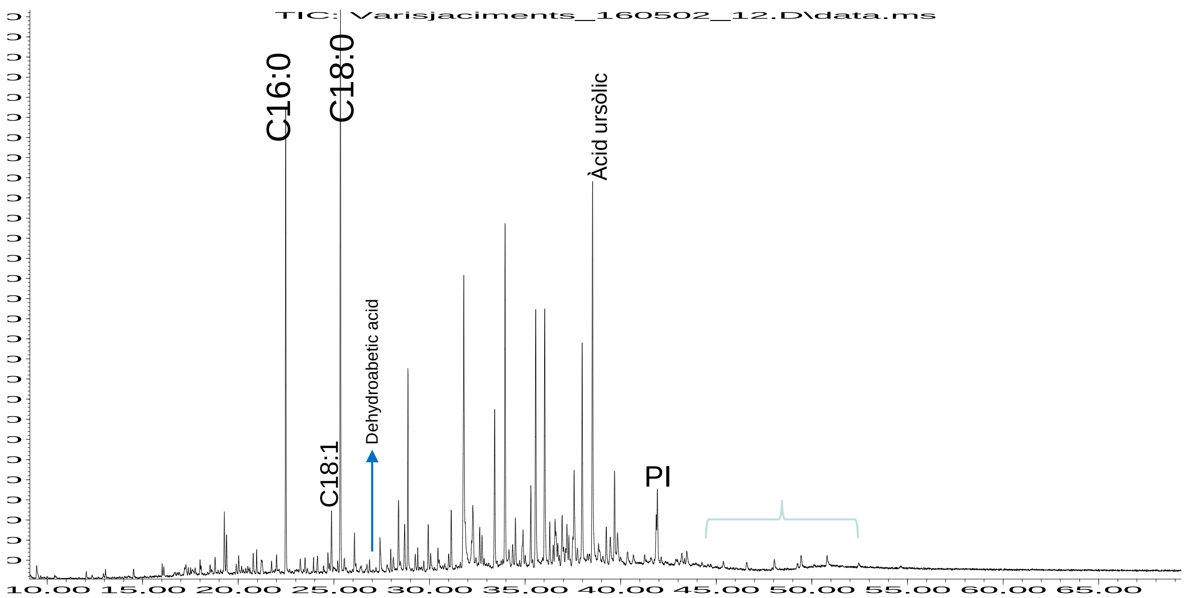
ID Lab	CTT07		
ID Sample	3006b	C18	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.53	-25.6	
TLE ($\mu g \cdot g^{-1}$)	29.4		
Interpretation	Pig adipose fat		

ID Lab	CTT08	
ID Sample	3006b	C15
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT09	
ID Sample	3006b	C13
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.76	-28.91
TLE (µg·g^{-1})	60.02	
Interpretation	Ruminant adipose fat	



ID Lab	CTT10		
ID Sample	3004	C9	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-22.88	-23.95	
TLE ($\mu g \cdot g^{-1}$)	523.3		
Interpretation	Ruminant adipose fat Pine resin		



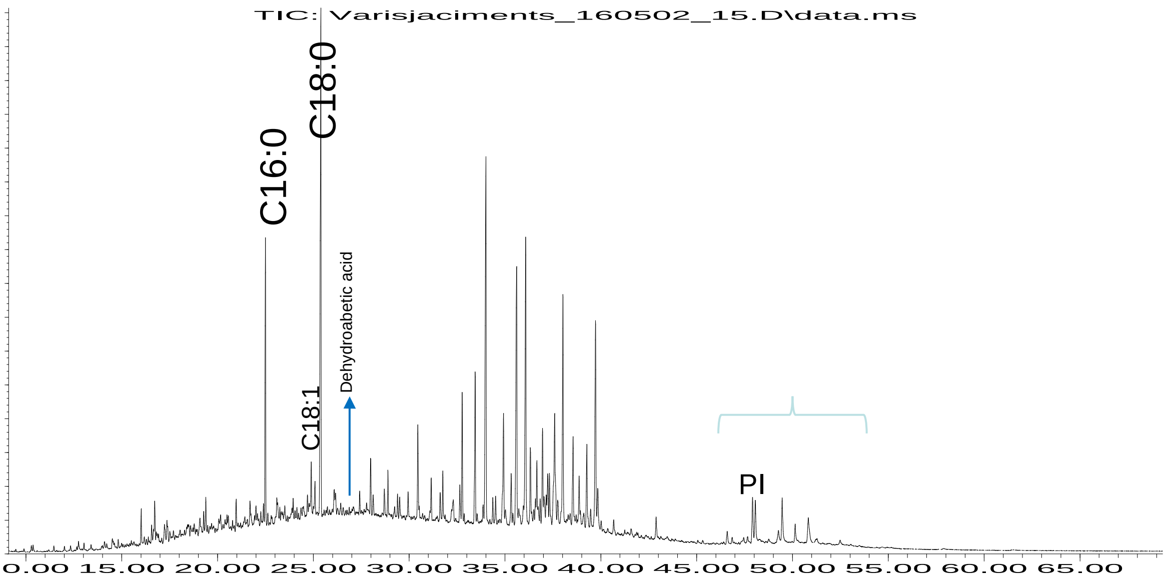
ID Lab	CTT11		
ID Sample	3011	C1	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTT12		
ID Sample	3005	C15	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTT13	
ID Sample	3007	C1
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.01	-28.23
TLE ($\mu g \cdot g^{-1}$)	212.1	
Interpretation	Ruminant adipose fat Pine resin	

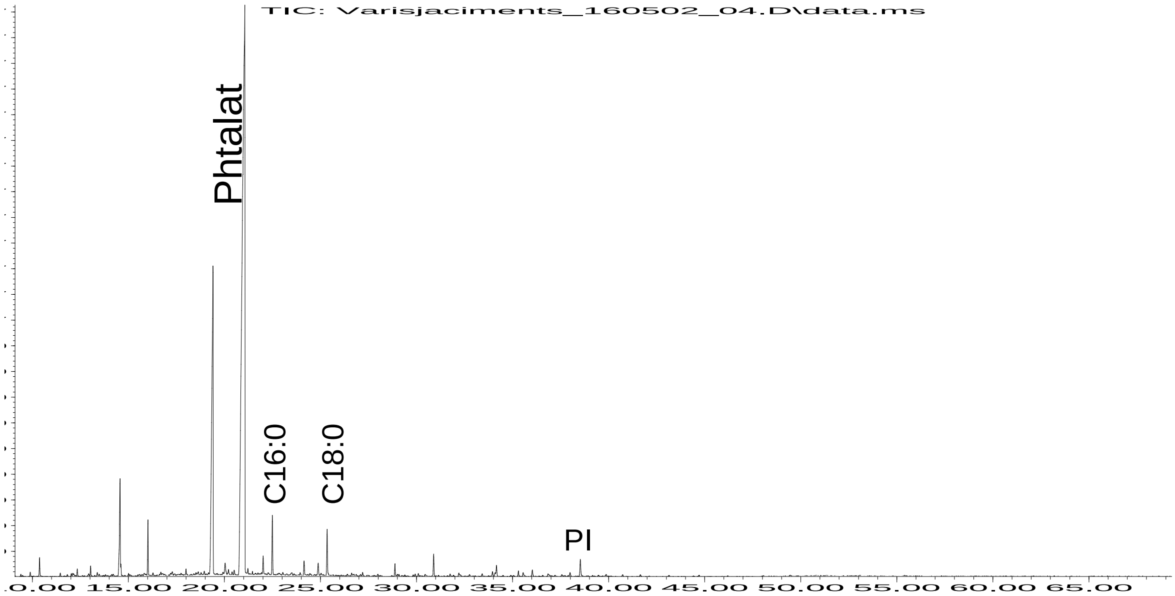
ID Lab	CTT14	
ID Sample	3007	C6
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (µg·g^{-1})		
Interpretation	Negligible	

ID Lab	CTT15	
ID Sample	3010	C6
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-28.92	-30.83
TLE ($\mu g \cdot g^{-1}$)	287.4	
Interpretation	Ruminant adipose fat Pine resin	



ID Lab	CTT16	
ID Sample	3005	C37
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT17	
ID Sample	3006	C2
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.18	-26.98
TLE (µg·g[-1])	23.1	
Interpretation	Wild boar adipose fat	



ID Lab	CTT18	
ID Sample	3010	C13
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT19		
ID Sample	3010	C2	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTT20	
ID Sample	3010	C20
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.93	-32.29
TLE ($\mu g \cdot g^{-1}$)	39.3	
Interpretation	Dairy fat	

ID Lab	CTT21	
ID Sample	2005	266
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT22	
ID Sample	2008	583
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT23		
ID Sample	2006	493	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTT24		
ID Sample	2004	214	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

ID Lab	CTT25	
ID Sample	2007	487
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT26		
ID Sample	2009	617	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

ID Lab	CTT27		
ID Sample	2009	618	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTT28		
ID Sample	2004	213	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

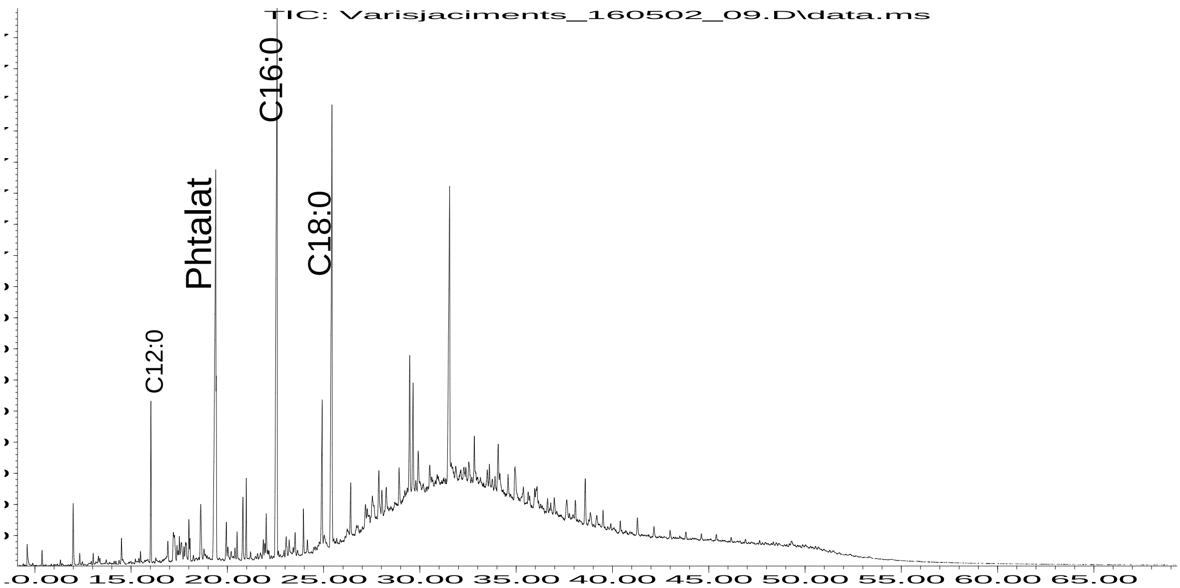
ID Lab	CTT29	
ID Sample	2008	584
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT30	
ID Sample	2005	233
Site	Coro Trasito	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

ID Lab	CTT31		
ID Sample	2005	232	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

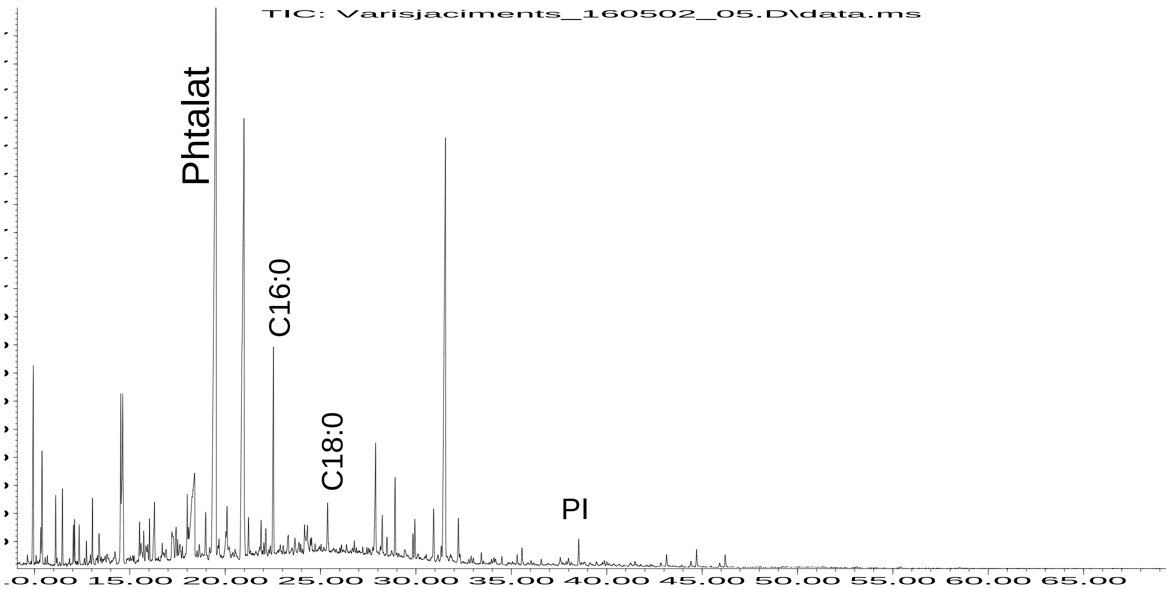
ID Lab	CTT32		
ID Sample	2008	585	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

ID Lab	CTT33		
ID Sample	2B26	TR712	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.03	-25.71	
TLE (µg·g⁻¹)	41.3		
Interpretation	Pig adipose fat		



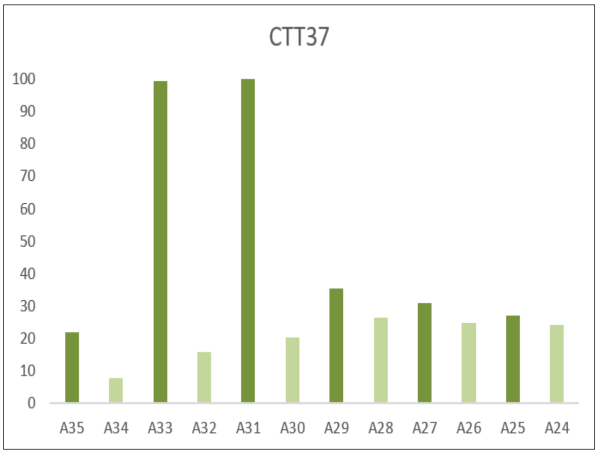
ID Lab	CTT34		
ID Sample	2A9	TR705	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.87	-21.46	
TLE ($\mu g \cdot g^{-1}$)	52.01		
Interpretation	Pig adipose fat		

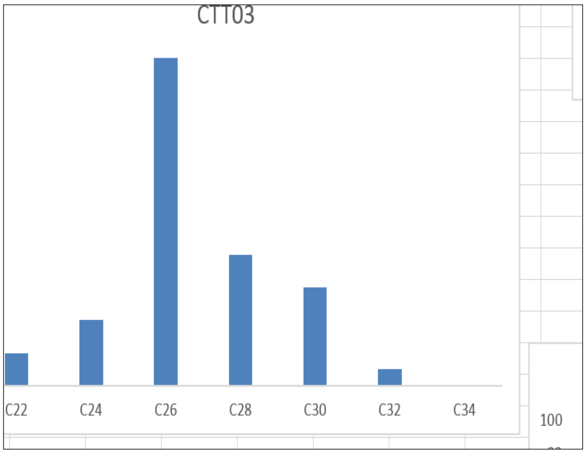
ID Lab	CTT35		
ID Sample		TR292	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.7	-27.48	
TLE (µg·g^{-1})	545.3		
Interpretation	Ruminant adipose fat		



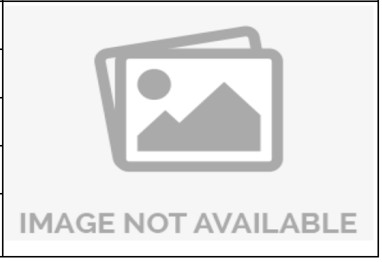
ID Lab	CTT36		
ID Sample	3A6	TR16-095	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.56	-23.33	
TLE (μg·g^{-1})	95.5		
Interpretation	Pig adipose fat		

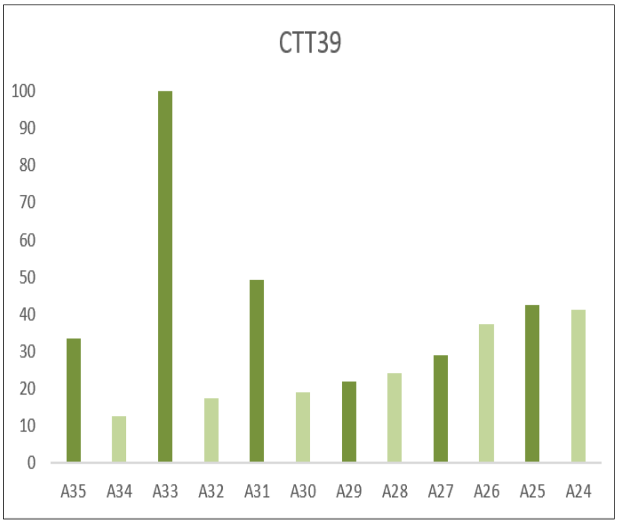
ID Lab	CTT37		
ID Sample		TR16-067	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.81	-28.49	
TLE (μg·g^{-1})	1786.6		
Interpretation	Dairy fat Epicuticular wax		

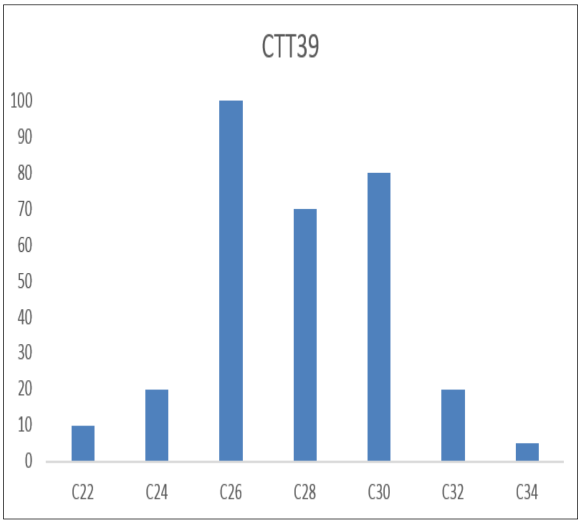




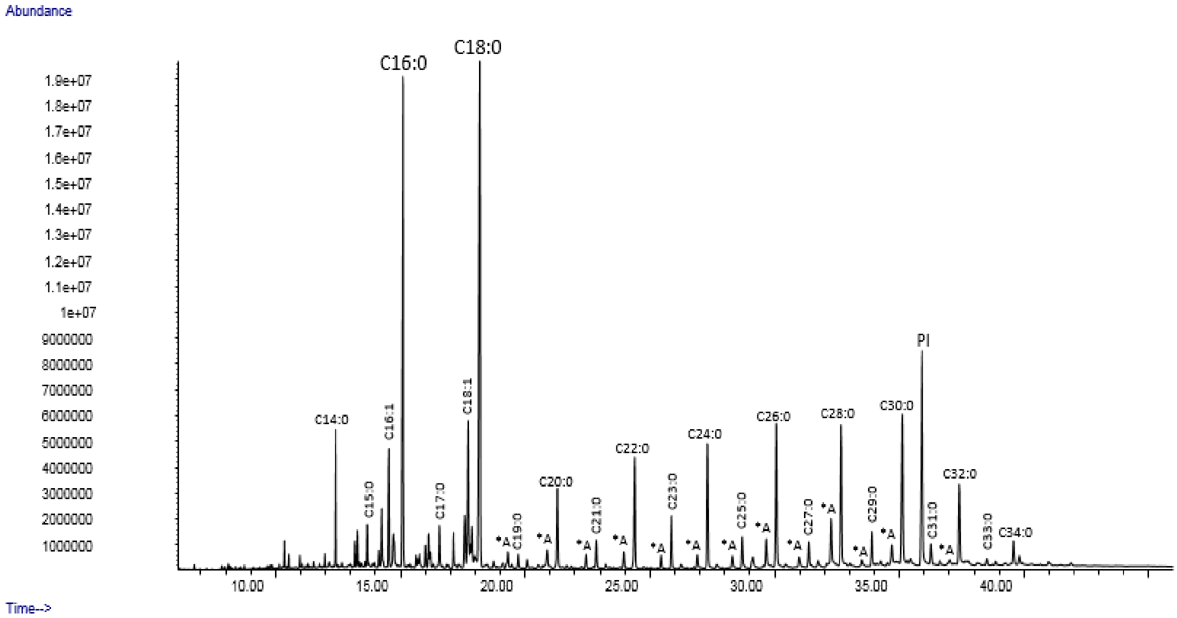
ID Lab	CTT38		
ID Sample	2B24	TR16-700	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.91	-21.07	
TLE ($\mu g \cdot g^{-1}$)	694.9		
Interpretation	Pig adipose fat		

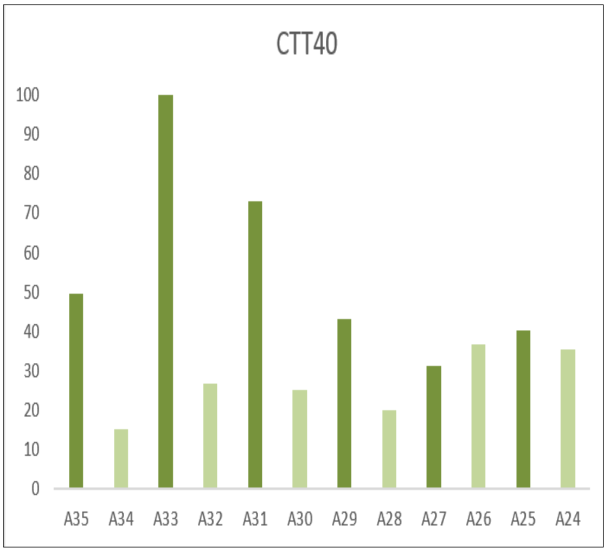
ID Lab	CTT39		
ID Sample		TR16-164	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.61	-27.47	
TLE ($\mu g \cdot g^{-1}$)	569.3		
Interpretation	Wild boar adipose fat Epicuticular wax		

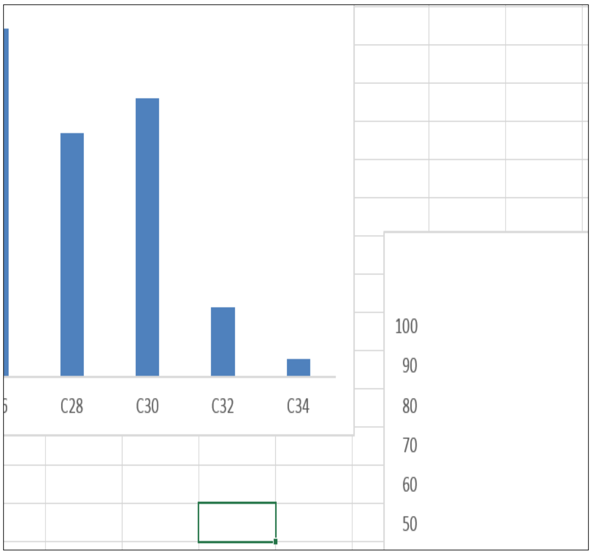


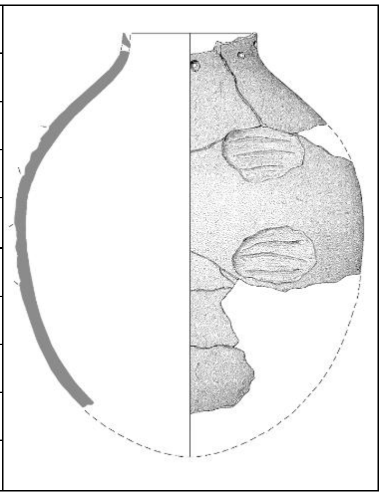


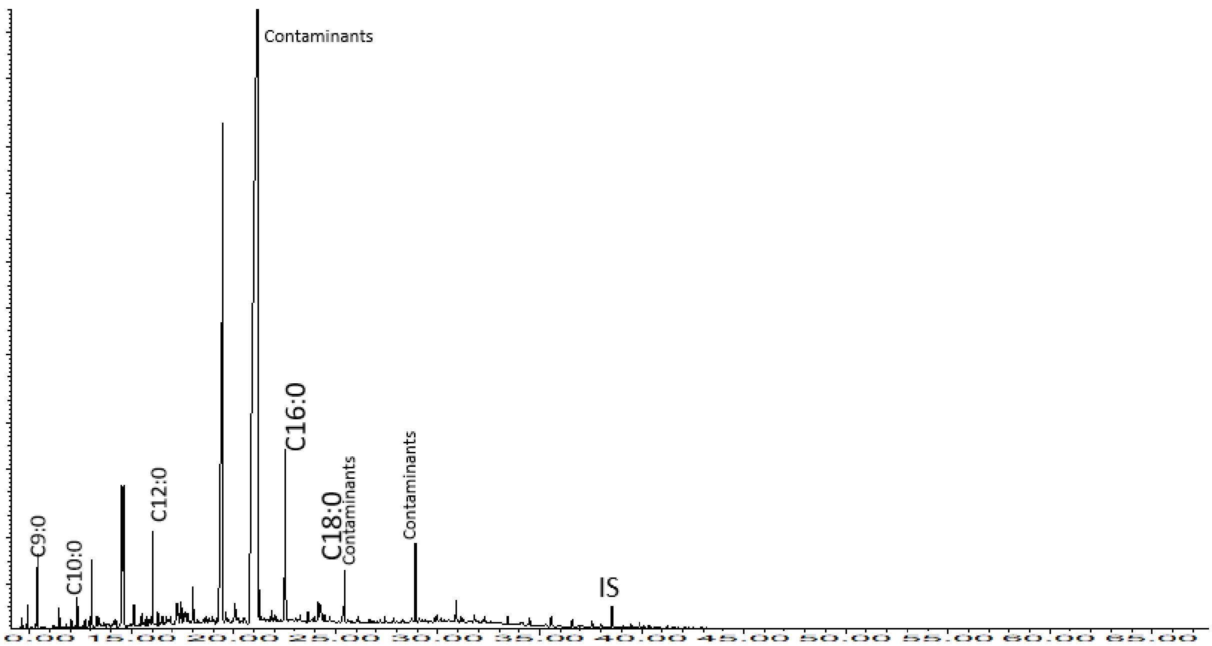
ID Lab	CTT40		
ID Sample		TR16-706	
Site	Coro Trasito		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.11	-31.74	
TLE ($\mu g \cdot g^{-1}$)	476.03		
Interpretation	Dairy fat Epicuticular wax		



