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Historical culinary practices in Southern Brazil: an organic residue approach

Alice Di Muro

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PhD Thesis

Historical culinary practices in Southern Brazil: an organic residue approach

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Nothing in life is to be feared, it is only to be understood

Maria Skłodowska-Curie

A Ginevra, che sta per aprire gli occhi sul mondo: che la curiosità ti porti a scrivere le pagine piu belle della tua vita

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Abstract

This thesis investigates historical culinary practices in Southern Brazil during the late 19th and early 20th centuries through an integrated bioarchaeological approach of ceramic artefacts, human and faunal materials. Focusing on archaeological sites in the regions of Babitonga Bay (Santa Catarina) and Patos Lagoon (Rio Grande do Sul), the research combines Organic Residue Analysis (ORA) of ceramics and Stable Isotope Analysis (SIA) of human and faunal remains to investigate dietary protein patterns, vessel use and food processing behaviours. These data are contextualised using historical sources, ceramic typology, and zooarchaeological evidence. The results of this thesis reveal distinct patterns in the use of marine and terrestrial resources, shaped by social contexts, local economies, historically documented identities, and access to commodities. ORA results indicate preserved ceramic residue patterns mainly compatible with C_3 plant inputs and terrestrial animal fats, with variable aquatic-compatible evidence, particularly in coastal contexts. SIA indicates regional contrasts in protein sources, suggesting differential access to fish, livestock, and cultivated plants across rural and urban settings. The evidence is consistent with the persistence of some practices rooted in Indigenous and Afro-Brazilian food traditions, alongside the adoption and adaptation of culinary practices associated with European immigration. By integrating molecular and isotopic data with material and documentary evidence, this study contributes to a more nuanced understanding of historical foodways, challenging narratives of cultural replacement and highlighting the complexity of daily domestic life during a period of major social transformation in Southern Brazil. The thesis demonstrates the potential of biomolecular archaeology to recover aspects of the past that are often absent from written records, offering a methodological framework for postcolonial and historical dietary research.

Resumen

Esta tesis investiga las prácticas culinarias históricas en el sur de Brasil durante finales del siglo XIX y principios del siglo XX mediante un enfoque bioarqueológico integrado de artefactos cerámicos, restos humanos y materiales faunísticos. Centrada en yacimientos arqueológicos de las regiones de la bahía de Babitonga (Santa Catarina) y la laguna de Patos (Rio Grande do Sul), la investigación combina el análisis de residuos orgánicos (ORA) en cerámicas y el análisis de isótopos estables (SIA) en restos humanos y faunísticos para investigar los patrones de proteína dietética, el uso de vasijas y los comportamientos relacionados con el procesamiento de alimentos. Estos datos se contextualizan mediante fuentes históricas, tipología cerámica y evidencia zooarqueológica. Los resultados de esta tesis revelan patrones diferenciados en el uso de recursos marinos y terrestres, configurados por los contextos sociales, las economías locales, las identidades históricamente documentadas y el acceso a productos de consumo. Los resultados de ORA indican patrones de residuos cerámicos preservados principalmente compatibles con aportes de plantas C_3 y grasas animales terrestres, con evidencia variable compatible con recursos acuáticos, particularmente en contextos costeros. El SIA indica contrastes regionales en las fuentes de proteína, lo que sugiere un acceso diferenciado al pescado, al ganado y a las plantas cultivadas en entornos rurales y urbanos. La evidencia es coherente con la persistencia de algunas prácticas arraigadas en tradiciones alimentarias indígenas y afrobrasileñas, junto con la adopción y adaptación de prácticas culinarias asociadas con la inmigración europea. Al integrar datos moleculares e isotópicos con evidencia material y documental, este estudio contribuye a una comprensión más matizada de las prácticas alimentarias históricas, cuestionando narrativas de reemplazo cultural y destacando la complejidad de la vida doméstica cotidiana durante un periodo de grandes transformaciones sociales en el sur de Brasil. La tesis demuestra el potencial de la arqueología biomolecular para recuperar aspectos del pasado que a menudo están ausentes de los registros escritos, y ofrece un marco metodológico para la investigación dietética postcolonial e histórica.

Resum

Aquesta tesi investiga les pràctiques culinàries històriques al sud del Brasil durant finals del segle XIX i principis del segle XX mitjançant un enfocament bioarqueològic integrat d'artefactes ceràmics, restes humanes i materials faunístics. Centrada en jaciments arqueològics de les regions de la badia de Babitonga, a Santa Catarina, i de la llacuna de Patos, a Rio Grande do Sul, la recerca combina l'anàlisi de residus orgànics (ORA) en ceràmiques i l'anàlisi d'isòtops estables (SIA) en restes humanes i faunístiques per investigar els patrons de proteïna dietètica, l'ús de recipients i els comportaments relacionats amb el processament dels aliments. Aquestes dades es contextualitzen mitjançant fonts històriques, tipologia ceràmica i evidència zooarqueològica.

Els resultats d'aquesta tesi revelen patrons diferenciats en l'ús de recursos marins i terrestres, configurats pels contextos socials, les economies locals, les identitats històricament documentades i l'accés a productes de consum. Els resultats d'ORA indiquen patrons de residus ceràmics preservats principalment compatibles amb aportacions de plantes C_3 i greixos animals terrestres, amb evidència variable compatible amb recursos aquàtics, particularment en contextos costaners. El SIA indica contrastos regionals en les fonts de proteïna, cosa que suggereix un accés diferenciat al peix, al bestiar i a les plantes cultivades en entorns rurals i urbans.

L'evidència és coherent amb la persistència d'algunes pràctiques arrelades en tradicions alimentàries indígenes i afrobrasileres, juntament amb l'adopció i adaptació de pràctiques culinàries associades amb la immigració europea. En integrar dades moleculars i isotòpiques amb evidència material i documental, aquest estudi contribueix a una comprensió més matisada de les pràctiques alimentàries històriques, qüestionant narratives de reemplaçament cultural i destacant la complexitat de la vida domèstica quotidiana durant un període de grans transformacions socials al sud del Brasil. La tesi demostra el potencial de l'arqueologia biomolecular per recuperar aspectes del passat que sovint estan absents dels registres escrits, i ofereix un marc metodològic per a la recerca dietètica postcolonial i històrica.

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List of Abbreviations

AD -Anno Domini

AE -Acidified methanol extraction

APAA -ω-(o-alkylphenyl) alkanoic acid

APAA-C18 -ω-(o-alkylphenyl) alkanoic acid with 18 carbon atoms

APAA-C20 -ω-(o-alkylphenyl) alkanoic acid with 20 carbon atoms

APAA C20/C18 -ratio of APAA-C20 to APAA-C18

AT2 -Arroio Tamarina 2

BC -Before Christ

BCE -Before Common Era

BP -Before Present

C_3 -C_3 photosynthetic pathway

C_4 -C_4 photosynthetic pathway

CAM -Crassulacean acid metabolism

CSIA -Compound-specific isotope analysis

CSIA-AA -Compound-specific isotope analysis of amino acids

DAG -Diacylglycerol

DC -Dicarboxylic acids

DHA -Dehydroabietic acid

EA-IRMS -Elemental analyser-isotope ratio mass spectrometry

E/H -E/H isomer ratio

EI -Electron ionisation

ERC -European Research Council

FA -Fatty acid

FC -Foz do Cubatão

FFA -Free fatty acid

FID -Flame ionisation detector

GC -Gas chromatography

GC-C-IRMS -Gas chromatography-combustion-isotope ratio mass spectrometry

GC-FID -Gas chromatography-flame ionisation detection

GC-MS -Gas chromatography-mass spectrometry

HT-GC-MS -High-temperature gas chromatography-mass spectrometry

ICTA-UAB -Institut de Ciència i Tecnologia Ambientals, Universitat Autònoma de Barcelona

IRMS -Isotope ratio mass spectrometry

ITN -Innovative Training Network

LAPArq -Laboratório de Arqueologia e Patrimônio Arqueológico

LEPAARQ -Laboratório de Ensino e Pesquisa em Antropologia e Arqueologia

MAG -Monoacylglycerol

MARSUL -Museu Antropológico do Rio Grande do Sul

MASJ -Museu Arqueológico de Sambaqui de Joinville

MG1 -Morro Grande 1

m/z -Mass-to-charge ratio

MeOH -Methanol

MS -Mass spectrometry

MUFA -Monounsaturated fatty acid

NIST -National Institute of Standards and Technology

ORA -Organic residue analysis

P/S -Palmitic/stearic ratio

PG-U21 -Praia Grande -Unidade 21

PNAS -Proceedings of the National Academy of Sciences

PUFA -Polyunsaturated fatty acid

RS -Rio Grande do Sul

SC -Santa Catarina

SE -Solvent extraction

SFA -Saturated fatty acid

SFS -São Francisco do Sul

SGR-Cat -Support for Research Groups of Catalonia

SIA -Stable isotope analysis

SIM -Selected ion monitoring

SRR -Standardised reference range

SUERC -Scottish Universities Environmental Research Centre

TAG -Triacylglycerol

TLE -Total lipid extraction

TMS -Trimethylsilyl

TMCS -Trimethylchlorosilane

TMTD -4,8,12-trimethyltridecanoic acid

TRADITION -Transformations in Diet and Culinary Practices

UAB -Universitat Autònoma de Barcelona

UFA -Unsaturated fatty acid

UFPEL -Universidade Federal de Pelotas

UNIVILLE -Universidade da Região de Joinville

VPDB -Vienna Pee Dee Belemnite

Author Declaration

I declare that this thesis is a presentation of original work and is the result of my own independent research. It has not been submitted, either in whole or in part, for a degree or diploma at this or any other university. Where work presented in this thesis has involved collaboration with others, their contributions are fully acknowledged in the acknowledgements section.

I acknowledge that I received assistance from **ChatGPT/OpenAI** for proofreading and language refinement during the revision of this thesis, in line with the University of York Policy on Transparency in Authorship in PGR Programmes. All academic content, interpretation, data analysis, and final wording remain my own responsibility.

This thesis contains material that has been submitted for publication. The contributions of co-authors and collaborators involved in these works are explicitly acknowledged in the general acknowledgements section.

Chapter 5 has been adapted from the manuscript and submitted as "*The rise of modern Brazilian cuisine revealed by lipid and protein residues in ceramic artefacts*", submitted to *PNAS Nexus* (manuscript ID: PNASNEXUS-2025-00349-T).

Chapter 6 has been adapted from the manuscript and it is currently in preparation for submission as *"Long-term variability in food habits in Southern Brazil revealed by organic residue analysis of archaeological cooking wares*".

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All sources are appropriately referenced, and I confirm that this thesis complies with the University of York's regulations and guidelines regarding academic integrity and research ethics.

1. Introduction

1.1. Rationale and general aims

Since the 1500s, the Atlantic Forest coast of Brazil has been of major interest for European colonisation, particularly the one led by the Portuguese (Bethell et al., 1989). Among the consequences of contact between Europeans and the native or Indigenous groups in Brazil were the disruption, extirpation and extinction of local socio-ecological systems that had endured for thousands of years across the region. In addition, new plants, animals, pathogens, and techniques for extracting natural resources were introduced, along with 4 to 5 million enslaved African people. Plants, animals, land, water and people (Indigenous populations) were transformed into commodities, leading to the emergence of new forms of management and governing for what had now become traded products (Schwartz, 1986; Dean, 1997). This convoluted historical process, commonly referred to as the Columbian Exchange (Rasmussen, 1973; Nunn and Qian, 2010), has set the scene for Brazil's emergence as one of the world's leading food producers, and its notorious social and economic inequalities (Dean, 1997).

Historians have long debated the origins and evolution of Brazil's economic and social history from a macroeconomic perspective. From sugarcane, cotton and gold to coffee plantations and rubber, and from slavery to systems of free labour, Brazil's history is often told through the lens of its successes and failures as a major commodity producer and exporter in the modern and contemporary world (Schwartz, 1986; Bethell et al., 1989; Skidmore, 1999; Fausto and Fausto, 2014). Over the last decades, however, there has been a growing effort to understand local historical narratives, by illuminating the experiences of people (peasants, fishers, artisans) and institutions (fisheries) who were not represented in official records, elite memories, biographies, naturalist accounts, or market statistics (Pereira et al., 2018; Gabriel and Silva, 2019; Herbst et al., 2023; Silva, 1988, 2001a). This new intellectual movement has allowed **Historical Archaeology** to gain momentum in Brazil and across Latin America as a discipline capable of uncovering the everyday experiences of vulnerable groups and

minorities, as well as their interactions with surrounding environments, in an attempt to provide a more holistic, just, and inclusive understanding of contemporary Brazil (Funari, 1994; Symanski and Zarankin, 2014; Symanski, 2010; Symanski and Souza, 2007; Symanski, 2009). However, historical archaeology in Brazil remains largely focused on ceramic artefacts, shipwrecks, infrastructure, housing and settlement patterns, while molecular approaches to historical archaeological materials are still underdeveloped (Funari, 1994; Symanski and Zarankin, 2014; Symanski, 2010; Symanski and Souza, 2007; Symanski, 2009; Borba, 2013).

The Atlantic Forest, recognised as a global biodiversity hotspot, and the Pampa are home to numerous coastal and inland communities that have historically relied on marine and terrestrial biodiversity for food and livelihood (Rezende et al., 2018; Colombo and Joly, 2010; Silvano and Begossi, 2012). In these regions, small-scale fishing communities who descend from Indigenous, African and European populations, such as the Caiçaras (Begossi and Richerson, 1993; Hanazaki and Begossi, 2003; Begossi, 2006b), hold local ecological knowledge that is increasingly valued in management and conservation efforts (Begossi, 2006a; Levis et al., 2024; Bennett et al., 2022; Bennett, 2022; Bennett et al., 2018). These knowledge systems are relevant to the UN's Ocean Literacy principles (Santoro et al., 2017) aimed at promoting better public understanding, communication and informed policy decisions for ocean sustainability (Intergovernmental Oceanographic Commission, 2021), but their tangible and intangible historical heritage still remains largely overlooked. In acknowledging their role in sustainable use, management and protection of their ecosystems (Bennett et al., 2022, 2018), and that "*sustainable development cannot be achieved without ocean literate societies*" (UNESCO-IOC, 2022), it became evident that efforts should be made to engage with the origin and evolving nature of small-scale fishing and farming communities, and their longstanding interactions with the natural environment.

The overarching aim of this thesis is to advance the research agenda in Molecular Historical Archaeology in Brazil. It seeks to integrate multiple lines of evidence, with a particular emphasis on biomolecular analysis of archaeological

artefacts and biological materials, which remains underdeveloped in the country. Conceptually, it is based on the premise that analysis of material culture related to household food consumption (ceramic artefacts) and biological remains (human, faunal materials) has the potential to reveal much about Brazil's environmental, economic and social history. This is because it reflects how local actors valued natural resources, and transformed those resources into community and regional food traditions that in turn were also exposed and influenced by local-regional environmental contexts (coastal plain, lagoons), social relations, historically documented identities, economic conditions and changes in technology.

The thesis is set within the social, economic, and environmental context of the second half of the 19th and early 20th centuries in Southern Brazil. These were critical decades marked by significant reorganisations of political and social spheres, beginning with Brazil's independence from Portugal in 1822. They were also economically significant decades, driven by the rise of large-scale livestock (*Charqueadas*) and coffee production, which gained prominence in foreign markets and moved considerable labour forces, particularly in the central and Southern regions (Symanski and Zarankin, 2014; Funari, 1994). Infrastructure improvements facilitated the transportation of dry meat, coffee, fish, timber and other commodities through coastal ports, making coastal areas increasingly important and expanded urban centres into previously underdeveloped regions (Cabral, 1937; Noble, 1967). In 1888, the abolition of slavery initiated the transition to free labour, followed by mass European migration to replace the enslaved workforce (Symanski, 2009; Funari, Zarankin and Salerno, 2009). This shift contributed to the rise of a local middle class and the expansion of regional markets in emerging urban centres. They also reshaped Brazil's geopolitical and economic landscapes, particularly in Southern regions such as Santa Catarina and Rio Grande do Sul, where European migrants, Afro-descendants and Luso-Brazilian populations constituted the majority of the workforce in both rural and urban settings.

Historical records indicate that fish, along with agricultural products (both crops and livestock), increasingly served to meet local and regional demand (Costa, 2018;

Fossile et al., 2024), attracting both public and private investors (Herbst et al., 2023). Unprecedented political efforts were documented during these decades and the following ones to boost Brazil's commercial fishing sector, a historical phase that Herbst et al. defined as the "incubation chamber" of Brazil's industrial fisheries (Herbst et al., 2023). But from a local perspective, studies have shown that fisheries policies driven by profit and efficiency accelerated the "economy of things" at the expense of the "economy of relationships" between communities and their environments (Manno, 2010), leading to intensified exploitation, increased competition and weakened governance in the decades that followed (Herbst et al., 2024, 2023). This market-driven logic has contributed to the dismantling of traditional forms of coastal management and socio-ecological interactions (Diegues, 1983). Questions remain about the extent to which non-elite communities had access to fish and other aquatic resources.

Considerable economic variability marked this historical process. In the coastal areas of Santa Catarina, small-scale fisheries and farming coexisted as subsistence systems serving domestic and local markets. In contrast, the Patos Lagoon region in Rio Grande do Sul also witnessed one of the first examples of industrial-scale food production outside the northern regions of the country, relying heavily on enslaved labour: the *Charqueadas* (cattle ranching industry). The meat industry fostered the rise of local elites who invested in urban centres and infrastructure, driving regional development in the years before the abolition of slavery in 1888 (Jordan, 1962; Duarte, 2014)

While extensive research has examined colonial and post-colonial impacts on broader food systems in Brazil (Gabriel and Silva, 2019), gaps remain in understanding 1) *how food habits evolved over time in the coastal regions of Santa Catarina and Rio Grande do Sul* 2) *to what extent were marine and terrestrial resources used and valued by historical groups with distinct ethnic backgrounds and social status in these regions* and 3) *how did local economies influence dietary habits and culinary traditions among rural communities and emerging urban elites.*

This thesis addresses these research questions by applying Organic Residue Analysis (ORA) to ceramic materials and Stable Isotope Analysis (SIA) to human and faunal remains recovered from historical archaeological sites in Babitonga Bay (Santa Catarina) and Patos Lagoon (Rio Grande do Sul), dating from the late 19th to early 20th centuries.

Molecular analysis of organic residues preserved in ceramic artefacts can be used to obtain information on past food preparation and consumption practices (Hendy et al., 2018; Evershed, 2008; Craig, 2021). For example, lipids are readily absorbed by porous ceramic vessels during food processing, retaining molecular and isotopic compositions that carry significant information on culinary practices (Craig, 2021; Evershed, 2008; Cramp and Evershed, 2014; Dudd and Evershed, 1998). Lipid residue analysis is currently expanding our understanding of prehistoric economies in coastal Brazil (Admiraal et al., 2023; Colonese et al., 2014; Hansel and Evershed, 2009; Lantos et al., 2024), but it has never been applied to historical contexts. Here the focus is on the historic ceramic material retrieved from five different historic sites in Babitonga Bay and Patos Lagoon.

Stable isotope analysis of human remains is a well-established approach for dietary reconstructions (Toso et al., 2021; Bastos et al., 2022, 2016; Alexander et al., 2019; Makarewicz and Sealy, 2015; Fernandes and Jaouen, 2017). Stable carbon and nitrogen isotopes from bone collagen have been extensively used to estimate sources of dietary proteins in pre-colonial human groups in Brazil (Toso et al., 2021; Bastos et al., 2022; Colonese et al., 2014; Bastos et al., 2014), but there has been very little research on historical human individuals (Bastos et al., 2016). Here we extend the SIA approach to human and faunal materials from historical sites in Babitonga Bay and Patos Lagoon (Porto Alegre, in Rio Grande do Sul) to document the main source of dietary proteins and to compare these results with evidence for food processing derived from contemporary ceramic vessels from the same regions.

1.2. Research questions and objectives

Using a suite of molecular techniques (SIA, ORA) combined with archaeological evidence (faunal remains, artefacts typology) and historical records we will address the following research questions:

1) **How have food habits evolved over time in the coastal regions of Santa Catarina and Rio Grande do Sul?**

ORA is performed on historical ceramic vessels recovered from archaeological contexts in Babitonga Bay and Patos Lagoon. The molecular and isotopic data obtained are compared with analyses conducted on pre-colonial Indigenous ceramic artefacts to identify continuities and changes in the use of natural resources and culinary practices through time. The results are further contextualised through a comparison with human skeletal isotopic data ($\delta^{13}C$ and $\delta^{15}N$) to better understand dietary shifts.

2) **To what extent were marine and terrestrial resources used and valued by historical groups with distinct ethnic backgrounds and social status in these regions?**

SIA on faunal remains and ORA from ceramic artefacts recovered from historically and archaeologically distinct contexts in Babitonga Bay and Patos Lagoon are used to assess variation in resource use, particularly in relation to marine and terrestrial resources. Lipid profiles and isotopic signatures are interpreted alongside historical, archaeological and ethnographic evidence to explore how cultural affiliation, social position, environmental setting and economic access may have shaped food practices.

3) **How did local economies influence dietary habits and culinary traditions among rural communities and emerging urban elites?**

Lipid residues and isotopic data from ceramics used in rural (Babitonga Bay) and urban settings (Patos Lagoon) are also contextualised within the economic frameworks of the time, considering the impact of subsistence farming, market economies, and trade networks. This analysis aims to investigate the relationship between economic systems and foodways, shedding light on how culinary traditions were shaped by access to resources and social hierarchies.

1.3. Thesis structure

Although this work is presented as a monograph, instead of following a classical monographic structure with a "methods-results-discussion" format, this thesis adopts a thematic and contextual organisation. This choice reflects both the interdisciplinary nature of the research, which combines organic residue analysis, stable isotope data, and historical sources, and the need to approach culinary practices in a contextual, integrated manner. A more traditional structure would risk fragmenting the narrative and separating data from their cultural and historical meaning.

After the initial Chapters of literature review (2 and 3), and a Chapter which focuses on the materials and methods (4), three result Chapters (5,6, and 7) are organised thematically and geographically, addressing specific case studies while contributing to the overarching research questions. This structure mirrors the complexity of the materials studied and the development of the research process itself. It also allows for a meaningful comparison across different historical and social contexts, highlighting the diversity of foodways and resource use in Southern Brazil during the nineteenth and early twentieth centuries.

Two of the core Chapters (Chapters 5 and 6), which discuss the main case studies and present the results of the organic residue analyses, are currently under review for publication (Chapter 5) or under final stages for submission (Chapter 6).

These articles have been adapted into Chapters for this thesis, ensuring consistency with the overall narrative and structure, while maintaining the academic standards of peer-reviewed outputs.

Chapter content

Chapter 2: In this Chapter, both the archaeological and environmental contexts of the study area will be introduced in detail. The discussion begins with an environmental overview of Southern Brazil, with particular attention to the biomes of the Mata Atlântica and Pampa. Following this, the Chapter will discuss the historical background of Brazil, tracing the development of the country from its colonial origins to modern times, and exploring how the cultural, social, and economic transformations impacted the region. Special focus will be given to the two states of interest: **Rio Grande do Sul** and **Santa Catarina.**

Chapter 3: This Chapter presents a literature review of the scientific methodologies employed in this study: Organic Residue Analysis (ORA) and Stable Isotope Analysis (SIA). It outlines the theoretical principles behind these techniques and examines how they have been applied in archaeological research, especially in the context of foodways, culinary practices, and diet reconstruction.

Chapter 4: This Chapter details the analytical strategy and laboratory protocols used in this study. It provides information on the archaeological materials analysed, including ceramic sherds, human bones, and faunal remains, recovered from sites in Babitonga Bay (Santa Catarina) and the Patos Lagoon region (Rio Grande do Sul).

Chapter 5: This Chapter first introduces the archaeological sites located in Babitonga Bay (Santa Catarina) and then the results of the Organic Residue Analysis conducted on historic ceramics. The analysis investigates food preparation and consumption practices among historically lower-income, small-scale communities. Results are discussed in relation to pre-colonial Indigenous ceramics and SIA of historic faunal remains, to contextualize food sources and culinary continuities or shifts.

Chapter 6: This Chapter first introduces the archaeological sites located in Patos Lagoon (Rio Grande do Sul) and then the results of the Organic Residue Analysis conducted on historic ceramics. The Chapter investigates how culinary traditions intersected with social class, ethnicity, and urban development in 19th and early 20th century Pelotas. It builds upon and contrasts the findings from Babitonga Bay to understand regional variability. Results are discussed in relation to pre-colonial Indigenous ceramics and SIA of historic faunal remains, to contextualize food sources and culinary continuities or shifts.

Chapter 7: This Chapter first introduces the archaeological sites located in Patos Lagoon (Rio Grande do Sul) and Babitonga Bay (Santa Catarina) and then the results of SIA of faunal and human remains. These results are then compared with ORA data from Chapters 5 and 6.

Chapter 8: This final Chapter synthesizes the main findings from the previous Chapters in light of the study's research questions. It discusses how culinary practices evolved across time and space in the Southern Brazilian context, evaluates the contribution of organic residue and isotopic data to archaeological research, addresses the limitations of the study, and suggests avenues for future research.

2.Environmental and historical background

Having outlined the research aims, questions, and methodological framework, the following Chapter situates the study within its geographical and historical setting. It examines the environmental characteristics and socio-economic history of Southern Brazil, focusing on Santa Catarina and Rio Grande do Sul, and introduces the contrasting local economies and foodways that underpin the archaeological case studies explored in later Chapters.

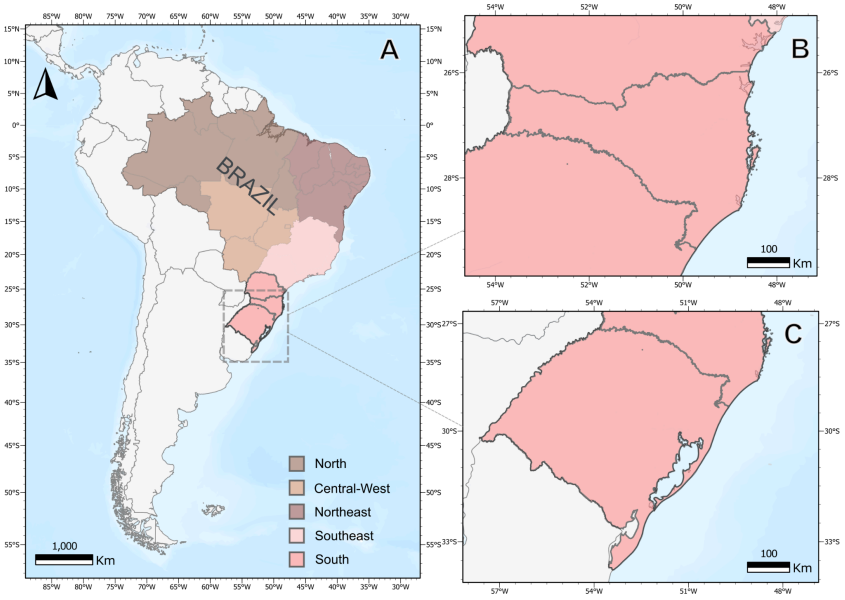


Figure 1 : *Map of Brazilian regional divisions, highlighting the Southern region (A), including the states of Santa Catarina (B) and Rio Grande do Sul (C), which are the focus of this study. Map created using ArcGIS Pro and edited in Inkscape.*

2.1. Geographical and ecological overview: Southern Brazil

Brazil is the largest country in South America, covering a wide array of climates, landscapes, and ecological zones. Administratively, the country is divided into five major regions: North, Northeast, Southeast, Central-West, and South (Figure 1A). Each region has distinct environmental characteristics, contributing to Brazil's ecological diversity. The South region of Brazil (Figure 1A), which includes the states of Santa

Catarina and Rio Grande do Sul, is characterised by a temperate climate and two key biomes: the **Mata Atlântica** (Atlantic Forest) and the **Pampas** (Overbeck et al., 2007). These biomes offer unique ecological settings that have historically shaped local food systems, agro-pastoral traditions, and culinary practices. Currently, the economy of both regions relies heavily on services, tourism, agriculture, livestock, and industry, which, while essential for economic growth, contribute to environmental challenges such as deforestation, soil erosion, and water pollution (de Area Leão Pereira, Diele-Viegas and de Santana Ribeiro, 2024; de Figueiredo et al., 2017).

2.1.1. The Mata Atlântica: ecology, history and conservation

The Mata Atlântica, or Atlantic Forest (Figure 2A-B), is a tropical and subtropical forest biome that historically extended along Brazil's eastern coastline, reaching into parts of Paraguay and Argentina. Originally covering approximately 1.5 million km², this biome has been drastically reduced due to centuries of deforestation, leaving behind only 12-28% of its original extent, fragmented across various regions (Rezende et al., 2018). Comparatively, the Mata Atlântica was historically almost twice the size of France. Even in its fragmented state today, it still covers an area equivalent to the United Kingdom and Portugal combined. The forest stretches from 5°S to 30°S, spanning multiple Brazilian states, including Bahia, Espírito Santo, Rio de Janeiro, São Paulo, Paraná, Santa Catarina, and Rio Grande do Sul (Marques et al., 2021). Due to its complex orography, ranging from coastal lowlands to mountainous regions, the biome has developed high biodiversity and endemism. However, unlike the Amazon, which retains large contiguous tracts of forest, the Atlantic Forest exists mostly as small, isolated patches, particularly in steep terrains, protected areas, and conservation units (Lins-e-Silva, Ferreira and Rodal, 2021). Despite this fragmentation, it continues to play an essential role in many ecosystem services such as water regulation, carbon storage, and biodiversity conservation, particularly for Brazil's most densely populated regions (Pires et al., 2021).

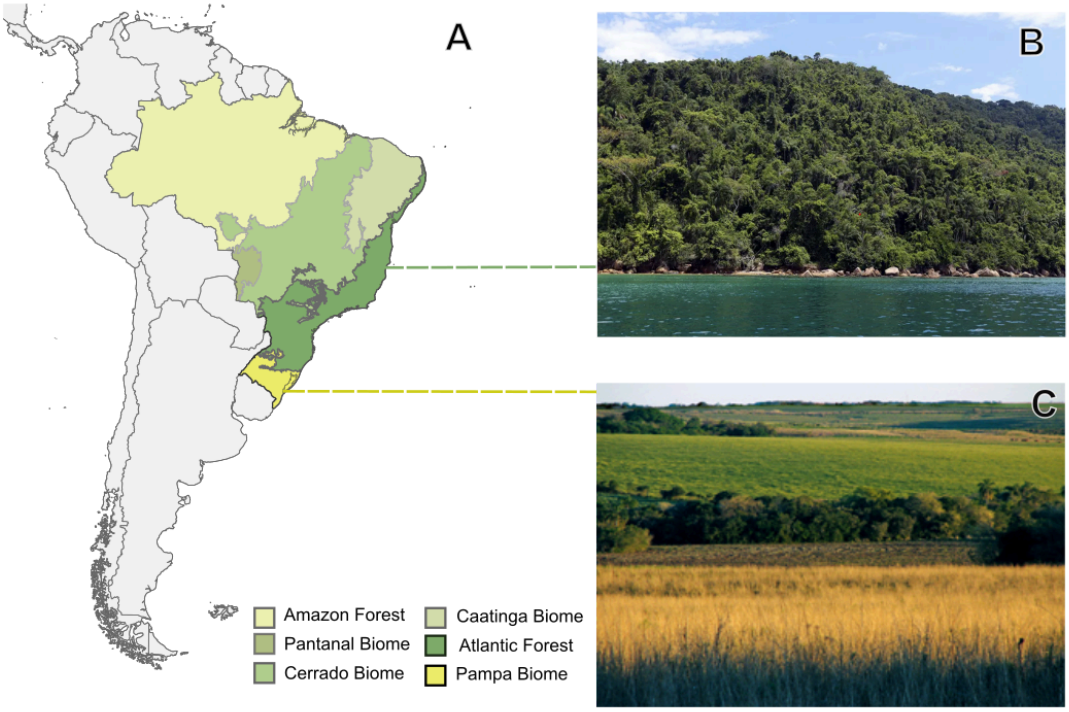


Figure 2: *(A) Map of Brazilian biomes, highlighting the Atlantic Forest and Pampa Biome in the Southern region. (B) Photograph of the Mata Atlântica biome (by Rhett A. Butler) and (C) Photograph of the Pampa biome (by Marcio Borges Martins). Map created using ArcGIS Pro and edited in Inkscape.*

This forest is one of the world's most diverse biomes, hosting approximately 20,000 plant species, nearly 40% of which are endemic (Marques et al., 2021; Guedes et al., 2005). This biome is home to different vegetation formations, including dense ombrophilous forests, seasonal semi-deciduous forests, mixed ombrophilous Forests (*Araucaria forests*), and non-forest ecosystems like restingas and mangroves (Carlucci, Marcilio-Silva and Torezan, 2021; Guedes et al., 2005). The dense ombrophilous forest, primarily found in coastal regions, is characterised by multi-layered canopies, epiphytes, and evergreen trees that thrive under consistently high humidity (Ibge, 2012; Guedes et al., 2005). In contrast, Seasonal Semi-Deciduous Forests, dominant in

the interior, exhibit tree species that shed their leaves during the dry season, a crucial adaptation to water scarcity (Guedes et al., 2005). The mixed ombrophilous forests of Paraná, Santa Catarina, and Rio Grande do Sul are dominated by the Paraná pine (*Araucaria angustifolia),* a conifer species that once covered vast expanses of the highland plateaus but is now endangered due to logging and agricultural expansion (Carlucci, Marcilio-Silva and Torezan, 2021; Guedes et al., 2005). Additionally, restingas, found along sandy coastal areas, and mangroves in estuarine environments, support specialised plant communities that contribute to coastal stability and serve as carbon sinks (Lins-e-Silva, Ferreira and Rodal, 2021; Guedes et al., 2005). The diversity of the biome, caused by historical climatic fluctuations and complex topography, means that species composition varies significantly across regions, with some forest fragments containing over 450 tree species per hectare, a density exceeding that of many Amazonian regions (Carlucci, Marcilio-Silva and Torezan, 2021; Guedes et al., 2005).

This biome also contains more than 2,200 vertebrate species, including approximately 270 mammal species, 850 birds, 370 amphibians, 200 reptiles, and over 350 freshwater fish species (Figueiredo et al., 2021). Endemism is particularly high among primates, birds, and amphibians, many of which are critically endangered due to habitat fragmentation (Figueiredo et al., 2021). Among the most iconic species are the golden lion tamarin (*Leontopithecus rosalia*), which has become a symbol of conservation efforts, the woolly spider monkey (*Brachyteles arachnoides*), and the maned three-toed sloth (*Bradypus torquatus*), all of which are endemic to the Atlantic Forest (Figueiredo et al., 2021). The forest also harbors significant bird diversity, with species such as the red-billed curassow (*Crax blumenbachii*) and the red-tailed parrot *(Amazona brasiliensis*), both highly dependent on intact forest areas for survival (Figueiredo et al., 2021). Amphibians are another key group, as the forest's humid environment supports one of the highest concentrations of endemic frog species in the world (Padial et al., 2021). These species play crucial ecological roles, including seed dispersal, pollination, and pest control, making their survival critical for the biome’s overall health (Padial et al., 2021).

Human presence in the Atlantic Forest dates back over 10,000 years, with evidence of early Indigenous societies practicing slash-and-burn agriculture, hunting, and fishing while maintaining a dynamic relationship with the forest (Lira, Portela and Tambosi, 2021; Solórzano, Brasil and de Oliveira, 2021). The arrival of Europeans in the 16th century marked a turning point, as Portuguese colonizers initiated large-scale deforestation for sugarcane plantations, logging, and cattle ranching (Solórzano, Brasil and de Oliveira, 2021). This pattern of exploitation continued into the 20th century, driven by urban expansion, coffee cultivation, and industrialization, leading to severe habitat loss and biodiversity decline (Carlucci, Marcilio-Silva and Torezan, 2021). Despite conservation efforts, many Indigenous communities and Quilombola populations (descendants of enslaved Black Africans) continue to struggle for land rights as agribusiness and urbanization encroach upon forested areas (Lira, Portela and Tambosi, 2021). Recent studies highlight the importance of recognizing these communities' traditional knowledge in sustainable forest management (Marques et al., 2021).

Currently, the Atlantic Forest is home to over 148 million people, representing nearly 70% of Brazil's population (Solórzano, Brasil and de Oliveira, 2021). It comprises major economic centres, including São Paulo, Rio de Janeiro, Curitiba, and Florianópolis, making it a key driver of Brazil's economic output (Carlucci, Marcilio-Silva and Torezan, 2021). The biome supports agriculture, tourism, hydropower, and industrial activities, but these economic drivers often come at the expense of forest conservation (Pires et al., 2021). Urbanization remains a significant challenge, as uncontrolled expansion and infrastructure projects continue to threaten remaining forest fragments (Lira, Portela and Tambosi, 2021). However, eco-tourism and sustainable forestry initiatives have emerged as potential solutions, aiming to balance economic development with conservation (Carlucci, Marcilio-Silva and Torezan, 2021).

2.1.2. The Pampa Biome: ecology, history and conservation

The Pampa biome, also known as the Campos Sulinos in Brazil, is a vast temperate grassland extending across Southern Brazil, Uruguay, and Argentina. In Brazil,

it occupies the state of Rio Grande do Sul, covering approximately 176,000 km², which represents about 2% of the national territory (Overbeck et al., 2007). The biome lies between latitudes 28°S and 35°S, surrounded by rolling plains, low hills, and coastal wetlands (Baez-Lizarazo, Köhler and Reginato, 2024). The Pampa is often compared in size to countries like Uruguay or the UK, yet remains one of South America's most neglected biomes in conservation efforts (Overbeck et al., 2015). The geological history of the Pampa is marked by Pleistocene climatic fluctuations, which influenced the evolution of its grassland ecosystems. Fossil and phylogenetic data suggest that C_3 grasses dominated the biome in the Paleogene (~34 Ma), followed by the expansion of C_4 grass species during the Miocene (~10 Ma) (Baez-Lizarazo, Köhler and Reginato, 2024). The Pampa is floristically connected to other South American open ecosystems, including the Cerrado, Chaco, and Andean foothills, through historical dispersal and biotic interchange (Baez-Lizarazo, Köhler and Reginato, 2024).

The Pampa is characterised by a rich diversity of grass species, with about 4,368 vascular plant species, 8% of which are endemic (Andrade et al., 2023). It is primarily composed of mesophytic grasslands, dominated by species from the Poaceae, Asteraceae, and Fabaceae families. These plant communities are influenced by fire, grazing, and seasonal rainfall variations (Overbeck et al., 2007). The northern Pampa, particularly in Rio Grande do Sul and Uruguay, has higher floristic diversity and shares species with the Atlantic Forest and Cerrado, while the Southern Pampa in Argentina is comparatively drier and less diverse (Baez-Lizarazo, Köhler and Reginato, 2024). Historical biogeographic analysis reveals that many Pampa species originated from Neotropical forests, particularly the Atlantic Forest, through dispersal events (Baez-Lizarazo, Köhler and Reginato, 2024). Key functional traits in Pampa vegetation include fire tolerance, drought resistance, and adaptations to grazing pressure (Andrade et al., 2023). The biome also features small patches of shrublands and riparian forests, particularly along rivers and rocky outcrops (Overbeck et al., 2015).

This biome supports a diverse fauna adapted to grassland conditions, including species from the Cervidae, Canidae, and Rodentia families. The biome is home to

native herbivores such as the pampas deer (*Ozotoceros bezoarticus*) and the plains viscacha (*Lagostomus maximus*), as well as predators like the maned wolf (*Chrysocyon brachyurus*) and the Geoffroy's cat (*Leopardus geoffroyi*) (Overbeck et al., 2007). Avian diversity is particularly rich, with over 500 recorded species, including endangered grassland birds like the saffron-cowled blackbird (*Xanthopsar flavus*) (Bencke, Dias and Fontana, 2024). Mammalian and avian communities in the Pampa have been shaped by historical climate fluctuations and human-driven landscape changes (Baez-Lizarazo, Köhler and Reginato, 2024). The biome's faunal diversity is at risk due to habitat fragmentation and agricultural expansion, which reduce native grassland cover and disrupt ecological corridors (Overbeck et al., 2015).

The Pampa has been inhabited for over 10,000 years, by groups such as the Charrua, Minuane, and Guarani (Baez-Lizarazo, Köhler and Reginato, 2024). These groups traditionally relied on hunting, gathering, and later cattle ranching, which influenced the region's landscape dynamics. The arrival of European settlers in the 17th and 18th centuries led to intensive livestock farming, which became the dominant economic activity (Overbeck et al., 2007). During the 19th and 20th centuries, large-scale cattle ranching, soy monoculture, and forestry plantations dramatically altered the biome (Oliveira et al., 2017). Native grasslands were replaced by exotic species such as Eucalyptus and Pinus, reducing biodiversity and affecting ecosystem services (Overbeck et al., 2015).

Today, the Brazilian Pampa is home to over 12 million people, with major urban centres including Porto Alegre, Pelotas, and Bagé (Ibge, 2012). The region's economy is centred on livestock production, accounting for a significant portion of Brazil's beef and wool exports. Additionally, soybean cultivation and afforestation projects have expanded in recent decades, often at the expense of native grasslands (Oliveira et al., 2017). Despite its economic importance, the Pampa remains one of Brazil's least protected biomes, with only 3% of its area under formal conservation status (Overbeck et al., 2015). The expansion of industrial agriculture and forestry plantations continues

to threaten biodiversity and ecosystem resilience (Baez-Lizarazo, Köhler and Reginato, 2024).

2.2. Setting the scene: historical overview on early modern Brazil (19th and 20th centuries)

While Brazil's history is deeply enriched by the heritage of its original inhabitants, whose presence extends back thousands of years (Denevan, 1992; Milheira and DeBlasis, 2014; Toso et al., 2021; Bastos et al., 2022; Admiraal et al., 2023), this section focuses primarily on the transformative events and developments that occurred in the second half of the 19th and early 20th centuries, which is the time interval covered by this thesis. Nevertheless, to provide essential context, earlier historical foundations that set the stage for these later changes will also be briefly presented. As discussed i**n Chapter 1,** the second half of the 19th century represents a particularly critical period in Brazil's political, social and economic history, marked by profound shifts in its social fabric, regime and economic institutions.

Before European colonisation (1500 AD), large ethnic groups belonging to the Gê, Tupi and Guarani, lived across Brazil, practicing distinct levels of agriculture, fishing and hunting, modifying the landscape and experimenting distinct social organisations (Denevan, 1992; Milheira and DeBlasis, 2014; Toso et al., 2021; Bastos et al., 2022; Admiraal et al., 2023). The Portuguese arrival in 1500 marked a turning point, initiating colonisation efforts that exploited natural resources like brazilwood and introduced sugar plantations, relying heavily on African slavery to sustain the colonial economy (Boxer, 1969; Schwartz, 1986).

The 17th and 18th centuries marked the discovery of gold in Minas Gerais (Southeast Region of the country), leading to a significant gold rush, attracting a large influx of migrants and increasing the revenue of the Portuguese crown. This period also saw the development of internal trade routes and the growth of the mining industry, which had profound economic and social impacts on central and north Brazil (Barman,

1988). The widespread use of slave labour, which dominated the labour market from the 16th century to the 19th, was a significant factor in the inequality of income distribution that persisted over centuries (Conrad, 1972; Schwartz, 1986)

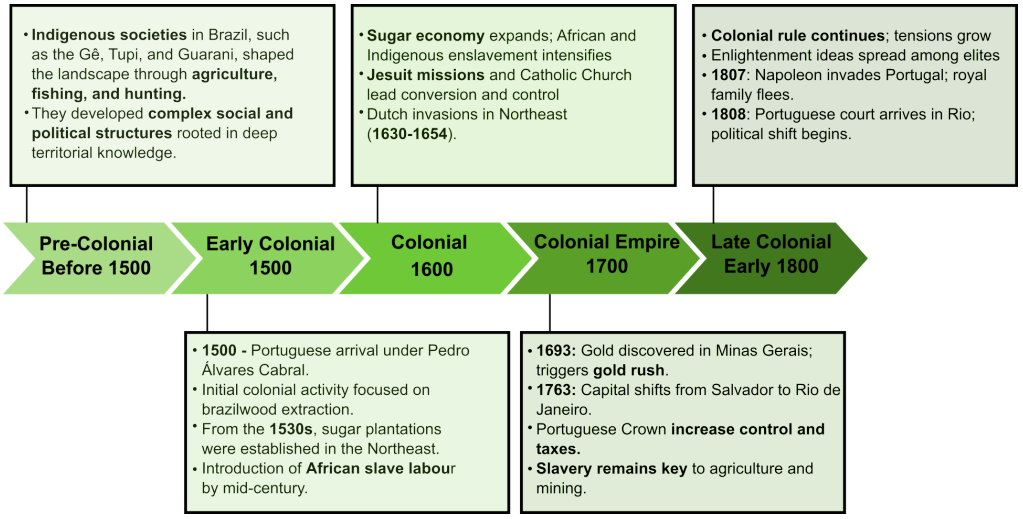


Figure 3: *Timeline of Early Modern Brazil (Pre-1500 to Early 1800s).*

In the early 19th century, the Portuguese royal family arrived in Brazil and elevated the country's status to the Kingdom of Brazil. This move ushered in a new and decisive phase in the history of the Empire, fostering nationalism and paving the way for independence in 1822, when Brazil became an empire under Dom Pedro I (Fausto, 1999; Barman, 1988). However, the persistence of slavery and concentrated wealth limited the impact of these changes. Indigenous groups had been largely exterminated or reduced to reservations. Their presence in coastal areas was minimal, while urban development expanded, particularly in central areas such as Santos (São Paulo State), Rio de Janeiro (Rio de Janeiro State), driven by the growth of coffee plantations (Fausto, 1999).

The late 19th and early 20th centuries marked a period of profound transformation. The abolition of slavery in 1888 ended forced labour and led to social and economic shifts (Jordan, 1962; Duarte, 2014). In 1889, Brazil transitioned from a monarchy to a republic, known as the "Old Republic," established by a military coup led by Marshal Deodoro da Fonseca with support from coffee planters (the *cafeicultores*)

and regional elites from São Paulo and Minas Gerais. This period was characterised by federalism, with political power concentrated in regional oligarchies through the system known as *coronelismo*, and a large export economy dominated by coffee exports and cattle products (Fausto and Fausto, 2014; Schwarcz and Starling, 2018). Following the abolition of slavery, the government actively promoted immigration to address labour shortages (Holloway, 2017).

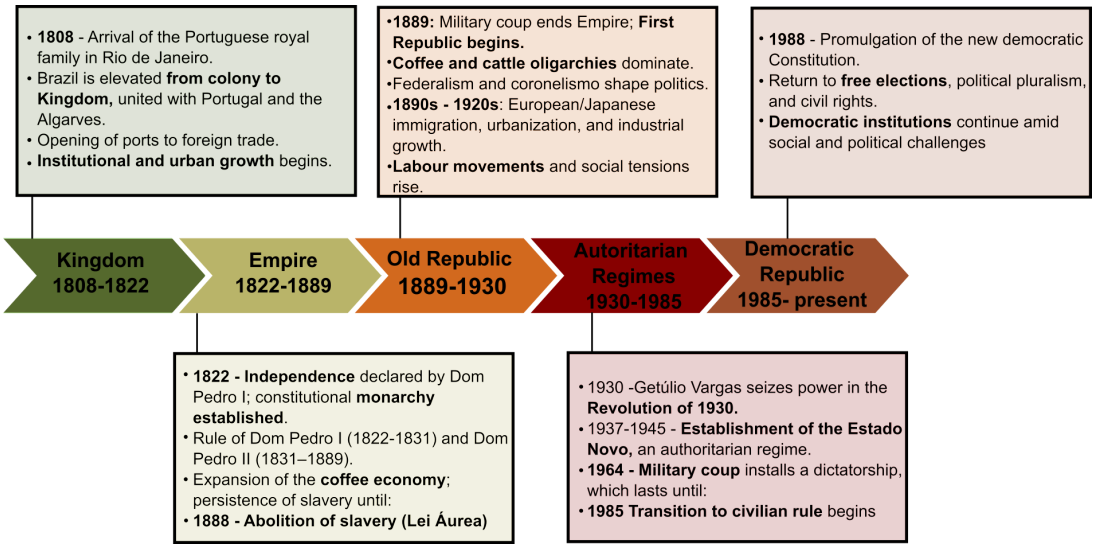


Figure 4: *Timeline of late 19th and early 20th centuries in Brazil.*

Millions of immigrants from Italy, Germany, Japan, Spain, among others, arrived in the central and Southern regions, introducing innovative agricultural practices and working on industries such as coffee, textiles, and ceramics (Skidmore, 1999). These immigrants not only diversified Brazil's workforce but also enriched its cultural fabric, particularly in Southern regions like Santa Catarina and Rio Grande do Sul, where European and African influences merged to create distinct local traditions (Ribeiro, 2017; Borba, 2014). Additionally, the legacy of slavery remained deeply ingrained, shaping patterns of inequality and economic development in the post-abolition period (Palma et al., 2021).

This era also witnessed Brazil's urban and industrial expansion. Cities like São Paulo, Rio de Janeiro, and Porto Alegre emerged as industrial hubs, with railways and ports facilitating the growth of regional and international trade (Skidmore, 1999; Vidal

Luna and Klein, 2014; Palma et al., 2021). Southern cities like Pelotas also developed at that time, with industrial growth fueled by the *charque* (salted meat) industry and immigration driving urbanization (da Silva Peixoto and Cerqueira, 2006). These transformations also ignited the rise of labour movements, which advocated for improved working conditions and influenced early social reforms (Bethell et al., 1989; Holloway, 2016). Cultural shifts were equally transformative. The blending of Indigenous, African, and European influences, particularly in culinary practices, reshaped Brazilian identity (Ribeiro, 2017; Borba, 2014).

Politically, the early 20th century was marked by the consolidation of republican ideals and the expansion of federal governance. While the Old Republic's reliance on coffee and cattle elites dominated national policies, emerging industrial centres began to challenge traditional power dynamics (Fausto, 1999). This period saw growing tensions between rural oligarchies and urban industrialists, reflecting broader struggles for political and economic modernization (Skidmore, 1999; Fausto, 1999). Although this study focuses on the transformations that took place between the late nineteenth and early twentieth centuries, it is worth noting that, following a prolonged period of authoritarianism beginning in 1930, Brazil entered a phase of redemocratization in 1985, culminating in the 1988 Constitution and the establishment of the current democratic regime (Fausto, 1999).

2.2.1. The State of Santa Catarina

Five of the eight archaeological sites analysed in this study, Morro Grande 1 (MG1), Praia Grande - Unidade 21 (PG-U21), Foz do Cubatão (FC), Arroio Tamarina 2 (AT2), and the Igreja Matriz de Nossa Senhora da Graça, are located in the state of Santa Catarina. These sites provide key insights into historical subsistence strategies, food production, and material culture of distinct ethnic and social groups that occupied the region, contributing to a broader understanding of household food consumption and culinary practices in early modern Brazil (Borba, 2014; Alves, 2017). A detailed analysis of the archaeological context of the sites, ceramic assemblages and ORA will

be presented in **Chapter 5**, while SIA of faunal and human remains, will be explored in **Chapter 7**.

Santa Catarina's colonial and modern history has been shaped by its strategic coastal location, fertile lands, and abundant natural resources. European colonisation intensified from the 17th century, with the founding of São Francisco do Sul (1642), Desterro, today's Florianópolis (1662) (Figure 5A-B). These towns became important hubs for maritime trade, agricultural surplus redistribution, and the circulation of goods and people. Enslaved Africans were introduced to work in agricultural and domestic contexts, bringing culinary knowledge and crops that merged with Indigenous and Portuguese traditions (Borba, 2014; Palma et al., 2021; Borba, 2017).

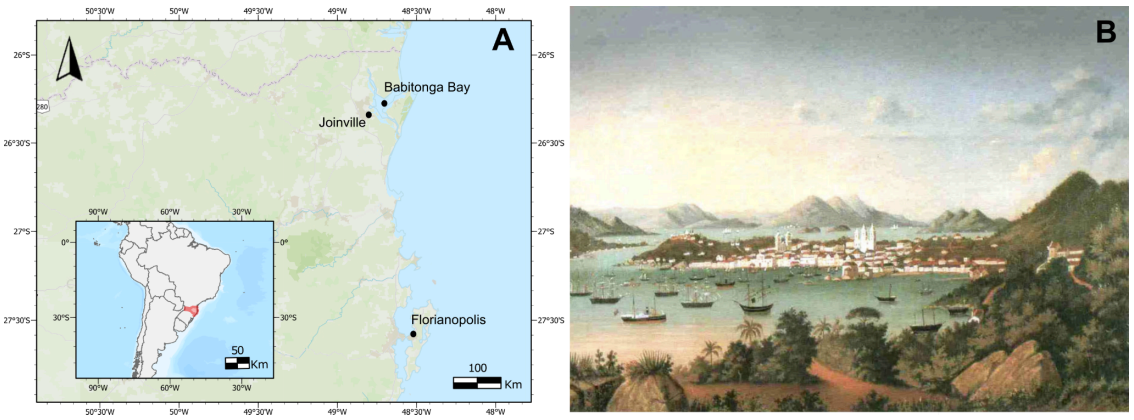


Figure 5: *(A) Location of Santa Catarina State, Brazil, highlighting key areas of interest, Babitonga Bay, Florianópolis, and Joinville. The map was created using ArcGIS Pro and edited in Inkscape. (B) View from the old city of Desterro 1868, now Florianópolis by Joseph Bruggemann (Corrêa, 2005)*

In the second half of the 19th century, Santa Catarina experienced successive waves of European immigration, particularly from Germany, Italy, and Poland. These newcomers contributed to agricultural diversification, cultivating wheat, maize, cassava, beans, and horticultural crops, as well as to early industrial activities such as ceramics, furniture, and food processing (Skidmore, 1999; Noble, 1967; Borba, 2014). Settlements like Joinville integrated Luso-Brazilian, immigrant, and regional traditions, becoming important agricultural and commercial centres.

By the late 19th and early 20th centuries, Santa Catarina's economy combined small-scale, locally oriented fishing and farming systems with growing participation in wider commercial markets (Fausto, 1999; Cabral, 1937; Noble, 1967). Railway expansion and the development of ports in São Francisco do Sul and Florianópolis strengthened regional trade links. Despite increasing integration into commercial networks, many communities retained strong artisanal and household-based food production systems (Borba, 2014). The Babitonga Bay area, situated in the northern part of the state, exemplifies these dynamics. It offers a clear example of how small-scale coastal economies adapted to environmental opportunities and shifting socio-economic conditions, a case study explored in the following section.

Small-scale coastal communities in Babitonga Bay

Babitonga Bay, located on the northern coast of Santa Catarina, is an ecologically diverse estuarine-lagoonal system that encompasses extensive mangrove forests, river mouths, and small islands between the cities of São Francisco do Sul and Joinville (Fossile et al., 2023). These mangroves, among the largest in Southern Brazil, support rich marine and terrestrial biodiversity and have historically served as vital nurseries for fish, crustaceans, and mollusks (Fossile et al., 2023; Herbst et al., 2023). This ecological abundance shaped the subsistence strategies of both pre-colonial and historic communities, who practiced small-scale fishing, shellfish gathering, and agriculture based on manioc and other staples (Alves, 2017; Borba, 2014).

Long before European settlers arrived, Indigenous groups using ceramic artefacts attributed to Taquara-Itararé tradition, and later to Guarani linguistic stock, occupied the region of Babitonga Bay. The Taquara-Itararé tradition appeared in the region around 1200 cal BP. This coincides with the decline of the Sambaqui fishing culture, which had previously dominated the coastal landscape (Admiraal et al., 2023). Sambaquis are large shell mounds constructed by coastal populations from ca. 7000 cal BP onwards, composed primarily of mollusc shells, faunal remains, and artefacts (Figure 6A). These sites reflect resilient economies centred on fishing and shellfish

gathering, often supplemented by plant use and serving both domestic and ceremonial functions (Admiraal et al., 2023).

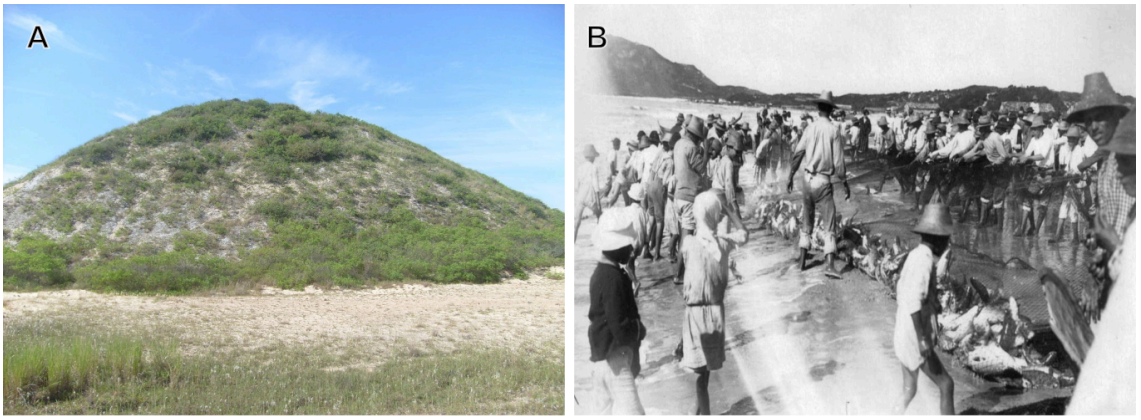


Figure 6: *(A) Picture of Sambaqui Figueirinha (SC) (Thigruner, 2006) ; (B) Photograph showing artisanal fishermen in Florianópolis (SC), early 20th century (by Baroness Edla von Wangenheim) (Fontana, 2024)*

The introduction of Taquara-Itararé ceramics marks this transition, but there is still debate over whether this represented a full replacement of Sambaqui populations or a more complex process of assimilation (Admiraal et al., 2023). The Taquara-Itararé exploited both marine and land resources like fish, maize, and cassava, and occupied regions from the highlands to coastal areas, with evidence of seasonal movements and interaction with different environments (Milheira and Blasis, 2010). Their ceramics, often plain with linear decorations, have been found at various archaeological sites, indicating their widespread presence and complex social structures. Organic residue analyses show that Taquara-Itararé pottery was used for processing fish and plants, with estuarine fish playing a significant role in their diet, (Admiraal et al., 2025, 2023).

The *Guarani* groups reached the Southern Brazilian coast around 1000 years ago (ca. 1000 cal BP), marking an important cultural change in the region prior to European colonisation. They also relied on fish and grew crops such as cassava and fruits. Guarani settlements in Southern Santa Catarina were strategically located near lagoons and rivers, taking advantage of abundant marine and freshwater resources, as well as accessible clay deposits for ceramic production (Milheira and Blasis, 2010).

Archaeological evidence reveals diverse ceramic styles used for daily cooking and ceremonial purposes, highlighting their advanced craftsmanship and cultural practices.

Stable isotope analyses of bone collagen and organic residue analysis on pottery have revealed insights into the diets and mobility patterns of these groups, emphasizing their adaptive strategies within coastal ecosystems. These analyses show a significant reliance on fish and other aquatic resources and their ability to adapt to the environment (Admiraal et al., 2023; Fossile et al., 2023; Admiraal et al., 2025).

The arrival of Portuguese settlers in the 16th century disrupted Indigenous systems but also resulted in the hybridization of food systems. Enslaved Africans who were brought to the region to work in agricultural and domestic settings, introduced tropical agriculture and culinary practices that blended with Indigenous and European traditions, creating a complex food system (Borba, 2014). During this period, cassava remained a dietary staple.

During the late 19th and early 20th centuries, Babitonga Bay underwent profound socio-economic transformations tied to Brazil's transition from Empire to Republic and the emergence of industrial and regional trade policies. This period was marked by the expansion of port infrastructure in São Francisco do Sul, then one of the most active maritime terminals in Santa Catarina, and the growth of Joinville as an urban and commercial hub, supported by railway connections and increased state investment (Fausto, 1999; Cabral, 1937; Noble, 1967). Immigration played a key role in shaping the region, with European settlers arriving through various colonisation initiatives, particularly in Joinville and surrounding areas. These communities introduced new agricultural and craft production systems, including the use of wheel-thrown ceramics, dairying, and intensified livestock farming (Herbst et al., 2023; Noble, 1967). Alongside traditional subsistence practices, commercial fishing and small-scale industries began to flourish, reflecting a blend of historical and modern approaches (Alves, 2017; Borba, 2014).

The reliance on marine resources persisted well into the late 19th and early 20th centuries, as evidenced by archaeological and historical records that point to sustained fish processing, shellfish collection, and manioc cultivation in coastal communities (Borba, 2014; Alves, 2017; Fossile et al., 2023) (Figure 6B). Excavated faunal assemblages from this period confirm the ongoing importance of estuarine species, while historical sources also document the commercialisation of dried fish and the importation of salted cod (*Gadus morhua*), illustrating both continuity and diversification in regional diets (Herbst et al., 2023). However, the intensification of resource use, driven by expanding trade and industrialisation, began to impact local ecosystems. Studies of biodiversity change in Babitonga Bay point to long-term pressures on marine and terrestrial environments, including the defaunation of medium- and large-bodied mammals and a shift in fish catches toward smaller, lower trophic level species, indicators of ecological degradation over time (Fossile et al., 2023).

Today, traditional ecological knowledge remains integral to the sustainable management of Babitonga Bay's resources. Practices such as rotational fishing, community-based marine conservation, and agroforestry systems highlight the resilience of coastal communities in balancing ecological preservation with economic needs. Archaeological and environmental studies emphasise the importance of bridging historical knowledge with contemporary conservation efforts to ensure the long-term sustainability of Babitonga Bay's natural and cultural heritage (Admiraal et al., 2023; Fossile et al., 2023).

2.2.2. The State of Rio Grande do Sul

Casa 2 is discussed in Chapter 6 in relation to the organic residue analysis of its ceramic assemblage. Casa 8 and the Cúria Metropolitana / Igreja Matriz de Nossa Senhora Madre de Deus are discussed in Chapter 7 in relation to the faunal and human stable isotope datasets from Rio Grande do Sul.

The state of Rio Grande do Sul, located at the Southern tip of Brazil, comprises a wide range of ecological zones, including grasslands, freshwater systems, and one of South America's largest coastal lagoons, the Patos Lagoon, part of the Pampas biome (Figure 7). This extensive estuarine and wetland region is among the most biologically productive ecosystems on the continent, having supported Indigenous societies for millennia through its abundance of aquatic life, fertile soils, and navigable waterways (Admiraal et al., 2025).

Before European colonisation, the lowland plains and lagoon margins were inhabited by Indigenous populations associated with the *cerritos* tradition-large earthen mounds constructed between ca. 3900 and 1000 cal BP. These mounds, distributed across the Patos Lagoon basin, served as residential areas, cemeteries, and ceremonial platforms (Admiraal et al., 2025; Milheira, Calippo and Haimovici, 2023) (Figure 8A). They reflect not only long-term settlement but also landscape engineering, enabling communities to adapt to seasonal flooding (Milheira, Calippo and Haimovici, 2023; Garcia, 2020). These societies formed part of a broader cultural phenomenon that extended across the Pampas, including regions of present-day Uruguay and Argentina.

Ethnohistorical and archaeological evidence suggests that Cerritos populations may have interacted with or been ancestral to later groups such as the Minuano and Charrúa, especially in the Guenoa-Minuan territory, which overlapped spatially and temporally with the mound-building tradition (Admiraal et al., 2025). These connections are supported by the continuity of certain settlement patterns, subsistence practices, and regional movement across the lowlands.

The Cerrito builders practiced mixed economies involving fishing, mollusc gathering, and plant cultivation, including maize (*Zea mays*), squash (*Cucurbita* sp.), beans (*Phaseolus* sp.), and palm fruits such as Butia capitata (Silva, Wagner and Ulguim, 2023; Admiraal et al., 2025). Organic residue analysis of ceramics from Cerritos sites has identified plant lipids consistent with both routine food preparation and

specialised activities, including fermentation, likely associated with feasting or ceremonial gatherings (Admiraal et al., 2025). Distinct patterns in vessel use suggest a separation between pots used for aquatic products and those for plant-based preparations, offering a comparative model for interpreting ceramic assemblages in this study (Admiraal et al., 2025).

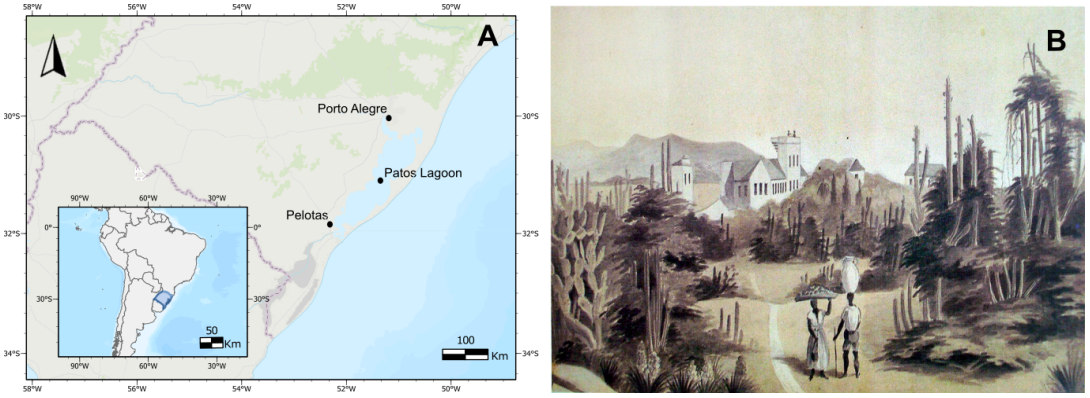


Figure 7: *(A) Location of Rio Grande do Sul state, Brazil, highlighting key areas of interest, Pelotas, Porto Alegre and Patos Lagoon. The map was created using ArcGIS Pro and edited in Inkscape. (B) Painting of Pelotas in 1852 by Hermann Rudof Wendroth, depicting the historical landscape of the city in the 19th century.*

The Portuguese presence in Rio Grande do Sul became more structured in the early 18th century, following the expansion of Luso-Brazilian cattle ranching from São Paulo and Santa Catarina into the Southern Pampas (Cerqueira and da Rosa, 2012; Kühn, 2020). The establishment of Jesuit missions by Spanish missionaries introduced new dynamics of Indigenous labour and religious conversion, creating an important network of settlements throughout the region (Fleck, 1999). From the mid-18th century, Portuguese and Spanish colonial disputes over this Southern frontier intensified, culminating in treaties such as the Treaty of Madrid (1750) and the Treaty of San Ildefonso (1777). These agreements sought to formalize territorial claims but were met with Indigenous resistance and local unrest (Borba and da Rocha Bandeira, 2013; Lesaffer, 2018; Owens, 1993). Several Jesuit missions were destroyed during this period, and the region became increasingly militarized as colonial powers sought to secure their influence (Lesaffer, 2018).

In response, the Portuguese Crown promoted the creation of strategic settlements and military outposts, such as Rio Grande (1737) and the relocation of Porto Alegre (1773), to assert colonial control and facilitate the movement of troops and goods (Kühn, 2020). These measures laid the groundwork for the development of the charque industry, which would later become a major economic force in the 19th century (Cerqueira and da Rosa, 2012). The displacement of Indigenous populations and the intensification of slavery, both African and Indigenous, significantly reshaped the region's social fabric. In the late 19th century, successive waves of European immigration, particularly Germans, Italians, and Poles, introduced new agricultural practices and artisanal skills that contributed to the diversification of the local economy (Peixoto, 2009).

Enslaved Africans also played a crucial role in Rio Grande do Sul's economic development. The charque industry in Pelotas, which depended on their labour, became a significant contributor to the region's wealth. (Palma et al., 2021; Peixoto, 2009; da Silva Peixoto and Cerqueira, 2006; Bastos, 2014). By the late 19th and early 20th centuries, Rio Grande do Sul had transitioned into a hub of agricultural and industrial activity. The growth of the timber industry and the development of urban centres such as Porto Alegre and Pelotas reflected the region's increasing economic diversification (Peixoto, 2009).

The charque industry of Pelotas

Pelotas, located in the Southern region of Rio Grande do Sul, played a crucial role in Brazil's *charque* industry, which emerged prominently in the late 18th and 19th centuries (Figure 8B). *Charque*, a type of salted and sun-dried beef, was a vital commodity due to its long shelf life, making it a staple for feeding enslaved labourers on sugarcane plantations and supporting Brazil's growing urban centres (Gutierrez, 2001; Vargas, 2017). The large-scale production of *charque* not only supplied local demand but also fueled exports to European markets and other regions of Brazil (Gutierrez, 2001). The economic prosperity brought by the charque industry also

promoted the urban expansion of Pelotas, with its infrastructure and historic buildings reflecting the wealth generated during the peak of charque production (Bastos, 2014).