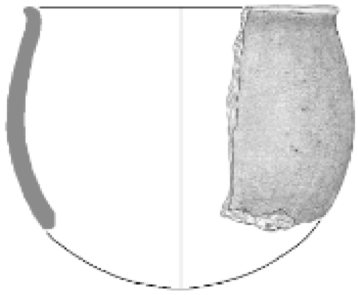




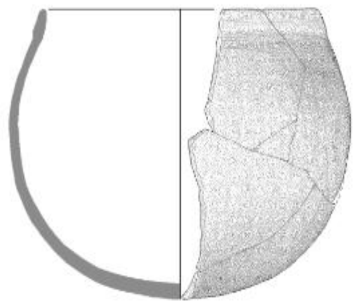
ID Lab	CTM01		
ID Sample	5307-1		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	2.15		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.61	-30.03	
TLE ($\mu g{\cdot}g^{-1}$)	40.07		
Interpretation	Ruminant adipose fat		

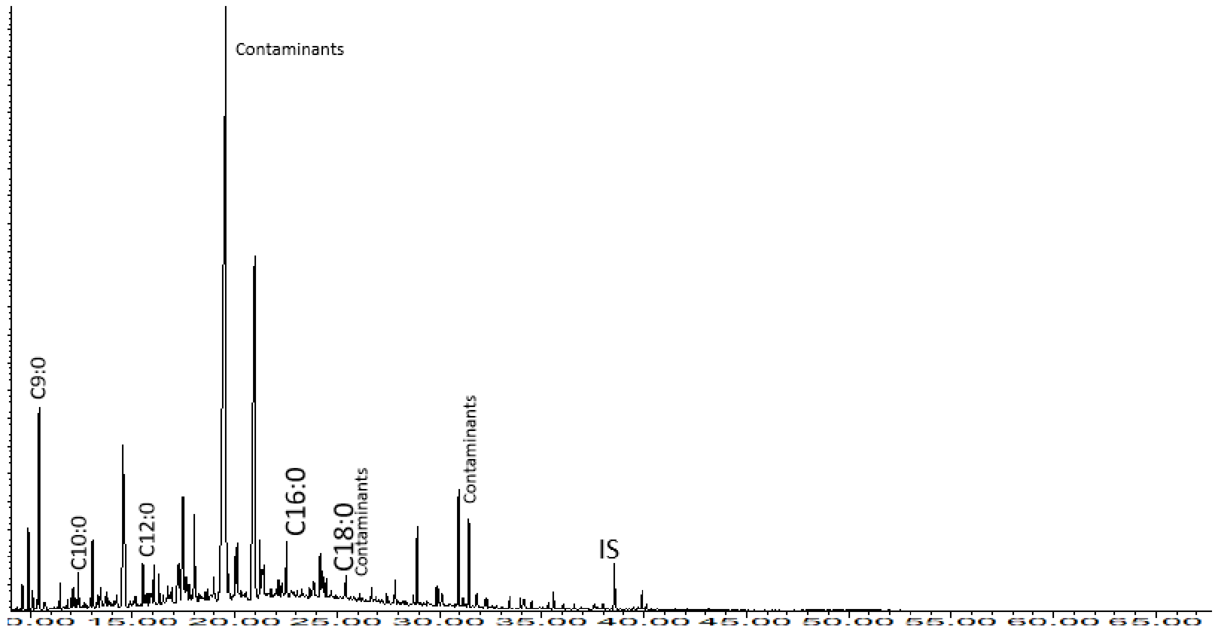

Contaminants
C16:0
C18:0
Contaminants
Contaminants
C9:0
C10:0
C12:0
IS
0.00
15.00
20.00
25.00
30.00
35.00
40.00
45.00
50.00
55.00
60.00
65.00

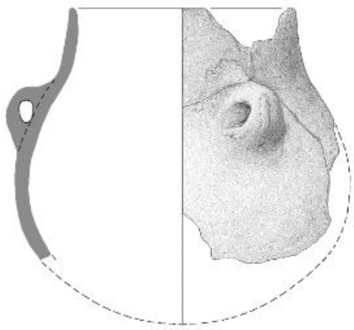
ID Lab	CTM02	
ID Sample	6888	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.37	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

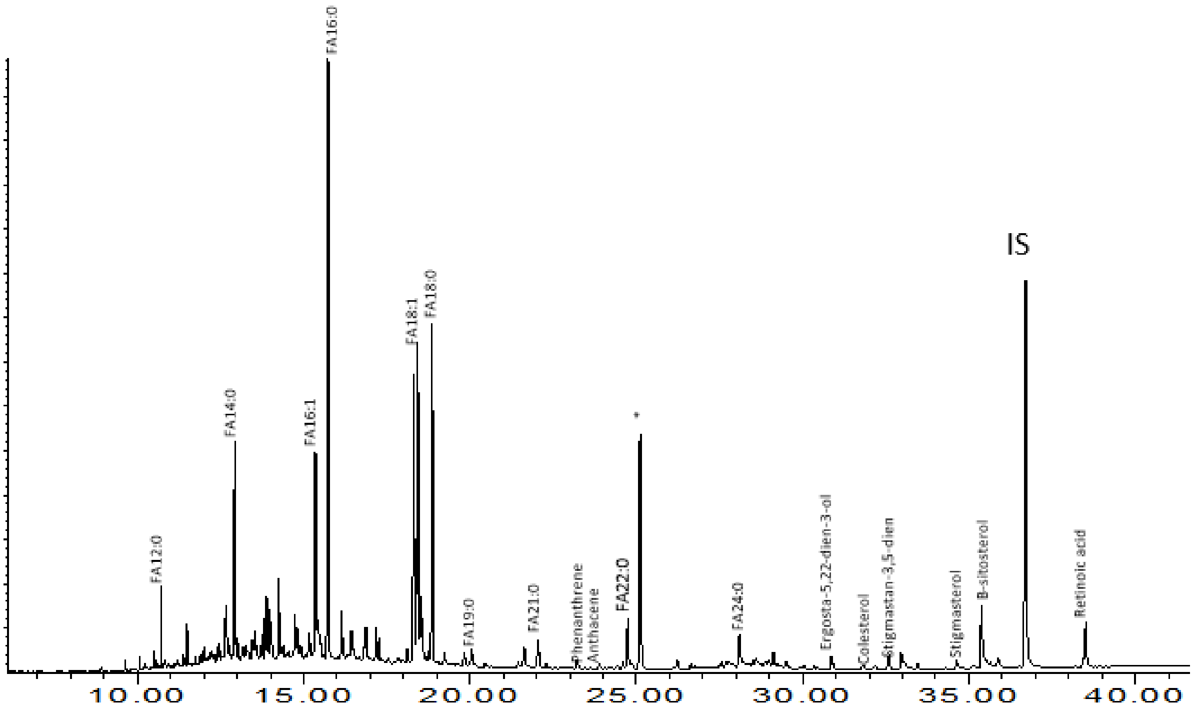


ID Lab	CTM03	
ID Sample	8330	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.76	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-31.28	-32.94
TLE (μg·g-1)	11.36	
Interpretation	Ruminant adipose fat	

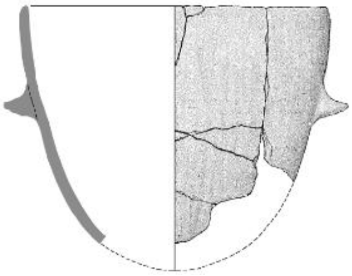




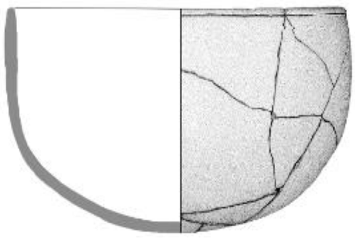
ID Lab	CTM04		
ID Sample	1047		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	0.95		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.83	-26.22	
TLE (µg·g^{-1})	374.25		
Interpretation	Non-ruminant adipose fat Plants		

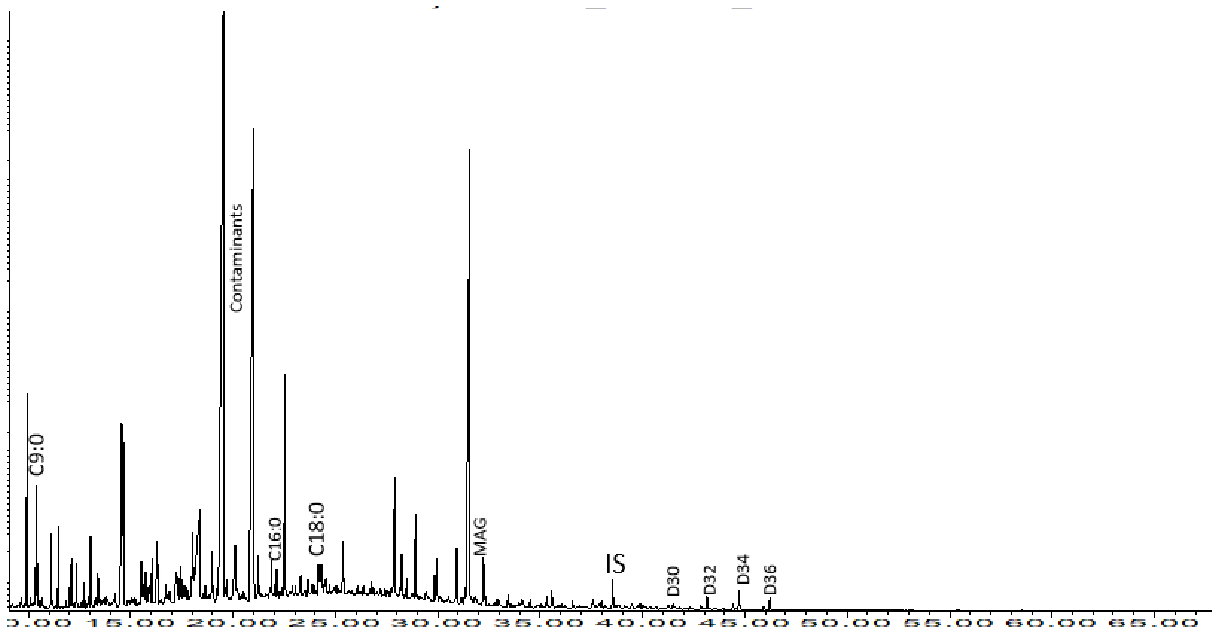


ID Lab	CTM05	
ID Sample	40692-6	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.77	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.11	-28.38
TLE (µg·g-1)	461.85	
Interpretation	Ruminant adipose fat Plants	

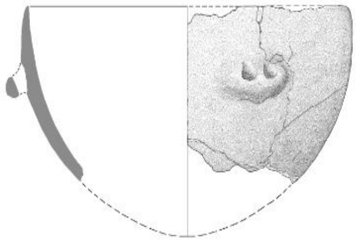


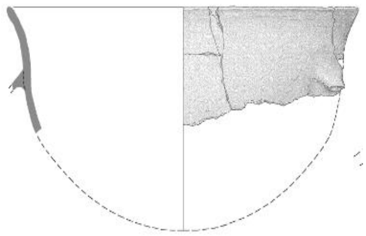
ID Lab	CTM07	
ID Sample	41905	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.64	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.65	-25.18
TLE (μg·g[-1])	9.13	
Interpretation	Non-ruminant adipose fat	

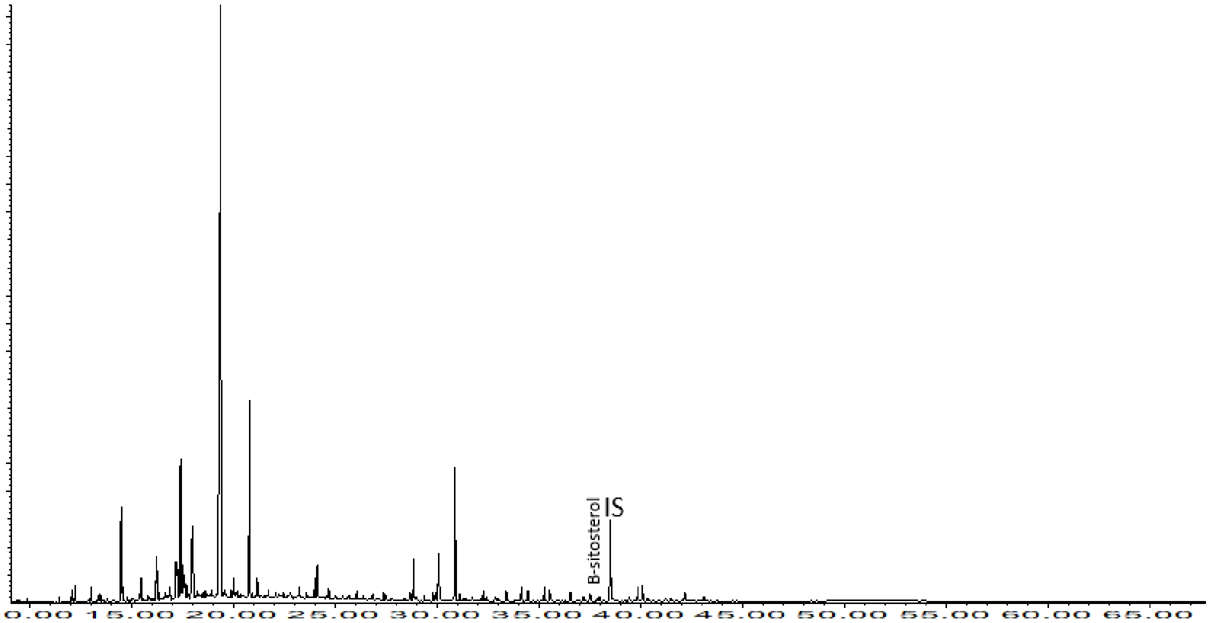




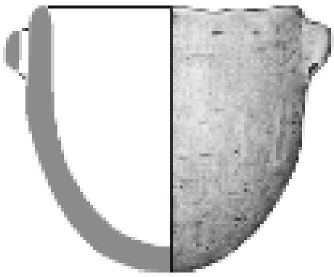
ID Lab	CTM08	
ID Sample	40688-9	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.98	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})		
Interpretation	Negligible	

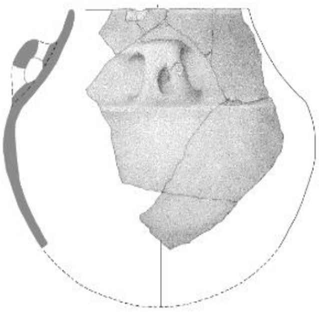


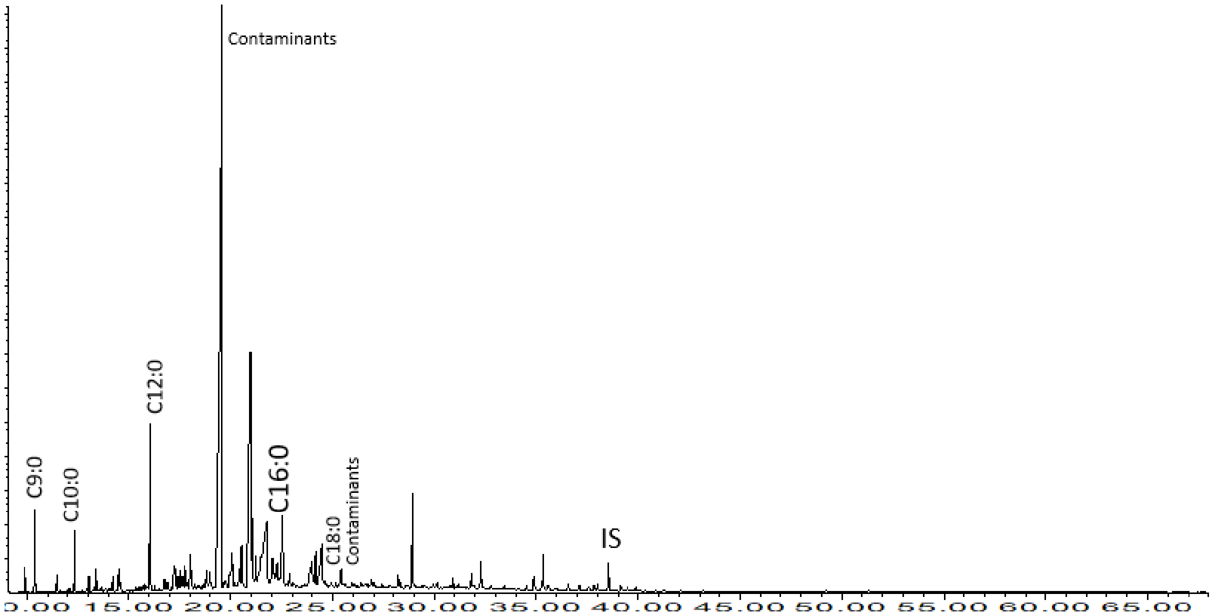
ID Lab	CTM09		
ID Sample	19711-1		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	1.02		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.95	-25.73	
TLE (μg·g^{-1})	46.46		
Interpretation	Non-ruminant adipose fat		



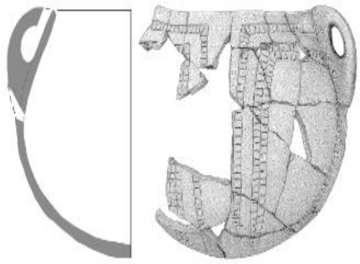
ID Lab	CTM10	
ID Sample	41900	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.11	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (μg·g^{-1})		
Interpretation	Negligible	

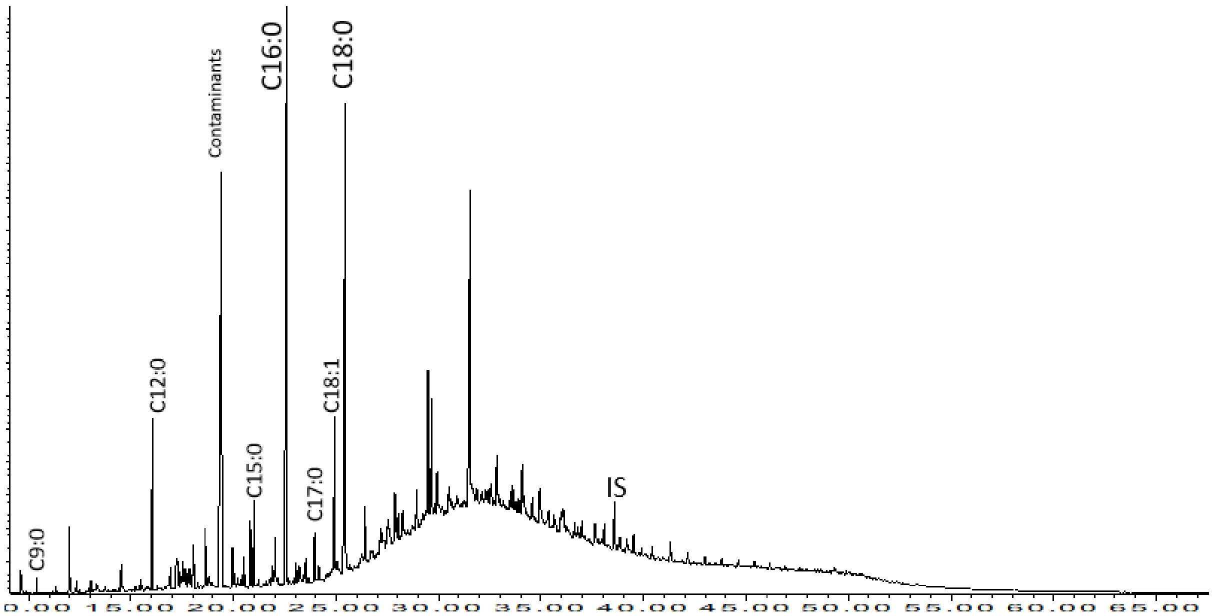


ID Lab	CTM11		
ID Sample	8521		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	1.03		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.07	-24.69	
TLE (µg·g^{-1})	12.83		
Interpretation	Non-ruminant adipose fat		

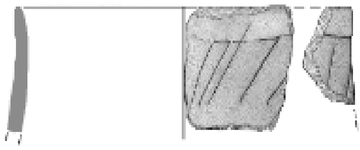


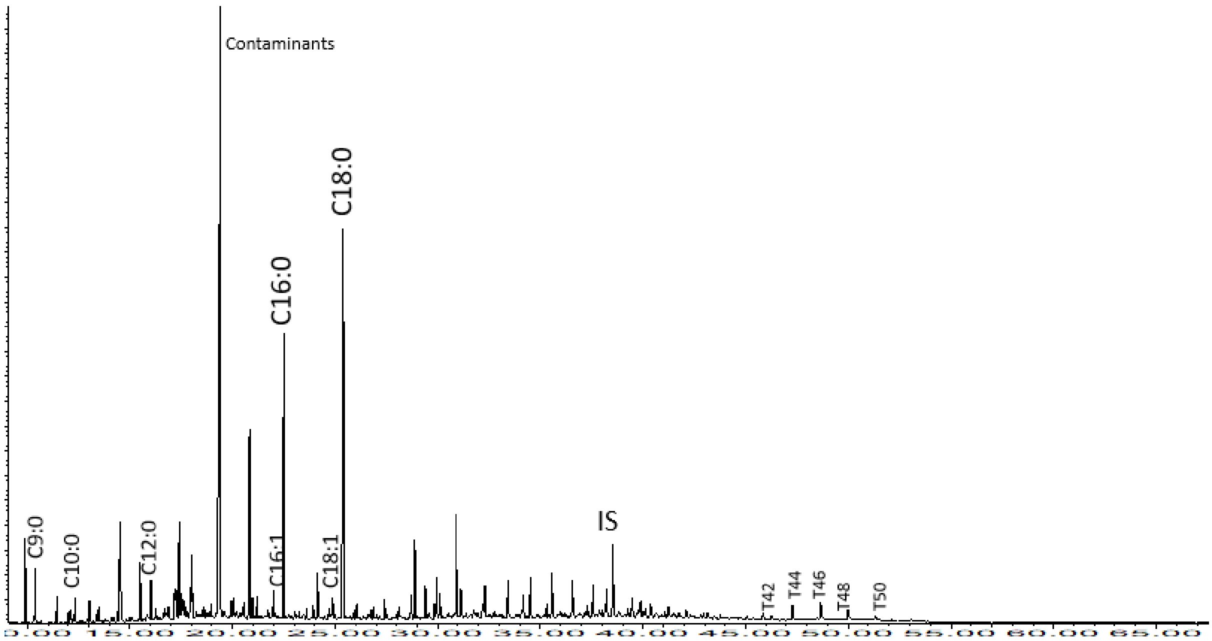
ID Lab	CTM12	
ID Sample	9457	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.52	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-23.82	-22.31
TLE (μg·g[-1])	32.84	
Interpretation	Non-ruminant adipose fat	



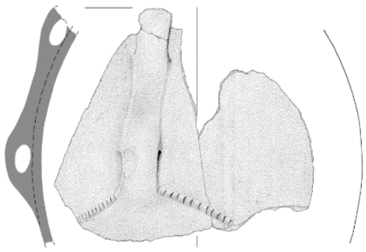


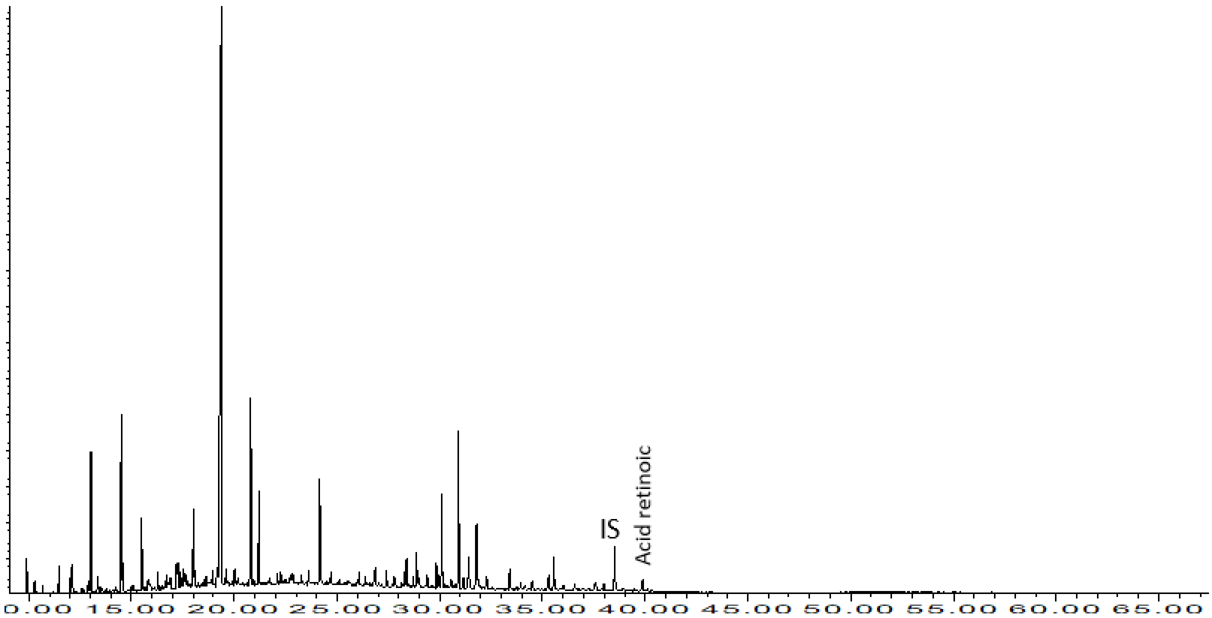
ID Lab	CTM13	
ID Sample	5661	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-28.18	-30.2
TLE (μg·g^{-1})	21.31	
Interpretation	Ruminant adipose fat	



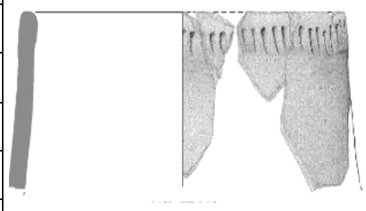


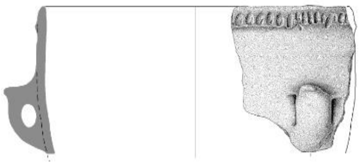
ID Lab	CTM14	
ID Sample	41056	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (μg·g-1)		
Interpretation	Negligible	