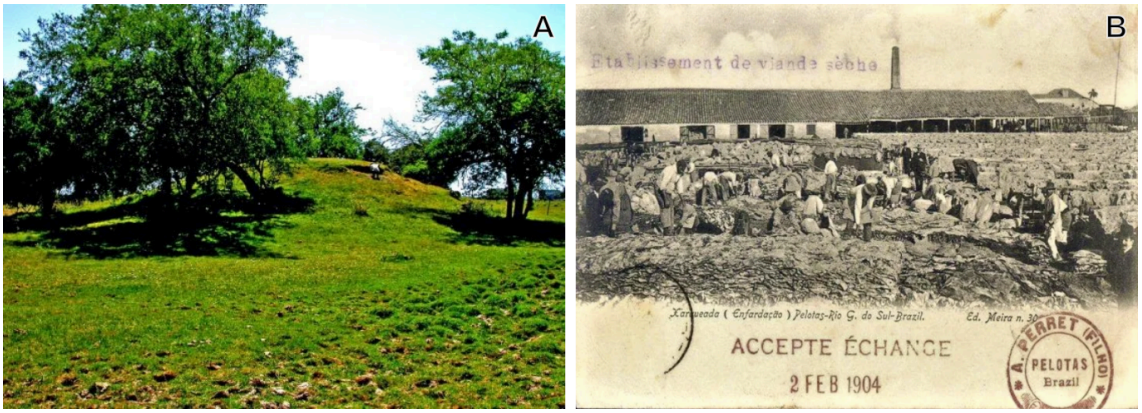


***Figure 8**: (A) Cerrito mound in Southern Brazil, representing pre-colonial Indigenous landscape modifications (Milheira, Calippo and Haimovici, 2023). (B) Early 20th-century postcard depicting a charqueada in Pelotas, Rio Grande do Sul, illustrating industrial meat production and labour practices (Universidade Federal de Pelotas - UFPel).*

The origins of the charque industry in Pelotas can be traced back to the 1780s, when José Pinto Martins established the first *charqueada* in the region along the Pelotas River (Gutierrez, 2001). This area, located strategically near waterways such as the Arroio Pelotas, the São Gonçalo Canal, and the Laguna dos Patos, facilitated the transportation of charque to both domestic and international markets (Cerqueira and da Rosa, 2012). The proximity to cattle-raising areas in the Pampas further solidified Pelotas' role as the "Charque Capital of Brazil" (da Silva Peixoto and Cerqueira, 2006). By the 19th century, Pelotas housed dozens of charqueadas, forming a robust industrial network that supplied salted beef, hides, and byproducts such as grease and tallow. These charqueadas, particularly in areas like Monte Bonito and Arroio Pelotas, were part of a spatially organised production system where the separation of cattle-raising (*estâncias*) from meat processing demonstrated the efficiency of the industry (Ribeiro, 2017). This specialisation was crucial for increasing productivity and sustaining the region's economic dominance. By 1911, over 30 charqueadas were operating in

Pelotas, serving as the backbone of the city's economy and shaping its spatial organisation (Gutierrez, 2001).

Charque production relied heavily on enslaved labour. Enslaved Africans and Afro-descendants were key in every stage of production, from herding and slaughtering cattle to salting and drying the meat. Historical records indicate that more than half of the enslaved population in charqueadas were specialised workers, such as *carneadores* (butchers), *marinheiros* (fishermen/sailmen), and craftsmen responsible for maintaining equipment and infrastructure (Palma et al., 2021). Many of these workers also contributed to Pelotas' architectural heritage by building the infrastructure necessary for sustaining the charque industry, including the warehouses and transportation networks that facilitated trade (Bastos, 2014). The 1833 census of Pelotas revealed that enslaved individuals constituted 51.7% of the population, with an additional 10.4% identified as freed Afro-descendants (Gutierrez, 2001). This demographic structure highlights the centrality of enslaved labour to the region's economy while reflecting the deep social inequalities that characterised Pelotas during this period (Gutierrez, 2001). This disparity perpetuated cycles of poverty and disenfranchisement that persisted long after the abolition of slavery in 1888 (Palma et al., 2021).

The decline of the charque industry began in the early 20th century, as the abolition of slavery and technological advancements (such as refrigeration) reduced the demand for salted beef. By the 1930s, most charqueadas had ceased operations, marking the end of an era for Pelotas. Despite this decline, the cultural and historical significance of the charque industry remains integral to the city's identity.

2.3. Culinary practices in Southern Brazil

The Southern region of Brazil encapsulates a rich blend of culinary practices shaped by different cultural influences, from Indigenous, to European and African societies and people that occupied the region over the last centuries.

2.3.1. Indigenous roots

In the coastal lowlands of Santa Catarina and Rio Grande do Sul, Indigenous populations developed diverse subsistence strategies that combined fishing, shellfish gathering, hunting, and the use of a wide variety of cultivated and foraged plants. At the heart of these food systems was cassava (*Manihot esculenta*), a long-established staple processed into *farinha* and other by-products that formed the dietary foundation across much of the region. Its preparation drew on a repertoire of processing techniques, roasting, drying, and, crucially, fermentation (Dória, 2012; Cascudo, 1967). Fermentation was particularly significant for cassava-based products such as *beiju* and *puba*, which were soaked, fermented, and dried to improve digestibility, reduce toxicity, and extend storage life (Dória, 2012; Cascudo, 1967). This practice was widespread throughout Brazil and, in regions such as the Rio Negro basin, was closely intertwined with cultural identity and food security (França, 2024). Fermentation also extended to beverages, notably *cauim* and *caxiri*, traditional cassava-based drinks that carried important ritual and social meanings within Indigenous communities (Lima et al., 2022). These techniques, embedded in a broader body of plant-processing knowledge, exemplify the sophistication of Indigenous culinary systems, which transformed staple crops into safe, storable, and culturally resonant foods. Alongside cassava, beans (*Phaseolus* spp.), particularly black beans, were another vital source of carbohydrates, forming part of a balanced subsistence base that long predates European arrival (Dória, 2012; Cascudo, 1967).

The botanical diversity of Brazil supported this plant-based foundation. Early naturalists such as Augustin Saint-Hilaire (Saint-Hilaire, 1936) documented the extensive use of native fruits, including feijoa, açaí, acerola, piquiá, caju, macaúba, guava, mango, papaya, pineapple, maracujá, and soursop, which were processed in multiple ways, from juice extraction to seed roasting (Saint-Hilaire, 1936). In Southern Brazil, nuts like cashew and a wide range of starchy crops, including yams and manioc derivatives such as tapioca and *tucupi*, were integral to everyday diets (Saint-Hilaire, 1936). The archaeological record confirms that these plant resources were prepared

using a variety of methods, including roasting, drying, and fermentation, and that fermentation often held both nutritional and symbolic importance (Dória, 2012; Saint-Hilaire, 1936).

Botanical remains such as starch grains and phytoliths, recoverable from sediments and ceramic residues, provide direct evidence of the processing, consumption, and in some cases domestication of these plants (Behling, 2006).

In Babitonga Bay, archaeological investigations at pre-colonial sites such as Morro do Ouro and Rio Comprido have documented the use of starchy tubers like yam (*Dioscorea* sp.), sweet potato (*Ipomoea batatas*), and plants from the Araceae family (e.g., taro-like roots), alongside fruits from palm trees (*Arecaceae*) and myrtles (*Myrtaceae*). The latter include several native fruit-bearing species such as guava (*Psidium guajava*), jabuticaba (*Plinia cauliflora),* pitanga or Surinam cherry (*Eugenia uniflora*), and araçá (*Psidium cattleianum*) (Pezo-Lanfranco et al., 2018). Microremains recovered from dental calculus, along with macrobotanical traces and grinding tools, suggest that plant collection and processing were integrated into daily routines.

Stable isotope data further support a mixed diet, combining plant carbohydrates with aquatic proteins. Archaeological evidence indicates the long-term importance of aquatic resources in Indigenous and historical diets (Pezo-Lanfranco et al., 2018). At pre-colonial sambaqui sites such as Morro do Ouro and Rio Comprido, faunal remains include abundant marine molluscs and estuarine fish species such as *Micropogonias furnieri* (whitemouth croaker), *Mugil* sp., and *Ariidae* (catfish), pointing to a consistent reliance on estuarine fishing and shellfish gathering throughout the Middle and Late Holocene (Pezo-Lanfranco et al., 2018). These resources complemented carbohydrate-rich diets based on tubers and native fruits.

In the Patos Lagoon region of Rio Grande do Sul, similar dietary patterns are observed among the mound-building Cerritos societies. These groups practiced a mixed economy that included fishing, hunting, gathering, and the management and cultivation of plant resources (Milheira and Gianotti Garcia, 2018). Archaeobotanical

and artefactual evidence indicates the use of maize (*Zea mays*), beans *(Phaseolus sp.)*, squash *(Cucurbita sp.)*, and tuberous plants such as Canna sp. and Calathea sp., alongside wild and semi-domesticated fruits (Milheira and Gianotti Garcia, 2018). These plant foods were integrated into long-term settlement strategies that included planned landscape use, permanent mound construction, and, in later periods, even maize cultivation areas located near some cerritos. Faunal assemblages from pre-colonial sites contain large quantities of fish bones, including estuarine species such as *Micropogonias furnieri* and *Pogonias cromis* (whitemouth croaker and black drum), suggesting that estuarine fishing was a stable and long-term component of subsistence over several millennia (Fossile et al., 2023). Some ceramic vessels from these sites also contained aquatic lipid biomarkers, supporting the interpretation that fish were processed in pottery (Admiraal et al., 2025). The integration of aquatic resources with cultivated and gathered plant foods illustrates the Cerritos societies' flexible adaptation to the wetland environments of the Southern Brazilian lowlands.

2.3.2. European influences

Portuguese colonisation introduced species and agricultural practices that reshaped Indigenous foodways, including crops like sugarcane and domesticated animals such as chickens, cattle, and pigs. They brought with them a variety of fruits and vegetables and citrus fruits such as oranges and lemons were introduced (Madeira, Reifschneider and de B Giordano, 2008) (Figure 9).



Figure 9: *Jean-Baptiste Debret:" Les fruits du Nouveau Monde" (1822 - musée Magnin / Thierry Le Mage)*

Portuguese settlers also introduced salting and drying techniques, which became central to the meat-based economy of Rio Grande do Sul and Santa Catarina (Ribeiro, 2017). One of the most debated aspects of Brazilian cuisine is the origin of feijoada, a stew of black beans and pork, often considered a quintessential Brazilian dish. While commonly believed to have originated in slave quarters, scholars argue that feijoada is more accurately linked to European stews such as the Portuguese *cozido* or the French *cassoulet* (Elias, 2010; Katz, 2014). This method involved browning meats in hot fat before slow cooking with beans and spices, a process that helped tenderize tougher cuts of meat and enhance flavour (Dória, 2012; Cascudo, 1967). The adaptation of European culinary techniques to local ingredients resulted in the creation of dishes that became central to Brazilian food culture. Feijoada is a clear example, typically served with black beans and *farofa* (a toasted cassava flour mixture), both legacies of Indigenous cuisine, alongside rice and sausages of European origin.

Smoking was another method introduced by European immigrants, particularly German and Italian settlers in Santa Catarina and Rio Grande do Sul used it to preserve sausages and cured meats (Borba, 2014). Portuguese techniques also included the extensive use of seasoning blends, such as those used in marinades and preserved

meats, which deeply influenced regional flavours (Cascudo, 1967). Additionally, the influence of conventional Portuguese cuisine, known for its methodical preparation of stews and layered dishes, played a role in shaping Brazil's slow-cooking traditions (Cascudo, 1967).

German immigrants in Santa Catarina introduced beer production based on European grains, with breweries established in Joinville as early as the 19th century (da Rocha Bandeira, 2017). They also influenced dairy farming, leading to the production of cheese varieties that became part of the local gastronomy (Borba, 2014). In addition to their influence on meat preservation, German communities contributed to baking traditions, introducing rye bread, pretzels, and pastries, which were later adapted with local ingredients (Borba, 2014).

Italian immigrants were central to wine-making traditions, particularly in the Serra Gaúcha region and highland areas of Santa Catarina, where vineyards flourished under their expertise (Serpa, 1998). They also brought pasta-making traditions, leading to the widespread consumption of polenta, and fresh handmade pasta, which became key elements of regional cuisine (Serpa, 1998). In addition to wine production, Italian immigrants contributed to cheese-making traditions, establishing dairy farms that produced a variety of cheeses that were incorporated into the regional diet (Bastos, 2014).

European influences were also evident in confectionery traditions, particularly in Pelotas and São Francisco do Sul, where elite Portuguese women and Afro-Brazilian cooks developed elaborate desserts like *quindim*, *camafeu*, and *doces finos*, adapting recipes with local ingredients (de Farias, 2012). The use of sugar, egg yolks, and tropical fruits in sweets became a defining characteristic of Brazilian pastries. In São Francisco do Sul, these confectionery techniques were also linked to trade and economic activities, as sweets and preserved fruits became significant commodities (de Farias, 2012).

2.3.3. African contribution

African culinary knowledge significantly contributed to the use of spices, slow-cooked stews, and frying techniques, as seen in dishes such as *acarajé*, *vatapá*, and *moqueca*. These methods were key to adapting local ingredients to African palates, ensuring that traditional dishes maintained their distinctive flavours despite the limitations of colonial life (Arsenault, 2010).

The use of *dendê* (palm oil), coconut milk, and okra reflects African culinary heritage (Souza, 2015). African cooking techniques, such as pounding grains with a mortar and pestle, were essential for preparing staples like rice and cassava-based dishes. This culinary influence extended to food preservation methods, including the use of smoked and dried fish, which became common in Afro-Brazilian communities (Carney, 2004). African women are believed to have brought rice grains hidden in their hair, ensuring that this staple was cultivated in the Americas (Carney, 2004). This African contribution was not limited to cultivation; enslaved populations brought specific rice-processing techniques from West Africa, including the use of large wooden mortars and pestles to dehusk grains. These techniques persisted well into the 19th century and influenced regional food systems. Food in Candomblé rituals, a religion with strong African roots in Brazil, further reflects the enduring impact of African food traditions. Ritual foods such as *acarajé* (deep-fried black-eyed pea fritters) and *amalá* (okra stew) are not only sacred offerings but also part of everyday Bahian cuisine (Souza, 2015). These culinary practices emphasise the deep interconnection between food, spirituality, and identity in Afro-Brazilian communities.

In Santa Catarina, archaeological research has identified ceramic artefacts associated with African culinary traditions, indicating the persistence of African cooking techniques in local food culture (Borba, 2014). Similarly, in Pelotas, Rio Grande do Sul, enslaved African women played a central role in food production, domestic kitchens, and confectionery, shaping what would become the region's renowned sweet-making tradition (Peixoto, 2009; Ribeiro, 2017). The preparation of *quindim*, *cocadas*, and

other egg-based desserts in colonial Pelotas was heavily influenced by African and Portuguese traditions, highlighting the adaptation of European recipes to local ingredients and Afro-Brazilian techniques (Figure 10).

Over the centuries, Brazilian cuisine became an important symbol of national identity. The Modernist movement contributed to the elevation of traditional foods, incorporating dishes such as *feijoada* into cultural narratives of Brazilian authenticity (Dória, 2012). The recognition of *feijoada* as an intangible cultural heritage in Rio de Janeiro in 2013 further reinforced its status as a national dish, deeply connected to historical processes of colonisation, migration, and adaptation (Bitelli, F. M. & Jurema, M. L. M., 2019).

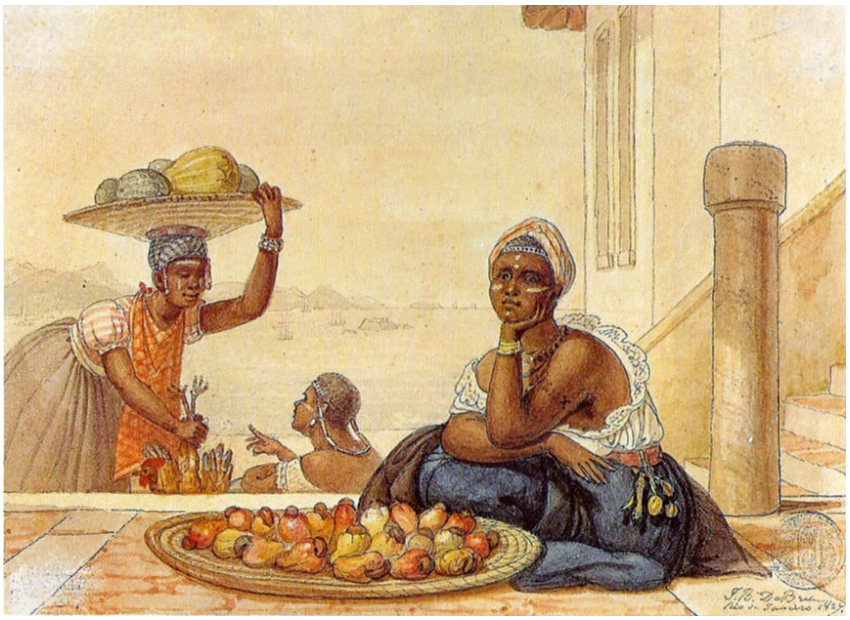


Figure 10: *Jean-Baptiste Debret "Tattooed black woman selling cashew fruits" (1827)*

In Rio Grande do Sul, the tradition of barbecue (*churrasco*), initially associated with gaucho culture, became emblematic of the region's culinary identity. As previously discussed, the development of *charqueadas* (salted meat processing sites) in Pelotas in the 19th century further consolidated the role of meat preservation and consumption in the local economy and food traditions (Palma et al., 2021). Regional meat consumption practices, particularly in Southern Brazil, were shaped by cattle ranching and became associated with both everyday meals and festive banquets (Cascudo, 1967).

The study of Brazilian culinary traditions reveals that its most iconic dishes are the result of centuries of cultural exchanges. Rather than being a product of a single group or moment in history, Brazilian cuisine embodies the complex interplay of Indigenous, African, and European influences, continuously evolving in response to social and economic changes (Elias, 2010). Cascudo's work on Brazilian cuisine emphasises adaptability, regional diversity, and the blending of ancestral food traditions with colonial innovations (Cascudo, 1967).

2.4. Brazilian pottery

From Indigenous communities to colonial settlements and Afro-Brazilian traditions, ceramics reflect evolving socio-economic conditions, technological adaptations, and cultural exchanges. Beyond its utilitarian role in food storage, preparation, and consumption, pottery serves as a key artefact for understanding the material realities of past communities, particularly through archaeological investigations (Symanski, 2010).

2.4.1. Indigenous pottery

Prior to European colonisation, Indigenous groups developed distinct pottery traditions that reflected regional adaptations and cultural expressions. These ceramics were often decorated, with motifs carrying symbolic and ritualistic meanings beyond their functional roles (Milheira and DeBlasis, 2011; Admiraal et al., 2023). Archaeological sites in Santa Catarina and Rio Grande do Sul reveal pottery fragments associated with both domestic and ceremonial use, indicating a reliance on both terrestrial and marine resources (Borba, 2013). Diagnostic ceramic assemblages from the Southern coast, including both Guarani and Taquara-Itararé styles, provide insight into long-term technological practices and shifting social structures.

The methods of Indigenous pottery-making varied across groups and regions, though most traditions relied on hand-building techniques such as coiling and modelling. Common tempering materials included sand, crushed shell, and mineral

fragments like feldspar and quartz, all selected to improve thermal resistance and overall durability (Milheira and DeBlasis, 2014; Admiraal et al., 2025). Firing was generally done in open bonfires, often resulting in uneven coloration and surface treatment, though some groups developed controlled firing practices to produce burnished or dark-smoked finishes. In many cases, the internal surfaces were carefully finished to prevent food absorption and enhance vessel performance (Ribeiro and Milheira, 2015).

Ceramics associated with the **Taquara-Itararé tradition**, linked to proto-Jê populations, began appearing in the Southern coast of Brazil around 1200 years ago and were widespread across both inland and coastal sites. These vessels are typically thin-walled, round-bottomed, and finely made, often exhibiting geometric decorations produced through incision, modelling, and punctuation (Figure 11).

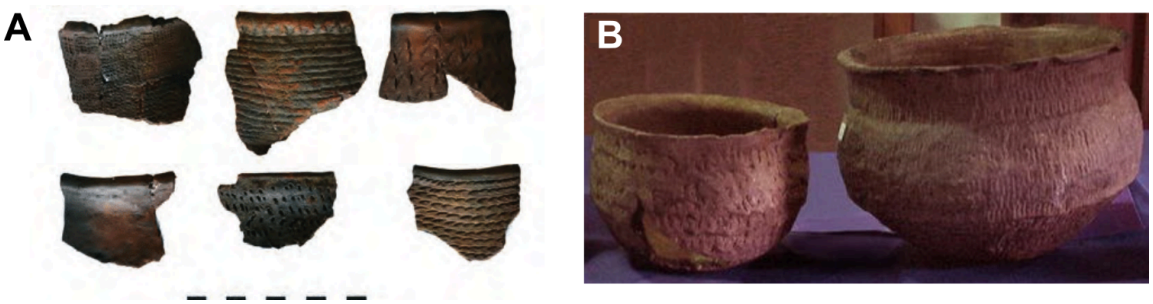


Figure 11: *(A) examples of Taquara-Itarare pottery (Faggi, 2015); (B) Guarani corrugated vessels (Milheira and DeBlasis, 2014)*

The shapes include cooking pots and storage jars, occasionally with pronounced rims and everted lips. Decoration appears to have served both aesthetic and communicative purposes, reinforcing group identity through shared stylistic codes (Milheira and DeBlasis, 2014).

The **Guarani tradition**, on the other hand, is characterised by thicker-walled vessels, often large and conical in shape, designed for storage, cooking, and possibly fermentation. These ceramics often exhibit red slip coatings, burnishing, and a wide

array of decorative motifs, including incised lines, corrugations, and finger impressions. Vessel forms include *Cambuchí* (large jars), bowls, and smaller pots suited for domestic use (Ribeiro and Milheira, 2015). In Guarani pottery, decoration was not merely ornamental, it was connected to social meaning, religious practice, and kinship systems. Some motifs appear consistently across wide geographical areas, suggesting shared ideological frameworks and strong inter-community ties. Notably, Guarani pottery from the Patos Lagoon region in Rio Grande do Sul presents a particularly rich typological diversity. Large conical jars with thick walls and flat bases are often found alongside smaller, more refined vessels, likely used for food serving or storage. These ceramics frequently display surface treatments such as burnishing or slip application, and decorations are concentrated around the rim and shoulder, areas most visible during handling and use (Ribeiro and Milheira, 2015; Borba, 2013). The placement and repetition of motifs suggest standardized conventions passed through generations of ceramicists, reinforcing both continuity and cultural memory (Ribeiro and Milheira, 2015).

While both Taquara-Itararé and Guarani ceramic traditions share certain techniques, such as coiling, low-temperature firing, and the use of local raw materials, they are typologically distinct. Taquara-Itararé pottery tends to be more delicate and uniform, reflecting perhaps a smaller scale of production oriented around household needs. Guarani ceramics, in contrast, point to larger-scale domestic production with more emphasis on large-volume storage and collective food preparation, possibly aligned with social practices like communal feasting (Milheira and DeBlasis, 2011; Admiraal et al., 2023). Ultimately, Indigenous pottery in Southern Brazil reflects a long-standing expertise in ceramic technology, adapted to different environments and cultural priorities. The diversity of forms, tempers, and surface treatments attests to a rich tradition of technical knowledge passed through generations. These ceramics serve not only as utilitarian tools but also as expressions of identity, territorial belonging, and cosmological meaning, anchoring material culture to broader Indigenous worldviews.

2.4.2. Historical pottery

The arrival of Portuguese settlers introduced new ceramic technologies, including the potter's wheel and glazed ceramics, which were incorporated into local traditions to create hybridized Luso-Brazilian styles (Borba and da Rocha Bandeira, 2013). In urban centres such as Pelotas and Florianópolis, ceramic production centres emerged in the 18th and 19th centuries, catering to both local needs and export markets. These workshops produced utilitarian ceramics like cooking pots, bowls, and water jars, many of which were adapted to local culinary practices (Figure 12). Glazed earthenware, tin-glazed ceramics, and painted faience became increasingly popular in urban homes, reflecting shifts in consumer preferences and technological advancements (Rodrigues, 2015).

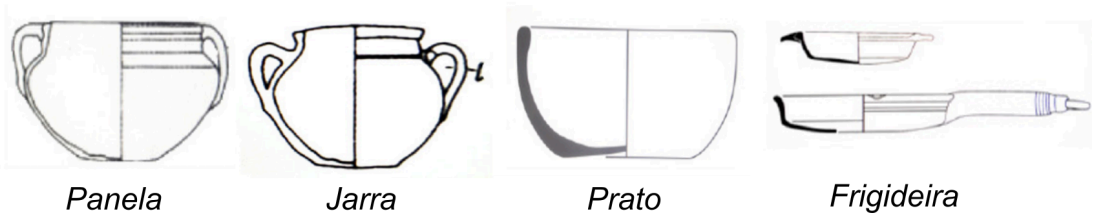


Figure 12: *Portuguese common styles of pottery (adapted from Noelli and Sallum, 2019)*

In Southern Brazil, archaeological investigations have highlighted the existence of two distinct yet often coexisting production traditions: wheel-thrown wares from formal olarias and handmade ceramics shaped using traditional techniques, often within domestic settings (Salvador, 2017). The coexistence of these traditions reflects a layered social landscape, where ceramic objects not only fulfilled utilitarian roles but also conveyed information about social hierarchy, ethnicity, and gendered divisions of labor. These differences were spatially encoded in the household: refined, wheel-thrown vessels (Figure 12) were typically used in dining areas, while hand-built pots with burnished or incised surfaces, common in Indigenous and Afro-Brazilian traditions, were relegated to kitchens and workspaces associated with enslaved and marginalised individuals (Salvador, 2017; Deminicis, 2017).

In addition to domestic production, historical records suggest that the ceramic industry in colonial Brazil was heavily influenced by trade networks that connected European markets with local artisanal production. Portuguese and Luso-Brazilian pottery often incorporated Indigenous and African influences, resulting in hybridized styles that catered to the specific culinary and storage needs of local communities (Symanski, 2010). Some workshop-produced vessels even show decorative patterns (e.g., *roletado*, *escovado*, *digitado*) linked to African-inspired motifs, suggesting that artisans and users both participated in the negotiation of transcultural forms (Salvador, 2017).

Pottery also played a crucial role in the material culture of enslaved African populations in Brazil. Enslaved individuals adapted local and African-inspired ceramic traditions for daily use, producing wide-mouthed cooking pots, water vessels, and coarse earthenware bowls that facilitated communal cooking and social interaction (Borba, 2013). These items, often produced in informal settings, provided enslaved individuals with a means of cultural expression and resilience under oppressive conditions. Some ceramic types associated with African traditions continued to be produced by Afro-Brazilian communities long after the abolition of slavery, maintaining their symbolic importance in Brazilian culture (Ribeiro, 2017).

In Santa Catarina and Rio Grande do Sul, these traditions are evident in archaeological contexts linked to both urban and rural Black communities, whose ceramics combine hand-built techniques with regionally distinctive stylistic elements (Salvador, 2017) (Figure 13). Further studies on ceramic composition and technological choices indicate that colonial pottery-makers adapted to Indigenous and African traditions in clay preparation, shaping techniques, and decorative motifs (Salvador, 2017).



Figure 13: *(A) Guarani ceramic fragment with polished red slip and refined internal surface. (B) Taquara-Itararé fragments with incised linear motifs typical of proto-Jê decorative style. Both images reproduced from Salvador (Salvador, 2017).*

Archaeological analysis suggests that Afro-Brazilian communities in Brazil continued using open-firing techniques and hand-built methods in producing domestic pottery, differentiating their ceramic styles from European wheel-thrown counterparts (Milheira and DeBlasis, 2014). These ceramics, frequently found in urban contexts such as Pelotas, Florianópolis, and Joinville, exhibit specific stylistic elements such as burnished surfaces, incised decorations, and tempering techniques designed for durability and heat resistance (Zanettini and Wichers, 2009). Many of these features align with what has been termed *cerâmica neobrasileira*, a broad typological category including hybrid forms developed in colonial contexts through the relationship of African, Indigenous, and European knowledge systems (Deminicis, 2017).

The production and use of ceramics in Afro-Brazilian communities also played a symbolic role in maintaining cultural identity. Some vessels have been linked to ritualistic and religious practices, demonstrating the role of pottery beyond its

utilitarian function. The persistence of these ceramic traditions throughout the post-colonial period underscores their importance in Afro-Brazilian material culture (Rodrigues, 2015). In Southern Brazil, where archaeological evidence supports the survival of Indigenous ceramic styles well into the 19th century, such traditions also reflect a resilient material vocabulary through which communities navigated and negotiated the pressures of colonial modernity (Salvador, 2017).

2.5. Historical archaeology in Brazil

Historical archaeology in Brazil has become an important field for examining colonial and post-colonial histories through material culture. Its contribution is particularly relevant for this thesis because it provides a framework for addressing inequality, labour, household practice, cultural interaction and the visibility of communities that are often underrepresented in written records.

2.5.1. The Beginning of historical archaeology in Brazil

Historical archaeology in Brazil emerged in the 1960s, influenced by prehistoric archaeology and the growing interest in cultural heritage management. Initially, research lacked differentiation in goals or methods compared to prehistoric studies but later developed into a distinct discipline emphasizing the historical and cultural particularities of the region (Funari, 1994; Symanski and Zarankin, 2014; Zarankin, 2012). Over the past two decades, the field has grown significantly, adopting theoretical frameworks rooted in contemporary social theory, focusing on inequality, cultural hybridity, and resistance (Symanski, 2009; Funari, Zarankin and Salerno, 2009).

Historical archaeology has also contributed to challenging dominant narratives of colonial and post-colonial history. One of the key contributions of Brazilian historical archaeology is its ability to challenge “master narratives” by addressing marginalised communities, such as enslaved peoples, Indigenous groups, and Afro-descendants (de Souza and Symanski, 2009; Symanski and Souza, 2007). These studies have revealed diverse perspectives on colonial and post-colonial histories, contributing to a more

nuanced understanding of Brazil's past (de Souza and Symanski, 2009; Symanski and Souza, 2007). Additionally, public archaeology and cultural heritage projects have fostered engagement with local communities, ensuring that these interpretations resonate beyond academia (Funari, 2004).

2.5.2. Colonial influences and the construction of national identity

The early focus of historical archaeology in Brazil centred on correlating material culture with documentary records to understand colonial interactions. For instance, studies of material remains from Jesuit missions in Rio Grande do Sul and Pernambuco States provided critical insights into Indigenous-European exchanges during the 16th to 18th centuries (Funari, 1999). These studies often reinforced narratives of colonial splendor but later evolved to include perspectives highlighting the resilience and agency of Indigenous groups.

The colonial period also shaped Brazil's national identity through its archaeological record. The material culture of Portuguese settlements, such as ceramics and architecture, symbolized the integration of European practices with local traditions. These artefacts were used not only for restoration projects but also to celebrate Brazil's colonial heritage during the post-independence period (Symanski, 2009; Lima, 1993). However, recent archaeological approaches have sought to dismantle such celebratory narratives by emphasizing the violence and exploitation inherent in colonial systems (Zarankin, 2012).

2.5.3. Material culture and the archaeology of enslaved populations

One of the most significant developments in Brazilian historical archaeology is the study of enslaved populations and maroon communities (*quilombos*). Studies highlight the variability in locally made ceramics across enslaved communities, showing how decorative patterns, production techniques, and vessel forms reflect cultural

hybridity and the process of creolization. For example, pottery in Chapada dos Guimarães transitioned from simpler designs during the early 18th century to highly decorative forms as new African identities emerged in Brazil. These changes reflect both the selective appropriation of colonial materials and the persistence of African cultural traditions. (de Souza and Symanski, 2009).

In addition to ceramics, archaeological investigations have uncovered evidence of internal economies within plantations, such as markets for enslaved individuals to purchase or trade goods. These findings challenge assumptions about enslaved populations' material scarcity, revealing their ability to maintain cultural identities and adapt to oppressive environments (Symanski, 2009; Funari, 1994). The visibility of enslaved groups in the archaeological record is critical for addressing questions of identity, agency, and resistance (de Souza and Symanski, 2009).

2.5.4. Contemporary advances and broader applications

In recent years, Brazilian historical archaeology has expanded its scope to include interdisciplinary approaches and marginalised perspectives. For instance, eco-historical archaeology examines the environmental impacts of colonial activities, such as deforestation and resource extraction, alongside their socio-cultural consequences (Costa, 2018). These studies highlight the interconnectedness of human actions and ecological transformations, particularly in regions like the Brazilian Amazon and sugar plantations in the Northeast (de Souza and Symanski, 2009).

Public archaeology has also played a vital role in promoting inclusive narratives and engaging descendant communities. The work of Funari on public archaeology emphasises the importance of making archaeological findings accessible and relevant to local populations (Funari, 2004). This approach has been instrumental in addressing issues of visibility and preservation, particularly for sites associated with enslaved groups (de Souza and Symanski, 2009). Additionally, the internationalization of Brazilian historical archaeology has fostered collaborations with global scholars, enriching theoretical frameworks and methodological approaches. Journals like

Vestígios: Revista Latino-Americana de Arqueologia Histórica have provided a platform for disseminating research across Latin America, further integrating Brazilian archaeology into broader academic debates (Funari, Zarankin and Salerno, 2009).

3. Organic residue and stable isotope analysis: a review

This Chapter outlines the methodological approaches used in this research to investigate culinary practices and resource use in Southern Brazil. It provides an overview of the chemical properties of lipids and stable isotopes, highlighting their relevance in archaeological and historical contexts and their potential to inform interpretations of food preparation, consumption and storage practices. The Chapter is organised into two main sections. The first focuses on organic residue analysis (ORA), describing the chemical characteristics of lipids, their preservation in archaeological pottery, and the analytical techniques used to extract, identify, and quantify them. Special attention is given to the interpretation of food-related biomarkers, including lipid markers associated with animal fats, plant products and aquatic resources, which have been particularly informative in archaeological contexts (e.g. Hansel and Evershed, 2009; Admiraal et al., 2023, 2025). While such approaches have greatly advanced our understanding of pre-colonial foodways in South America, their application to historical ceramics in the Neotropics remains rare. This study emphasises their untapped potential for exploring post-contact and colonial contexts across the region. The second section addresses stable isotope analysis (SIA) of bulk collagen, explaining the principles of isotopic fractionation and their application to palaeodietary studies. It also outlines the sampling protocols and analytical techniques employed in this research.

3.1. Organic residue analysis

ORA is a well-established scientific method in archaeology used to detect and interpret molecular traces of past biological materials preserved in various substrates, including ceramics, soils, tools and skeletal remains (e.g. Evershed et al., 2001b; Evershed, 2008; see also Polla and Springer, 2022 for a recent review). While ceramics are among the most commonly studied materials due to their widespread use and

porous structure, ORA is not limited to pottery and can be applied to a wide range of archaeological matrices. Pottery, in particular, has long been a key resource for reconstructing past human behaviours, initially through typological and stylistic analysis to investigate cultural identities, technological traditions, and exchange networks (Rice, 2015). The integration of ORA has added a biochemical dimension to these interpretations, by providing evidence for substances processed, stored or absorbed in archaeological materials.

Organic residues, comprising lipids, proteins, carbohydrates, and plant-derived compounds, can adhere to pottery surfaces or become absorbed into the ceramic matrix through prolonged exposure to food processing activities such as cooking, boiling, fermenting, or storing liquids and semi-solid substances (Craig et al., 2013a; Cramp and Evershed, 2014). Unlike macroscopic residues, which may degrade over time, absorbed biomolecules can persist within the ceramic structure for thousands of years, protected from environmental factors such as erosion and microbial activity (Regert, 2011). Their identification requires specialised chemical techniques, as they are not directly visible and cannot be inferred through typological studies alone.

Among the biomolecules preserved in archaeological ceramics, lipids are particularly resilient due to their hydrophobic and chemically stable nature, which enhances their survival in burial environments (Heron and Evershed, 1993). These compounds originate from a wide range of organic sources, including animal fats, plant oils, and waxes, and can provide valuable insights into food processing, subsistence strategies, and trade in perishable commodities. The application of techniques such as gas chromatography-mass spectrometry (GC-MS) and GC-combustion isotope ratio mass spectrometry (IRMS) enables archaeologists to distinguish between different sources of lipids and reconstruct ancient culinary practices with a high degree of specificity. By integrating ORA with other archaeological and environmental data, researchers can move beyond traditional pottery classification schemes to explore questions related to diet, subsistence, and cultural exchange, broadening our understanding of past societies and their interactions with food.

3.1.1. Lipids: chemical overview

Lipids are a diverse group of organic compounds primarily composed of carbon, hydrogen, and oxygen atoms (Fahy et al., 2011). These molecules serve essential biological functions such as energy storage and cell membrane structure (Fahy et al., 2009). In archaeological contexts, lipids are particularly valuable due to their chemical stability, which allows them to persist in artefacts such as pottery for millennia (Heron and Evershed, 1993; Charters et al., 1995; Evershed, 2008; Roffet-Salque et al., 2017a).

Lipids are generally classified based on their molecular structure, solubility, or physical state (Fahy et al., 2005). They include simple lipids, such as triglycerides and waxes, and complex lipids, which incorporate additional components like phosphates, carbohydrates, or proteins (Fahy et al., 2011). A third category, derived lipids, includes molecules like free fatty acids and alcohols that result from the hydrolysis or breakdown of simple or complex lipids (Fahy et al., 2009). A defining characteristic of lipids is their solubility in non-polar organic solvents, such as chloroform, benzene, and methanol, which has made their extraction and analysis feasible in archaeological studies (Fahy et al., 2011).

The physical properties of lipids vary according to their molecular structure. For example, oils and fats are distinguished by their physical state at room temperature, with oils generally liquid and fats generally solid. This difference is partly related to the degree of saturation of the fatty acids they contain. Saturated fatty acids contain only single carbon-carbon bonds, whereas unsaturated fatty acids contain one or more carbon-carbon double bonds. This affects molecular packing, melting behaviour and susceptibility to oxidation. Lipids are also characterised by polarity: polar lipids, such as phospholipids, contain hydrophilic groups, whereas neutral lipids, such as triacylglycerols, are predominantly hydrophobic (Fahy et al., 2005, 2009).

Fatty acids

N-alkanoic acids (fatty acids) (Figure 14) are the main constituents of lipids (Lund and Rustan, 2020). They are molecules consisting of an aliphatic chain of carbon

atoms, ending with an acidic carboxyl group (-COOH). While the hydrocarbon chain is typically linear, it can occasionally be branched or cyclic, adding to the structural diversity (Lund and Rustan, 2020). The classification of fatty acids depends on several factors. One of the key determinants is the chain length, which categorizes fatty acids as short-chain (≤6 carbons), medium-chain (7-12 carbons), or long-chain (≥13 carbons). As the chain length increases, the solubility of fatty acids in water decreases, and their melting points rise, making longer chains more solid at room temperature (Lund and Rustan, 2020).

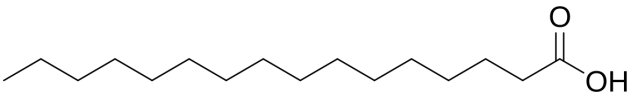


Figure 14: *Chemical structure of palmitic acid ($C_{16:0}$ - hexadecanoic acid)*

In archaeological lipid residues, the distinction between saturated and unsaturated fatty acids is particularly important for preservation. Saturated fatty acids, including palmitic acid ($C_{16:0}$) and stearic acid ($C_{18:0}$), are generally more stable and more frequently preserved in archaeological ceramics (Lund and Rustan, 2020). Unsaturated fatty acids (Figure 15), by contrast, are more susceptible to oxidation and degradation because of the presence of one or more carbon-carbon double bonds.

Unsaturated fatty acids can be further subdivided into monounsaturated fatty acids (MUFA), which contain one double bond, and polyunsaturated fatty acids (PUFA), which contain two or more double bonds. Oleic acid ($C_{18:1}$; cis-9-octadecenoic acid or (9Z)-octadec-9-enoic acid) is a common MUFA in olive oil and many vegetable oils, while linoleic acid ($C_{18:2}$; cis,cis-9,12-octadecadienoic acid or (9Z,12Z)-octadeca-9,12-dienoic acid) is a common PUFA in plant oils. Some PUFA, including linoleic acid, are essential fatty acids and are also important components of plant and fish oils (Lund and Rustan, 2020).



Figure 15: *Chemical structure of MUFA oleic acid ($C_{18:1}$ (9Z)-octadec-9-enoic acid) and PUFA linoleic acid ($C_{18:2}$ cis, cis-9,12-Octadecadienoic acid)*

The configuration of double bonds also plays a significant role in the classification of unsaturated fatty acids. In the *cis*-configuration, the hydrogen atoms attached to the carbons involved in the double bond are located on the same side, resulting in a bent molecular structure. This configuration is common in most natural unsaturated fats. Conversely, in the *trans*-configuration, the hydrogen atoms are on opposite sides of the double bond, leading to a straighter molecular structure. Trans fatty acids are rare in nature but are commonly produced during industrial hydrogenation processes (Lund and Rustan, 2020).

In archaeological studies, fatty acids are among the most commonly preserved lipids (Roffet-Salque et al., 2017a; Evershed, 2008; Craig et al., 2012). They are absorbed into the porous matrices of ceramic vessels during cooking and storage. Their analysis provides direct evidence of food processing, including the use of animal fats, plant oils, and aquatic resources. For example, the ratio of saturated to unsaturated fatty acids can help indicate the origin of the residues, while isotopic analysis can further differentiate between terrestrial and aquatic sources.

Mono, di and triacylglycerols

Triacylglycerols (TAGs) are among the most abundant natural lipids and are key components of animal fats and vegetable oils (Domínguez et al., 2022) (Figure 16). Structurally, TAGs consist of three fatty acid chains esterified to a glycerol backbone

(Domínguez et al., 2022). Depending on the type of fatty acids attached, TAGs can be classified as simple (with identical fatty acids) or mixed (with different fatty acids).

Monoacylglycerols (MAGs) and diacylglycerols (DAGs) are intermediates in lipid metabolism and play crucial roles in energy storage and membrane biosynthesis (Domínguez et al., 2022). MAGs contain a single fatty acid chain bound to a glycerol backbone, while DAGs have two. These molecules are essential in the biosynthesis of more complex lipids and serve as key intermediates in lipid metabolism (Domínguez et al., 2022). MAGs and DAGs are often found in degraded lipid residues and provide insights into the partial breakdown of TAGs in archaeological contexts (Evershed et al., 2002a; Cramp and Evershed, 2014; Drieu et al., 2025). Their presence may indicate partial hydrolysis of TAGs due to degradation processes or specific food preparation methods (Roffet-Salque et al., 2017a; Regert, 2011). Their analysis can help reconstruct past culinary practices and lipid preservation conditions.

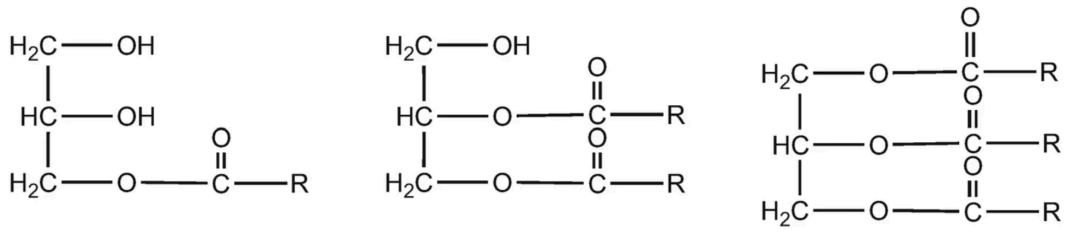


Figure 16: *General structures of monoacylglycerols, diacylglycerols and triacylglycerols, showing fatty acyl chains esterified to a glycerol backbone.*

TAGs exhibit distinct physical and chemical properties based on their fatty acid composition. For instance, those rich in saturated fatty acids form highly stable crystalline structures, resulting in solids at room temperature, such as animal fats. Conversely, TAGs with unsaturated fatty acids, particularly cis-configured double bonds, exhibit lower melting points and are typically liquid, as seen in vegetable oils (Domínguez et al., 2022).

In archaeological contexts, TAGs are frequently identified in ceramic residues, providing valuable insights into the types of oils and fats processed or stored in vessels (Dudd, Regert and Evershed, 1998; Steele, Stern and Stott, 2010; Regert, 2011). The presence of TAGs and their degradation products can support interpretations of lipid processing, particularly when considered alongside fatty acid profiles, biomarkers and isotopic data.

Isoprenoids: steroids and terpenes

Isoprenoids are a diverse class of lipids derived from isoprene units (C_5H_8) (Domínguez et al., 2022). This structural backbone gives rise to a vast array of molecules, including sterols, carotenoids, fat-soluble vitamins, and plant resins. Among isoprenoids, steroids and terpenes represent two major subclasses with distinct biological and functional roles. Steroids, including sterols, are essential structural components of membranes and metabolic precursors in animals and plants, while terpenes are primarily known for their aromatic properties, roles in plant defence, and medicinal applications (Wise, 1999; Domínguez et al., 2022). Despite their differences, both steroids and terpenes share a common biosynthetic origin from isoprene units, making them crucial for a wide range of physiological and ecological processes (Domínguez et al., 2022).

Steroids are a broad class of lipids characterised by a core structure of four fused rings (cyclopentanoperhydrophenanthrene). Among them, sterols form a distinct subclass, defined by the presence of a hydroxyl (-OH) group at the C_3 position, which makes them amphipathic molecules. This structural feature allows sterols to integrate into cell membranes, where they regulate fluidity, permeability, and cellular signalling (Pateiro et al., 2019; Domínguez et al., 2022).

In animal systems, *cholesterol* (Figure 17) is the most abundant sterol, primarily derived from dietary sources such as meat, dairy, and eggs. It plays an essential role in membrane integrity and serves as a precursor for bile acids, vitamin D, and steroid hormones (Björkhem, 2009; Domínguez et al., 2022). Archaeological lipid analysis

frequently detects cholesterol and its oxidation products in ceramic residues, indicating the processing of animal fats in food preparation and storage (O'Keefe, 2002). The identification of cholesterol in pottery provides direct evidence of dietary reliance on domesticated livestock and marine resources, as well as potential uses of animal lipids for illumination, waterproofing, or medicinal purposes (Hammann et al., 2018; Roffet-Salque et al., 2017a). However, it is important to consider that cholesterol may also derive from exogenous sources, including degraded animal remains in surrounding soils or soil-dwelling organisms, and should consequently be interpreted with caution and in context (Evershed, Heron and Goad, 1990) .

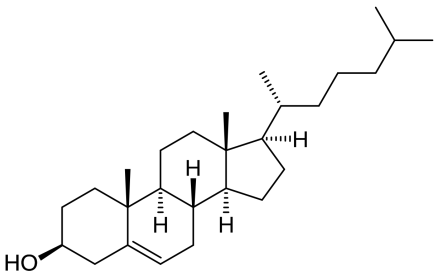


***Figure 17**: Chemical structure of cholesterol ($C_{27}H_{46}O$)*

In plants, *phytosterols*, such as *β-sitosterol, campesterol, and stigmasterol* (Figure 18), function similarly to cholesterol in animals, contributing to membrane stability. Phytosterols are usually found in vegetable oils, nuts, seeds, and whole grains, have been extensively studied for their ability to lower cholesterol absorption in humans (O'Keefe, 2002). Their presence in archaeological ceramics and organic residues is significant, as it suggests the processing of plant oils, medicinal extracts, and resin-based materials. Some phytosterol-containing substances, such as tree resins and medicinal salves, were likely used not only in food but also in ritual and therapeutic applications (Pateiro et al., 2019).

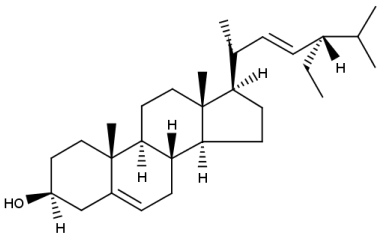


***Figure 18**: Chemical structure of stigmasterol ($C_{29}H_{48}O$)*

Terpenes, another major isoprenoid subclass, are primarily found in plants, fungi, and some marine organisms. Structurally, terpenes range from monoterpenes (C_{10}), found in herbs and citrus oils, to tetraterpenes (C_{40}), which include carotenoids and vitamin A precursors (Wise, 1999; Domínguez et al., 2022). These compounds not only enhance flavour and aroma in foods but also serve medicinal and technological functions.

Carotenoids (Figure 19), a group of tetraterpenes, provide pigmentation to fruits and vegetables such as carrots, tomatoes, and peppers. Of particular archaeological interest are diterpenoids and triterpenoids, which are major constituents of conifer resins and plant waxes. Diterpenoids such as abietic acid and its oxidation products, including dehydroabietic acid (DHA) and 7-oxo DHA, are commonly identified in archaeological ceramics and are often associated with the use of pine resins for vessel sealing, waterproofing, or as fuel (Hammann et al., 2020; Regert et al., 2003). Similarly, triterpenoids may point to the use of birch bark tar or frankincense-type resins, which played roles in craft production and ritual contexts (Evershed et al., 1997a). These compounds are highly resistant to degradation and thus provide robust biomarkers for non-culinary uses of pottery and organic coatings (Stern et al., 2003; Regert, 2011; Domínguez et al., 2022).

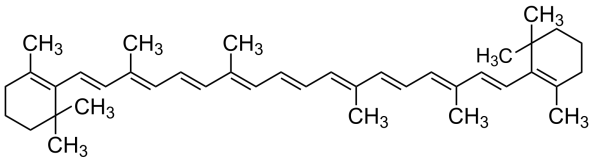


Figure 19: *Chemical structure of the terpene beta-carotene ($C_{40}H_{56}$)*

Wax esters

Waxes (Figure 20) are simple lipids composed of long-chain fatty acids esterified to long-chain alcohols (Domínguez et al., 2022). Due to their chemical structure, waxes are hydrophobic and malleable at room temperature, with melting points generally ranging between 40-90°C, depending on their molecular composition (Domínguez et al., 2022). These compounds are highly hydrophobic and function as protective coatings in plants, insects, and marine organisms. Their resistance to hydrolysis and oxidation makes them chemically stable, allowing them to persist in various environmental and archaeological contexts (Regert et al., 2003). Beeswax, one of the most well-documented sources of wax esters, consists predominantly of C_{40}-C_{52} wax esters, along with hydrocarbons, free fatty acids, and minor flavonoid-derived compounds (Tulloch, 1973). The unique composition of wax esters in beeswax, dominated by even-numbered carbon chains, serves as a valuable biomarker in archaeological lipid analysis (Regert et al., 2003).

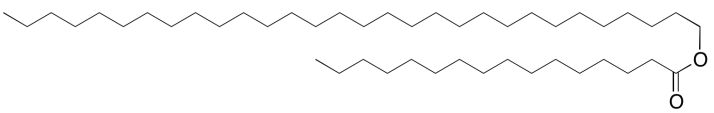


Figure 20: *Chemical structure of triacontanyl palmitate ($C_{46}H_{92}O_2$) wax ester - major component of beeswax*

In archaeology, wax esters are commonly identified in ceramic vessels, wooden artefacts, and preserved organic residues, providing insights into ancient technologies and culinary practices. The use of beeswax in pottery has been associated with waterproofing, adhesive applications, and medicinal uses. Chemical analysis of

Neolithic and Roman-period vessels has revealed the presence of wax esters alongside n-alkanes and free fatty acids, indicating the use of beeswax for various functions (Regert et al., 2003).

N-alkanes

Alkanes (Figure 21) are the simplest class of hydrocarbons, consisting only of carbon and hydrogen atoms arranged in single bonds. They follow the general molecular formula CnH_2n+2, meaning each carbon atom is fully saturated with hydrogen. This structural simplicity results in chemical stability, as alkanes exhibit relatively low reactivity due to the strength of their C-C and C-H bonds (Clayden, Greeves and Warren, 2012).



Figure 21: *Chemical structure of tetracosane ($C_{24}H_{50}$), a C24 n-alkane*

Alkanes can exist as linear, branched, or cyclic molecules. Their physical properties vary with chain length: shorter alkanes are gases at room temperature, medium-length alkanes are liquids, and longer chains form waxy solids. This variability influences their function in biological and archaeological contexts. In nature, n-alkanes are widely produced by plants, bacteria, and marine organisms, often forming protective wax layers on leaves or acting as metabolic byproducts in microbes. Their composition and distribution patterns can serve as biomarkers for distinguishing different plant sources (Clayden, Greeves and Warren, 2012). Archaeologically, n-alkanes can indicate the presence of plant materials used in food preparation or other cultural practices, such as fuel for cooking (Charters et al., 1993; Evershed, 2008; Cramp et al., 2014).

Lipid degradation and preservation

The preservation of lipids in archaeological ceramics is governed by a complex set of chemical and environmental variables. While lipids are more stable than proteins or carbohydrates due to their hydrophobic nature, they are nonetheless subject to degradation through processes such as hydrolysis, oxidation, and microbial activity (Evershed, 2008; Regert, 2011). These mechanisms alter the original composition of lipid residues and result in the formation of specific degradation products that serve as secondary biomarkers (Raven et al., 1997; Hansel, Bull and Evershed, 2011).

Hydrolysis is one of the primary mechanisms of lipid breakdown. It involves the cleavage of ester bonds in triacylglycerols (TAGs), producing free fatty acids (FFAs), monoacylglycerols (MAGs), and diacylglycerols (DAGs). This reaction is catalysed by heat and the presence of water or enzymatic activity, and is often intensified during cooking or early post-depositional processes (Dudd, Regert and Evershed, 1998; Evershed et al., 2002a)

Oxidation, particularly of unsaturated fatty acids, leads to the production of a variety of oxygenated degradation products, including ketones, α,ω-dicarboxylic acids (diacids), and hydroxy fatty acids. These compounds provide important markers for identifying thermal processing or prolonged exposure to oxygen-rich environments (Evershed et al., 1995; Regert et al., 1998; Hansel, Bull and Evershed, 2011). For example, odd-numbered mid-chain ketones (e.g., C_{29}-C_{35}) form through free radical-induced pyrolytic reactions and are typically observed when lipids are heated above 300°C.

In addition to chemical processes, microbial degradation plays a significant role, particularly in temperate and tropical burial environments. Lipids can be metabolized by soil microbes, which produce branched-chain fatty acids and sterol derivatives that complicate residue interpretation (Heron and Evershed, 1993; Dudd, Regert and Evershed, 1998). Preservation also depends on the distinction between free and bound lipids. Free lipids, loosely absorbed into the ceramic matrix, are more vulnerable to

leaching and microbial breakdown. In contrast, bound lipids are covalently or physically trapped within the ceramic's mineral structure, making them more resistant to post-depositional alterations and essential for long-term survival (Roffet-Salque et al., 2017a; Hammann et al., 2020). Environmental conditions such as soil pH, moisture, aeration, and temperature further influence lipid survival. While some studies suggest that acidic soils favour lipid preservation due to limited microbial activity, other research highlights the complexity of this relationship and emphasises that waterlogged, frozen, or arid environments offer the best overall preservation outcomes (Evershed, 1993; Regert et al., 1998; Copley et al., 2005a).

Ultimately, understanding the mechanisms of lipid degradation is essential for interpreting molecular profiles in archaeological ceramics. The presence, absence, or transformation of particular compounds not only reflects vessel use but also the physical and chemical histories of the objects themselves.

Lipid degradation products

Building on the overview of lipid degradation mechanisms and preservation factors, the following section examines the main chemical products that result from these processes, including ketones, dicarboxylic acids, and hydroxy fatty acids, and explores their significance in archaeological interpretations.

Ketones are a class of organic compounds characterised by the presence of a carbonyl functional group (C=O) bonded to two carbon atoms. They are commonly produced through the oxidation of secondary alcohols and play significant roles in both biological and industrial processes. In lipid chemistry, ketones can also be found as natural components of plant epicuticular waxes, serving protective functions against water loss and environmental stress (Evershed et al., 1995; Raven et al., 1997). However, in the context of archaeological residues, ketones are primarily formed as degradation products of lipids, particularly through thermal processing or oxidation of fatty acids. Because of their high stability, long-chain ketones are used as biomarkers

for identifying heat-altered lipids in pottery residues, providing valuable insights into past cooking and technological practices (Raven et al., 1997).

In archaeology, the presence of long-chain ketones in archaeological ceramics has been widely interpreted as evidence of repeated vessel heating and lipid processing. The characteristic 1:2:1 ratio of C_{31}, C_{33}, and C_{35} ketones has been observed in experimental studies, confirming their formation during intensive lipid heating in porous ceramic matrices (Matlova et al., 2017; Drieu et al., 2019). These compounds are particularly useful in distinguishing between vessels used for dry heating or roasting versus those used for boiling, where ketone formation is less likely. Interestingly, ketones have also been detected in vessels not directly associated with cooking, suggesting alternative functions such as waterproofing, post-firing oil treatments, or fuel processing (Longoni et al., 2024). The persistence of long-chain ketones in lipid residues provides an essential tool for reconstructing ancient culinary practices, vessel heating techniques, and post-production treatments in ceramic manufacture (Longoni et al., 2024).

Dicarboxylic acids (diacids) are a class of organic compounds containing two carboxyl (-COOH) functional groups, which makes them more polar and reactive than their monocarboxylic counterparts (Figure 22). These compounds play an essential role in lipid oxidation and degradation processes, both in biological and archaeological contexts. Chemically, α,ω-dicarboxylic acids form through a sequence of reactions involving hydroxylation, oxidative cleavage, and ω-oxidation of unsaturated fatty acids (Passi et al., 1993). Their formation is often associated with the oxidative breakdown of lipids, particularly polyunsaturated fatty acids (PUFAs), which are highly susceptible to autoxidation (Frankel et al., 1977). Due to their high stability, dicarboxylic acids can persist in ancient residues, providing valuable markers for lipid degradation and post-depositional processes in organic materials (Hansel, Bull and Evershed, 2011).

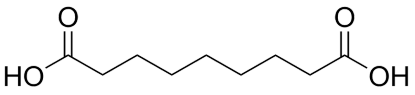


Figure 22: *Chemical structure of azelaic acid ($C_9H_{16}O_4$; C_9 dicarboxylic acid; nonanedioic acid).*

In archaeological studies, α,ω-dicarboxylic acids are frequently identified in ceramic lipid residues, where they serve primarily as indicators of lipid oxidation and degradation rather than source-specific biomarkers (Regert et al., 2003; Demirci et al., 2021). The presence of diacids in archaeological pottery suggests the prolonged exposure of lipids to oxygen, heat, or microbial activity, indicating that vessels were used to process or store lipid-rich substances. In particular, C_9 to C_{11} dicarboxylic acids are often detected in marine-derived lipid residues, whereas C_7 to C_{14} diacids have been linked to the degradation of terrestrial fats and plant oils (Hansel, Bull and Evershed, 2011). The detection of α,ω-dicarboxylic acids in ceramic vessels also suggests that they were exposed to high-temperature processing, potentially used for rendering fats, boiling oils, or heating waxy substances. These oxidation markers provide an important tool for reconstructing ancient food practices and the technological applications of lipids in early societies (Hansel, Bull and Evershed, 2011).

Hydroxy fatty acids are oxygenated derivatives of fatty acids characterised by the presence of one or more hydroxyl (-OH) functional groups attached to their hydrocarbon chains. These compounds arise from enzymatic oxidation, lipid autoxidation, or microbial metabolism and play crucial roles in biological membranes, signalling pathways, and lipid metabolism (Hansel, Bull and Evershed, 2011). They can be categorized into monohydroxy, dihydroxy, and polyhydroxy fatty acids, depending on the number of hydroxyl groups. One of the most studied subgroups is vicinal dihydroxy fatty acids, such as 9,10-dihydroxystearic acid, which are oxidation products of monounsaturated fatty acids, preserving valuable information about lipid degradation pathways (Hansel, Bull and Evershed, 2011).

In archaeological contexts, hydroxy fatty acids are useful indicators of lipid oxidation and degradation processes involving plant oils, animal fats and marine lipids. Their detection in ceramic residues provides information on lipid transformation and preservation rather than direct source identification on its own (Hansel, Bull and Evershed, 2011). These compounds are often found as part of the bound phase in potsherds and may reflect polymerised lipid residues that underwent long-term oxidative change. The position of hydroxyl groups can help identify the original unsaturation site of precursor lipids, but interpretations of marine or terrestrial contributions should be made cautiously and in combination with other molecular, isotopic and archaeological evidence.

3.1.2. Interpreting molecular and isotopic data: analytical approaches

The identification of organic compounds in archaeological materials requires the integration of multiple analytical techniques, each providing distinct chemical signatures that help classify and compare residues with reference substances. This multi-method approach is essential for obtaining a complete characterisation of the complex mixtures present in ancient ceramics and for evaluating both the potential and limitations of ORA (Oudemans, 2007; Mazzeo, 2017).

Gas chromatography (GC) is one of the most widely used techniques for analysing organic residues in archaeological ceramics (Evershed, Heron and Goad, 1990; Evershed, 2008). It allows for the separation of volatile and semi-volatile compounds, which can then be detected and analysed using different methods depending on the analytical goal. Two of the most common detection techniques employed in ORA are flame ionisation detection (GC-FID) and Mass Spectrometry (GC-MS). While GC-FID provides quantitative analysis of organic compounds, GC-MS enables structural identification of individual molecules (Dudd, Regert and Evershed, 1998; Regert, 2011). These complementary methods are often used together to maximise analytical accuracy and obtain a more detailed molecular profile of

archaeological residues (Heron and Evershed, 1993; Colombini, 2009).

In addition to these methods, Gas chromatography-combustion-isotope ratio mass spectrometry (GC-C-IRMS) is increasingly used in ORA to measure compound-specific stable isotope ratios of the two main fatty acids (stearic and palmitic), helping to distinguish between different biological sources such as terrestrial vs. marine resources or C_3 vs. C_4 plants (Craig et al., 2013a; Evershed et al., 1997b; Dunne et al., 2012).

GC-FID: quantitative analysis of organic compounds

Gas chromatography-flame ionisation detection (GC-FID) is a widely used quantitative detection method for organic compounds containing carbon-hydrogen (C-H) bonds. The flame ionisation detector (FID) operates by burning organic compounds in a hydrogen-air flame, producing ions and free electrons, which generate an electrical current. The ionisation current is directly proportional to the quantity of carbon present, making GC-FID a powerful tool for precise quantification of organic compounds (Sparkman, Penton and Kitson, 2011). Since the ionisation current is proportional to the number of carbon atoms in a molecule, the technique provides an accurate measurement of compound concentration.

However, FID does not generate mass spectral data, meaning it cannot provide information about the molecular structure of an analyte (Sparkman, Penton and Kitson, 2011). GC-FID is in fact primarily used for quantification rather than structural identification. For this reason, GC-FID is often used alongside GC-MS, where FID provides quantitative data, and MS is responsible for compound identification.

GC-MS: structural identification of compounds

Gas chromatography-mass spectrometry (GC-MS) is a widely used analytical technique that combines the separation capabilities of gas chromatography (GC) with the identification and structural elucidation potential of mass spectrometry (MS).

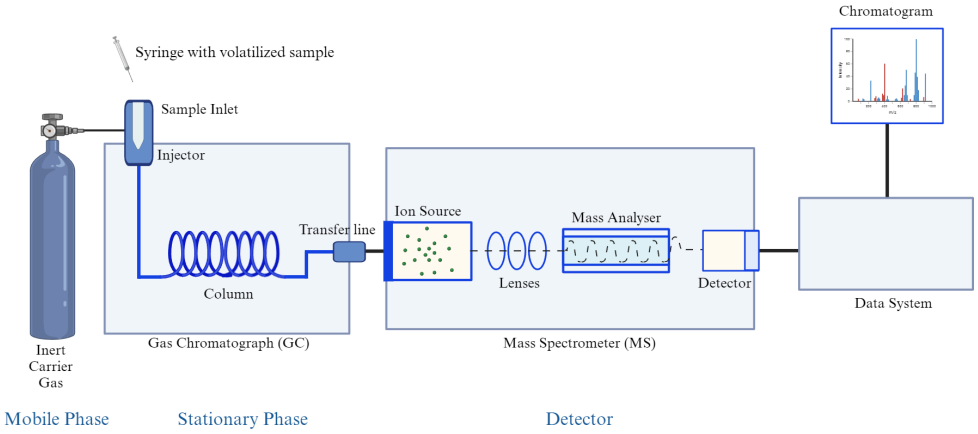


Figure 23: *Schematic representation of a GC-MS system, showing the key components involved in compound separation, ionisation and detection.*

A typical GC-MS system consists of a gas chromatograph (GC) for compound separation, a mass spectrometer (MS) for detection, and a computerized data processing system (Figure 23). The GC unit includes an injector, a capillary column housed in a temperature-controlled oven, and a carrier gas system. The mass spectrometer comprises an ionisation source, a mass analyser (such as a quadrupole, ion trap, or time-of-flight), and a detector that records the mass-to-charge ratio (m/z) of fragment ions. These components work together to provide precise chemical identification and quantification of organic compounds (Sparkman, Penton and Kitson, 2011).

The gas chromatography component of the technique is responsible for the separation of complex mixtures of volatile and semi-volatile compounds. This process relies on the differential interaction of analytes with the stationary phase of a capillary column and a carrier gas, typically helium or hydrogen, which serves as the mobile phase (Sparkman, Penton and Kitson, 2011). The sample is introduced through an

injector, where it is vaporized before entering the column. As the sample passes through the column, its components interact with the stationary phase based on their physicochemical properties, such as volatility and polarity. Less volatile compounds, or those with greater affinity for the stationary phase, exhibit longer retention times, while more volatile compounds elute earlier (Sparkman, Penton and Kitson, 2011). The column is housed within an oven that operates under a controlled temperature program, allowing for improved separation efficiency by gradually increasing the temperature to facilitate the elution of compounds with higher boiling points (Sparkman, Penton and Kitson, 2011).

After separation, the analytes enter the mass spectrometer, where they undergo ionisation. As compounds elute from the column at different retention times, they are detected, generating a chromatogram, a graphical representation of signal intensity over time. Each peak in the chromatogram corresponds to a separated compound, which is then analysed by the mass spectrometer for identification and structural elucidation. The most commonly used ionisation method in GC-MS is electron ionisation (EI), in which high-energy electrons collide with the molecules, generating charged fragments (Sparkman, Penton and Kitson, 2011). These fragments then pass through the mass analyser, which sorts them based on their mass-to-charge ratio (*m/z*). The most frequently used mass analysers in GC-MS include quadrupole, time-of-flight (TOF), and ion trap systems, each offering specific advantages in terms of resolution, sensitivity, and speed (Sparkman, Penton and Kitson, 2011). Once separated, the ions are detected, and the resulting data is converted into a mass spectrum, which provides a unique fingerprint for each compound. This spectral information allows for the identification of unknown substances by comparing the obtained spectra with reference databases such as the NIST or Wiley mass spectral libraries (Sparkman, Penton and Kitson, 2011). One of the key advantages of GC-MS is its high sensitivity, enabling the detection of trace amounts of compounds in complex mixtures. This makes it particularly useful in fields requiring precise chemical identification, such as toxicology, environmental monitoring, and food safety testing (Sparkman, Penton and Kitson, 2011).

In archaeology, GC-MS has proven to be a useful method in the identification of organic residues preserved in pottery and other porous materials. The technique allows for the detection of lipid biomarkers such as saturated and unsaturated fatty acids, sterols, terpenes, wax esters, and specific degradation products like ω-(o-alkylphenyl) alkanoic acids (APAAs), which can be associated with marine resources or thermally processed plant and animal fats (Hansel et al., 2004; Evershed, 2008; Regert, 2011). Through its application in case studies across Europe, Africa, and the Americas, GC-MS has enabled archaeologists to reconstruct ancient dietary practices, vessel use, and food processing techniques (Dudd and Evershed, 1998; Dunne et al., 2012; Drieu et al., 2021a).

GC-c-IRMS

A major advancement in organic residue analysis has been the application of Gas chromatography-combustion-isotope ratio mass spectrometry (GC-c-IRMS) to archaeological ceramics (Matthews and Hayes, 1978; Evershed et al., 1994, 1997b). This method separates individual molecular components, oxidises them to CO_2, and measures the $^{13}C/^{12}C$ ratio for each compound. It has been applied in determining the isotopic composition of fatty acids, particularly palmitic acid ($C_{16:0}$) and stearic acid ($C_{18:0}$), which are the most commonly found in ORA (Evershed et al., 1994, 1997b; Dudd, Regert and Evershed, 1998). During analysis, gas chromatography (GC) isolates compounds, which are then combusted to CO_2, with water removal to prevent the formation of interfering ions (Evershed et al., 1994). The resulting ionised CO_2 is analysed by mass spectrometry to determine the isotopic composition relative to the VPDB standard.

The ability of GC-c-IRMS to distinguish between different food sources relies on the inherent isotopic variation among plants and animals. These variations, driven by photosynthetic pathways and metabolic processes, provide critical insights into dietary choices and resource use in past societies. The following section outlines the chemical principles underlying these isotopic measurements, providing the broader context for their application in organic residue analysis.

Stable isotope analysis in organic residues

The integration of SIA into organic residue research has significantly contributed to the understanding of past culinary practices by helping identify the original contents processed in archaeological pottery. In this context, stable isotope measurements are typically applied through compound-specific isotope analysis (CSIA), which targets individual lipids such as fatty acids and sterols to provide insights into their biological and environmental origins. While this section focuses on the role of isotopes specifically within organic residue analysis, a broader and more detailed overview of isotopic principles, including their applications to bulk materials such as bone collagen, is provided in the following section on stable isotope analysis in bulk collagen.

Stable isotopes are atoms of the same chemical element that share the same number of protons and electrons, but differ in the number of neutrons in their nuclei, resulting in differences in atomic mass (Ellam, 2016; Hoefs, 2021). While this variation does not significantly affect their chemical reactivity, since reactions are governed by electron configurations, it does influence the physical behaviour of isotopes during natural processes. These mass-dependent differences give rise to isotope fractionation, where lighter isotopes tend to react or diffuse slightly faster than their heavier counterparts (Fry, 2006). Fractionation processes can be biological (e.g., photosynthesis, metabolism), chemical (e.g., precipitation reactions), or physical (e.g., evaporation, diffusion), and result in subtle but measurable differences in isotopic composition between materials or compartments. These differences, in turn, become informative tracers of environmental and biological processes in archaeological materials (Mariotti, 1983; Bronk Ramsey, 2008).

Fractionation is typically expressed as the ratio of the heavier to the lighter isotope of a given element (e.g., $^{13}C/^{12}C$ for carbon), and this ratio is reported relative to an international reference standard using the delta (δ) notation:

$$\delta X = (R_{sample}/R_{standard} - 1) \times 1000\ (‰)$$

Here, X represents the heavier isotope (e.g., ^{13}C for carbon), and R denotes the ratio between heavier and lighter isotopes in both the sample and the reference standard (Hoefs, 2021). For carbon, the Vienna Pee Dee Belemnite (VPDB) standard is commonly used (O'Leary, 1988; Coplen, 1995; Hoefs, 2021). Less negative $\delta^{13}C$ values indicate enrichment in the heavier isotope (^{13}C), while more negative values reflect relative depletion, corresponding to a higher abundance of ^{12}C (O'Leary, 1988). For example, C_3 plants typically have $\delta^{13}C$ values between -20‰ and -35‰, whereas C_4 plants fall between -9‰ and -16‰, reflecting distinct photosynthetic pathways (O'Leary, 1988; Fry, 2006; Hoefs, 2021). In archaeological ceramics, to support interpretation, $\delta^{13}C$ values of fatty acids ($C_{16:0}$ and $C_{18:0}$) were compared to modern reference datasets for plant, animal and marine lipids. These were visualised as 68% confidence ellipses (Figure 24), derived from published studies and experimental reference samples (appendix 7). These insights, especially when combined with lipid biomarker analysis, enhance the specificity of dietary reconstructions, allowing researchers to infer not only what was cooked in vessels, but also how it was processed and consumed in different cultural settings.

Compound-specific isotope analysis of fatty acids

Carbon isotope fractionation in plants results from differences in photosynthetic pathways, leading to distinct $\delta^{13}C$ signatures (Tieszen, 1991). The $\delta^{13}C$ values of plant lipids, particularly n-alkanoic acids such as $C_{16:0}$ and $C_{18:0}$, are preserved during cooking and degradation, making them reliable proxies for identifying plant inputs in organic residue analysis (Evershed, 2009). The Calvin cycle, used by C_3 plants, preferentially incorporates ^{12}C, yielding relatively negative $\delta^{13}C$ values.

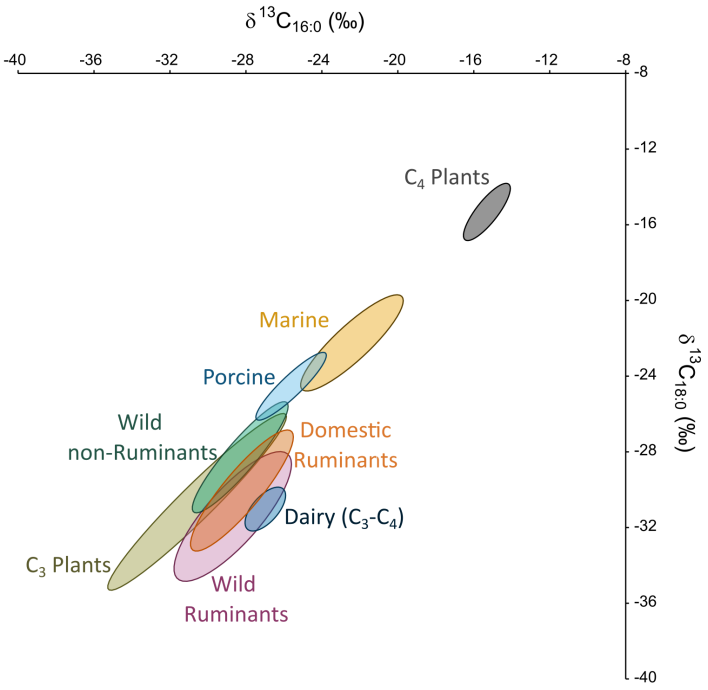


Figure 24: *Reference δ¹³C values of $C_{16:0}$ and $C_{18:0}$ fatty acids for various plant and animal products, based on modern reference datasets (Appendix 7). The ellipses represent 68% confidence intervals and were used in this thesis to interpret archaeological lipid residues by comparison with known isotopic signatures of C₃ and C₄ plants, marine resources, dairy, and different types of animal fats.*

In contrast, C₄ plants, which follow the Hatch-Slack pathway, show a preference for ¹³C, resulting in more positive δ¹³C values (Spangenberg and Ogrinc, 2001). Plants using crassulacean acid metabolism (CAM) exhibit a broad range of δ¹³C values due to their ability to switch between C₃ and C₄ pathways (O'Leary, 1988). The bimodal distribution of δ¹³C values in C₃ and C₄ plants allows archaeologists to infer the use of plant-derived resources in ceramic vessels (Figure 24 and 25). The reference values used in this study and represented in Figures 24 and 26 are primarily derived from modern datasets established in European or global settings, which have proven effective in distinguishing major food groups such as C₃ vs. C₄ plants, ruminant vs. non-ruminant fats, and marine vs. terrestrial inputs. However, given the regional specificity of this study, efforts were made to incorporate relevant reference data from

South America. For instance, previous studies analysed modern maize (*Zea mays*) and manioc (*Manihot esculenta*) cultivated in Southern Brazil, providing $\delta^{13}C$ values that align with known C_4 and C_3 ranges, respectively (Admiraal et al., 2023). These comparative values enhance the relevance of the isotopic framework for interpreting residues recovered from the study region. Early studies by Evershed et al. (Evershed et al., 1994) demonstrated that $\delta^{13}C$ analysis of long-chain lipids could identify the processing of specific plant taxa, such as Brassica species, in medieval pottery. More recent work has combined lipid biomarkers with isotopic analysis to confirm the presence of millet, a C_4 crop, in ancient food residues (Heron et al., 2016).

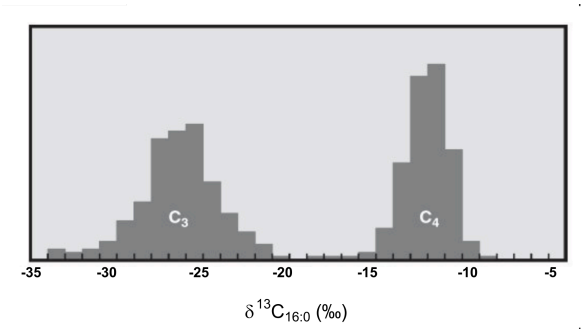


Figure 25: *Comparison of plant C_3 and C_4 characteristic $\delta^{13}C$ ranges (Evershed, 2009).*

Carbon isotope signatures of animals reflect their diets, with herbivores incorporating the $\delta^{13}C$ values of the plants they consume (DeNiro and Epstein, 1978; Tieszen, 1991). The digestive processes of ruminants and monogastric animals lead to characteristic differences in the $\delta^{13}C$ values of fatty acids (Evershed et al., 1997b). Ruminants, such as cattle, sheep, and goats, synthesize palmitic acid ($C_{16:0}$) primarily from acetate produced by microbial fermentation in the rumen, whereas stearic acid ($C_{18:0)}$ is mainly derived from the hydrogenation of dietary unsaturated fatty acids (Craig et al., 2012; Copley et al., 2003). In contrast, non-ruminants like pigs incorporate both acetate and glucose into lipid biosynthesis, resulting in more ^{13}C-enriched fatty acids overall (Dudd, Evershed and Gibson, 1999; Vernon, 1980). By measuring the $\delta^{13}C$ values of both $C_{16:0}$ and $C_{18:0}$, it is possible to distinguish between different animal fat sources when compared with modern reference datasets (Dudd and Evershed, 1998; Copley et al., 2003). The resulting isotopic offset, expressed as $\Delta^{13}C$ ($\delta^{13}C_{18:0}$ - $\delta^{13}C_{16:0}$), typically

falls below -1‰ for ruminant adipose fats, while values more negative than -3.3‰ are generally associated with dairy fats (Mottram et al., 1999; Evershed et al., 1997b) (Figure 26)

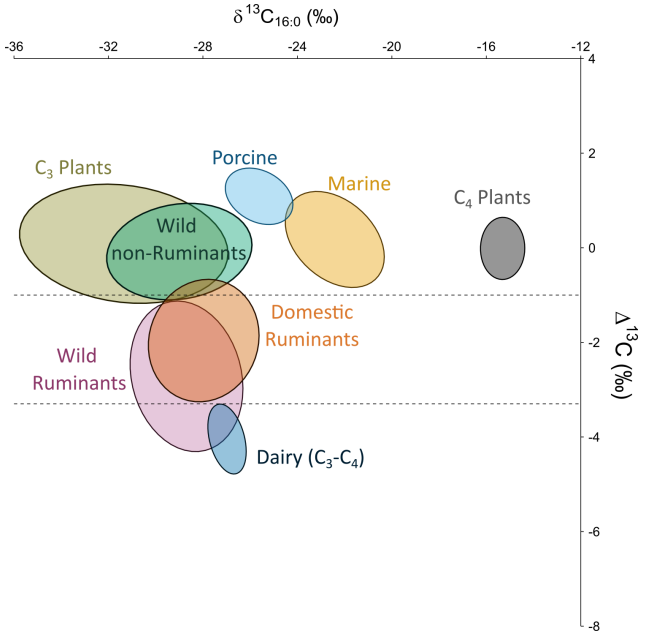


Figure 26: *Reference δ¹³C values of C16:0 and* $\Delta^{13}C_{18:0 - 16:0}$ *values for various plant and animal products, based on modern reference datasets (Appendix 7). The ellipses represent 68% confidence intervals and were used in this thesis to interpret archaeological lipid residues by comparison with known isotopic signatures of C₃ and C₄ plants, marine resources, dairy, and different types of animal fats.*

Another important application of this technique is the attempt to distinguish between adipose and dairy products. In dairy-producing animals, the metabolic routing of fatty acids during lactation differs from that of adipose tissue: $C_{18:0}$ in milk lipids tends to reflect the animal's diet, while $C_{16:0}$ is synthesized de novo in the mammary gland from acetate (Evershed et al., 2002b, 2002a; Copley et al., 2003). This leads to particularly negative $\Delta^{13}C$ values (-4/-8 ‰) in dairy fats compared to adipose fats from the same species (Figure 26). When interpreted cautiously and in conjunction with modern references from the same region, this offset can also be used to explore the presence of dairy fats in archaeological ceramics. In addition, comparisons with reference values may help differentiate between ruminant and non-ruminant fats and

have also been applied to tentatively identify poultry fats (Evershed et al., 2001a; Colonese et al., 2017).

Marine ecosystems exhibit distinct $\delta^{13}C$ signatures compared to terrestrial ecosystems, due to differences in carbon sources (Figure 26). Marine organisms tend to be enriched in ^{13}C partly because of the isotopic fractionation that occurs when atmospheric CO_2 dissolves in seawater and forms bicarbonate ions, which are then used in photosynthesis (Chisholm, Nelson and Schwarcz, 1982), allowing for their distinction from terrestrial resources in isotopic analyses (Shoda et al., 2018; Lucquin et al., 2016a). However, the overlap between marine and C_4 plant $\delta^{13}C$ values introduces interpretative challenges that require careful consideration (Heron et al., 2016; Courel et al., 2020; Admiraal et al., 2023; Casanova et al., 2022b). In such cases, contextual data and complementary lipid biomarkers are often necessary to support interpretations of marine resource use in archaeological ceramics.

3.1.3. Interpreting food biomarkers in archaeology: the archaeological biomarker concept

The archaeological biomarker concept was formalized in the 1990s when Evershed highlighted the value of integrating biomolecular and archaeological approaches to interpret organic remains (Evershed, 2008). Biomarkers, chemically distinct molecules, or suites of molecules (fingerprints) derived from specific biological sources, can persist in archaeological contexts and, when studied alongside archaeological data, help reconstruct past human-environment interactions, culinary practices, and technological choices. This concept includes a range of biomolecules, including lipids, proteins, and carbohydrates, although lipids remain the best-preserved and most frequently studied class (Stacey, 2009; Evershed, 2008).

Among archaeological materials, pottery plays a key role in biomarker research due to its abundance and physical structure. The porosity of ceramics allows organic compounds to be absorbed and retained within the matrix, while the hydrophobic nature of lipids enhances their preservation (Roffet-Salque et al., 2017a; Stacey, 2009).

Lipids absorbed during cooking or storage can become embedded within the ceramic's micropores, where they are shielded from microbial and chemical degradation. These absorbed residues are often better preserved than surface deposits, which are more susceptible to cleaning, erosion, or leaching (Dudd, Regert and Evershed, 1998). Importantly, absorbed organic residues may derive not only from culinary practices, such as boiling, roasting, fermenting, or storing food, but also from non-culinary uses including vessel sealing, surface treatments, or even repair (Charters et al., 1997; Stacey, 2009).

Experimental studies have shown that lipid concentrations vary within vessels depending on cooking methods and vessel shape. Boiling fatty substances tends to concentrate lipids near the rim, while roasting or dry-heating leads to more uniform lipid absorption throughout the vessel (Figure 27) (Charters et al., 1997; Stacey, 2009). Such patterns, however, are also influenced by other factors, including vessel reuse, sealing practices, and depositional environments, complicating simple functional interpretations.

To successfully identify and quantify food biomarkers, researchers must understand the processes that alter residues. These include pre-burial processes like heating and cleaning, post-depositional factors such as microbial activity and water exposure, and modern contamination from excavation or storage (Stacey, 2009). For example, lipid degradation may result in hydrolysis or oxidation of fatty acids, leading to the formation of new compounds, such as ketones and alkylphenols, which are diagnostic of thermal processing (Raven et al., 1997; Evershed, 2008). Conversely, the disappearance of unsaturated fatty acids is often due to their susceptibility to oxidation or microbial degradation (Solazzo and Erhardt, 2007).

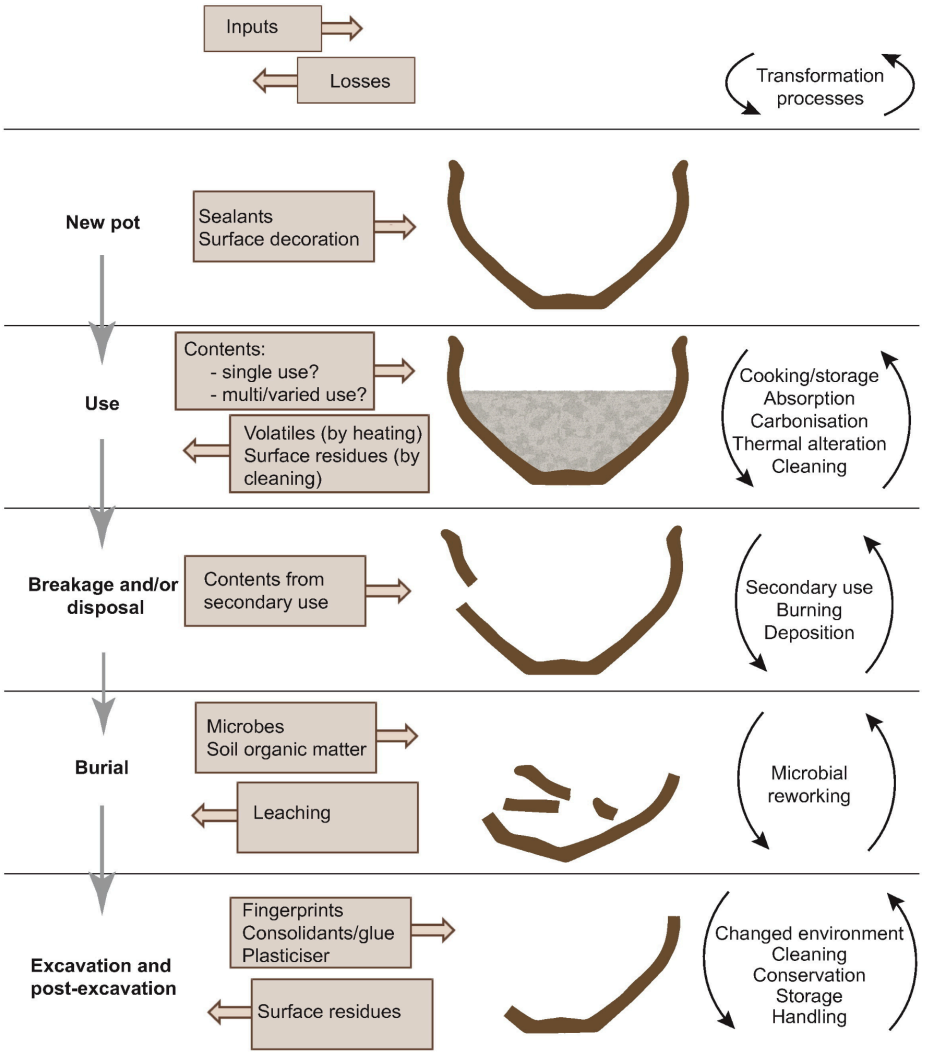


Figure 27: *Overview of the main processes influencing the preservation and composition of absorbed organic residues in archaeological ceramics, including inputs, losses, and transformation stages throughout the vessel's lifecycle (adapted from Roffet-Salque et al., 2017a).*

As outlined above, archaeologists apply a range of techniques to detect and interpret these residues, including gas chromatography, mass spectrometry (GC-MS), isotope ratio mass spectrometry (IRMS), and in some cases, proteomic or immunological methods for detecting proteins (Craig et al., 2000; Solazzo et al., 2008). GC-MS provides molecular fingerprints of lipid residues, including their degradation products, while GC-C-IRMS helps distinguish between animal carcass fats and dairy products through $\delta^{13}C$ values of individual fatty acids (Dudd and Evershed, 1998; Evershed, 2008). While

carbohydrate and protein residues are less commonly preserved, being more hydrophilic and more vulnerable to microbial attack, emerging research demonstrates that under exceptional conditions, such as anaerobic or frozen environments, protein residues can be identified (Craig et al., 2000; Solazzo et al., 2008). Specific instrument settings applied in this study are explained in the following Chapter (Chapter 4).

The following section reviews the main food-related biomarkers relevant to archaeological pottery, with particular attention to the resources considered in this project in Southern Brazil.

Terrestrial Animal Fats

Animal fats are among the most commonly detected organic residues in archaeological ceramics, due to their high lipid content and frequent use in food preparation. Even when degraded, they can be traced through specific molecular markers and stable isotope signatures, offering insight into culinary practices and resource exploitation in the past (Dudd and Evershed, 1998; Regert, 2011).

Triacylglycerols (TAGs), the primary constituents of fresh animal fats, offer valuable insights into ancient dietary practices when preserved in archaeological ceramics. Although TAGs are prone to degradation, advancements in high-temperature gas chromatography-mass spectrometry (HT-GC-MS) have enhanced our ability to detect and analyse these compounds (Evershed et al., 2002a; Mirabaud, Rolando and Regert, 2007). The specific profiles of TAGs can be diagnostic of the fat's origin, distinguishing between ruminant adipose tissue, dairy fats, and non-ruminant sources. Incorporating TAG analysis into lipid residue studies thus provides a more nuanced understanding of ancient food processing and consumption (Evershed et al., 2002a).

The main preserved constituents of degraded animal fats are saturated fatty acids, especially palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids. Their relative abundance, often expressed as the P/S ratio (palmitic/stearic), helps distinguish between fat types: a ratio below 1 suggests ruminant adipose tissue, while values above 1 may indicate non-ruminant or dairy fats (Romanus et al., 2007; Baeten et al., 2013). However,

degradation processes can lead to the preferential loss of shorter-chain acids like $C_{16:0,}$ complicating interpretation (Dudd, Regert and Evershed, 1998; Evershed et al., 2002a).

Unsaturated fatty acids, especially $C_{18:1}$, are generally less abundant due to oxidation, but their positional isomers can provide information about the origin: ruminants show a broader range of $C_{18:1}$ isomers (Regert, 2011; Evershed et al., 1997b).

Degraded fats also contain a variety of secondary products. Branched-chain fatty acids such as iso and anteiso $C_{15:0}$ and $C_{17:0}$ are typically associated with bacterial activity in the rumen, and thus with ruminant fats, although they can also be produced in horses and microbes (Heron and Evershed, 1993). Because no single compound is fully diagnostic, confident interpretations are based on a combination of lipid classes and contextual evidence (Roffet-Salque et al., 2017a).

Thermal processing also produces distinct biomarkers. Dicarboxylic acids (diacids) and hydroxy/dihydroxy fatty acids can result from the oxidation of unsaturated fatty acids, and their presence often indicates heating (Regert et al., 1998; Copley et al., 2005b). Similarly, odd-numbered mid-chain ketones (C_{29}-C_{35}) form through pyrolytic decarboxylation reactions at temperatures exceeding 300 °C. Their distribution across vessel surfaces can reflect specific cooking practices, such as boiling in the upper parts of the pot or roasting over direct heat (Evershed et al., 1995; Cramp and Evershed, 2014; Roffet-Salque et al., 2017a).

Finally, sterols such as cholesterol, while present in animal tissues, are rarely preserved due to cooking and degradation. When detected, they may appear alongside other degradation products such as squalene, a sterol common in fingerprints, requiring careful consideration of potential contamination (Evershed, 1993; Hammann et al., 2018).

Plant oils

Although plant-derived products have long been central to subsistence strategies in many ancient societies, their detection in archaeological ceramics is often hindered by their relatively low lipid content and the tendency for animal fats to dominate mixed residues. This is particularly relevant in regions like Brazil, where plant resources have historically played a major dietary and cultural role (Saint-Hilaire, 1936).

Two main lipid classes are typically recovered from plants: oils and waxes. Plant oils are composed primarily of triacylglycerols (TAGs), but these are highly prone to hydrolysis and oxidation, particularly due to their high content of unsaturated fatty acids. As a result, the most commonly preserved components in archaeological contexts are free fatty acids (FAs), especially oleic ($C_{18:1}$), linoleic ($C_{18:2}$), and linolenic acid ($C_{18:3}$) (Heron and Evershed, 1993; Baeten et al., 2013). The relative abundance of these FAs can sometimes offer clues to specific oil types, such as olive oil, typically rich in $C_{18:1}$ or linseed oil, which contains higher levels of $C_{18:2}$ and $C_{18:3}$. However, oxidative degradation can significantly alter these profiles, making direct identification challenging (Copley et al., 2005b). Oxidation of unsaturated fatty acids also leads to the formation of diagnostic degradation products such as α,ω-dicarboxylic acids (diacids) and dihydroxy fatty acids. Azelaic acid (C_9 diacid), for example, is a major product of oleic acid oxidation. Some hydroxylated compounds, such as 11,12-dihydroxyeicosanoic acid and 13,14-dihydroxydocosanoic acid, are more specific to oils from Brassicaceae plants like mustard, turnip, or radish. Similarly, 12-hydroxyoctanoic acid is a known marker for castor oil (Copley et al., 2005b; Romanus et al., 2007).

Recent experimental work has also demonstrated that ω-(o-alkylphenyl) alkanoic acids (APAAs), especially the C_{18}, can form from the heating of various plant oils (Figure 28). While APAAs were once associated primarily with aquatic fats, their formation under experimental cooking conditions from purely vegetal sources indicates that their presence is not diagnostic on its own (Bondetti et al., 2021). In particular, Bondetti and colleagues also demonstrated that the E/H isomer ratio of APAA-C_{18} offers

novel potential for distinguishing plant types. In the experimental reference dataset, APAA-C_{18} E/H ratios above 4 are associated mainly with some non-leafy and cereal-derived plant products, whereas several other starchy or non-leafy resources, including beans and cassava, fall below this threshold. For this reason, the threshold is used here as a plant-processing proxy and not as a definitive distinction between plant categories (Figure 28).

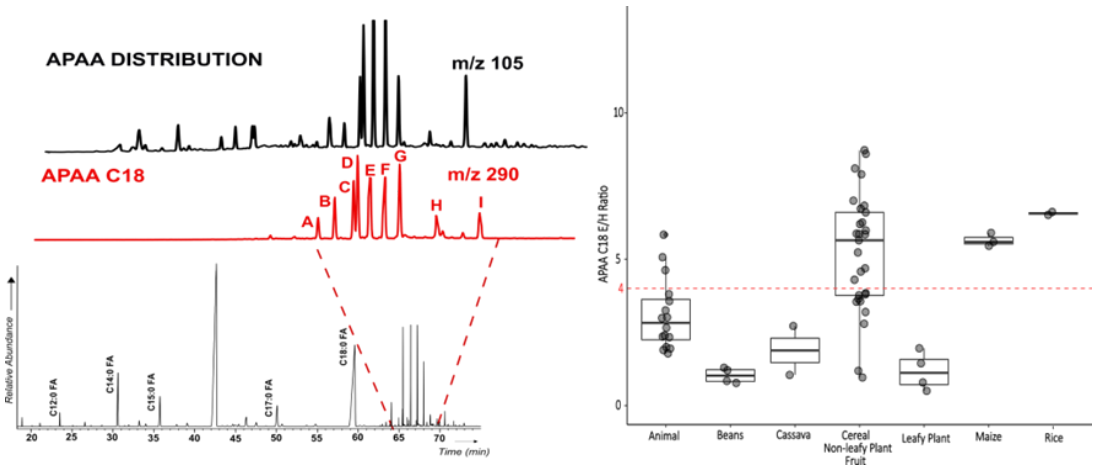


Figure 28: *Chromatographic identification and distribution of ω-(o-alkylphenyl) alkanoic acids (APAAs), with a focus on APAA-C_{18}, alongside E/H isomer ratio values from experimental reference samples (Appendices 3 and 7). The chromatograms on the left display diagnostic ion traces (m/z 105 and 290) used to identify APAAs, with peaks corresponding to the elution of APAA-C_{18} isomers highlighted. The dashed line marks an APAA-C_{18} E/H value of 4. In this thesis, values above this threshold are treated as evidence compatible with plant processing under the experimental framework of Bondetti et al. (2021), rather than as a definitive distinction between plant categories; some starchy or non-leafy resources, including beans and cassava, may fall below this value.*

These findings offer new avenues for detecting plant processing in ceramic vessels, especially when supported by additional biomarkers and contextual data. In tropical settings like Brazil, the diversity of lipid-rich native vegetation is remarkable. Historical sources such as Saint-Hilaire and Johann Pohl documented the dietary use of oily fruits like *feijoa, açaí, acerola, caju, piquiá, marolo,* and *macaúba*. Other notable plants include purslane, chicha, and a variety of palms, all known for their lipid content.

Additionally, starchy crops like cassava (manioc) and yams were widely processed into flours and fermented liquids such as tucupi, which are also potential sources of absorbed lipids (Saint-Hilaire, 1936).

Among the preserved saturated fatty acids, palmitic acid ($C_{16:0}$) is particularly stable and found in both plant and animal fats. The palmitic/stearic acid (P/S) ratio, expressed as $C_{16:0}/C_{18:0}$, has been explored as a tentative indicator of plant oil residues. Ratios above 3.0 may suggest plant inputs, while values between 1.0 and 2.0 are more typical of degraded animal fats (Mottram et al., 1999; Copley et al., 2005a; Evershed, 2008). However, this ratio is not definitive and must be interpreted cautiously, as it can be influenced by degradation, thermal processing, and the presence of aquatic fats (Regert, 2011; Roffet-Salque et al., 2017a).

Plant waxes, or epicuticular waxes, form protective layers on leaves, stems, and fruits. They include long-chain aliphatic compounds such as odd-numbered n-alkanes (C_{21}-C_{37}), n-alkanols, n-ketones, and wax esters (Eglinton and Hamilton, 1967). The specific composition of plant waxes is often species-dependent, with a dominance of n-alkanes around C_{29} and C_{31} typical of terrestrial plants (Baeten et al., 2013).

Recent studies have explored the potential of long-chain *n*-alkanols, particularly *n*-dotriacontanol (C_{32}), as compound-specific biomarkers for the identification of maize (*Zea mays*) in archaeological pottery. It was demonstrated that *n*-dotriacontanol is a major constituent of maize kernel waxes and is relatively rare in non-maize plants, making it a promising candidate for maize detection when combined with $\delta^{13}C$ analysis (Reber et al., 2004). This compound, which is part of the epicuticular wax layer, was shown to preserve well in archaeological ceramics and to retain the characteristic C_4 isotopic signal of maize even in the presence of mixed residues. However, as Reber and Evershed (2004) later cautioned, the detection of maize is strongly affected by degradation, and its identification through lipid analysis alone remains challenging, especially when maize is mixed with other high-lipid foods that mask its signature (Reber and Evershed, 2004). Despite these limitations, *n*-dotriacontanol offers a

valuable tool for maize identification in ceramic residues, particularly when contextual isotopic and archaeobotanical data support its presence.

As discussed, interpreting isotopic signals from plant-derived residues is particularly complex. The $\delta^{13}C$ values of C_3 plants often overlap with those of freshwater and terrestrial ruminant fats, while C_4 plants can yield values similar to marine organisms or non-ruminant animals (Steele, Stern and Stott, 2010). Moreover, the mixing of plant and animal products within a vessel can significantly skew $\delta^{13}C$ values. Experimental work with simulated mixtures (e.g., dairy with barley) has shown that the resulting isotopic signatures can mimic pure animal fats, further complicating interpretation (Hendy et al., 2018).

Aquatic oils

Aquatic organisms, both marine and freshwater, contain distinctive lipid profiles marked by high levels of polyunsaturated (PUFAs) and monounsaturated fatty acids (MUFAs), as well as isoprenoid fatty acids. In modern contexts, these profiles are relatively easy to characterise (Ackman and Hooper, 1970), but their identification in archaeological ceramics remains challenging due to degradation processes.

The unsaturated fatty acids that define aquatic fats degrade rapidly during vessel use and burial, particularly through oxidation (Heron, Evershed and Goad, 1991; Evershed et al., 2008b). However, oxidation can lead to the formation of diagnostic degradation products such as dicarboxylic acids (diacids) and vicinal dihydroxy fatty acids (DHYAs). Some DHYAs, like 9,10-dihydroxypalmitic, 9,10-dihydroxyarachidic, and 11,12-dihydroxydocosanoic acids, originate from MUFAs (e.g., $C_{16:1}$, $C_{18:1}$, $C_{20:1}$) that are abundant in aquatic organisms, offering indirect evidence of their original presence (Copley et al., 2005b; Hansel, Bull and Evershed, 2011).

Isoprenoid fatty acids (IFAs), including phytanic acid, pristanic acid, and 4,8,12-trimethyltetradecanoic acid (TMTD), are more stable due to their branched molecular structure and often survive better than PUFAs. These compounds form from phytol, a constituent of chlorophyll, which is abundant in aquatic organisms (Cramp

and Evershed, 2014). However, IFAs are not exclusive to marine or freshwater sources. For instance, phytanic acid is also found in ruminant and dairy fats, complicating interpretations (Brown et al., 1993).

Building on the discussion of plant-derived APAAs, particular attention has been given to longer-chain ω-(o-alkylphenyl) alkanoic acids (APAAs), especially APAA-C_{20} and APAA-C_{22}, because these compounds can derive from polyunsaturated fatty acids that are abundant in aquatic organisms (Hansel et al., 2004; Cramp and Evershed, 2014). Experimental research has demonstrated that these compounds can form under realistic cooking conditions, such as boiling in ceramic pots over open fires, and that their formation may be enhanced by the catalytic role of the ceramic matrix (Bondetti et al., 2021). A useful metric for assessing possible aquatic inputs is the ratio of APAA-C_{20} to APAA-C_{18} (APAA-$C_{20/C18}$). Values greater than 0.06 have been associated with aquatic commodities in the experimental dataset of Bondetti et al. (2021), but this threshold is used here as an aquatic-compatible proxy rather than as an exclusive or definitive aquatic marker.

For this reason, APAA-$C_{20/C18}$ values above 0.06 are interpreted only in combination with lipid concentration, chromatographic profile, other molecular indicators, CSIA results and archaeological context. When long-chain APAAs occur alongside additional aquatic-compatible indicators, such as isoprenoid fatty acids including phytanic and pristanic acid, the evidence for aquatic resource processing is strengthened, although it remains proxy-based rather than source-diagnostic on its own (Craig et al., 2007; Bondetti et al., 2021).

Compound-specific isotope analysis (CSIA) of palmitic and stearic acid can also help to better distinguish aquatic products. Marine organisms typically exhibit enriched $\delta^{13}C$ values compared to terrestrial species. This isotopic signal is primarily due to differences in carbon sources, marine food webs are based on bicarbonate (HCO_3^-) rather than atmospheric CO_2, leading to a higher incorporation of ^{13}C into fatty acids (Schoeninger and DeNiro, 1984). However, some overlap exists between marine fats

and non-ruminant terrestrial fats, which can complicate interpretation. In such cases, combining isotopic data with molecular biomarkers (e.g., isoprenoid fatty acids or APAAs) is critical for confident identification.

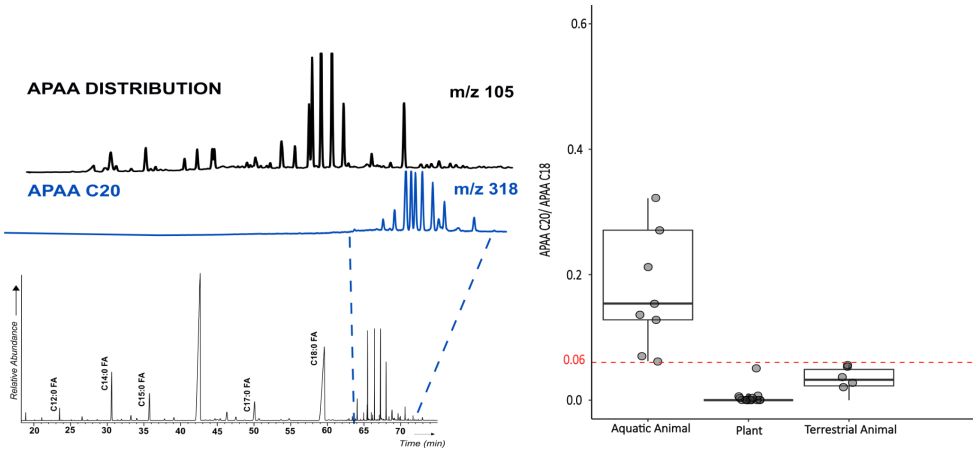


Figure 29: *Chromatographic identification and distribution of ω-(o-alkylphenyl) alkanoic acids (APAAs), with a focus on APAA-C_{20}, alongside APAA-$C_{20/C18}$ values from experimental reference samples. The chromatograms on the left display diagnostic ion traces (m/z 105 and 318) used to identify APAAs, with peaks corresponding to the elution of APAA-C_{20} isomers highlighted. The dashed red line marks the APAA-$C_{20/C18}$ threshold value of 0.06. In this thesis, values above this threshold are treated as aquatic-compatible proxy evidence, to be interpreted alongside lipid concentration, molecular profiles, CSIA results and archaeological context.*

While more isotopic reference data are being developed globally, particularly for marine and freshwater species, datasets for tropical and South American contexts remain limited. Expanding these regional reference frameworks is essential to improving the resolution of isotopic interpretations for aquatic lipid residues in archaeological pottery from coastal and estuarine environments, such as those in Southern Brazil.

3.1.4. Challenges of organic residue analysis in neotropical contexts

The application of Organic residue analysis (ORA) in tropical South American contexts presents several methodological challenges that affect both the recovery and interpretation of molecular data. One major limitation is environmental preservation.

The hot, humid, acidic, and biologically active conditions of neotropical environments accelerate lipid degradation, particularly of labile compounds such as polyunsaturated fatty acids. As a result, only the most stable lipids, such as saturated long-chain fatty acids, are likely to survive burial (Heron and Evershed, 1993; Dudd, Regert and Evershed, 1998; Evershed, 2008). Even these can be transformed by oxidation into degradation products, further complicating interpretation (Regert et al., 1998; Copley et al., 2005c). Consequently, ceramic vessels from rainforest or coastal settings frequently yield only trace residues, often insufficient to distinguish between different foodstuffs.

A second challenge lies in the limited availability of reference datasets for Indigenous South American plants and animals. Many native taxa remain chemically uncharacterised, forcing researchers to rely on generic or extraregional lipid profiles, which may lead to misidentifications (Lantos et al., 2024). For instance, distinguishing marine lipids from those of C_4 plants like maize is notoriously difficult, as both can produce similarly enriched $\delta^{13}C$ values. This is particularly problematic in coastal Brazil, where diets historically included both marine and maize-based resources (Schoeninger and DeNiro, 1984; Bastos et al., 2014). Recent studies have shown that the use of compound-specific isotope analysis, calibrated against local faunal baselines, can help disentangle such overlaps (Bondetti et al., 2021), but the absence of regionally relevant biomolecular references continues to limit interpretive confidence.

A third difficulty concerns the detection of starchy crops such as manioc. These plants contain little lipid content, and what they do contain degrades quickly or is easily masked by higher-fat ingredients. Experimental studies have shown that maize residues are rarely preserved beyond a few months of burial, and manioc, rich in carbohydrates and water, typically leaves no lipid trace at all (Reber and Evershed, 2004; Lantos et al., 2015).

To address these limitations, scholars increasingly advocate for integrated, multi-analytical approaches. These include the use of GC-MS profiling,

compound-specific isotope analysis, and targeted biomarker ratios such as the C_{20}/C_{18} ω-(o-alkylphenyl) alkanoic acid index, which can help distinguish aquatic from terrestrial resources (Bondetti et al., 2021). Experimental studies replicating tropical taphonomic conditions, such as humidity, pH, and microbial degradation, are also essential to understand the diagenesis of native food residues (Reber and Evershed, 2004). Recent research in tropical colonial settings, such as Isla de Mona in the Caribbean, demonstrates that even under poor preservation conditions, ORA can still successfully detect labile plant residues like manioc and fermented beverages such as wine, provided targeted biomolecular approaches are employed (Briggs et al., 2023).

Ultimately, building comprehensive reference libraries for South American resources, especially for lesser-known fruits, roots, and palms, is crucial. Combined with contextual data and microfossil analyses, these methods will enhance our ability to reconstruct foodways in tropical environments. Nevertheless, interpretations must remain cautious: under such preservation-challenging conditions, absence of evidence does not equate to evidence of absence.

3.1.5. Food biomarkers in South American archaeology

In South America, the application of ORA has expanded over the past two decades, offering new insights into food processing, dietary strategies, and cultural resilience. The following overview highlights key regional case studies that demonstrate the potential of lipid biomarker analysis to illuminate subsistence patterns, technological adaptation, and economic transformation from pre-colonial to colonial periods.

In recent years, ORA has gained increasing relevance in Brazilian archaeology, particularly in the study of pre-colonial societies across diverse ecological and cultural contexts. Notable examples include research on the Cerritos mound builders of Southern Brazil, where lipid residues from pottery demonstrated the specialised processing of estuarine fish and fermented plant products, likely linked to seasonal feasting practices (Admiraal et al., 2025). In coastal Guarani settlements, ORA has also

been employed to trace shifts in subsistence strategies, revealing a transition from marine-based diets to maize-dominated economies, as reflected in the organic signatures recovered from ceramic vessels (Admiraal et al., 2023).

Studies in the Amazon and northern Brazil have identified lipid residues indicative of diverse subsistence strategies that integrated both local and introduced species (Lantos et al., 2024). Research in pre-colonial pottery has shown evidence of marine and plant-based products, reinforcing the notion of mixed subsistence economies (Hansel et al., 2006). Additionally, dihydroxy acids have been identified as biomarkers for the processing of marine resources, demonstrating long-term reliance on fish and other seafood (Hansel and Evershed, 2009).

In coastal Brazil, research has explored the long-term resilience of subsistence strategies, demonstrating that pottery technology was integrated into existing maritime foodways without immediately replacing them (Colonese et al., 2014). These studies highlight the adaptability of Indigenous communities to environmental and cultural changes across different regions of Brazil.

The Andean region has also been important for ORA, particularly regarding maize, an essential staple. Studies in western Tinogasta, Argentina, identified maize starch and lipid biomarkers in pre-Hispanic pottery, revealing its role in food and beverage production, particularly in chicha fermentation (Lantos et al., 2015). Further south, an ORA study in La Ciénega valley, Argentina, analysed ceramic residues from early agricultural societies (200 BCE-900 CE), detecting biomarkers associated with maize and other plant-based resources, alongside limited evidence of animal fats (Fiorani et al., 2022).

In Chile, research in Araucanía examined organic residues in pottery from the El Vergel cultural complex, revealing the consumption of both marine and terrestrial resources, including C_3 and C_4 plants (Montalvo-Cabrera et al., 2024). Another study in Southern Chile found that maize was not a dietary staple but was likely used in fermented beverages consumed in feasting contexts (Montalvo-Cabrera et al., 2024).

Further evidence from Mocha Island highlights mixed subsistence strategies combining marine resources and cultivated plants, showing how island economies adapted to their environmental constraints (Montalvo-Cabrera et al., 2024).

In Argentinian Patagonia, a study using $\delta^{13}C$ and $\delta^{15}N$ values from ceramic residues demonstrated regional variations in food processing, with some vessels showing evidence of animal fat cooking, while others reflected plant-based diets (Chaile et al., 2018). Further studies in the region highlight the role of pottery in food processing among hunter-gatherer groups, showcasing the adaptability of these communities to diverse environments. Organic residue analysis has also identified mixed diets, suggesting a combination of marine and terrestrial resources in different ecological zones. These findings contribute to broader discussions on long-term human-environment interactions in Patagonia (Chaile et al., 2018).

Recently, studies in the Caribbean have been a focal point of ORA studies related to historical contexts and European colonisation. A study on Isla de Mona, Puerto Rico, analysed ceramic residues using gas chromatography-mass spectrometry (GC-MS) and compound-specific isotope analysis (GC-C-IRMS). The findings revealed the predominance of plant residues, with evidence of cassava processing and the earliest detection of wine residues in the Americas, reflecting early colonial food exchanges (Briggs et al., 2023). This study highlights the potential of ORA in uncovering early colonial foodways and interactions between Indigenous and European culinary traditions.

The studies reviewed demonstrate the growing impact of ORA in Latin American archaeology, illustrating regional dietary patterns, cultural exchanges, and economic transformations over time. However, a critical gap remains in the study of organic residues from historical and colonial contexts. While pre-colonial ORA research has advanced considerably, studies focusing on the material culture of post-contact periods, including interactions between Indigenous and European foodways, remain scarce. Addressing this gap is essential for understanding culinary and economic

transformations in Latin America's early modern history. Future research integrating multiproxy approaches and expanding datasets will continue to refine our understanding of Latin America's diverse culinary past. Expanding ORA applications to historic and colonial archaeological sites will provide new perspectives on trade, adaptation, and cultural resilience in food practices during and after European colonisation.

3.2. Stable isotope analysis of bulk collagen

Bulk collagen stable isotope analysis is a well established method in bioarchaeological research, enabling the reconstruction of past diets by examining the ratios of carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) isotopes preserved in bone collagen (Richards and Hedges, 1999; Hedges and Reynard, 2007; O'Connell and Hedges, 1999; Schwarcz and Schoeninger, 1991; Craig et al., 2013b). Because collagen is a durable, protein-rich component of the skeletal system, it retains biochemical signatures of dietary protein sources, offering a time-averaged reflection of an individual's nutritional intake over several years (DeNiro and Epstein, 1978; Ambrose and Norr, 1993). This approach is especially powerful for investigating subsistence strategies, ecological adaptations, and food web dynamics across archaeological populations and periods (Katzenberg and Waters-Rist, 2008; Hedges and Reynard, 2007; Toso, 2018). As the main structural protein in bone, collagen is biochemically stable and metabolically conservative, meaning it remodels slowly over time. This characteristic allows collagen to record an integrated isotopic signal of dietary protein consumption accumulated over the final years of an individual's life (Bocherens and Drucker, 2003; Ambrose and Norr, 1993). Unlike other tissues that may reflect short-term dietary changes, collagen provides a long-term average that is especially useful in identifying broad dietary patterns. These isotopic profiles can be compared across individuals of different ages, sexes, or social groups, and between sites, enabling researchers to assess both intra- and inter-population variability (Hedges and Reynard, 2007). In addition, collagen isotopes are valuable for tracing diachronic changes in subsistence practices associated

with cultural transitions, migration, or environmental shifts (Richards, Fuller and Molleson, 2006; O'Connell and Hedges, 1999).

In this project SIA was employed as a complementary approach to ORA in order to enhance the interpretation of lipid residues and broaden the scope of dietary reconstruction. Recent studies emphasise that ORA of pottery can reveal aspects of ancient food consumption that other methods (including bone isotope analysis) cannot (Craig et al., 2011; Regert, 2011). By using multiple proxies, the analysis leverages the strengths of both techniques: bone collagen $\delta^{13}C$ and $\delta^{15}N$ values reflect a long-term average of an individual's dietary protein intake (Hedges and Reynard, 2007), whereas lipids and molecular residues preserved in ceramics provide more context-specific information about foods processed in particular vessels (Lucquin et al., 2016b). Combining these methods helps overcome the limitations of each single approach and yields a more comprehensive understanding of past foodways (Craig et al., 2011; Lucquin et al., 2016b).

Additionally, isotopic analysis of faunal remains was conducted to establish local ecological baselines and to explore ancient animal feeding strategies. Defining a faunal baseline is crucial for contextualizing human isotope values (Schwarcz and Schoeninger, 1991), as environmental variations and animal diets can shift $\delta^{13}C/\delta^{15}N$ signatures at the base of the food web. By measuring contemporary animal bones from the same archaeological context, we can account for local plant $\delta^{13}C$ (e.g. C_3 vs. C_4 vegetation) and other ecological factors when interpreting human collagen values (Schwarcz and Schoeninger, 1991). This approach also provides insight into husbandry and ecology, for instance, revealing whether domesticated animals were consuming C_4 crops or marine resources, which in turn aids the interpretation of residue evidence (Lucquin et al., 2016b). In sum, integrating SIA with ORA (and anchoring interpretations with faunal isotope baselines) produces a more robust, multi-scalar reconstruction of historical diet and resource use, an approach increasingly advocated in archaeological science (Craig et al., 2011; Regert, 2011).

Stable carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) isotopes serve as key indicators of dietary inputs in archaeological populations. Carbon isotope values primarily reflect the types of plants consumed, either directly or indirectly through the food chain, by distinguishing between photosynthetic pathways, namely C_3 and C_4 plants (Schoeninger and DeNiro, 1984; Bocherens and Drucker, 2003). In contrast, nitrogen isotopes increase with trophic level due to metabolic fractionation, offering insights into the proportion of animal versus plant protein in the diet. When analysed together, $\delta^{13}C$ and $\delta^{15}N$ values allow researchers to assess variability in dietary patterns, including differential use of terrestrial, marine, and freshwater resources. Moreover, isotopic data from associated fauna can be used to establish local baselines and infer subsistence practices such as hunting, fishing, and animal husbandry (Hedges and Reynard, 2007; Schoeninger and DeNiro, 1984).

A major strength of bulk collagen isotope analysis is its capacity to reconstruct diet at the individual level, making it possible to assess intra-population variability and detect socially or culturally mediated dietary differences (Katzenberg and Waters-Rist, 2008). Variations in $\delta^{13}C$ and $\delta^{15}N$ values between individuals can reveal patterns of food access related to social status, occupation, gender, or religious practices (Müldner and Richards, 2005; Reitsema, 2013). For instance, elevated $\delta^{15}N$ values may indicate preferential consumption of animal-derived protein, often associated with elite or specialised diets. Conversely, lower values might suggest dietary restrictions or reduced access to high-status foods. Furthermore, stable isotope analysis has proven particularly informative in studies of infant feeding behaviour. Elevated $\delta^{15}N$ values in infants relative to adult females are consistent with breastfeeding, while a gradual decline indicates the weaning process and the introduction of complementary foods (Katzenberg and Waters-Rist, 2008; Fuller et al., 2006).

Collagen isotope analysis also offers significant advantages in terms of preservation and applicability across a wide range of environmental conditions. In arid or oxidizing environments, where organic residues in ceramics degrade rapidly and lipid preservation is poor, collagen often tends to remain stable and can yield reliable

isotopic data (van Klinken, 1999). Similarly, in temperate or humid regions, where soil chemistry and microbial activity may compromise residue analysis, bone collagen can still preserve dietary isotopic signals. This resilience makes collagen a particularly valuable substrate for reconstructing diet, even in cases where other types of bioarchaeological evidence are not well preserved. Its methodological robustness across taphonomic settings contributes to its widespread use in archaeological science and paleoecological studies (Ambrose, 1990; van Klinken, 1999).

When applied to skeletal remains across different time periods and cultural settings, bulk collagen stable isotope analysis enables the reconstruction of long-term dietary shifts and broader social transformations. It has proven instrumental in identifying the adoption of domesticated plants and animals, transitions from foraging to farming, and changing degrees of reliance on marine and freshwater resources (Cubas et al., 2019; Craig et al., 2007). Additionally, isotopic differences within populations have revealed patterns of social inequality, where access to high-status or culturally significant foods reflects hierarchical structures or evolving food ideologies. These insights contribute to a deeper understanding of human-environment interactions and the emergence of complex food economies, aligning biological data with archaeological, historical, and environmental narratives.

3.2.1. Isotopes: chemical overview

As explained above (section **3.1**), isotopes are atoms of the same element with the same number of protons but different numbers of neutrons, resulting in distinct atomic masses (Soddy, 1949; Ellam, 2016). While these differences do not substantially affect chemical reactivity, they do influence the physical behaviour of isotopes in processes such as evaporation, diffusion, and metabolic transformation.

Isotopic fractionation, the preferential use or loss of lighter isotopes during natural processes, produces variations in isotopic ratios that reflect biological and environmental pathways (Hoefs, 2021; Fry, 2006). As described earlier, the degree to which an isotope fractionates in a given system is measured using the delta (δ)

notation, which represents the deviation of a sample's isotope ratio relative to a standard reference material. The general formula used to express isotopic ratios has already been explained before (section **3.1**)**.** For carbon isotope measurements, the internationally accepted reference standard is the Vienna Pee Dee Belemnite (VPDB), while for nitrogen isotopes, the standard is atmospheric nitrogen (AIR) (Mariotti, 1983). These standards provide a consistent baseline for comparing isotopic values across different studies and materials.

Stable Carbon Isotopes

Carbon isotope fractionation in bone collagen occurs as a result of metabolic processes that selectively incorporate dietary carbon into body tissues. As previously discussed, $\delta^{13}C$ values in collagen primarily reflect the protein component of the diet and not the total macronutrient intake (Ambrose and Norr, 1993). This is because collagen synthesis relies on amino acids derived from dietary protein, not on carbohydrates or lipids, which means that isotopic signals from non-protein food sources may not be fully represented in collagen $\delta^{13}C$ values (Tykot, 2004).

As highlighted in section 3.1, a key distinction lies between C_3 and C_4 plants, which follow different photosynthetic pathways and produce characteristically different $\delta^{13}C$ signatures. C_3 plants, including temperate-climate species such as wheat, barley, legumes, and most fruits and vegetables, typically exhibit $\delta^{13}C$ values between -22‰ and -35‰. In contrast, C_4 plants such as maize, millet, and sorghum are enriched in ^{13}C, with values ranging from -9‰ to -14‰ (O'Leary, 1988; Tieszen, 1991). These isotopic differences are transferred up the food chain and are preserved in herbivores and, ultimately, in the tissues of human consumers.

Collagen $\delta^{13}C$ values can also reflect the consumption of marine resources, as marine ecosystems generally have higher baseline $\delta^{13}C$ values compared to terrestrial systems due to differences in carbon cycling and primary production (Chisholm, Nelson and Schwarcz, 1982). Individuals with relatively enriched $\delta^{13}C$ values may have consumed significant amounts of marine or estuarine protein. However, interpretation

is not always straightforward: the use of C_4 plants as fodder, or the consumption of animals raised on C_4-based diets, can mimic isotopic signatures typically associated with marine inputs (Hedges and Reynard, 2007). As a result, contextual information and multi-isotope approaches are essential for accurate dietary reconstruction.

Stable Nitrogen Isotopes

Nitrogen isotopes are usually measured for reconstructing trophic relationships and protein source contributions in past diets. During metabolism, organisms preferentially excrete the lighter nitrogen isotope (^{14}N), resulting in a stepwise enrichment of the heavier isotope (^{15}N) in body tissues with each trophic level increase (Hedges and Reynard, 2007). This process typically leads to a $\delta^{15}N$ increase of approximately 3-5‰ per trophic step, making it possible to distinguish between herbivores, omnivores, and carnivores in both human and faunal samples (Richards and Hedges, 1999).

Herbivores, consuming only plants, display $\delta^{15}N$ values that mirror the local vegetational baseline, usually ranging from about 2‰ to 6‰. Higher $\delta^{15}N$ values in omnivores and carnivores reflect their elevated trophic positions. In archaeological contexts, enriched $\delta^{15}N$ values in human remains often suggest increased consumption of animal-derived proteins such as meat, dairy, or fish, whereas lower values point toward diets richer in plant proteins (Schoeninger and DeNiro, 1984; Bocherens and Drucker, 2003).

Interpretation becomes more complex when aquatic ecosystems are involved. Marine and freshwater food webs often exhibit higher baseline $\delta^{15}N$ values due to extended trophic chains and nitrogen recycling within aquatic environments (Dufour, Bocherens and Mariotti, 1999). As a result, individuals with high $\delta^{15}N$ values may have consumed aquatic resources, though overlaps can occur, especially when freshwater species have variable or unexpectedly low $\delta^{15}N$ values. Local ecological baselines and faunal isotope data are consequently essential for accurate dietary reconstruction (Schoeninger and DeNiro, 1984).

3.2.2. Interpreting bulk isotopic data: EA-IRMS

Elemental analyser-isotope ratio mass spectrometry (EA-IRMS) is the standard method used to determine carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) isotope ratios in bone collagen. This technique combines the combustion capabilities of an Elemental analyser (EA) with the precision of an Isotope Ratio Mass Spectrometer (IRMS), enabling the accurate measurement of stable isotope composition in organic material (Hoefs, 2021).

The analytical workflow begins with the preparation of purified, freeze-dried collagen, which is weighed (typically 0.5-1.0 mg) and sealed in tin capsules. These capsules are introduced into the EA, where the sample undergoes high-temperature combustion in the presence of oxygen. During this process, carbon and nitrogen in the sample are converted into simple gases, CO_2 and N_2, respectively (DeNiro and Epstein, 1978; Hedges and Reynard, 2007). The resulting gases are carried by a continuous helium stream through a reduction column, where residual oxygen and nitrogen oxides are removed, and then separated chromatographically before entering the IRMS.

Within the IRMS, the gases are ionised and passed through a magnetic field that separates isotopic species based on their mass-to-charge (m/z) ratios. The instrument records the relative abundances of heavy and light isotopes, which are converted into δ-values relative to internationally recognised standards (Brand, 2004). Each run includes a suite of control materials, international standards (e.g., IAEA-600, USGS62) and internal collagen references with known $\delta^{13}C$ and $\delta^{15}N$ values, to ensure analytical precision and accuracy (Ambrose, 1990).

Instrument drift, combustion completeness, and analytical error are monitored through repeated measurements of standards and duplicates. Measurement precision typically falls within ±0.2‰ for both $\delta^{13}C$ and $\delta^{15}N$ (Katzenberg and Waters-Rist, 2008). All unknowns are measured against calibration curves derived from these standards, ensuring data comparability across samples and batches. Specific instrument settings applied in this study are explained in the following Chapter **(Chapter 4)**.

4. Material and Methods

The analytical principles and interpretative frameworks outlined in **Chapter 3** are applied to the archaeological materials presented in the following Chapter. Chapter 4 details the laboratory protocols used to generate the datasets of this thesis, together with information on sample numbers and provenance.

4.1. Recovering lipids from archaeological pottery

4.1.1. Sample selection

For this study, organic residue analysis was conducted on ceramic sherds recovered from five archaeological sites in Southern Brazil. The selected materials derive from existing archaeological collections assembled during previous excavations before the beginning of the present doctoral research, and not from newly excavated material. These collections were held in archaeological repositories or museum holdings, meaning that excavation, handling, labelling, packaging and post-excavation storage conditions were not fully controlled as part of this project. This has implications for organic residue analysis, because post-excavation treatment, storage materials and incomplete contextual documentation may affect residue preservation, contamination risk and the interpretation of negative or low-yield results. The sites include Morro Grande 1 (MG1), Praia Grande - Unidade 21 (PG-U21), Arroio Tamarina 2 (AT2), Foz do Cubatão (FC), located in Babitonga Bay (Santa Catarina), and Casa 2 in Pelotas (Rio Grande do Sul).

Radiocarbon dating and associated material culture indicate that the sites of Morro Grande 1 (MG1) and Praia Grande, Unidade 21 (PG-U21), were occupied between 1750 and 1950 AD. Faunal bone samples yielded calibrated dates ranging from 1510 to 1950 AD, with median values between 1650 and 1850 AD (Fossile et al., 2023). However, the interpretation of radiocarbon data for these historical contexts is challenged by the plateaus and reversals in the calibration curve between ca. 1700 and

1950 AD, which result in broad calibrated ranges and low chronological resolution. As discussed by Fossile et al. (2023), this limits the ability to securely date post-colonial sites based solely on radiocarbon, requiring a combined approach with relative dating methods, such as diagnostic artefacts and stratigraphy. The presence of early 20th-century English pottery at PG-U21 and 19th-century coins at MG1 supports a more conservative occupation range between the late 18th and mid-20th centuries. For all other sites in this study, the chronology was established based on contextual archaeological and documentary sources.

Where possible, the selection process prioritised ceramic sherds associated with cooking practices, based on typological and morphological characteristics such as vessel shape, size, and visible signs of use, including sooting and thermal alteration. Special attention was given to the preservation state of the ceramics to maximise the potential recovery of absorbed organic residues. Where possible, selection was also informed by stratigraphic context and associated archaeological documentation.

However, the full typological identification of all sherds was not always possible. In some cases, archaeological reports lacked detailed ceramic classification, as the original research objectives focused primarily on broader site characterisation rather than ceramic typology. In addition, the small size and fragmented condition of some samples limited their morphological assessment. Despite these challenges, the assemblage still provides a robust and representative dataset for investigating culinary practices, food processing, and resource use in historical Southern Brazil. Further archaeological details on the ceramic assemblages and sites analysed, including photographs and contextual information, are provided in **Chapters 5** and **6.**

4.1.2. Lipid Extraction Strategies

Lipid extraction from archaeological ceramics is commonly performed using solvent-based total lipid extraction (TLE; also referred to here as solvent extraction, SE) and acidified methanol extraction (AE). These approaches recover partly overlapping but not identical lipid fractions and preserve different types of analytical information.

Solvent-based extraction methods, including chloroform/methanol or dichloromethane/methanol mixtures, have long been used in archaeological organic residue analysis to extract free and solvent-extractable lipids from ceramic matrices (Bligh and Dyer, 1959; Condamin et al., 1976; Evershed, Heron and Goad, 1990; Evershed, 2008). One advantage of TLE is that it can retain compositional information on intact or partially preserved acyl lipids, including triacylglycerols (TAGs), diacylglycerols (DAGs), monoacylglycerols (MAGs) and wax esters, which may be altered by hydrolytic or methylating extraction methods (Evershed, Heron and Goad, 1990; Regert, 2011; Reber, 2021). Before gas chromatographic analysis, solvent extracts usually require derivatisation to improve volatility and chromatographic performance, particularly for polar compounds such as free fatty acids and alcohols (Regert, 2011; Řezanka, Pádrová and Sigler, 2016).

However, TLE has limitations when applied to archaeological ceramics, especially where residues are highly degraded, strongly absorbed or present at low concentration. Solvent extraction may recover lower lipid yields than acidified methanol extraction in some archaeological samples and is less effective at releasing lipids strongly associated with the ceramic matrix or polar degradation products such as dicarboxylic acids and dihydroxy fatty acids (Regert et al., 1998; Correa-Ascencio and Evershed, 2014; Hammann et al., 2020). These factors can reduce interpretative resolution, particularly in preservation-challenging contexts or when large assemblages require systematic screening (Evershed et al., 1999; Evershed, 2008; Roffet-Salque et al., 2017a).

Acidified methanol extraction (AE), developed for archaeological ceramics by Correa-Ascencio and Evershed (2014), was designed as a direct extraction and methylation method for absorbed lipid residues. The method uses acidic methanol to release lipid components from powdered ceramic and simultaneously convert fatty acids into fatty acid methyl esters suitable for GC-FID, GC-MS and GC-c-IRMS analysis (Correa-Ascencio and Evershed, 2014). This approach has been shown to recover higher concentrations of lipid residues than conventional solvent extraction in many

archaeological samples and to reduce processing time, making it particularly useful for high-throughput screening of large ceramic assemblages (Correa-Ascencio and Evershed, 2014; Roffet-Salque et al., 2017a). Acid extraction may also yield substantially higher lipid contents than solvent extraction in some archaeological ceramics, highlighting its value for residues preserved within the ceramic matrix (Hammann et al., 2020).

The main limitation of AE is that it hydrolyses and methylates complex acyl lipids, including TAGs, MAGs, DAGs and wax esters, breaking them down into constituent fatty acid methyl esters and alcohols. As a result, some structural information on intact lipid classes is lost (Roffet-Salque et al., 2017a; Reber, 2021). For this reason, AE and TLE/TMS were used in this study in a structured but non-equivalent way. AE provided the principal dataset for lipid recovery, abundance estimates, molecular screening and compound-specific isotope analysis of fatty acids, with isotope values corrected for the added methyl carbon where applicable. TLE/TMS was applied to selected subsets to retain complementary information on solvent-extractable lipid fractions where chromatographic quality allowed.

The table below summarises the initial number of ceramic records analysed by site, the number of AE extracts retained for molecular and isotopic interpretation after screening, the number of AE records not retained, and the TLE/TMS subset analysed as complementary analytical evidence, noting where outputs were interpretable or retained only as analytical documentation.

Site	Region	Initial ORA records	AE retained for interpretation	AE not retained	TLE/TMS subset analysed
Foz do Cubatão	Santa Catarina	45	40	5	14
Morro Grande 1	Santa Catarina	40	34	6	17
Arroio Tamarina 2	Santa Catarina	41	36	5	16
Praia Grande - Unidade 21	Santa Catarina	55	54	1	31
Casa 2	Rio Grande do Sul	20	14	6	6

Table 1: *Initial ORA records, AE extracts retained for molecular and isotopic interpretation, AE records not retained, and TLE/TMS subsets analysed by site.*

For the Babitonga Bay assemblages, the initial ORA dataset comprised 181 unique ceramic records: 45 from Foz do Cubatão, 40 from Morro Grande 1, 41 from Arroio Tamarina 2 and 55 from Praia Grande - Unidade 21. Of these, 164 AE extracts yielded interpretable lipid profiles above the 5 μg g^{-1} threshold and were retained for molecular and isotopic interpretation. The remaining 17 Babitonga records were not retained for AE-based interpretation: 12 produced low-yield or non-interpretable lipid signals, while five Arroio Tamarina 2 extracts were excluded because chromatographic contamination compromised reliable interpretation of the AE results. A complementary Babitonga TLE/TMS subset of 78 extracts was also analysed, of which 30 produced low-yield, non-diagnostic or contamination-affected TLE/TMS outputs. For Casa 2, the initial dataset comprised 20 records, of which 14 AE extracts yielded interpretable lipid profiles above the 5 μg g^{-1} threshold and were retained for molecular and isotopic interpretation; six samples were also analysed by TLE/TMS. These distinctions are important because low-yield, contaminated and non-diagnostic extracts form part of

the analytical record, but are not treated as equivalent to the main interpretable dataset used for molecular or isotopic interpretation. Detailed sample-level information, analytical outcomes and chromatographic evidence are provided in Chapters 5 and 6 and in the corresponding appendices.

AE was applied to all archaeological sherds selected for ORA because it provided the most efficient and analytically robust way to evaluate lipid preservation and recovery across the full assemblage. It was used as the principal screening strategy because it recovers free and bound/degraded lipid fractions effectively, allows quantitative comparison of lipid abundance, and produces fatty acid methyl esters suitable for GC-FID, GC-MS and GC-c-IRMS analysis (Correa-Ascencio and Evershed, 2014; Roffet-Salque et al., 2017a; Hammann et al., 2020). The 5 $\mu g\ g^{-1}$ threshold used in this thesis applies to AE-based lipid recovery and abundance estimates derived from GC-FID quantification.

TLE/TMS was not intended as a second full-screening method applied systematically to every sherd. Instead, it was used as a complementary extraction on selected samples to retain additional information on solvent-extractable lipid fractions, including partially preserved acyl lipids that may be altered during AE (Evershed, Heron and Goad, 1990; Regert, 2011; Reber, 2021). This strategy avoided duplicating the full AE dataset while still allowing TLE/TMS to contribute useful compositional information where chromatographic quality was sufficient.

TLE/TMS outputs were interpreted only when chromatographic quality allowed reliable assessment of the target compounds. In the Babitonga Bay subset, some TLE/TMS chromatograms showed early-eluting background or contaminant patterns. Where these features did not co-elute with the retention-time windows used to assess target compounds, they were retained as analytical records but were not treated as evidence for archaeological lipid inputs. In the Casa 2 subset, the TLE/TMS chromatograms were more extensively affected by laboratory hexane/background contamination. For this reason, the Casa 2 TLE/TMS results were retained as analytical

documentation only, and no TLE/TMS-derived MAG, DAG, TAG, sterol or plant-wax assignments were used for source-specific interpretation.

These TLE/TMS limitations are distinct from the exclusion of specific AE extracts affected by low yield or chromatographic contamination. AE records retained or not retained for interpretation are reported at sample level in the relevant results chapters and appendices, while TLE/TMS outputs are presented as complementary analytical documentation where appropriate.

The following analytical procedures are based on the current Standard Operating Procedure (SOP) for ORA at the University of York (BioArCh), which builds upon established methodologies widely adopted in archaeological science (e.g. Evershed et al., 1999; Craig et al., 2013a; Correa-Ascencio and Evershed, 2014).

Acidified methanol extraction

Approximately 1 g of homogenised ceramic powder was weighed into a clean, labelled glass tube. A C34 n-alkane internal standard was added to each sample prior to extraction, and one method blank and one standard were included in each extraction batch to monitor procedural contamination and analytical consistency. Methanol (MeOH; 4 mL) was then added to the powdered sample, and the mixture was sonicated for 15 minutes at approximately 25 °C to promote solvent penetration and dispersion of the ceramic powder. Concentrated sulfuric acid (H_2SO_4; 800 µL) was added slowly under a fume hood, and the samples were capped and heated at 70 °C for 4 hours to promote acid-catalysed hydrolysis and methylation of free and bound lipids.

After heating, the samples were cooled to room temperature and centrifuged at 3000 rpm for 5 minutes. The acidified methanol extract was transferred to a clean, labelled glass tube, taking care to avoid the transfer of ceramic sediment. Hexane was then added to the remaining powder, vortexed and centrifuged to recover additional lipid material; this supernatant was combined with the acidified methanol extract. The methanol/acid extract was subsequently extracted with hexane in repeated

solvent-exchange steps. The upper hexane layer, containing the extracted lipids, was collected and passed through a potassium carbonate filter to neutralise residual acid and remove polar contaminants.

The combined hexane extracts were reduced under a gentle stream of nitrogen with low heat and then evaporated to dryness. The dried extract was resuspended in hexane and transferred to an autosampler vial containing a C36 n-alkane recovery standard prior to instrumental analysis. Method blanks were processed alongside the archaeological samples and analysed by GC-FID and GC-MS to identify potential contaminants introduced during sample preparation and extraction.