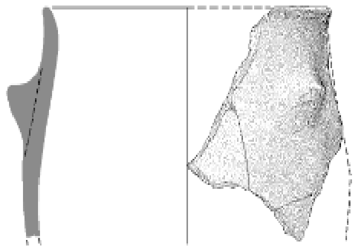


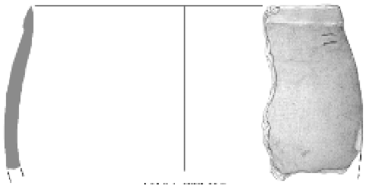
ID Lab	CTM15	
ID Sample	9435	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

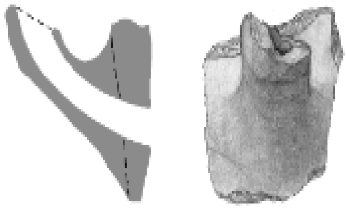


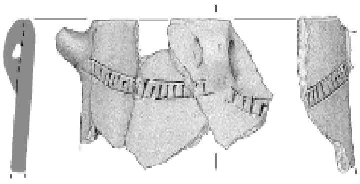
ID Lab	CTM16		
ID Sample	9862		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.46	-26.61	
TLE ($\mu g \cdot g^{-1}$)	22.4		
Interpretation	Non-ruminant adipose fat		

ID Lab	CTM17	
ID Sample	17475	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (μg·g^{-1})		
Interpretation	Negligible	



ID Lab	CTM18		
ID Sample	46104 (Ap4)		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTM19		
ID Sample	46308		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

ID Lab	CTM20		
ID Sample	17730		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

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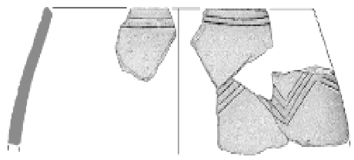
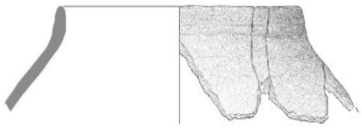
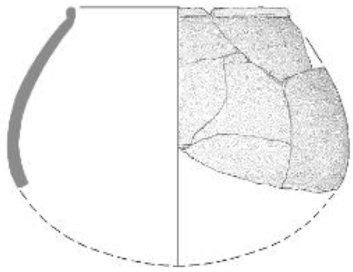
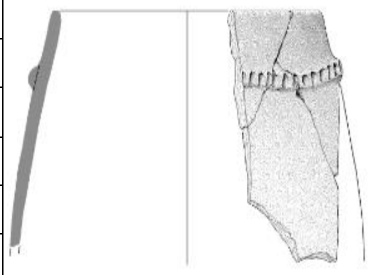
<tr><td>ID Lab</td><td colspan="2">CTM21</td><td rowspan="10"></td></tr>
<tr><td>ID Sample</td><td colspan="2">40882-7</td></tr>
<tr><td>Site</td><td colspan="2">Cueva de El Toro</td></tr>
<tr><td>Morphology</td><td colspan="2"></td></tr>
<tr><td>Handles</td><td colspan="2"></td></tr>
<tr><td>Volume (L)</td><td colspan="2"></td></tr>
<tr><td>Heating biomarkers</td><td colspan="2"></td></tr>
<tr><td>δ13C values [C16:0 / C18:0]</td><td></td><td></td></tr>
<tr><td>TLE ($\mu g \cdot g^{-1}$)</td><td colspan="2"></td></tr>
<tr><td>Interpretation</td><td colspan="2">Negligible</td></tr>
</table>

ID Lab	CTM22
ID Sample	7040
Site	Cueva de El Toro
Morphology	
Handles	
Volume (L)	
Heating biomarkers	
δ13C values [C16:0 / C18:0]	
TLE (µg·g^{-1})	
Interpretation	Negligible

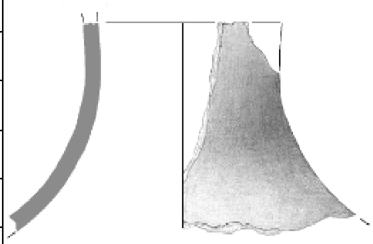


ID Lab	CTM23	
ID Sample	6939	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.65	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})		
Interpretation	Negligible	



ID Lab	CTM24		
ID Sample	1509		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-31.65	-27.14	
TLE (µg·g^{-1})	35.97		
Interpretation	Non-ruminant adipose fat		

ID Lab	CTM25	
ID Sample	1019	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (µg·g^{-1})		
Interpretation	Negligible	



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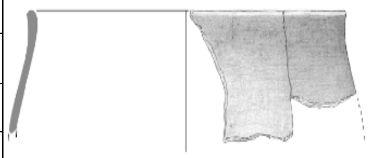
<tr><td>ID Lab</td><td colspan="2">CTM26</td><td rowspan="10"></td></tr>
<tr><td>ID Sample</td><td colspan="2">19326</td></tr>
<tr><td>Site</td><td colspan="2">Cueva de El Toro</td></tr>
<tr><td>Morphology</td><td colspan="2"></td></tr>
<tr><td>Handles</td><td colspan="2"></td></tr>
<tr><td>Volume (L)</td><td colspan="2"></td></tr>
<tr><td>Heating biomarkers</td><td colspan="2"></td></tr>
<tr><td>δ13C values [C16:0 / C18:0]</td><td></td><td></td></tr>
<tr><td>TLE ($\mu g \cdot g^{-1}$)</td><td colspan="2"></td></tr>
<tr><td>Interpretation</td><td colspan="2">Negligible</td></tr>
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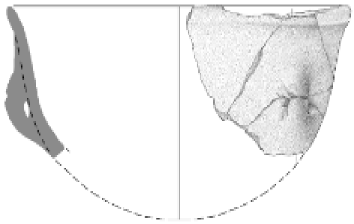
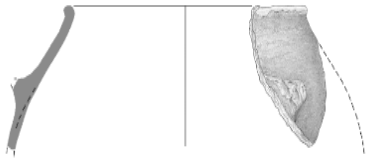
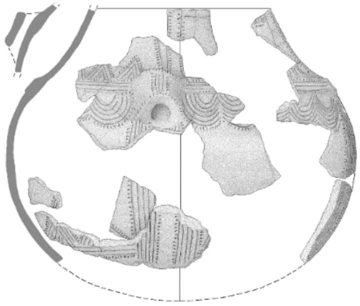
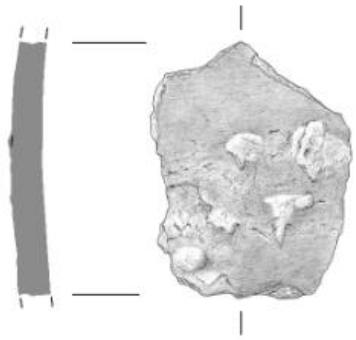
ID Lab	CTM27		
ID Sample	7259		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	0.39		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})			
Interpretation	Negligible		

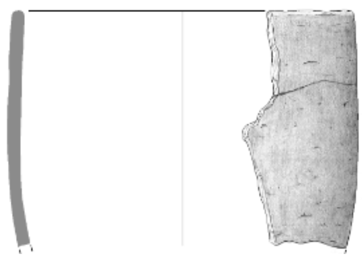
ID Lab	CTM28		
ID Sample	5413 (Ap9)		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTM29		
ID Sample	1407		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	3.66		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

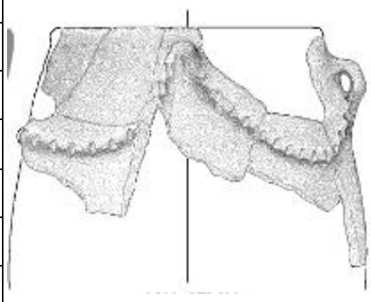
ID Lab	CTM30	
ID Sample	40763-4	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})		
Interpretation	Negligible	

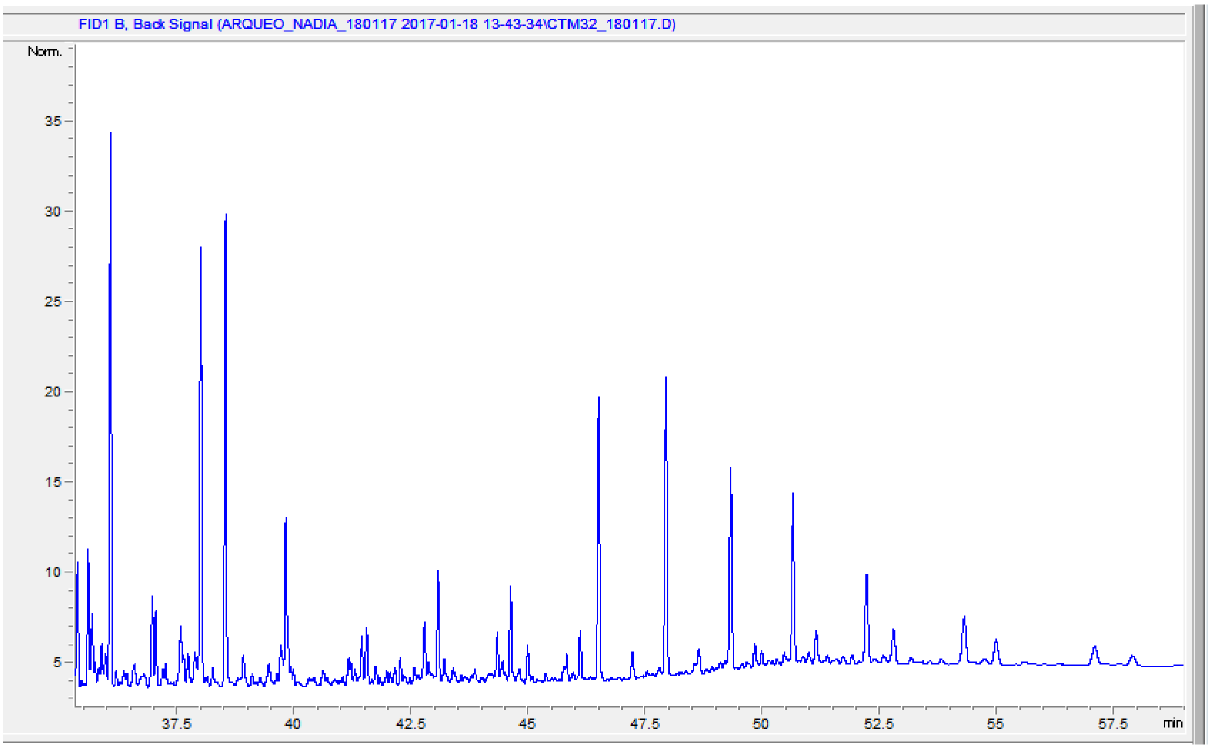


ID Lab	CTM31	
ID Sample	19325 (Ap6)	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

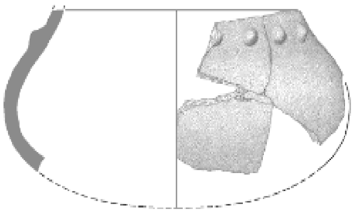


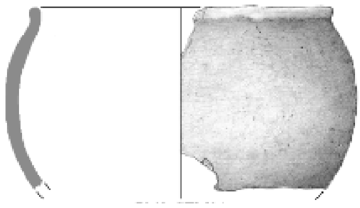
ID Lab	CTM32	
ID Sample	8704	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.74	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.75	-30.13
TLE (μg·g^{-1})	78.4	
Interpretation	Non-ruminant adipose fat	



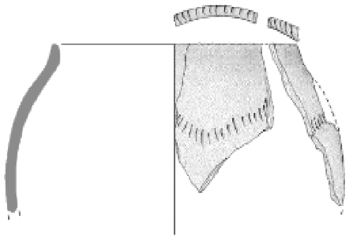


ID Lab	CTM33	
ID Sample	18197	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.47	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.58	-24.39
TLE (μg·g^{-1})	14.93	
Interpretation	Non-ruminant adipose fat	



ID Lab	CTM34		
ID Sample	7962		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	0.47		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CTM35	
ID Sample	6922	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.76	-23.88
TLE ($\mu g \cdot g^{-1}$)	58.41	
Interpretation	Non-ruminant adipose fat	



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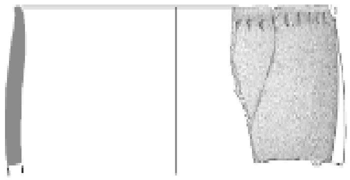
<tr><td>ID Lab</td><td colspan="2">CTM36</td><td rowspan="10"></td></tr>
<tr><td>ID Sample</td><td colspan="2">19865</td></tr>
<tr><td>Site</td><td colspan="2">Cueva de El Toro</td></tr>
<tr><td>Morphology</td><td colspan="2"></td></tr>
<tr><td>Handles</td><td colspan="2"></td></tr>
<tr><td>Volume (L)</td><td colspan="2"></td></tr>
<tr><td>Heating biomarkers</td><td colspan="2"></td></tr>
<tr><td>δ13C values [C16:0 / C18:0]</td><td></td><td></td></tr>
<tr><td>TLE ($\mu g \cdot g^{-1}$)</td><td colspan="2"></td></tr>
<tr><td>Interpretation</td><td colspan="2">Negligible</td></tr>
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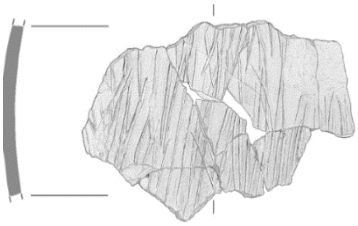
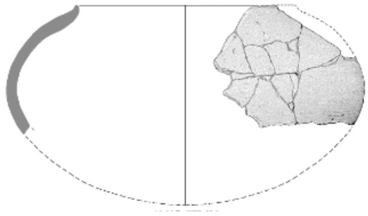
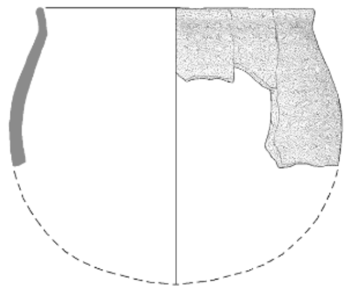
ID Lab	CTM37	
ID Sample	1291	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	



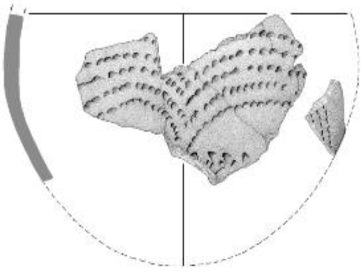
ID Lab	CTM38		
ID Sample	40698		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	0.80		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.4	-29.11	
TLE (µg·g^{-1})	537.2		
Interpretation	Ruminant adipose fat Plants		

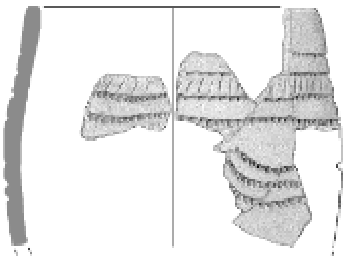
ID Lab	CTM39	
ID Sample	14597	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	1.1	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

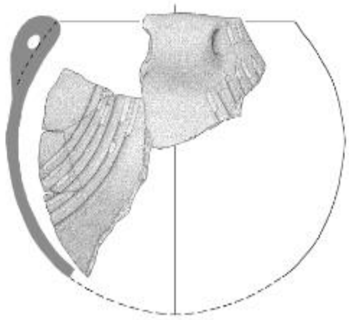


ID Lab	CTM40		
ID Sample	1215		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.82	-23.88	
TLE ($\mu g \cdot g^{-1}$)	40.65		
Interpretation	Non-ruminant adipose fat		

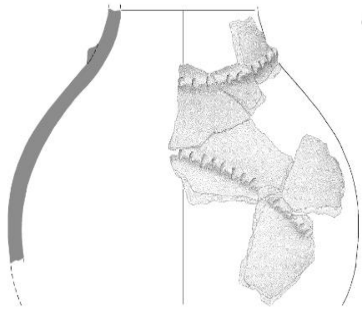
ID Lab	CTM41	
ID Sample	40938	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	0.70	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.14	-26.01
TLE (µg·g^{-1})	43.69	
Interpretation	Non-ruminant adipose fat	

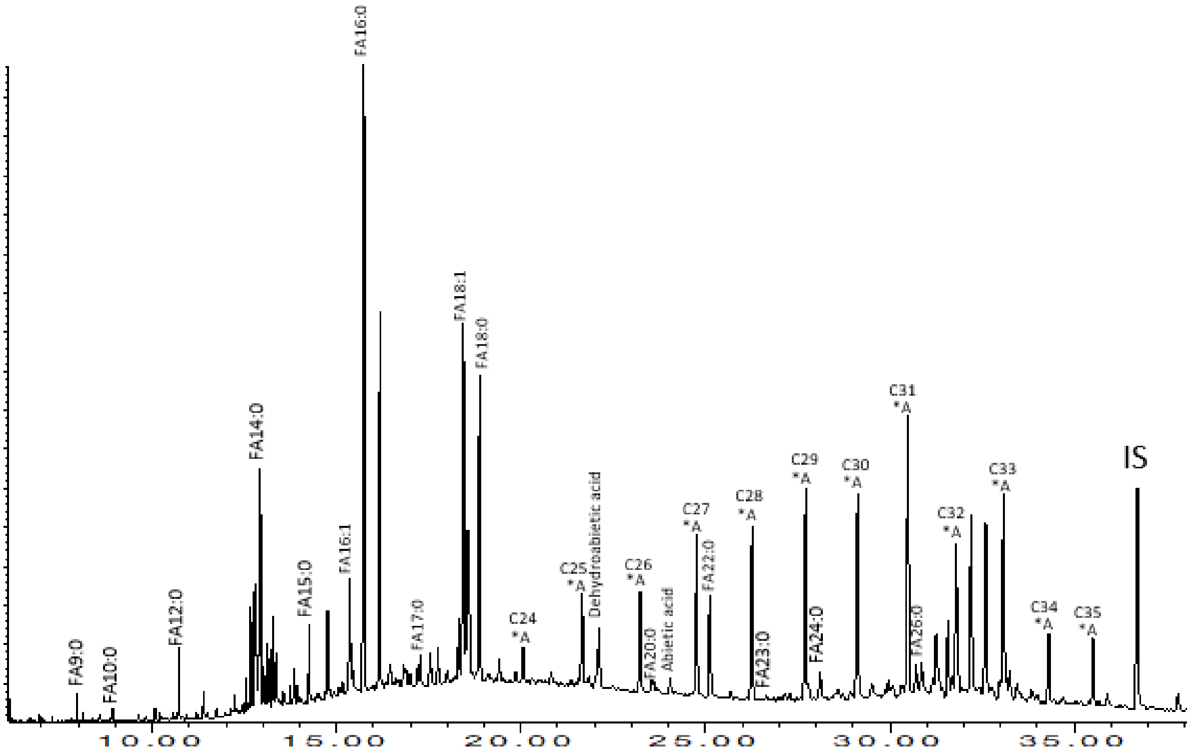


ID Lab	CTM42		
ID Sample	40939		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.76	-27.54	
TLE (μg·g^{-1})	15.3		
Interpretation	Non-ruminant adipose fat		

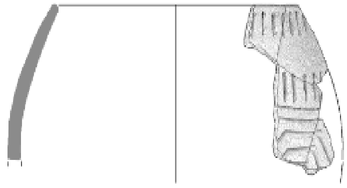
ID Lab	CTM43		
ID Sample	40887		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)	0.64		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.42	-24.05	
TLE (µg·g^{-1})	834.48		
Interpretation	Non-ruminant adipose fat Pine resin		

ID Lab	CTM44	
ID Sample	46587	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)	2.26	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-30.56	-32.43
TLE (µg·g^{-1})	325.3	
Interpretation	Ruminant adipose fat Pine resin	



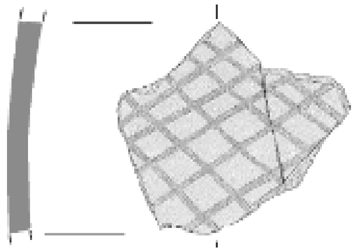


ID Lab	CTM45	
ID Sample	18552	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})		
Interpretation	Negligible	

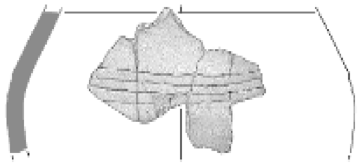


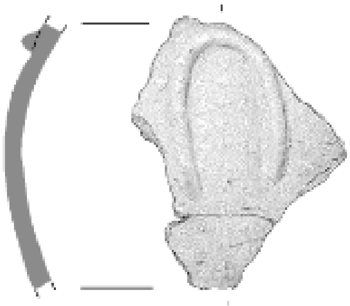
ID Lab	CTM46		
ID Sample	5811		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.82	-28.33	
TLE ($\mu g \cdot g^{-1}$)	53.22		
Interpretation	Ruminant adipose fat		

ID Lab	CTM47	
ID Sample	45687	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (μg·g^{-1})		
Interpretation	Negligible	

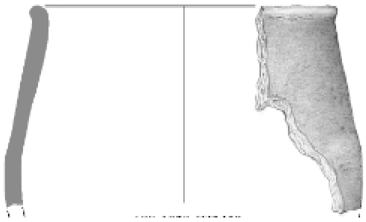


ID Lab	CTM48	
ID Sample	22087	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g-1)		
Interpretation	Negligible	



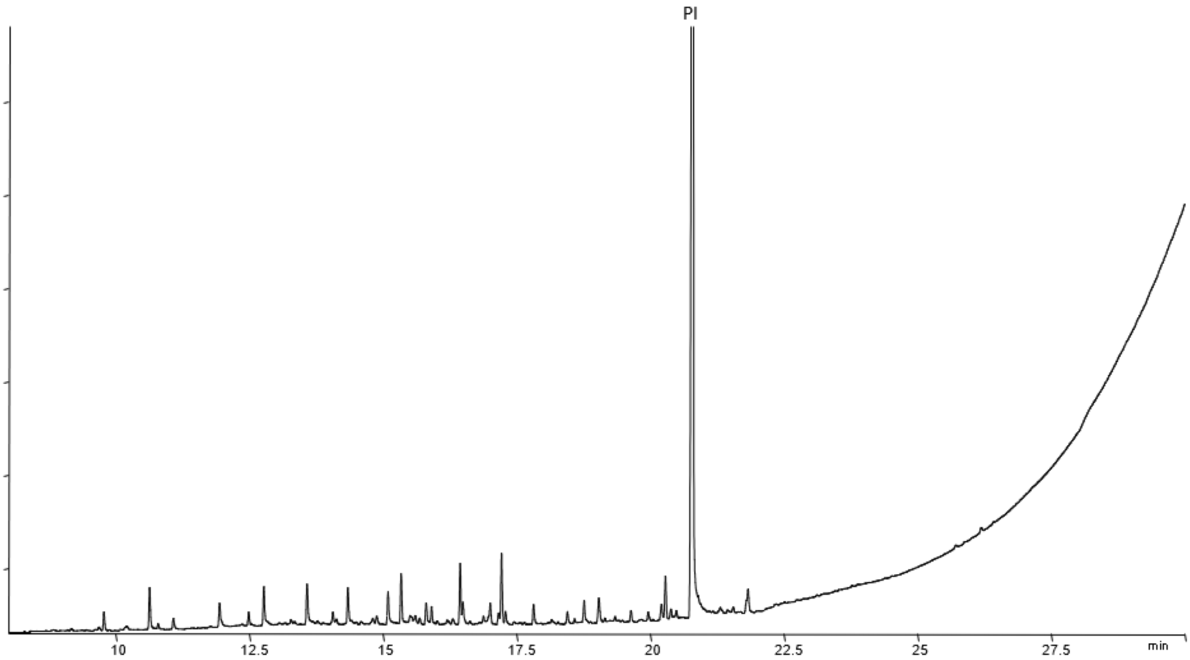
ID Lab	CTM49		
ID Sample	46716		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.56	-28.84	
TLE ($\mu g \cdot g^{-1}$)	33.84		
Interpretation	Ruminant adipose fat		

ID Lab	CTM50	
ID Sample	1038	
Site	Cueva de El Toro	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-29.31	-30.37
TLE ($\mu g \cdot g^{-1}$)	78.36	
Interpretation	Ruminant adipose fat	



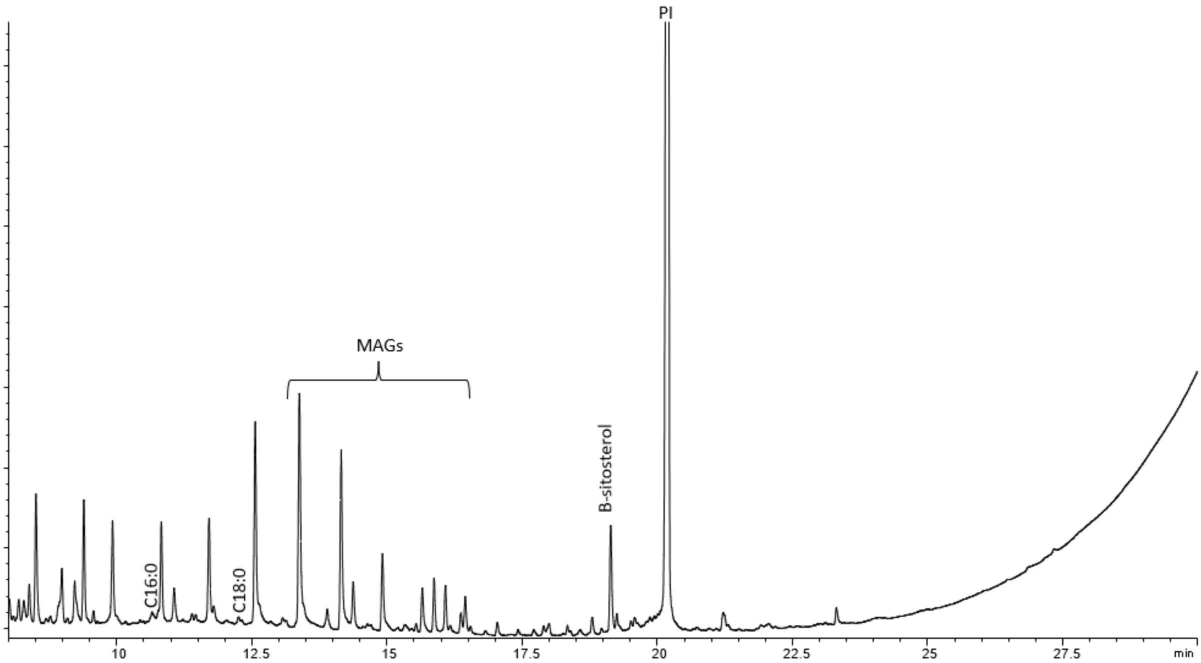
ID Lab	CTM51		
ID Sample	322		
Site	Cueva de El Toro		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.55	-30.44	
TLE ($\mu g \cdot g^{-1}$)	722.11		
Interpretation	Dairy fat		

ID Lab	MIG02	
ID Sample	M108	
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (μg·g^{-1})		
Interpretation	Negligible	