Modern reference extraction

Acidified methanol extraction was also performed on modern reference samples of rice (*Oryza sativa* subsp. *indica*, n = 2), beans (*Phaseolus vulgaris*, Pinto Group, n = 4) and manioc flour (*Manihot esculenta*, n = 2) to assess the isomer distribution of ω-(o-alkylphenyl) alkanoic acids (APAAs) produced under experimental heating conditions. These reference samples were included to support the interpretation of APAA-C_{18} E/H ratios in relation to plant processing, following the approach developed by Bondetti et al. (2021).

Dried plant samples (65 mg) were mixed with 100 mg of sterile pottery powder drilled from wheel-thrown replica vessels made with red clay. The mixtures were placed in sterile glass tubes and covered with deionised water to simulate contact between plant material, ceramic fabric and moisture during heating. The samples were heated at 270 °C for 5 hours and then extracted using the same acidified methanol protocol applied to the archaeological ceramic powders. The resulting APAA distributions are reported in Appendix 3.

Solvent extraction

Total lipid extraction (TLE) was applied to a selected subset of ceramic samples to recover free and weakly bound lipids that may retain compositional information lost during acidified methanol extraction. Ceramic powder was prepared following the same general surface-cleaning and powdering procedure used for AE. Where necessary, the outer surface layer was removed to reduce potential contamination, and approximately 1 g of ceramic powder was weighed into a clean, labelled glass tube. A C_{34} n-alkane internal standard was added to each sample prior to extraction, and method blanks and standards were included in each batch.

A dichloromethane:methanol mixture (DCM:MeOH, 2:1 v/v; 5 mL) was added to each sample. This solvent mixture was used to dissolve free and weakly bound non-polar and moderately polar lipids. Samples were sonicated for 15 minutes at approximately 25 °C and then centrifuged at 3000 rpm for 10 minutes to separate the solvent extract from the ceramic powder. The supernatant was transferred to a clean, labelled glass tube, taking care to avoid the transfer of fine ceramic particulates.

The extraction was repeated twice more using fresh DCM:MeOH (2:1 v/v), and the three solvent extracts were combined. The combined total lipid extract was then reduced to approximately 2 mL under a gentle stream of nitrogen with low heat. Where sufficient extract was available, the TLE was divided into fractions for subsequent derivatisation and analysis. This method was used to provide complementary information on solvent-extractable lipid classes, including acyl lipids such as monoacylglycerols, diacylglycerols and triacylglycerols, which may be hydrolysed during acidified methanol extraction.

Derivatisation of extracts - silylation

Solvent extracts selected for silylation were derivatised using BSTFA + TMCS (99:1; N,O-bis(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane). The

dried extract was first resuspended in a small volume of hexane to ensure that the residue was fully dissolved. BSTFA + TMCS was then added, and the vial was gently flushed with nitrogen and capped. Samples were heated at 70 °C for 60 minutes to form trimethylsilyl derivatives of compounds containing active hydrogen groups, including alcohols, sterols and hydroxy fatty acids.

After heating, the samples were allowed to cool, and excess reagent and solvent were evaporated under a gentle stream of nitrogen. The derivatised extracts were then resuspended in hexane, transferred to autosampler vials containing the C36 n-alkane recovery standard, and analysed by GC-FID and GC-MS. Derivatisation was carried out shortly before instrumental analysis to minimise degradation of the TMS derivatives.

4.1.3. Analytical approaches: specific GC-FID, GC-MS and GC-c-IRMS settings

The following section describes the instrumental conditions used for GC-FID, GC-MS and GC-c-IRMS analysis. Analyses were carried out between BioArch, University of York, and ICTA-UAB, Universitat Autònoma de Barcelona. In this study, GC-FID and GC-MS were used as complementary analytical approaches with different functions. GC-FID provided the primary basis for the quantification of derivatised lipid extracts, consistent with its routine use for the quantitative assessment of separated organic compounds, whereas GC-MS was used principally for compound identification and confirmation of source-informative biomarkers (Sparkman, Penton and Kitson, 2011; Regert, 2011). GC-c-IRMS was used for compound-specific carbon isotope analysis of selected fatty acid methyl esters.

Acidified methanol extracts were initially screened using an Agilent 7890A Series gas chromatograph (Agilent Technologies, Cheadle, Cheshire, UK) equipped with a flame ionisation detector (FID). Chromatographic separation was performed on a DB-1HT column composed of 100% dimethylpolysiloxane (15 m × 0.32 mm × 0.1 µm; J&W Scientific, Folsom, CA, USA). An aliquot of 1 µL was introduced in splitless mode,

with the injector maintained at 300 °C. The oven temperature was held at 100 °C for 2 min, then increased at a rate of 20 °C min^{-1} to 325 °C, where it was held for a further 2 min. Helium was used as the carrier gas at a constant flow of 2 mL min^{-1}. The FID was maintained at 300 °C, with a hydrogen flow of 30 mL min^{-1}. The total run time for this short screening programme was 20 min.

Derivatised solvent extracts were analysed using the same GC-FID system and DB1-HT column described above. For these extracts, the oven programme was extended to improve the detection of higher molecular weight lipid compounds, including monoacylglycerols (MAGs), diacylglycerols (DAGs) and triacylglycerols (TAGs). The initial oven temperature was held at 50 °C for 2 min, then increased at 10 °C min^{-1} to 375 °C, followed by a final hold at 375 °C for 10 min.

GC-FID was used to estimate lipid abundance from integrated peak areas, while GC-MS provided structural information needed to confirm compound identifications and distinguish archaeological lipid signals from exogenous contaminants. Lipid abundance was calculated from the summed GC-FID peak areas assigned to archaeological lipid compounds, after excluding internal standards and identified exogenous contaminant peaks. The resulting values were normalised to the internal standard response and to the mass of ceramic powder analysed, and are reported as µg lipid g^{-1} ceramic. Where GC-MS indicated that specific peaks represented exogenous compounds, including phthalate-related plasticiser peaks, these were excluded from the summed lipid peak area. Peak areas were integrated using the instrument software and manually inspected to ensure consistent baseline placement, peak assignment and exclusion of identifiable contaminants where appropriate. Manual adjustment was applied only where necessary, for example in cases of unresolved shoulders, baseline drift or incorrectly assigned peaks. Concentration values were used to assess lipid recovery and chromatographic quality, rather than as absolute measures of original vessel contents.

Full-scan GC-MS analysis was performed at ICTA-UAB using a Thermo Trace Ultra gas chromatograph coupled to a Thermo ISQ7000 quadrupole mass spectrometer. Prior to analysis, samples were diluted in isooctane, and 1 µL of each extract was injected onto a DB-5MS fused-silica capillary column. The injector temperature was set at 310 °C. The GC oven was initially held at 90 °C for 1 min, then increased to 320 °C at 6 °C min^{-1}, with a final hold at 320 °C for 20 min. Helium was used as the carrier gas. The mass spectrometer was operated in electron ionisation mode at 70 eV, with data acquired in full-scan mode over the range m/z 70–550 and a scan time of 0.2 s $scan^{-1}$. The transfer line and ion source temperatures were maintained at 300 °C and 260 °C, respectively. Compounds were identified by comparison of their mass spectra with the NIST 14.0 mass spectral library.

In this configuration, the column effluent was divided after chromatographic separation. Approximately 10% of the effluent was directed to the MS detector for compound identification, while the remaining 90% was directed to the GC-c-IRMS system for stable carbon isotope analysis of fatty acid methyl esters. This split-flow configuration allowed molecular identification and compound-specific isotope measurement to be obtained from the same chromatographic run where sample quality and concentration were sufficient.

Stable carbon isotope ($\delta^{13}C$) values of methyl palmitate ($C_{16:0}$) and methyl stearate ($C_{18:0}$), corresponding to the methylated derivatives of palmitic and stearic acids, were analysed by GC-c-IRMS at ICTA-UAB. Samples were injected in splitless mode on a DB5-MS UI fused silica column (60 m × 0.25 mm i.d., 0.25 µm film thickness, J&W Scientific, Folsom, CA, USA). The $\delta^{13}C$ values of individual fatty acids were determined using a Delta V Advantage mass spectrometer (Thermo Fisher, Bremen) linked to a ConFlo IV interface. During the initial 10 minutes, the effluent from the GC was diverted away from the combustion reactor (backflush mode). During the solvent-divert period, CO_2 reference gas was automatically introduced into the IRMS in a series of pulses and its $^{13}C/^{12}C$ measured. After the solvent-divert period, the effluent from the GC was allowed to enter the combustion reactor and IRMS. The IRMS

automatically measured the ion intensities of m/z 44, 45, and 46 in its three Faraday cups corresponding to $^{12}C^{16}O_2$, $^{13}C^{16}O_2$, and $^{12}C^{16}O^{18}O$, respectively. The Isodat 3.0 software automatically computed the $^{13}C/^{12}C$ ratio of each sample peak, referenced to the standard CO_2 gas and its known isotopic content. The results were expressed in per mil (‰) relative to the VPDB standard. To ensure data comparability, the instrument was calibrated using the Indiana University F8 standard, which contains a mixture of eight methylated and ethylated fatty acids of known isotopic composition. Additionally, quality control standards were intercalated every 5-6 injections to monitor the stability of the isotopic measurements through time. Instrumental precision and accuracy were better than ±0.5‰. All lipid extracts selected for GC-c-IRMS were analysed in duplicate to assess reproducibility. Values were also corrected using mass balance to account for the methylation of the carboxyl group that occurs during acid extraction. Corrections were based on comparisons with a standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition processed in each batch as a sample.

To investigate plant and aquatic-related biomarkers that may occur at low abundance, all acidified methanol extracts retained for molecular interpretation were further analysed by targeted GC-MS in selected ion monitoring (SIM) mode at BioArch, University of York. SIM was used to increase sensitivity for pre-selected compounds by monitoring specific mass-to-charge ratios.

Analyses were performed using an Agilent 7890A GC connected to an Agilent 5975C Inert XL mass-selective detector with a quadrupole mass analyser. A polar DB-23 column was used (50%-cyanopropyl-methylpolysiloxane; 60 m × 0.250 mm × 0.25 µm). The oven programme began at 50 °C, held for 2 minutes, followed by temperature ramps of 10 °C min^{-1} to 100 °C, 4 °C min^{-1} to 140 °C, 0.5 °C min^{-1} to 160 °C, and 20 °C min^{-1} to 250 °C, where it was held for 10 minutes. Helium was used as the carrier gas at a constant flow rate of 2.4 mL min^{-1}.

The SIM method targeted ions selected for compounds relevant to plant and aquatic lipid interpretation. For 4,8,12-trimethyltridecanoic acid (TMTD), ions m/z 74,

87, 213 and 270 were monitored. For pristanic acid, ions m/z 74, 88, 101 and 312 were monitored, while phytanic acid was monitored using ions m/z 74, 101, 171 and 326. APAAs were monitored using ions m/z 74, 105, 262, 290, 318 and 346, allowing targeted detection of APAA homologues including APAA-C18 and APAA-C20. Phytanic acid diastereomers were assessed by integrating ion m/z 101.

The selection of the SIM target ions was based on the use of fragment ions that are widely recognised as diagnostically informative for the compounds of interest in archaeological residue analysis. For TMTD, the ion set m/z 74, 87, 213 and 270 was selected because it captures the characteristic fragmentation pattern of this isoprenoid fatty acid derivative and improves confidence in its detection in complex archaeological extracts. For pristanic acid, the ions m/z 74, 88, 101 and 312 were selected, while for phytanic acid, the ions m/z 74, 101, 171 and 326 were monitored; similar ion sets are routinely used in archaeological residue studies targeting branched isoprenoid fatty acids linked to aquatic resource processing. For APAAs, the ion set m/z 74, 105, 262, 290, 318 and 346 was chosen because these ions allow the targeted detection of APAAs of different chain lengths, including APAA-C_{18} and APAA-C_{20}, which are particularly informative in the interpretation of heated plant and aquatic-derived commodities (Hansel et al., 2004; Craig et al., 2007; Cramp and Evershed, 2014, Colonese et al., 2017, Bondetti et al., 2021). In the present study, these ions were selected to maximise confidence in the targeted detection of aquatic- and plant-related biomarkers while minimising the uncertainty associated with low-abundance compounds in full scan acquisition.

Because scan and SIM acquisition serve different analytical purposes, they were not treated as directly equivalent sources of information. Full scan GC-MS was used to obtain broad compositional information and support general compound identification, whereas SIM was used as a more sensitive targeted mode for the detection of selected biomarkers of interest. Greater sensitivity in SIM relative to full scan is expected because acquisition time is concentrated on a restricted set of ions rather than distributed across a broader mass range, thereby improving the detectability of

low-abundance target compounds. In the present study, SIM was used to strengthen confidence in the detection of compounds such as APAAs and isoprenoid fatty acids.

The 5 μg g^{-1} threshold was applied specifically to the GC-FID-derived concentration estimates, after exclusion of identified exogenous contaminant peaks, including phthalate-related plasticiser peaks, and was used here as a practical screening threshold. This is consistent with recent ORA studies in which values above 5 μg g^{-1} are treated as yielding interpretable amounts of absorbed lipids in potsherds, while interpretation still depends on chromatographic quality and the presence or absence of diagnostic compounds (Courel et al., 2020; Briggs et al., 2023). Consequently, values above this threshold were not treated automatically as diagnostically informative, and values below it were not treated automatically as devoid of archaeological significance. Instead, lipid concentration was considered together with chromatographic quality, biomarker presence and agreement between analytical approaches where available.

Because these analyses formed part of a workflow distributed between BioArch, University of York, and ICTA-UAB, a limited between-laboratory comparison was undertaken to assess the comparability of compound-specific carbon isotope measurements of fatty acids. The comparison was based primarily on the repeated analysis of shared isotopic reference and quality-control materials used in the GC-c-IRMS workflow, including the standard mixture of $C_{16:0}$ and $C_{18:0}$ fatty acids of known isotopic composition processed in each batch. This was supplemented by a limited subset of archaeological extracts analysed across both laboratory settings. The use of shared reference materials, repeated quality-control measurements, and replicate analyses to assess comparability is consistent with established good practice in isotope ratio mass spectrometry, particularly when the aim is to evaluate analytical consistency rather than to derive formal inter-laboratory reproducibility statistics from a large replicated sample set. In the present study, this comparison did not reveal any systematic discrepancies that would alter the main isotopic interpretations. Instead, the results were broadly consistent in terms of the $\delta^{13}C$ patterning of the major fatty

acids and the overall interpretative trends observed in the dataset. Any minor differences between laboratories are considered most likely to reflect differences in instrumental configuration and analytical sensitivity and not contradictory archaeological signals.

Analytical uncertainty and expected variability were treated as explicit components of interpretation throughout this study. Interpretative confidence was based not on isolated numerical differences alone, but on the convergence of multiple lines of evidence, including lipid abundance, chromatographic quality, the presence of diagnostic biomarkers, and isotopic patterning. As a consequence, small differences were treated with caution, particularly in low-yield or borderline samples, and greater interpretative weight was given to patterns that were supported by more than one analytical criterion. This approach is consistent with good practice in both archaeological residue analysis and isotope ratio mass spectrometry, where quality control, duplicate analysis, calibration, and cautious treatment of low-abundance compounds are central to robust interpretation.

Replication was incorporated where possible. Lipid extracts analysed by GC-c-IRMS were run in duplicate, repeated standards and quality-control materials were intercalated throughout the runs, and instrumental precision and accuracy for isotopic measurements were better than ±0.5‰, as assessed through repeated measurement of reference materials and control samples. Because the between-laboratory comparison was based on shared standards and only a limited subset of archaeological samples, it was used here to demonstrate broad analytical consistency rather than to calculate formal estimates of inter-laboratory error such as standard deviation across a large replicated sample set. These criteria are applied in Chapters 5 and 6, where analytical outcomes are reported separately from the initial extraction counts and evaluated in relation to lipid concentration, chromatographic quality, biomarker preservation, contamination and site-specific preservation conditions. The resulting ORA dataset is reported at two levels in this thesis. Chapters 5 and 6 present the interpretable molecular and isotopic results used for

archaeological interpretation, while the appendices provide sample-level analytical outcomes, including low-yield, non-interpretable and contamination-affected records. This separation is intended to distinguish clearly between the analytical record generated during the study and the subset of results used for source-specific interpretation.

4.2. Recovering collagen from archaeological bones

4.2.1.Sample selection

As discussed in the previous Chapter, SIA was employed as a complementary methodology to ORA, with the aim of broadening the dietary and ecological interpretation of the ceramic residue results. Faunal isotope data were used to establish local isotopic baselines and to explore animal feeding regimes, both of which are important for interpreting the sources and variability of organic residues identified through ORA. While the primary focus of this thesis lies in the application of ORA, the integration of SIA provides an additional comparative line of evidence. The isotopic data are interpreted in light of the ORA findings presented in **Chapters 5** and **6**, allowing for a more nuanced discussion of food resource use, culinary practices and subsistence strategies.

Stable isotope analysis was conducted on human and faunal bone samples from five historical sites representing urban, rural and ecclesiastical contexts The selected bones derive from existing curated collections assembled prior to the present doctoral research. The sites include Morro Grande 1 (MG1), Praia Grande - Unidade 21 (PG-U21), and Igreja Matriz Nossa Senhora da Graça in São Francisco do Sul, all in the state of Santa Catarina, as well as Casa 8 in Pelotas and Igreja da Matriz Nossa Senhora Madre de Deus in Porto Alegre (Cúria Metropolitana), both in Rio Grande do Sul. All samples are associated with 19th to early 20th century occupations. Where possible, rib fragments were selected due to their turnover rates, which enhance comparability in dietary reconstruction. In cases where ribs were not available or poorly preserved,

other dense cortical bones (e.g., femur or humerus) were chosen based on preservation quality and contextual integrity. The table below summarises the number of samples analysed for each site. Further archaeological and contextual information on the bone samples is provided in Chapter 7.

Site	Region	Context	Faunal samples	Human samples	Tot.
Casa 8	Rio Grande do Sul	Urban/domestic	Marine Fish (2); Cattle (3); Chicken (2); Dog (1); Opossum (1); Pig (1)	0	10
Igreja da Matriz (Porto Alegre)	Rio Grande do Sul	Church	Cattle (6)	16	22
Igreja Matriz (São Francisco do Sul)	Santa Catarina	Church	0	7	7
Morro Grande 1	Santa Catarina	Rural/coastal	Chicken (5); Cattle (10); Deer (12); Marine Fish (11); Pig (1); Wild Pig (Tayassuidae) (4)	0	43
PG - Unidade 21	Santa Catarina	Rural/coastal	Chicken (14); Cattle (6); Marine Fish (37); Armadillo (3); Pig (1)	0	61

***Table 2**: Summary of faunal and human samples analysed from the five archaeological sites included in this Chapter*

4.2.2. Collagen extraction

Collagen extraction for stable isotope analysis was carried out using a modified Longin-based protocol designed to isolate the collagen-rich organic fraction of bone while reducing exogenous contamination and diagenetic alteration (Longin, 1971; Brown et al., 1988). The aim of the procedure was to obtain collagen suitable for carbon and nitrogen stable isotope analysis, allowing dietary protein signals to be assessed where preservation was adequate.

The procedure began with mechanical cleaning of the bone surface using scalpels or drills to remove adhering sediment, surface contamination and visibly altered material. The cleaned bone samples were then demineralised in 0.5 M

hydrochloric acid (HCl) at approximately 4 °C to preserve collagen integrity. The acid was periodically replaced, and demineralisation was considered complete when the mineral fraction had dissolved and the remaining material became pliable and translucent, indicating the preservation of the collagen-rich organic fraction (DeNiro and Epstein, 1978; Collins and Galley, 1998).

After demineralisation, the samples were rinsed to neutrality with ultrapure water and gelatinised in a weakly acidic solution, usually pH 3 HCl, at 80 °C for up to 48 hours. The solubilised collagen was then transferred to sterile straight-sided tubes and filtered through EEZE™ filters to remove insoluble debris and particulate matter. This filtration step improved the clarity of the extract before freeze-drying.

The purified gelatin was freeze-dried to obtain a collagen powder suitable for isotopic analysis. The extracted collagen was weighed and stored in airtight containers to minimise post-processing contamination. Collagen quality was assessed using standard preservation criteria, particularly the atomic carbon-to-nitrogen (C:N) ratio, with acceptable values falling between 2.9 and 3.6 (DeNiro, 1985; van Klinken, 1999). Samples falling outside this range were considered potentially contaminated or poorly preserved and were excluded from isotopic interpretation (Toso, 2018).

4.2.3. Specific instrumental settings: elemental analysis-isotope ratio mass spectrometry (EA-IRMS)

Stable isotope analysis of bone collagen was performed at SUERC, East Kilbride (UK), using a Delta V Advantage continuous-flow isotope ratio mass spectrometer coupled via a Conflo IV to an IsoLink elemental analyser (Thermo Scientific, Bremen, Germany) as described in Sayle et al. (Sayle et al., 2019). Bone collagen (~0.7 mg) was combusted in the presence of oxygen in a single reactor containing tungstic oxide and copper wires at 1020 °C to produce N_2, CO_2 and SO_2. A magnesium perchlorate trap was used to eliminate water produced during the combustion process, and the gases were

separated in a GC column heated between 70 °C and 240 °C. Helium was used as a carrier gas throughout the procedure. N_2, CO_2 and SO_2 entered the mass spectrometer via an open split arrangement within the ConfloIV and were analysed against their corresponding reference gases. The International Atomic Energy Agency (IAEA) reference materials USGS40 (L-glutamic acid, $\delta^{13}C$ (V-PDB) = -26.39 ± 0.04‰, $\delta^{15}N$ (AIR) = -4.52 ± 0.06‰) and USGS41a (L-glutamic acid, $\delta^{13}C$ (V-PDB) = 36.55 ± 0.08‰, $\delta^{15}N$ (AIR) = 47.55 ± 0.15‰) were used to normalise $\delta^{15}N$ and $\delta^{13}C$ values. Normalisation was checked using the well characterised Elemental Microanalysis IRMS fish gelatin standard B2215 ($\delta^{13}C$ (V-PDB) = -22.92 ± 0.10‰, $\delta^{15}N$ (AIR) = 4.26 ± 0.12‰, $\delta^{34}S$ (V-CDT) = 1.21 ± 0.24‰) and/or USGS88 (marine collagen, $\delta^{13}C$ (V-PDB) = -16.06 ± 0.07‰, $\delta^{15}N$ (AIR) = 14.96 ± 0.14‰, $\delta^{34}S$ (V-CDT) = 17.10 ± 0.44‰). Precision was determined to be ± 0.2‰ for $\delta^{15}N$, ± 0.1‰ for $\delta^{13}C$ and ±0.4‰ for $\delta^{34}S$ and is based on repeated measurements of Elemental Microanalysis IRMS fish gelatin standard B2215. 19% of samples were analysed in duplicate. Although $\delta^{34}S$ was measured as part of the SUERC analytical sequence and quality-control workflow, sulphur isotope values are not interpreted in this thesis because the research questions are addressed using carbon and nitrogen isotope data in combination with ORA results.

5. Organic residue analysis results from Santa Catarina: exploring ethnic and socio-economic variability in foodways

Parts of this Chapter have been adapted from Di Muro et al., submitted. "The rise of modern Brazilian cuisine revealed by lipid and protein residues in ceramic artefacts." PNAS Nexus, manuscript ID: PNASNEXUS-2025-00349-T.

5.1. Introduction

This Chapter presents the results of ORA conducted on ceramic assemblages from four late 19th and early 20th century sites in Babitonga Bay, Santa Catarina. It builds on the historical and methodological foundations discussed in previous Chapters (**2** and **3**) to explore how food preparation and consumption varied across cultural and socio-economic contexts during a time of major regional change.

As previously discussed, on Brazil's Atlantic Forest coast, colonial and postcolonial processes, such as industrialization, land commodification, and waves of migration, deeply reshaped local food systems, often adapting and mixing with subsistence practices rooted in Indigenous, Afro-Brazilian, and Luso-Brazilian traditions (Schwartz, 1986; Dean, 1997; Gabriel and Silva, 2019). These processes of culinary and cultural change are not fully understood because historical records tend to focus on elite consumption (Ribeiro, 2017).

ORA offers a complementary line of evidence by identifying food molecular traces preserved in ceramics, providing molecular evidence for substances processed or absorbed in ceramic vessels (Evershed, 2008; Regert, 2011; Cramp and Evershed, 2014). Despite its potential, it remains underused in studies of colonial and postcolonial Brazil (Hansel and Evershed, 2009; Colonese et al., 2014). Here, the analysis of lipid biomarkers and carbon isotopes is integrated with archaeological and historical data to examine foodways as expressions of adaptation and identity.

Comparisons with published lipid datasets from pre-colonial Indigenous contexts in the same region (Admiraal et al., 2023, 2025) allow for a diachronic perspective on culinary change.

5.2. Sites and ceramic assemblages

The ceramic assemblage analysed in this Chapter comes from four archaeological sites (Figure 30), each representing distinct ethnic and possibly economic contexts.

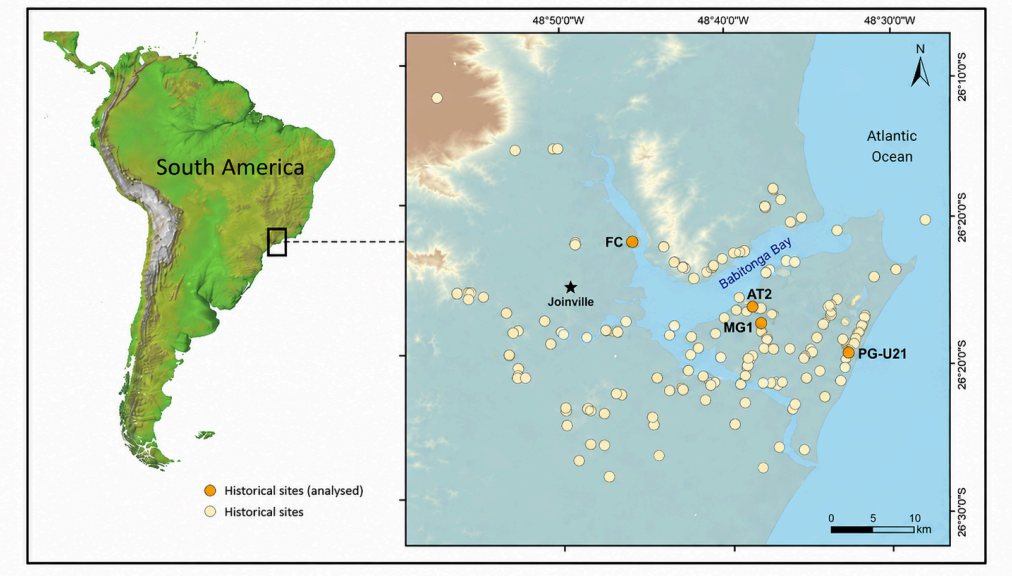


Figure 30: *Geographic distribution of the historical archaeological sites in Babitonga Bay, Santa Catarina, recorded by Bandeira and Alves (2012), including those analysed in this Chapter (orange dots): FC - Foz do Cubatão, MG1 - Morro Grande 1, AT2 - Arroio Tamarina 2, and PG-U21 - Praia Grande Unidade 21. The inset indicates the location of Santa Catarina within Brazil, with the study area highlighted by a box.*

Foz do Cubatão (FC) and Morro Grande 1 (MG1) are associated with Luso-Brazilian, Indigenous and Afro-Brazilian descendant communities, whose food preparation practices were likely shaped by local subsistence strategies, historical inequalities and colonial influences. Praia Grande - Unidade 21 (PG-U21) is similarly associated with non-elite rural populations and ceramic assemblages that may reflect a degree of localised production and self-sufficiency. By contrast, Arroio Tamarina II (AT2)

is archaeologically associated with a German migrant community, although any relationship between migrant identity, ceramic technology and food preparation must be evaluated through the combined residue, isotopic, ceramic and historical evidence and not assumed from site attribution alone.

The assemblages analysed here derive from previously excavated and curated collections, and the selection of sherds was constrained by availability, fragmentation, documentation and preservation. Where possible, sampling prioritised ceramic fragments likely to have been associated with cooking or food processing, based on vessel morphology, visible use-alteration and contextual information. This strategy increased the likelihood of recovering absorbed food residues, but may also have introduced a preservation and functional bias towards ceramic fabrics, vessel forms or surface conditions more favourable to lipid survival. Consequently, the results are interpreted as patterns of residue preservation and vessel use within the sampled assemblage(Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

No visible charred surface residues were recorded among the sampled sherds. This absence is treated as a macroscopic observation instead of as evidence that the vessels were not heated, since absorbed lipid residues and molecular heating markers may preserve even where carbonised surface residues are absent. Heating-related evidence is assessed through molecular indicators, including APAA distributions.

The initial Babitonga sherd database included 181 unique records from FC, MG, AT2 and PG-U21. The final AE dataset used for molecular and isotopic interpretation retained 164 extracts with interpretable lipid profiles above the 5 μg g^{-1} screening threshold. Seventeen records were not retained for AE-based interpretation: 12 produced low-yield or non-interpretable lipid signals, while five AT2 extracts were excluded because chromatographic contamination compromised reliable interpretation of the AE results. These excluded records are retained in the sample-level appendices as part of the analytical record, but they are not included in the interpreted AE dataset.

5.2.1. Foz do Cubatão

The site of Foz do Cubatão is situated near the Cubatão River, opposite Babitonga Bay, in what is now the city of Joinville (Figure 30). It was first identified during inspections by the archaeological team of the Joinville Sambaqui Archaeological Museum (MASJ), who recorded ceramic fragments along the riverbank and in the surrounding area (MASJ, 2001).



Figure 31: *(A) Excavated house structure at Foz do Cubatão. (B) Examples of ceramic fragments from FC selected for organic residue analysis. Scale = 1 cm.*

These materials were interpreted as evidence of pre-colonial Indigenous occupation, likely associated with the Tupi-Guarani tradition (Borba, 2014; MASJ, 2001). Further investigations revealed the remains of a small rural house, reportedly inhabited until around the 1950s, according to oral histories provided by local residents (Borba, 2014; MASJ, 2001) (Figure 31A).

Excavations confirmed the presence of a domestic structure with clay brick walls and compacted earth floors, and uncovered a material assemblage that spanned Indigenous and historical contexts (Figure 31A) (MASJ, 2001). Among the artefacts recovered were low-fired handmade ceramics with incised and combed surface treatments, maiolica sherds, glass fragments, copper coins, iron tools, and domestic utensils. These materials indicate a household of modest means, with technological features that reflect overlapping Indigenous, African, and European influences (MASJ,

2001). Faunal remains, including otoliths and fish bones from catfish (*bagre*) and whitemouth croaker (*pescada*), along with shell tools and fishing gear, point to the importance of estuarine resources in daily subsistence (MASJ, 2001). The combination of ceramics, faunal material, and tool types suggests a hybrid domestic economy shaped by cultural interaction and sustained engagement with the local aquatic environment.

The initial FC dataset included 45 ceramic records. Of these, 40 AE extracts were retained for molecular and isotopic interpretation, while five records were not retained because they produced low-yield or non-interpretable lipid signals. Fourteen FC samples were also analysed by TLE/TMS as complementary analytical evidence.

5.2.2. Morro Grande 1

Morro Grande 1 (MG1) is located on São Francisco do Sul Island (Figure 30), a historically significant area on Santa Catarina's northern coast. São Francisco do Sul, one of the oldest colonial settlements in Southern Brazil, was originally occupied in pre-colonial times by Indigenous groups. By the mid-17th century, Portuguese settlers, such as Antônio Fernandes and Manoel Lourenço de Andrade, began requesting land concessions from the Portuguese Crown to develop agriculture and industry. These early colonial endeavors established São Francisco do Sul as a prominent hub for trade and production, including cordage, dried fish, sugarcane, cotton, and cassava (Fossile et al., 2023; Silva, 2001b).

The Morro Grande area formed part of Andrade's *sesmaria* (land grant), but its early history remains unclear due to the lack of written records. By the 19th century, documents mention additional land grants in the area, and oral accounts indicate that MG1 was predominantly occupied during the early 20th century. At this time, the area was an agricultural community, with residents cultivating manioc and raising livestock such as chickens, cattle, and pigs (Borba, 2014).

Artefacts from MG1 reveal a rich and diverse material culture. Ceramics include white and decorated earthenware, porcelain, stoneware, and locally produced clay pottery - the latter being the ceramic category analysed in this study (Figure 32). Decorations feature straight, curved, or diagonal lines, as well as brushed patterns on the rims and bodies, suggesting a mix of Indigenous, African, and European influences (Chmyz et al., 1976; Silva, 2001b).

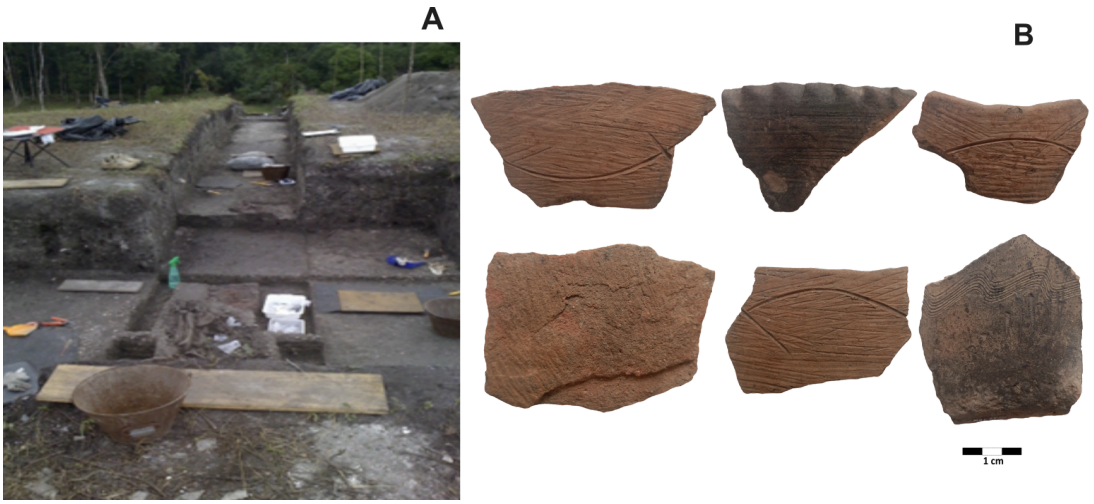


Figure 32: (A) *Excavation photograph from Morro Grande (MASJ, 2001). (B) Examples of ceramic fragments from the Morro Grande assemblage selected for organic residue analysis. Scale = 1 cm.*

The utilitarian nature of these ceramics, primarily medium to large bowls and pans used for food preparation, reflects the practical needs of the community (Borba, 2014). Other recovered items include metal objects (nails, coins, buttons, spoons, knives), glassware, lithic flakes, pipes, slingshot pellets, and couscous steamer fragments (Chmyz et al., 1976). These findings highlight the enduring traditions of enslaved Indigenous peoples and African descendants, as well as Portuguese settlers, within a context of low socioeconomic status (Chmyz et al., 1976; Silva, 2001b). Further evidence suggests local manufacturing processes for pottery, as indicated by the absence of wheel use, underscoring an adaptation to traditional methods to their environment. The initial Morro Grande dataset included 40 ceramic records. Of these, 34 AE extracts were retained for molecular and isotopic interpretation, including 33 from Morro Grande 1 and one from Morro Grande 3, while six records were not

retained because they produced low-yield or non-interpretable lipid signals. Seventeen Morro Grande samples were also analysed by TLE/TMS as complementary analytical evidence.

5.2.3. Praia Grande - Unidade 21

Praia Grande - Unidade 21 (PG-U21) is also located on São Francisco do Sul Island (Figure 30). The site was discovered during pipeline construction in 2001, which revealed the remains of an old residence accompanied by a mill, an "oven-like" structure, wooden and stone architectural features, and fragments of artefacts and food remains (Alves and Oliveira, 2002; Fossile et al., 2023). The archaeological context suggests a rural settlement primarily dedicated to manioc cultivation and processing, with radiocarbon dating placing its occupation in the late 19th and early 20th centuries (Silva et al., 2001; Fossile et al., 2023).

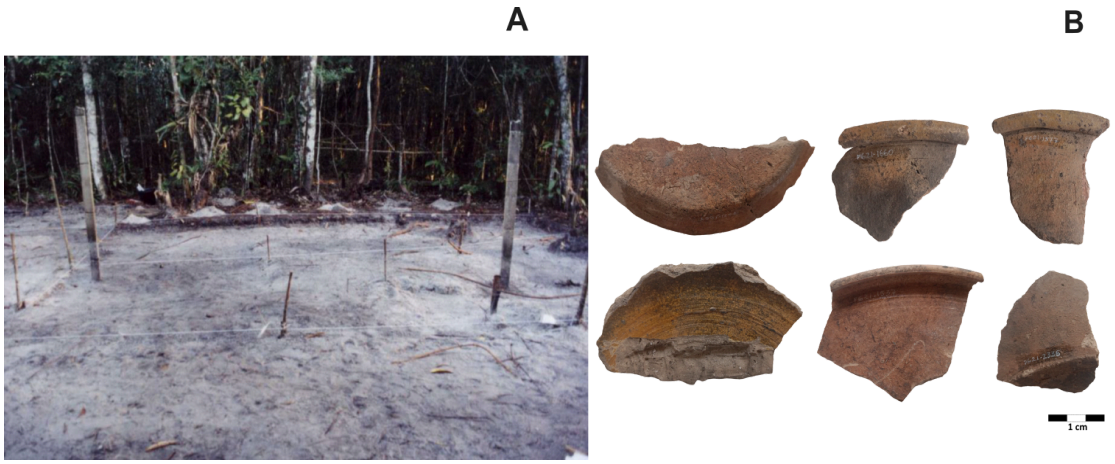


Figure 33: *(A) Excavation photograph of Praia Grande - Unidade 21 (Alves and Oliveira, 2002). (B) Examples of ceramic fragments from PG-U21 selected for organic residue analysis. Scale = 1 cm.*

The presence of domestic milling and combustion facilities, as well as utilitarian ceramics, indicates a community engaged in both subsistence and market-oriented production. Faunal remains from PG-U21 indicate the exploitation of fish, hunted fauna

and domestic animals, including chickens, cattle and pigs (Silva et al., 2001; Fossile et al., 2023).

Sherds analysed in this study were attributed to wheel-made cooking wares likely originating from external markets (Alves and Oliveira, 2002; Fossile et al., 2023) (Figure 33, B). The presence of porcelain further suggests that the community had access to long-distance trade networks or connections to wealthier residents (Alves and Oliveira, 2002; Fossile et al., 2023). Additionally, fragments of glass and refined ceramics imply a degree of economic integration with urban centres, potentially indicating that some goods were imported via coastal trade routes. The initial PG-U21 dataset included 55 ceramic records. Of these, 54 AE extracts were retained for molecular and isotopic interpretation, while one record was not retained because it produced a low-yield or non-interpretable lipid signal. Thirty-one PG-U21 samples were also analysed by TLE/TMS as complementary analytical evidence.

5.2.4. Arroio Tamarina II

The Arroio Tamarina II (AT2) site is situated in São Francisco do Sul Island (Figure 30), within dense vegetation characterised by bromeliads, orchids, and humid soils (de Farias, 2012) (Figure 34). Excavations conducted by The Museu Arqueológico de Sambaqui de Joinville (MASJ) uncovered the remains of a historic house, including structural beams and various everyday artefacts, such as a mortar containing mollusc remains. Other materials included glass, porcelain, and animal bones from domesticated species like chickens and cattle (Borba, 2014).



Figure 34: *(A) Excavation photograph of Arroio Tamarina 2 (Borba, 2014). (B) Examples of ceramic fragments from AT2 selected for organic residue analysis. Scale = 1 cm.*

The ceramic corpus from AT2 resembles that of PG-U21, consisting mainly of wheel-made, smoothed/undecorated red clay cooking wares (Figure 34B). Residential structures and associated artefacts suggest that the site was occupied by German migrants during the early 20th century (de Farias, 2012). Material culture recovered includes glass bottles, faience, wire, animal bones, and tile fragments. Ceramics sampled from the site exhibited utilitarian features and were consistent with low socioeconomic status (Borba, 2014). Additionally, oral histories associated with the site hint at interactions between German settlers and neighboring communities, further enriching the narrative of cultural exchange in this rural region. These findings suggest that AT2 was a rural settlement shaped by migrant histories, local resources and interactions with neighbouring communities (Borba, 2014). The initial AT2 dataset included 41 ceramic records. Of these, 36 AE extracts were retained for molecular and isotopic interpretation, while five records were excluded because chromatographic contamination compromised reliable interpretation of the AE results. These five excluded AT2 records are TRD_1335, TRD_1336, TRD_1342, TRD_1349 and TRD_1368. Sixteen AT2 samples were also analysed by TLE/TMS as complementary analytical evidence.

5.3. Results

The results are presented by analytical approach in order to distinguish between information obtained from acidified methanol extraction (AE) and total lipid extraction followed by TMS derivatisation (TLE/TMS). AE provided the main dataset for lipid concentration, fatty acid distributions, APAA formation, isoprenoid compounds and compound-specific isotope analysis. TLE/TMS was used as a complementary method to assess the preservation of intact or partially degraded acyl lipid fractions, particularly MAGs, DAGs and TAGs, where chromatographic quality allowed. TLE/TMS-derived compounds were not treated as source-specific biomarkers on their own. Within the Babitonga Bay TLE/TMS subset, some outputs showed early-eluting background or contaminant patterns; these regions were excluded from interpretation where they did not co-elute with the later retention-time windows used to assess intact or partially degraded acyl lipid classes and other target compounds, including MAGs, DAGs, TAGs and wax esters (Evershed, Heron and Goad, 1990; Regert, 2011). Outputs affected by low yield, non-diagnostic chromatographic quality or contamination are retained in the appendices as analytical documentation, but are not used as source-specific evidence; sample-level outcomes are reported in Appendix 1 and chromatographic documentation is provided in Appendix 2. The interpretation thus relies on the combined pattern of AE lipid profiles, APAA distributions, isoprenoid compounds, CSIA values and archaeological context, not on any single compound class alone (Evershed, 2008; Regert, 2011; Correa-Ascencio and Evershed, 2014; Roffet-Salque et al., 2017a).

5.3.1. Lipid recovery and composition

Lipid concentrations above the practical screening threshold used in this thesis for absorbed residues (>5 μg g^{-1}) were obtained from 164 AE extracts retained for molecular and isotopic interpretation, with concentrations ranging from 7.05 to 1932.22 μg g^{-1} (Appendix 1). The highest and lowest concentrations were both recorded within the Morro Grande assemblage, illustrating the substantial variability in

lipid recovery even within a single site. The 17 Babitonga records not retained for AE-based interpretation are reported in Appendix 1 as part of the analytical record and include 12 low-yield or non-interpretable outputs and five AT2 extracts excluded because AE chromatographic contamination compromised reliable interpretation.

The AE extracts were dominated by saturated fatty acids ($C_{7:0}$ - $C_{26:0}$), with palmitic ($C_{16:0)}$ and stearic ($C_{18:0}$) acids as the most abundant compounds (Figure 35A). P/S ratios ranged from 0.40 to 9.98 across the retained AE dataset, with 136 samples showing values between 1.1 and 9.98. These values are compatible with mixed lipid inputs, including degraded plant, animal and aquatic-derived fats, but P/S ratios alone are not diagnostic of source and were, for this reason, interpreted only in combination with APAA distributions, isoprenoid compounds and CSIA values (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Monomethyl-branched fatty acid candidates were recorded in 162 samples, while dicarboxylic acid candidates were recorded in 133 samples. Branched fatty acids may be compatible with bacterial contributions, including those associated with ruminant-derived fats, but they may also reflect microbial activity during deposition, burial or post-excavation storage. Dicarboxylic acids are better interpreted as oxidation products formed during lipid degradation, rather than as source-specific evidence for ruminant adipose fats. For this reason, these compounds are treated here as supporting and non-diagnostic indicators, and ruminant-related interpretations are considered only where supported by CSIA values, faunal evidence and archaeological context (Dudd, Regert and Evershed, 1998; Regert, 2011; Roffet-Salque et al., 2017a). Figure 35A provides an example of the general AE chromatographic profile used for molecular interpretation, while additional chromatograms, including low-yield, non-diagnostic and contamination-affected outputs, are provided in Appendix 2.

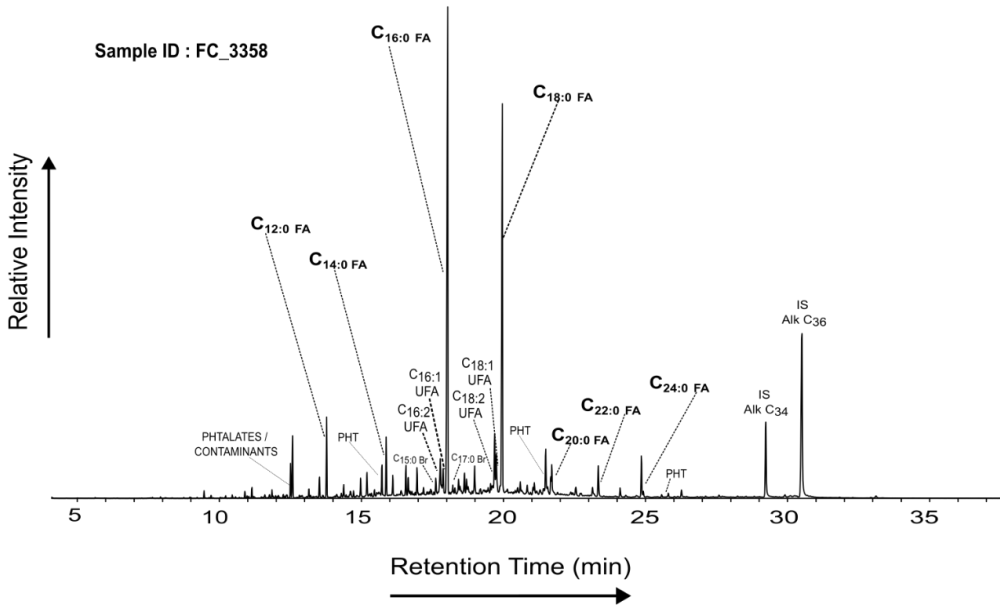


Figure 35A: *AE chromatogram from the Babitonga Bay - Foz do Cubatão ceramic assemblage. The chromatogram shows an interpretable acidified methanol extract from sample FC_3358-AE, dominated by palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) fatty acids, with additional medium- and long-chain fatty acids, unsaturated fatty acids and branched fatty acids indicated where identifiable. Peaks labelled as PHT correspond to phthalate-related plasticiser contaminants and were not used for lipid abundance estimates or source interpretation. The internal standards, alkane C_{34} and alkane C_{36}, are also indicated.*

In the TLE/TMS dataset, the compounds retained for interpretation were limited to MAGs, DAGs and TAGs. TLE/TMS was applied to 78 extracts from the Babitonga assemblages, of which 30 produced low-yield or non-diagnostic results. Where preserved, MAGs, DAGs and TAGs provide complementary evidence for the survival of intact or partially degraded acyl lipids, but they were not used as source-specific biomarkers. This distinction is important because hydrolysis, oxidation and microbial alteration can modify original lipid distributions during vessel use, burial and post-excavation storage (Dudd, Regert and Evershed, 1998; Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

As described in Chapter 4, identifiable modern contaminant peaks, including phthalate-derived plasticiser peaks, were excluded from lipid abundance estimates and were not used in source interpretation. The interpretations presented here are based on contaminant-corrected lipid abundances, fatty acid distributions, APAA ratios, isoprenoid compounds and CSIA values.

Possible resin-related oxidation products, including tentative DHA-related compounds, were recorded in the appendix as non-diagnostic compounds. These identifications remain uncertain in the present dataset and were not used as primary evidence for vessel contents. Such compounds may reflect exposure to resinous wood during firing or cooking, combustion residues, smoked products, post-depositional inputs, or post-excavation handling and storage. For this reason, they are considered contextual indicators of possible exposure to resinous materials but not direct evidence for specific food processing practices (Regert, 2011; Roffet-Salque et al., 2017a).

Long-chain ketones were not confidently identified in the analysed samples, despite the presence of APAAs in part of the assemblage. This absence is not necessarily contradictory, as different heating markers form under different conditions and from different lipid precursors. Long-chain ketones can form through the pyrolysis of acyl lipids, whereas APAAs are formed through the heating of unsaturated fatty acids; as a consequence, their formation may be affected differently by lipid composition, heating conditions, ceramic matrix and subsequent degradation (Raven et al., 1997; Hansel et al., 2004; Regert, 2011; Bondetti et al., 2021).

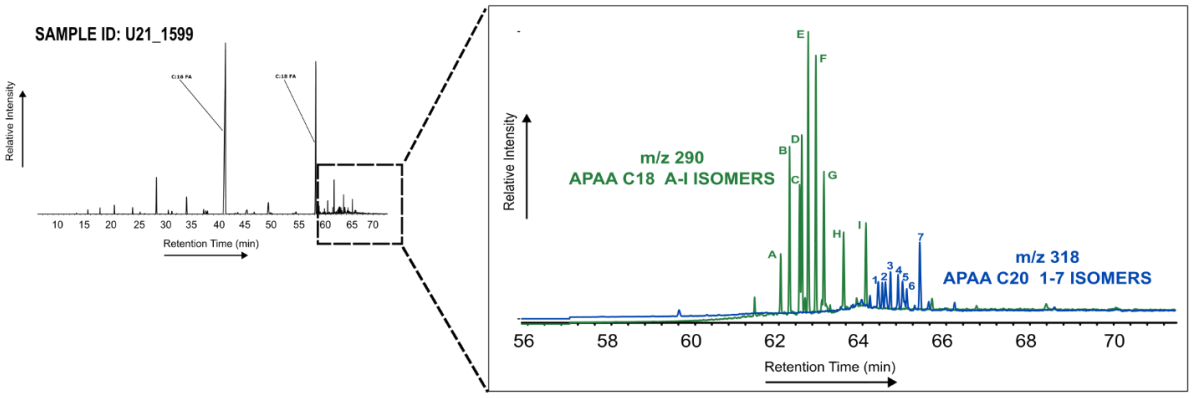


Figure 35B: *Representative DB-23/SIM chromatogram for APAA identification in sample U21-1599. The left panel shows the broader chromatographic profile obtained during the targeted DB-23/SIM analysis. The highlighted late-eluting region is expanded in the right panel, showing selected ion traces used to visualise the APAA series: APAA-C_{18} isomers A-I monitored at m/z 290 and APAA-C_{20} isomers 1-7 monitored at m/z 318. These traces illustrate the chromatographic basis for calculating APAA-C_{18} E/H and APAA-$C_{20/C18}$ values.*

APAA-C_{18} isomers were recorded in 73 samples, of which 38 had E/H values above 4 (Figure 35B; Appendix 1). These values are compatible with the heating of unsaturated plant-derived lipids and are treated here as plant-processing proxy evidence under the experimental framework described by Bondetti et al. (2021), rather than as a definitive distinction between plant categories. APAA-C_{18} E/H values above 4 were most frequent in PG-U21 (n = 19), followed by MG1 (n = 10), FC (n = 7) and AT2 (n = 2) (Figure 36; Appendix 1). Other samples with APAA-C_{18} E/H values above 4 also showed relatively high lipid concentrations and enriched $\delta^{13}C$ values. These results are compatible with mixed lipid inputs, including possible C_4 plant and/or aquatic-derived contributions, but they cannot be assigned to maize, marine products or any single source on isotopic grounds alone because APAA formation, lipid preservation and $\delta^{13}C$ values may overlap across different resources and mixed residues (Evershed, 2008; Regert, 2011; Bondetti et al., 2021; Roffet-Salque et al., 2017a). It is worth noting that cooking experiments on other modern plants widely consumed in the region yielded

relatively low APAA-C^{18} E/H values, including manioc (1.9) and beans (1.0) (Appendix 3; Bondetti et al., 2021).

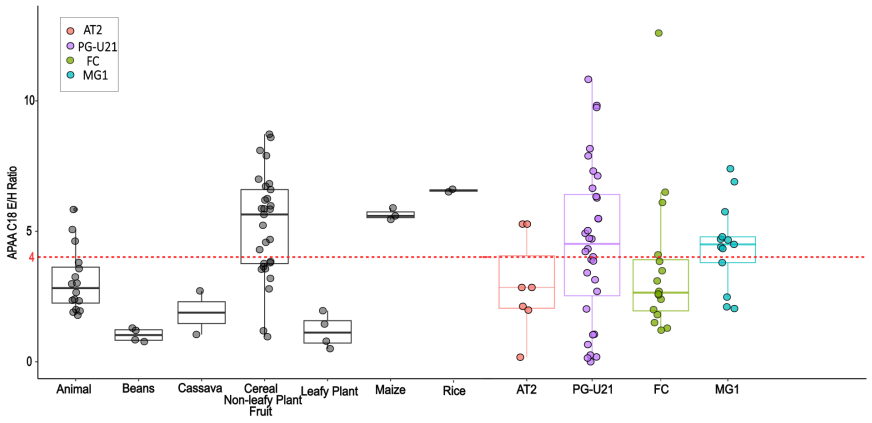


Figure 36: *Boxplot showing the distribution of APAA-C_{18} ω-(o-alkylphenyl)alkanoic acid E/H isomer ratios across experimental reference materials (grey; Appendix 3) and archaeological samples from AT2, PG-U21, FC and MG1. The dashed line marks the threshold value of 4, above which elevated APAA-C18 E/H ratios have been associated with the heating of non-leafy/starchy plant products under experimental conditions (Bondetti et al., 2021). These values are used here as a plant-processing proxy and are interpreted in combination with lipid concentration, molecular profiles and CSIA results.*

These experimental results suggest that APAA-C_{18} values should be interpreted as a plant-processing proxy. Minor amounts of lauric ($C_{12:0}$) and myristic ($C_{14:0}$) acids were recorded in the assemblage, but these compounds are not source-specific and may derive from multiple plant, animal or mixed lipid inputs, including dairy fats (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). For this reason, they were not used as independent evidence for plant processing (Figure 35A; Appendix 1). Higher relative abundances of $C_{12:0}$ in selected AT2 and PG-U21 samples may reflect the contribution of lipid sources enriched in medium-chain fatty acids, but no specific source assignment is proposed on this basis alone. Monounsaturated fatty acids ($C_{16:1}$-$C_{22:1}$), polyunsaturated fatty acids ($C1_{6:2}$ and $C_{18:2}$) and long-chain alkanes (C_{19}-C_{33}), where recorded, were treated as supporting and non-diagnostic compounds because their presence can reflect mixed lipid inputs, plant-associated contributions, degradation products or post-depositional processes (Heron and Evershed, 1993;

Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Consequently, they were interpreted only in combination with APAA distributions, lipid concentration, CSIA values and the wider archaeological context.

Among the samples yielding APAA-C_{18}, 28 also contained trace amounts of APAA-C_{20} (Figure 37; Appendix 1). The formation of longer-chain APAAs has been associated with the heating of aquatic-derived lipids, although experimental and archaeological studies indicate that APAA distributions should be interpreted cautiously because terrestrial products and mixed residues may also contribute to their formation (Hansel et al., 2004; Cramp and Evershed, 2014; Bondetti et al., 2021). APAA-$C_{20/C18}$ values above 0.06 were recorded in 21 samples, with the highest frequencies in MG1 (n = 7) and FC (n = 6), followed by PG-U21 (n = 5) and AT2 (n = 3) (Figure 37; Appendix 1).

This distribution provides evidence compatible with the heating of aquatic-derived lipids, particularly when considered alongside isoprenoid compounds and CSIA values. However, APAA-$C_{20/}C_{18}$ values are not treated as definitive evidence for marine resources in isolation, since heated terrestrial products and mixed residues may also contribute to APAA formation, and the isotopic distinction between marine resources and C_4 plants remains difficult to resolve in ceramic lipid residues (Hansel et al., 2004; Bondetti et al., 2021; Admiraal et al., 2023).

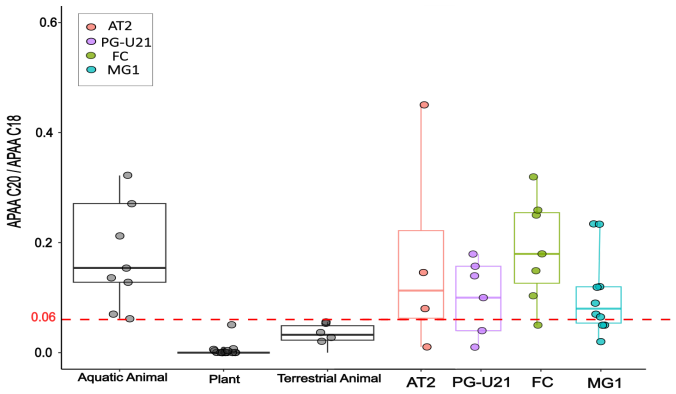


Figure 37: *Boxplot showing the distribution of APAA-C_{20}/C_{18} values across experimental reference materials (grey; Appendix 3) and archaeological samples from AT2, PG-U21, FC and MG1. The dashed line marks the threshold value of 0.06, above which APAA-C_{20}/C_{18} values have been associated with aquatic-derived lipids in archaeological ceramics (Bondetti et al., 2021).*

5.3.2. Compound-specific isotope analysis

Compound-specific carbon isotope analysis of palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids was used to assess broad isotopic patterning in the Babitonga assemblages. The results are shown in Figure 38. These values were interpreted alongside molecular evidence rather than as direct single-source assignments, because repeated vessel use, mixing of animal and plant products, lipid degradation and overlap between marine and C_4-derived carbon sources can complicate interpretation (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a; Admiraal et al., 2023). As analytical and interpretative uncertainties affect the separation of overlapping reference ranges, differences between sites are discussed as assemblage-level tendencies (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

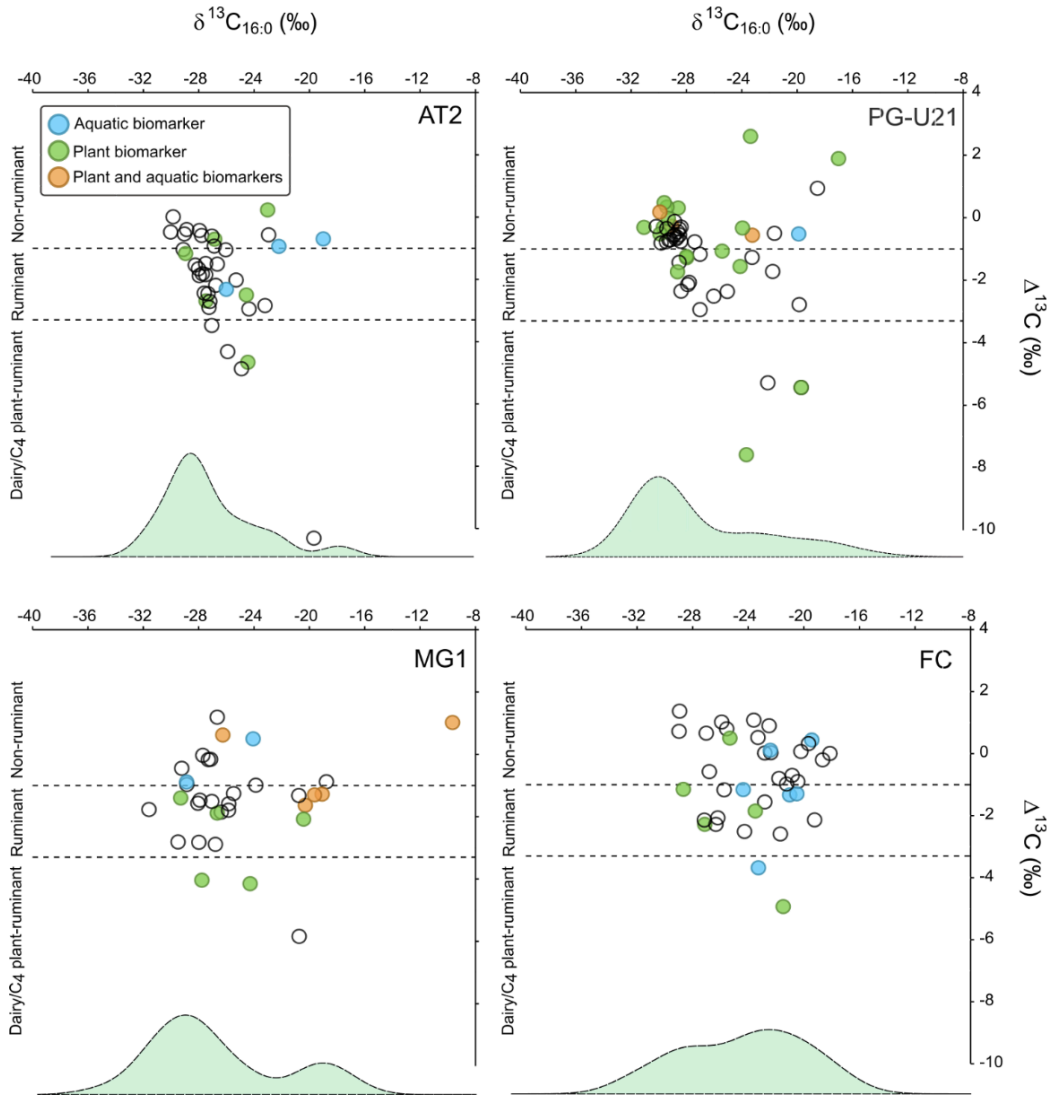


Figure 38: *Compound-specific carbon isotope results for Babitonga ceramic lipid residues, plotted by site as $\delta^{13}C_{16:0}$ values against $\Delta^{13}C_{18:0\text{-}16:0}$ values. The four panels show samples from AT2, PG-U21, MG1 and FC. Horizontal dashed lines indicate the broad interpretative ranges used to distinguish non-ruminant, ruminant adipose and dairy/C_4 plant-ruminant compatible lipid signatures. Coloured points indicate samples with APAA-derived proxy evidence: APAA-C_{18} E/H > 4 as plant-processing proxy positive (green), APAA-C_{20}/C_{18} > 0.06 as aquatic-compatible proxy positive (blue), and samples with both proxy indicators (orange). These markers are used as interpretative aids and not as definitive source assignments (Evershed, 2008; Bondetti et al., 2021; Roffet-Salque et al., 2017a).*

Samples from PG-U21 and AT2 mostly plotted within or close to the reference areas associated with C_3 plants, C_3-fed non-ruminant fats and ruminant adipose fats. A smaller number of samples from these sites plotted closer to dairy-compatible or mixed C_3/C_4-fed ruminant reference ranges. By contrast, MG1 and FC showed a broader spread of $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values, compatible with more variable inputs, including terrestrial animal fats, C_3/C_4 plant resources and/or aquatic-derived lipids (Figure 38). This broader isotopic range supports assemblage-level differences between sites, but does not allow marine and C_4 plant contributions to be fully separated without considering molecular biomarkers and contextual evidence (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a; Admiraal et al., 2023).

The bulk collagen $\delta^{13}C$ values of ruminant animals from PG-U21 and MG1, which will be discussed in detail in Chapter 7, provide contextual evidence for the presence of C_4-influenced animal food webs in Babitonga Bay. Similarly, collagen $\delta^{13}C$ and $\delta^{15}N$ values of chickens and pigs suggest variable feeding ecologies. These data may help explain enriched $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values in the ceramic lipid residues, but they are used here as contextual support and not as direct evidence for specific vessel contents (Evershed, 2008; Regert, 2011).

5.4. Discussion

The lipid and isotopic results from Babitonga Bay suggest assemblage-level differences in pottery use between the four sites. These differences are not inferred from single biomarkers, but from the combined distribution of lipid concentrations, APAA-C_{18} E/H values, APAA-$C_{20/C18}$ ratios, isoprenoid compounds, CSIA values and archaeological context. The patterns should be understood as comparative tendencies in the sampled ceramic assemblages, not as a complete reconstruction of household diet (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

The quality and interpretability of the Babitonga ORA dataset were shaped by preservation, analytical recovery and the previously excavated nature of the

archaeological collections. While 164 AE extracts produced interpretable lipid profiles above the practical screening threshold and were retained for molecular and isotopic interpretation, 17 records from the initial database were not retained for AE-based interpretation. Twelve of these produced low-yield or non-interpretable lipid signals, while five AT2 extracts were excluded because AE chromatographic contamination compromised reliable interpretation. As described in Chapter 4, identifiable modern contaminant peaks, including phthalate-derived plasticiser peaks, were excluded from lipid abundance estimates and were not used in source interpretation. In addition, some Babitonga TLE/TMS outputs showed early-eluting background or contaminant patterns and were retained as analytical documentation and not treated as source-specific evidence where chromatographic quality did not allow reliable interpretation. These issues do not invalidate the dataset, but they affect the scale at which interpretations can be made. Absence of a compound is not treated as straightforward evidence for absence of a resource, since lipid survival is influenced by vessel use, ceramic fabric, heating conditions, burial environment, microbial activity, post-excavation handling and laboratory detection limits. For this reason, the interpretations presented here prioritise recurring assemblage-level patterns and combinations of evidence, not the presence of single compounds or individual samples (Dudd, Regert and Evershed, 1998; Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

The late 19th and early 20th centuries represented a key turning point for food production and consumption habits in Southern Brazil, marked by significant economic and demographic shifts driven by nationalistic agendas, industrialization, and the rise of a middle class. Commercial agriculture and fishing expanded to meet the growing demand from urban centres and national and regional markets (Herbst et al., 2024, 2023; Noble, 1967). The abolition of slavery led to an influx of European labour through state-funded colonies, particularly in Southern Brazil (Duarte, 2014; Jordan, 1962). In Babitonga Bay, European immigrants intensified agricultural production in an area previously dominated by small-scale manioc cultivation and fishing by Luso-Brazilians, Africans, and Indigenous peoples (Cabral, 1937; Noble, 1967;

Saint-Hilaire, 1936). Through subsistence and incipient market-oriented production, these historical groups relied on agriculture (e.g. manioc, rice, beans, maize, sugar cane, peanuts) and domestic animal production (cattle, pigs, chicken), along with fishing and hunting wildlife (Fossile et al., 2023; Saint-Hilaire, 1936; Silva, 2019).

The AT2 assemblage shows a stronger tendency towards terrestrial/C_3-compatible lipid signatures than FC and MG1, based on CSIA values and the relatively lower frequency of aquatic-compatible APAA-$C_{20/C18}$ signals. This pattern is interpreted cautiously and at assemblage level, given the variable chromatographic quality observed in part of the retained AT2 AE dataset and the broader interpretative limitations of degraded archaeological lipid residues (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Only three AT2 samples had APAA-C20/C18 values above 0.06, compared with FC (n = 6), MG1 (n = 7) and PG-U21 (n = 5). Considering the archaeological attribution of AT2 to a German migrant community, this pattern may be compatible with food preparation practices more strongly oriented towards terrestrial plant and animal products. However, the wheel-thrown red clay vessels from AT2 may reflect access to external supply networks or local-regional markets, and ceramic technology should not be treated as a direct proxy for ethnicity or status without supporting contextual evidence. By contrast, FC and MG1 show a higher frequency of aquatic-compatible indicators than AT2. Together with faunal remains, fishing gear and historical accounts, these results are consistent with the continued importance of aquatic resources in the daily food economy of these communities. However, the ORA data alone cannot quantify the dietary importance of fish or distinguish fully between marine resources and C_4 plant inputs (Hansel et al., 2004; Bondetti et al., 2021; Admiraal et al., 2023).

More intensive use of C_3-compatible plant and terrestrial animal products at PG-U21 compared with FC and MG1 may reflect differences in ceramic use, household production and access to local-regional markets. These differences may partly relate to socio-economic status, but status cannot be isolated from other factors such as site

location, vessel form, ceramic supply networks, market integration and preservation conditions (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

These interpretations gain further support when considered within the broader economic and environmental context of late 19th and early 20th century Santa Catarina. Agricultural policies promoted maize, sugarcane and rice cultivation, while fishing became increasingly commercialised but also remained important for local subsistence strategies (Alves, 2017; Borba, 2014; Noble, 1967). This context may help explain the presence of C_4-compatible and aquatic-compatible signals in ceramic residues. However, these signals should not be treated as direct evidence for specific crops or fish consumption in isolation, because marine and C_4 plant inputs can overlap isotopically and ceramic residues may represent repeated or mixed vessel use (Evershed, 2008; Regert, 2011; Admiraal et al., 2023).

The ceramic evidence provides an important contextual framework for interpreting the lipid results: wheel-thrown wares at AT2 and PG-U21 may reflect access to external markets or different supply networks, while handmade and unglazed wares at FC and MG1 may indicate the persistence of local manufacturing traditions. These technological differences could have influenced both cooking practices and lipid preservation, but their relationship with ethnicity, status and food choice depends on wider archaeological and contextual evidence (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

The broader isotopic variability of the historical assemblages becomes more apparent when the historical data are compared with pre-colonial isotopic values (Figure 39). When considered over the longer term, the historical ceramic vessels show broader isotopic variability than ceramic artefacts attributed to pre-colonial Taquara-Itararé and Guaraní groups in the region (Admiraal et al., 2023). This broader range suggests greater variability in the products processed in historical ceramic vessels, not a simple increase in the use of any single resource category. Such

variability may reflect changes in pottery function, household economies and access to market-oriented resources during the late 19th and early 20th centuries.

The role of São Francisco do Sul as a major port and transportation route in Santa Catarina provides an additional context for interpreting the historical assemblages. Increased circulation of commodities and market-oriented production may have affected both the availability of food resources and the ceramic forms used in household food preparation. Comparisons with pre-colonial pottery need to account for differences in vessel function, chronology, ceramic technology and sampling strategy, which may also contribute to the observed isotopic contrasts. The historical assemblages are interpreted as reflecting changing and more variable pottery use within a transformed economic landscape, rather than as a direct measure of dietary replacement.

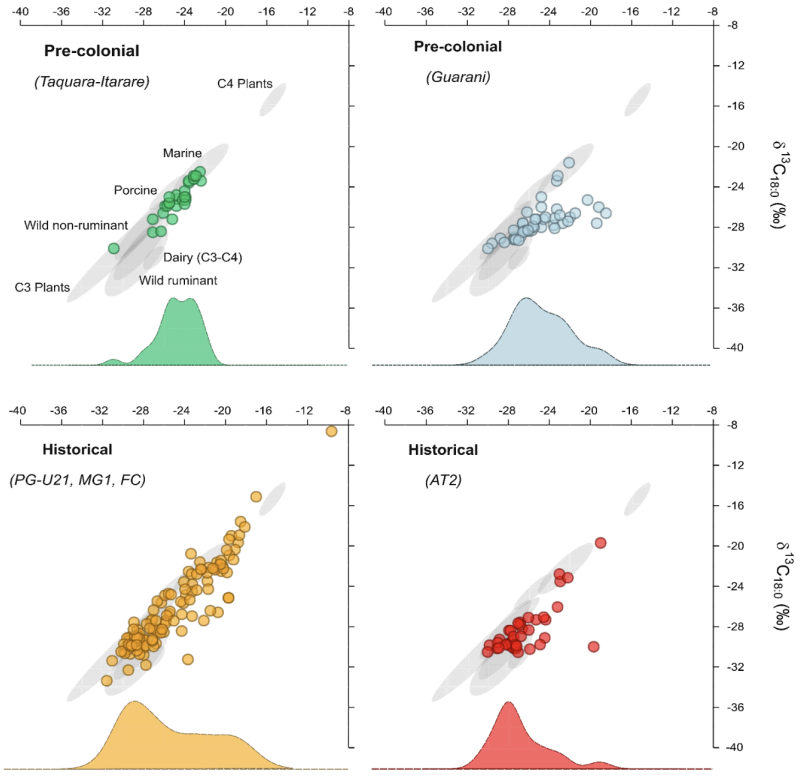


Figure 39: *Long-term variability in pottery use reflected in the $\delta^{13}C$ values of palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids from pre-colonial pottery attributed to Taquara-Itararé and Guarani contexts (Admiraal et al., 2023) and historical pottery analysed in this study from PG-U21, FC, MG1 and AT2. Data are compared with modern reference ellipses representing 68% confidence intervals (Appendix 7).*

Overall, the Babitonga Bay assemblages suggest that historical food practices in these coastal communities were shaped by the interaction of local ecology, household provisioning, ceramic use and changing market conditions, rather than by a single cultural or economic factor. The combined presence of plant-processing proxy evidence, aquatic-compatible indicators and variable terrestrial/C_3-compatible lipid signatures points to flexible vessel use within communities that remained connected to estuarine and small-scale provisioning systems while also participating in broader nineteenth- and early twentieth-century economic transformations. At the same time, the uneven distribution of these signals between sites shows that continuity in coastal resource use should not be interpreted as a fixed or homogeneous culinary tradition. Instead, the results indicate situated and variable food-processing practices, shaped by preservation, access to resources, vessel technology and household-level choices.

Future work should integrate more systematic ceramic form, function and fabric classifications with the molecular residue data. This would allow lipid patterns to be assessed not only by site, but also by vessel technology, morphology, surface treatment and probable use (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

In addition, targeted analysis of fruit acids and other plant-associated compounds could help evaluate whether decorated or functionally distinctive vessels were associated with specific plant-processing practices. Such work would be particularly useful for testing whether plant-related signals detected through APAA-C_{18} distributions correspond to particular vessel categories or broader patterns of repeated use (Bondetti et al., 2021; Regert, 2011).

This Chapter contributes to the reconstruction of historical foodways in Babitonga Bay by showing that ceramic lipid residues can preserve evidence for varied combinations of terrestrial, plant and aquatic-compatible resources across socially and historically distinct sites. The results point to flexible and locally situated food preparation practices shaped by environmental availability, ceramic technology, household economy and market integration.

6.Organic residue analysis results from Rio Grande do Sul: long-term changes in foodways and social adaptation

Parts of this Chapter have been adapted from Di Muro et al., (submission in progress). "Long-term variability in food habits in Southern Brazil revealed by organic residue analysis of archaeological cooking wares."

6.1. Introduction

Building on the analysis of rural and coastal communities in Babitonga Bay **(Chapter 5)**, this Chapter shifts focus to an urban and industrialized context in Southern Brazil. It presents the ORA of ceramic cooking wares from Casa 2 (Figure 40C), a 19th century elite residence in Pelotas, in the Patos Lagoon area (Rio Grande do Sul).

By examining foodways within the framework of a rapidly expanding agribusiness economy, this case study investigates how industrialization, urbanization, and social stratification shaped household dietary practices. Comparisons with Chapter 5 highlight regional contrasts between market-dependent and ecologically flexible food systems.

While the economic impact of the *charque* industry is well documented, household-level food practices remain poorly understood. Historical and archaeological sources indicate that while enslaved and working-class populations relied on salted beef as a dietary staple, elite families had access to a more varied repertoire, including fresh meat, dairy, imported goods, and regional produce (Ribeiro, 2017). This Chapter explores how such culinary choices are reflected in the lipid and isotopic profiles of ceramic vessels from Casa 2, offering a window into class-based consumption and dietary change in an emerging urban elite setting.

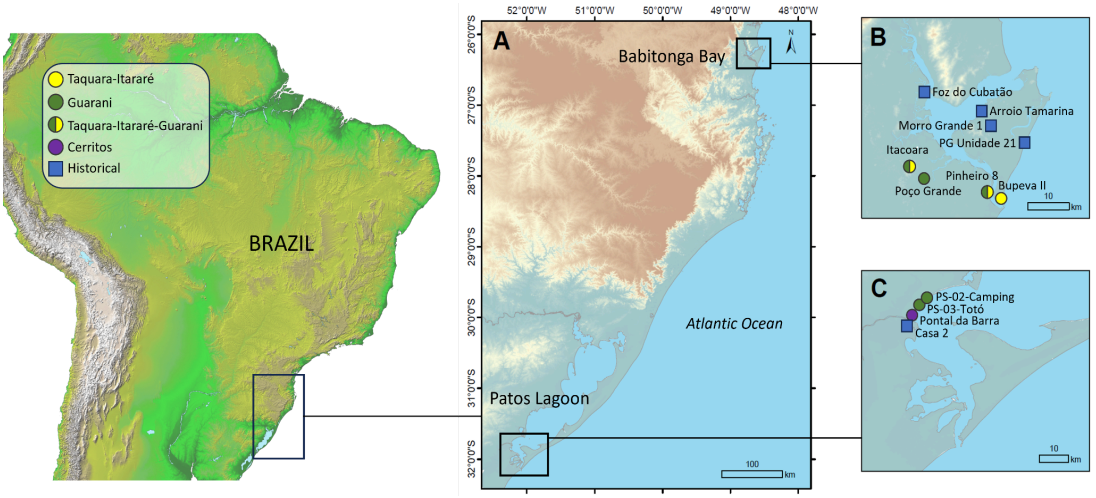


Figure 40: *Location of archaeological sites mentioned in the study, with emphasis on Casa 2, the primary focus of this Chapter. Panels A-B show the wider regional and local setting of the Patos Lagoon area and Pelotas, while panel C indicates the location of Casa 2. Map created using ArcGIS Pro and edited in Inkscape.*

As outlined in **Chapters 1** and **2**, the 19th and early 20th centuries were marked by major transformations in Brazilian food production and consumption, especially with the rise of export-oriented industries. In Rio Grande do Sul, Pelotas became a major hub of industrial agriculture through the development of *charqueadas*, large-scale establishments for producing dried and salted meat which structured the local economy and reinforced rigid social hierarchies (Schwartz, 1986; Gabriel and Silva, 2019). Urban growth brought with it new material cultures, domestic goods, and imported commodities, reflecting the growing power and aspirations of elite households (Vargas, 2017; Ribeiro, 2017).

6.2. Site and ceramic assemblage

6.2.1. Casa 2

Located in Pelotas, Rio Grande do Sul (Figure 40 A-C), and dated to around 1830, Casa 2 represents a 19th century urban residence associated with Brazil's emerging agribusiness elite (Figure 41A). The house was owned by Baron Butuí, a

prominent figure who profited from the region's booming cattle industry and the expansion of *charqueadas* (Bastos, 2014). As such, Casa 2 offers a valuable case study for understanding how industrialization and economic specialisation shaped domestic life, particularly in terms of food consumption and culinary practices.

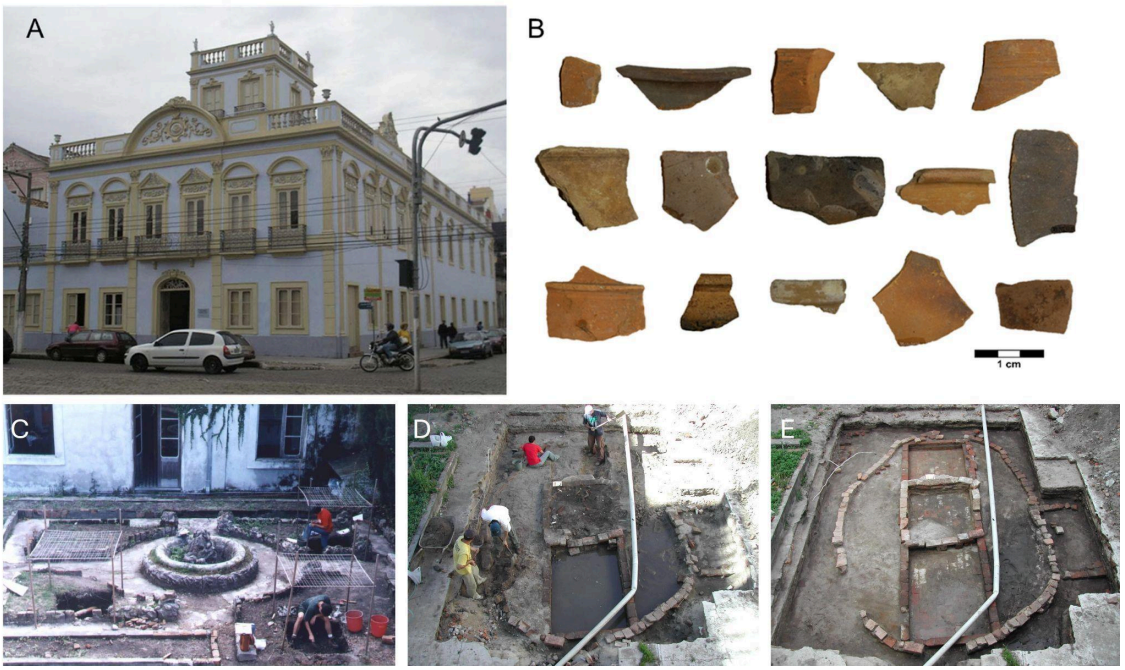


Figure 41: *A) A view of Casa 2 in Pelotas and B) ceramic sherds analysed in this study. C-E) View of the excavation in the backyard of House 2, where a structure used for whitewash production, and also as a domestic refuse pit, was found. Pictures: Archive of LEPAARQ-UFPEL.*

Casa 2 functioned not only as a residence but also as a commercial hub, possibly involved in the storage and sale of *charque* and cattle trading (Grillo, 2020). This dual-purpose nature suggests that food preparation and consumption were closely tied to broader economic activities. Historical records indicate that similar urban households in Pelotas operated as mixed-use spaces, reflecting the interweaving of domestic and commercial spheres in the daily lives of elite families (Grillo, 2020).

Archaeological excavations at Casa 2 recovered a substantial assemblage of ceramic cooking wares. These artefacts, primarily locally produced and undecorated, offer direct evidence of cooking techniques, food processing, and dietary habits in a stratified urban context (Figure 41B). The predominance of utilitarian ceramics may

indicate that these vessels were associated with everyday food preparation activities, possibly carried out by enslaved or low-status domestic workers and not by elite household members (da Silva Peixoto and Cerqueira, 2006, Peixoto, 2009). However, this association should be treated as a contextual interpretation, instead of a direct inference from vessel form alone. Their functional nature, as opposed to imported or decorated tableware, aligns with patterns observed in other working-class or enslaved domestic contexts (Peixoto, 2009; Ribeiro, 2017).

While Casa 2 reflects a highly market-oriented, industrialized food economy, the Babitonga Bay sites illustrate more flexible subsistence strategies, where communities continued to rely on a combination of marine and terrestrial resources (Costa, 2018; Fossile et al., 2024). Meanwhile, pre-colonial settlements offer a glimpse into older culinary traditions, revealing both continuity and rupture in food practices under colonial and post-colonial pressures. The initial Casa 2 sherd list included 20 records. The dataset for this Chapter retained 14 extracts with interpretable lipid concentrations above the minimum threshold. Six additional records did not produce interpretable lipid data during initial screening and were not taken forward for further molecular or isotopic analysis. Six of the interpretable samples were also analysed by total lipid extraction followed by TMS derivatisation (TLE/TMS). However, the available Casa 2 TLE/TMS chromatograms were affected by laboratory hexane contamination and for this reason were retained as analytical documentation and not used for source-specific interpretation. The main molecular interpretation of the Casa 2 assemblage is consequently based on the AE, SIM/APAA, isoprenoid and CSIA datasets.

6.3. Results

6.3.1. Lipid recovery and composition

Lipid concentrations above the practical screening threshold used in this thesis for absorbed residues (>5 μg g^{-1}) were obtained from 14 AE extracts retained for molecular and isotopic interpretation, with concentrations ranging from 5.22 to 175.4

µg g^{-1} (Appendix 4). Six additional records from the initial Casa 2 list were not retained for AE-based interpretation because they produced low-yield or non-interpretable lipid signals during initial screening. These records are retained in Appendix 4 as part of the analytical record but are not included in the interpreted AE dataset used in this Chapter. Figure 42 presents a representative chromatogram from the Casa 2 assemblage, included to illustrate the principal compounds and overall chromatographic quality of the extracts that will be discussed in this Chapter. The AE extracts were dominated by saturated fatty acids ($C_{12:0}$ - $C_{24:0}$), with palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids as the most abundant compounds.

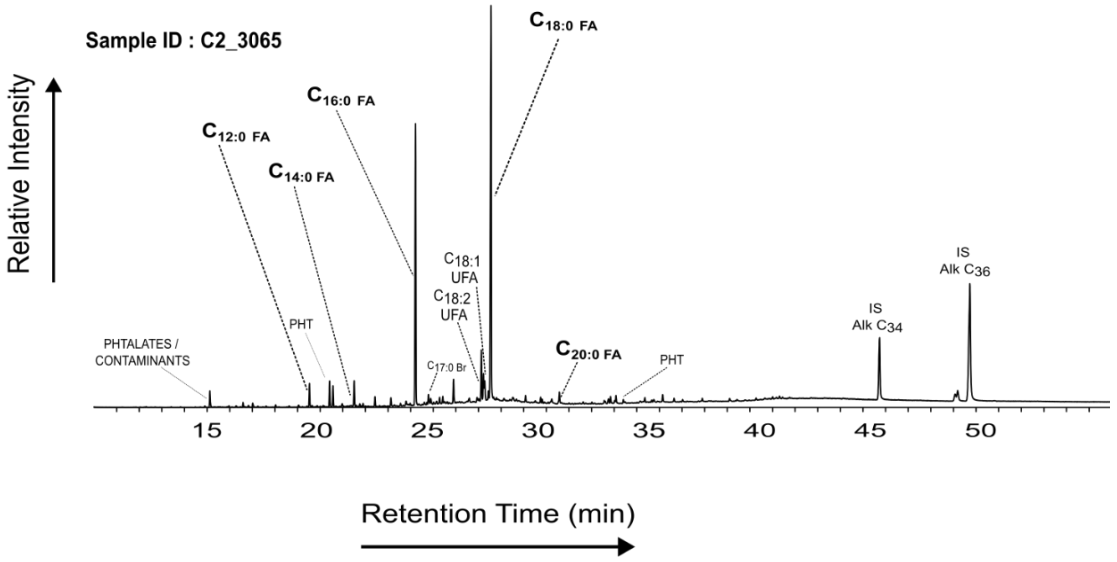


Figure 42: *Representative AE chromatogram from the Casa 2 ceramic assemblage. The chromatogram shows an interpretable acidified methanol extract from sample C2_3065, illustrating the principal fatty acid profile used for molecular interpretation in Chapter 6. Major fatty acids, internal standards and identifiable contaminant peaks are labelled where relevant. Additional Casa 2 chromatograms, including TLE/TMS outputs affected by hexane/ laboratory background contamination and retained as analytical documentation only, are provided in Appendix 5.*

P/S ratios ranged from 0.6 to 6.9 across the 14 AE extracts retained for molecular and isotopic interpretation. Six samples had P/S ratios between 1.1 and 6.9, but these values were not treated as source-specific evidence for plant-derived lipids. P/S ratios are compatible with degraded animal, plant or mixed lipid inputs, but they are not diagnostic of source and were interpreted only alongside APAA distributions, isoprenoid compounds, CSIA values and archaeological context (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Trace amounts of monomethyl-branched fatty acid candidates ($C_{15:0Br}$ and $C_{17:0Br)}$, short-chain dicarboxylic acid candidates and cholesterol-related compounds were recorded in the retained AE dataset (Appendix 4). These compounds are compatible with degraded animal or mixed lipid inputs, lipid oxidation and/or microbial contributions, but they are not treated as source-specific evidence in isolation (Dudd, Regert and Evershed, 1998; Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Lauric ($C_{12:0}$) and myristic ($C_{14:0}$) acids were recorded in trace amounts, but these compounds may derive from multiple plant, animal or mixed lipid inputs, including dairy fats, and were not used as independent indicators of plant processing (Evershed, 2008; Regert, 2011). Monounsaturated and polyunsaturated fatty acids, where present, were treated as supporting but non-diagnostic evidence because they may reflect mixed lipid inputs, degradation or background contributions. Possible abietic acid and terpenoid-related compounds were also treated cautiously and were not used as direct evidence for specific fuel, resin or plant-processing practices.

APAA-C_{18} E/H values were recorded in two Casa 2 samples. One sample had an APAA-C_{18} E/H value above 4 and is treated as plant-processing proxy positive, while the other remained below this threshold (TRD_693/C2-3065 and TRD_702A-B/C2-3453, respectively; Appendix 4; Bondetti et al., 2021). APAA-C_{20} was also recorded in two samples, both of which had APAA-$C_{20/C18}$ values above 0.06 and are treated here as aquatic-compatible proxy positive (TRD_687/C2-5773 and TRD_702A-B/C2-3453; Appendix 4). These proxy values are interpreted cautiously and in combination with lipid concentration, fatty acid distributions, isoprenoid compounds, CSIA values and

archaeological context, not as definitive source assignments (Hansel et al., 2004; Evershed, 2008; Regert, 2011; Bondetti et al., 2021).

Six Casa 2 samples were also analysed by TLE/TMS as a complementary extraction strategy. However, the available TLE/TMS chromatograms were affected by laboratory hexane contamination, which limited their interpretative value. For this reason, TLE/TMS-derived MAG, DAG, TAG, sterol or plant-wax assignments were not retained for source-specific interpretation in the Casa 2 dataset. The TLE/TMS outputs are reported separately in Appendix 5 as part of the analytical record, while the interpretation presented here is based on the AE dataset, including lipid concentration, fatty acid distributions, APAA values, isoprenoid compounds and CSIA results.

6.3.2. Compound-specific isotope analysis

Compound-specific carbon isotope analysis of palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids was used to assess broad isotopic patterning in the Casa 2 assemblage. APAA-C_{18} E/H values were recorded in two Casa 2 samples, one of which exceeded the threshold of 4 and is treated as plant-processing proxy positive. APAA-$C_{20/C18}$ values above 0.06 were recorded in two samples, providing limited aquatic-compatible proxy evidence (Appendix 4; Hansel et al., 2004; Bondetti et al., 2021). One aquatic-compatible proxy positive sample also showed enriched $\delta^{13}C_{16:0}$ and $\delta^{13}C_{18:0}$ values, but this pattern is interpreted cautiously because enriched carbon isotope values may reflect aquatic-derived lipids, C_4 plant inputs or mixed residues. The Casa 2 dataset provides limited molecular and isotopic evidence for plant-processing and aquatic-compatible inputs, but these results should not be treated as direct evidence for specific plant foods, fish, marine resources or other specific aquatic resources.

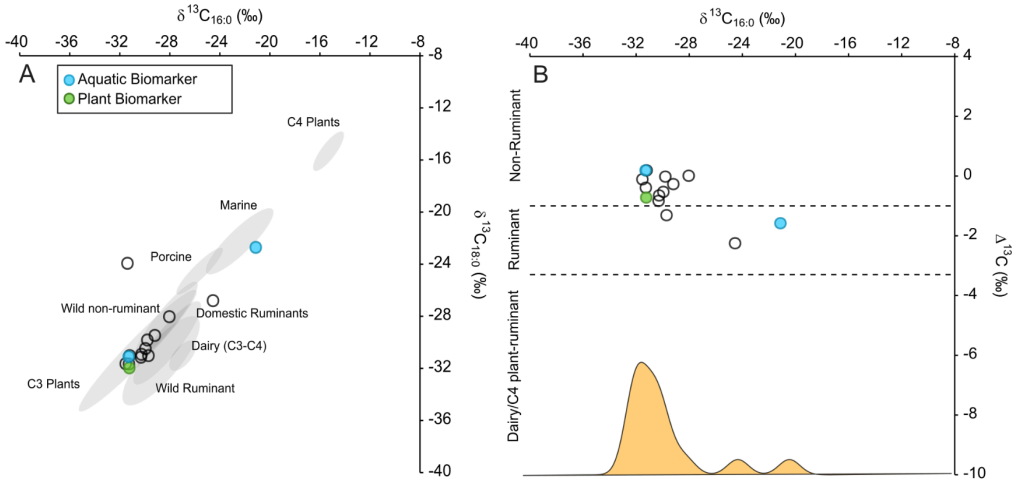


Figure 43: *A) Stable carbon isotope values of fatty acids ($C_{16:0}$ and $C_{18:0}$) from Casa 2, compared with modern reference ellipses (68% confidence) (Appendix 7). B) $\Delta^{13}C_{18:0\text{-}16:0}$ values against $\delta^{13}C_{16:0}$. Plant-processing proxy-positive samples are indicated by APAA-C_{18} E/H > 4; aquatic-compatible proxy-positive samples are indicated by APAA-$C_{20/C18}$ > 0.06.*

The remaining samples plotted within or close to broad reference areas compatible with C_3 plant and terrestrial animal lipid inputs. These values suggest that the preserved residues in the sampled Casa 2 vessels were more strongly associated with terrestrial/C_3-compatible signatures than with aquatic-compatible or C_4-enriched inputs (Figure 43).

6.4. Discussion

The Casa 2 lipid results provide evidence for the types of residues preserved in a small assemblage of historical ceramic vessels from an urban context in Pelotas. Instead of offering a direct reconstruction of household diet, these results are interpreted as evidence for vessel use, residue preservation and food-processing tendencies within the sampled ceramic assemblage. The dataset suggests a predominance of terrestrial/C_3-compatible lipid signatures, with limited plant-processing and aquatic-compatible proxy evidence. These patterns can be

discussed in relation to the broader social and economic context of 19th century Pelotas, including urban provisioning systems and the economic influence of the charque industry, while recognising the limits of a small ceramic residue dataset.

The Casa 2 lipid data do not provide direct evidence for social resistance, oppression or ethnic identity in isolation. Rather, they provide household-level evidence for the range of products processed in the analysed vessels, which can be interpreted in relation to the archaeological context of Casa 2, its association with an urban elite household, and the broader historical setting of Pelotas as a centre of charque production, slavery, post-abolition labour reorganisation and market expansion. Within this framework, the molecular and isotopic results contribute to the discussion of culinary practice, access to resources and household consumption, without acting as direct biomarkers of social identity or political experience.

The preserved residues from Casa 2 are more consistent with terrestrial/C_3-compatible inputs than with strongly aquatic-compatible or C_4-enriched signatures (Figure 43; Appendix 4). However, this pattern should not be treated as direct evidence for the absence of fish, C_4 plants or other resources from the wider diet, since ceramic lipid residues reflect preserved vessel-use signals rather than complete dietary assemblages (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Instead, the results indicate that these resources were only weakly represented in the absorbed lipid residues preserved in the sampled ceramic vessels. One sample showed APAA-C_{18} E/H values above 4, providing limited plant-processing proxy evidence, while two samples had APAA-C_{20}/C_{18} values above 0.06, providing limited aquatic-compatible proxy evidence (Hansel et al., 2004; Bondetti et al., 2021). These results are not sufficient to identify specific plant foods, fish or marine resources, but they indicate that occasional plant-processing and aquatic-compatible inputs were present in the assemblage.

The weak representation of C_4-enriched lipid signatures in the Casa 2 residues is also shaped by preservation, lipid yield and the kinds of products likely to leave

detectable traces. Low-lipid plant products such as sugarcane may be poorly represented in absorbed ceramic residues, while maize-derived lipids may be detectable under some circumstances, as shown in regional archaeological studies (Huang et al., 2017; Admiraal et al., 2023). In addition, C_4 plant inputs may enter ceramic residues indirectly through animal products when domestic animals consumed maize or other C_4 fodder. Although the Casa 2 CSIA values do not provide strong evidence for such inputs in the sampled vessels, this does not demonstrate their absence from the wider food system (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). Similarly, the lack of clear dairy-compatible lipid signatures should not be read as evidence that dairy products were absent from 19th century Pelotas. It only indicates that dairy was not clearly represented in the preserved residues analysed here, despite the broader historical importance of cattle and dairy products in regional foodways and social practices (Azevedo and Zanotto, 2023).

The Casa 2 results may also reflect the selective use of ceramic vessels alongside other cooking technologies, such as metalware, or glassware. Ceramic vessels may have been used for particular preparations, while other foods, including those associated with elite consumption or market provisioning, may have been processed in different containers or left limited lipid traces (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a). This is particularly relevant in an urban household context where food preparation, service and consumption may have involved different social actors and material practices. Where the archaeological context supports an association between parts of the assemblage and low-status or enslaved individuals, the lipid results may provide insight into the kinds of food-processing activities preserved in ceramics used within that social context. Nevertheless, interpretations involving social resistance, food segregation or inequality should be treated as historically informed possibilities and not direct conclusions from the lipid data alone (Lima, 2009; Lunsford, Arthur and Porter, 2021).

Compared with the Babitonga Bay assemblages, Casa 2 shows fewer aquatic-compatible molecular indicators and a narrower isotopic spread. This contrast

may reflect differences between an urban, market-oriented context in Pelotas and the more coastal/rural Babitonga Bay sites, where aquatic resources were more strongly represented in the archaeological and historical record (Saint-Hilaire, 1936; Fossile et al., 2023, 2024). The more restricted isotopic variability observed in Casa 2 may reflect differences in vessel use, cooking practices, ceramic fabric, preservation, access to resources and the use of other cooking technologies alongside pottery (Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

The Casa 2 results also differ from the broader isotopic variability reported for pre-colonial ceramic vessels from the Patos Lagoon region, where previous studies have identified evidence compatible with aquatic resources and C_4 plant inputs, including possible maize use (Admiraal et al., 2023, 2025) (Figure 44). However, this comparison should be made cautiously because the assemblages differ in chronology, cultural context, vessel function, ceramic technology, sample size and preservation history. The narrower range of Casa 2 values may indicate more restricted or more specialised ceramic use in the sampled historical assemblage, instead of a simple decline in food diversity. The comparison with Babitonga Bay similarly suggests regional differences in the products represented in ceramic residues and also contrasts in preserved pottery-use.

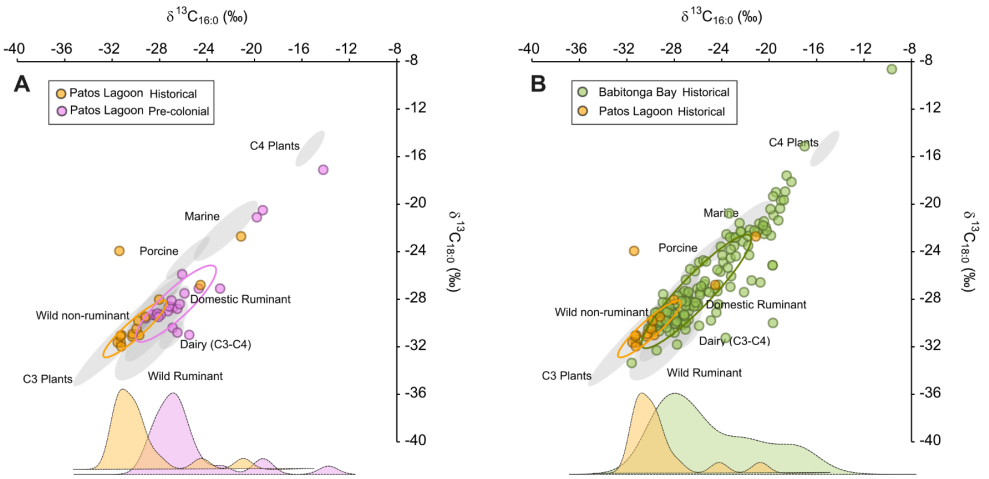


Figure 44: *Comparison of compound-specific $\delta^{13}C$ values of palmitic ($C_{16:0}$) and stearic ($_{C18:0}$) acids from historical Casa 2 vessels and comparative ceramic assemblages. (A) Casa 2 historical vessels compared with pre-colonial ceramic artefacts from the Patos Lagoon region. (B) Historical vessels from Casa 2/Pelotas compared with historical ceramic assemblages from Babitonga Bay. Kernel density estimates represent $\delta^{13}C_{16:0}$ values. Data are compared with modern reference ellipses representing 68% confidence intervals.*

Taken together, the Casa 2 results suggest that the sampled ceramic vessels preserved a relatively narrow range of terrestrial/C_3-compatible lipid signatures, with limited plant-processing and aquatic-compatible proxy evidence. This pattern can be interpreted in relation to the urban and industrial context of 19th century Pelotas, where food provisioning, labour relations and the charque economy shaped access to resources and culinary practices (Bastos, 2014; Bastos et al., 2016).

The interpretative scale of the Casa 2 dataset is shaped by sample size, preservation and analytical recovery. 14 AE extracts produced interpretable lipid profiles above the practical screening threshold and were retained for molecular and isotopic interpretation, while six additional records from the initial Casa 2 list were not retained for AE-based interpretation because they produced low-yield or non-interpretable lipid signals during initial screening. These non-interpretable results are relevant because they highlight the preservation and recovery limits of the

assemblage. In addition, the Casa 2 TLE/TMS subset was affected by laboratory hexane contamination and was retained as analytical documentation and not used to support source-specific assignments. Lipid survival and analytical recovery may be affected by vessel use, ceramic fabric, heating conditions, burial environment, microbial activity, post-excavation handling, storage conditions and laboratory background contamination (Dudd, Regert and Evershed, 1998; Evershed, 2008; Regert, 2011; Roffet-Salque et al., 2017a).

Within this framework, Chapter 6 contributes to the wider thesis by showing how an urban historical assemblage from Pelotas differs from both pre-colonial Patos Lagoon pottery and contemporary historical assemblages from Babitonga Bay (Admiraal et al., 2023, 2025; Fossile et al., 2023, 2024). These differences suggest variation in pottery use, resource representation and culinary practice across regions and social contexts, while also demonstrating the need to integrate ORA results with ceramic, faunal, isotopic and historical evidence when discussing foodways in post-colonial Southern Brazil.

7. Integrating organic residue analysis and stable isotope analysis: an overview from Santa Catarina and Rio Grande do Sul

7.1. Introduction

This Chapter presents the results of an integrative approach to dietary reconstruction in historical Southern Brazil by combining stable carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) isotope analysis of human and faunal bone collagen with the ORA findings previously presented in Chapters 5 and 6. The stable isotope dataset focuses on five archaeological assemblages: Igreja Matriz de Nossa Senhora da Graça (São Francisco do Sul, Santa Catarina), Cúria Metropolitana / former Igreja Matriz de Nossa Senhora Madre de Deus (Porto Alegre, Rio Grande do Sul), Casa 8 (Pelotas, Rio Grande do Sul), and the Babitonga Bay faunal assemblages from Morro Grande 1 (MG1) and Praia Grande - Unidade 21 (PG-U21), all associated with occupations dating to the 19th and early 20th centuries (Figure 45). These data are interpreted alongside the ORA results from the ceramic assemblages discussed in Chapters 5 and 6, particularly Casa 2 in Pelotas, which provides the main ceramic-residue dataset for Rio Grande do Sul. The human remains analysed here derive from ecclesiastical contexts, whereas the faunal remains were recovered from rural, domestic and religious settings. The faunal dataset includes cattle, pigs, chickens, fish and wild species, reflecting diverse livelihood strategies such as local husbandry, market provisioning and informal trade networks.

For clarity, the assemblages are referred to consistently in this Chapter as follows: Casa 8 refers to the Pelotas domestic faunal assemblage; Porto Alegre assemblage refers to the human and faunal remains from the Cúria Metropolitana / former Igreja Matriz de Nossa Senhora Madre de Deus; São Francisco do Sul sample refers to the human remains from the Igreja Matriz de Nossa Senhora da Graça; and MG1 and PG-U21 refer to the Babitonga Bay faunal assemblages from Morro Grande 1 and Praia Grande - Unidade 21.

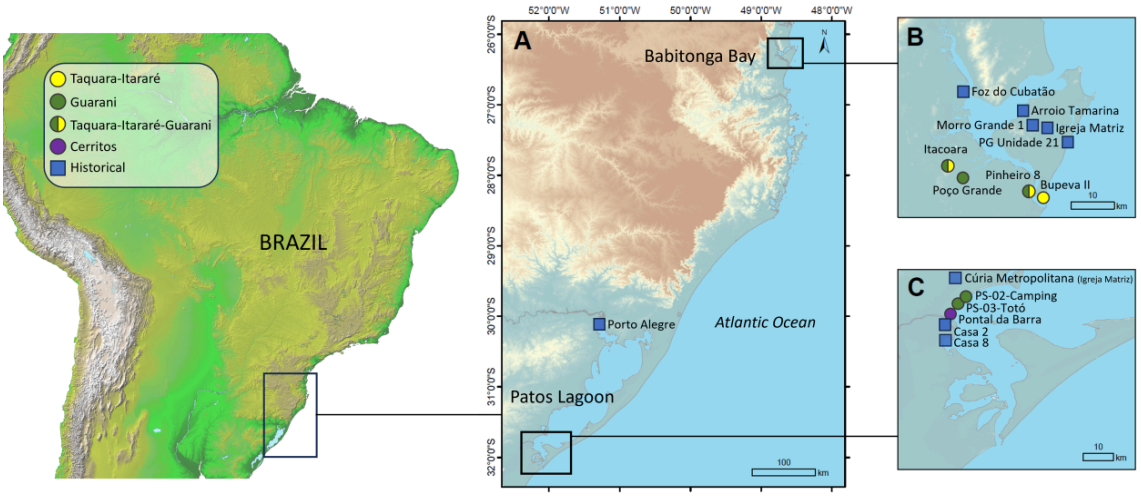


Figure 45: *Location of the archaeological sites discussed in Chapter 7. Blue squares indicate historical sites analysed in this thesis, while coloured circles indicate pre-colonial comparative sites used in the discussion: yellow = Taquara-Itararé, green = Guarani, half yellow/green = Taquara-Itararé-Guarani, and purple = Cerritos. Panel A shows the wider Southern Brazilian setting, Panel B focuses on Babitonga Bay, and Panel C focuses on the Patos Lagoon region. Map created using ArcGIS Pro and edited in Inkscape.*

Building on the methodological framework introduced in **Chapter 3 and 4,** SIA is used here to reconstruct the relative contributions of terrestrial and aquatic protein sources to human and faunal diets. Combined with the ORA results discussed in **Chapters 5 and 6**, alongside zooarchaeological and historical documentary evidence, this integrated approach offers a holistic understanding of how environmental factors, social stratification, and economic systems shaped culinary practices in colonial and post-colonial Brazil. The Chapter also situates these results within the broader Brazilian stable isotope record, in order to assess whether the patterns observed in Babitonga Bay and Rio Grande do Sul align with, or differ from, published isotopic evidence from coastal, lagoonal, inland, and historical contexts in Brazil.

7.2. Archaeological context and material

7.2.1. Casa 8 - Pelotas

Casa 8, also known as the Residência Conselheiro Antunes Maciel, is located in the historic centre of Pelotas, Rio Grande do Sul (Figure 46). The residence was excavated between 2004 and 2005 as part of a salvage archaeology project conducted by the Universidade Federal de Pelotas (UFPel) in collaboration with the *Programa Monumenta*, during urban revitalisation works in the city's historic district. The site forms part of a dense archaeological zone associated with continuous urban occupation since the late 18th century, and its excavation revealed stratified deposits that reflect multiple phases of domestic activity (Nobre, 2012; Cerqueira, Peixoto and Viana, 2004). In this Chapter, Casa 8 is used as the principal faunal and contextual comparator for the Rio Grande do Sul assemblage, while the human isotope data derive from the Porto Alegre ecclesiastical context discussed below (Cúria Metropolitana) .



Figure 46: *Casa do Conselheiro Antônio Maciel, (Casa 8) located in the historic centre of Pelotas, Rio Grande do Sul (Image source: acasasenhorial.org, 2023).*

Historically associated with the Maciel Family, part of Pelotas' urban elite during the late 19th century, Casa 8 preserves architectural elements typical of 19th century urban dwellings in Pelotas, including a symmetrical façade, internal courtyard,

and rear service areas (Cerqueira, Peixoto and Viana, 2004; Fredel, 2014). These spatial arrangements mirror broader patterns of elite domestic architecture in Southern Brazil, combining public reception spaces with secluded kitchen and utility zones used by enslaved and hired labourers (Ribeiro, 2017; Fredel, 2014).

Archaeological investigations recovered a wide variety of material culture, including imported ceramics (e.g., Portuguese faience, English pearlware and creamware), local low-fired earthenware, glass, metal fragments, and faunal remains (Cerqueira, Peixoto and Viana, 2004; Nobre, 2012). The faunal assemblage analysed in this study (n = 10), which derives from these excavations, includes remains of cattle (*Bos taurus*), pig (*Sus scrofa*), chicken (*Gallus gallus*), black drum (*Pogonias cromis*), opossum (*Didelphis* sp.), and domestic dog (*Canis lupus familiaris*), offering insight into provisioning strategies, dietary preferences, and the interplay between local and global foodways (see Table 2, Chapter 4). The co-occurrence of exotic tablewares and diverse faunal remains suggests a hybrid household economy that combined elite consumption practices with utilitarian provisioning patterns, potentially mediated by the labour of enslaved or marginalised workers. This configuration aligns with other historical residences in the region, where material culture and food remains reveal the social complexity and household dynamics of urban elites in 19th century Pelotas.

7.2.2. Cúria Metropolitana: *Igreja Matriz de Nossa Senhora Madre de Deus, Porto Alegre*

The archaeological site of the Cúria Metropolitana de Porto Alegre is located in the historic centre of Porto Alegre, Southern Brazil (Figure 47). This area once housed the **Igreja Matriz de Nossa Senhora Madre de Deus**, the city's first parish church, founded in the late 18th century (Cappelletti, 2013). Construction of the Igreja Matriz began in 1772, and the church quickly became the primary religious centre for the early colonial settlement. It served not only as a spiritual hub but also as a burial ground for the community, with an associated cemetery extending around the church structure (Cappelletti, 2013). In the following sections, the human and faunal remains

from the Cúria Metropolitana / Igreja Matriz de Nossa Senhora Madre de Deus are referred to as the Porto Alegre assemblage.



Figure 47: *Two views of the Cúria Metropolitana: Igreja Matriz de Nossa Senhora Madre de Deus, located in Porto Alegre, Rio Grande do Sul. The left image shows the historical appearance of the church in an earlier 20th-century photograph, while the right image presents its present-day façade (Cappelletti, 2013).*

The original Igreja Matriz was demolished in 1929 to make way for the construction of the modern Catedral Metropolitana, yet archaeological and historical evidence confirm that significant portions of the old sacred space remained beneath the urban fabric (Cappelletti, 2013).

In 2012 and 2013, archaeological excavations at the church were conducted during restoration and infrastructure works on the grounds of the Cúria Metropolitana. The interventions focused on an external courtyard and revealed the presence of both primary and secondary funerary contexts (Cappelletti, 2013). Primary inhumations were encountered in articulated condition, with individuals buried in extended dorsal position, oriented northward toward the former altar, in accordance with Catholic burial traditions. Some burials included associated artefacts such as rosaries, crucifixes, and glass bead necklaces, reflecting religious and cultural identities of the deceased. These primary burials are interpreted as having been located outside the original church building, but within its consecrated grounds (Cappelletti, 2013).

In addition to the primary graves, excavations revealed a large secondary deposit of human remains, referred to in an area which is called in the archaeological report as the *Concentração de Ossos* (Bones Concentration) *(Cappelletti, 2013)*. This deposit contained at least ninety-nine crania and hundreds of postcranial bone fragments, all disarticulated and without anatomical association. Historical documents and stratigraphic evidence suggest that these remains were likely displaced during 19th and early 20th century building activities, possibly linked to the demolition of the Igreja Matriz itself or to earlier modifications of the church's funerary spaces, such as the dismantling of an associated catacomb recorded in 19th century maps (Cappelletti, 2013).

The human bone assemblage analysed in this study (n = 16) derives from both of these archaeological contexts (see Table 2, Chapter 4). A first set of samples consists of cranial fragments recovered from the secondary deposit. These remains are fragmentary, disarticulated, and cannot be associated with specific individuals or burial events (Cappelletti, 2013). A second set of samples comes from primary inhumations. These remains, including mandibles, long bones, and other anatomical elements, are associated with specific graves and correspond to individuals buried in situ. Preliminary osteological analysis indicates that the buried population included individuals of both European and African ancestry, consistent with the demographic composition of Porto Alegre during the colonial and early post-colonial periods. Crania exhibiting African-derived morphological traits, particularly among individuals associated with ritual artefacts such as glass bead necklaces, point to the presence of Afro-descendant individuals within the cemetery population (Cappelletti, 2013). In addition to the human remains, the excavations also yielded a substantial quantity of animal bones, primarily cattle, recovered from both primary and secondary contexts, often intermingled with human material (Cappelletti, 2013). Selected cattle fragments from this faunal assemblage were included in the present study (n = 6). Because part of the Porto Alegre assemblage derives from secondary deposits, the isotope values are interpreted primarily at the sample level instead as tightly contextualised individual biographies.

7.2.3. Igreja Matriz Nossa Senhora da Graça, São Francisco do Sul

The Igreja Matriz Nossa Senhora da Graça, located in the historic centre of São Francisco do Sul, is one of the most significant colonial religious structures in Southern Brazil (Figure 48). Constructed between 1768 and 1771, the church replaced earlier chapel structures that had served the town's population since the 17th century (Alves, 2004; Borba, 2017). Over time, the building underwent numerous renovations and expansions, including the addition of a second bell tower in the 19th century, the construction of the churchyard walls, and other architectural modifications (Alves, 2004; Borba, 2017). These changes reflect the growing prominence of São Francisco do Sul as a regional administrative and religious centre during the colonial and imperial periods. As shown in Figure 46, the church's appearance has changed substantially from the 19th century to the present, including the completion of a second bell tower and the restoration of the façades during modern conservation efforts.



Figure 48: *Igreja Matriz Nossa Senhora da Graça, São Francisco do Sul. Left: early photograph showing the church prior to full symmetrical tower construction in 1783-1926. Right: current view following restoration works (photo credit: Hinsching, 2019).*

In the following sections, the human remains from Igreja Matriz Nossa Senhora da Graça are referred to as the São Francisco do Sul sample, while Babitonga Bay is used for the wider regional context that also includes MG1 and PG-U21.

Archaeological excavations carried out in 2004 as part of the *Programa Monumenta* restoration project yielded important information on the material culture, spatial organisation, and social history of the site. The investigations focused on the churchyard and adjacent spaces, revealing construction phases, refuse deposits, and a variety of artefact types (Alves, 2004; Borba, 2017). Recovered materials included domestic and fine ceramics (e.g., faiança, pearlware, creamware), glassware, metal objects, and faunal remains, indicating both ecclesiastical and residential uses of the structure. These findings support historical interpretations that the church also functioned as a residence and hospitality space for clergy and visiting elites between 1752 and 1797 (Alves, 2004; Borba, 2017).

The human bone assemblage analysed in this study (n= 7) derives from the archaeological excavations conducted during the 2004 restoration of the *Igreja Matriz Nossa Senhora da Graça* in São Francisco do Sul. A first group of samples comprises disarticulated cranial and postcranial fragments recovered from secondary deposits across the churchyard, particularly from the western and northern sectors (Alves, 2004; Borba, 2017). These remains were fragmentary and lacked anatomical articulation, precluding associations with specific individuals or burial events.

A second group of samples consists of anatomical elements such as mandibles, long bones, and vertebrae recovered from primary inhumations corresponding to individuals buried *in situ*. Although seventeen graves were identified, only two adult individuals were documented, one of whom was confidently identified as male (Alves, 2004; Borba, 2017). The remaining skeletons correspond to subadults, and sex estimation was not possible due to skeletal immaturity. Collagen preservation was generally favourable, particularly in specimens recovered from compact argillaceous layers containing charcoal and shell inclusions (Alves, 2004; Borba, 2017).

The funerary context of these individuals suggests that they belonged to lower socioeconomic strata. Burials were densely arranged, frequently overlapping, and in several cases disturbed by subsequent interments, indicating a lack of permanent grave

markers or spatial segregation. Most individuals were interred without coffins or formal grave goods, with the exception of a few simple bone artefacts-possibly beads or pendants-found in association with child burials (Alves, 2004; Borba, 2017). These patterns contrast with documented practices for wealthier members of society, who were buried within the church interior, often with greater care and material investment. The overall configuration of the cemetery supports the interpretation that the churchyard served primarily as a burial ground for impoverished or marginalised individuals, including enslaved persons and others excluded from elite funerary spaces (Alves, 2004; Borba, 2017).

Overall, 143 samples were selected for stable isotope analysis, comprising 23 human samples and 120 faunal remains (see Chapter 4, Table 2).

7.3. Results

7.3.1.Faunal isotopic results by region

The faunal isotope values are summarised in Table 3 and Figure 49. Presenting the data by region, site and faunal/resource group makes the local baselines clearer than listing each taxon separately in the text, while still retaining the main isotopic contrasts needed for interpretation.

In Rio Grande do Sul, the faunal assemblage from Casa 8 and the Porto Alegre assemblage indicates a strong C_4-influenced terrestrial baseline (Figure 49). C_4-fed domestic herbivores have a mean $\delta^{13}C$ value of -10.83 ± 1.71‰ and a mean $\delta^{15}N$ value of 6.24 ± 1.17‰, consistent with cattle raised on C_4-rich pasture or fodder. Domestic and commensal omnivores occupy higher $\delta^{15}N$ positions, with chickens, pig, dog and opossum reflecting mixed feeding practices and possible access to household-derived or animal-protein resources. The brackish-marine black drum from Casa 8 provides an aquatic reference point for the region, with mean values of -12.40 ± 1.56‰ for $\delta^{13}C$ and 11.05 ± 0.21‰ for $\delta^{15}N$, consistent with assimilation of benthic microalgae and marine phytoplankton in estuarine and coastal food webs (Garcia et al., 2007).

Region	Site	Faunal group	$\delta^{13}C$ mean(‰)	± SD (‰)	$\delta^{15}N$ mean(‰)	± SD (‰)
Rio Grande do Sul	Casa 8	C_4-fed domestic herbivores (cattle)	-10.83	± 1.71	6.24	± 1.17
Rio Grande do Sul	Casa 8	Domestic omnivores (chicken and pig)	-9.33	± 1.11	9.23	± 2.94
Rio Grande do Sul	Casa 8	Commensal/opportunistic omnivores (dog, opossum)	-12.65	± 2.90	10.35	± 0.21
Rio Grande do Sul	Casa 8	Brackish-marine fish	-12.40	± 1.56	11.05	± 0.21
Santa Catarina	PG-U21 / MG1	C_4-fed domestic herbivores (cattle)	-12.15	± 2.25	5.76	± 0.65
Santa Catarina	MG1	C_3 wild terrestrial fauna (deer)	-23.31	± 0.52	4.51	± 1.81
Santa Catarina	PG-U21 / MG1	Domestic/commensal omnivores (chicken, pig,armadillo)	-18.93	± 2.42	9.05	± 2.00
Santa Catarina	MG1 / PG-U21	Brackish-marine fish	-11.63	± 0.73	13.02	± 0.88

Table 3: *Summary of faunal isotope values by region, site and faunal/resource group. Values are reported as mean and standard deviation. Groups were defined to summarise the main local terrestrial, omnivorous and brackish-marine baselines used to interpret the human isotope results.*

In Santa Catarina, the faunal baseline is broader and includes C_4-fed domestic herbivores, C_3 wild terrestrial fauna, domestic and commensal omnivores, and brackish-marine fish. Cattle from PG-U21 and MG1 have mean values of -12.15 ± 2.25‰ for $\delta^{13}C$ and 5.76 ± 0.65‰ for $\delta^{15}N$, indicating substantial C_4 inputs, in line with historically documented foddering and agricultural practices in the region (Cabral, 1937; Noble, 1967). By contrast, wild terrestrial fauna, including deer and Tayassuidae, occupies a more depleted C_3 range, with a mean $\delta^{13}C$ value of -23.31 ± 0.52‰. Domestic and commensal omnivores, including chickens, domestic pigs and armadillos, show more variable values, consistent with mixed plant and animal feeding. The clearest aquatic baseline is provided by brackish-marine fish from MG1 and PG-U21, which show enriched $\delta^{13}C$ values and high $\delta^{15}N$ values, with mean values of -11.63 ± 0.73‰ and 13.02 ± 0.88‰ respectively (Figure 49). These values are consistent with

marine-brackish food webs and provide the main comparative baseline for assessing aquatic protein consumption in the São Francisco do Sul human sample (Fossile et al., 2024).

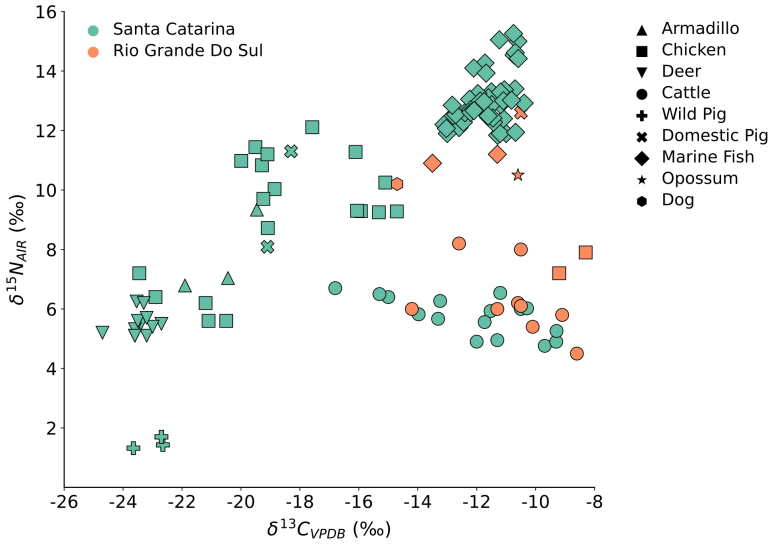


Figure 49: *Scatterplot of faunal δ¹³C and δ¹⁵N values from archaeological sites in Santa Catarina and Rio Grande do Sul. Colours distinguish region, with Santa Catarina shown in green and Rio Grande do Sul in orange, while labels/symbols distinguish faunal taxa or resource groups. The plot illustrates the local faunal baselines used to interpret the human isotope values presented in Figure 50.*

7.3.2.Human isotopic results by region

The human isotope values are summarised in Table 4 and Figure 50. Presenting the data by region and site clarifies the main differences between the Porto Alegre and São Francisco do Sul samples, while the full sample-level results are provided in Appendix 6.

In Rio Grande do Sul, isotopic data from 16 individuals recovered from the Porto Alegre assemblage reveal a dietary signal characterised by relatively high δ¹³C values, ranging from -17.8‰ to -12.1‰ (mean = -14.60‰ ± 1.68), and δ¹⁵N values between 8.33‰ and 15.6‰ (mean = 12.34‰ ± 2.01). This pattern suggests a mixed

diet with substantial contributions from animal protein and C_4-derived products, while the high $\delta^{15}N$ values observed in some individuals may also indicate access to higher-trophic-level protein, including possible aquatic or estuarine resources.

Region	Site / human sample	n	$\delta^{13}C$ range (‰)	$\delta^{13}C$ mean ± SD (‰)	$\delta^{15}N$ range (‰)	$\delta^{15}N$ mean ± SD (‰)
Rio Grande do Sul	Porto Alegre assemblage / Igreja Matriz Nossa Senhora Madre de Deus	16	-17.80 to -12.10	-14.60 ± 1.68	8.33 to 15.60	12.34 ± 2.01
Santa Catarina	São Francisco do Sul sample / Igreja Matriz Nossa Senhora da Graça	7	-16.80 to -14.60	-16.10 ± 0.75	12.90 to 15.70	14.36 ± 0.86

Table 4: *Summary of Human isotope values by region and site. Values are reported as mean and standard deviation.*

The wide range of $\delta^{15}N$ values points to variability in protein intake within the Porto Alegre sample, potentially associated with differences in status, occupation, household provisioning, or individual dietary choices within the urban population (Figure 50B). These values should not be interpreted as evidence for a wholly terrestrial diet, but neither do they require a simple marine/non-marine distinction.

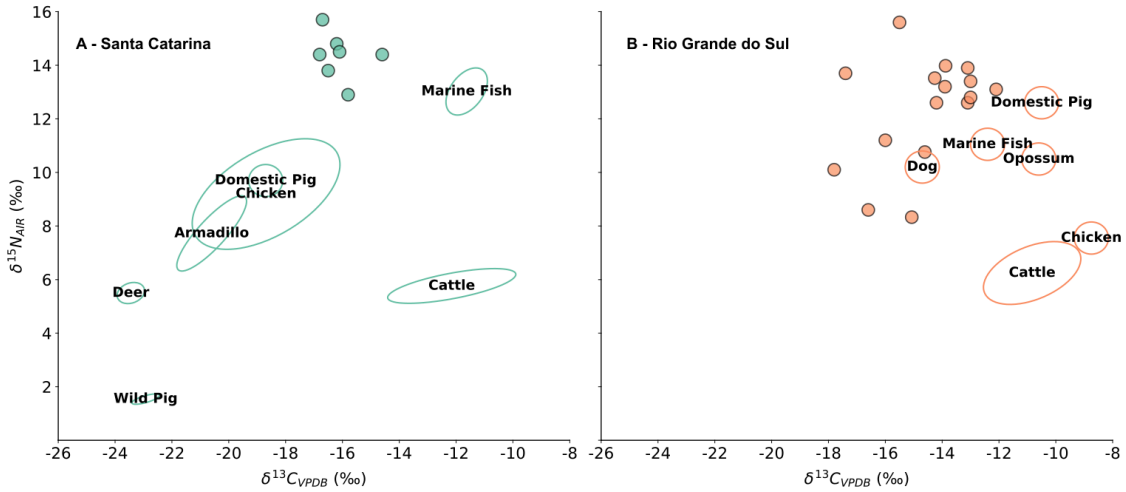


Figure 50: *Human δ¹³C and δ¹⁵N values from Santa Catarina (A) and Rio Grande do Sul (B), plotted against local faunal isotope baselines. Ellipses represent 68% confidence intervals for faunal taxonomic/resource groups by regio**n.***

Because elevated δ¹⁵N values can reflect several processes, including higher-trophic-level terrestrial protein, aquatic or estuarine protein intake, local baseline variation and individual life-history factors, the Porto Alegre data are interpreted here as evidence for dietary heterogeneity and not as definitive evidence for or against marine consumption.

In contrast, human remains from the São Francisco do Sul sample (n = 7) show lower δ¹³C values (range: -16.8‰ to -14.6‰; mean = -16.10‰ ± 0.75) and consistently higher δ¹⁵N values (range: 12.90‰ to 15.70‰; mean = 14.36‰ ± 0.86). These results suggest a protein-rich diet with a stronger marine or estuarine component than that observed for the Porto Alegre sample, in line with the coastal location of the site and the isotopically enriched fish baselines identified for the region (Figure 50A).

7.4. Discussion

The stable isotope results presented in this Chapter provide a comparative perspective on dietary practices in two historical Southern Brazilian settings: Babitonga Bay, represented here by the human remains from the São Francisco do Sul sample,

and Rio Grande do Sul, represented primarily by the Porto Alegre assemblage. By anchoring the human data to locally established faunal baselines, this Chapter allows a comparative interpretation of protein source exploitation and dietary breadth across the two regions. At the same time, the contrast between them is better understood as one of degree and variability instead of as a simple opposition between a "marine" coast and a wholly "terrestrial" urban centre. Human values from São Francisco do Sul cluster within a relatively narrow range, with $\delta^{13}C$ values between -16.8‰ and -14.6‰ and $\delta^{15}N$ values between 12.9‰ and 15.7‰, whereas the Porto Alegre sample is notably broader, with $\delta^{13}C$ values ranging from -17.8‰ to -12.1‰ and $\delta^{15}N$ values from 8.33‰ to 15.6‰. This difference is important because it suggests a more consistent isotopic pattern in the Santa Catarina sample and a more heterogeneous dietary signal in Porto Alegre.

In Babitonga Bay, the isotope values of individuals from São Francisco do Sul remain compatible with a substantial contribution of marine-brackish or estuarine protein, especially when considered in relation to the regional faunal baselines and to the strong aquatic signatures already identified along the Atlantic Forest coast of Southern Brazil. This interpretation also accords with the ORA evidence presented in Chapter 5, where lipid residue analyses from the Babitonga Bay ceramic assemblages identified aquatic-compatible molecular indicators and mixed residue profiles (Admiraal et al., 2023). Instead of reading these patterns as evidence for an exclusively marine diet, however, it is more accurate to see them as indicating a strong aquatic component within a broader subsistence system in which coastal fishing, local horticulture, and mixed resource use continued to structure everyday food practices. In this sense, the isotopic evidence complements the wider archaeological and historical picture, which suggests that, despite colonisation, market integration, and immigration, communities in Babitonga Bay maintained food practices closely tied to local ecological affordances and to long-standing coastal traditions (Silvano and Begossi, 2012; Colonese et al., 2014; Begossi, 2006b; Gabriel and Silva, 2019).

The Porto Alegre sample, by contrast, is better interpreted as reflecting a more heterogeneous dietary setting. The broader isotopic spread suggests variable combinations of animal protein, C_4-derived foods, and possibly, in some individuals, aquatic inputs. For this reason, the data do not support a rigid contrast in which Babitonga Bay is treated as marine and Porto Alegre as wholly non-marine. Instead, the Porto Alegre values point to a less uniform dietary range, likely shaped by urban provisioning systems, social differences, and uneven access to food resources. This reading is also more consistent with the broader archaeological and historical evidence discussed in Chapter 6, where the Pelotas-Porto Alegre sphere emerges as a socially differentiated setting shaped by cattle economies, market supply, and urbanisation, but not necessarily detached from more varied food practices. In this context, access to charque, fresh beef, dairy, maize-based foods, and other market-supplied products was likely unevenly distributed, reflecting broader patterns of class and labour organisation in nineteenth-century Rio Grande do Sul (Bastos et al., 2016; Bastos, 2014; Vargas, 2017).

Taken together, these patterns suggest that the two regions should not be understood as representing mutually exclusive dietary regimes, but rather different emphases within a wider spectrum of historical Southern Brazilian foodways. In Babitonga Bay, the isotope data point more consistently towards protein intake strongly linked to coastal and estuarine ecologies. In Porto Alegre, the broader spread of values suggests a less uniform pattern shaped by urban supply, social variability, and mixed access to different food sources. It is from this contrast that the broader contextualisation of the Chapter can be developed.

The broader isotopic range observed in the Porto Alegre sample is consistent with greater dietary heterogeneity within an urban context. The enriched $\delta^{13}C$ values suggest the contribution of C_4-derived foods or C_4-fed livestock, while the high $\delta^{15}N$ values observed in some individuals indicate access to higher-trophic-level protein, potentially including aquatic or estuarine resources. This pattern should not be interpreted as evidence for a wholly terrestrial diet, but as a mixed and socially variable

dietary signal. The faunal data from Casa 8 and the Porto Alegre assemblage support this interpretation, since cattle show C_4-influenced diets and brackish-marine fish provide a possible aquatic reference point for the region. Within the historical economy of Rio Grande do Sul, access to beef, charque, dairy, pork and other market-supplied products was likely shaped by the cattle economy, urban provisioning, and social differentiation (Bastos et al., 2014; Vargas, 2017).

The variation within the Porto Alegre dataset, particularly the spread in $\delta^{15}N$ values, suggests that access to protein was not uniform across the sampled population. Individuals with lower $\delta^{15}N$ values may have consumed lower proportions of high-trophic-level animal protein, although these values cannot be translated directly into food insecurity without additional osteological or documentary evidence. They are better interpreted as evidence for variability in protein access, potentially shaped by status, occupation, household provisioning, or other social factors within the urban population. This interpretation is consistent with historical and bioarchaeological work documenting marked social inequalities in late 19th century Brazilian urban contexts, particularly among enslaved, freed, and lower-status Afro-Brazilian populations, whose access to meat and higher-value foods was often constrained by labour relations, poverty, and unequal provisioning systems (Bastos, 2014; Ribeiro, 2017).

These results provide a useful comparative layer to the lipid residue data from Casa 2 discussed in Chapter 6. The Casa 2 ceramics show a predominance of C_3 plant and terrestrial animal products, while the Porto Alegre human isotope values suggest variable access to animal protein and C_4-derived foods within the wider urban region. Although caution is warranted because the datasets derive from different sites and material categories, the combined evidence is consistent with heterogeneous provisioning and socially differentiated access to food resources in nineteenth-century Rio Grande do Sul.

The comparison between Porto Alegre and São Francisco do Sul also points to the cultural and ecological dimensions of dietary practice. While Pelotas and Porto

Alegre reflect stronger engagement with market provisioning, urban supply, and the cattle economy, São Francisco do Sul appears more closely tied to coastal and estuarine resource use. This does not imply direct continuity from pre-colonial foodways, but it does suggest that long-standing coastal subsistence practices remained relevant within historical communities in Santa Catarina (Toso et al., 2021; Silvano and Begossi, 2012).

The isotopic patterns discussed above become clearer when the results from São Francisco do Sul and Porto Alegre are situated within the broader Brazilian isotopic record, not considered only at the scale of the two study areas. As shown in Figure 51, the human isotope values generated in this study were compared with published Brazilian human collagen isotope data compiled by Borges et al. (2022). This comparison is not intended to produce direct one-to-one analogies between the present samples and all other Brazilian groups, but to evaluate how the São Francisco do Sul and Porto Alegre individuals fall within the wider isotopic variability already documented for Brazil.

The compilation by Borges et al. (2022) provides a useful national-scale framework because it brings together isotopic and chronological data from archaeological sites across Brazil, including human and faunal samples from different ecological, chronological and cultural contexts. Within this broader framework, the São Francisco do Sul individuals occupy a position consistent with coastal and aquatic-influenced dietary patterns. Their consistently high $\delta^{15}N$ values, when compared with the local faunal baseline, support the interpretation of a strong contribution of marine or estuarine protein to the diet, while still allowing for mixed food practices that are not fully visible through collagen isotopes alone.

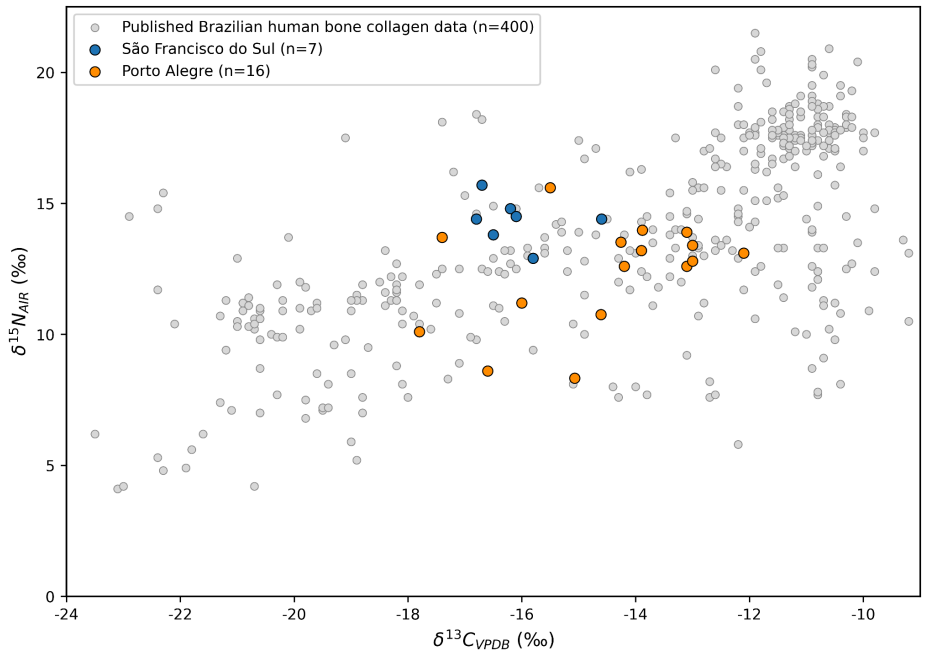


Figure 51: *Human collagen δ¹³C and δ¹⁵N values from São Francisco do Sul and Porto Alegre plotted against published Brazilian human bone collagen isotope records compiled in the IsoArcH dataset associated with Borges et al. (2022). Grey points represent published Brazilian human bone collagen records with paired δ¹³C and δ¹⁵N values, excluding one record with a negative δ¹⁵N value, while the highlighted points show the individuals analysed in this study. The Figure situates the two historical Southern Brazilian samples within the broader isotopic variability documented for archaeological human remains in Brazil.*

This interpretation is also relevant when considered alongside previous isotopic work from coastal Santa Catarina, including Forte Marechal Luz. Although Bastos et al. (2011) focused primarily on strontium isotope evidence for mobility and not directly on collagen-based palaeodietary reconstruction, the study is useful here because it highlights the importance of mobility, local/non-local identities and coastal environmental variability when interpreting skeletal assemblages from Santa Catarina. The São Francisco do Sul results should be understood not simply as an isolated historical sample, but as part of a broader coastal setting in which marine baselines, local ecology and individual life histories may all shape isotopic values.

The Porto Alegre sample occupies a different position within this broader Brazilian isotopic range. Its wider spread in both $\delta^{13}C$ and $\delta^{15}N$ values suggests greater dietary heterogeneity than observed in the São Francisco do Sul sample. This variability is important when considered alongside historical Brazilian isotope studies such as Bastos et al. (2016), which examined enslaved Africans buried in Rio de Janeiro and Salvador and showed that isotopic data can reveal marked differences in dietary access, origin and life history within historically unequal populations. The Porto Alegre individuals analysed here do not represent the same social or historical context as those cemeteries, and the comparison is not used as a direct analogy. Rather, it shows that historical-period Brazilian populations can display substantial isotopic variability linked to mobility, social inequality, provisioning systems and differential access to animal protein.

Read against this broader Brazilian and Southern Brazilian background, the contrast between São Francisco do Sul and Porto Alegre becomes more meaningful. The São Francisco do Sul sample fits within a coastal pattern in which aquatic or estuarine protein made an important contribution to diet, whereas the Porto Alegre sample reflects a broader and more heterogeneous urban dietary signal. Previous isotopic studies from Southern Brazil have shown that human diets varied substantially between coastal, lagoonal, inland Guarani and highland Taquara-Itararé contexts, reflecting different balances between aquatic protein, C_3-based resources and maize consumption (Loponte et al., 2021; Toso et al., 2021; Chanca et al., 2021). The results presented here do not simply add two further case studies to the Brazilian isotopic record, but help contextualise historical-period dietary variability in Southern Brazil, showing how local ecology, urban provisioning and social differentiation shaped access to food resources.