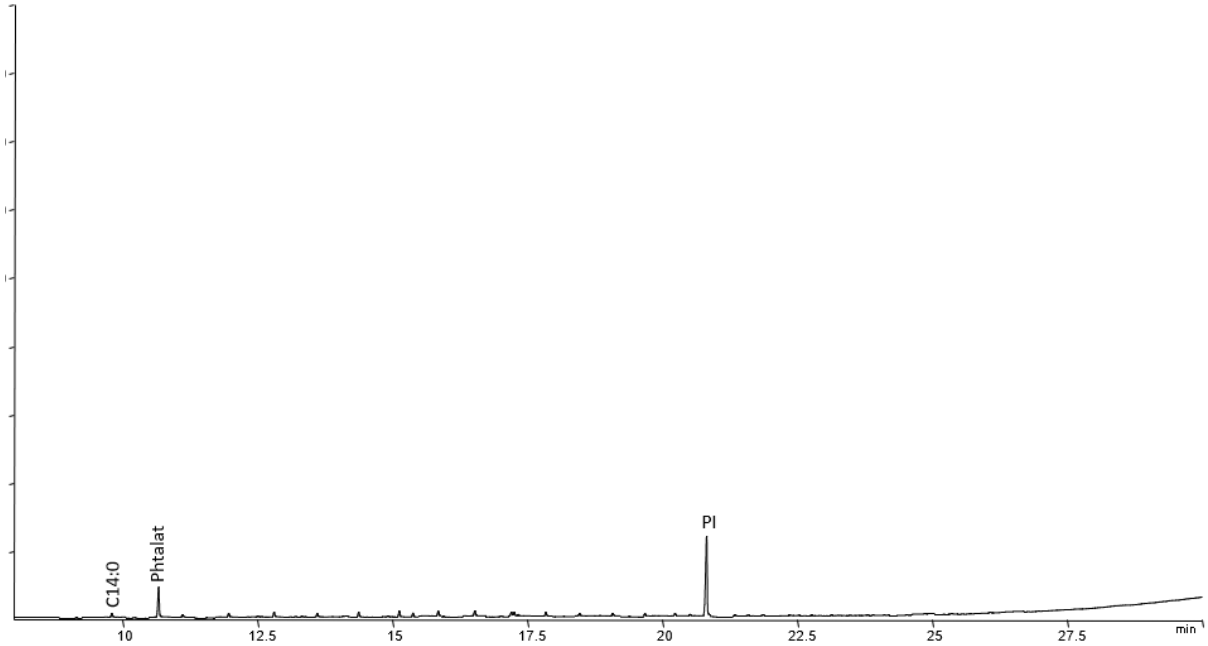


ID Lab	MIG07		
ID Sample	M41	NI	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

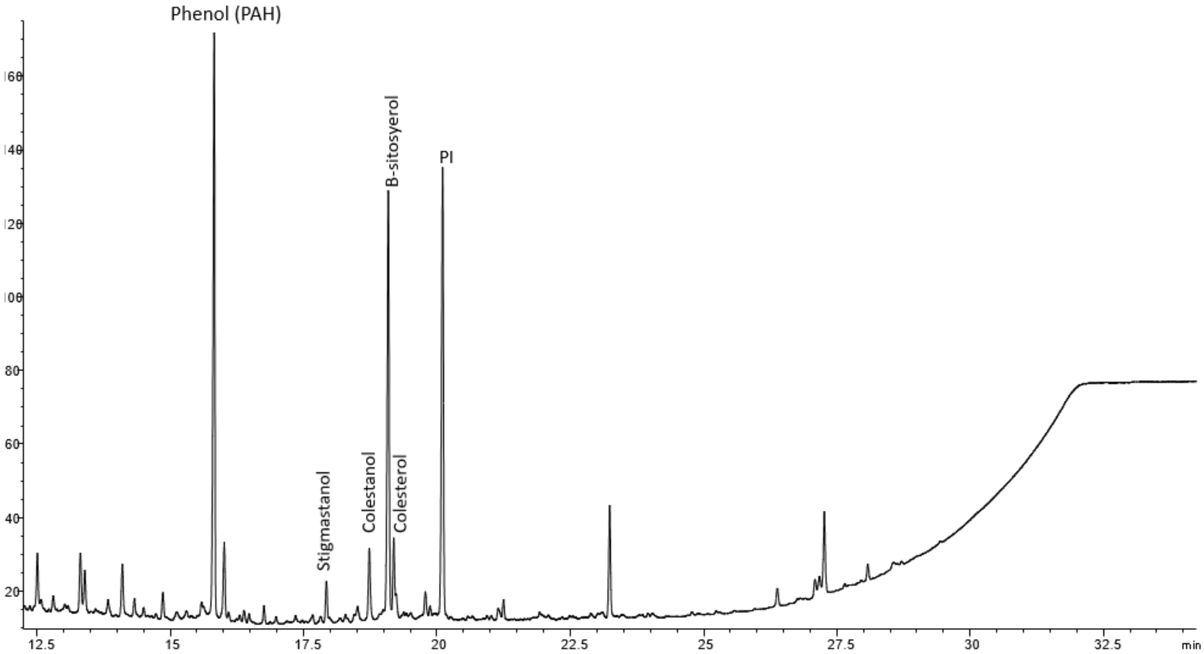
ID Lab	MIG08		IMAGE NOT AVAILABLE
ID Sample	M89		
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		



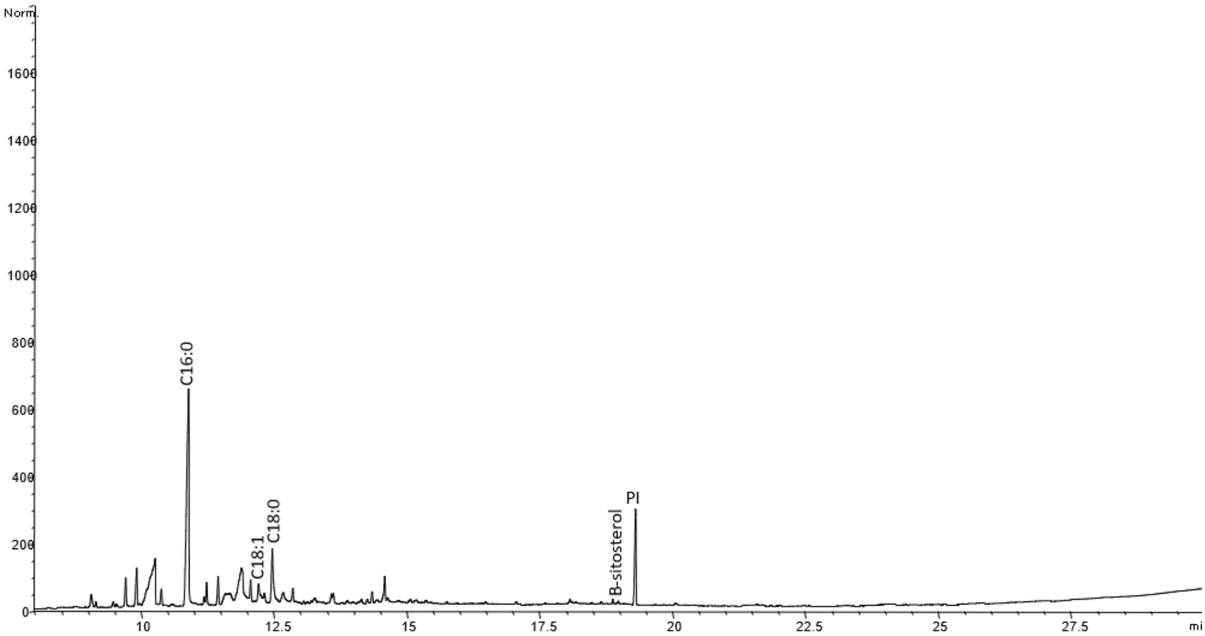
ID Lab	MIG09		
ID Sample	M7	390-33	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.1	-22.99	
TLE ($\mu g \cdot g^{-1}$)	103.2		
Interpretation	Non ruminant adipose fat		



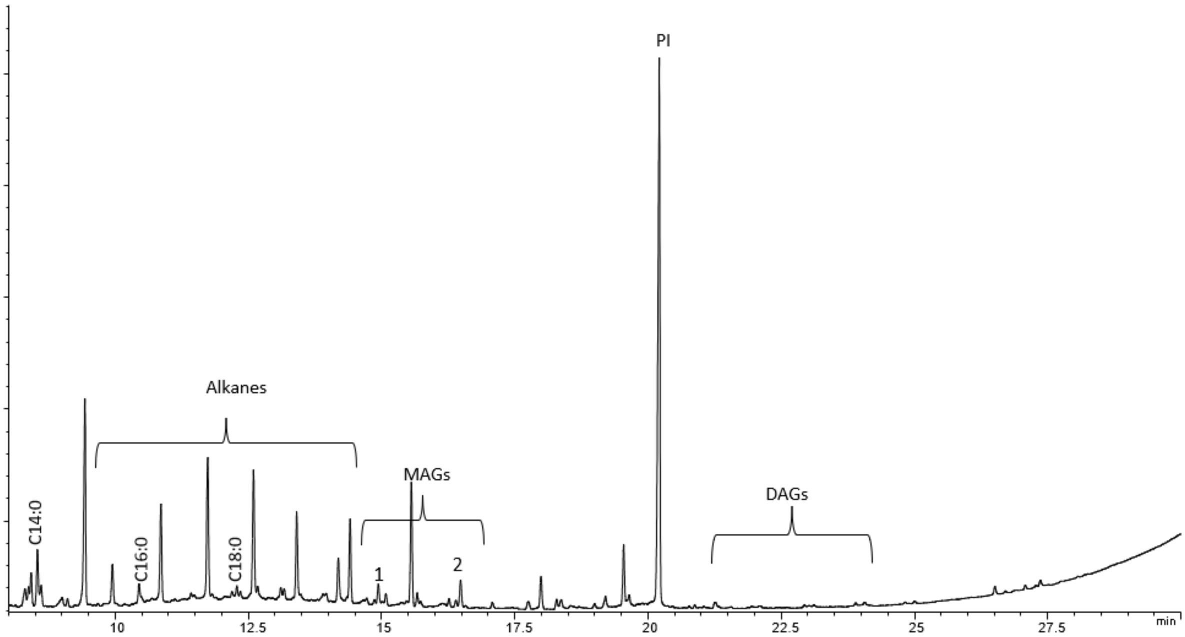
ID Lab	MIG10	
ID Sample	M41	1224
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-22.94	-23.27
TLE ($\mu g \cdot g^{-1}$)	99.89	
Interpretation	Non ruminant adipose fat	



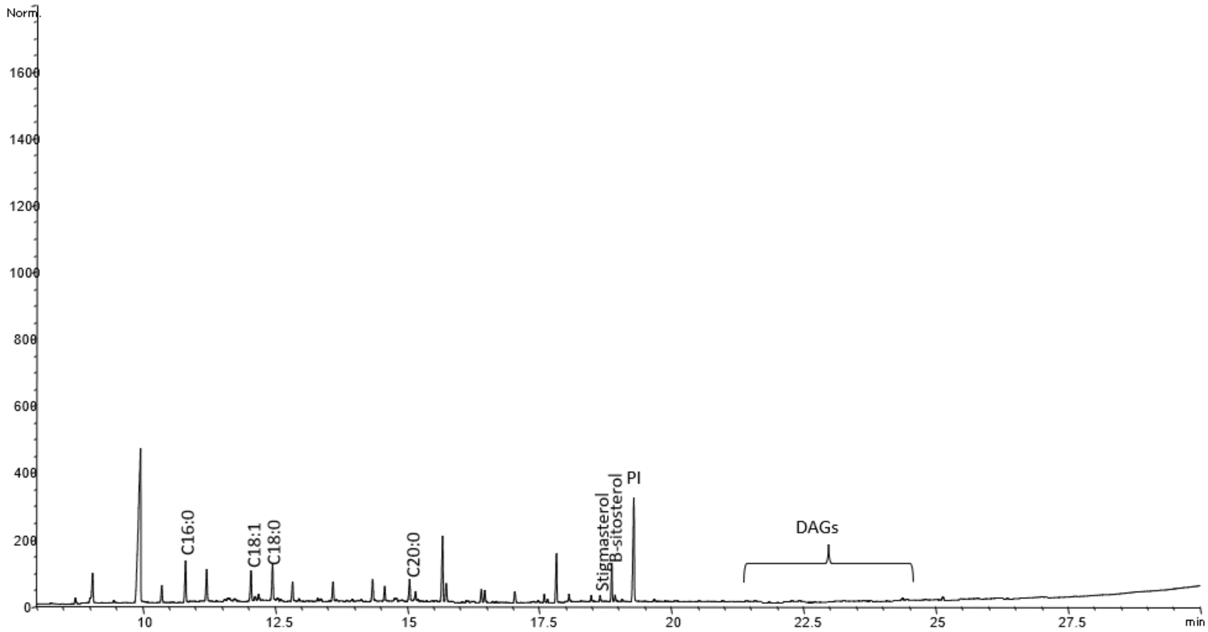
ID Lab	MIG11	
ID Sample	M83	
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.2	-26.83
TLE (µg·g[-1])	224.35	
Interpretation	Non ruminant adipose fat	



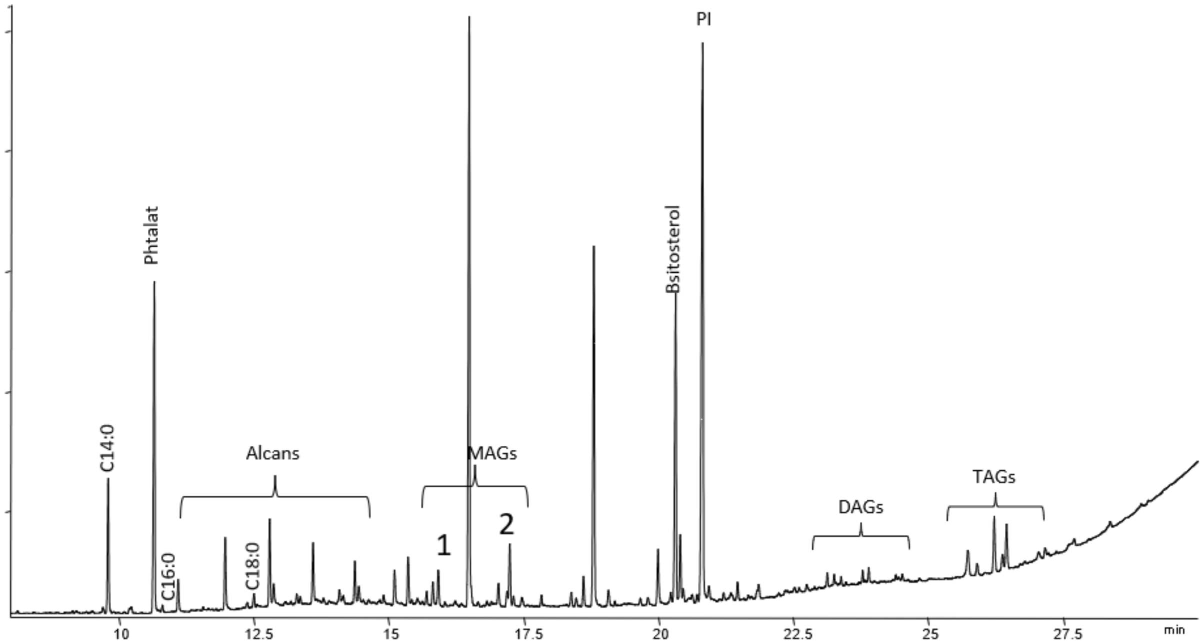
ID Lab	MIG12	
ID Sample	M6	408-1-NII-SF
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-28.93	-31.17
TLE (μg·g^{-1})	115.91	
Interpretation	Ruminant adipose fat	



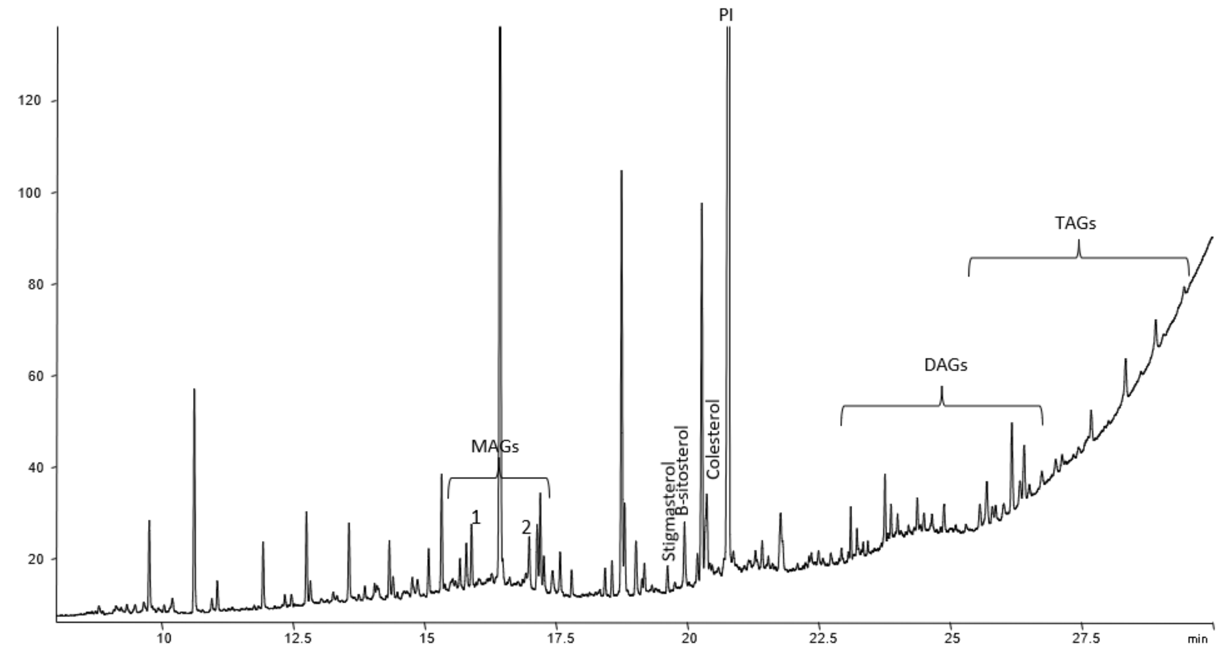
ID Lab	MIG13	
ID Sample	M68	11
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.11	-25.27
TLE (µg·g^{-1})	202.2	
Interpretation	Non ruminant adipose fat Plants	



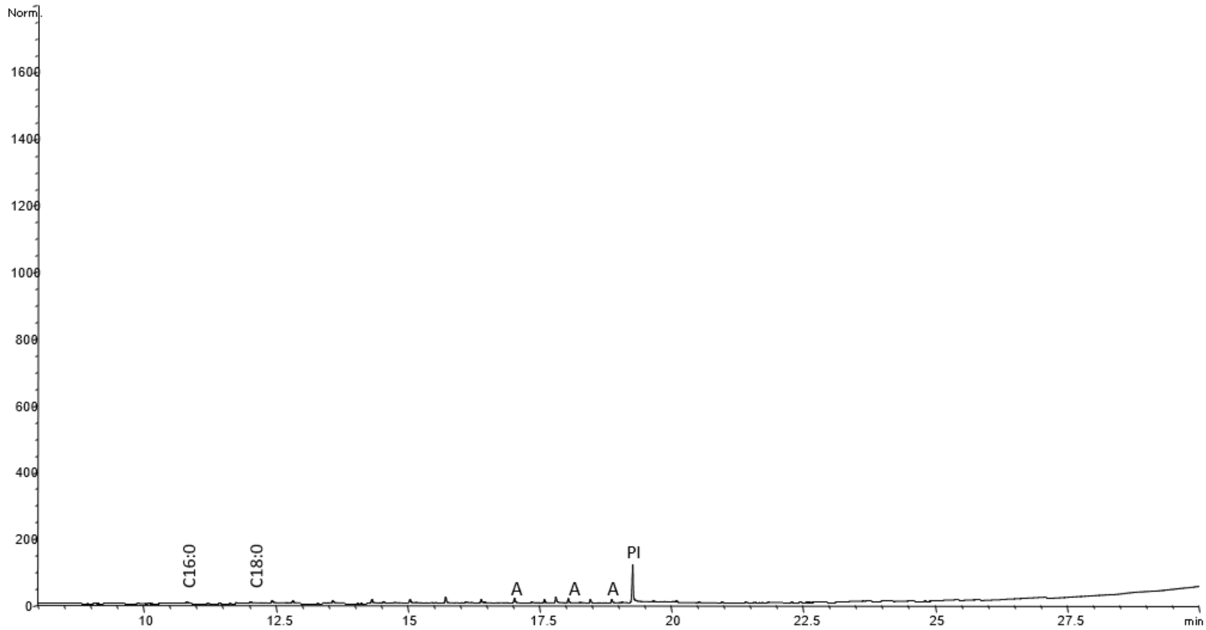
ID Lab	MIG14	
ID Sample	M6	408-2
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.5	-23.42
TLE (μg·g^{-1})	104.03	
Interpretation	Non ruminant adipose fat	



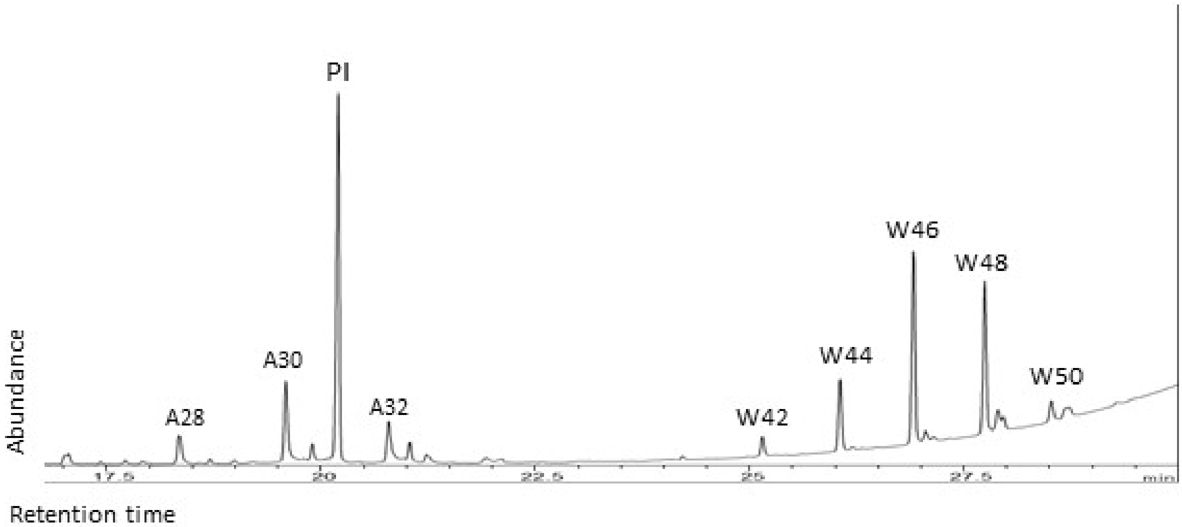
ID Lab	MIG16	
ID Sample	M8	418-16
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.57	-25.57
TLE (μg·g[-1])	225.69	
Interpretation	Non ruminant adipose fat	

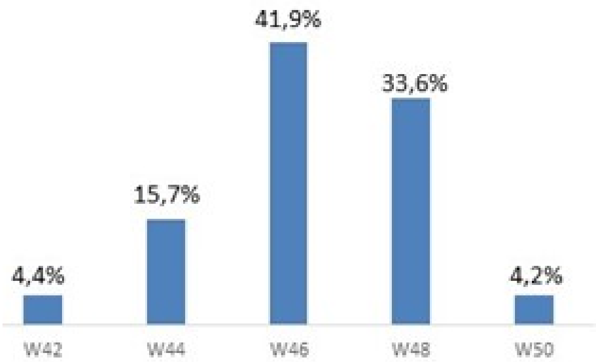


ID Lab	MIG17	
ID Sample	M85	CT41164
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.91	-29.23
TLE ($\mu g \cdot g^{-1}$)	89.71	
Interpretation	Ruminant adipose fat	



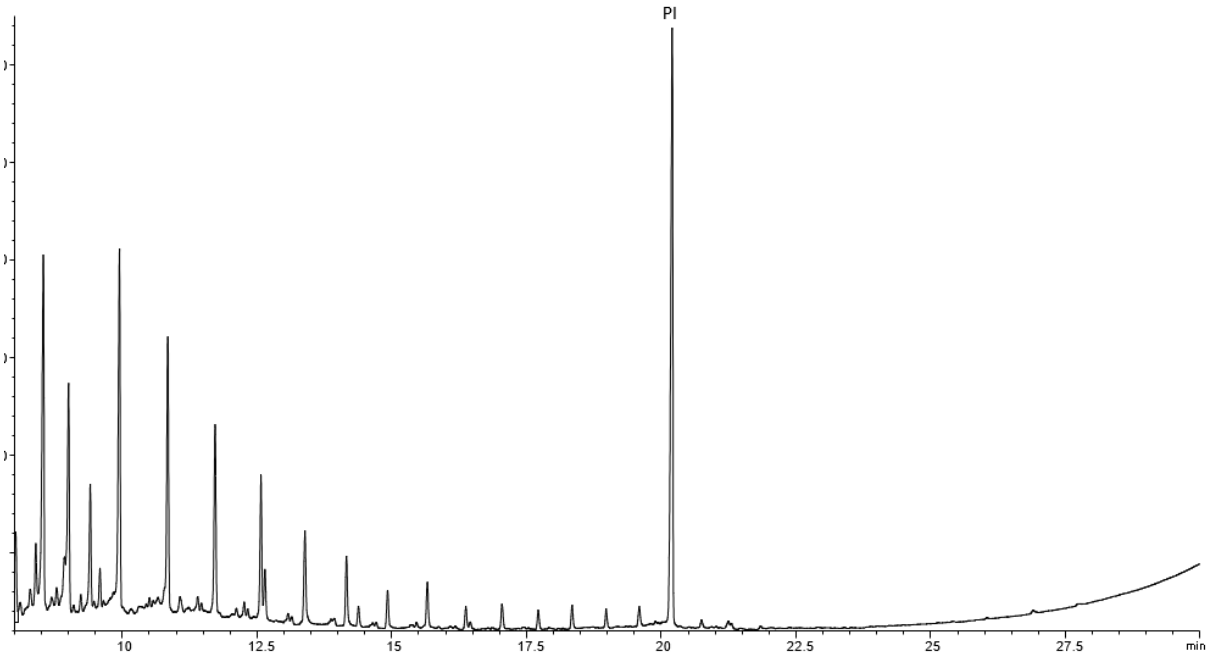
ID Lab	MIG18		
ID Sample	M84		
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	X	X	
TLE (µg·g-1)	89.01		
Interpretation	Beeswax		



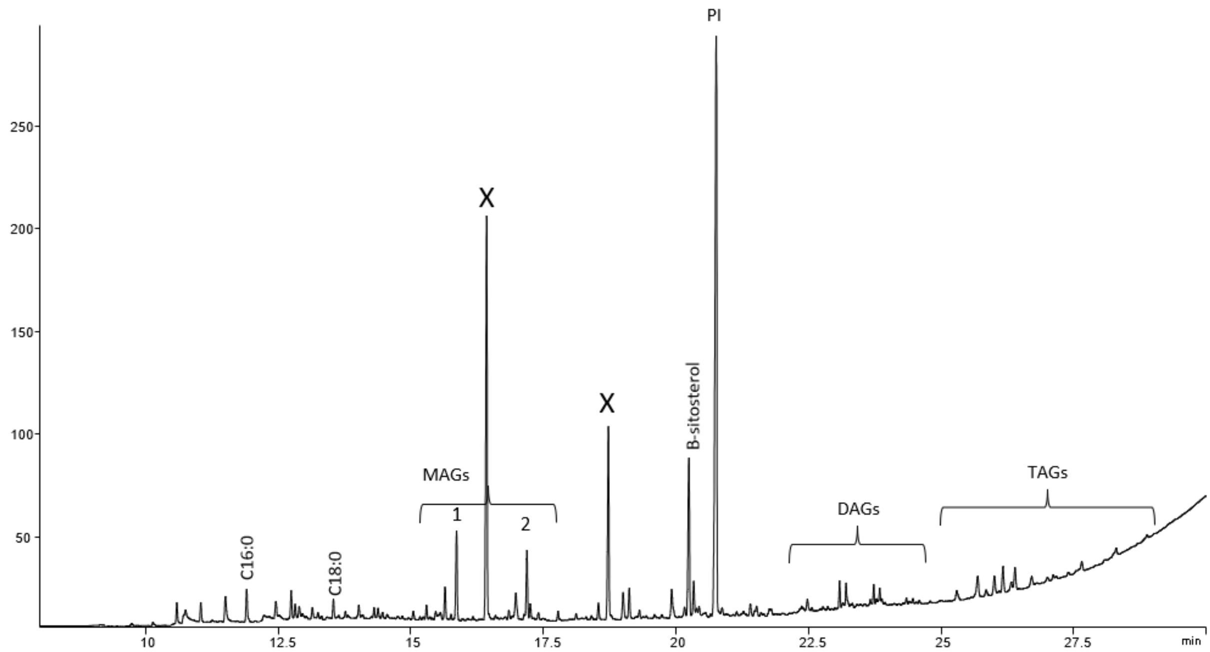


ID Lab	MIG19	
ID Sample	M84	214
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.4	-29.88
TLE (μg·g^{-1})	232.65	
Interpretation	Ruminant adipose fat	

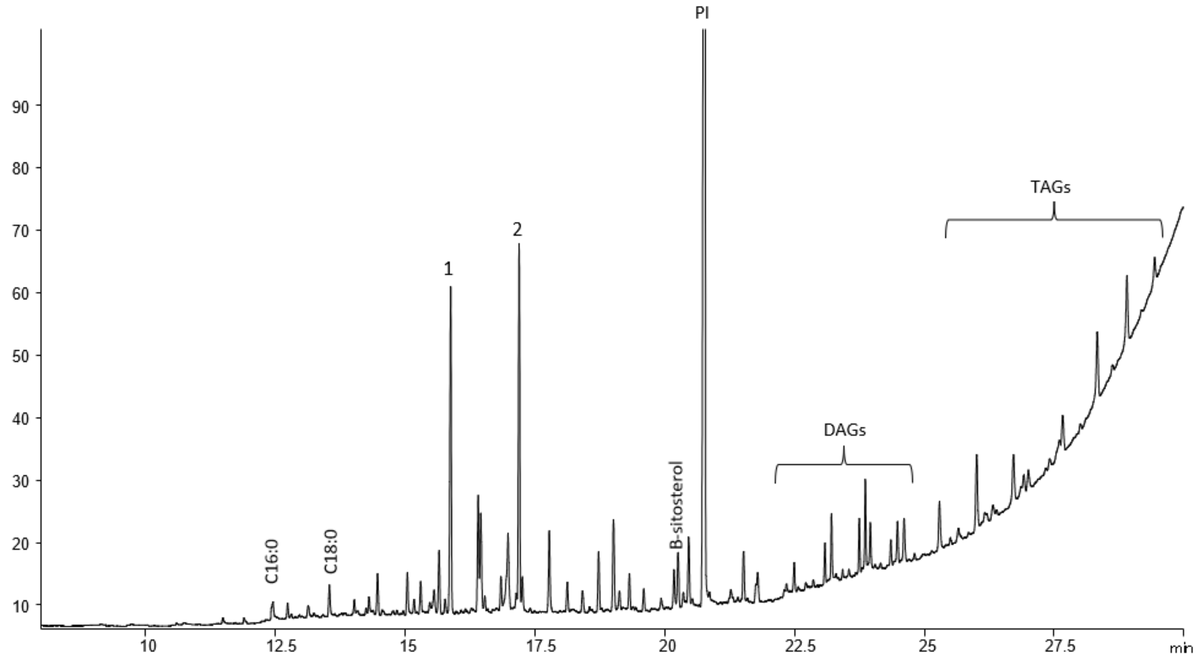
ID Lab	MIG20	
ID Sample	M84	1
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.97	-24.6
TLE ($\mu g \cdot g^{-1}$)	187.4	
Interpretation	Non ruminant adipose fat	



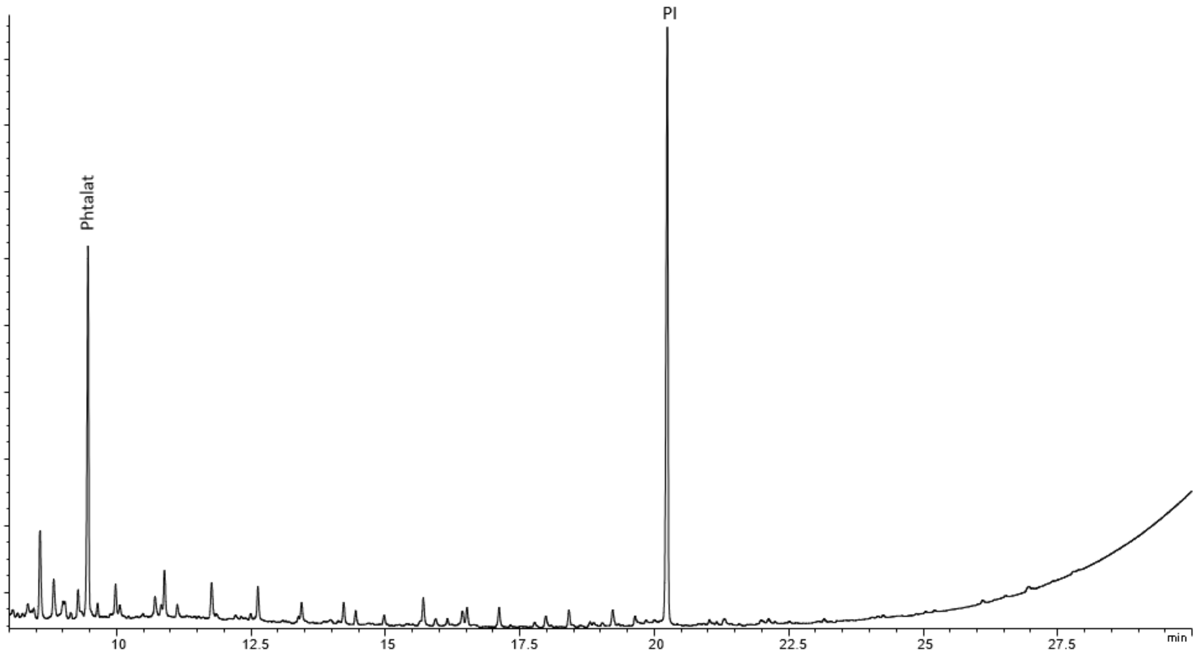
ID Lab	MIG21		
ID Sample	M84	1	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.53	-25.67	
TLE (μg·g^{-1})	408.36		
Interpretation	Non ruminant adipose fat		



ID Lab	MIG22	
ID Sample	M90	458
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.85	-25.43
TLE ($\mu g \cdot g^{-1}$)	545.1	
Interpretation	Non ruminant adipose fat	

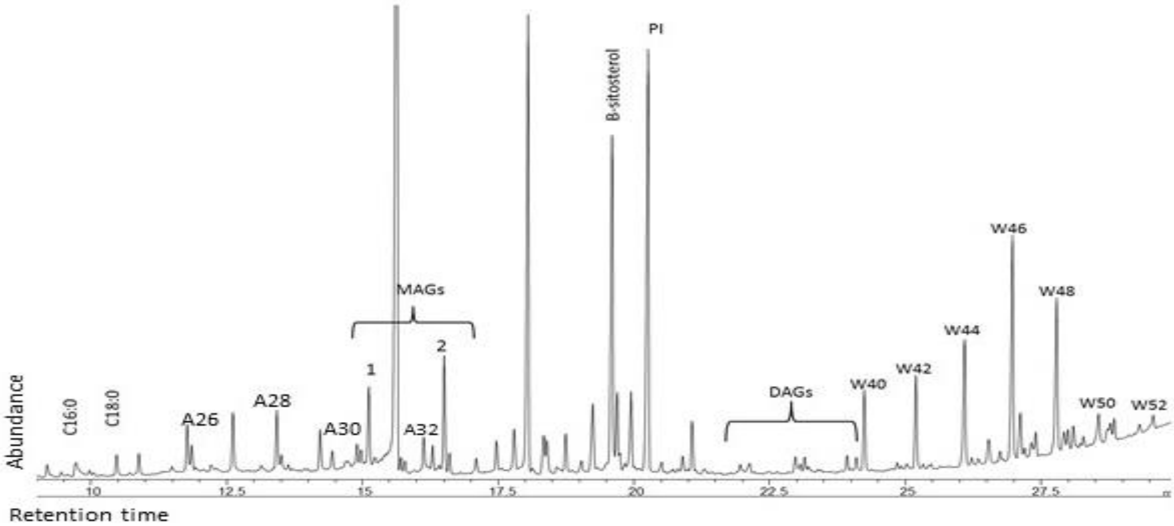


ID Lab	MIG23		
ID Sample	M90	14	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g-1)			
Interpretation	Negligible		

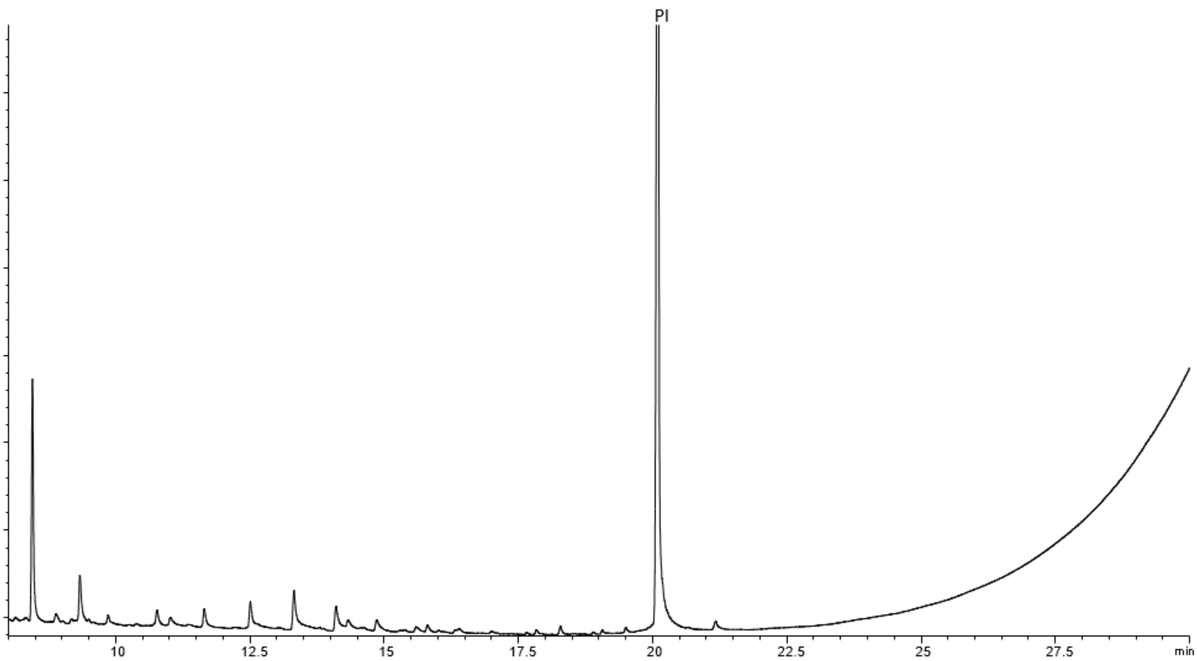


ID Lab	MIG24	
ID Sample	M84	
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	

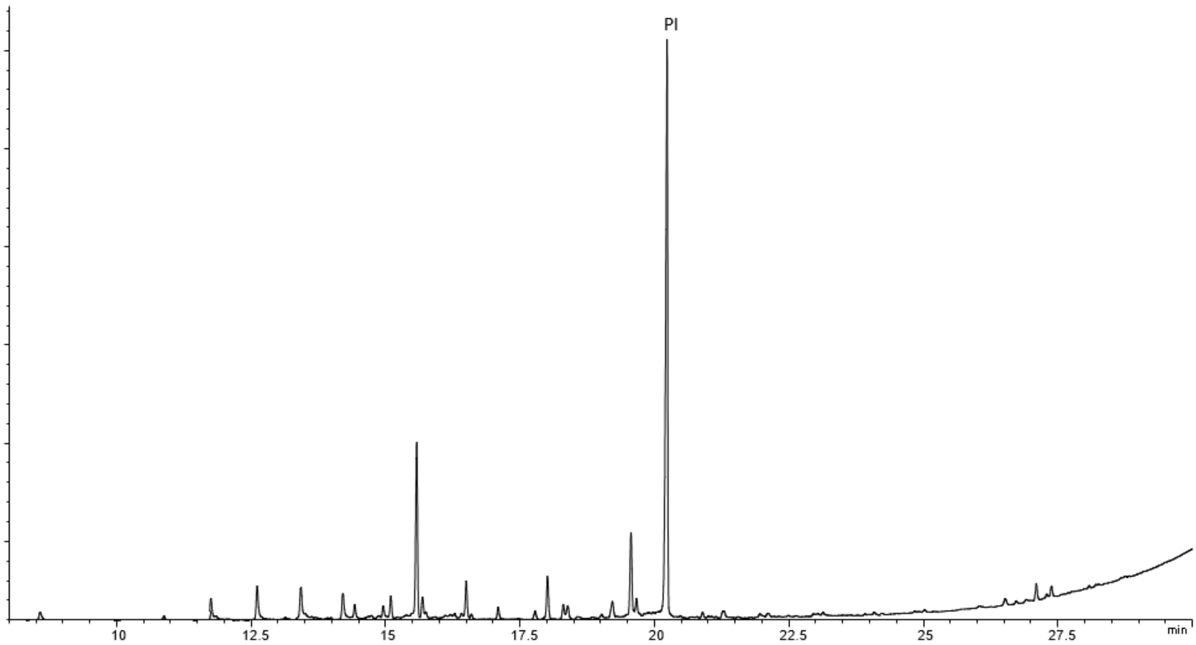
ID Lab	MIG25	
ID Sample	M41	1090-26-NI
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	X	X
TLE (μg·g^{-1})	76.69	
Interpretation	Beeswax Animal fat	



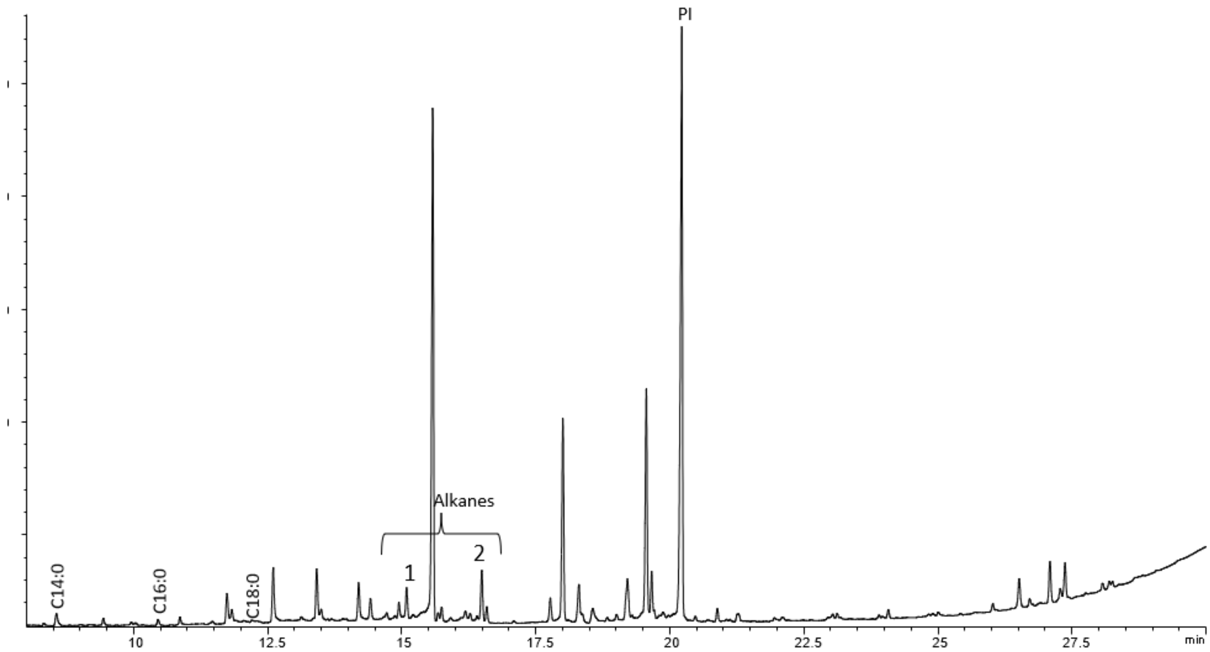
ID Lab	MIG26		
ID Sample	M68	91-2004	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

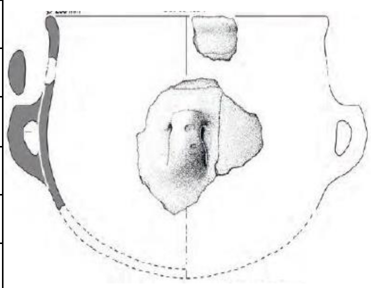


ID Lab	MIG27		
ID Sample	M68	68-19	
Site	Mines de Gavà		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (µg·g^{-1})			
Interpretation	Negligible		

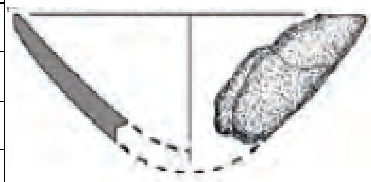


ID Lab	MIG28	
ID Sample	M68	19-2004
Site	Mines de Gavà	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.5	-27.54
TLE ($\mu g \cdot g^{-1}$)	80.2	
Interpretation	Ruminant adipose fat	

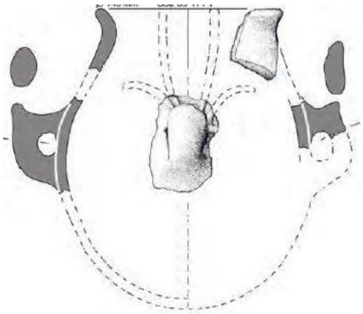


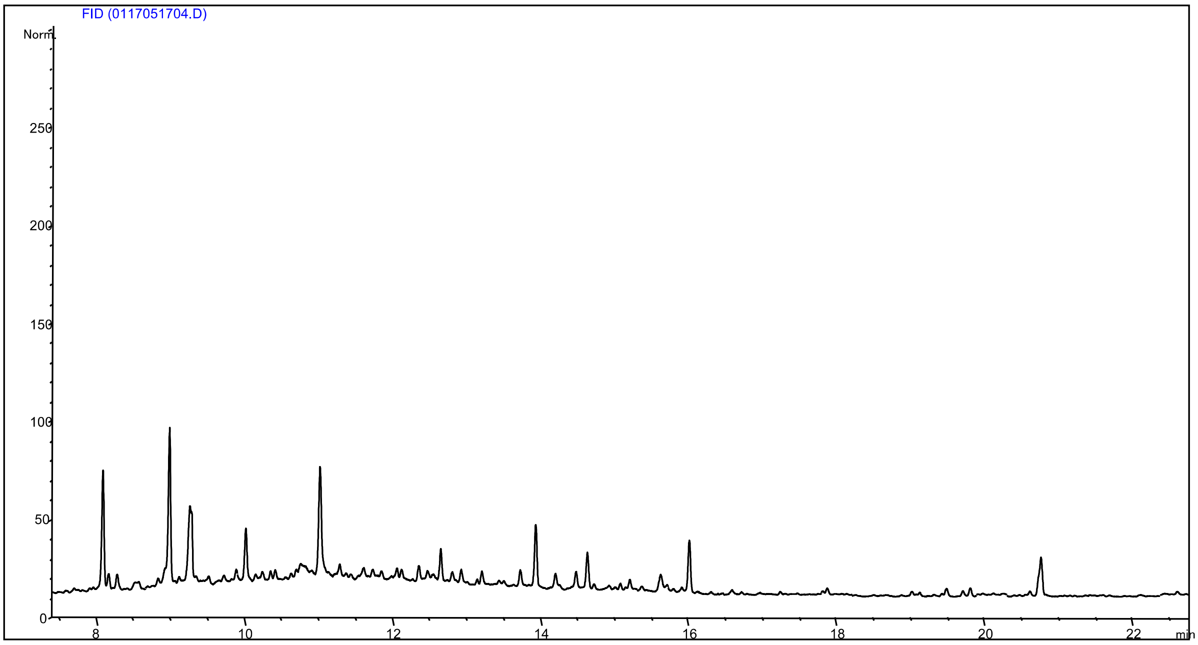
ID Lab	JBA01		
ID Sample	CCJ09	206-SJ1-UE105-100	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)	1.86		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.28	-29.28	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Ruminant adipose fat		

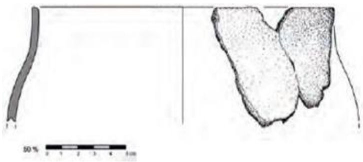
ID Lab	JBA02	
ID Sample	CCJ09	128-1
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)	0.06	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-23.96	-22.85
TLE (µg·g^{-1})		
Interpretation	Pig adipose fat	

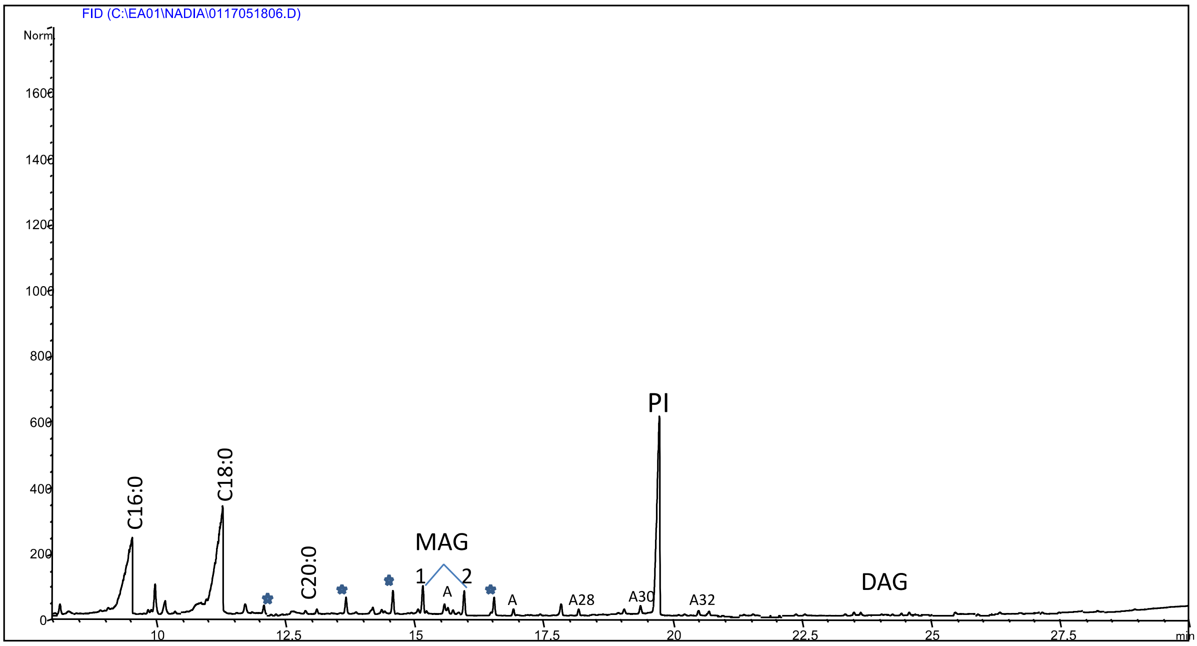


ID Lab	JBA03	
ID Sample	CCJ09	171-1
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)	1.06	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g-1)		
Interpretation	Negligible	

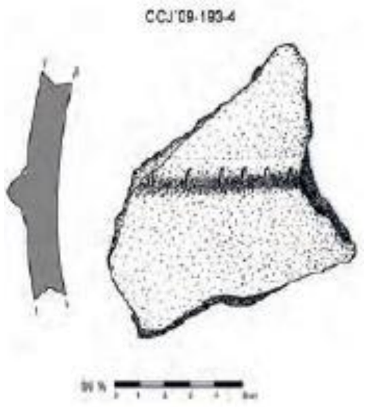




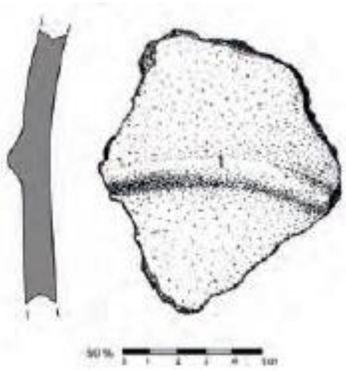
ID Lab	JBA04		
ID Sample	CCJ09	205-ZONA2-SJ08-UE128-3	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.73	-32.62	
TLE (μg·g-1)			
Interpretation	Dairy product		