The comparison with the Borges et al. (2022) dataset also highlights the contribution and limitations of the present study. The São Francisco do Sul and Porto Alegre samples add new historical-period data from Southern Brazil, a region and time frame still underrepresented in biomolecular studies when compared with pre-colonial

contexts. At the same time, the small sample sizes, especially for São Francisco do Sul, and the absence of detailed individual-level osteological and life-history information limit the extent to which dietary differences can be attributed to status, ethnicity, occupation or household identity. For this reason, the broader Brazilian comparison is used here to contextualise isotopic variability and strengthen the interpretation of regional dietary patterns.

8. Conclusions

This final Chapter synthesises the results of this thesis in relation to the three research questions set out in Chapter 1. It brings together the organic residue data from the ceramic assemblages, the stable isotope results from human and faunal remains, and the archaeological and historical contexts discussed in Chapters 5-7. The aim is to evaluate what can be concluded from the biomolecular evidence produced in this thesis, while distinguishing these conclusions from broader historical interpretations based on comparative literature and contextual evidence.

This distinction is necessary because the methods used in this thesis do not measure the same dimensions of past foodways. Organic residue analysis provides evidence for preserved residues and vessel use in the analysed ceramic assemblages, while bulk stable isotope analysis provides information on longer-term dietary protein intake in humans and animals. These proxies are complementary, but neither provides a complete reconstruction of diet, cuisine, identity or social practice on its own (Evershed, 2008; Roffet-Salque et al., 2017a; Craig, 2021). For this reason, the conclusions presented here distinguish between analytical results, interpretations supported by the convergence of multiple lines of evidence, and broader social or historical inferences.

The same caution is especially relevant when discussing ethnicity, status and cultural identity. Comparative studies of colonial and post-colonial foodways have shown that food practices and material culture can contribute to identity formation, but rarely operate as direct or fixed ethnic markers. They are shaped through daily practice, access to resources, household labour, colonial constraint, market participation and social negotiation (Mintz and Price, 1992; Ferguson, 1992; Lightfoot, Martinez and Schiff, 1998; Silliman, 2001, 2005). In this Chapter, this literature is used as an interpretative caution and not as a direct analogy for Southern Brazil. It helps frame why ethnicity, status and social position are treated as contextual dimensions that must be interpreted through the convergence of biomolecular data, archaeological

evidence and historical sources, not as categories directly measurable through lipid residues or bulk collagen isotopes.

The Chapter first considers how food habits changed over time across Babitonga Bay and the Patos Lagoon region. It then evaluates the extent to which marine and terrestrial resources were used and valued across different regional and social contexts, before discussing how local economies and provisioning systems may have shaped culinary practice. The final sections reflect on the methodological contribution of the thesis and on the limitations imposed by preservation, sample size, analytical resolution, contamination, low-yield extracts and negative results. These limitations are not treated as peripheral problems, but as central to defining the strength and scope of the conclusions drawn from the dataset.

8.1. How have food habits evolved over time?

Addressing how food habits evolved over time requires a comparative perspective that links the historical data produced in this thesis with published pre-colonial, colonial and historical evidence from Southern Brazil. Although this study focuses on the nineteenth and early twentieth centuries, historical foodways need to be understood as part of longer-term processes shaped by regional ecologies, colonial disruption, migration, economic change and shifting social relations. The central issue is not whether historical foodways represent either continuity or rupture, but how older resource-use practices, new provisioning systems and changing ceramic habits became combined, transformed or differentially preserved across the study regions.

This comparison is necessarily proxy-dependent. The historical ORA results discussed in Chapters 5 and 6 reflect preserved residues and vessel use in the analysed ceramic assemblages, while the SIA results presented in Chapter 7 reflect longer-term dietary protein intake in humans and animals. These datasets can be compared with earlier zooarchaeological, isotopic and organic residue evidence to identify broad patterns of continuity and change, but they cannot demonstrate direct cultural

transmission, cultural replacement or intentional resistance on their own. The focus is on recurring resource-use patterns, changes in vessel use and differences in dietary protein sources, not on fixed ethnic or cultural categories.

Published pre-colonial evidence from the two study regions provides an important comparative baseline. In Babitonga Bay, stable isotope, zooarchaeological and organic residue studies indicate a strong and long-term reliance on aquatic and estuarine resources, especially fish and shellfish, alongside plant use and later ceramic-associated food processing practices (da Rocha Bandeira, 2000; Colonese et al., 2014; Toso et al., 2021; Bastos et al., 2022; Admiraal et al., 2023). In the Patos Lagoon region, zooarchaeological and organic residue evidence from late Holocene contexts similarly indicates the importance of lagoonal and estuarine resources, combined with plant foods and wild terrestrial fauna (Fossile et al., 2023; Admiraal et al., 2025; see Chapters 2 and 6). These earlier datasets attest that aquatic resources and plant processing formed part of long-term coastal and lagoonal food systems in Southern Brazil, but they do not provide a direct template for interpreting the historical assemblages. They instead establish a regional baseline against which the historical patterns from Babitonga Bay, Casa 2, São Francisco do Sul and Porto Alegre can be evaluated. The key question is not whether historical communities simply continued pre-colonial foodways, but how older resource-use traditions, local ecologies, ceramic practices, migration, urban provisioning and new market conditions interacted during the nineteenth and early twentieth centuries.

In Santa Catarina, the rural assemblages from Praia Grande - Unidade 21 (PG-U21), Morro Grande 1 (MG1), and Foz do Cubatão (FC) provide evidence for the continued relevance of coastal and estuarine resources in some nineteenth- and early twentieth-century food practices, although this should not be interpreted as direct or unbroken continuity from pre-colonial foodways (see Chapter 5). The organic residue results indicate that some analysed vessels preserved aquatic-compatible signals, plant-associated biomarkers and mixed residue profiles, suggesting that estuarine resources and plant foods formed part of the range of substances processed in these

ceramic assemblages (Chapter 5; Figures 36-39; Appendix 1). These results are broadly comparable with earlier evidence from the same region, but the comparison is strongest when framed as continuity in resource use and ecological orientation, instead of continuity of identity, ethnicity or specific dishes.

The stable isotope data from São Francisco do Sul provide a complementary but different line of evidence. As discussed in Chapter 7 (Figures 49-51; Appendix 6), the human collagen values are consistent with substantial aquatic or high-trophic-level protein intake, alongside variable contributions from C_3 and C_4 resources. This strengthens the interpretation that aquatic foods remained important in at least some coastal historical diets, but it does not indicate which foods were prepared in the analysed vessels, nor whether the same resources carried the same social meanings as in earlier periods. When read alongside archaeological and historical evidence for fishing, manioc cultivation, small-scale farming and mixed rural communities in Babitonga Bay, the ORA and SIA results suggest that local coastal practices remained relevant during a period of immigration, market expansion and urbanisation (Borba, 2014; Fossile et al., 2023; Herbst et al., 2023).

Arroio Tamarina II (AT2), historically associated with European immigrant occupation, particularly German settlers, reveals a different preserved residue pattern. The analysed vessels from AT2 produced fewer aquatic-compatible signals and a stronger visibility of terrestrial animal fats and plant-related residues (Chapter 5; Figures 36-39; Appendix 1). This contrast is important because it suggests that historical change in Babitonga Bay did not involve a single regional trajectory. Instead, different rural communities appear to have combined local resources, agricultural practices, animal management and ceramic use in different ways. Historical sources describe the introduction of new agricultural practices, livestock management strategies and food traditions by European immigrant communities in Santa Catarina, including German settlers (Borba, 2014). The organic residue evidence from AT2 is therefore better read as a different preserved vessel-use pattern within an immigrant-associated rural context, rather than as evidence for a “German” diet or a

fixed ethnic cuisine. It is more cautious to interpret AT2 as showing a different preserved vessel-use pattern within an immigrant-associated rural context, in which terrestrial animal products and plant foods are more visible in the analysed ceramic residues than aquatic resources.

Overall, the Santa Catarina evidence points to variability instead of a single model of coastal persistence. PG-U21, MG1 and FC show greater visibility of aquatic-compatible and plant-associated signals, while AT2 shows stronger visibility of terrestrial animal products and plant foods in the analysed ceramic residues (Chapter 5; Figures 36-39; Appendix 1). These differences are likely to reflect overlapping factors, including local ecology, household provisioning, ceramic use, post-depositional preservation, access to animals and crops, and participation in wider economic networks. Ethnic background and immigrant history provide important context for interpreting these patterns, but they should not be treated as direct explanations on their own. The Babitonga Bay results suggest that historical food habits were shaped by the selective persistence, transformation and reorganisation of earlier coastal practices under new social and economic conditions (Borba, 2014; Fossile et al., 2023; Herbst et al., 2023; see Chapter 5).

The faunal isotope dataset presented in Chapter 7 adds a broader ecological and provisioning perspective to this interpretation. Animal remains from Santa Catarina yielded variable $\delta^{13}C$ values, suggesting that livestock and other animals were not managed or fed in a uniform way. The presence of both C_3 and C_4 signals may reflect differences in foddering, grazing environments or access to maize- or sugarcane-derived resources, although no direct biomolecular evidence links these animal values to specific foodstuffs processed in the ceramic vessels. This matters because it shows that historical food change was not limited to what people cooked in pots, but also involved the management, circulation and feeding of animals within changing rural economies. Taken together, the ORA and SIA data support a model in which rural food habits in Santa Catarina were diverse and context-dependent,

combining local coastal resources, plant processing, animal management and some novel inputs associated with agricultural change and market integration.

The Patos Lagoon region provides a second, but not directly equivalent, diachronic comparison. As discussed in Chapter 6, late Holocene and pre-colonial evidence from this region indicates the importance of lagoonal and estuarine resources, wild terrestrial fauna and plant foods within local food systems (Fossile et al., 2023; Admiraal et al., 2025). Organic residue data from pre-colonial pottery show fatty acid $\delta^{13}C$ values that differ in part from the historical Casa 2 assemblage, suggesting that the range of substances processed in pottery changed between the pre-colonial and historical periods (see Chapter 6, Figure 43). This comparison is best treated as a comparison of preserved ceramic residue patterns, not as a direct comparison of whole diets or social identities. Differences may reflect changes in resource use, vessel function, ceramic technology, cooking practice, preservation and the broader historical context of urbanisation and market provisioning.

The historical Casa 2 assemblage from Pelotas presents a different preserved residue pattern from the Babitonga Bay assemblages. As discussed in Chapter 6, the interpretable ceramic extracts from Casa 2 are mainly compatible with C_3 plant inputs and terrestrial animal fats, while aquatic-compatible biomarkers are less visible in the analysed vessels (Chapter 6; Figures 42-44; Appendix 4). This pattern suggests that, within the sampled vessels, terrestrial and plant-derived resources were more visible than aquatic products. This is an important result, but it should be interpreted as a ceramic-use and preservation signal, not as a complete reconstruction of household diet.

The Casa 2 results are consistent with the broader historical context of Pelotas, where cattle ranching, charque production and urban provisioning were central to the regional economy during the nineteenth and early twentieth centuries (da Silva Peixoto and Cerqueira, 2006; Bastos, 2014; Ribeiro, 2017; Palma et al., 2021). Nevertheless, the organic residue data do not provide direct evidence for charque itself. Rather, the lipid

and compound-specific isotope results are compatible with the processing of C_3-associated plant foods and terrestrial animal products in some of the analysed vessels. The presence of lipid oxidation and degradation products, including dicarboxylic acids, also indicates altered or degraded residues, but these compounds should not be treated as source-specific evidence for particular dishes or cooking techniques.

The difference between Casa 2 and the Babitonga Bay assemblages points to regional variability in preserved pottery-use patterns. This variability may reflect differences in local economies, household provisioning, ceramic function, cooking technologies, resource availability and preservation conditions. The comparison is also affected by the smaller size of the Casa 2 assemblage relative to the combined Babitonga Bay dataset. In Pelotas, the greater visibility of terrestrial animal fats in the ceramic residues can be discussed in relation to the cattle-based economy and the historical availability of beef and charque, but the archaeological evidence does not support a direct claim that aquatic foods were absent from household consumption or that urban diet can be reconstructed from ceramic residues alone.

The human collagen data discussed in Chapter 7 add a complementary perspective to the ceramic residue evidence, although they reflect longer-term dietary protein intake and not specific cooking practices. This distinction is important because the ceramic residues and the collagen isotope values do not necessarily record the same meals, vessels or social behaviours. The Porto Alegre assemblage shows isotopic variability, including enriched $\delta^{13}C$ values in some individuals, which may indicate contributions from C_4 plants, C_4-fed livestock or mixed provisioning systems. At the same time, some individuals also show high $\delta^{15}N$ values, which should not be interpreted as evidence for a wholly terrestrial diet (Figures 50-51; Appendix 6). These values may reflect access to higher-trophic-level protein, including possible aquatic or estuarine resources, alongside terrestrial animal products and urban market foods.

When situated within the broader Brazilian isotopic record discussed in Chapter 7, including the comparative datasets compiled by Borges, Chanca and Salesse (2022), these results suggest that the historical individuals analysed here fall within a wider range of dietary variability already documented across Brazilian archaeological contexts (Bastos et al., 2011, 2016; Borges, Chanca and Salesse, 2022). This broader comparison is useful because it prevents the Porto Alegre and São Francisco do Sul values from being reduced to a simple marine/terrestrial opposition. Instead, the SIA data point to heterogeneous dietary protein sources, shaped by regional ecology, urban provisioning, animal management, access to crops, and possibly social differences in food availability.

These patterns can be discussed in relation to the historical context of inequality, slavery, post-abolition labour, land tenure and market integration in Southern Brazil (Bastos, 2014; Ribeiro, 2017; Palma et al., 2021). However, the isotope data alone cannot assign individuals to specific social groups or demonstrate elite, poor, Afro-Brazilian, Indigenous or immigrant diets directly. This limitation is particularly important because contextual information on several burials is incomplete: some human remains derive from secondary, disturbed or disarticulated deposits, and in several cases they cannot be securely associated with specific individuals, burial events, grave goods, sex, age or social position. The SIA results provide evidence for dietary variability that can be interpreted alongside archaeological context, documentary sources and the broader social history of the region, but they should not be used to reconstruct fixed social or ethnic dietary categories or to make unsupported individual-level interpretations.

The evidence discussed in this section indicates that food habits in Southern Brazil changed unevenly across regions, sites and forms of evidence. In Babitonga Bay, the ORA and SIA results are compatible with the continued importance of aquatic resources and plant processing in some historical coastal contexts, although they do not demonstrate direct cultural continuity with pre-colonial practices. In Pelotas, the Casa 2 assemblage shows a different preserved residue pattern, in which C_3 plant

inputs and terrestrial animal fats are more visible than aquatic-compatible biomarkers. This can be interpreted in relation to the cattle-based economy, urban provisioning and the historical importance of charque production, but it remains a contextual inference (Chapter 6; Figures 42-44; Appendix 4).

The overall pattern is therefore not one of simple cultural replacement, but neither should it be presented as direct proof of unbroken continuity or intentional resistance. A more cautious interpretation is that older coastal resource-use practices, plant-based food traditions, ceramic habits, immigrant-associated agricultural practices and urban market provision overlapped in different ways across the study regions. Historical sources on Southern Brazilian foodways and rural economies support the importance of manioc, beans, fish, pork, cattle products and small-scale horticulture in different settings, but the biomolecular evidence presented here captures only those elements that were preserved in ceramic residues or reflected in collagen isotope values (Borba, 2014; Ribeiro, 2017; Fossile et al., 2023; Herbst et al., 2023).

In this sense, the first research question can be answered by emphasising variability instead of a single trajectory of change. Food habits did not evolve uniformly from Indigenous or coastal traditions toward European or urban foodways. Instead, the evidence points to regionally specific combinations of continuity, adaptation and change, shaped by local ecologies, household provisioning, ceramic use, migration, market integration and preservation conditions. The strongest conclusion is not that one culinary system replaced another, but that historical foodways in Southern Brazil were reorganised through the interaction of older resource-use practices, new economic structures and changing domestic technologies.

8.2. To what extent were marine and terrestrial resources used and valued by historical groups with distinct ethnic backgrounds and social status?

The second research question concerns not only whether marine and terrestrial resources were present in the biomolecular record, but also how far their use and social significance can be interpreted in relation to ethnicity, status and historical context. The evidence presented in this thesis can address resource use more directly than resource valuation. Organic residue analysis can identify preserved vessel-use signals compatible with aquatic, plant or terrestrial animal products, while stable isotope analysis can indicate longer-term dietary protein sources. The social value attached to particular foods is more difficult to assess, because it does not leave a direct biomolecular signature. It must be inferred more cautiously, through the combination of biomolecular data, archaeological context, historical sources and comparative literature. This distinction is important because the same resource may have held different practical, economic or symbolic meanings depending on context, access, labour organisation and household practice. This is consistent with broader approaches to colonial and post-colonial foodways, which emphasise that food practices and material culture can contribute to the negotiation of identity, social difference and inequality, but only when interpreted in relation to daily practice, access, labour, household organisation and colonial power relations (Ferguson, 1992; Mintz and Price, 1992; Lightfoot, Martinez and Schiff, 1998; Silliman, 2001, 2005; Deagan, 2004).

Comparative studies of colonial and post-colonial foodways in North America and the Caribbean are useful here because they show that food practices and material culture rarely correspond directly to fixed ethnic identities. Work on creolisation and African-American cultural formation has emphasised how culinary practices could emerge from the interaction of inherited knowledge, displacement, coercion and unequal access to resources, and not from the simple preservation of bounded cultural

traditions (Ferguson, 1992; Mintz and Price, 1992; Singleton, 1995). Similarly, studies of plantation, urban and multi-ethnic colonial households have shown that diet and material culture were shaped by household provisioning, labour organisation, gendered practice, market participation and status negotiation, as well as by ancestry or cultural affiliation (Lightfoot, Martinez and Schiff, 1998; Armstrong and Kelly, 2000; Wilkie, 2000; Silliman, 2001, 2005; Deagan, 2004; Voss, 2008; Galle, 2010).

This literature provides a cautionary framework for the present thesis because the Babitonga Bay and Patos Lagoon assemblages were also formed within historically unequal settings marked by Indigenous dispossession, African slavery and post-abolition labour, European immigration, rural smallholding, urbanisation and market expansion. The comparison is not intended to equate Southern Brazil directly with North American or Caribbean case studies. Rather, it supports a broader interpretative principle: foodways are produced through everyday practice, access, labour, household organisation and political economy, as well as through cultural affiliation. For this reason, aquatic-compatible residues, plant-processing markers, terrestrial animal fats and collagen isotope values are interpreted here as evidence for resource use and dietary variability shaped by ecology, access, ceramic use, household provisioning, market integration, social position and preservation, rather than as direct indicators of Indigenous, Afro-Brazilian, Luso-Brazilian or immigrant identity.

In the Babitonga Bay region of Santa Catarina, the evidence for marine and terrestrial resource use is strongest when the ORA results are read together with zooarchaeological, isotopic and historical information. Aquatic-compatible APAA-C20/C18 values, plant-processing proxy evidence, faunal remains and historical accounts of fishing, manioc cultivation and small-scale provisioning all point to the practical importance of estuarine and coastal resources in some historical communities (Cascudo, 1967; Silva, 2001a; Borba, 2014; Fossile et al., 2023; Herbst et al., 2023; Chapter 5; Appendix 1). However, this practical importance should not be translated directly into a fixed ethnic or cultural interpretation. The value of aquatic and plant resources in these communities was likely produced through everyday practice: access

to estuarine environments, household labour, fishing knowledge, plant processing, ceramic use, and the organisation of small-scale provisioning.

This means that the Babitonga Bay evidence can support a discussion of local coastal foodways, but not a direct claim that specific residues represent Indigenous, Afro-Brazilian or Luso-Brazilian identity. The persistence of aquatic-compatible and plant-associated signals is better understood as a historically situated pattern of resource use, shaped by local ecology and household practice under changing conditions of migration, market expansion and social inequality. In this sense, the data are compatible with the continued relevance of local and non-hegemonic food knowledge, but they do not demonstrate the intentional preservation of bounded cultural traditions or a direct correspondence between aquatic residues and ethnic identity.

At PG-U21, where some of the analysed vessels were wheel-thrown, aquatic-compatible residues indicate that local or estuarine resources could still be processed in vessels that were not necessarily handmade or technologically conservative (Chapter 5; Appendix 1). This is important because it shows that ceramic technology and food practice did not always change together. Wheel-thrown vessels may reflect changing production or supply systems, while the residues preserved within them suggest that locally available aquatic resources continued to form part of vessel use. The evidence is better interpreted as a combination of technological change and culinary continuity at the level of resource processing, but not as either cultural replacement or unchanged tradition.

The São Francisco do Sul human isotope values provide a complementary perspective on this pattern. As discussed in Chapter 7, these values suggest that aquatic or high-trophic-level protein contributed to some coastal diets. They can be compared with the Porto Alegre assemblage, but the contrast should not be framed as a simple marine versus terrestrial opposition. Some individuals from Porto Alegre show high $\delta^{15}N$ values, which may indicate access to higher-trophic-level protein, including

possible aquatic or estuarine resources, alongside terrestrial animal products and urban market foods (Chapter 7; Figures 49-51; Appendix 6). The difference between São Francisco do Sul and Porto Alegre is better understood as variation in the range, combination and visibility of dietary protein sources.

The Rio Grande do Sul evidence points to a different configuration of resource use and archaeological visibility. At Casa 2, the interpretable ceramic residues are mainly compatible with C_3 plant inputs and terrestrial animal fats, while aquatic-compatible biomarkers are less frequent in the analysed vessels (Chapter 6; Figures 42-44; Appendix 4). This pattern can be interpreted in relation to the historical importance of cattle ranching, charque production and urban provisioning in Pelotas, where terrestrial animal products were economically significant and historically prominent (da Silva Peixoto and Cerqueira, 2006; Bastos, 2014; Ribeiro, 2017; Palma et al., 2021). This does not mean that the residues directly identify charque, beef consumption or elite diet. They show which lipid signals were preserved in a smaller urban ceramic assemblage, not the full range of foods consumed, prepared or valued in the household.

Thus, the question of value is more difficult than the question of use. The stronger visibility of terrestrial animal fats in Casa 2 may reflect several overlapping processes: the availability of cattle products within the regional economy, household access to market goods, the use of particular vessels for preparing animal fats or C_3 plant foods, and preservation or recovery conditions that favoured some lipid signals over others. Comparative studies of plantation, urban and post-emancipation households have shown that differences in food remains and material culture often reflect provisioning systems, household strategies, labour relations, market participation and status negotiation, but not ethnicity or class alone (Wilkie, 2000; Voss, 2008; Galle, 2010). In this sense, Casa 2 can be discussed as part of an urban provisioning system, but not as a direct biomolecular marker of elite or Luso-Brazilian identity.

The Porto Alegre human isotope data add another layer to this interpretation. As discussed in Chapter 7, enriched $\delta^{13}C$ values in some individuals may indicate contributions from C_4 plants, C_4-fed animals or mixed market provisioning, while high $\delta^{15}N$ values in some individuals may reflect higher-trophic-level protein, including possible aquatic or estuarine resources. The Porto Alegre assemblage should be understood as a heterogeneous urban dietary context, and not treated as evidence for a wholly terrestrial diet or a simple replacement of aquatic foods by beef. This is important because it prevents the Rio Grande do Sul evidence from being reduced to a single cattle-based dietary model. Cattle products and charque were historically central to the regional economy, but the biomolecular evidence points instead to mixed and variable food access across ceramic and human isotope records.

Historical literature on Southern Brazil shows that cattle products, charque, wheat, maize, beans, manioc and fish all circulated within changing systems of provisioning, labour and social distinction during the nineteenth and early twentieth centuries (Cascudo, 1967; Silva, 2001a; Bastos, 2014; Ribeiro, 2017; Gabriel and Silva, 2019; Palma et al., 2021). Within this context, the greater visibility of terrestrial animal fats in the Casa 2 ceramic residues can be discussed as part of a cattle-based regional economy and an urban provisioning system. However, claims about the social devaluation of fish, cultural distancing from aquatic foods, or the association of beef with whiteness and elite identity cannot be demonstrated directly from the biomolecular evidence presented here. These remain important historical questions, but they require closer integration of documentary sources, faunal assemblages, household artefacts, vessel function, ceramic form and comparative urban datasets.

The contrast between Santa Catarina and Rio Grande do Sul indicates that marine and terrestrial resources were used in different ways across the analysed sites, but the evidence for how they were valued is more indirect. In Babitonga Bay, marine and estuarine resources are visible across multiple lines of evidence, including ceramic residues, faunal remains, historical accounts and human isotope values. This supports the interpretation that aquatic resources remained practically important in some

coastal historical communities. In Pelotas, by contrast, the Casa 2 ceramic residues show greater visibility of C_3 plant inputs and terrestrial animal fats, while the Porto Alegre isotope data point to heterogeneous protein sources rather than to a simple absence of aquatic input.

The second research question can be answered by distinguishing between resource use, resource visibility and resource value. Marine and terrestrial resources were both used, but their visibility varied according to region, site, proxy and archaeological context. In Santa Catarina, aquatic-compatible and plant-associated evidence suggests continued engagement with coastal and estuarine resources in some rural communities. In Rio Grande do Sul, terrestrial animal fats and C_3 plant inputs are more visible in the Casa 2 ceramic assemblage, while the Porto Alegre human isotope data indicate mixed urban protein access. These differences are best explained through a combination of environmental setting, household provisioning, ceramic use, market access, animal management, labour organisation, preservation and broader historical conditions.

Ethnic background and social status remain important historical dimensions for interpreting these assemblages, but they cannot be directly measured through ORA or bulk SIA. Their role is best discussed through the convergence of biomolecular evidence, historical documents, archaeology, faunal data and comparative studies of colonial and post-colonial foodways. In this sense, the thesis supports a model of socially and regionally variable foodways, in which marine and terrestrial resources were meaningful not because they map neatly onto fixed ethnic or class categories, but because they were embedded in everyday practices of access, labour, provisioning, cooking and preservation.

8.3. How did local economies influence dietary habits and culinary traditions among rural communities and emerging urban elites?

The third research question concerns the relationship between local economies, food access and culinary practice. The evidence presented in Chapters 5-7 suggests that local economies influenced the range of resources available to households, the ways in which foods were provisioned and prepared, and the visibility of different resources in the archaeological record. However, local economies did not produce uniform diets or fixed culinary identities; rather, they interacted with ecology, household labour, ceramic use, market access, preservation and social position.

In Pelotas, the Casa 2 ceramic assemblage provides evidence for a preserved ceramic-use pattern in which C_3 plant inputs and terrestrial animal fats are more visible than aquatic-compatible residues (Chapter 6; Figures 42-44; Appendix 4). This interpretation is consistent with comparative studies of colonial and post-emancipation households, where food remains and material culture have been shown to reflect provisioning systems, labour relations, household strategies and market access, rather than social status or ethnicity alone (Armstrong and Kelly, 2000; Wilkie, 2000; Galle, 2010). However, the Casa 2 evidence should not be treated as a direct reconstruction of the household diet or as proof that aquatic resources were absent from consumption.

The economic context of Pelotas is nevertheless important for interpreting why terrestrial animal products are more visible in the analysed ceramic residues. The charque industry structured labour, wealth accumulation, urban growth and access to cattle products in the region, while market expansion and immigration contributed to changes in food availability, cooking technologies and domestic consumption (Bastos, 2014; Ribeiro, 2017; Palma et al., 2021). Within this context, the Casa 2 residue pattern is compatible with greater engagement with cattle-based provisioning and urban food networks. At the same time, the available data cannot determine whether these

residues reflect social preference, economic access, vessel function, preservation bias or the use of other cooking technologies for foods not visible in the ceramic record.

Historical studies of domestic labour and post-slave society in Brazil show that food preparation was closely connected to gendered labour, slavery, freed labour and household organisation (Ribeiro, 2017; Gabriel and Silva, 2019; Palma et al., 2021). These themes are relevant for interpreting urban foodways in Pelotas. The Casa 2 assemblage contributes to understanding urban provisioning and ceramic use, while broader questions of labour, class and culinary transmission require integration with documentary sources, household archaeology and comparative material evidence.

In the rural Babitonga Bay sites, the local economy was more closely connected to coastal and estuarine resources, small-scale cultivation and household-level provisioning. Zooarchaeological and historical evidence indicates the importance of fishing, shellfish gathering, manioc cultivation, small livestock and wild terrestrial resources in the region, although the relative importance of each resource varied by site and context (Saint-Hilaire, 1936; Hanazaki and Begossi, 2003; Borba, 2014; Fossile et al., 2023; Herbst et al., 2023). The ORA evidence from PG-U21, MG1 and FC is compatible with this broader pattern, since some analysed vessels preserved aquatic-compatible signals, plant-associated biomarkers and mixed residue profiles (Chapter 5; Figures 36-39; Appendix 1).

These data suggest that rural food practices in Babitonga Bay were shaped by locally available resources and small-scale provisioning systems, but they should not be reduced to subsistence alone. Historical and ethnographic studies of coastal communities in Southern Brazil show that fishing, horticulture and household food preparation were embedded in social relationships, local ecological knowledge and community-based resource use (Hanazaki and Begossi, 2003; Begossi, 2006b; Fossile et al., 2023; Herbst et al., 2023). The biomolecular evidence from ceramics is better understood as indicating broad categories of processed resources, rather than specific dishes. Such examples are useful as historically documented culinary possibilities, but

the residue data more cautiously indicate the processing of aquatic-compatible, plant-associated and mixed resources in some vessels.

The contrast with Casa 2 is economic and technological as much as dietary. In Babitonga Bay, ceramic residues, faunal remains and historical evidence point to food systems more closely linked to local coastal ecologies and household provisioning. In Pelotas, the Casa 2 residues are more visible for C_3 plant inputs and terrestrial animal fats, within a region structured by cattle production, charque and urban markets. These differences show how local economies influenced what foods were available, how they were prepared, and which resources became visible in the archaeological record. They do not, however, allow a simple opposition between “traditional” rural foodways and “modern” urban diets.

The human isotope data from Porto Alegre and Pelotas add further complexity to this picture. As discussed in Chapter 7, some individuals show enriched $\delta^{13}C$ values, compatible with contributions from C_4 plants, C_4-fed animals or mixed market foods, while high $\delta^{15}N$ values in some Porto Alegre individuals may indicate higher-trophic-level protein, including possible aquatic or estuarine inputs (Chapter 7; Figures 49-51; Appendix 6). These patterns suggest heterogeneous access to protein sources within urban or peri-urban contexts, rather than a single elite, terrestrial or market-based dietary model.

Historical literature indicates that urban foodways in nineteenth- and early twentieth-century Brazil were shaped by social inequality, domestic labour, slavery and post-abolition conditions, immigration, market access and changing ideals of household consumption (Bastos, 2014; Ribeiro, 2017; Gabriel and Silva, 2019; Palma et al., 2021). These factors make it possible to discuss food as part of social differentiation, but the present biomolecular dataset cannot demonstrate deliberate avoidance of fish, the social devaluation of aquatic foods, or the use of meat as a direct marker of whiteness or elite identity. Such interpretations remain important historical

questions, but they require closer integration of documentary records, faunal assemblages, household artefacts, vessel function and comparative urban datasets.

Overall, the comparative evidence from rural and urban contexts indicates that local economies influenced food habits and culinary practice by shaping resource availability, provisioning systems, cooking technologies and the archaeological visibility of different foods. In Babitonga Bay, the combined ORA, zooarchaeological and historical evidence points to food systems closely linked to estuarine resources, plant cultivation, small-scale animal keeping and household-level provisioning. In Pelotas, the Casa 2 residue pattern is more closely associated with C_3 plant inputs and terrestrial animal fats, within a regional economy structured by cattle ranching, charque production and urban markets. Porto Alegre adds further complexity, since the human isotope data suggest heterogeneous access to protein sources.

The third research question can be answered by emphasising the role of local economies as structuring conditions instead of direct determinants of culinary identity. Rural and urban contexts did not simply correspond to “traditional” and “modern” foodways, nor can social class or ethnicity be read directly from the biomolecular data. Instead, the results suggest that local economies shaped the range of foods available, the ways in which resources entered household provisioning, and the likelihood that particular foods became visible in ceramic residues or collagen isotope values. The influence of economic systems is clearest when these results are interpreted together with local ecology, ceramic use, market access, labour history, household organisation and preservation conditions.

8.4. Methodological considerations, limitations and further approaches

This thesis demonstrates the value of combining organic residue analysis, stable isotope analysis, zooarchaeological evidence and historical sources for the study of historical foodways in Southern Brazil. The integration of these approaches made it

possible to compare preserved vessel-use patterns, longer-term dietary protein intake, faunal baselines and historical context across Babitonga Bay and the Patos Lagoon region. ORA contributed information on substances absorbed and preserved in ceramic vessels, while SIA provided a broader view of human and faunal dietary protein sources. When interpreted together, these methods offered a more nuanced picture than either proxy could provide alone.

At the same time, the results of this thesis also show the importance of defining the limits of each line of evidence. ORA does not reconstruct the complete diet of a household or community, but rather the subset of substances that were processed in ceramic vessels, absorbed into the ceramic fabric, preserved after burial, recovered during extraction and detected during instrumental analysis (Evershed, 2008; Roffet-Salque et al., 2017a; Craig, 2021). Bulk collagen SIA, by contrast, reflects longer-term dietary protein intake and cannot identify specific dishes, cooking practices or the full range of plant foods consumed. For these reasons, the strength of the interpretations depends not only on the presence of diagnostic or compatible biomarkers and isotope values, but also on preservation, sample size, analytical resolution and the archaeological context of each assemblage.

For this reason, negative, low-yield and contamination-affected results are an important part of the dataset and not peripheral analytical failures. In the Babitonga Bay assemblages, the initial ORA dataset comprised 181 ceramic records, of which 164 AE extracts were retained for molecular and isotopic interpretation and 17 records were not retained for AE-based interpretation. These 17 records include 12 low-yield or non-interpretable outputs and five Arroio Tamarina 2 extracts excluded because AE chromatographic contamination compromised reliable interpretation. In the Casa 2 assemblage, the initial list included 20 records, of which 14 AE extracts produced interpretable lipid profiles above the practical screening threshold and were retained for molecular and isotopic interpretation, while six records were not retained for AE-based interpretation because they produced low-yield or non-interpretable lipid signals during initial screening. These non-retained cases limit the extent to which the

interpretable residues can be generalised to entire sites or communities. They also provide useful evidence for differential lipid preservation and analytical recovery across contexts, ceramic fabrics, use histories and post-excavation storage conditions. The Casa 2 TLE/TMS subset provides a further example of the need to distinguish between analytical records and interpretable datasets. Although the TLE/TMS chromatograms were retained for transparency, they were excluded from source-specific interpretation because they were affected by laboratory hexane contamination.

The inclusion of representative chromatograms in the results Chapters and the full chromatogram appendix was not only a matter of documentation, but also part of the interpretative framework of the thesis. Good-quality chromatograms support the identification of recurring lipid patterns, while low-quality, low-yield or contaminated chromatograms help define the limits of interpretation. Where plasticiser contamination or other modern exogenous compounds were identified, these were treated cautiously and excluded from lipid abundance estimates where possible, as described in Chapter 4. However, the presence of such contamination also highlights the challenges of working with previously excavated and curated collections, where post-excavation handling and storage conditions cannot always be reconstructed or controlled.

One important consideration is the preservation and detectability of organic residues over long periods. The molecular composition of absorbed residues can be altered by diagenesis, heating, oxidation, microbial activity, burial conditions and post-excavation storage. Fragile compounds, including unsaturated lipids and short-chain fatty acids, are particularly prone to degradation, while more stable saturated fatty acids and some thermal alteration products may survive more readily (Evershed, 2008; Roffet-Salque et al., 2017a; Eusebio, 2020; Mallol et al., 2025). As a result, the preserved lipid profile may only represent a partial and transformed record of the original contents of a vessel.

An absence of a specific residue is not definitive proof that the food was never used. Highly processed plant foods, lipid-poor ingredients, diluted broths, manioc flour, leafy vegetables or fruits may leave faint, non-specific or undetectable chemical traces. This is also relevant for aquatic products and heating biomarkers: their absence in a sample may reflect degradation, low original lipid input, vessel cleaning, repeated reuse, ceramic fabric, burial environment or analytical recovery, instead of absence from the broader cuisine. Caution is warranted before concluding that a food was absent from the assemblage, since taphonomic processes and analytical thresholds may be responsible for its non-detection.

A further methodological consideration concerns the stable isotope component of this study, which was based on bulk collagen analysis. Bulk $\delta^{13}C$ and $\delta^{15}N$ values are useful for identifying broad dietary protein patterns and for comparing humans and animals within regional isotopic baselines, but they cannot distinguish all possible combinations of marine, freshwater, terrestrial, C_3 and C_4 resources. This is particularly important in mixed-source contexts such as Southern Brazil, where aquatic foods, C_3 plants, C_4 plants, C_4-fed livestock and terrestrial animal products may overlap isotopically.

Future application of compound-specific isotope analysis of amino acids (CSIA-AA), targeting both $\delta^{13}C$ and $\delta^{15}N$ values of individual amino acids in bone collagen, could refine the interpretation of dietary inputs, especially in cases where bulk isotope values are difficult to separate (Soncin et al., 2021; Choy et al., 2024). By distinguishing source and trophic amino acids, this approach may help to separate baseline effects from trophic-level effects and provide a more detailed assessment of marine, terrestrial and plant contributions. However, CSIA-AA also requires well-preserved collagen, robust ecological baselines and careful consideration of trophic offsets and overlapping signals. For this reason, it should be seen as a complementary future approach rather than as a replacement for contextual interpretation based on faunal baselines, archaeology, ORA and historical sources.

Another promising avenue for future research involves targeted analysis of fruit-derived organic acids and other polar compounds, which could complement lipid residue analysis by increasing the range of plant-based preparations detectable in archaeological ceramics. Compounds such as tartaric, malic, succinic, fumaric and syringic acids have been used in studies of grape products, fruit processing and possible fermented beverages, but their interpretation is not straightforward. These compounds may vary in preservation, extraction efficiency and source specificity, and their presence or absence should not be interpreted without reference materials, quantitative criteria and archaeological context (Garnier and Valamoti, 2016; Drieu et al., 2020; Chassouant et al., 2021; McGovern et al., 2021; Irto et al., 2022).

In the contexts examined in this thesis, such analyses would be useful not because they would automatically identify specific drinks or recipes, but because they could test hypotheses that remain difficult to address through lipid analysis alone. These include the possible processing of fruits, plant-based preparations or fermented products in particular vessel types. This would be especially valuable if combined with ceramic form, function, fabric and decorative treatment, since different vessel categories may have been used for different culinary or storage practices. Future work could examine whether specific forms or decorated ceramics are associated with particular organic acid profiles, and not treating all sherds as functionally equivalent.

This approach would help clarify whether differences between assemblages reflect resource use, vessel function, ceramic technology, preservation or household practice. However, organic acid analysis should be treated as a complementary approach, not as a direct route to identifying social identity or specific culinary traditions. Its interpretative value would depend on experimental reference materials, robust analytical protocols, vessel typology, archaeological context and historical evidence.

Chronological resolution remains a limiting factor in this study. Although the archaeological contexts broadly date between the late eighteenth and early twentieth

centuries, it was not always possible to define more precise timeframes for individual ceramic assemblages or residue formation events. Chronological estimates were based primarily on archaeological context and ceramic typology, supported in some cases by radiocarbon dates on associated faunal remains (Fossile et al., 2023). As discussed in earlier Chapters, this period also coincides with sections of the radiocarbon calibration curve that can limit chronological precision, meaning that small chronological differences may not always be resolvable through conventional radiocarbon dating alone (Bronk Ramsey, 2009; Hogg et al., 2020; Reimer et al., 2020).

Compound-specific radiocarbon dating of absorbed lipid residues offers a promising future approach because it can, in principle, date carbon associated more directly with vessel use (Stott et al., 2003; Berstan et al., 2008; Casanova et al., 2022b). This would be particularly useful for testing whether changes in pottery use and food processing occurred gradually or whether they corresponded more closely with historical processes such as immigration, urban growth, market expansion or changes in household provisioning.

However, CSRD should not be treated as a complete solution to the chronological limitations of this study. It requires sufficiently preserved and source-specific compounds, careful control of modern contamination, and enough lipid concentration for reliable measurement (Casanova et al., 2022b). The resulting dates still require calibration and may be affected by the same calibration-curve limitations as other radiocarbon dates. In addition, aquatic or mixed carbon sources may introduce reservoir effects, and absorbed residues may represent one or more episodes of vessel use rather than the deposition of the sherd itself. For these reasons, CSRD would be most useful if applied alongside ceramic typology, stratigraphic information, faunal dating, residue composition and historical documentation.

Beyond laboratory analysis, future work would benefit from a stronger integration of experimental, ethnographic and material-culture approaches. Controlled cooking experiments can help clarify how different ingredients, recipes, heating

regimes, cleaning practices and repeated vessel use affect residue formation and preservation in ceramic fabrics (Evershed, 2008; Roffet-Salque et al., 2017a; Craig, 2021). This would be particularly useful for evaluating why some foods, such as fatty animal products, are more visible in lipid residues than others, such as lipid-poor plant preparations, diluted broths or highly processed ingredients.

Ethnographic and ethnohistorical research with coastal and rural communities in Brazil could also provide useful comparative information on vessel use, cooking practice, cleaning, reuse and the relationship between ceramic and non-ceramic technologies. Such work would need to be applied cautiously, since contemporary practices cannot be treated as direct survivals of nineteenth- or early twentieth-century foodways. However, studies of small-scale fishing, horticulture and household provisioning in coastal Brazil can help frame the kinds of practical decisions that shape daily food preparation and resource use (Hanazaki and Begossi, 2003; Begossi, 2006b; Fossile et al., 2023; Herbst et al., 2023).

This is especially important in historical contexts, where ceramics represented only one part of a broader culinary repertoire. Metal cookware, glass containers, industrial packaging and other non-ceramic tools were increasingly available during the nineteenth and early twentieth centuries. These materials may have been used for particular foods or storage practices without leaving direct lipid evidence in the ceramic record. As a result, the ORA data presented in this thesis should be interpreted as evidence for selected ceramic vessel-use practices, not as a complete record of all foods prepared or consumed.

Future research should integrate organic residue analysis with ceramic form, function, fabric, use-wear, faunal remains, archaeobotanical evidence, documentary sources and ethnographic comparison. This would make it possible to assess whether differences between assemblages reflect food choice, vessel function, technological change, preservation, access to resources or broader economic conditions. Such an approach would be particularly valuable for historical Brazilian contexts, where

foodways were shaped by overlapping Indigenous, African, European, rural, urban, market and household traditions, but where these dimensions cannot be isolated through biomolecular data alone.

8.5. Final considerations

Although this thesis is presented as an (almost) linear academic product, the process behind it was anything but. Doing research is often a conflicting experience, one shaped by uncertainty, solitude, and doubt as much as by curiosity and discovery. Over the course of this project, I developed a conflicted yet enduring love for research. I learned that it is not an exercise in quick results, but a practice of patience. It takes time before the pieces align, and strength to keep going when they don't. That, more than any analytical insight, is one of the greatest lessons this project has taught me.

In the final stages of writing, I engaged more deeply with the material, the communities, and the broader implications of this work than I had in the years before. What had often felt like disconnected data and fragmented case studies began to take form as a coherent, meaningful narrative. I came to realise that the value of this research lies not only in what it reveals about the past, but also in how it speaks to present-day dynamics, and in the ways it can be shared through inclusive, collaborative "knowledge-making."

Lastly, collaborating with local scholars and archaeological teams in Brazil reminded me that research in postcolonial contexts must go beyond recovering data or retelling history. It must also involve listening, co-producing knowledge, and recognising the lived experiences and voices that have long been excluded from dominant narratives. This is not about exporting theory or correcting memory: it is about building bridges between places, perspectives, and people.

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APPENDICES

APPENDIX 1 - Organic residue analysis of ceramics from Babitonga Bay - Morro Grande, Arroio Tamarina 2, Foz Do Cubatao and Unidade 21 - Praia Grande - CHAPTER 5

Compounds marked with an asterisk **(*)** are reported as possible or tentative assignments only. They are retained for transparency but are not treated as diagnostic biomarkers unless supported by additional evidence, including lipid concentration, chromatographic profile, APAA/DB23 indicators, isoprenoids and/or compound-specific isotope values.

SFA: saturated fatty acids; **DC:** dicarboxylic acids; **UFA:** unsaturated fatty acids; **br:** branched fatty acids; **pris:** pristanic acid; **phy:** phytanic acid; - Targeted DB-23/SIM screening corresponds to the AQUASIM.m method used to monitor selected plant and aquatic-related compounds, including APAAs, TMTD, pristanic acid and phytanic acid. Where available, these outputs are reported in the relevant columns below.

To improve legibility, the Babitonga Bay organic residue dataset is divided into complementary tables instead of being presented as a single wide table. Table **1A** reports sample information, lipid recovery, the main AE molecular results and the targeted DB-23/SIM compounds detected for the retained extracts. Table **1B** reports APAA and isoprenoid proxy data, CSIA results, TLE/TMS status. Table **1C** reports the 17 records from the initial Babitonga ORA dataset that were not retained for AE-based molecular or isotopic interpretation, including 12 low-yield or non-interpretable outputs and five Arroio Tamarina 2 extracts excluded because AE chromatographic contamination compromised reliable interpretation.

APPENDIX 1A: Sample information, lipid recovery, AE molecular summary and DB-23/SIM compounds detected

Note on highlighted retained AT2 records: Highlighted rows indicate retained AE extracts from Arroio Tamarina 2 that showed moderate chromatographic background, contaminant peaks, elevated P/S ratios and/or incomplete quality-control information during chromatographic review. Highlighting does not indicate exclusion from the retained dataset. These samples were retained because $C_{16:0}$, $C_{18:0}$, internal standards and associated molecular and/or isotopic data were considered sufficiently interpretable, but their interpretation should be treated cautiously. Samples excluded from AE-based molecular and isotopic interpretation are reported separately in Appendix 1C.

TRD_ID	Sample_ID	Site	Method	Fragment	Decoration	Thickness	Concentration (µg/g)	AE molecular compounds/classes retained*	DB-23/SIM targeted compounds detected	P/S ratio	C12:0 %	C14:0 %
TRD_1469	U21-1642	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	9.3	12.03	SFA (C12-C24), UFA (C18:1, C18:2, C22:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid;	1.72	0.51	4.15

									phytanic acid			
TRD_1470	U21-2336	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	4	42.97	SFA (C12-C20), UFA (C18:1), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.64	0.79	4.34
TRD_1471	U21-1502	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	6.7	23.1	SFA (C12-C22), UFA (C18:1), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.01	0.85	2.07
TRD_1472	U21-2344	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	5.6	31	SFA (C12-C22), UFA (C18:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.91	0.38	3.39

TRD_1473	U21-1506	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	6.24	63.78	SFA (C12-C20), UFA (C16:1, C18:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.5	0.57	1.27
TRD_1474	U21-1512	Unidade 21	AE, TLE (tms)	Body	Smoothed	7.05	29.03	SFA (C12-C20), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.98	0.7	2.56
TRD_1476	U21-2303	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	5.74	8.56	SFA (C12-C20), UFA (C16:1), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	3.43	0.22	2.68

TRD_1477	U21-1688	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	5.23	61.86	SFA (C12-C24), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.89	0.54	1.87
TRD_1478	U21-2251	Unidade 21	AE, TLE (tms)	Base	Smoothed	17.35	50.5	SFA (C12-C20), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	0.98	0.09	2.5
TRD_1479	U21-1514	Unidade 21	AE, TLE (tms)	Rim	Internal Glaze	2.36	13.24	SFA (C12-C18), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.29	0.6	3.21

TRD_1480	U21-1696	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	7.49	18.69	SFA (C12-C22), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	2.75	0.45	1.4
TRD_1481	U21-2294	Unidade 21	AE	Body	Smoothed	5.6	18.69	SFA (C12-C20), UFA (C16:1, C18:1, C18:2), DC (C10-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.3	2.85	6.61
TRD_1482	U21-1643	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	6.99	26.18	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C8-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.73	6.3	9.28

TRD_ 1483	U21-2207	Unidade 21	AE	Body	Internal Glaze	8.5	20	SFA (C9- C20), UFA (C16:1, C18:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.22	0.47	1.79
TRD_ 1484	U21-2246	Unidade 21	AE	Body	Internal Glaze	6.1	49.5	SFA (C12-C20), UFA (C16:1, C18:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.32	0.32	2.4
TRD_ 1485	U21-1630	Unidade 21	AE	Base	Internal Glaze	9.4	103.95	SFA (C12-C20), UFA (C16:1, C18:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.13	1.67	3.14

TRD_1486	U21-2395	Unidade 21	AE	Base	Internal Glaze	11.51	18.77	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.86	0.21	1.75
TRD_1487	U21-2394	Unidade 21	AE, TLE (tms)	Body	Smoothed	9.4	11.1	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.59	1.79	7.29
TRD_1488	U21-1659	Unidade 21	AE, TLE (tms)	Base	Smoothed	8.8	77.95	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.22	4.73	3.52
TRD_1489	U21-2372	Unidade 21	AE	Body	Smoothed	6.63	19.91	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*,	TMTD; pristanic acid; phytanic acid	2.82	1.61	4.65

								branched FA candidates*				
TRD_1490	U21-1626	Unidade 21	AE	Body	Smoothed	5.49	43.52	SFA (C12-C20tr), UFA (C16:1, C16:2), branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.95	0.34	2.26
TRD_1491	U21-1527	Unidade 21	AE, TLE (tms)	Body	Smoothed	4.45	93.54	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.53	0.38	5.5
TRD_1492	U21-1578	Unidade 21	AE, TLE (tms)	Base	Smoothed	7.11	102.84	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2	1.24	2.48

TRD_1493	U21-1592	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	6.23	14.37	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.12	2.15	3.99
TRD_1494	U21-1577	Unidade 21	AE, TLE (tms)	Body	Smoothed	5.77	10.75	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	3.11	0.18	1.55
TRD_1495	U21-1614	Unidade 21	AE, TLE (tms)	Rim	Internal Glaze	5.64	52.72	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.36	0.29	1.53

TRD_1496	U21-1616	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	9.29	24.66	SFA (C12-C20 (even only)), UFA (C16:1, C18:1, C18:2), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.62	0.25	1.1
TRD_1497	U21-1529	Unidade 21	AE, TLE (tms)	Body	Smoothed	4.97	104.12	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.34		
TRD_1498	U21-1690	Unidade 21	AE, TLE (tms)	Body	Smoothed	5.32	64.99	SFA (C14- C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.56	0.56	3
TRD_1499	U21-2382	Unidade 21	AE, TLE	Body	Smoothed	4.58	28.74	SFA (C12-C22 (even only)), UFA (C16: tr, C18:1	C18 APAAs; TMTD;	3.06	0.18	2.33

			(tms)					tr), branched FA candidates*	pristanic acid; phytanic acid			
TRD_ 1500	U21-1599	Unidade 21	AE	Body	Internal Glaze	5.63	277.87	SFA (C12-C24tr), UFA (C18:1, C18:2), DC (C9-C13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.94	0.59	4.77
TRD_ 1501	U21-1700	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	6.37	36.36	SFA (C12-C18), UFA (C16:1, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	0.93	0.79	5.36
TRD_ 1502	U21-2234	Unidade 21	AE	Body	Smooth ed	6.04	14.74	SFA (C12-C20), UFA (C16:1 tr), DC (C9, C10) candidates*,	TMTD; pristanic acid; phytanic acid	0.77	1.53	2.23

								branched FA candidates*				
TRD_1503	U21-2380	Unidade 21	AE, TLE (tms)	Body	Smoothed	6.6	12.63	SFA (C12-C18), UFA (C16:1, C18:1, C18:2), DC (C9tr) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	2.09	0.15	1.49
TRD_1504	U21-2349	Unidade 21	AE	Body	Smoothed	3.84	51.72	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.6	0.46	2.21

TRD_1505	U21-1582	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	7.16	17.98	SFA (C12-C24 (no 19, 21)), UFA (C16:1, 16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	2.86	0.72	5.03
TRD_1506	U21-1609	Unidade 21	AE	Body	Smoothed	4.41	8.3	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.21	0.98	2.39
TRD_1507	U21-2273	Unidade 21	AE	Base	Smoothed	14.73	9.81	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid;	2.01	0.32	11.74

									phytanic acid			
TRD_1508	U21-2202	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	8.65	49.92	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.05	0.43	1.26
TRD_1509	U21-2242	Unidade 21	AE	Body	Internal Glaze	4.25	8.48	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	3.15	0.15	1.56
TRD_1510	U21-1660	Unidade 21	AE, TLE (tms)	Body	Smoothed	8.54	35.92	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	2.61	0.1	1.3

TRD_1511	U21-1598	Unidade 21	AE, TLE (tms)	Rim	Internal Glaze	5.1	304.19	SFA (C12-C20(even only)), UFA (C18:1, C18:2), branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.49	0.37	2.09
TRD_1512	U21-2240	Unidade 21	AE	Body	Internal Glaze		32.22	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.7	9.35	10.53
TRD_1513	U21-2296	Unidade 21	AE	Base	Smoothed	10.7	88.73	SFA (C12-C20tr), UFA (C16:1tr, 16:2tr, C18:1, C18:2), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.79	0.83	3.13

TRD_1514	U21-2275	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	6.65	64.86	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	3.05	2.54	13.68
TRD_1515	U21-1641	Unidade 21	AE, TLE (tms)	Body	Smoothed	5.24	265.14	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	3.84	1.71	2.5
TRD_1516	U21-1607	Unidade 21	AE	Body	Smoothed	6.27	50.06	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.66		

TRD_1517	U21-1565	Unidade 21	AE	Body	Smoothed	6.75	33.86	SFA (C12-C22tr), UFA (C16:1tr, 16:2tr, C18:1, C18:2), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.65	0.3	3.22
TRD_1518	U21-2261	Unidade 21	AE	Rim	Internal Glaze	3.64	38.62	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.11	0.4	2.33
TRD_1519	U21-1575	Unidade 21	AE, TLE (tms)	Base	Internal Glaze	11	76.29	SFA (C12-C20tr), UFA (C16:1, 16:2, C18:1, C18:2), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.55	2.38	5
TRD_1520	U21-1663	Unidade 21	AE, TLE (tms)	Body	Internal Glaze	5.55	28.88	SFA (C12-C22tr), UFA (C16:1, C18:1, C18:2), DC (C10) candidates*,	TMTD; pristanic acid;	1.96	5.22	6.4

								branched FA candidates*	phytanic acid			
TRD_1521	U21-1555	Unidade 21	AE	Body	Internal Glaze	10.97	26.53	SFA (C12-C22), UFA (C16:1, 16:2, C18:1, C18:2), branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.07	0.54	2.66
TRD_1522	U21-2350	Unidade 21	AE	Body	Smoothed	9.61	92.83	SFA (C12-C20), UFA (C16:1, C18:1, C18:2), DC (C9-C13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.99	0.54	7.02
TRD_1523	U21-1537	Unidade 21	AE	Body	Internal Glaze	4.03	43.21	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.03	0.65	7.82

TRD_1289	MG1-6625	Morro Grande 1	AE, TLE (tms)	Body	Brushed Straight	7.6	11.92	SFA (C9-C22), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.6	2.27	7.14
TRD_1290	MG1-6335	Morro Grande 1	AE, TLE (tms)	Body	Smoothed	8.1	446.09	SFA (C12-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C18) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.4	0.29	2.67
TRD_1292	MG1-5494	Morro Grande 1	AE	Rim	Brushed Pattern	6.7	10.85	SFA (C10-C19), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C14) candidates*, branched FA candidates*; minor	—	1.2	3.58	9.09

								non-diagnostic peaks*				
TRD_1293	MG1-5200	Morro Grande 1	AE, TLE (tms)	Body	Smoothed	9.2	291.94	SFA (C9-C20, C22), UFA (C16:1, C18:1, C20:1, C22:1), DC (C7-C14) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	2.5	1.25	3.17
TRD_1294	MG1-6841	Morro Grande 1	AE	Body	Smoothed	7.4	47.09	SFA (C9-C30), UFA (C16:1 tr, C18:1 tr), DC (C9-C12) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	0.9	1.45	3.76
TRD_1295	MG1-5287	Morro Grande 1	AE	Body	Brushed Straight	5.8	53.4	SFA (C12-C24), UFA (C16:1, C18:1, C20:1),	TMTD; pristanic acid;	0.6	1.6	4.06

								branched FA candidates*	phytanic acid			
TRD_1296	MG1-5233	Morro Grande 1	AE, TLE (tms)	Body	Brushed Straight	9.3	33.45	SFA (C9-C26), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C14) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.6	1.3	3.74
TRD_1297	MG1-5514	Morro Grande 1	AE, TLE (tms)	Rim	Brushed Pattern	5.6	12.12	SFA (C10-C24), UFA (C16:1, C18:1, C20:1, C22:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.4	4.81	8.71
TRD_1299	MG1-6434	Morro Grande 1	AE	Body	Smoothed	5.6	1062.53	SFA (C12-C24), UFA (C16:1, C18:1, C20:1), DC (C8-C18) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid;	0.6	0.1	1.12

									phytanic acid			
TRD_1300	MG1-6254	Morro Grande 1	AE, TLE (tms)	Body	Brushed Straight	5.4	738.74	SFA (C10-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C7-C16) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1	0.42	4.05
TRD_1301	MG1-6340	Morro Grande 1	AE, TLE (tms)	Body	Corrugated	6.1	325.49	SFA (C8-C24), UFA (C16TR, C18), DC (C8-C14) candidates*	TMTD; pristanic acid; phytanic acid	2.6	0.33	
TRD_1302	MG1-6544	Morro Grande 1	AE, TLE (tms)	Body	Smoothed	6.3	540.66	SFA (C9-C26), UFA (C16:1 tr, C18:1 tr), DC (C6-C16) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.7	0.58	2.15

TRD_1303	MG3-276	Morro Grande 3	AE, TLE (tms)	Body	Smoothed	8.3	312.94	SFA (C9-C24), UFA (C16:1 tr, C18:1 tr), DC (C7-C15) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; phytanic acid	4.1	1.37	2.05
TRD_1304	MG1-6507	Morro Grande 1	AE	Body	Smoothed	8.9	18.56	SFA (C7-C20), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.1	2.77	7.47
TRD_1305	MG1-6062	Morro Grande 1	AE, TLE (tms)	Rim	Brushed Straight	6.1	488.96	SFA (C9-C20), UFA (C16:1, C18:1), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.9	0.69	3.86
TRD_1306	MG1-5190	Morro Grande 1	AE, TLE (tms)	Body	Corrugated	7.7	322.52	SFA (C9-C24), UFA (C16:1, C18:1, C20:1), DC (C7-C14, C20) candidates*,	C18 APAAs; C20 APAAs; TMTD; pristanic	2.2	1.64	6.25

								branched FA candidates*; minor non-diagnostic peaks*	acid; phytanic acid			
TRD_1308	MG1-5277	Morro Grande 1	AE, TLE (tms)	Body	Brushed Pattern	6.4	7.05	SFA (C9-C18), UFA (C16:1, C18:1, C20:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.2	4.82	12.19
TRD_1309	MG1-6476	Morro Grande 1	AE	Body	Smoothed	7.4	9.8	SFA (C12-C24, C26), UFA (C16:1, C18:1, C22:1), DC (C9-C14) candidates*	TMTD; pristanic acid; phytanic acid	1.3	1	4.56
TRD_1310	MG1-6775	Morro Grande 1	AE	Body	Brushed Pattern	8.3	100.66	SFA (C10-C28), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C22, C24) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.2	1.6	6

TRD_1311	MG1-5297	Morro Grande 1	AE	Body	Brushed Straight	6.9	11.36	SFA (C10-C18, C20, C22, C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C14) candidates*, branched FA candidates*	—	1.7	2.36	7.06
TRD_1314	MG1-6129	Morro Grande 1	AE	Body	Brushed Straight	6.2	50.1	SFA (C9-C22), UFA (C16:1, C18:1), branched FA candidates*	pristanic acid	1.7	0.38	2.54
TRD_1315	MG1-5296	Morro Grande 1	AE, TLE (tms)	Body	Brushed Pattern	6.3	168.66	SFA (C10-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C20) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; C20 APAAs; phytanic acid	1.7	1.97	6.68

TRD_1316	MG1-5513	Morro Grande 1	AE, TLE (tms)	Rim	Brushed Straight	10.01	17.23	SFA (C12-C20, C22, C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C7-C15) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.86	0.87	6.79
TRD_1318	MG1-6506	Morro Grande 1	AE	Rim	Brushed Pattern	7.2	9.51	SFA (C12-C20), UFA (C16:1 tr, c18:1 tr), DC (C9, C13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.6	1.17	3.93
TRD_1319	MG1-6519	Morro Grande 1	AE, TLE (tms)	Base	Brushed Straight	9.2	16.39	SFA (C9-C22), UFA (C16:1 tr, C18:1 tr), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.9	0.41	3.89
TRD_1320	MG1-6334	Morro Grande 1	AE	Body	Brushed Straight	6.8	983.58	SFA (C9-C24), UFA (C16:1, C18:1), DC (C9-C15) candidates*,	C18 APAAs; C20 APAAs; TMTD;	1	0.07	0.31

								branched FA candidates*	pristanic acid; phytanic acid			
TRD_1321	MG1-275	Morro Grande 1	AE, TLE (tms)	Body	Smoothed	8.1	1932.22	SFA (C14-C18, C20-C24), UFA (C16:1, C18:1), DC (C8-C20) candidates*, branched FA candidates*	pristanic acid; phytanic acid	2.2	1.35	6.93
TRD_1322	MG1-6718	Morro Grande 1	AE	Body	Smoothed		33.17	SFA (C12-C26), UFA (C16:1, C18:1), DC (C10-C13) candidates*, branched FA candidates*	pristanic acid; phytanic acid	2.15	0.84	2.66
TRD_1323	MG1-5182	Morro Grande 1	AE	Rim	Brushed Straight	6.5	88.18	SFA (C12-C22), UFA (C16:1, C18:1, C22:1), DC (C9-C14) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.4	0.57	5.42

TRD_1324	MG1-6543	Morro Grande 1	AE	Rim	Brushed Straight	6	18.96	SFA (C12-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C9-C14) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.33	1.71	4.76
TRD_1325	MG1-6071	Morro Grande 1	AE	Rim	Brushed Straight	6.3	73.5	SFA (C8-C30), UFA (C16:1 tr, C18:1 tr), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.16	0.59	2.22
TRD_1326	MG1-6354	Morro Grande 1	AE	Body	Brushed Pattern	6.8	25.36	SFA (C12-C26), UFA (C16:1, C18:1, C20:1, C22:1), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.72	1.32	6.17

TRD_1327	MG1-6529	Morro Grande 1	AE	Body	Brushed Straight	6.6	17.91	SFA (C12-C22), UFA (C16:1, C18:1), DC (C13?) candidates*, branched FA candidates*	TMTD	1.69	0.86	6.34
TRD_1328	MG1-6171	Morro Grande 1	AE, TLE (tms)	Body	Brushed Straight	5.6	301.45	SFA (C9-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C7-C16, C19, C20) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; C20 APAAs; TMTD	1.73	1.17	9.06
TRD_461	FC-3087	Foz do Cubatão	AE	Body	Brushed Straight	6	249.3	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid;	1.09		

									phytanic acid			
TRD_462	FC-3022	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	0.8	87.2	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.47		
TRD_463	FC-3017	Foz do Cubatão	AE	Rim	Brushed Straight	6.3	199.8	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.21		
TRD_464	FC-3365	Foz do Cubatão	AE	Rim	Brushed Straight	1	156	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	3.1	15.35	11.82

TRD_465	FC-337	Foz do Cubatão	AE	Body	Smoothed	7.7	67.5	SFA (C10-24), UFA (C16:1, C18:1, C22:1), DC (13) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.37		
TRD_466	FC-3553	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	0.7	9.2	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	2.5	0.83	7.33
TRD_467	FC-1966	Foz do Cubatão	AE	Rim	Brushed Straight	0.8	270.9	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.11		

TRD_469	FC-4309	Foz do Cubatão	AE	Rim	Smoothed	8.6	23.4	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	3.1	1.59	5.03
TRD_470	FC-7140	Foz do Cubatão	AE	Base	Smoothed	9	192.3	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.08		
TRD_471	FC-2773	Foz do Cubatão	AE, TLE (tms)	Body	Brushed Straight	2	368.7	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.79		

TRD_472	FC-3761 (3811)	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	6.9	179.3	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.27		
TRD_473	FC-7548K8	Foz do Cubatão	AE	Body	Smoothed	2.5	751.8	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	0.65		
TRD_475	FC-2752Al5	Foz do Cubatão	AE, TLE (tms)	Body	Brushed Straight	6.3	538.4	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*,	TMTD; pristanic acid; phytanic acid	2.1		

								branched FA candidates*				
TRD_476	FC-2747	Foz do Cubatão	AE	Rim	Brushed Straight	1	34.8	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	0.9		
TRD_477	FC-7406	Foz do Cubatão	AE	Rim	Brushed Straight	0.7	116.3	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	0.56		
TRD_478	FC-6400	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	6.2	252.1	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C16) candidates*, branched FA candidates*; minor	C18 APAAs; pristanic acid; phytanic acid	1.9	0.38	1.9

								non-diagnostic peaks*				
TRD_479	FC-6500	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	0.7	14.8	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	2.4	1.07	6.22
TRD_480	FC-2583	Foz do Cubatão	AE	Rim	Brushed Straight	6.4	10.7	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	0.94		
TRD_481	FC-3118	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	6.3	91.8	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.2	0.83	3.65

TRD_483	FC-5825	Foz do Cubatão	AE	Rim	Brushed Straight	1.2	155.1	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.35		
TRD_484	FC-216	Foz do Cubatão	AE	Rim	Brushed Straight	1	204	SFA (C12-C24), UFA (C16:1, C18:1, C22:1), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs	1.26		
TRD_485	FC-2677	Foz do Cubatão	AE, TLE (tms)	Body	Brushed Straight	2.5	37.8	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1		

TRD_486	FC-6302	Foz do Cubatão	AE	Rim	Brushed Straight	7.8	31.4	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2	2.77	6.33
TRD_487	FC-6875	Foz do Cubatão	AE, TLE (tms)	Rim	Brushed Straight	5.8	376.3	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.07		
TRD_488	FC-3202	Foz do Cubatão	AE	Body	Smoothed	5	17.1	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.1	0.9	5.68
TRD_489	FC-6240	Foz do Cubatão	AE	Rim	Brushed Straight	1	142	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*,	C18 APAAs; C20 APAAs; TMTD; pristanic acid;	0.7	0.37	1.45

								branched FA candidates*	phytanic acid			
TRD_492	FC-3651	Foz do Cubatão	AE	Rim	Brushed Straight	1	24	SFA (C12-C26), UFA (C16:1, C16:2, C22:1), DC (C9-C14) candidates*, branched FA candidates*	C18 APAAs	3.1	0.75	5.89
TRD_493	FC-5704	Foz do Cubatão	AE, TLE (tms)	Rim	Smoothed	1	70.6	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.2	0.68	3.6
TRD_494	FC-6964	Foz do Cubatão	AE	Rim	Brushed Straight	0.4	88.1	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic	1.14		

									acid; phytanic acid			
TRD_495	FC-5765	Foz do Cubatão	AE, TLE (tms)	Body	Smoothed	3.4	14.7	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	4.6	1.84	6.78
TRD_496	FC-2775	Foz do Cubatão	AE	Body	Brushed Straight	9.6	78.6	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.1		
TRD_497	FC-6018	Foz do Cubatão	AE	Rim	Smoothed	8.8	18	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.1		

TRD_498	FC-2428	Foz do Cubatão	AE	Rim	Smoothed	5.4	34.2	SFA (C14- C24), UFA (C16:1, C18:1, C22:1), DC (C8-C16) candidates*, branched FA candidates*	C18 APAAs; pristanic acid; phytanic acid	2.2	1.91	7.72
TRD_499	FC-2453	Foz do Cubatão	AE	Rim	Brushed Pattern	7	112.2	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.6	0.73	5.79
TRD_500	FC-5758	Foz do Cubatão	AE		Smoothed	7	22.9	SFA (C12-C26), UFA (C16:1, C18:1, C20:1, C22:1), DC (C8-C16) candidates*, branched FA candidates*	pristanic acid; phytanic acid	2.3	1.27	5.99

TRD_501	FC-3358	Foz do Cubatão	AE	Rim	Smoothed	7.9	67	SFA (C12-C22), UFA (C16:1, C18:1), DC (C9-C13) candidates*, branched FA candidates*	C18 APAAs; pristanic acid; phytanic acid	1.6	1.4	3.76
TRD_502	FC-5824	Foz do Cubatão	AE	Rim	Brushed Straight	8	369.4	SFA (C12-C24), UFA (C16:1, C18:1), DC 8C9-C12), branched FA candidates*	C18 APAAs; C20 APAAs; pristanic acid; phytanic acid	1.4	1.57	3.67
TRD_503	FC-SN003	Foz do Cubatão	AE	Rim	Brushed Straight	1	230.7	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	0.9	0.14	1.29

TRD_504	FC-339	Foz do Cubatão	AE	Rim	Brushed Straight	1	58.4	SFA (C12-C24), UFA (C16:1, C16:2, C18:1, C18:2), DC (C8-C18) candidates*, branched FA candidates*	C18 APAAs; TMTD	3.8	1.43	4.41
TRD_505	FC-3906	Foz do Cubatão	AE	Rim	Brushed Straight	1.5	375.7	SFA (C12-C24), UFA (C16:1, C18:1, C20:1, C22:1), DC (C9-C16, C18-C20) candidates*, branched FA candidates*	—	0.4	0.19	0.71
TRD_1223	AT02-311	Arroio Tamarina 2	AE	Body	Smoothed	5.3	78.14	SFA (C12-C26), UFA (C18:1, C18:2, C22:1), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.89	2.78	12.53

TRD_1329	AT02-232	Arroio Tamarina 2	AE	Body	Smoothed	11.3	31.25	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	0.62	1.56	3.78
TRD_1330	AT02-233	Arroio Tamarina 2	AE	Body	Smoothed	10.8	34.95	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.42	0.94	7.54
TRD_1331	AT02-315	Arroio Tamarina 2	AE	Base	Smoothed	3.2	48.22	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.17	0.63	5.97
TRD_1332	AT02-268	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	13.7	20.58	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*,	TMTD; pristanic acid; phytanic acid	0.71	0.47	3.94

								branched FA candidates*				
TRD_1333	AT02-302	Arroio Tamarina 2	AE, TLE (tms)	Body	Brushed	11.2	31.43	SFA (C12-C24), UFA (C16:1, C16:2, C18:1, C22:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	0.95	2.22	11.55
TRD_1334	AT02-231	Arroio Tamarina 2	AE	Body	Smoothed	1.5	42.76	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	0.9	0.51	1.94
TRD_1337	AT02-310	Arroio Tamarina 2	AE, TLE (tms)	Body	Painted	11.2	22.77	SFA (C12-C24), UFA (C18:1, C18:2, C22:1), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.35	1.86	9.31

TRD_1338	AT02-270	Arroio Tamarina 2	AE	Base	Painted	13.3	45.96	SFA (C14-C18, C20, C22, C24), UFA (C16:1tr, C16:2 tr, C18:1tr, C22:1tr), DC (C10-C13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.79	0.52	3.49
TRD_1339	AT02-277	Arroio Tamarina 2	AE, TLE (tms)	Body	Brushed	5.4	41.69	SFA (C12-C26), UFA (C16:1, C16:2 tr, C18:1, C22:1), DC (C9, C10) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.05	2.59	15.4
TRD_1340	AT02-304	Arroio Tamarina 2	AE	Base	Corrugated	1.2	11.51	SFA (C12-C26), UFA (C16:1, C18:1, C18:2, C22:1), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.34	2.44	9.48

TRD_1341	AT02-230	Arroio Tamarina 2	AE	Body	Brushed	5.1	28.57	SFA (C12-C28), UFA (C16:1 tr, C18:1 tr), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.61	1.26	1.6
TRD_1343	AT02-309	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	11.8	18.05	SFA (C12-C24), UFA (C16:1, C18:1, C18:2), DC (C9-C13) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.72	2.58	8.6
TRD_1344	AT02-346	Arroio Tamarina 2	AE	Body	Smoothed	12.7	22.04	SFA (C14-C24 (even only)), UFA (C16:1 tr, C18:1 tr, C22:1 tr), branched FA candidates*;	—	0.85		

								minor non-diagnostic peaks*				
TRD_1345	AT02-286	Arroio Tamarina 2	AE	Body	Painted	8.5	166.81	SFA (C10-C22), UFA (C18:1), DC (C8-C14) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	0.83	1.26	4.67
TRD_1346	AT02-307	Arroio Tamarina 2	AE	Body	Smoothed	12.2	19.65	SFA (C10-C28tr), UFA (C16:1tr, C16:2 tr, C18:1tr, C22:1tr), DC (C9-C12) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.63	0.87	7.85
TRD_1347	AT02-291	Arroio Tamarina 2	AE	Body	Smoothed	2.5	32.4	SFA (C12-C24), UFA (C16:1, C18:1, C22:1), DC (C9-C13) candidates*,	C18 APAAs; TMTD; pristanic acid;	1.33	0.9	6.66

								branched FA candidates*	phytanic acid			
TRD_1348	AT02-272	Arroio Tamarina 2	AE, TLE (tms)	Body	Brushed	12.4	31.94	SFA (C14-C18 C20 and C22 tr), UFA (C16:1, C18:1 tr), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.73	2.22	3.5
TRD_1350	AT02-400	Arroio Tamarina 2	AE	Body	Brushed	5.3	82.32	SFA (C12-C26), UFA (C16:1, C18:1), DC (C9-C10) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; TMTD; pristanic acid; phytanic acid	2.17	2.33	15.2
TRD_1351	AT02-318	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	11.9	27.14	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.89	0.62	2.27

TRD_1352	AT02-273	Arroio Tamarina 2	AE, TLE (tms)	Body	Brushed	12.3	11.56	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.41	1.29	6.23
TRD_1353	AT02-375	Arroio Tamarina 2	AE	Body	Smoothed	3.6	31.74	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.71	3.32	14.72
TRD_1354	AT02-260	Arroio Tamarina 2	AE				30.25	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.8	3.47	7.04

TRD_1355	AT02-293	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	11.1	7.94	SFA (C9-C22), UFA (C16:1. C16:2, C18:1 tr C18:2 tr, C22:1tr), DC (C9-C10) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	9.98	3.17	11.99
TRD_1356	AT02-313	Arroio Tamarina 2	AE	Body	Smoothed	7.4	18.8	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	TMTD; pristanic acid; phytanic acid	1.99	4.48	15.76
TRD_1357	AT02-328	Arroio Tamarina 2	AE	Body	Brushed	5.3	134.75	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*,	C18 APAAs; C20 APAAs;	0.74	0.78	3.11

								branched FA candidates*	TMTD; pristanic acid; phytanic acid			
TRD_1358	AT02-384	Arroio Tamarina 2	AE	Body	Smoothed	4	25.5	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.37	2.05	9.37
TRD_1359	AT02-378	Arroio Tamarina 2	AE	Body	Smoothed	4.4	54.03	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.4	1.29	6.21
TRD_1360	AT02-377	Arroio Tamarina 2	AE, TLE (tms)	Body	Brushed	7.9	113.87	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	2.22	0.91	2.6

TRD_1361	AT02-1361	Arroio Tamarina 2	AE	Body	Smoothed	6.4	79.74	SFA (C12-C24 (20, 24 tr)), UFA (C16:1 tr, C18:1 tr), DC (C9-C12) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	1.4	1.13	2.63
TRD_1362	AT02-370	Arroio Tamarina 2	AE	Body	Smoothed	6.4	37.38	SFA (C12-C24), UFA (C16:1, C18:1), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	C18 APAAs; TMTD; pristanic acid; phytanic acid	1.29	1.13	6.18
TRD_1363	AT02-388	Arroio Tamarina 2	AE	Body	Smoothed	5.4	68.2	SFA (C12-C20), UFA (C16:1), DC (C11-13) candidates*,	TMTD; pristanic acid; phytanic acid	1.6	1.15	5.91

								branched FA candidates*				
TRD_1364	AT02-262	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	6.9	54.31	SFA (C12-C20), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9) candidates*, branched FA candidates*	C18 APAAs; C20 APAAs; TMTD; pristanic acid; phytanic acid	2.73	1.24	4.66
TRD_1365	AT02-215	Arroio Tamarina 2	AE	Body	Painted	7.8	43.37	SFA (C12-C18), UFA (C16:1, C18:1), branched FA candidates*	TMTD; pristanic acid; phytanic acid	1.03	1.52	1.84
TRD_1366	AT02-330	Arroio Tamarina 2	AE, TLE (tms)	Body	Smoothed	9.6	16.73	SFA (C12-C22), UFA (C16:1, C16:2, C18:1, C18:2), DC (C9-C11) candidates*, branched FA candidates*	TMTD; pristanic acid; phytanic acid	8.18	3.75	17.55

TRD_1367	AT02-217	Arroio Tamarina 2	AE	Body	Smoothed	4	23.88	SFA (C12-C26), UFA (C16:1, C18:1, C18:2 tr, C22:1tr), DC (C9-C11) candidates*, branched FA candidates*; minor non-diagnostic peaks*	—	1.33	1.19	3.94

APPENDIX 1B: Targeted biomarkers, CSIA, TLE/TMS

Note on highlighted retained AT2 records: Highlighted rows indicate retained AE extracts from Arroio Tamarina 2 that showed moderate chromatographic background, contaminant peaks, elevated P/S ratios and/or incomplete quality-control information during chromatographic review. Highlighting does not indicate exclusion from the retained dataset. These samples were retained because $C_{16:0}$, $C_{18:0}$, internal standards and associated molecular and/or isotopic data were considered sufficiently interpretable, but their interpretation should be treated cautiously. Samples excluded from AE-based molecular and isotopic interpretation are reported separately in Appendix 1C.