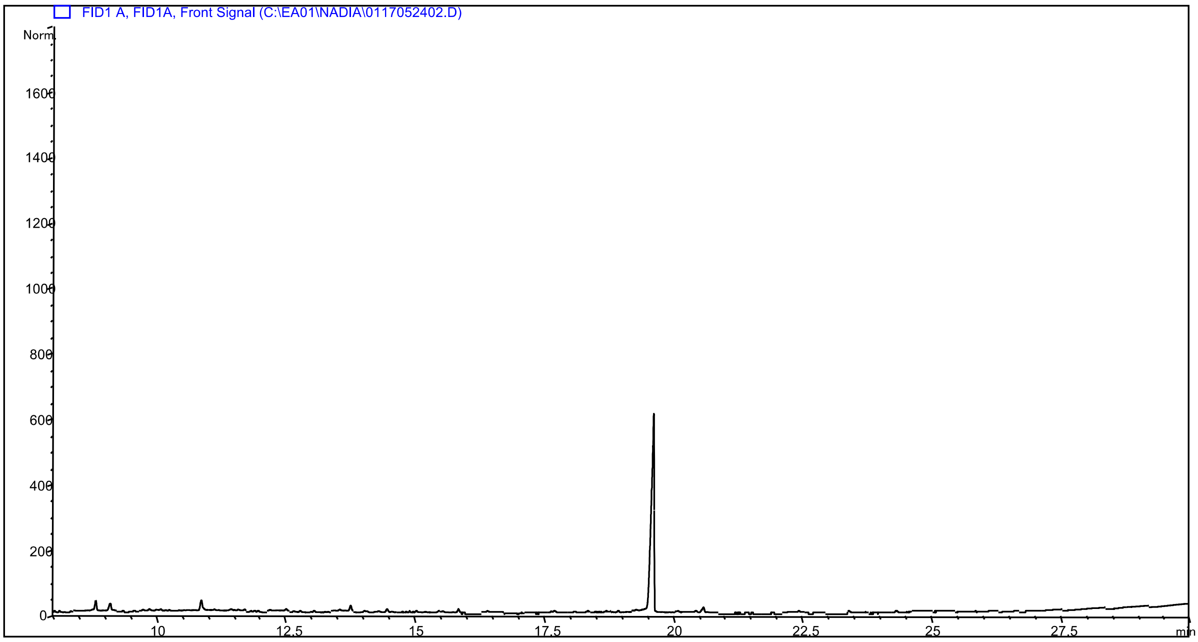


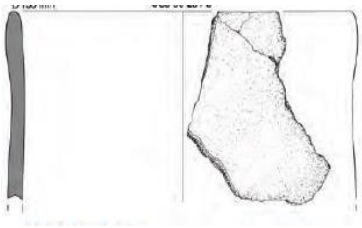
ID Lab	JBA05	
ID Sample	CCJ09	193-4
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	



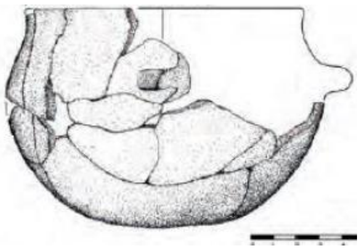
ID Lab	JBA06		
ID Sample	CCJ09	171-3	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})			
Interpretation	Negligible		

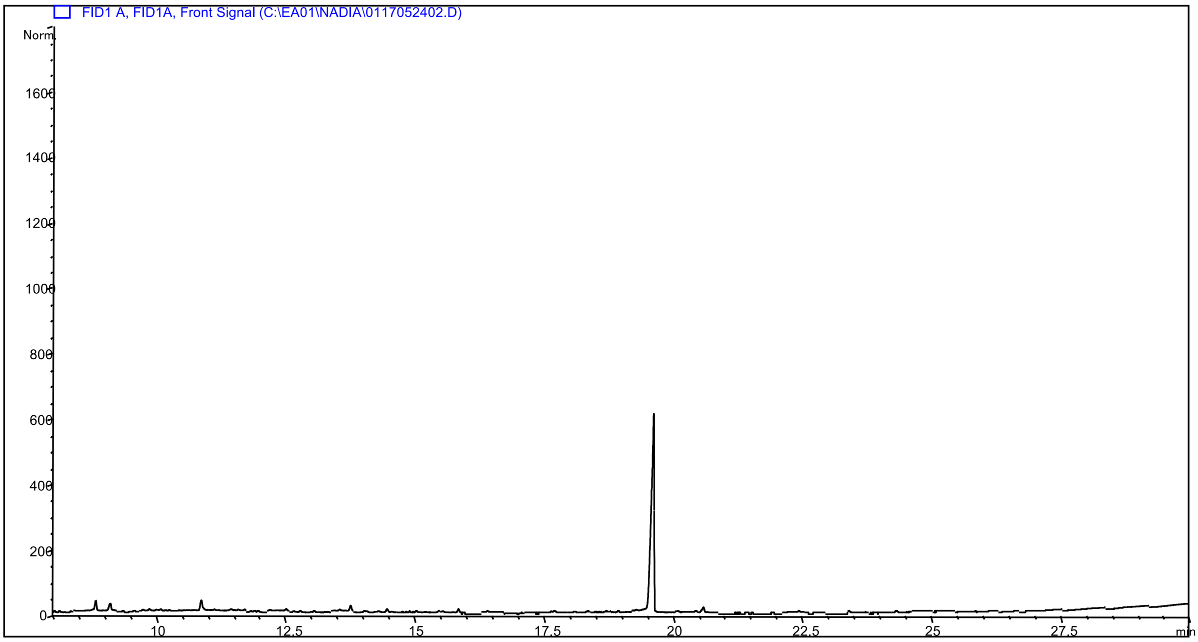




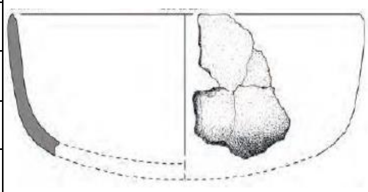
ID Lab	JBA07		
ID Sample	CCJ09	206-FS39-UE231-3	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

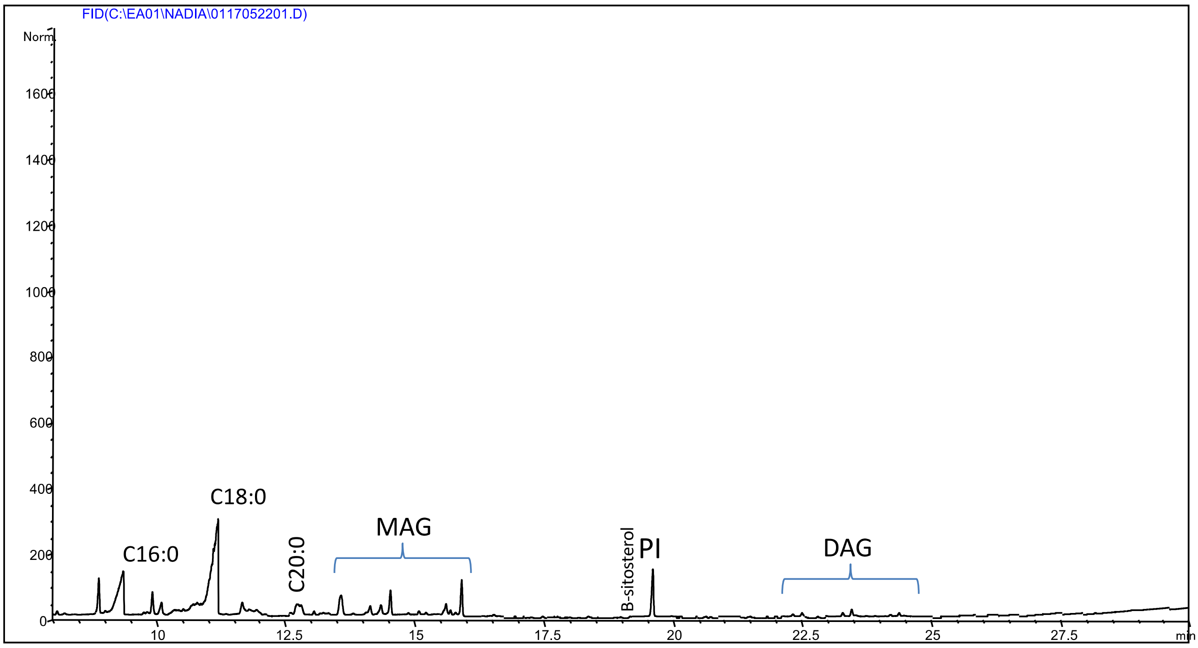
ID Lab	JBA09	
ID Sample	CCJ09	205-537-SJ07-UE127-2537
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)	0.30	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (μg·g^{-1})		
Interpretation	Negligible	



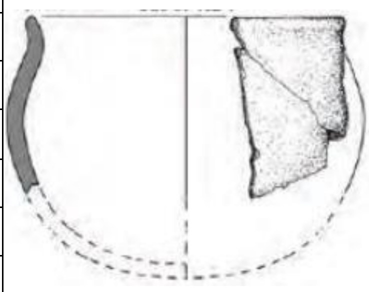


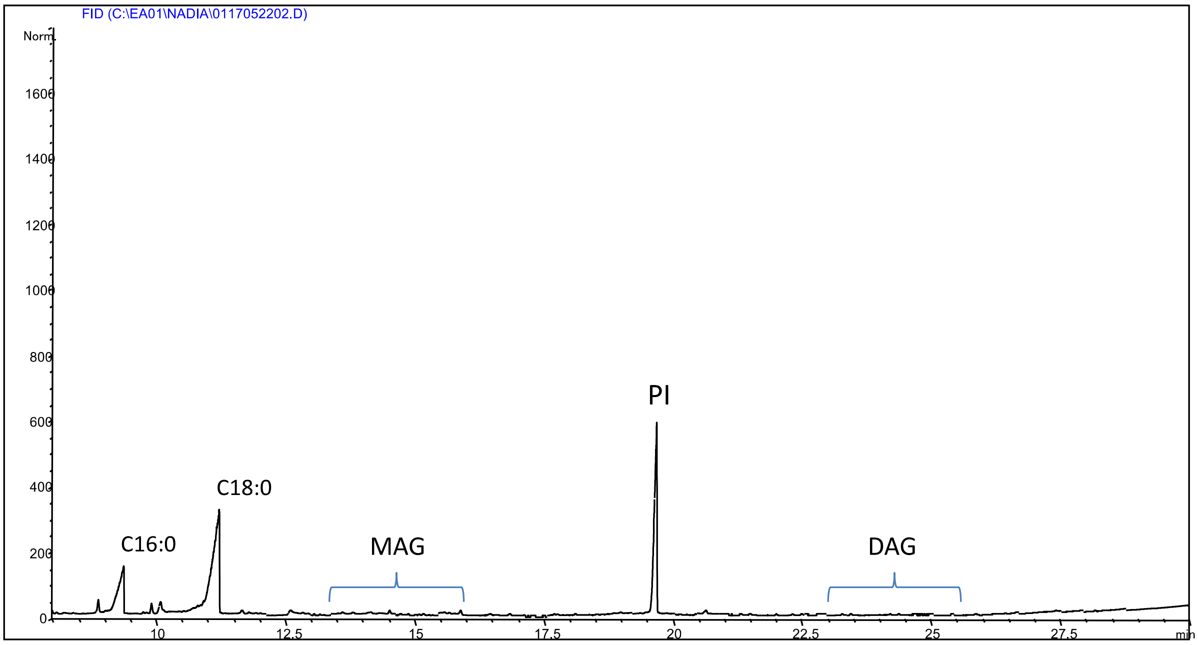
ID Lab	JBA10	
ID Sample	CCJ09	206-FS39-231-1
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-32.34	-35.18
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Dairy product	

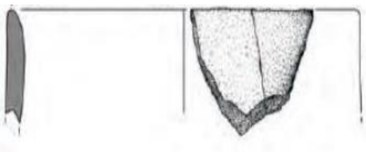


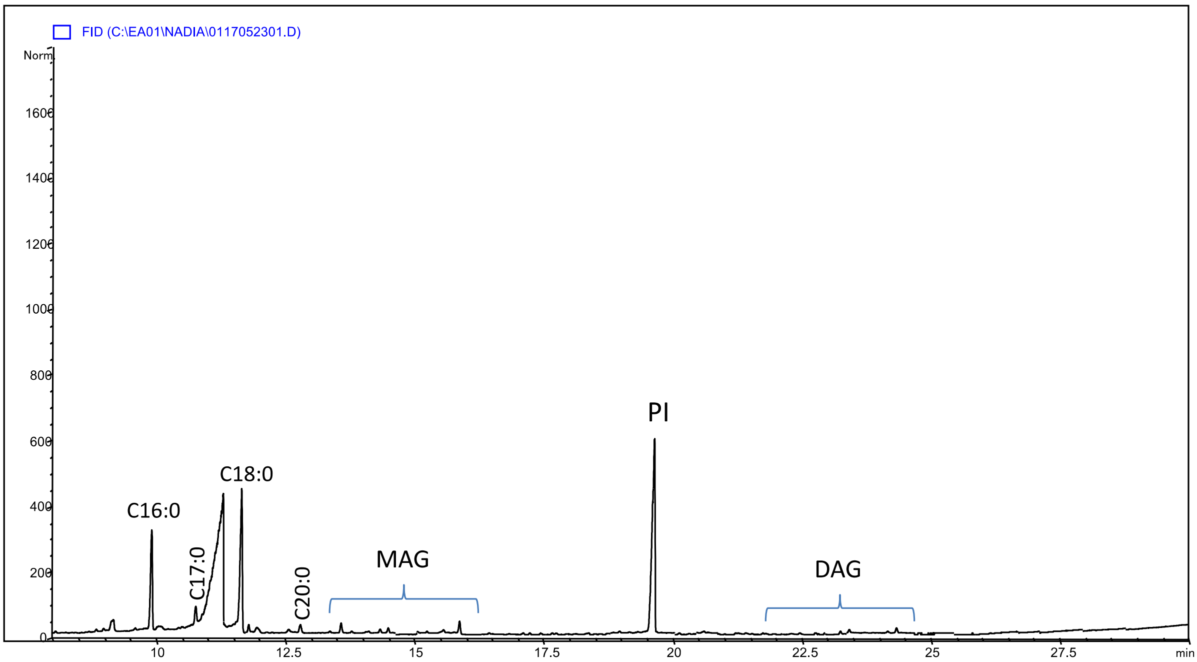


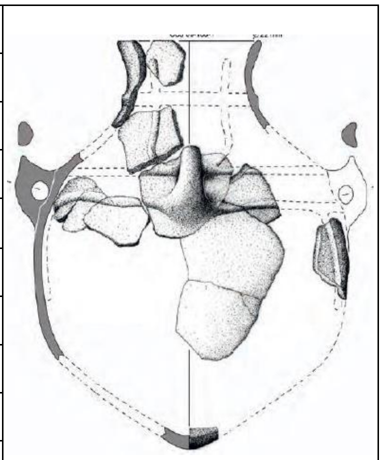
ID Lab	JBA11	
ID Sample	CCJ09	132-1
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)	0.27	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-29.58	-31.84
TLE (µg·g-1)		
Interpretation	Wild ruminant adipose fat	

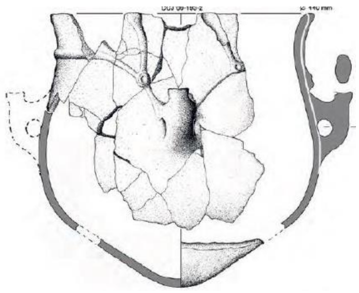




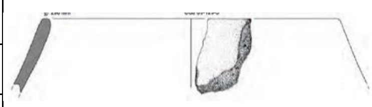
ID Lab	JBA13		
ID Sample	CCJ09	UE127-2	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.16	-24.07	
TLE (μg·g-1)			
Interpretation	Pig adipose fat		

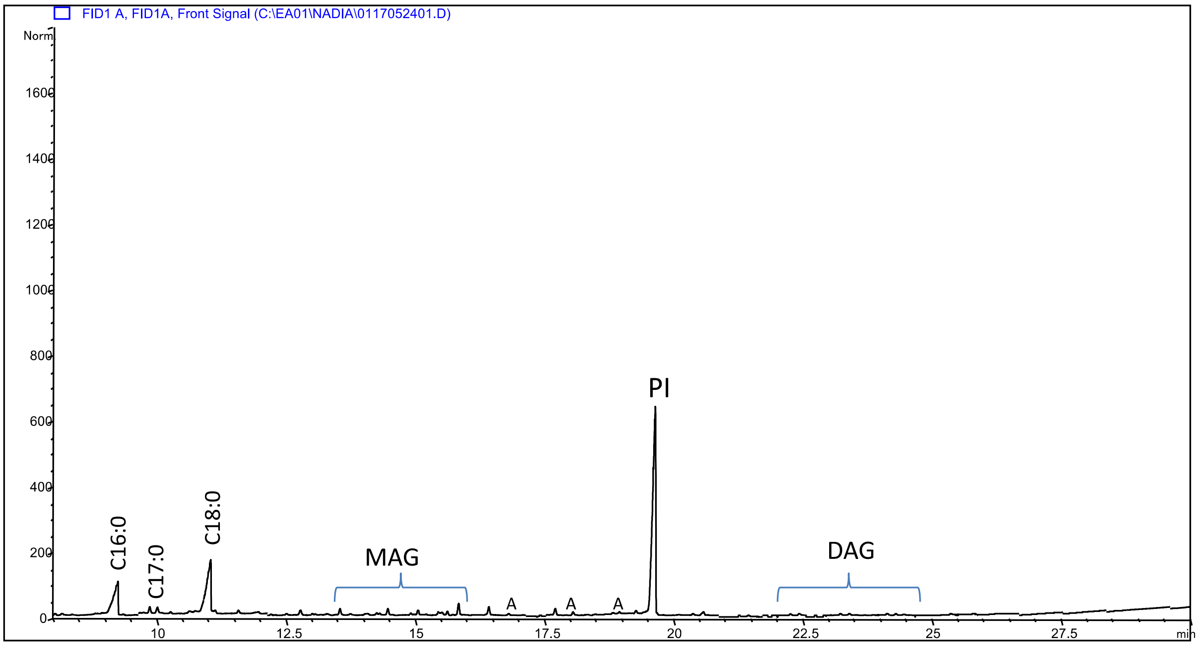


ID Lab	JBA14		
ID Sample	CCJ09	208-106-1-SJ1	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)	6.53		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-21.78	-22.22	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Pig adipose fat		

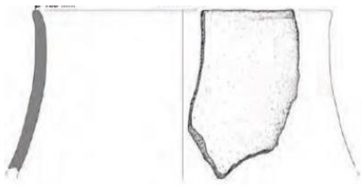
ID Lab	JBA17		
ID Sample	CCJ09	207-SJ24-193-2	
Site	Camp del Colomer		
Morphology			
Handles			
Volume (L)	5.21		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

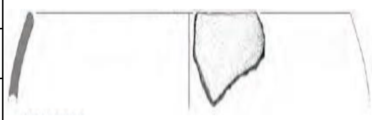
ID Lab	JBA28	
ID Sample	CCJ09	ZONA2-SJ08-UE129-3
Site	Camp del Colomer	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-27.17	-25.94
TLE (μg·g^{-1})		
Interpretation	Wild boar adipose fat	



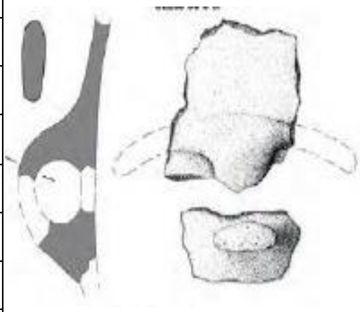


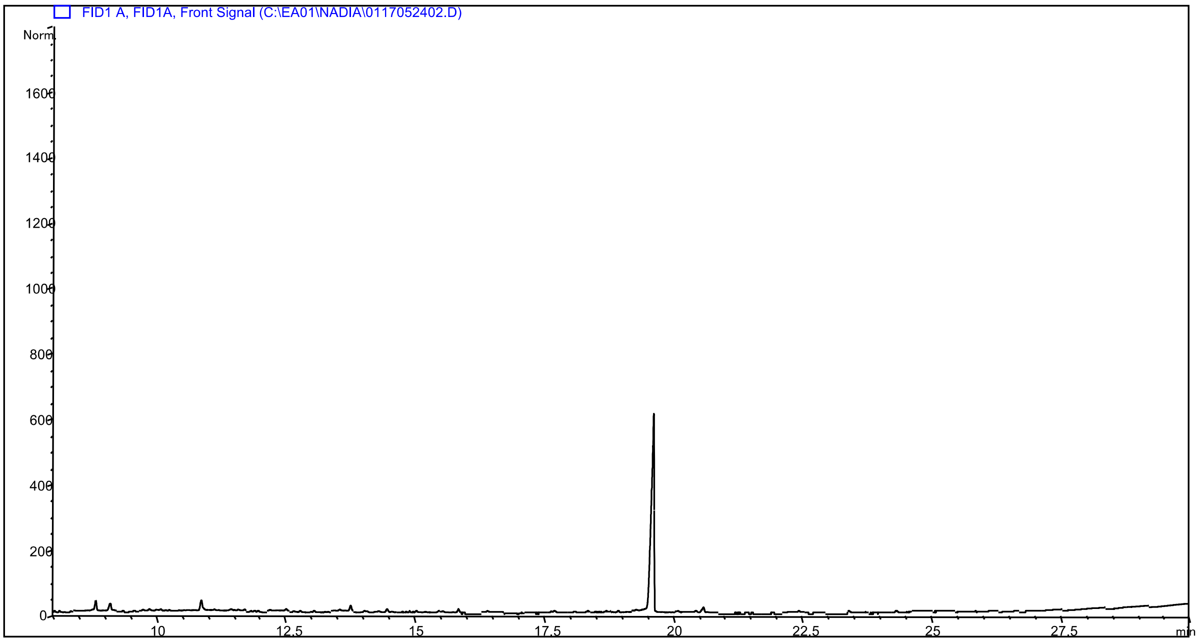
ID Lab	JBA08	
ID Sample	CL28J09	60-4-3
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)	0.49	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	



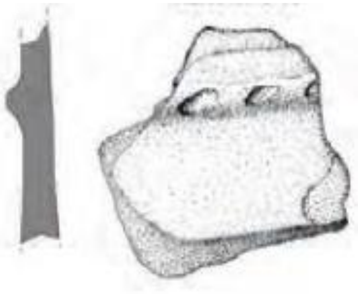
ID Lab	JBA12		
ID Sample	CL28J09	60-8-6	
Site	Carrer Llinàs 28		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-30.75	-31.75	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Ruminant adipose fat		

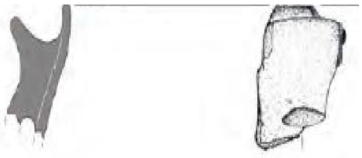
ID Lab	JBA16	
ID Sample	CL28J09	8-37
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Negligible	



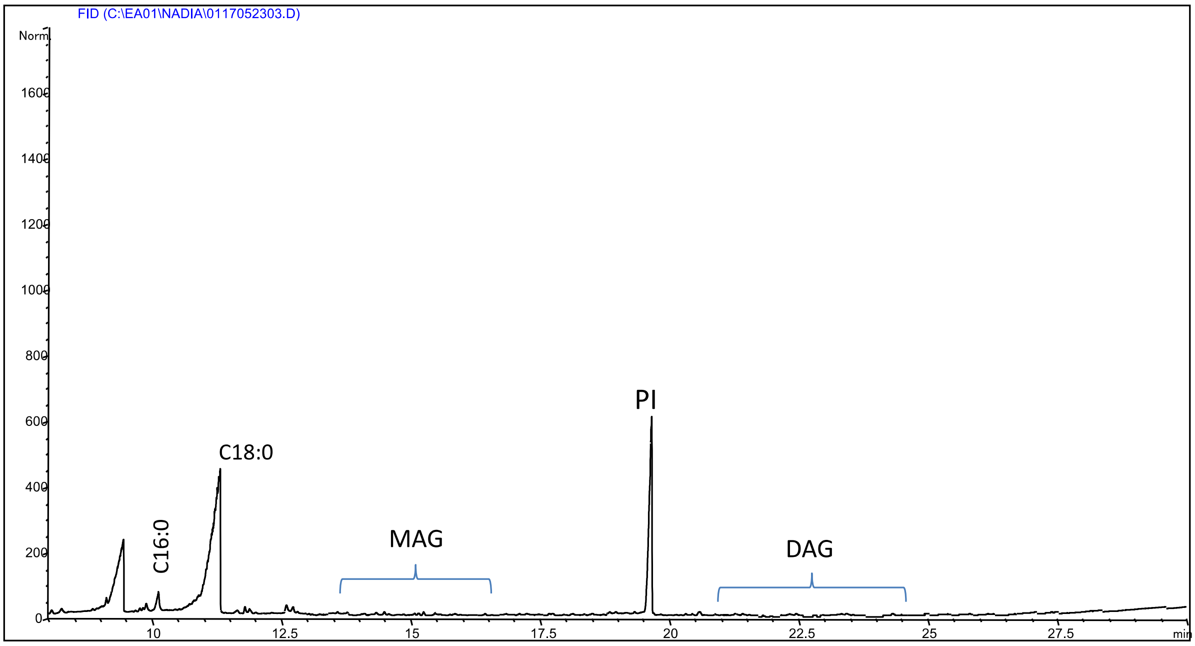


ID Lab	JBA19	
ID Sample	CL28J09	58-ARQ178-UE8-42
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.29	-24.45
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Pig adipose fat	

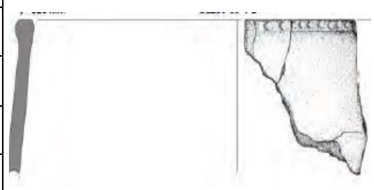


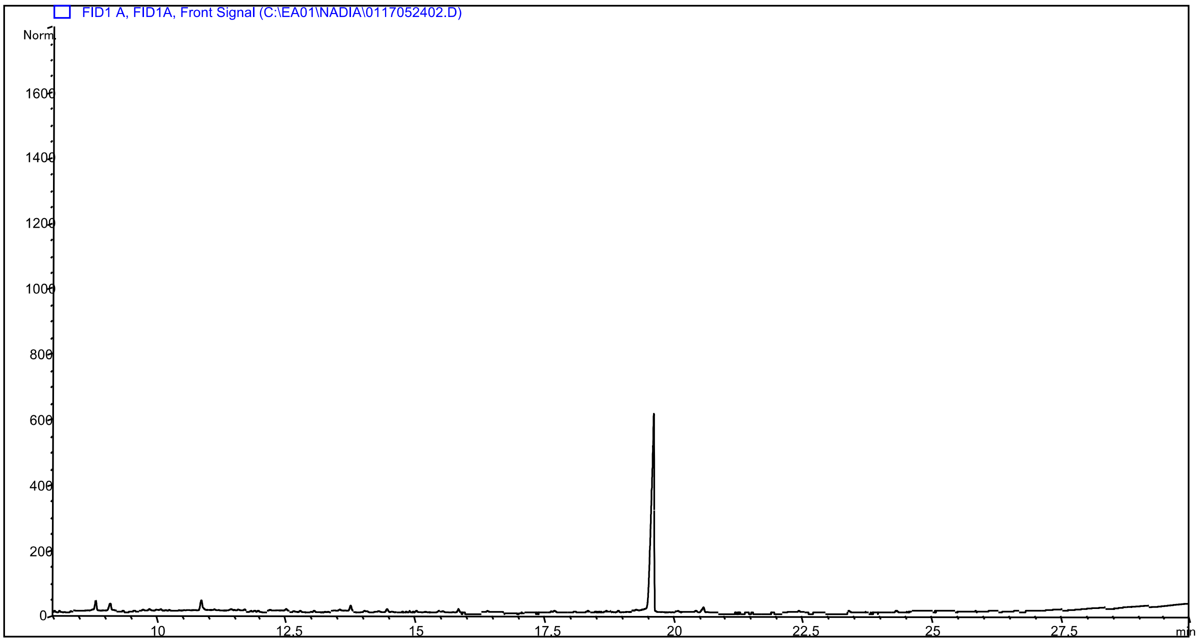
ID Lab	JBA20		
ID Sample	CL28J09	58-ARQ178-UE10-1	
Site	Carrer Llinàs 28		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	JBA21	
ID Sample	CL28J09	60-8-7
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-29.31	-29.99
TLE (μg·g-1)		
Interpretation	Ruminant adipose fat	

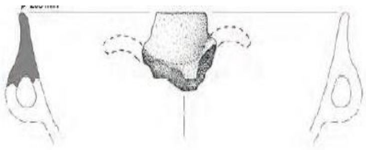


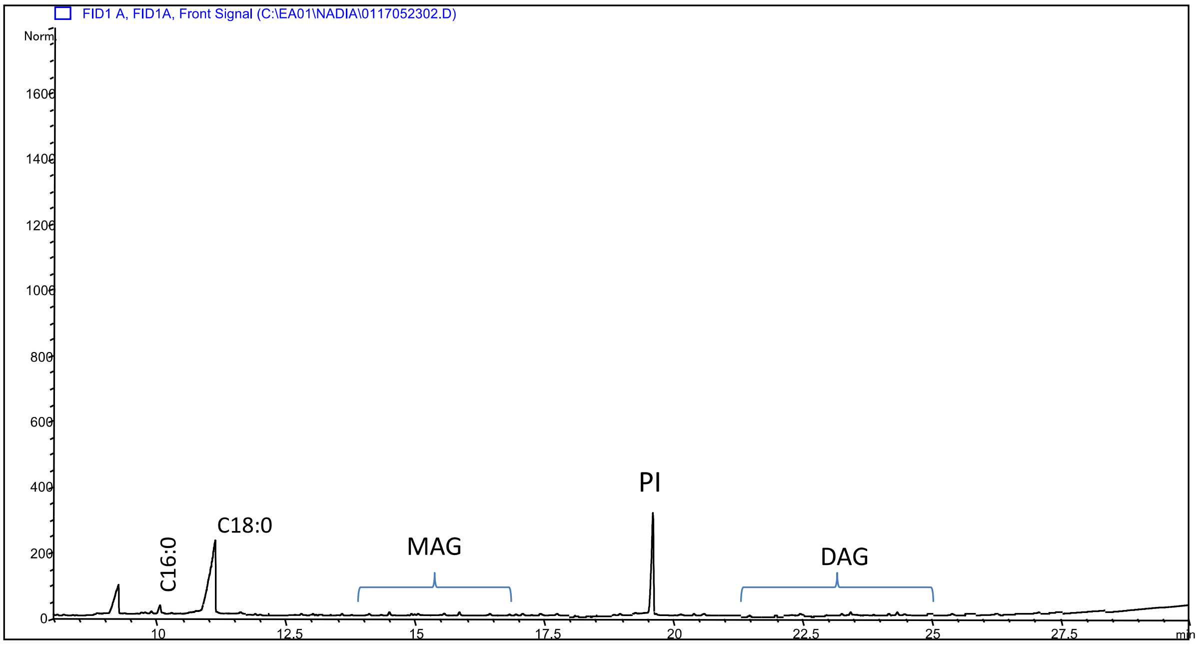
ID Lab	JBA22	
ID Sample	CL28J09	58-ARQ181-UE4-2
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (μg·g-1)		
Interpretation	Negligible	



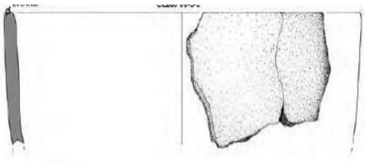


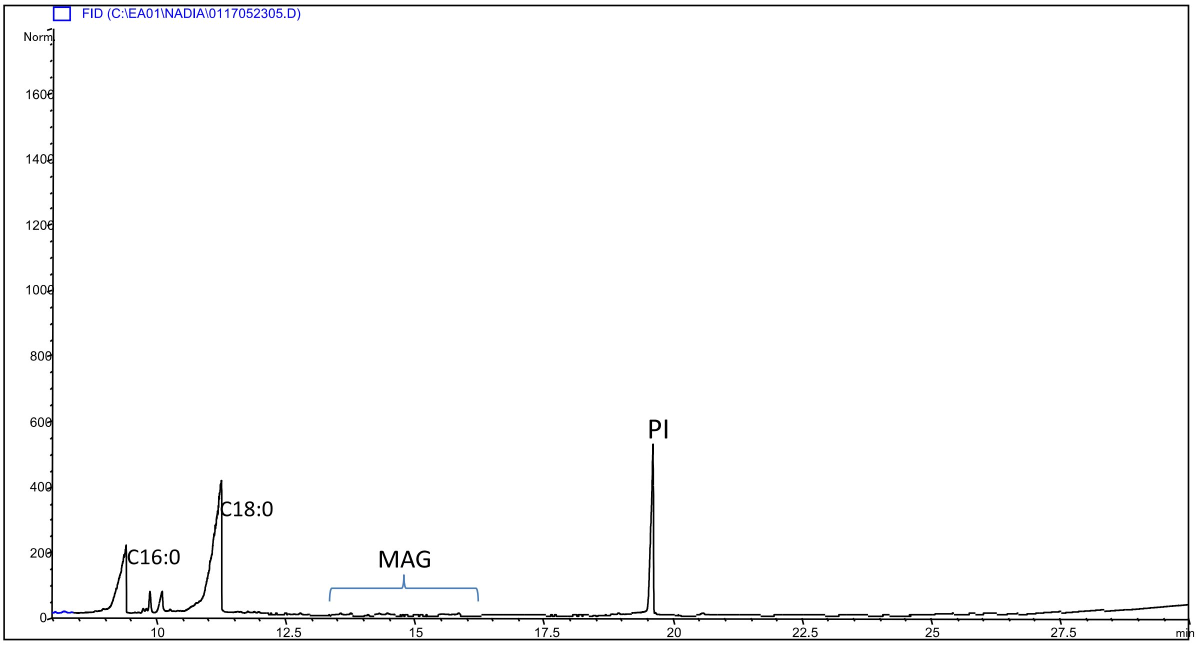
ID Lab	JBA23	
ID Sample	CL28J09	60-8-2
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-28.5	-30.61
TLE (μg·g-1)		
Interpretation	Ruminant adipose fat	



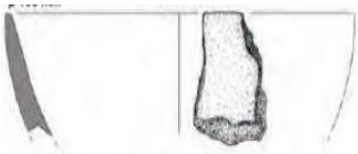


ID Lab	JBA26	
ID Sample	CL28J09	8-3
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.08	-26.82
TLE (µg·g^{-1})		
Interpretation	Dairy product	

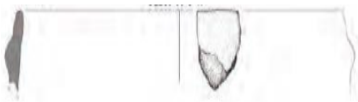


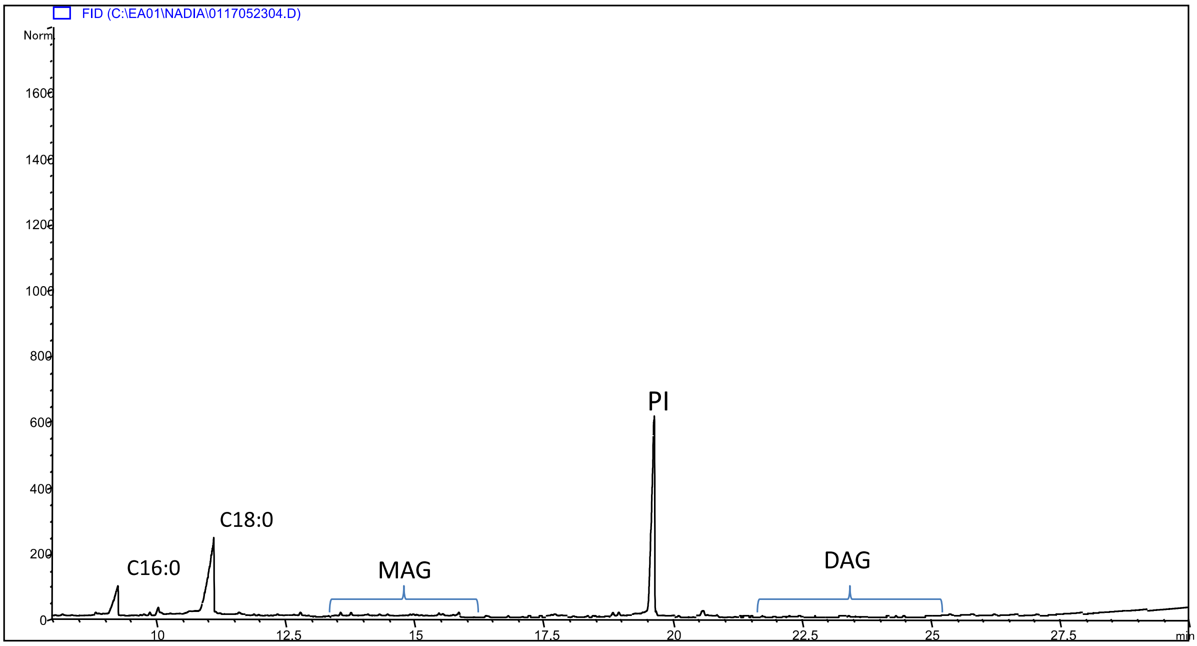


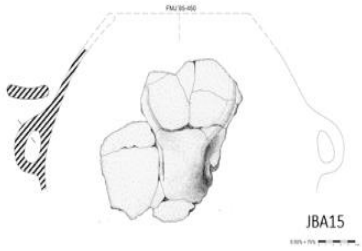
ID Lab	JBA27	
ID Sample	CL28J09	8-12
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-23.13	-22.74
TLE (µg·g^{-1})		
Interpretation	Pig adipose fat	



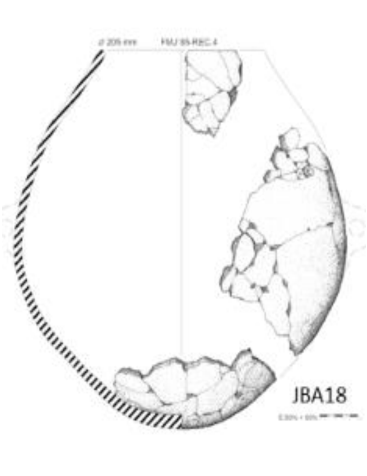
ID Lab	JBA29	
ID Sample	CL28J09	8-17
Site	Carrer Llinàs 28	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-28.46	-30.22
TLE (µg·g^{-1})		
Interpretation	Ruminant adipose fat	

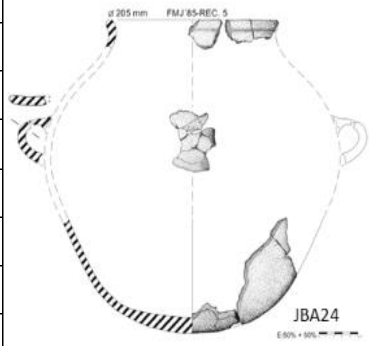




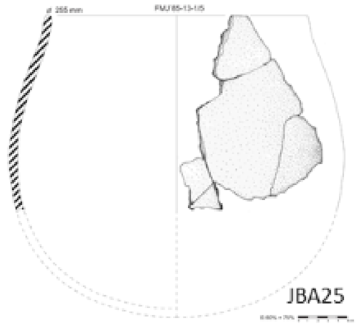
ID Lab	JBA15		
ID Sample	FMJ85	65-450	
Site	Feixa del Moro		
Morphology			
Handles			
Volume (L)	2.20		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	JBA18	
ID Sample	FMJ85	65-REC4
Site	Feixa del Moro	
Morphology		
Handles		
Volume (L)	3.78	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-21.03	-22.83
TLE ($\mu g \cdot g^{-1}$)		
Interpretation	Ruminant adipose fat	

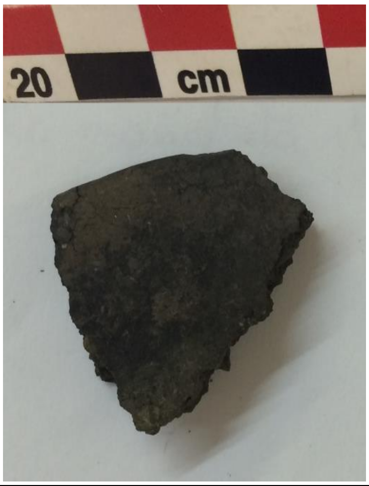


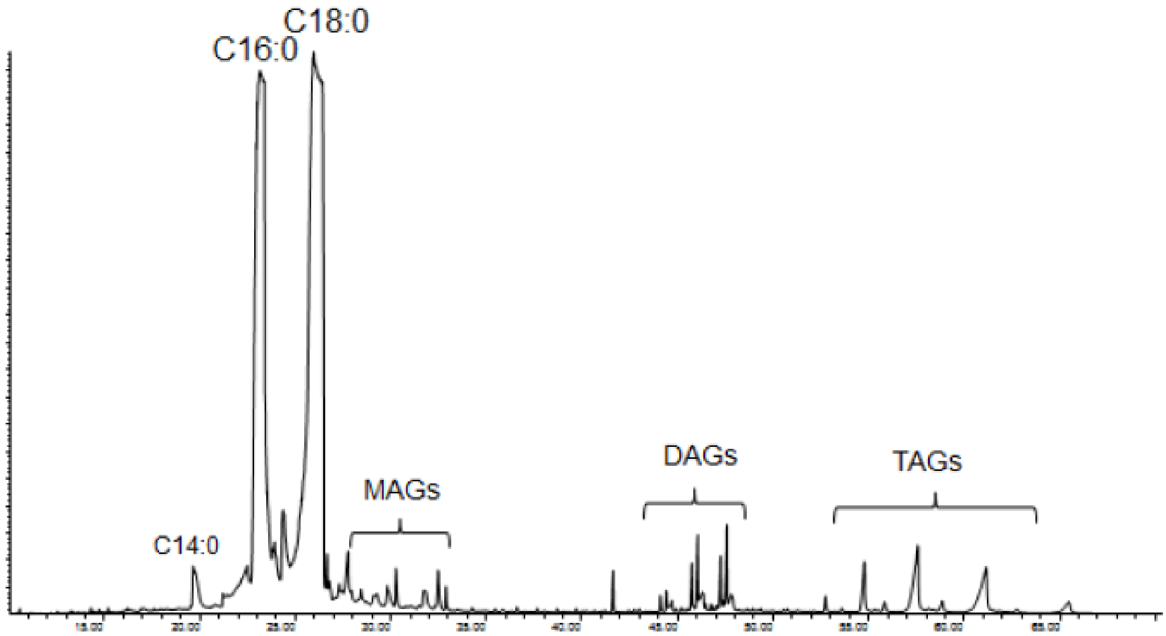
ID Lab	JBA24		
ID Sample	FMJ85	65-REC5	
Site	Feixa del Moro		
Morphology			
Handles			
Volume (L)	2.81		
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-22.09	-21.76	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Pig adipose fat		

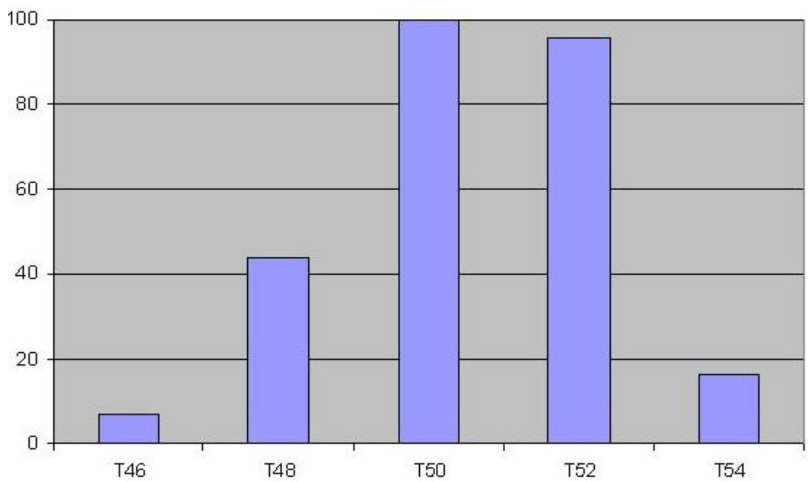
ID Lab	JBA25	
ID Sample	FMJ85	13-5-171c
Site	Feixa del Moro	
Morphology		
Handles		
Volume (L)	2.18	
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.66	-27.17
TLE (μg·g^{-1})		
Interpretation	Ruminant adipose fat	



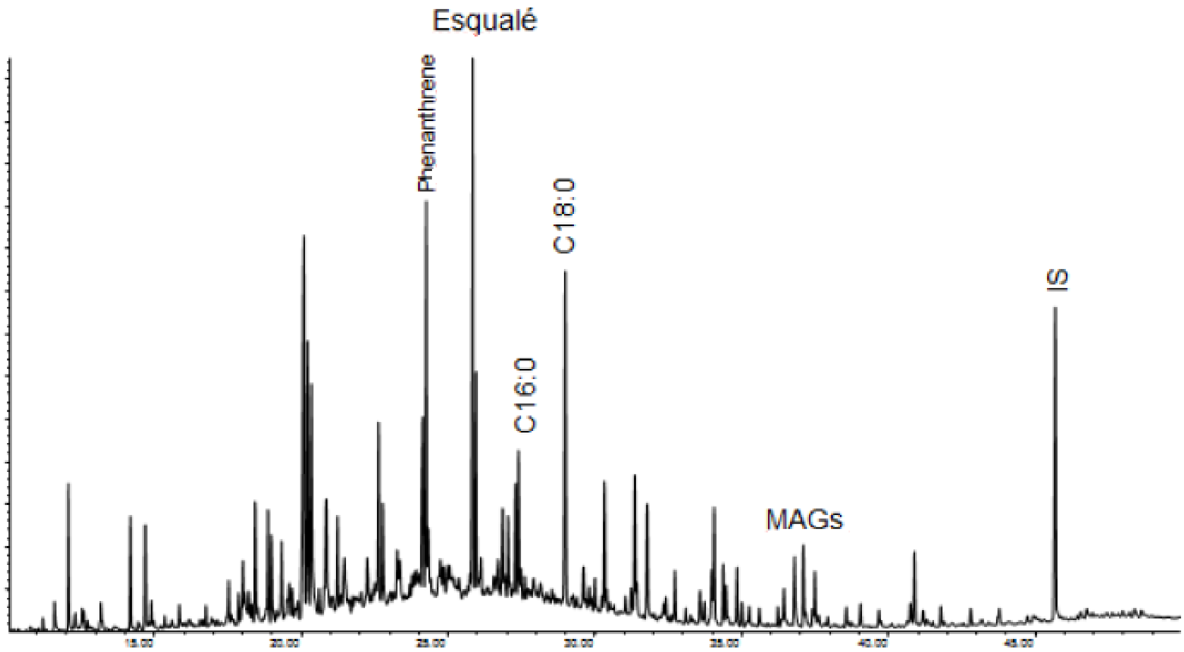
ID Lab	SAR02	
ID Sample	A8	VB14/999
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-25.26	-27.22
TLE (μg·g^{-1})	1794.27	
Interpretation	Ruminant adipose fat	



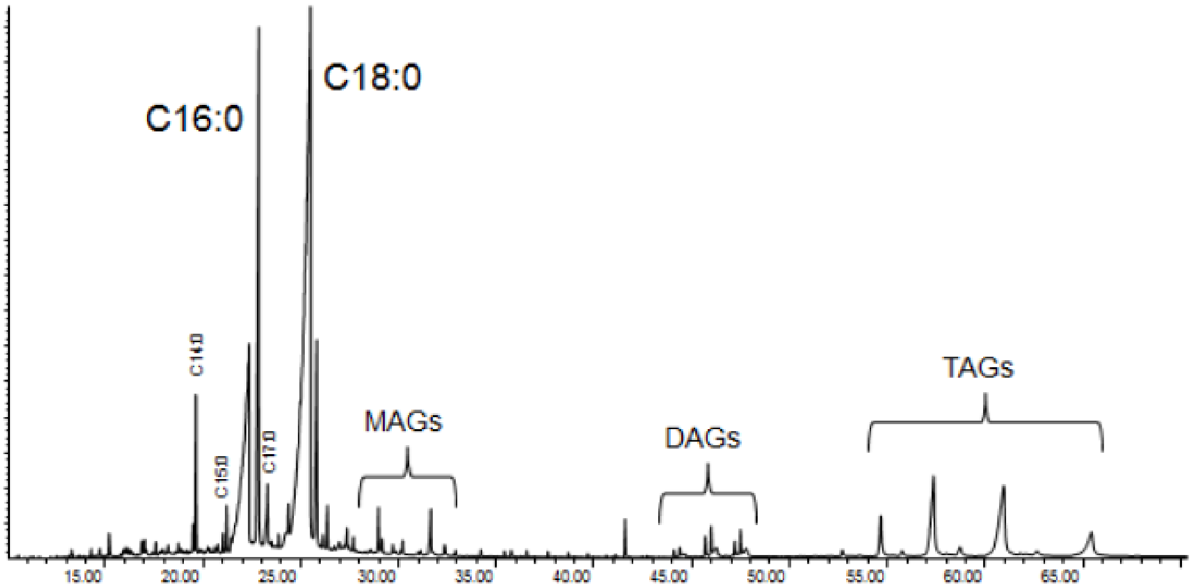


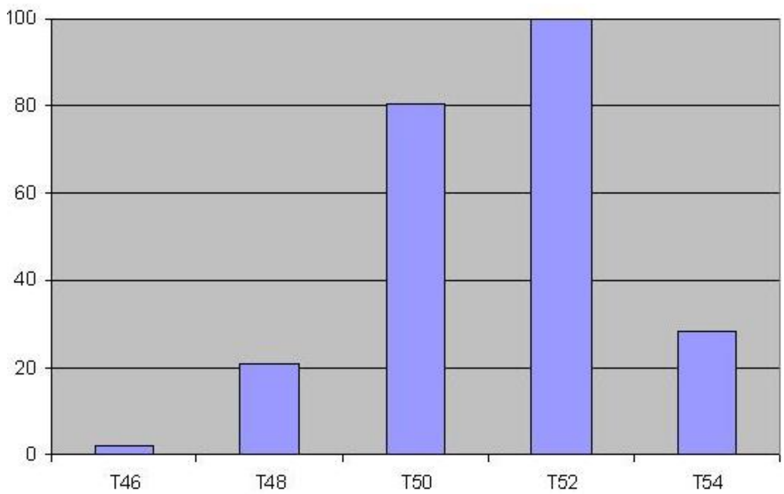


ID Lab	SAR03	
ID Sample	A7	VB14/3324
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.36	-28.91
TLE ($\mu g \cdot g^{-1}$)	447.7	
Interpretation	Ruminant adipose fat	

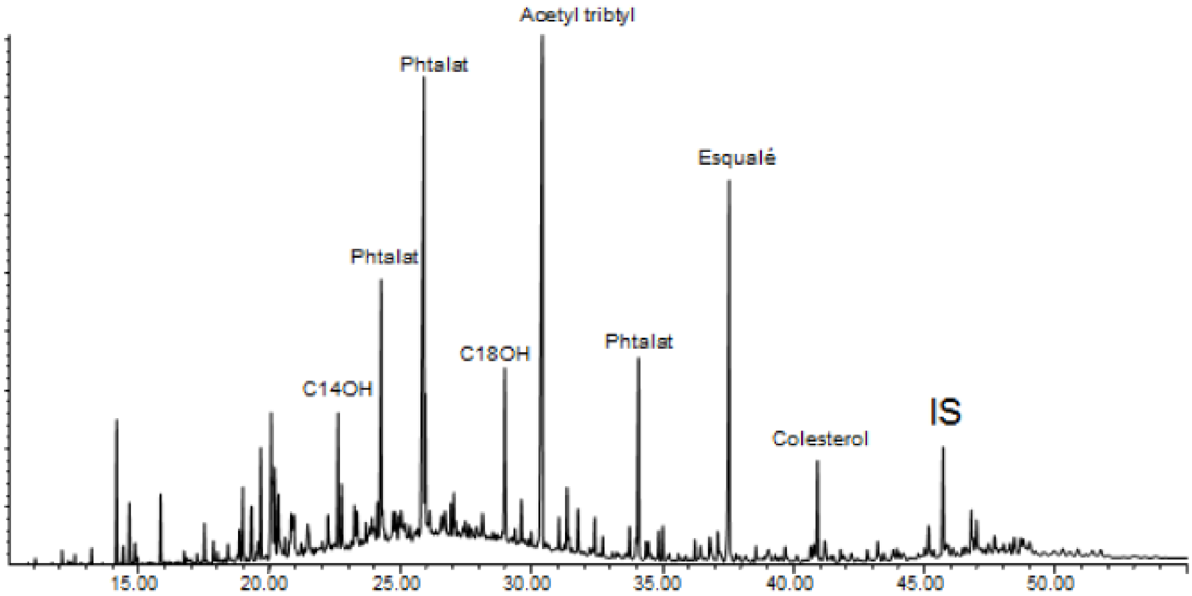


ID Lab	SAR04	
ID Sample	A5	VB14/719
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.96	-26.13
TLE (μg·g-1)	899.23	
Interpretation	Ruminant adipose fat	