TRD_ID	Sample_ID	Site	APAA C18 E/H DB23	APAA C20/C18 DB23	Proxy summary	SRR%	δ13C16:0 (‰)	δ13C18:0 (‰)	Δ13C (C18:0-C16:0)	TLE/TMS
TRD_1469	U21-1642	Unidade 21	4.93	0.16	Plant + aquatic-compatible proxies	60.4	-29.06	-29.33	-0.27	MAGs*; hydroxy FA candidates*; MAGs* / monopalmitin candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1470	U21-2336	Unidade 21	5.48		Plant-processing proxy	77	-24.12	-25.68	-1.56	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*; phorbol-like markers*
TRD_1471	U21-1502	Unidade 21	2.02		No APAA proxy	62.7	-27.02	-28.19	-1.17	hydroxy FA candidates*; resin diterpenoid markers*

TRD_1472	U21-2344	Unidade 21	5.49	0.1	Plant + aquatic-compatible proxies	79.1	-23.24	-23.8	-0.56	MAGs*; DAGs*; TAGs*; hydroxy FA candidates*
TRD_1473	U21-1506	Unidade 21	3.41		No APAA proxy	45.5	-28.6	-28.29	0.31	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*
TRD_1474	U21-1512	Unidade 21	1.05		No APAA proxy	76.2	-27.99	-29.27	-1.28	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1476	U21-2303	Unidade 21	5.03		Plant-processing proxy	46.1	-29.41	-29.07	0.34	
TRD_1477	U21-1688	Unidade 21	0		No APAA proxy	52.3	-28	-29.24	-1.24	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1478	U21-2251	Unidade 21	0.19		No APAA proxy	72.6	-21.64	-22.14	-0.5	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1479	U21-1514	Unidade 21	4.71	0.04	Plant-processing proxy	37.1	-25.42	-26.49	-1.07	
TRD_1480	U21-1696	Unidade 21	7.13	0.01	Plant-processing proxy	57.5	-31.07	-31.38	-0.31	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1481	U21-2294	Unidade 21	3.92		No APAA proxy	45.2	-28.89	-29.43	-0.54	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1482	U21-1643	Unidade 21	4.73		Plant-processing proxy	57	-28.53	-29.96	-1.43	MAGs*
TRD_1483	U21-2207	Unidade 21	0.26	1.09	Aquatic-compatible proxy	57.2	-27.91	-30.05	-2.14	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1484	U21-2246	Unidade 21	1.04		No APAA proxy	74.7	-25.05	-27.41	-2.36	hydroxy FA candidates*
TRD_1485	U21-1630	Unidade 21	4.02		Plant-processing proxy	49.3	-28.53	-29.96	-2.4	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1486	U21-2395	Unidade 21			No APAA proxy	76.4	-28.39	-30.74	-2.35	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1487	U21-2394	Unidade 21			No APAA proxy	58.5	-27.8	-29.87	-2.07	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1488	U21-1659	Unidade 21			No APAA proxy	49.9	-29.67	-29.98	-0.31	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1489	U21-2372	Unidade 21			No APAA proxy	50.7	-28.52	-28.88	-0.36	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1490	U21-1626	Unidade 21			No APAA proxy	53.2	-27.4	-28.17	-0.77	resin diterpenoid markers*
TRD_1491	U21-1527	Unidade 21			No APAA proxy	78.2	-21.77	-23.49	-1.72	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_1492	U21-1578	Unidade 21	8.17		Plant-processing proxy	61	-29.32	-29.34	-0.02	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1493	U21-1592	Unidade 21			No APAA proxy	61.6	-28.5	-29.05	-0.55	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1494	U21-1577	Unidade 21	10.83		Plant-processing proxy	61.2	-28.65	-30.38	-1.73	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1495	U21-1614	Unidade 21	6.28		Plant-processing proxy	68.7	-29.61	-29.13	0.48	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1496	U21-1616	Unidade 21			No APAA proxy	68.7	-28.66	-29.29	-0.63	MAGs*; MAGs* / monopalmitin candidates*; resin diterpenoid markers*

TRD_1497	U21-1529	Unidade 21	3.86		No APAA proxy	37.6	-18.53	-17.59	0.94	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_1498	U21-1690	Unidade 21			No APAA proxy	72.4	-23.26	-24.53	-1.27	resin diterpenoid markers*
TRD_1499	U21-2382	Unidade 21	7.89		Plant-processing proxy	49.3	-29.91	-30.43	-0.52	MAGs*; MAGs* / monopalmitin candidates*
TRD_1500	U21-1599	Unidade 21	3.14	0.18	Aquatic-compatible proxy	66.7	-19.89	-20.41	-0.52	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1501	U21-1700	Unidade 21			No APAA proxy	95.6	-27.02	-29.96	-2.94	MAGs*; resin diterpenoid markers*
TRD_1502	U21-2234	Unidade 21			No APAA proxy	64.6	-29.45	-30.22	-0.77	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1503	U21-2380	Unidade 21			No APAA proxy	47.8	-28.76	-29.44	-0.68	MAGs*; resin diterpenoid markers*
TRD_1504	U21-2349	Unidade 21	6.33		Plant-processing proxy	66.8	-23.67	-31.25	-7.58	resin diterpenoid markers*

TRD_1505	U21-1582	Unidade 21			No APAA proxy	81.5	-26.03	-28.54	-2.51	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1506	U21-1609	Unidade 21	0.15		No APAA proxy	58.6	-28.39	-28.69	-0.3	resin diterpenoid markers*
TRD_1507	U21-2273	Unidade 21	0.66		No APAA proxy	80.9	-19.85	-22.63	-2.78	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1508	U21-2202	Unidade 21	9.83		Plant-processing proxy	53.9	-28.91	-29.49	-0.58	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1509	U21-2242	Unidade 21			No APAA proxy	55	-29.83	-30.64	-0.81	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1510	U21-1660	Unidade 21	6.65	1.14	Plant + aquatic-compatible proxies	43.9	-29.92	-29.74	0.18	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1511	U21-1598	Unidade 21	7.31		Plant-processing proxy	80.5	-17.01	-15.12	1.89	MAGs*; DAGs*; TAGs*; MAGs* / monopalmitin candidates*; resin diterpenoid markers*
TRD_1512	U21-2240	Unidade 21			No APAA proxy	64.4	-28.42	-29.18	-0.76	MAGs*; resin diterpenoid markers*; phorbol-like markers*

TRD_1513	U21-2296	Unidade 21			No APAA proxy	50.5	-30.18	-30.46	-0.28	resin diterpenoid markers*
TRD_1514	U21-2275	Unidade 21			No APAA proxy	48	-29.27	-29.98	-0.71	
TRD_1515	U21-1641	Unidade 21	2.7		No APAA proxy	86.9	-19.71	-25.14	-5.43	MAGs*; DAGs*; TAGs*; resin diterpenoid markers*
TRD_1516	U21-1607	Unidade 21	9.75		Plant-processing proxy	70.8	-19.71	-25.14	-5.43	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1517	U21-1565	Unidade 21			No APAA proxy	74.6	-28.86	-28.98	-0.12	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1518	U21-2261	Unidade 21			No APAA proxy	51.1	-29.43	-29.78	-0.35	
TRD_1519	U21-1575	Unidade 21	4.33		Plant-processing proxy	67.8	-23.37	-20.77	2.6	resin diterpenoid markers*
TRD_1520	U21-1663	Unidade 21			No APAA proxy	72.3	-28.63	-29.08	-0.45	MAGs*; MAGs* / monopalmitin candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1521	U21-1555	Unidade 21			No APAA proxy	51.7	-29.16	-29.87	-0.71	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1522	U21-2350	Unidade 21	4.22		Plant-processing proxy	81.5	-23.95	-24.28	-0.33	resin diterpenoid markers*
TRD_1523	U21-1537	Unidade 21			No APAA proxy	81.3	-22.12	-27.4	-5.28	
TRD_1289	MG1-6625	Morro Grande 1			No APAA proxy	80.9	-27.3	-27.47	-0.17	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1290	MG1-6335	Morro Grande 1			No APAA proxy	49.3	-18.77	-19.65	-0.88	MAGs*
TRD_1292	MG1-5494	Morro Grande 1			No APAA proxy	65.1	-28.05	-29.62	-1.57	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1293	MG1-5200	Morro Grande 1	7.36	0.09	Plant + aquatic-compatible proxies	78.8	-9.66	-8.63	1.02	MAGs*; hydroxy FA candidates*
TRD_1294	MG1-6841	Morro Grande 1			No APAA proxy	58.3	-26.37	-28.22	-1.85	resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1295	MG1-5287	Morro Grande 1			No APAA proxy	78.5	-29.22	-29.67	-0.45	hydroxy FA candidates*
TRD_1296	MG1-5233	Morro Grande 1			No APAA proxy	65.4	-28.86	-29.83	-0.97	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1297	MG1-5514	Morro Grande 1			No APAA proxy	61.3	-26.79	-29.68	-2.89	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1299	MG1-6434	Morro Grande 1	4.44	0.12	Plant + aquatic-compatible proxies	85.9	-26.26	-25.64	0.62	
TRD_1300	MG1-6254	Morro Grande 1	4.68	0.07	Plant + aquatic-compatible proxies	64.1	-19.08	-20.35	-1.27	MAGs*; TAGs*
TRD_1301	MG1-6340	Morro Grande 1			No APAA proxy		-26.66	-25.46	1.19	hydroxy FA candidates*
TRD_1302	MG1-6544	Morro Grande 1	5.75	0.07	Plant + aquatic-compatible proxies	57.4	-20.3	-21.93	-1.63	MAGs*; DAGs*; hydroxy FA candidates*
TRD_1303	MG3-276	Morro Grande 3	3.8	0.02	No APAA proxy		-26.66	-28.54	-1.89	MAGs*; DAGs*

TRD_1304	MG1-6507	Morro Grande 1			No APAA proxy	60.4	-25.83	-27.41	-1.59	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1305	MG1-6062	Morro Grande 1	4.67		Plant-processing proxy	48.5	-20.42	-22.5	-2.08	MAGs*; DAGs*; TAGs*; hydroxy FA candidates*; long-chain alcohol candidates*
TRD_1306	MG1-5190	Morro Grande 1	4.79	0.05	Plant-processing proxy	59.7	-29.3	-30.7	-1.4	hydroxy FA candidates*; phorbol-like markers*
TRD_1308	MG1-5277	Morro Grande 1			No APAA proxy	62.3	24.22	23.55	-0.67	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1309	MG1-6476	Morro Grande 1			No APAA proxy	63.1	-20.77	-22.09	-1.32	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1310	MG1-6775	Morro Grande 1	4.48	0.16	Plant + aquatic-compatible proxies	49.1	-20.74	-26.58	-5.84	hydroxy FA candidates*
TRD_1311	MG1-5297	Morro Grande 1			No APAA proxy	82.1	-29.49	-32.3	-2.81	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1314	MG1-6129	Morro Grande 1			No APAA proxy	84.2	-27.91	-29.38	-1.47	

TRD_1315	MG1-5296	Morro Grande 1	2.48	0.12	Aquatic-compatible proxy	59.5	-24.07	-23.57	0.5	hydroxy FA candidates*
TRD_1316	MG1-5513	Morro Grande 1			No APAA proxy	57.4	-28.67	-29.82	-1.15	MAGs*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1318	MG1-6506	Morro Grande 1			No APAA proxy	60.1	-21.24	-22.22	-0.98	
TRD_1319	MG1-6519	Morro Grande 1			No APAA proxy	62.9	-25.84	-27.64	-1.79	hydroxy FA candidates*
TRD_1320	MG1-6334	Morro Grande 1	4.34	0.05	Plant-processing proxy	44.6	-27.77	-31.8	-4.04	hydroxy FA candidates*
TRD_1321	MG1-275	Morro Grande 1			No APAA proxy		-31.59	-33.36	-1.77	MAGs*; DAGs*; TAGs*; hydroxy FA candidates*
TRD_1322	MG1-6718	Morro Grande 1			No APAA proxy	65.8	-27.14	-27.3	-0.16	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1323	MG1-5182	Morro Grande 1			No APAA proxy	90.9	-20.74	-26.58	-5.84	oxidised resin diterpenoid markers*

TRD_1324	MG1-6543	Morro Grande 1			No APAA proxy	50.4	-27.7	-27.73	-0.03	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1325	MG1-6071	Morro Grande 1	2.14		No APAA proxy	49.4	-23.87	-24.86	-0.99	oxidised resin diterpenoid markers*; triterpenoid candidates*
TRD_1326	MG1-6354	Morro Grande 1	6.87		Plant-processing proxy		-24.28	-28.43	-4.15	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1327	MG1-6529	Morro Grande 1			No APAA proxy	64.8	-27.05	-28.56	-1.51	oxidised resin diterpenoid markers*
TRD_1328	MG1-6171	Morro Grande 1	4.54	0.24	Plant + aquatic-compatible proxies	92.2	-19.63	-20.93	-1.3	MAGs*
TRD_461	FC-3087	Foz do Cubatão	3.8		No APAA proxy	51.1	-27.2	-29.91	-2.71	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_462	FC-3022	Foz do Cubatão	4.5		Plant-processing proxy	39.8	-26.05	-27.1	-1.05	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_463	FC-3017	Foz do Cubatão	4.4		Plant-processing proxy	87.8	-27.02	-26.36	0.66	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_464	FC-3365	Foz do Cubatão			No APAA proxy		-21.79	-22.67	-0.8	hydroxy FA candidates*; resin diterpenoid markers*
TRD_465	FC-337	Foz do Cubatão	6.1		Plant-processing proxy	46.7	-20.84	-21.63	-0.7	resin diterpenoid markers*
TRD_466	FC-3553	Foz do Cubatão			No APAA proxy	48	-28.94	-27.57	1.37	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_467	FC-1966	Foz do Cubatão			No APAA proxy	84.6	-27.13	-29.41	-2.28	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_469	FC-4309	Foz do Cubatão			No APAA proxy	55	-20.44	-21.44	-0.9	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_470	FC-7140	Foz do Cubatão	1.8	0.1	Aquatic-compatible proxy	36.1	-19.43	-18.99	0.44	hydroxy FA candidates*; resin diterpenoid markers*
TRD_471	FC-2773	Foz do Cubatão	3.2		No APAA proxy	100	-28.98	-28.26	0.72	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_472	FC-3761 (3811)	Foz do Cubatão	3.5	0.18	Aquatic-compatible proxy	83.7	-19.43	-18.99	0.44	MAGs*; resin diterpenoid markers*; phorbol-like markers*

TRD_473	FC-7548K8	Foz do Cubatão	2.6	0.26	Aquatic-compatible proxy	64.7	-22.5	-21.59	0.9	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_475	FC-2752AI5	Foz do Cubatão			No APAA proxy		-21.02	-22.35	-1.33	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_476	FC-2747	Foz do Cubatão	2.9		No APAA proxy	51	-23.27	-26.95	-3.68	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_477	FC-7406	Foz do Cubatão	2.4		No APAA proxy	79.3	-25.91	-24.89	1.02	hydroxy FA candidates*; resin diterpenoid markers*
TRD_478	FC-6400	Foz do Cubatão	1.5		No APAA proxy	48	-18.65	-18.93	-0.2	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_479	FC-6500	Foz do Cubatão			No APAA proxy	46	-25.91	-24.89	1.02	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_480	FC-2583	Foz do Cubatão			No APAA proxy		-19.66	-19.34	0.33	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_481	FC-3118	Foz do Cubatão			No APAA proxy	73	-25.73	-26.9	-1.17	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_483	FC-5825	Foz do Cubatão			No APAA proxy		-18.12	-18.12	0.01	hydroxy FA candidates*; resin diterpenoid markers*
TRD_484	FC-216	Foz do Cubatão	1.3		No APAA proxy	72.2	-22.37	-22.34	0.03	
TRD_485	FC-2677	Foz do Cubatão			No APAA proxy	48	-20.21	-22.23	0.07	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_486	FC-6302	Foz do Cubatão			No APAA proxy	49	-22.37	-22.34	0.03	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_487	FC-6875	Foz do Cubatão			No APAA proxy		-26.8	-27.4	-0.58	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_488	FC-3202	Foz do Cubatão			No APAA proxy	47	-27.16	-29.3	-2.14	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_489	FC-6240	Foz do Cubatão	2.7	0.25	Aquatic-compatible proxy	62	-25.55	-24.72	0.8	hydroxy FA candidates*; resin diterpenoid markers*
TRD_492	FC-3651	Foz do Cubatão	3.1		No APAA proxy	77	-26.19	-28.26	-2.07	resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_493	FC-5704	Foz do Cubatão			No APAA proxy	72	-20.5	-21.8	-1.29	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_494	FC-6964	Foz do Cubatão	1.2	0.32	Aquatic-compatible proxy	80.8	-22.81	-24.37	-1.56	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_495	FC-5765	Foz do Cubatão			No APAA proxy	47	-19.23	-21.36	-2.14	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_496	FC-2775	Foz do Cubatão	6.5		Plant-processing proxy	77.3	-24.36	-25.52	-1.16	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_497	FC-6018	Foz do Cubatão			No APAA proxy		-21.48	-26.42	-4.93	hydroxy FA candidates*; resin diterpenoid markers*
TRD_498	FC-2428	Foz do Cubatão	4.1		Plant-processing proxy	75.3	-23.6	-22.53	1.08	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_499	FC-2453	Foz do Cubatão			No APAA proxy	87.1	-23.3	-22.78	0.52	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_500	FC-5758	Foz do Cubatão			No APAA proxy	44.3	-23.49	-25.34	-1.85	resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_501	FC-3358	Foz do Cubatão	2		No APAA proxy	62.4	-21.69	-24.28	-2.59	resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_502	FC-5824	Foz do Cubatão	6.5	0.05	Plant-processing proxy	69.8	-24.27	-26.78	-2.51	
TRD_503	FC-SN003	Foz do Cubatão	2.6	0.15	Aquatic-compatible proxy	72.7	-22.81	-22.79	0.02	
TRD_504	FC-339	Foz do Cubatão	12.6		Plant-processing proxy	51.8	-25.32	-24.82	0.5	
TRD_505	FC-3906	Foz do Cubatão			No APAA proxy	56.6	-22.41	-22.3	0.11	
TRD_1223	AT02-311	Arroio Tamarina 2			No APAA proxy	47	-25.48	-26.73	-1.26	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1329	AT02-232	Arroio Tamarina 2			No APAA proxy	47	-27.98	-30.81	-2.82	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1330	AT02-233	Arroio Tamarina 2			No APAA proxy	39.9	-22.95	-23.52	-0.57	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1331	AT02-315	Arroio Tamarina 2			No APAA proxy	54.9	-28.02	-29.68	-1.66	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1332	AT02-268	Arroio Tamarina 2			No APAA proxy	50.6	-27.95	-28.38	-0.43	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1333	AT02-302	Arroio Tamarina 2			No APAA proxy	71.4	-27.96	-29.84	-1.88	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*
TRD_1334	AT02-231	Arroio Tamarina 2			No APAA proxy	57.3	-27.79	-28.38	-0.59	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_1337	AT02-310	Arroio Tamarina 2			No APAA proxy	55.2	-27.47	-30.16	-2.69	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1338	AT02-270	Arroio Tamarina 2			No APAA proxy	52.5	-19.68	-29.99	-10.31	hydroxy FA candidates*; resin diterpenoid markers*; phorbol-like markers*
TRD_1339	AT02-277	Arroio Tamarina 2			No APAA proxy	55.7	-27.47	-30.16	-2.69	MAGs*; resin diterpenoid markers*; oxidised resin diterpenoid markers*; phorbol-like markers*
TRD_1340	AT02-304	Arroio Tamarina 2			No APAA proxy	43.2	-27.74	-29.56	-1.82	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1341	AT02-230	Arroio Tamarina 2			No APAA proxy	86.9	-23.21	-26.05	-2.84	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1343	AT02-309	Arroio Tamarina 2			No APAA proxy	43.8	-25.3	-27.32	-2.02	MAGs*; DAGs*; hydroxy FA candidates*; resin diterpenoid markers*
TRD_1344	AT02-346	Arroio Tamarina 2			No APAA proxy	51.9	-26.66	-28.17	-1.51	resin diterpenoid markers*
TRD_1345	AT02-286	Arroio Tamarina 2	2.12	0.08	Aquatic-compatible proxy	51.9	-27.6	-30.04	-2.44	
TRD_1346	AT02-307	Arroio Tamarina 2			No APAA proxy	51.2	-29.12	-30.16	-1.04	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1347	AT02-291	Arroio Tamarina 2	0.7		No APAA proxy	57.5	-19	-19.7	-0.7	resin diterpenoid markers*; phorbol-like markers*
TRD_1348	AT02-272	Arroio Tamarina 2			No APAA proxy	74.8	-27.5	-29.35	-1.85	MAGs*; resin diterpenoid markers*
TRD_1350	AT02-400	Arroio Tamarina 2	5.3		Plant-processing proxy	56.8	-26.85	-27.56	-0.71	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1351	AT02-318	Arroio Tamarina 2			No APAA proxy	41.5	-27.06	-30.54	-3.48	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1352	AT02-273	Arroio Tamarina 2			No APAA proxy	62.9	-24.45	-29.11	-4.66	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1353	AT02-375	Arroio Tamarina 2			No APAA proxy	44.8	-28.87	-29.27	-0.4	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1354	AT02-260	Arroio Tamarina 2			No APAA proxy	66.4	-29.04	-29.58	-0.54	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1355	AT02-293	Arroio Tamarina 2			No APAA proxy	57.3	-26.88	-27.81	-0.93	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1356	AT02-313	Arroio Tamarina 2			No APAA proxy	48.7	-27.05	-27.66	-0.61	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_1357	AT02-328	Arroio Tamarina 2	2.85	0.15	Aquatic-compatible proxy	57.5	-27.5	-28.99	-1.49	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1358	AT02-384	Arroio Tamarina 2			No APAA proxy	71.7	-24.56	-27.06	-2.5	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*

TRD_1359	AT02-378	Arroio Tamarina 2			No APAA proxy	71.7	-22.21	-23.15	-0.94	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1360	AT02-377	Arroio Tamarina 2			No APAA proxy	72.7	-27.33	-29.79	-2.46	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1361	AT02-1361	Arroio Tamarina 2	1.98	0.45	Aquatic-compatible proxy	56.4	-26.77	-28.96	-2.19	hydroxy FA candidates*; resin diterpenoid markers*
TRD_1362	AT02-370	Arroio Tamarina 2	0.18		No APAA proxy	56.9	-28.25	-29.79	-1.54	MAGs*; resin diterpenoid markers*; phorbol-like markers*
TRD_1363	AT02-388	Arroio Tamarina 2			No APAA proxy	74.9	-26.01	-28.33	-2.32	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1364	AT02-262	Arroio Tamarina 2	5.28	0.01	Plant-processing proxy	54.6	-28.25	-29.79	-1.54	MAGs*; hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1365	AT02-215	Arroio Tamarina 2			No APAA proxy	60.1	-24.91	-29.78	-4.87	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*
TRD_1366	AT02-330	Arroio Tamarina 2			No APAA proxy	60.1	-28.95	-30.12	-1.17	hydroxy FA candidates*; resin diterpenoid markers*

TRD_1367	AT02-217	Arroio Tamarina 2			No APAA proxy		-30.03	-30.51	-0.48	hydroxy FA candidates*; resin diterpenoid markers*; oxidised resin diterpenoid markers*; phorbol-like markers*

APPENDIX 1C: Babitonga Bay records not retained for AE-based interpretation

Records from the initial Babitonga ORA dataset that were not retained for AE-based molecular or isotopic interpretation. They are included here to document low-yield, non-interpretable or contamination-affected results as part of the analytical record.

TRD ID	Site	Final AE status	Reason / analytical note
TRD_468	Foz do Cubatão	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_474	Foz do Cubatão	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_482	Foz do Cubatão	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_490	Foz do Cubatão	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.

TRD_491	Foz do Cubatão	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1291	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1298	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1307	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1312	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1313	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1317	Morro Grande 1	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.

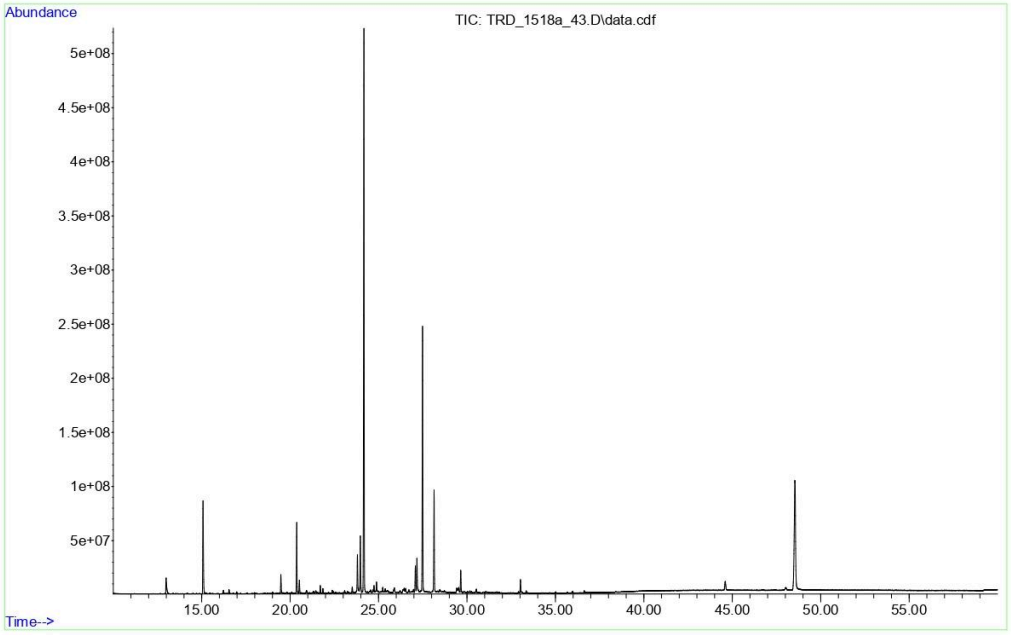
TRD_1475	Praia Grande - Unidade 21	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal.
TRD_1335	Arroio Tamarina 2	Not retained	AE extract excluded from molecular and isotopic interpretation because chromatographic contamination compromised reliable interpretation.
TRD_1336	Arroio Tamarina 2	Not retained	AE extract excluded from molecular and isotopic interpretation because chromatographic contamination compromised reliable interpretation.
TRD_1342	Arroio Tamarina 2	Not retained	AE extract excluded from molecular and isotopic interpretation because chromatographic contamination compromised reliable interpretation.
TRD_1349	Arroio Tamarina 2	Not retained	AE extract excluded from molecular and isotopic interpretation because chromatographic contamination compromised reliable interpretation.
TRD_1368	Arroio Tamarina 2	Not retained	AE extract excluded from molecular and isotopic interpretation because chromatographic contamination compromised reliable interpretation.

APPENDIX 2 - Chromatograms from the Babitonga Bay assemblages

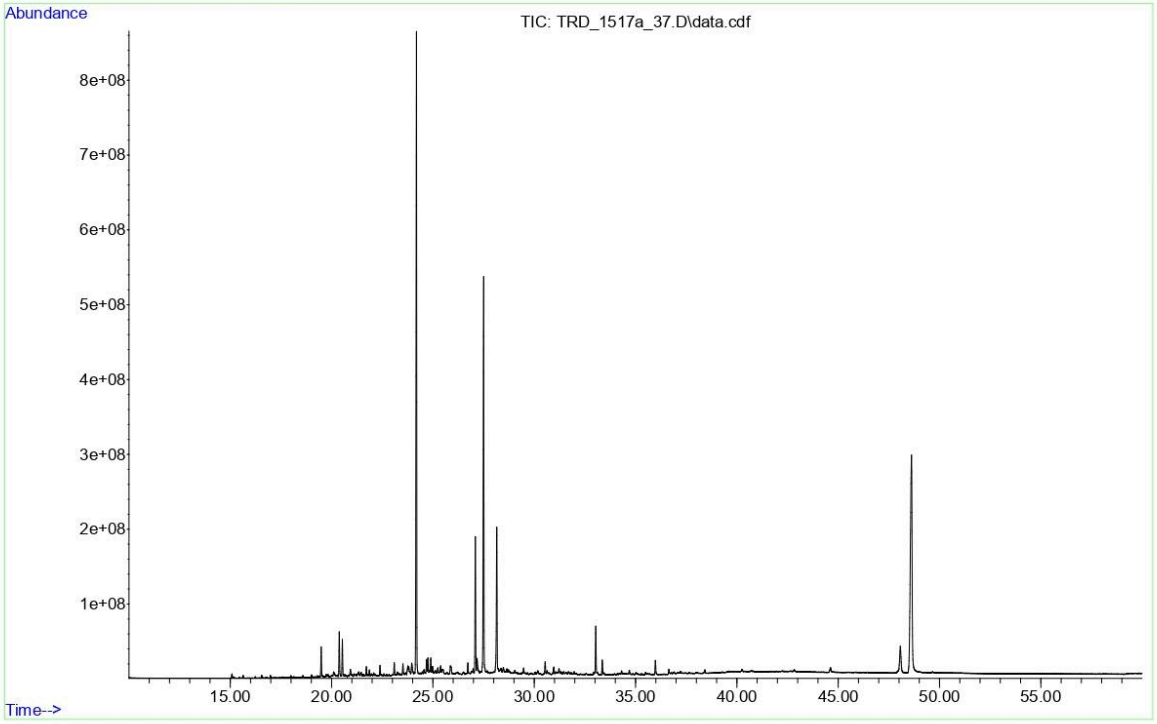
This appendix presents the available chromatograms from the Babitonga Bay ceramic assemblages analysed in Chapter 5. Chromatograms are organised by site and extraction method. Acidified methanol extraction chromatograms are presented separately from TLE/TMS chromatograms because the two methods contributed different forms of analytical information. Low-quality, low-yield and non-diagnostic chromatograms are retained where available as part of the analytical record and to document the limits of lipid preservation and recovery.

Praia Grande -Unidade 21

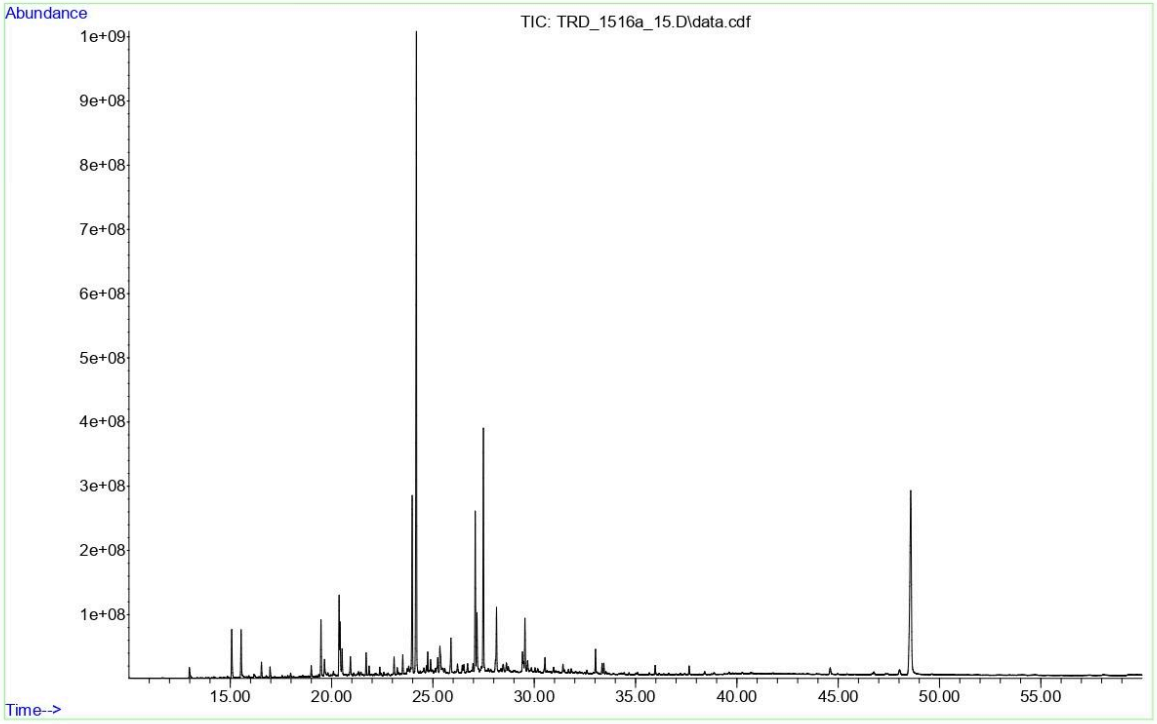
Acidified methanol extraction chromatograms