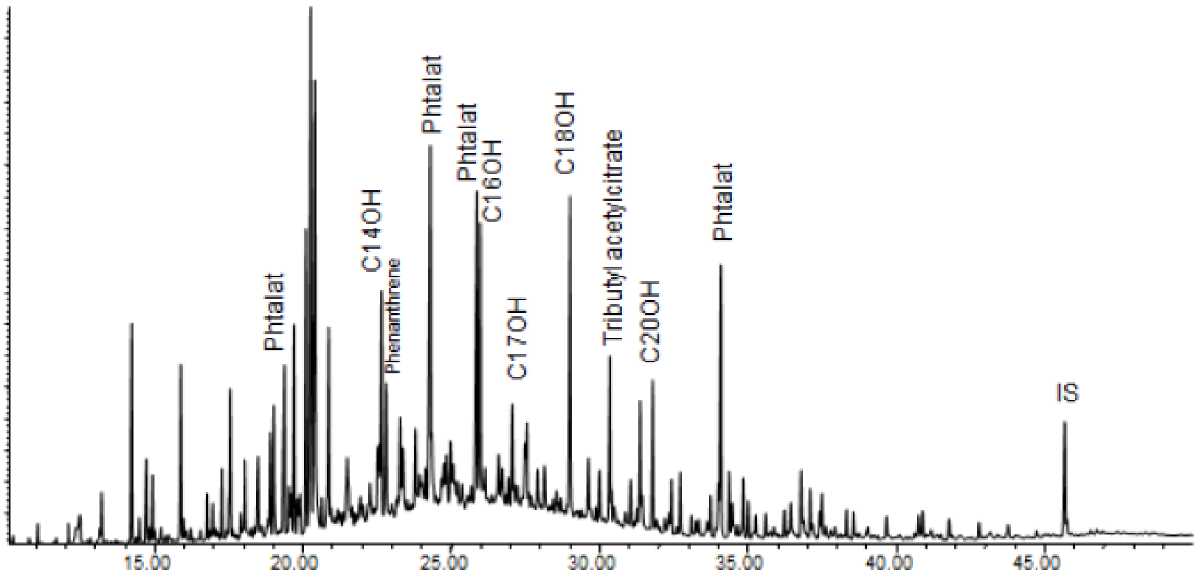




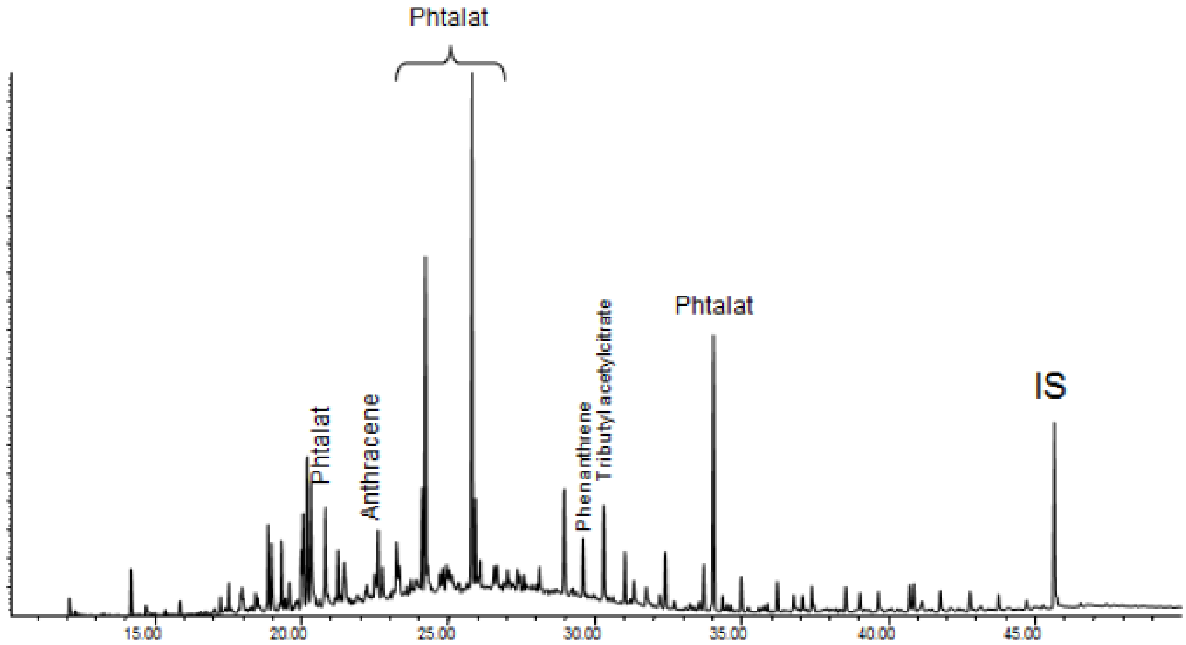
ID Lab	SAR05		
ID Sample	A7	VB14/3324.1	
Site	Cova del Sardo		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (μg·g^{-1})	52.63		
Interpretation	Negligible		



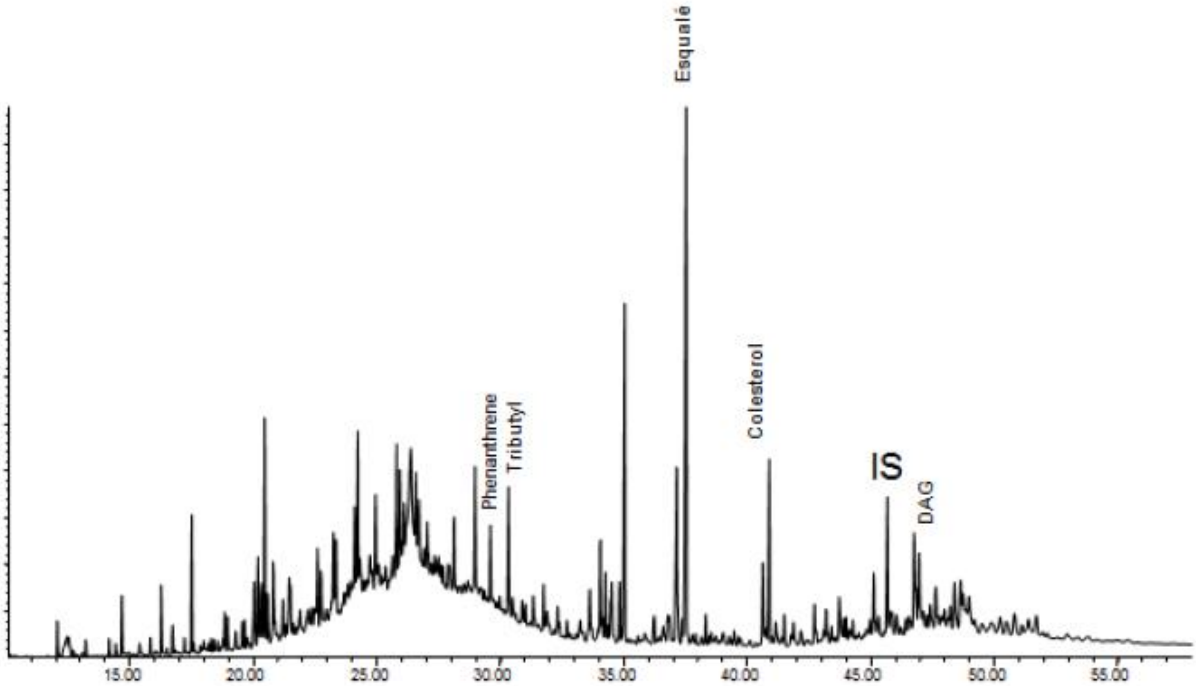
ID Lab	SAR06	
ID Sample	A7	VB14/747
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)	132.74	
Interpretation	Negligible	



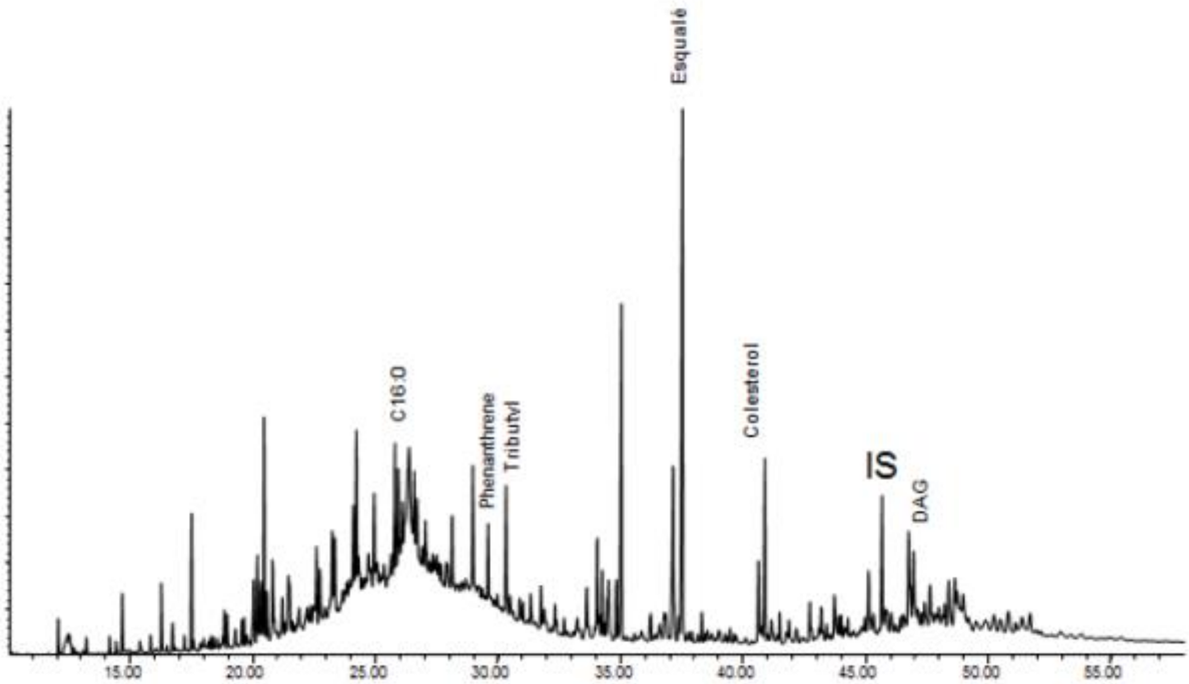
ID Lab	SAR08	
ID Sample	A5	VB14/630
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE ($\mu g \cdot g^{-1}$)	36.70	
Interpretation	Negligible	



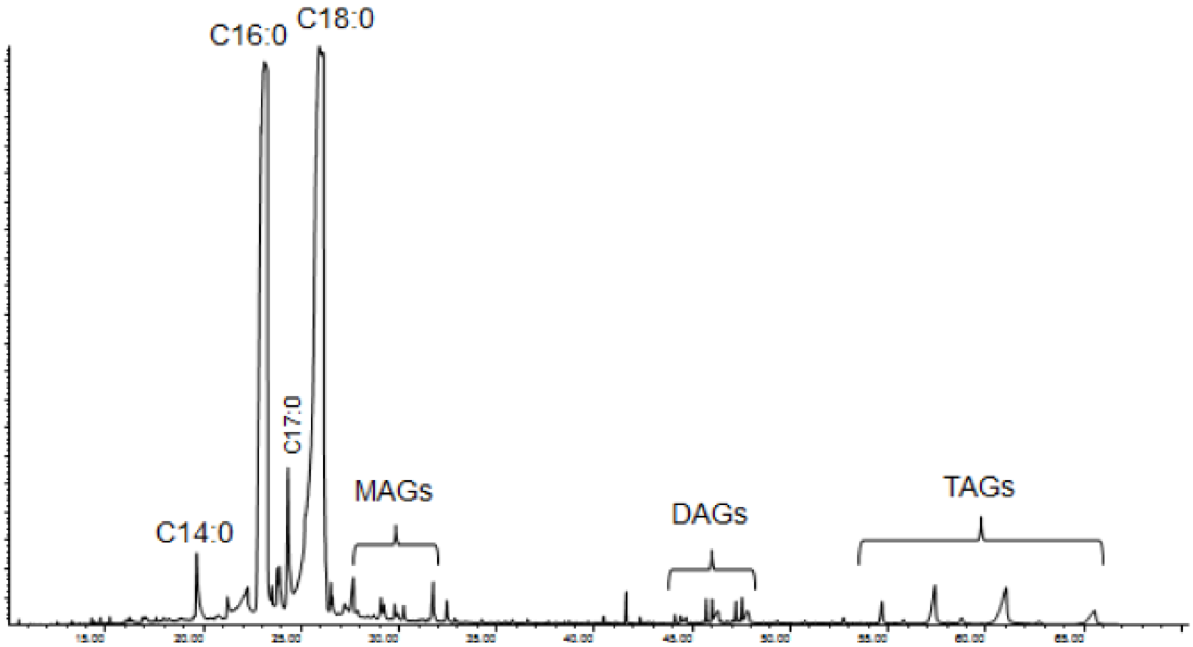
ID Lab	SAR09		
ID Sample	A7	VB14/743.2	
Site	Cova del Sardo		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE (μg·g^{-1})	22.70		
Interpretation	Negligible		

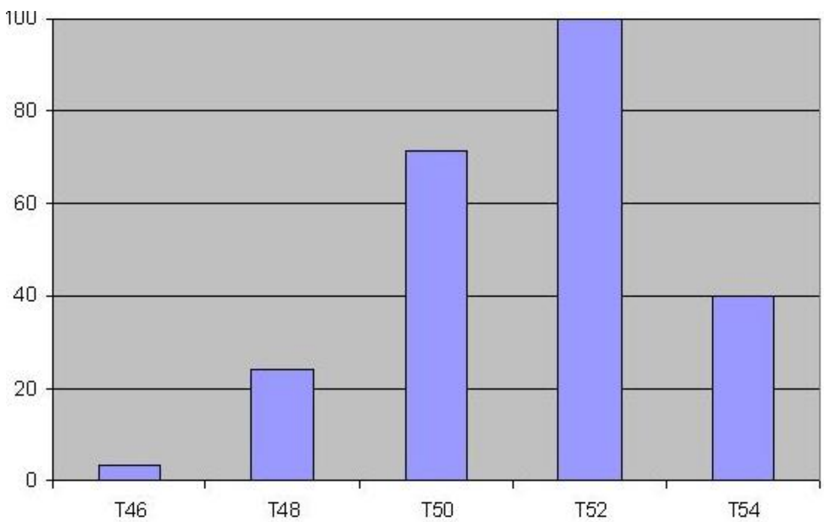


ID Lab	SAR10		
ID Sample	A7	VB14/569	
Site	Cova del Sardo		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]			
TLE ($\mu g \cdot g^{-1}$)	22.50		
Interpretation	Negligible		

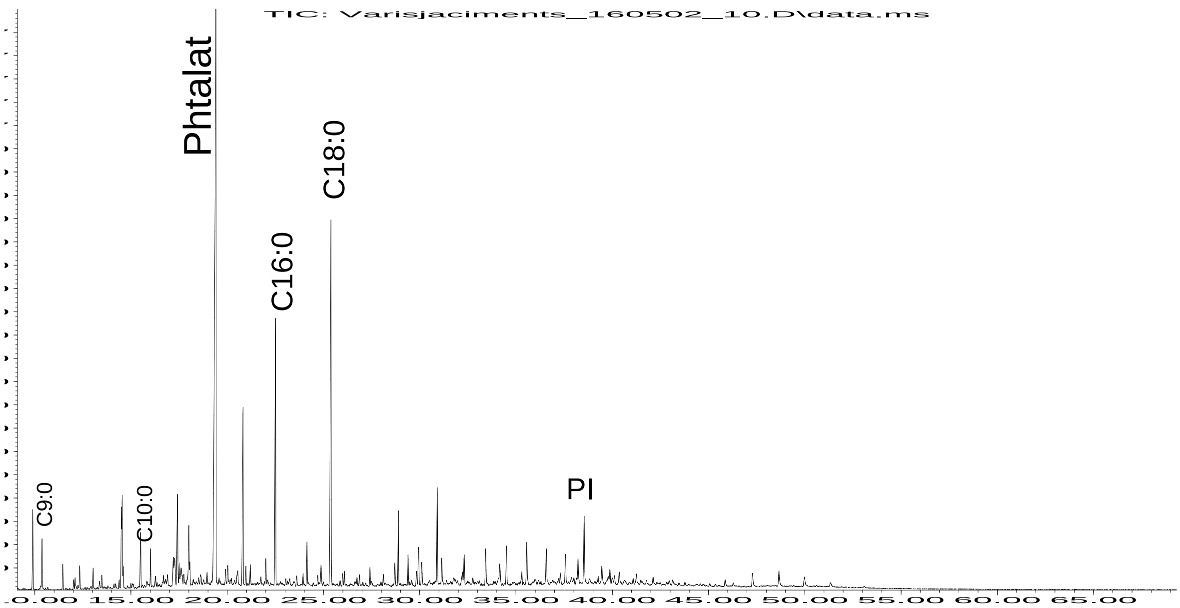


ID Lab	SAR11	
ID Sample	A7	VB14/728
Site	Cova del Sardo	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-24.77	-27.03
TLE (µg·g-1)	2640.96	
Interpretation	Ruminant adipose fat	





ID Lab	CNP01		
ID Sample	5	7/261	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.54	-27.45	
TLE (µg·g^{-1})	86.9		
Interpretation	Non-ruminant adipose fat		



ID Lab	CNP02		
ID Sample	5	445/1	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.89	-27.18	
TLE ($\mu g \cdot g^{-1}$)	51.3		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP03		
ID Sample	10	628	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g{\cdot}g^{-1}$)			
Interpretation	Negligible		

<table>
<tr><td>ID Lab</td><td colspan="2">CNP04</td><td rowspan="5">
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<tr><td>ID Sample</td><td>10</td><td>256</td></tr>
<tr><td>Site</td><td colspan="2">Cabecicos Negros</td></tr>
<tr><td>Morphology</td><td colspan="2"></td></tr>
<tr><td>Handles</td><td colspan="2"></td></tr>
<tr><td>Volume (L)</td><td colspan="2"></td><td rowspan="5">
</td></tr>
<tr><td>Heating biomarkers</td><td colspan="2"></td></tr>
<tr><td>δ13C values [C16:0 / C18:0]</td><td>-</td><td>-</td></tr>
<tr><td>TLE ($\mu g \cdot g^{-1}$)</td><td colspan="2"></td></tr>
<tr><td>Interpretation</td><td colspan="2">Negligible</td></tr>
</table>

ID Lab	CNP05		
ID Sample	10	80	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CNP06		
ID Sample	8	219	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.47	-25.66	
TLE ($\mu g \cdot g^{-1}$)	91.4		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP07		
ID Sample	8	131	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g{\cdot}g^{-1}$)			
Interpretation	Negligible		

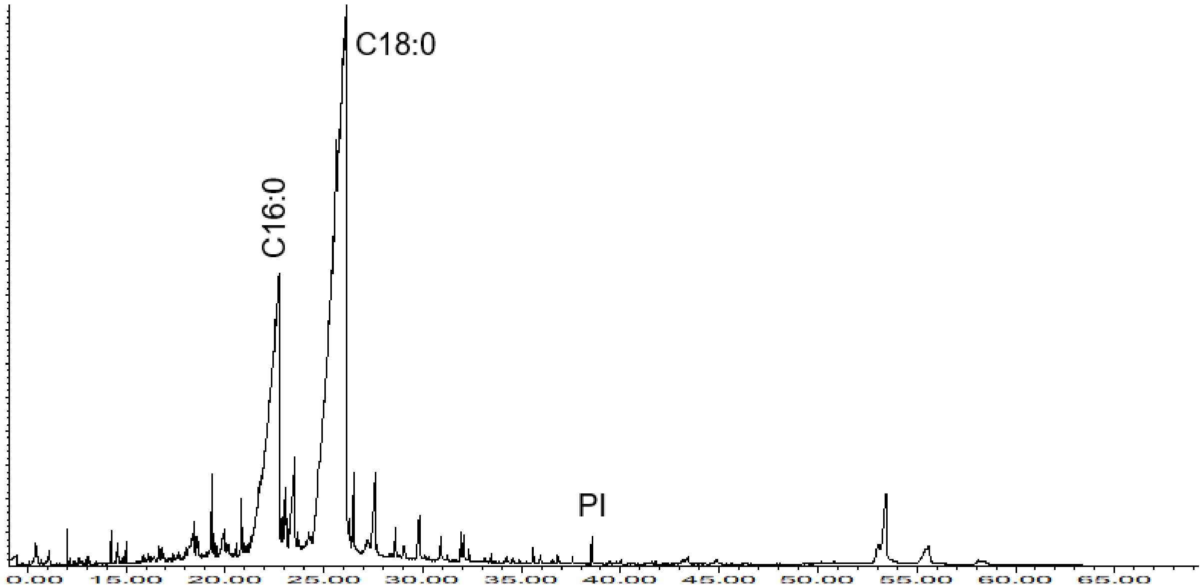
ID Lab	CNP08		
ID Sample	14	736	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CNP09		IMAGE NOT AVAILABLE
ID Sample	14	779	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE ($\mu g \cdot g^{-1}$)			
Interpretation	Negligible		

ID Lab	CNP10		
ID Sample	9	196	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-	-	
TLE (μg·g^{-1})			
Interpretation	Negligible		

ID Lab	CNP11	
ID Sample	9	231/1
Site	Cabecicos Negros	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (μg·g^{-1})		
Interpretation	Negligible	

ID Lab	CNP12	
ID Sample	14	538
Site	Cabecicos Negros	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-26.84	-31.92
TLE ($\mu g \cdot g^{-1}$)	896.65	
Interpretation	Dairy product	



ID Lab	CNP13		
ID Sample	8	332	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.09	-25.14	
TLE ($\mu g \cdot g^{-1}$)	65.2		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP14		
ID Sample	10	646	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-24.32	-24.65	
TLE (µg·g^{-1})	66.8		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP15		
ID Sample	14	427	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.65	-25.42	
TLE ($\mu g \cdot g^{-1}$)	87.1		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP16		
ID Sample	14	236	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.23	-24.60	
TLE ($\mu g \cdot g^{-1}$)	95.4		
Interpretation	Non-ruminant adipose fat		

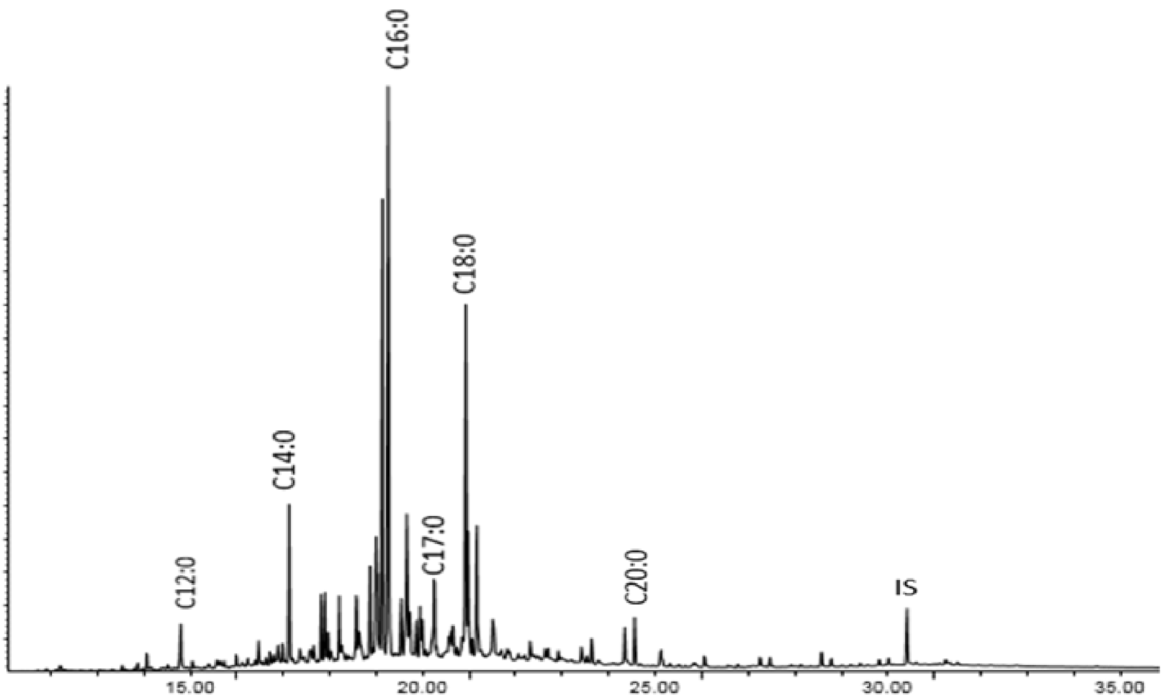
ID Lab	CNP17		
ID Sample	8	471	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-23.88	-22.78	
TLE ($\mu g \cdot g^{-1}$)	69.3		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP18		
ID Sample	5	570	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.33	-27.81	
TLE ($\mu g \cdot g^{-1}$)	88.9		
Interpretation	Ruminant adipose fat		

ID Lab	CNP19		
ID Sample	14	8	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.72	-25.53	
TLE ($\mu g \cdot g^{-1}$)	45.3		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP20	
ID Sample	14	855
Site	Cabecicos Negros	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]	-	-
TLE (µg·g^{-1})		
Interpretation	Negligible	

ID Lab	CNP21		IMAGE NOT AVAILABLE
ID Sample	8	19/1	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			IMAGE NOT AVAILABLE
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-28.37	-25.25	
TLE (μg·g⁻¹)	132.45		
Interpretation	Non-ruminant adipose fat		



ID Lab	CNP22		
ID Sample	10	353	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.88	-26.03	
TLE ($\mu g \cdot g^{-1}$)	21.8		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP23		
ID Sample	10	294	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-25.42	-25.71	
TLE (μg·g^{-1})	84.3		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP24		
ID Sample	14	542	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.89	-26.71	
TLE ($\mu g \cdot g^{-1}$)	74.2		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP25	
ID Sample	14	473
Site	Cabecicos Negros	
Morphology		
Handles		
Volume (L)		
Heating biomarkers		
δ13C values [C16:0 / C18:0]		
TLE (µg·g^{-1})		
Interpretation	Negligible	

ID Lab	CNP26		
ID Sample	9	156	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.21	-25.79	
TLE (μg·g^{-1})	39.5		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP27		
ID Sample	5	462	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-26.78	-26.32	
TLE (µg·g^{-1})	18.1		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP28		
ID Sample	9	127	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.54	-25.89	
TLE (µg·g^{-1})	23.6		
Interpretation	Non-ruminant adipose fat		

ID Lab	CNP29		
ID Sample	8	232	
Site	Cabecicos Negros		
Morphology			
Handles			
Volume (L)			
Heating biomarkers			
δ13C values [C16:0 / C18:0]	-27.54	-25.89	
TLE (µg·g^{-1})	49.5		
Interpretation	Non-ruminant adipose fat		