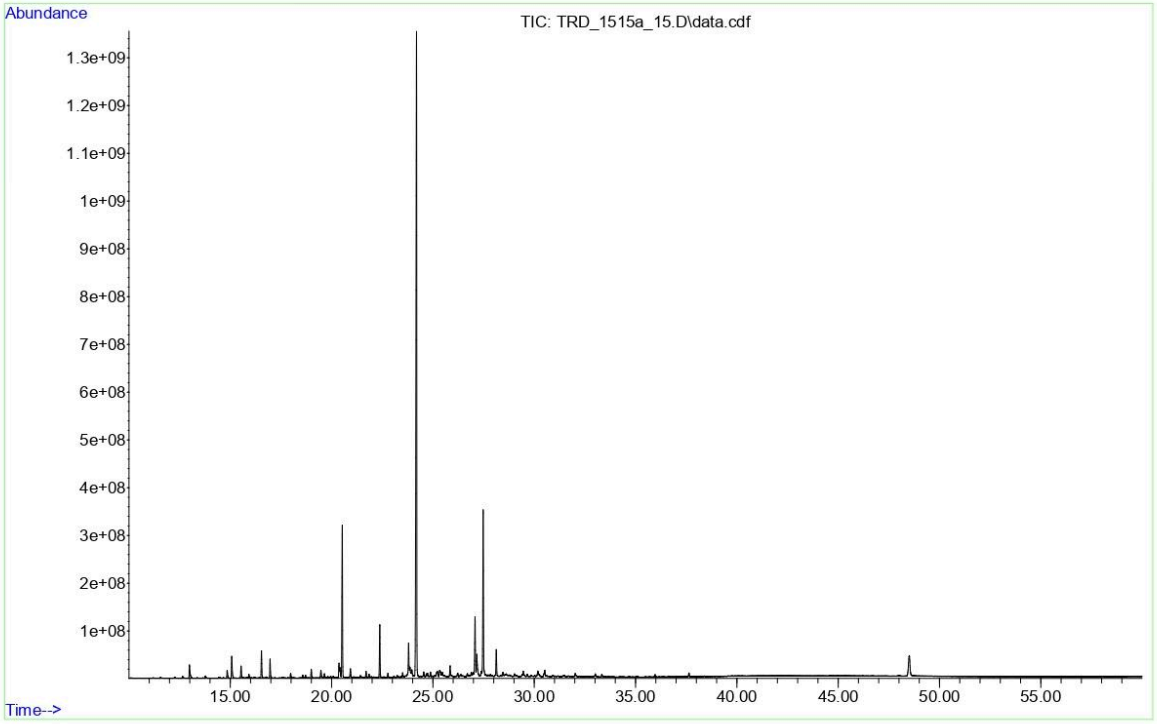
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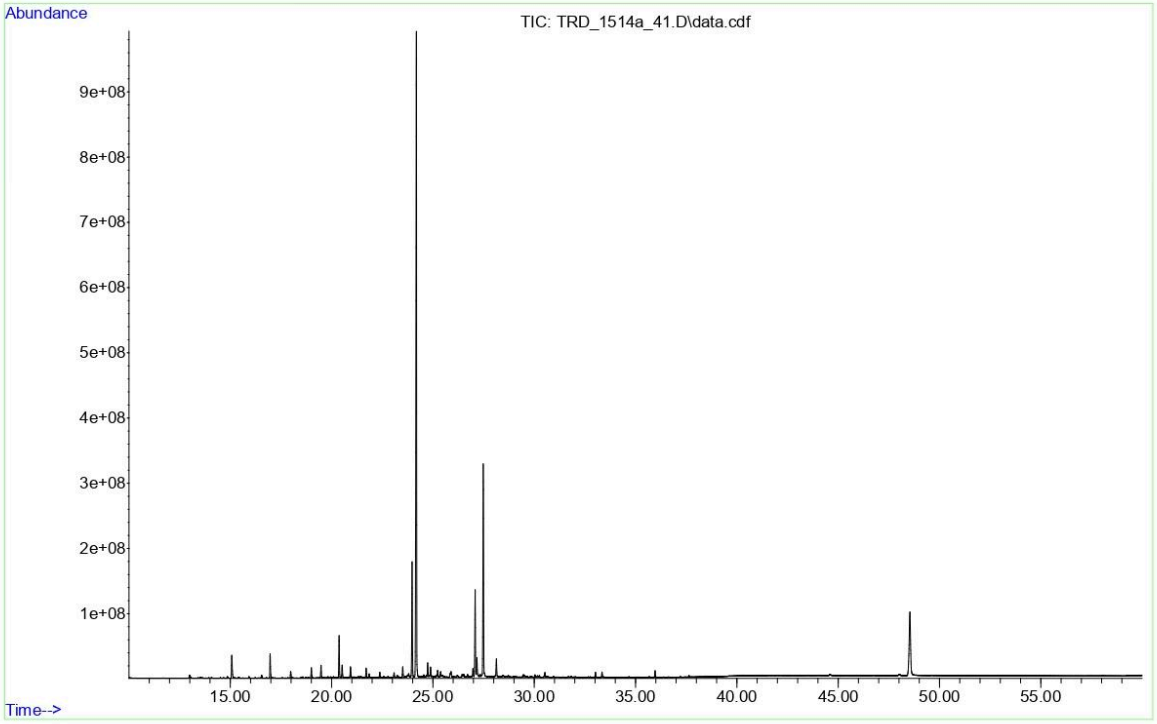
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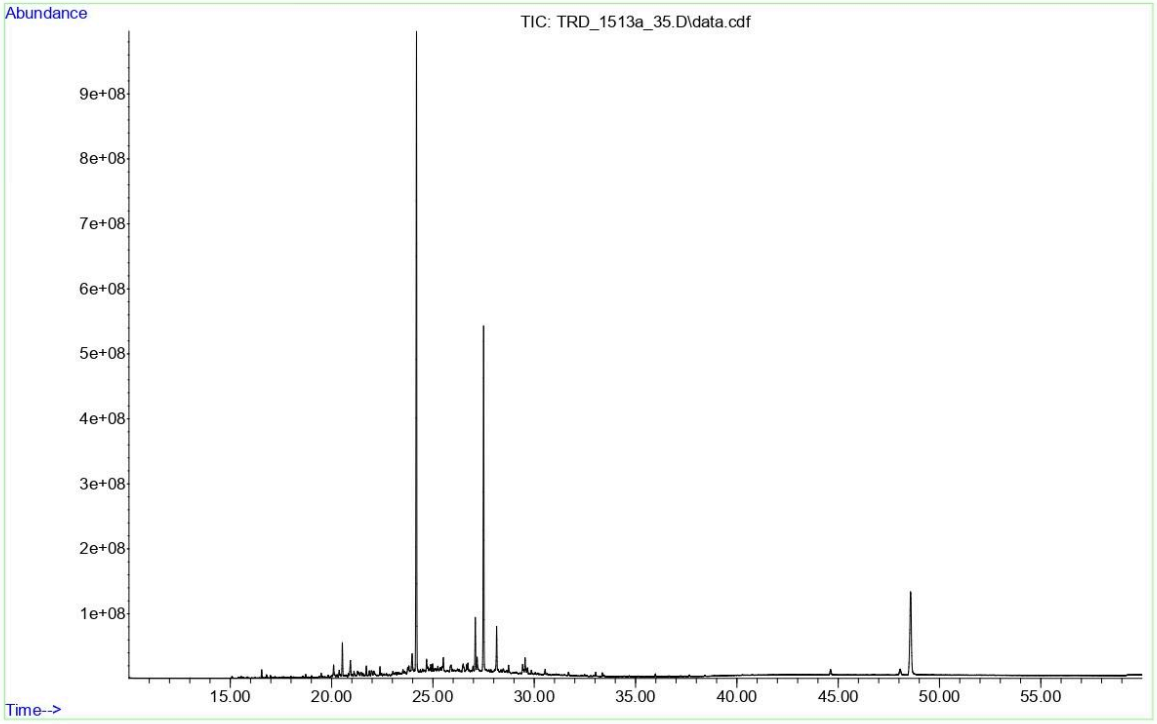
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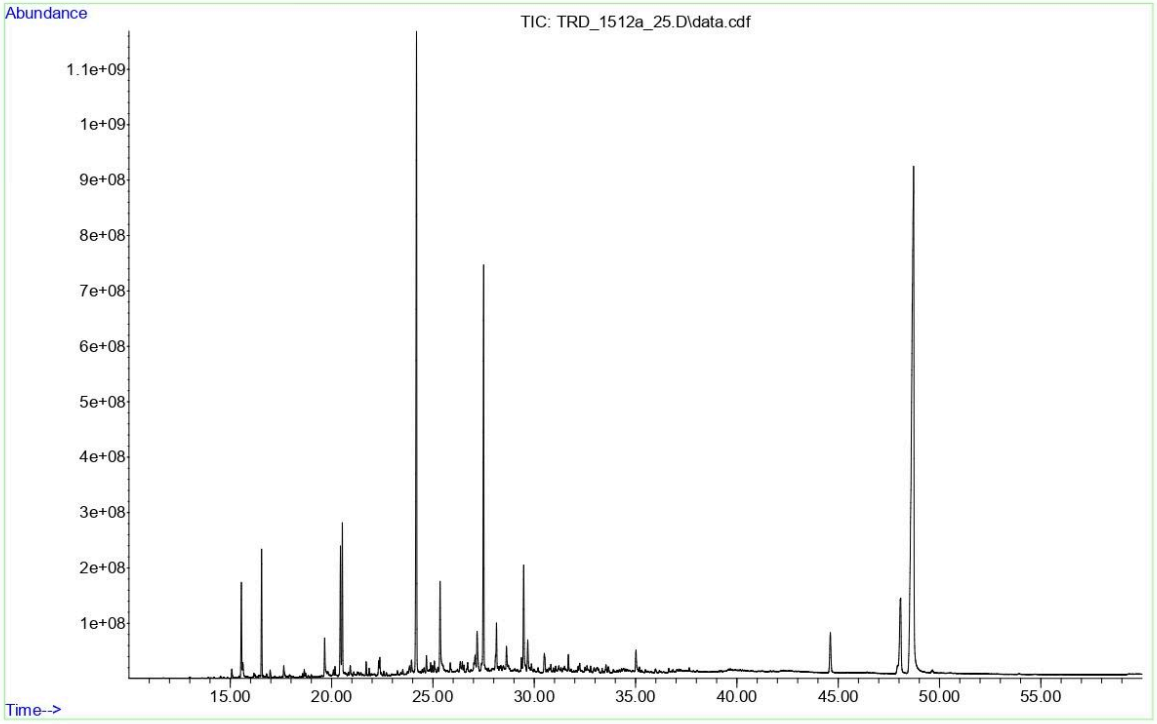
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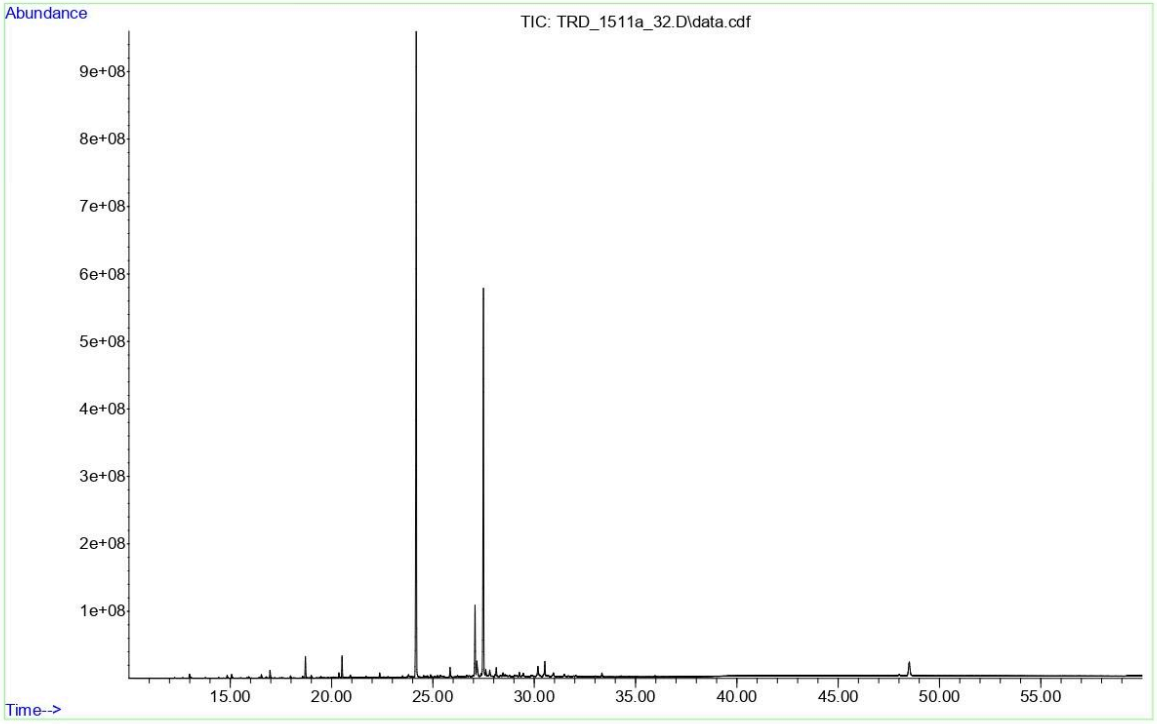
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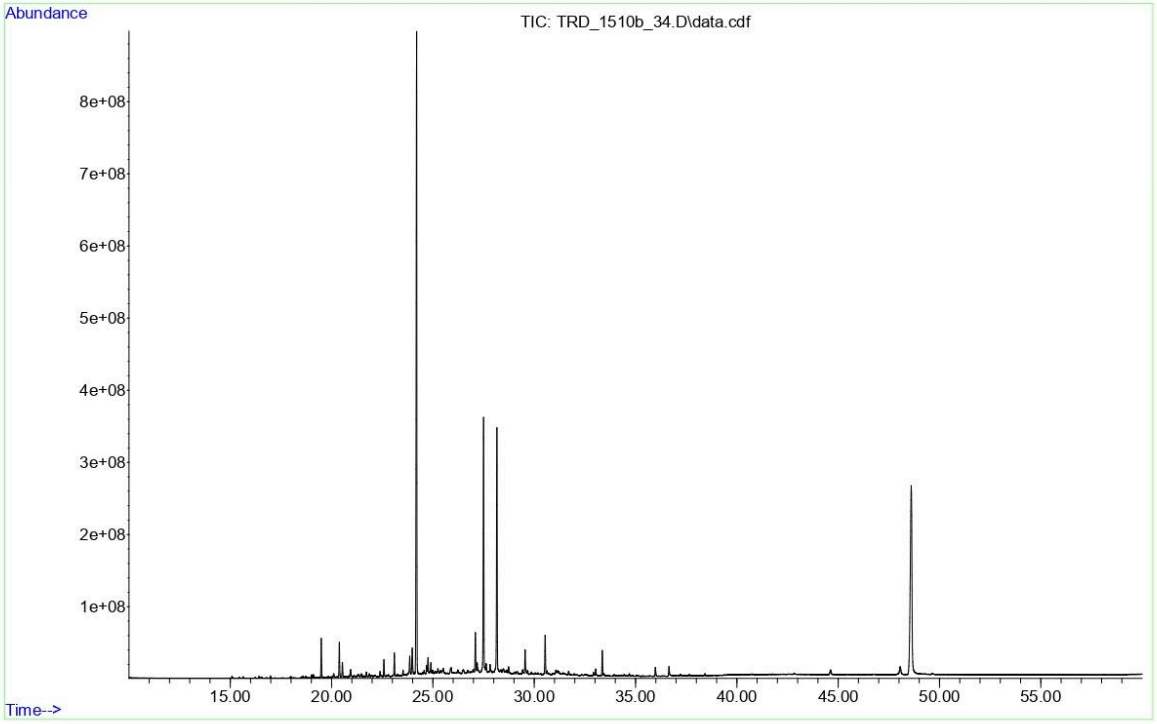
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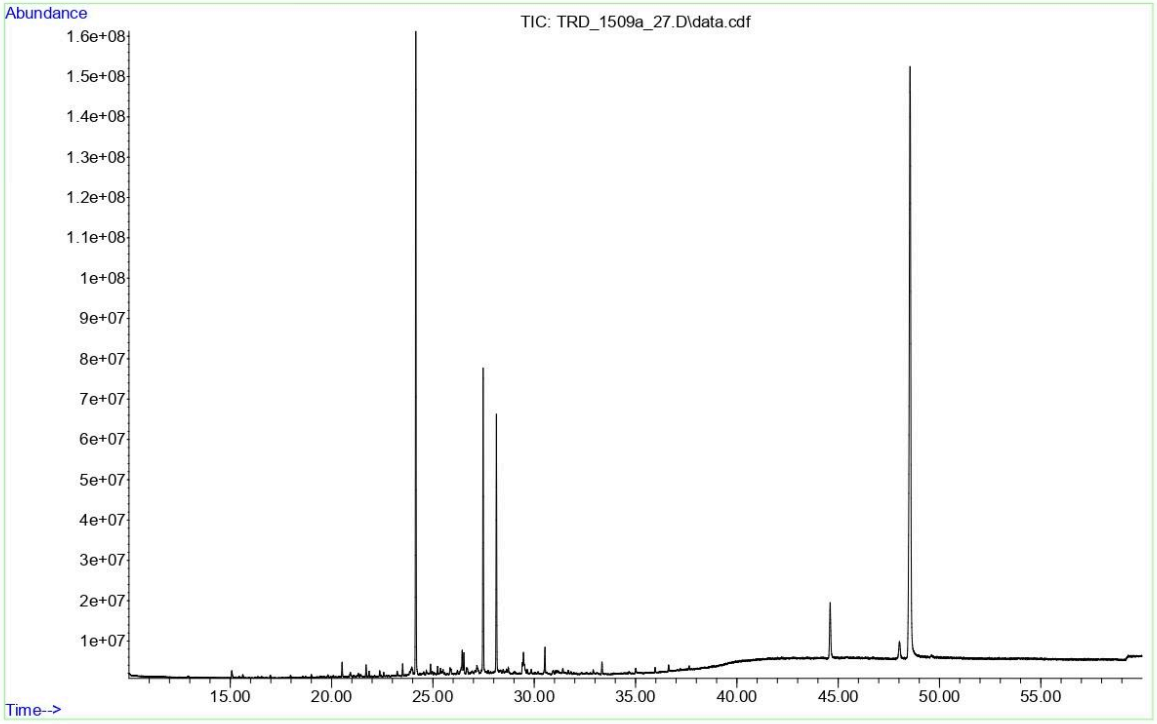
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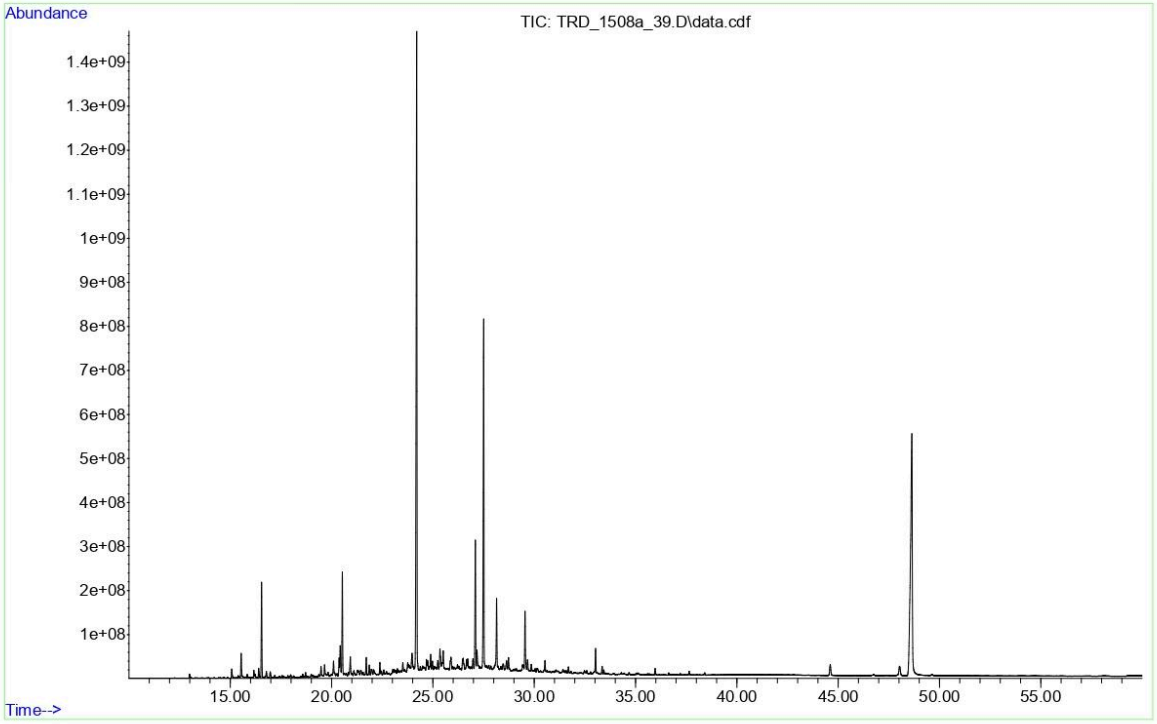
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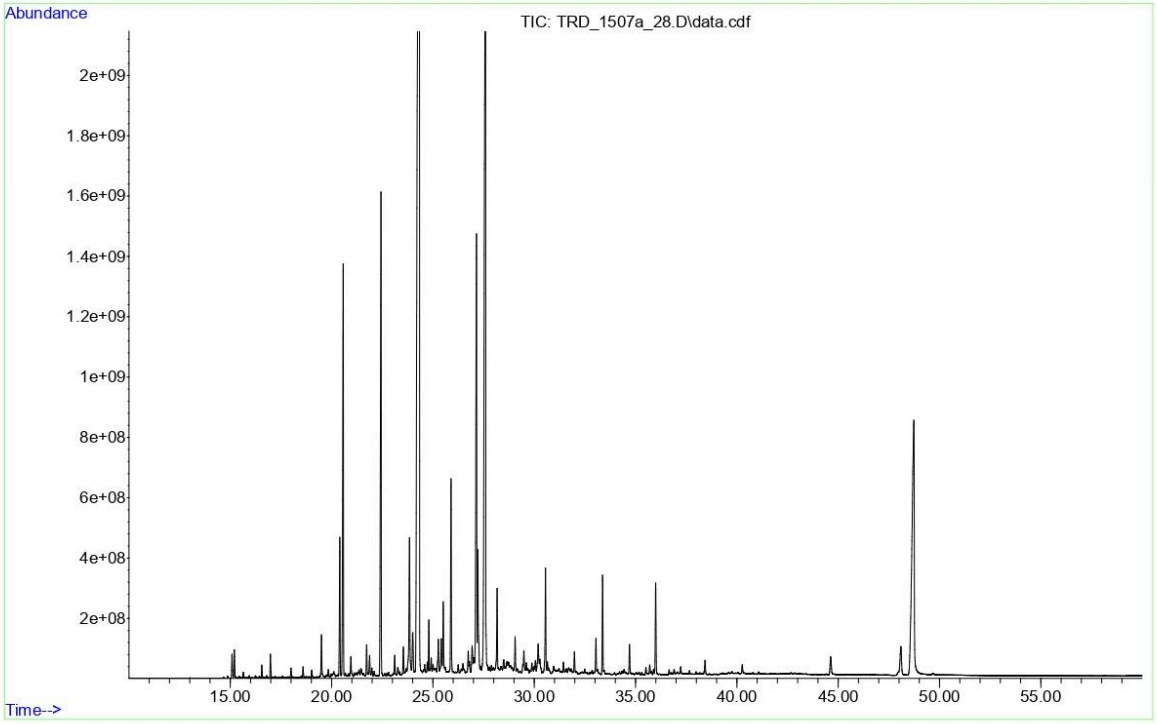
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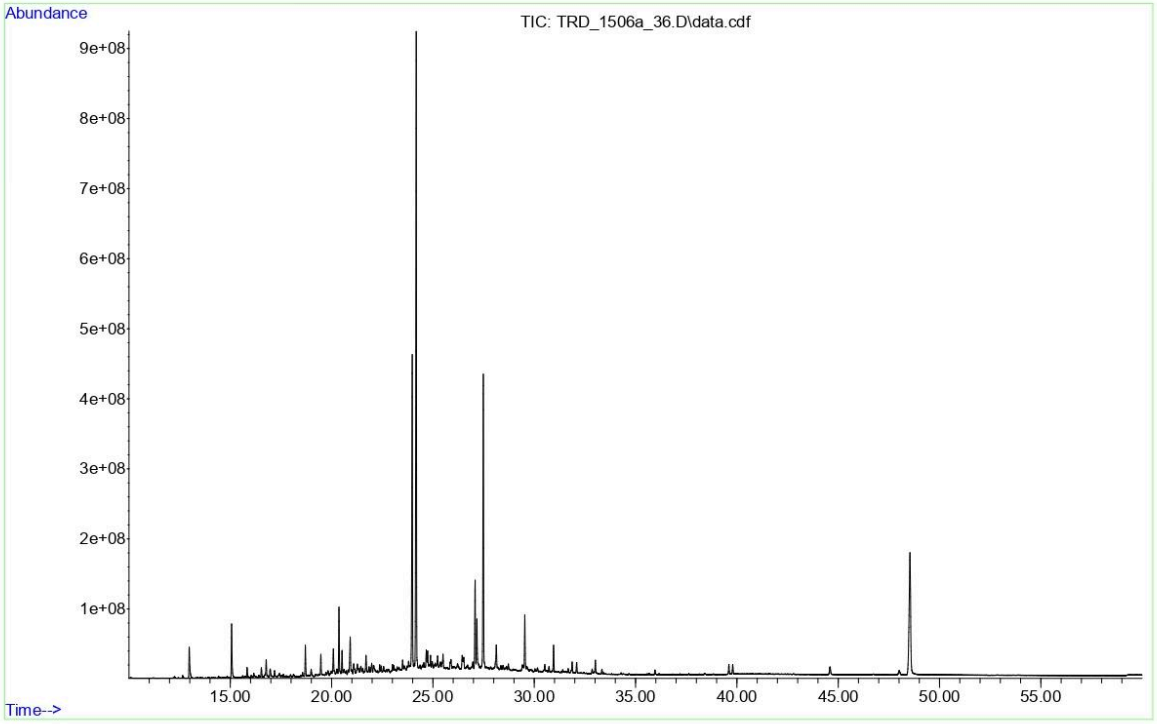
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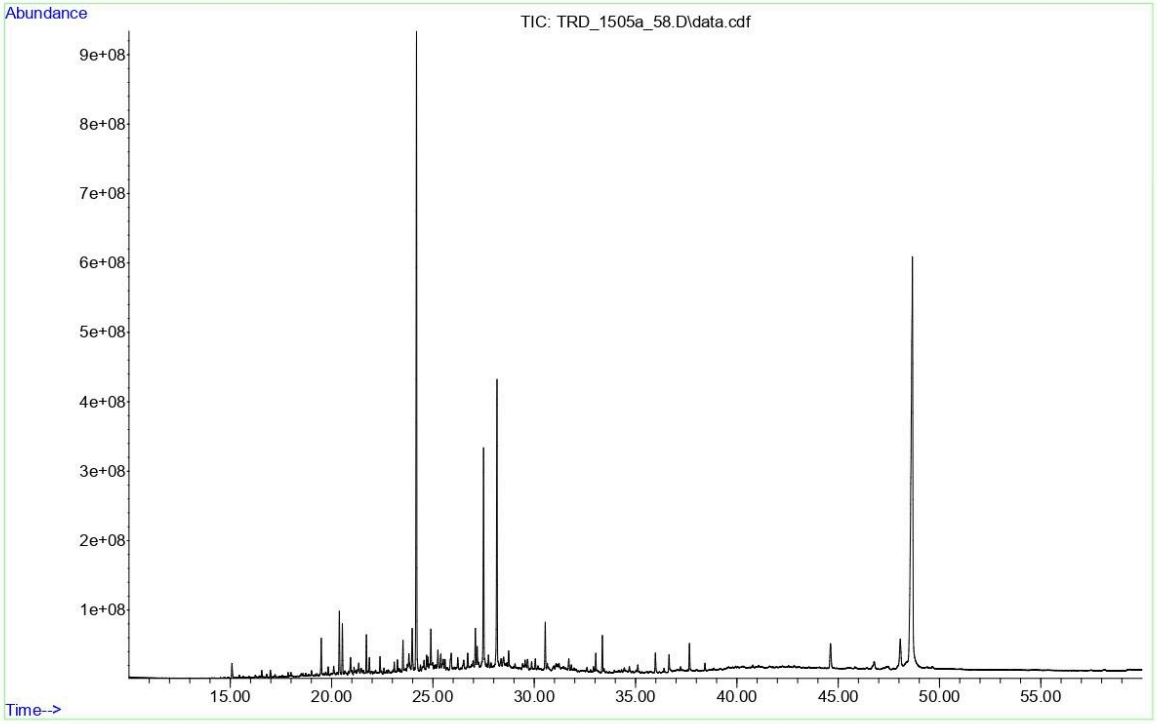
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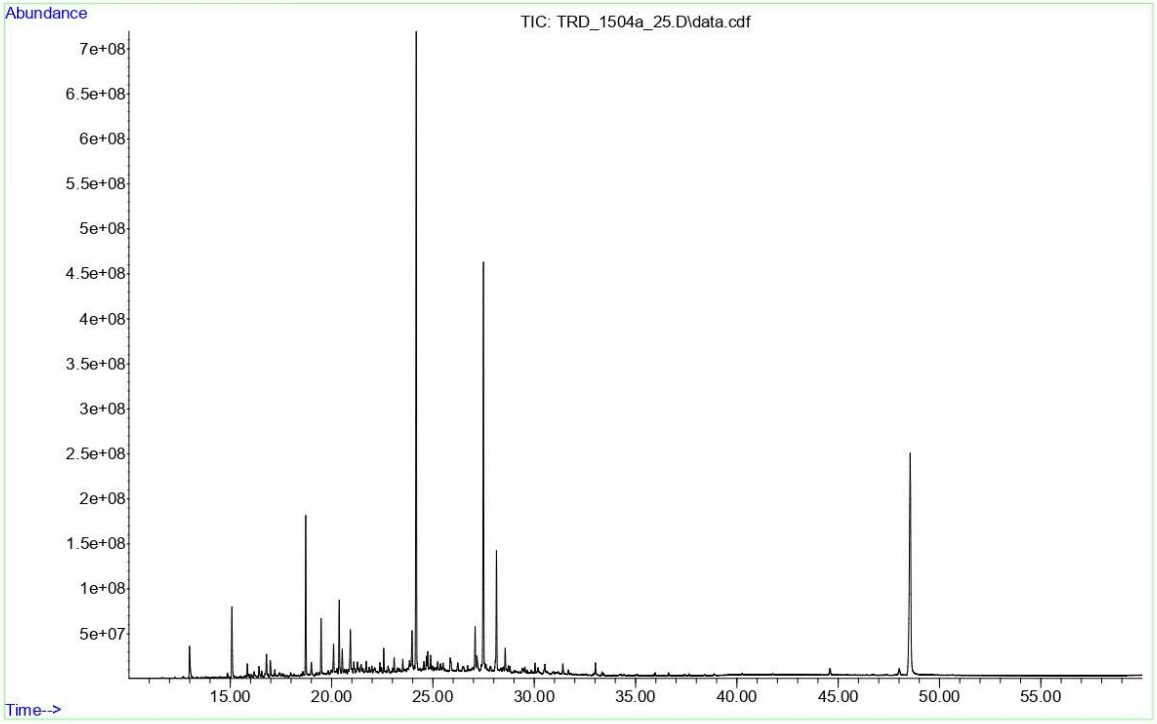
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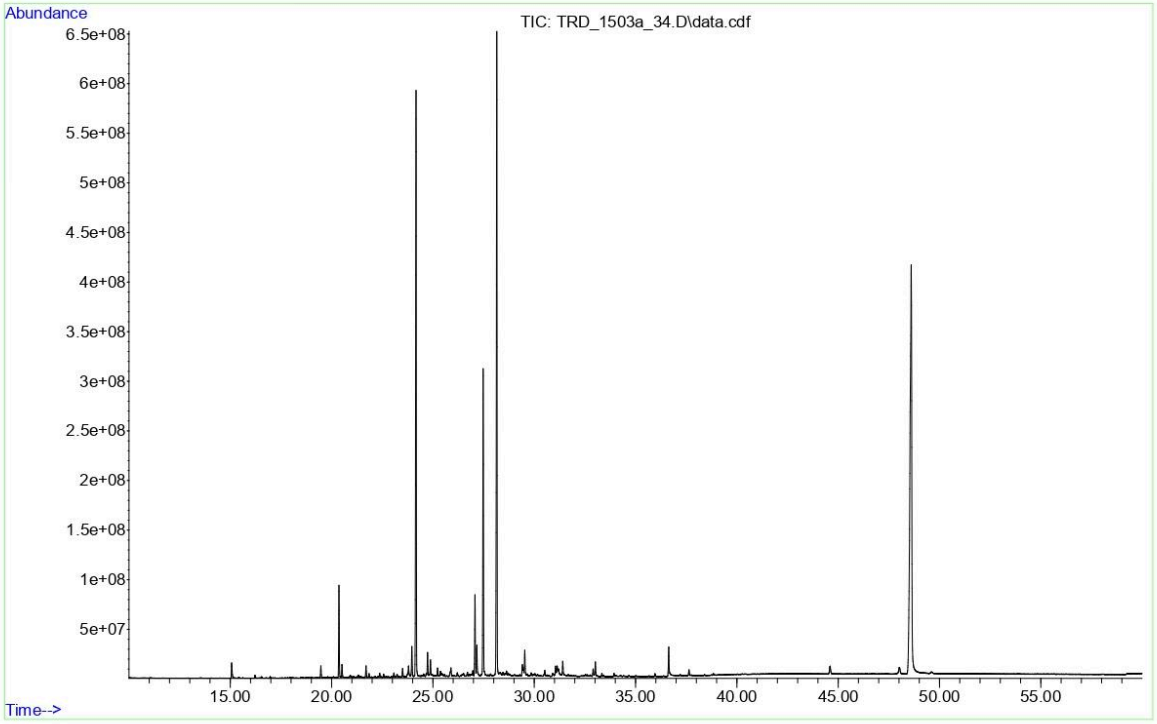
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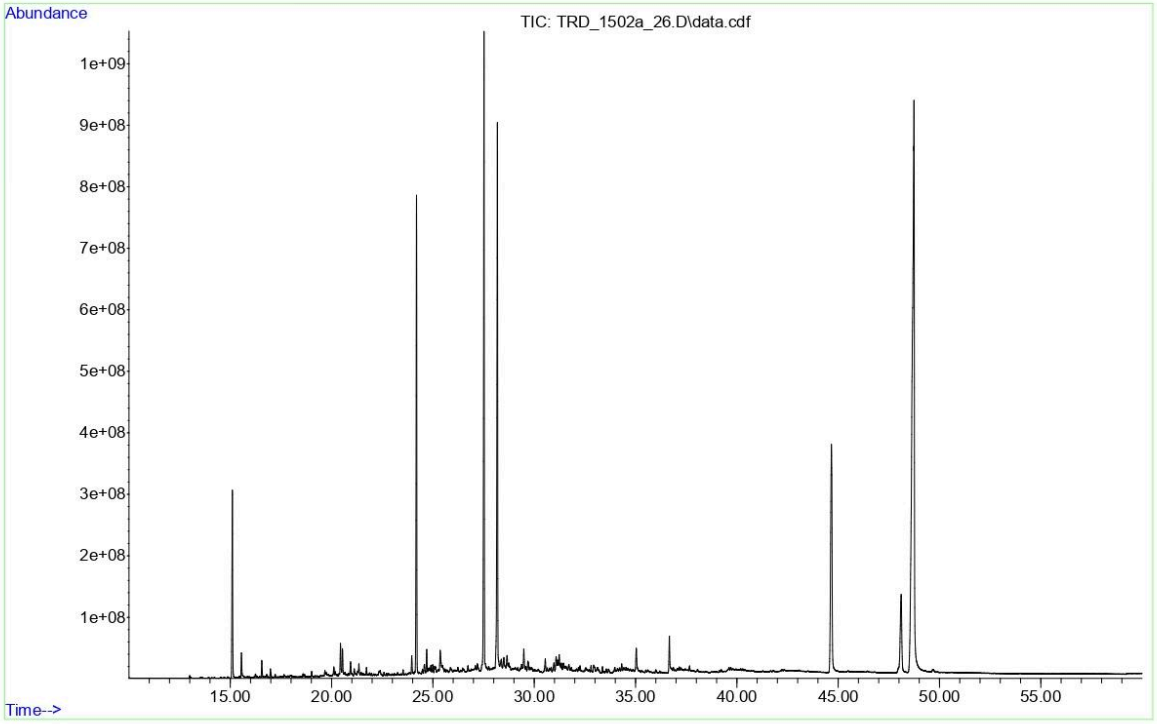
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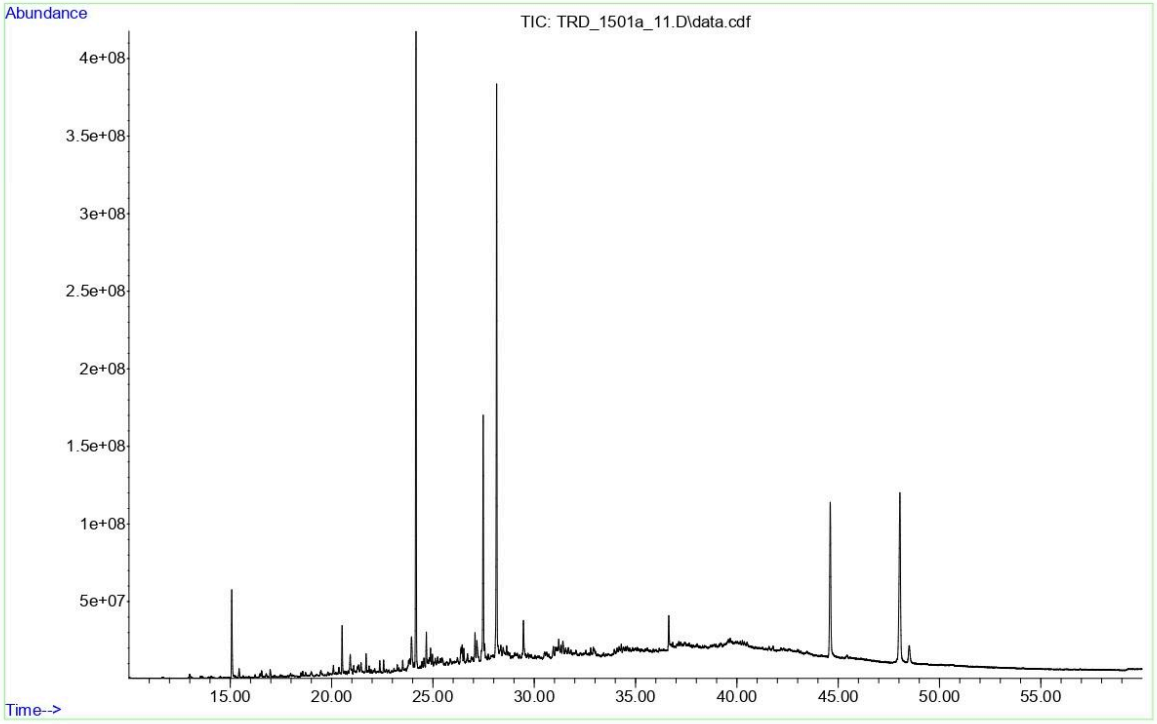
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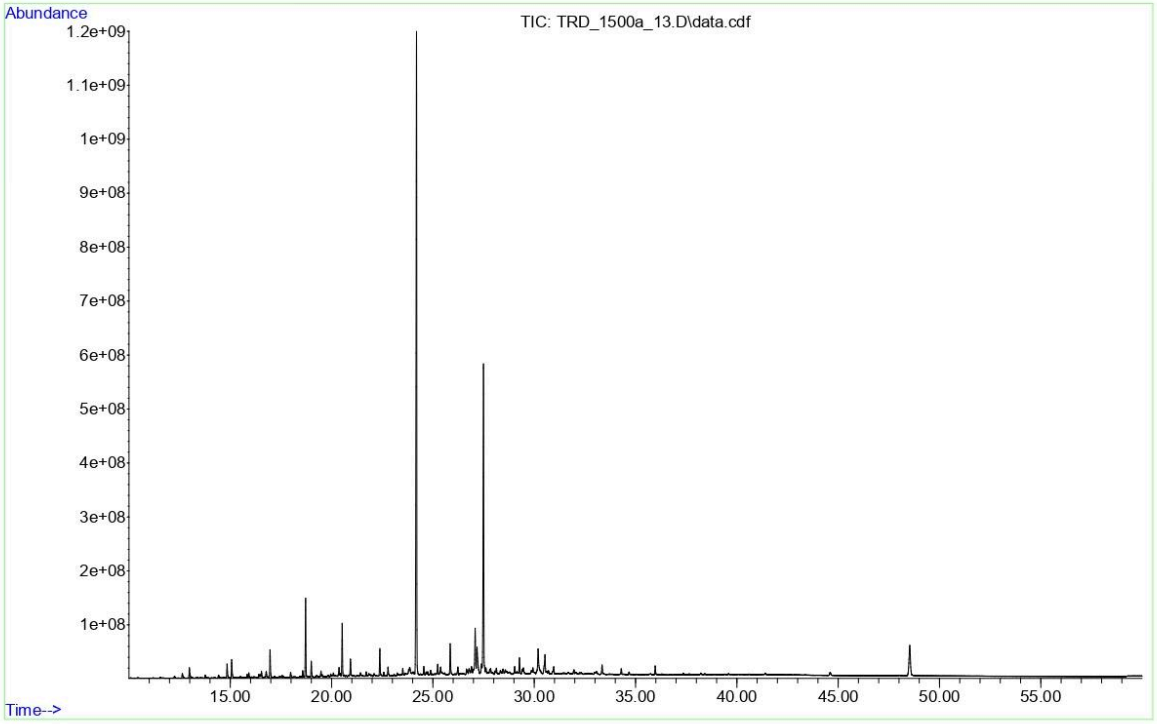
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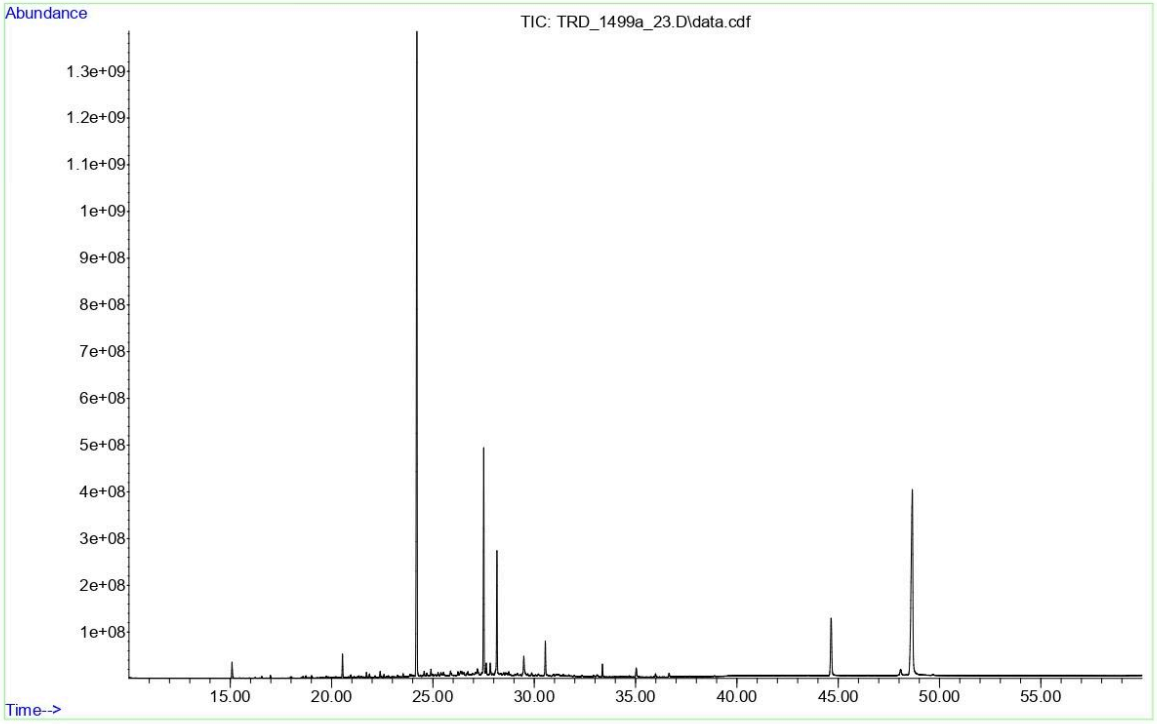
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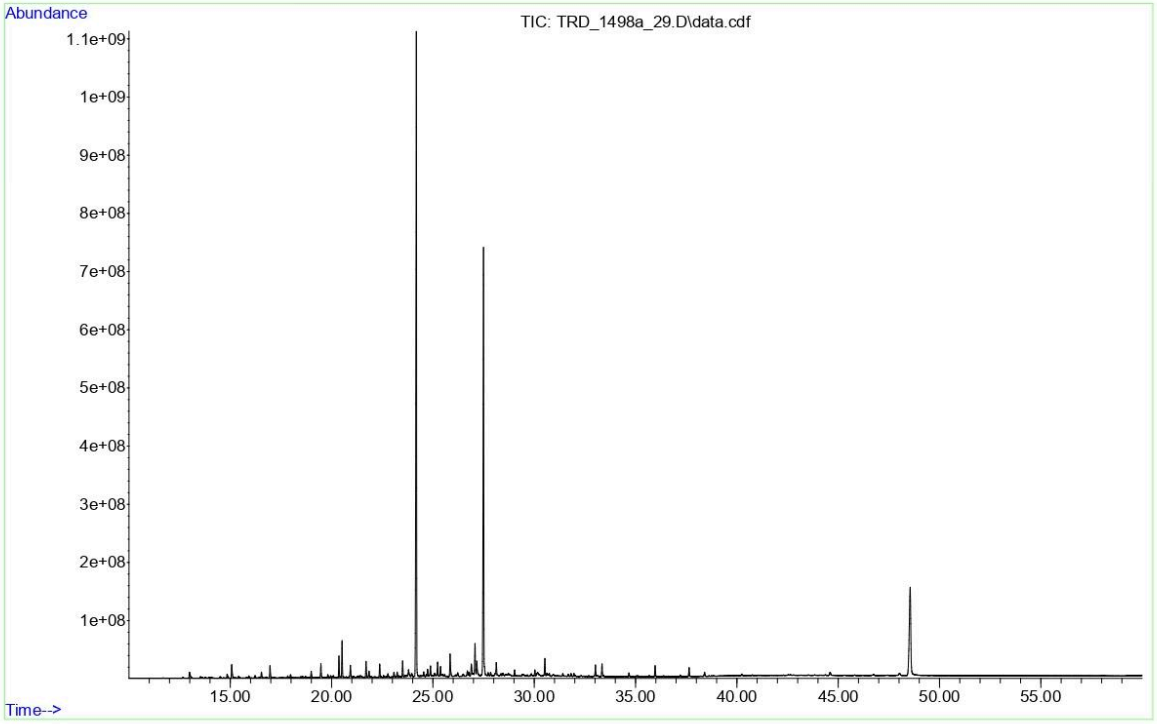
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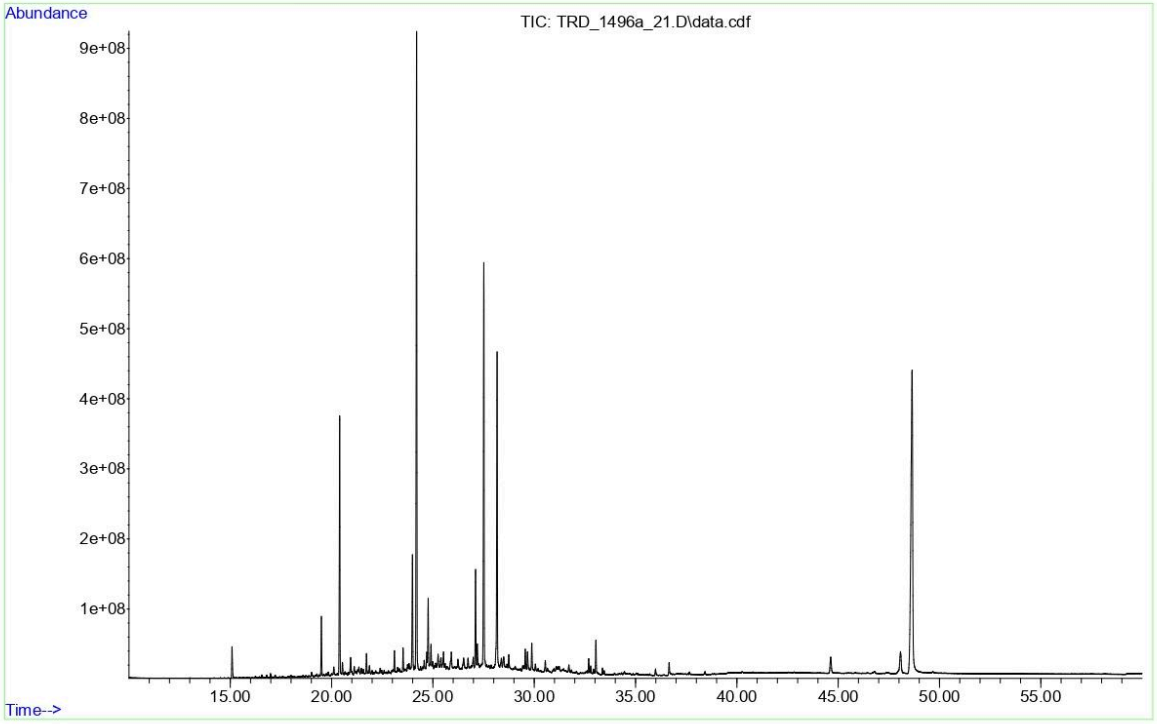
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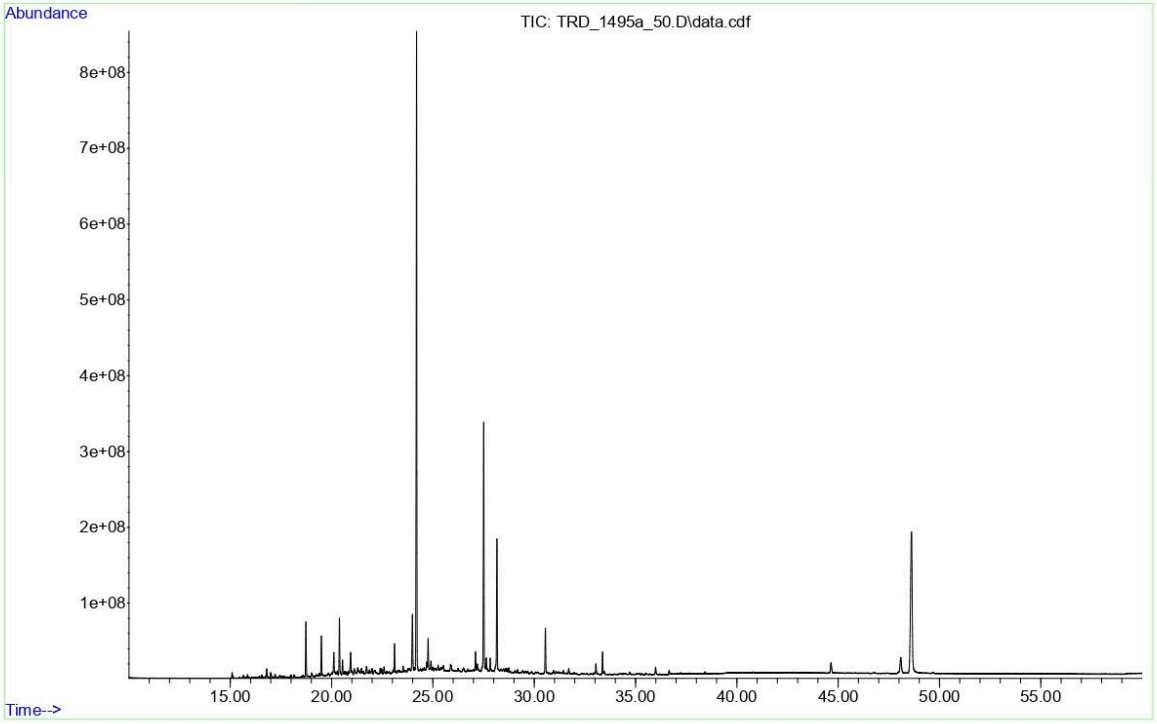
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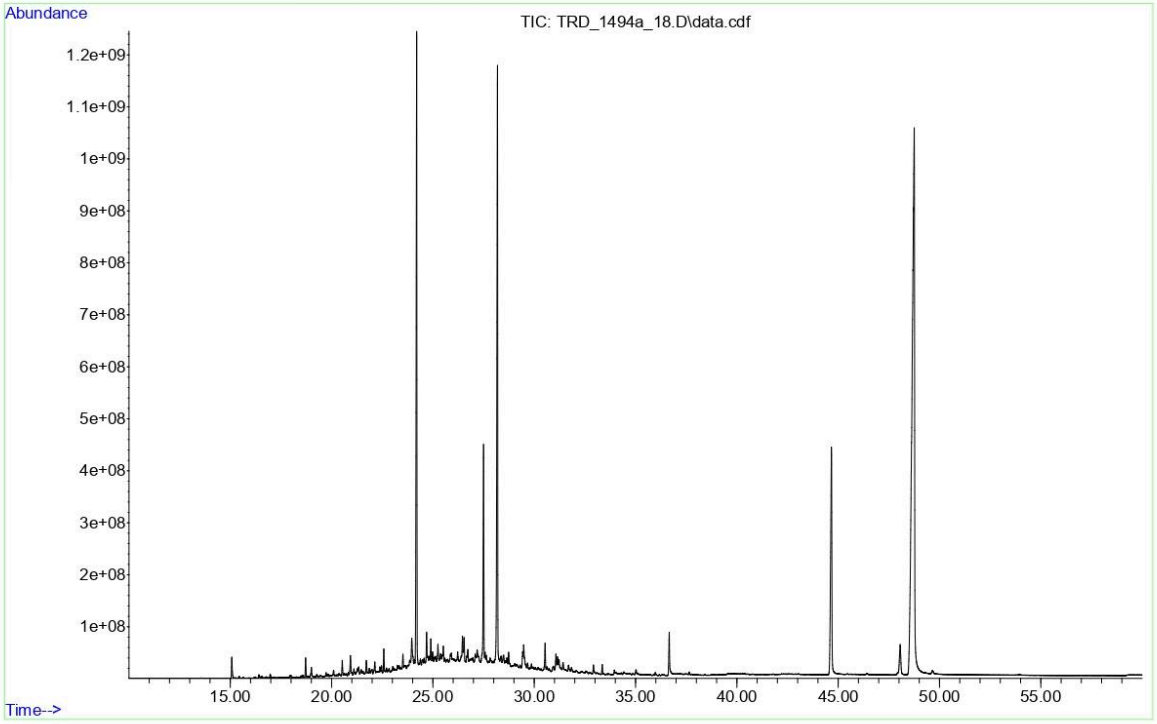
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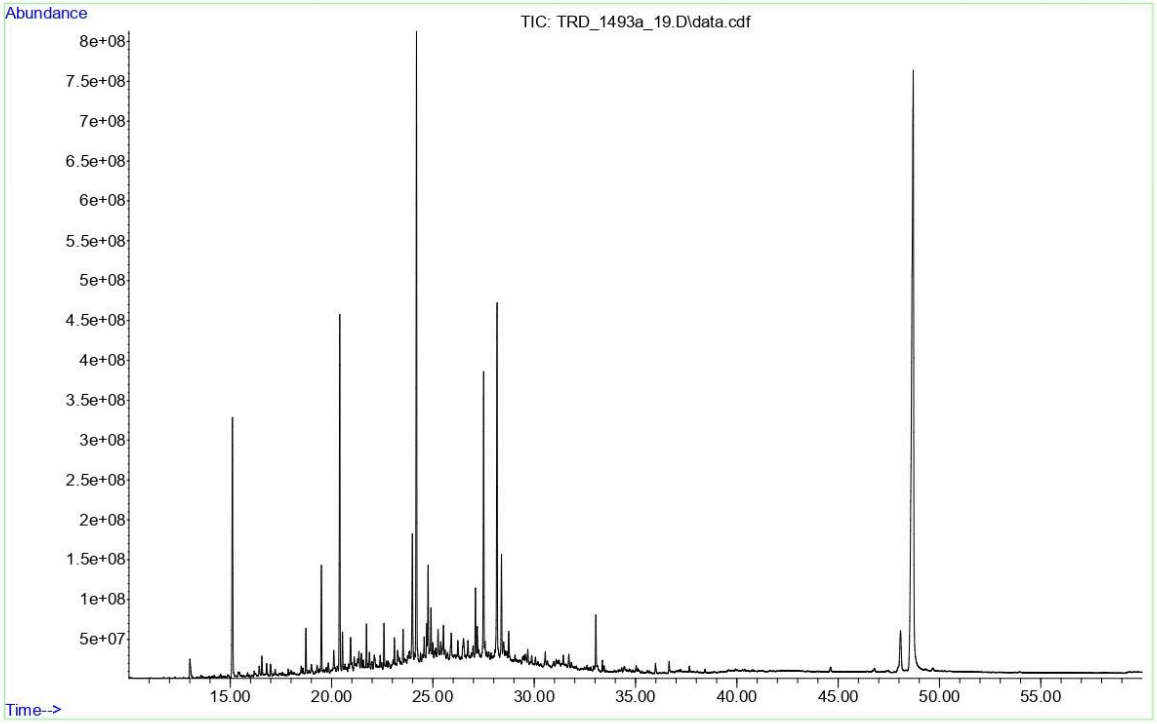
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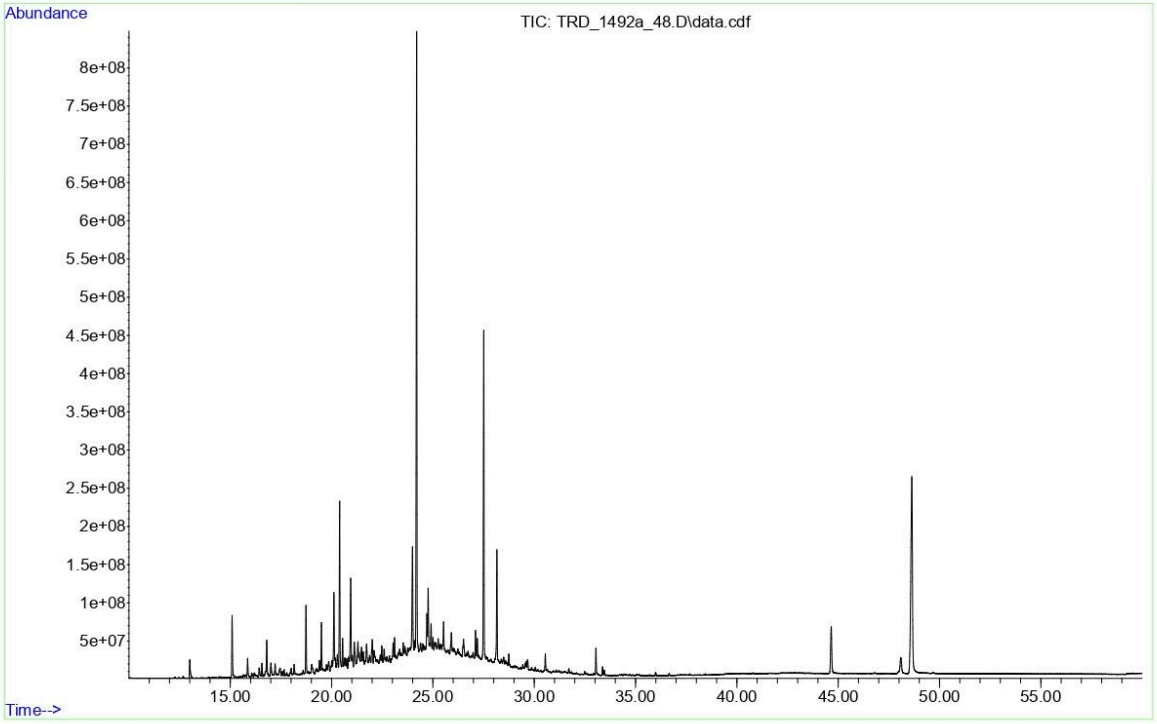
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Acquired : 20221122103636+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



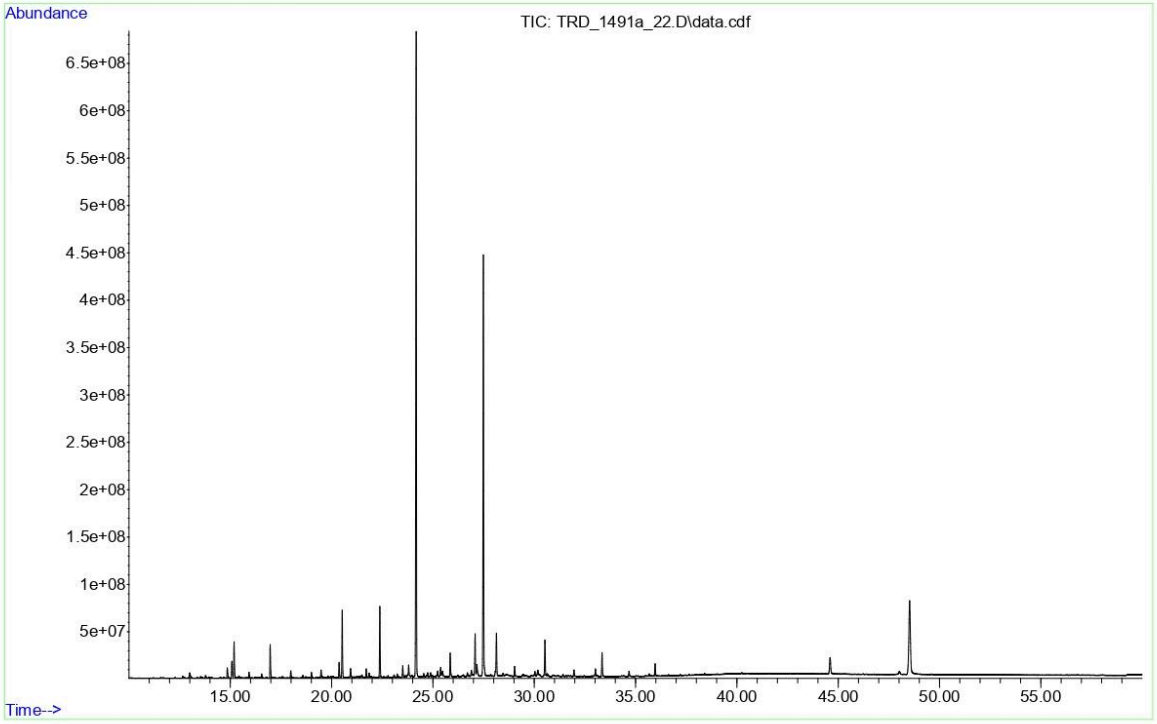
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1493a_19.D
Operator : SYSTEM
Acquired : 20221118090904+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



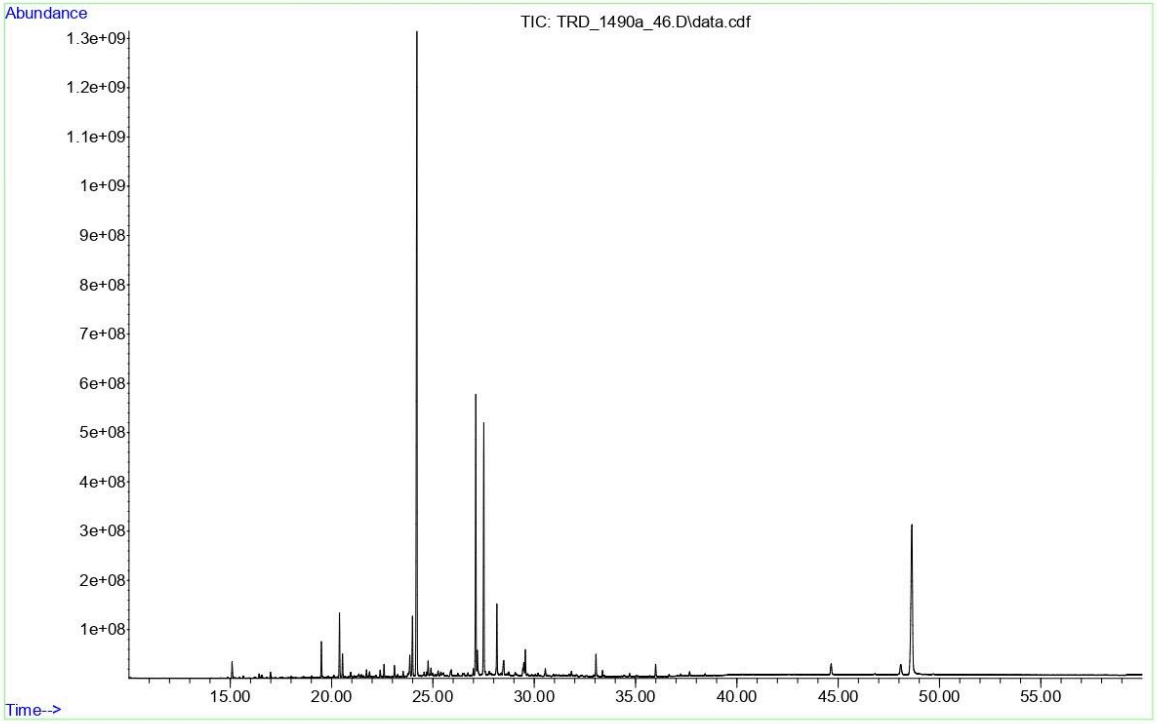
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Operator : SYSTEM
Acquired : 20221116212546+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



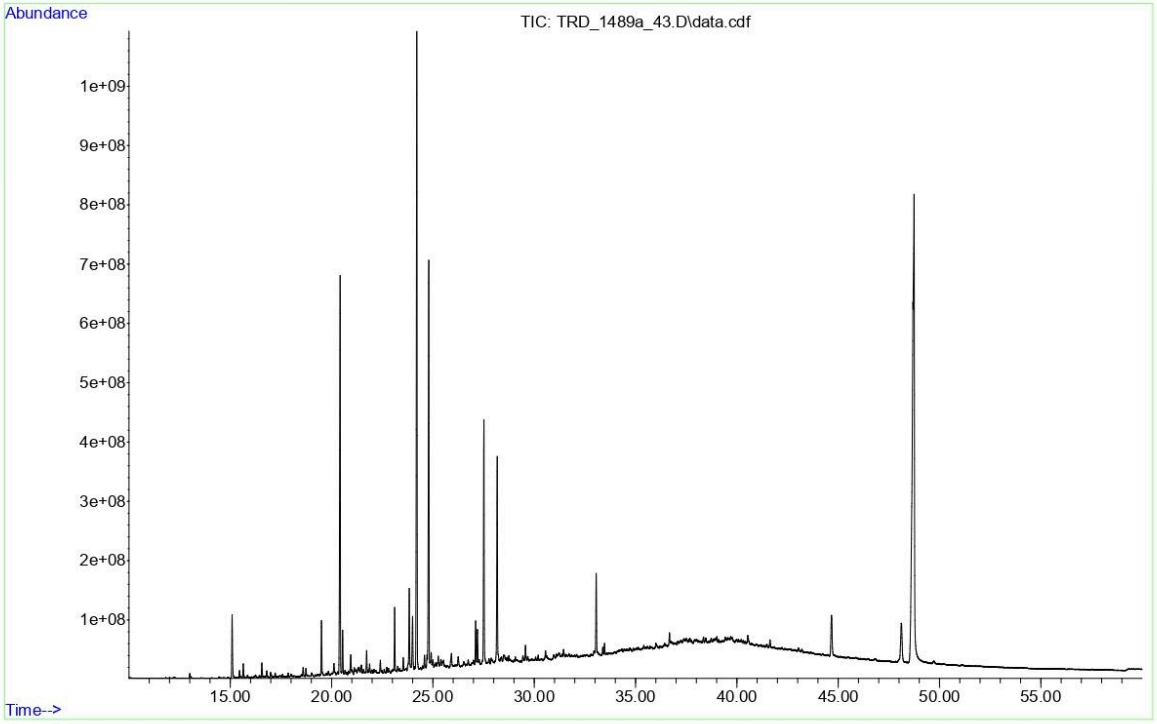
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1491a_22.D
Operator : SYSTEM
Acquired : 20221125121611+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



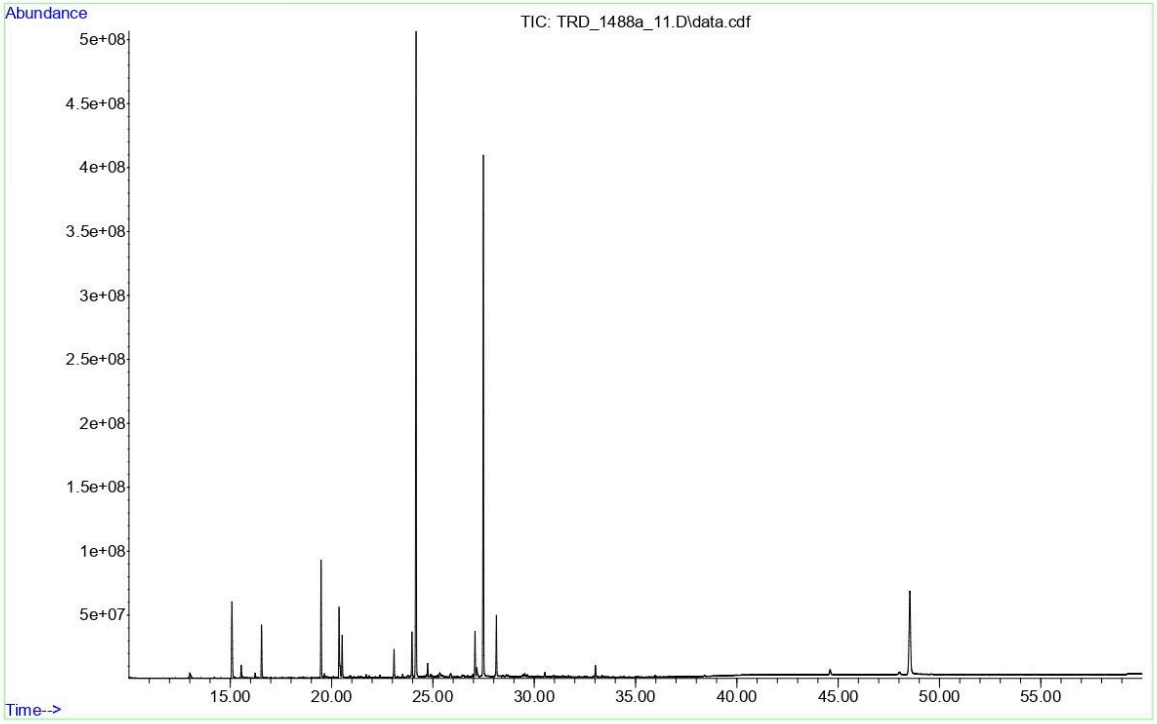
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1490a_46.D
Operator : SYSTEM
Acquired : 20221116190113+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



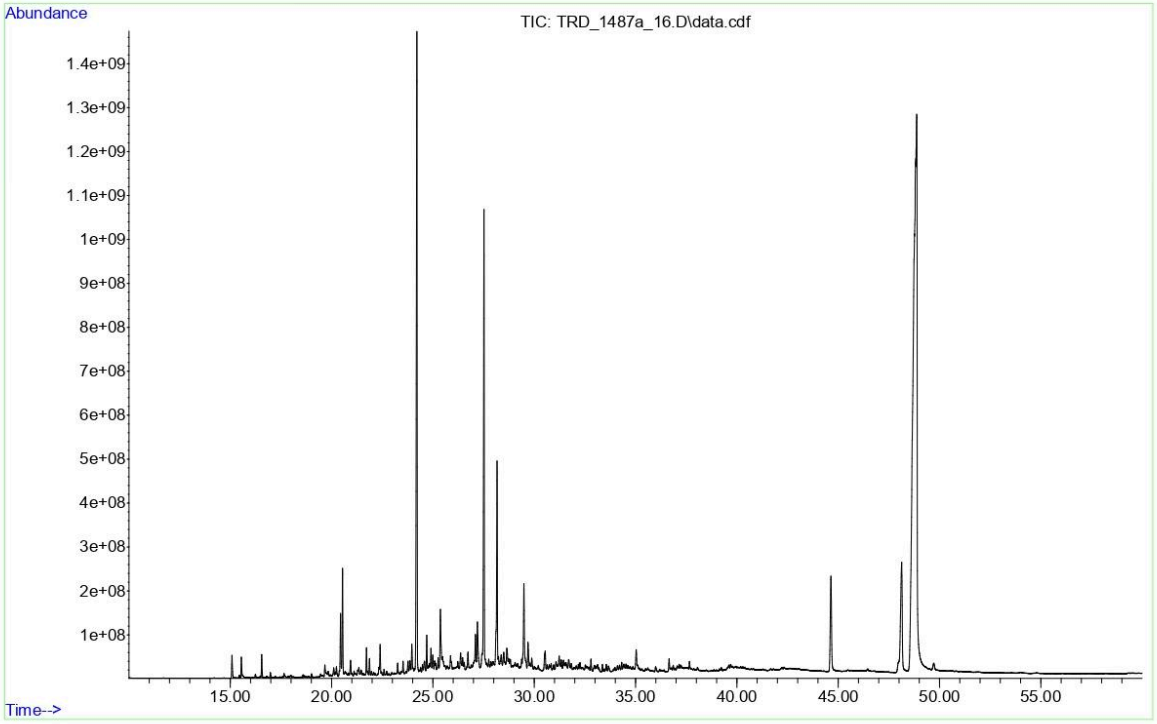
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1489a_43.D
Operator : SYSTEM
Acquired : 20221116152417+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



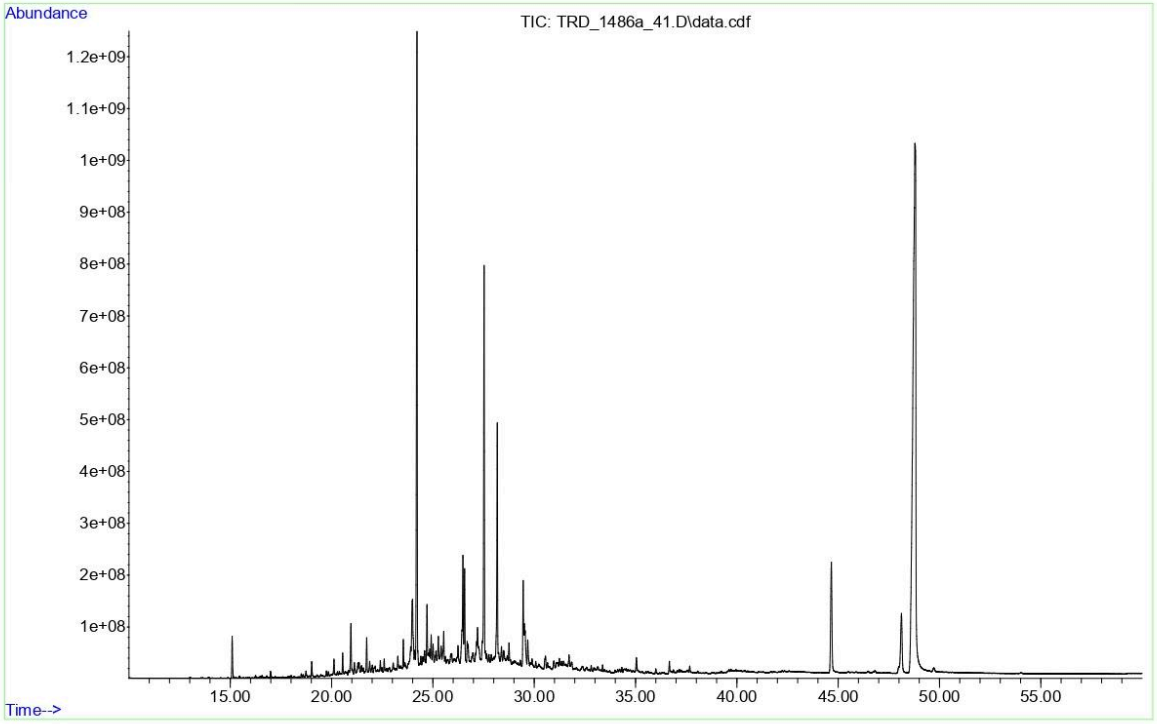
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1488a_11.D
Operator : SYSTEM
Acquired : 20221122021057+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



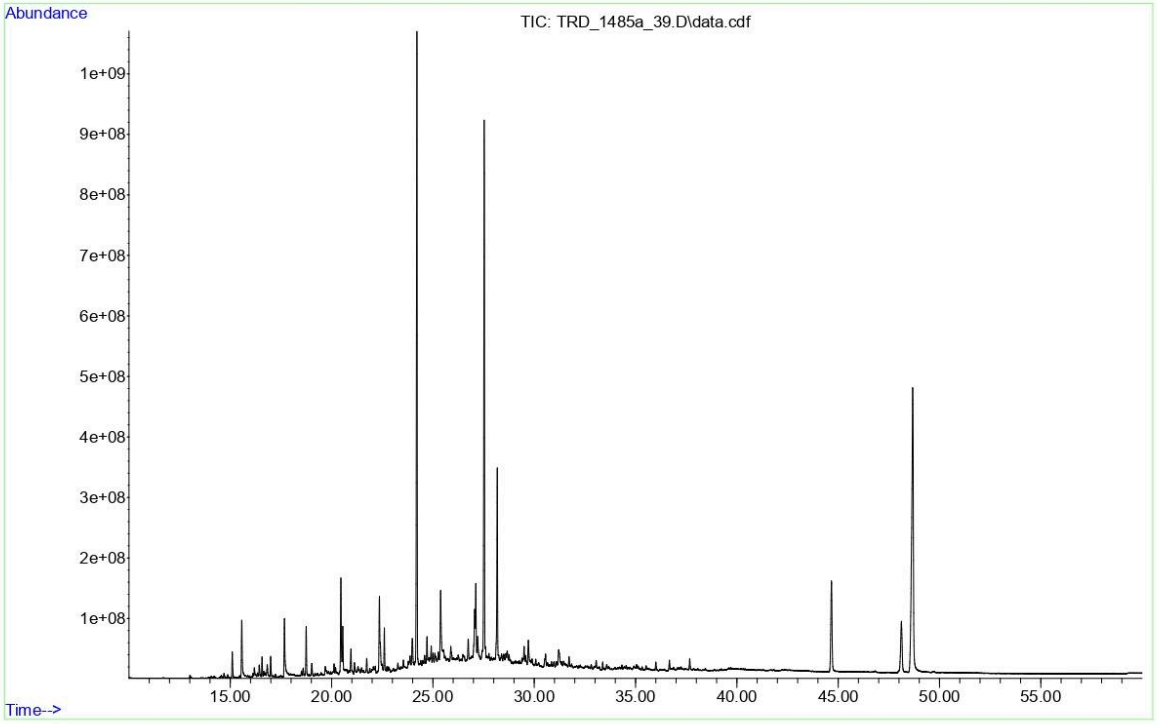
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1487a_16.D
Operator : SYSTEM
Acquired : 20221118053221+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



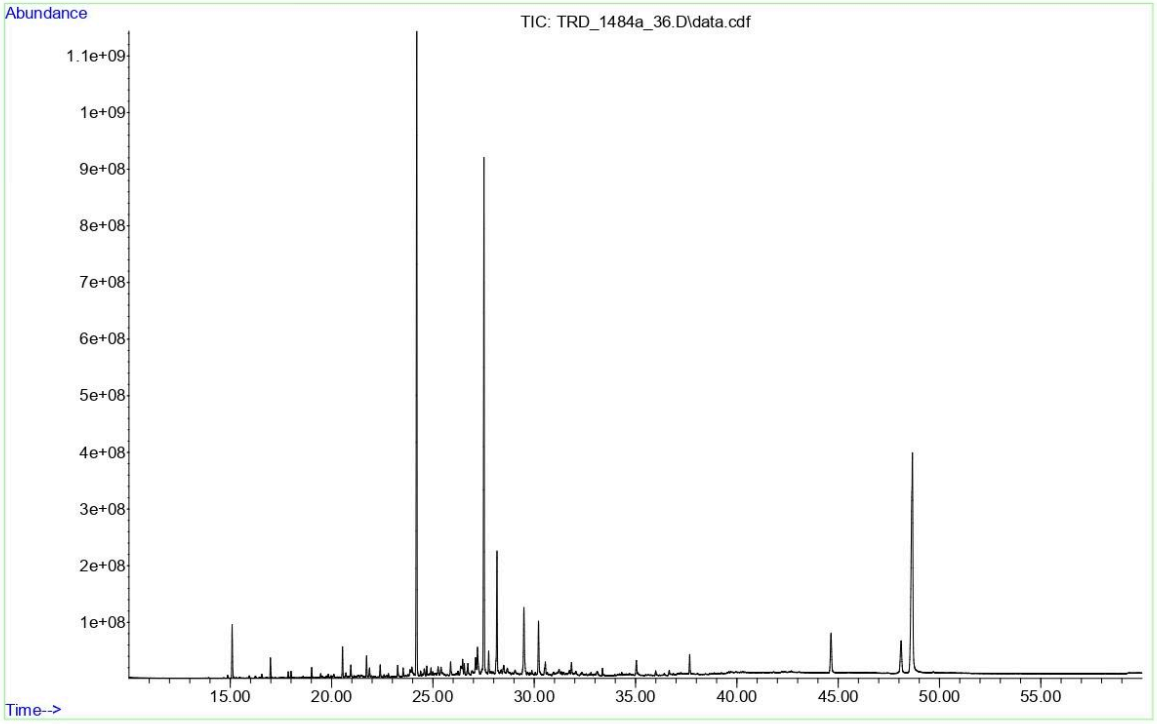
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1486a_41.D
Operator : SYSTEM
Acquired : 20221116125939+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



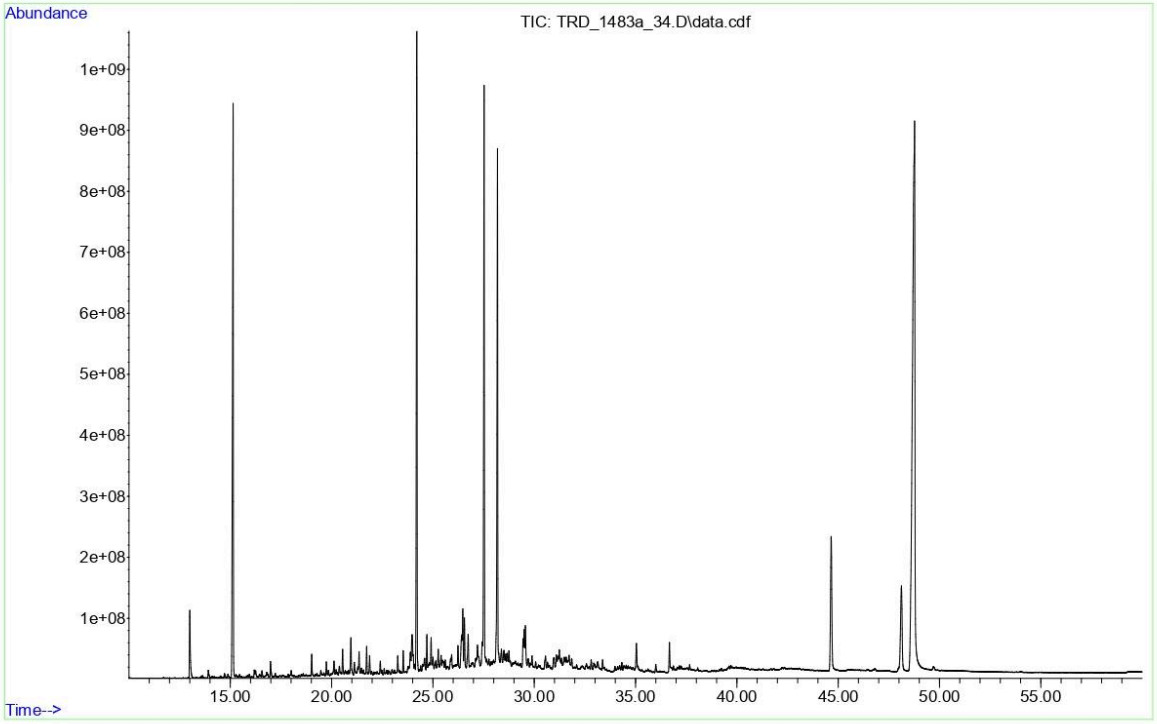
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1485a_39.D
Operator : SYSTEM
Acquired : 20221116103501+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



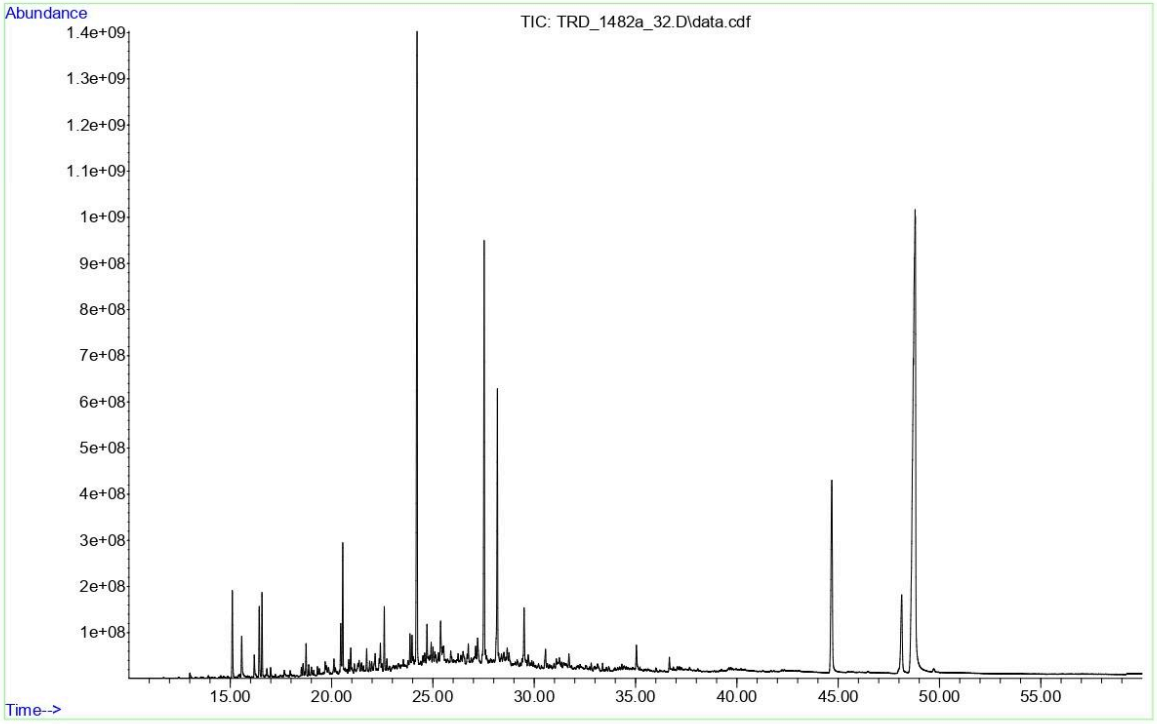
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1484a_36.D
Operator : SYSTEM
Acquired : 20221116065809+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



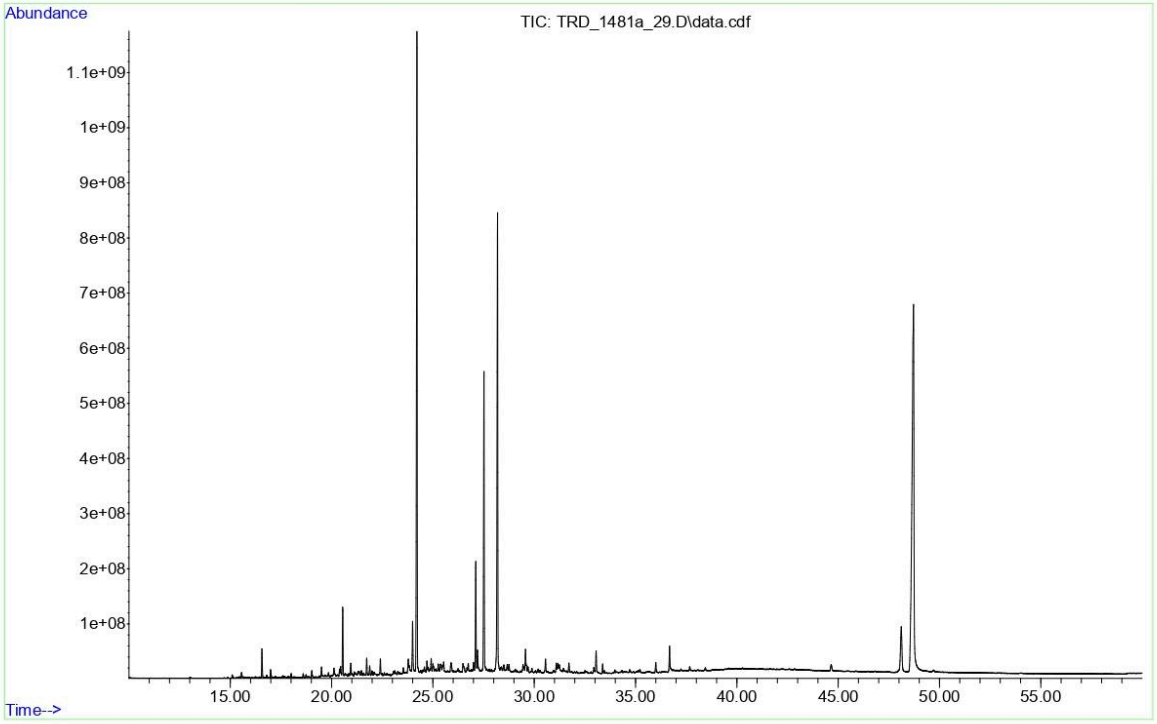
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1483a_34.D
Operator : SYSTEM
Acquired : 20221116043338+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



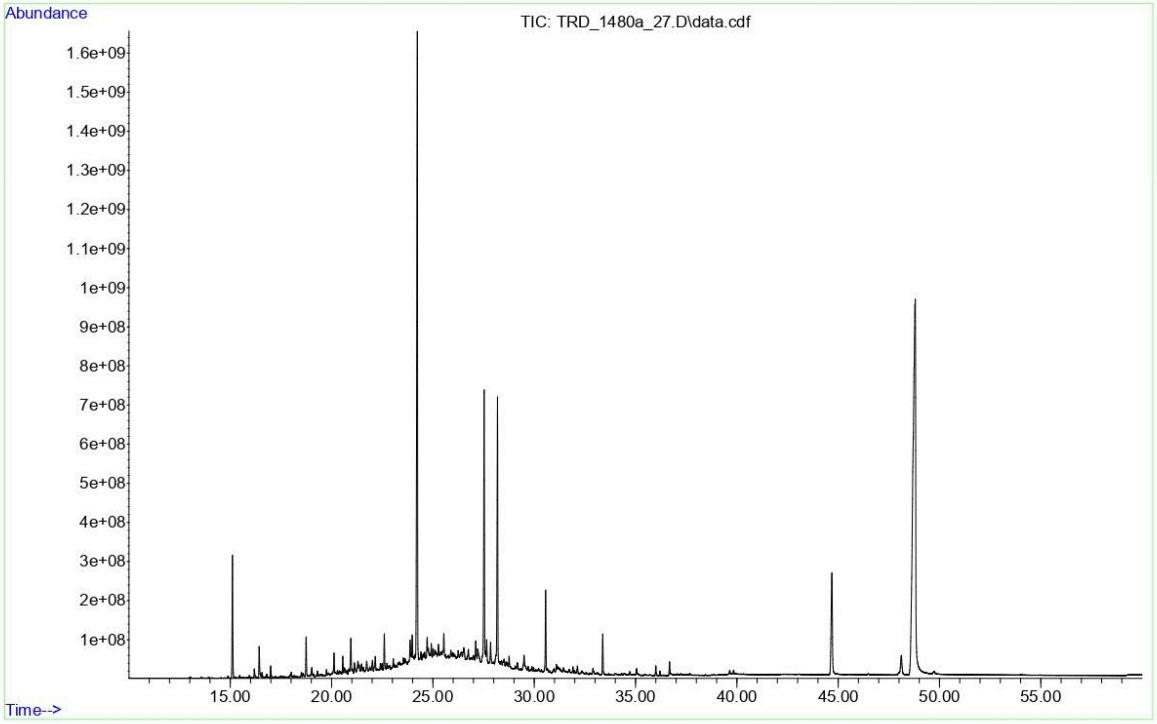
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1482a_32.D
Operator : SYSTEM
Acquired : 20221116020906+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



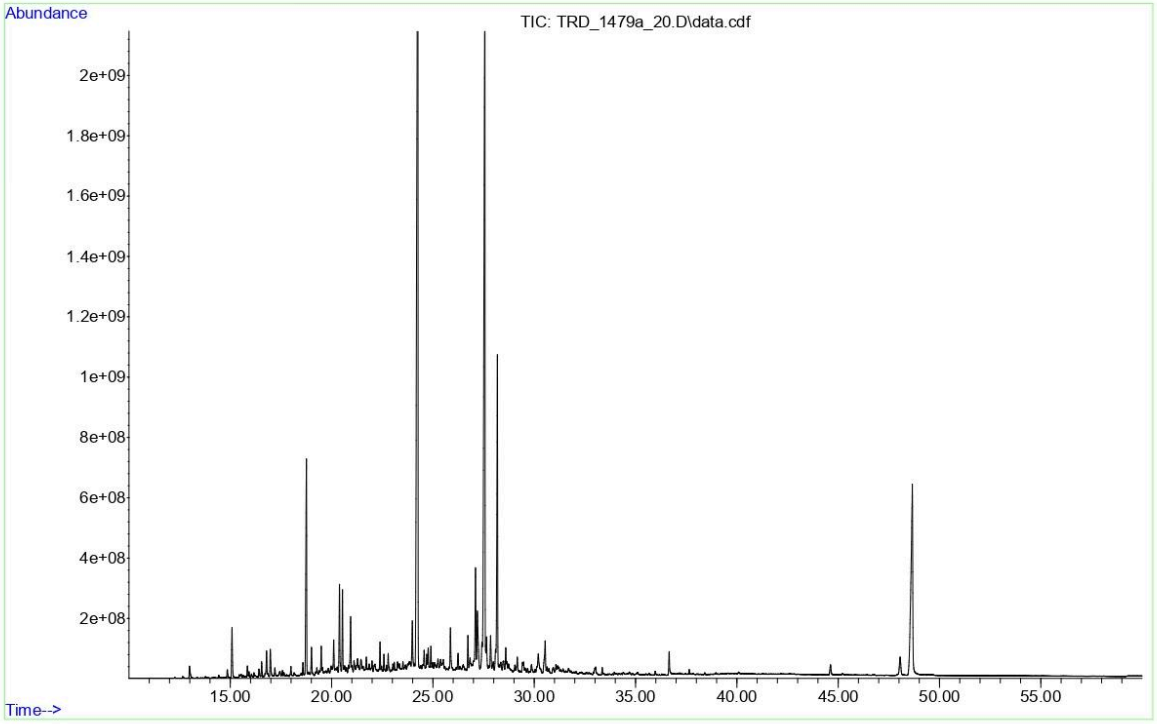
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1481a_29.D
Operator : SYSTEM
Acquired : 20221115223216+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



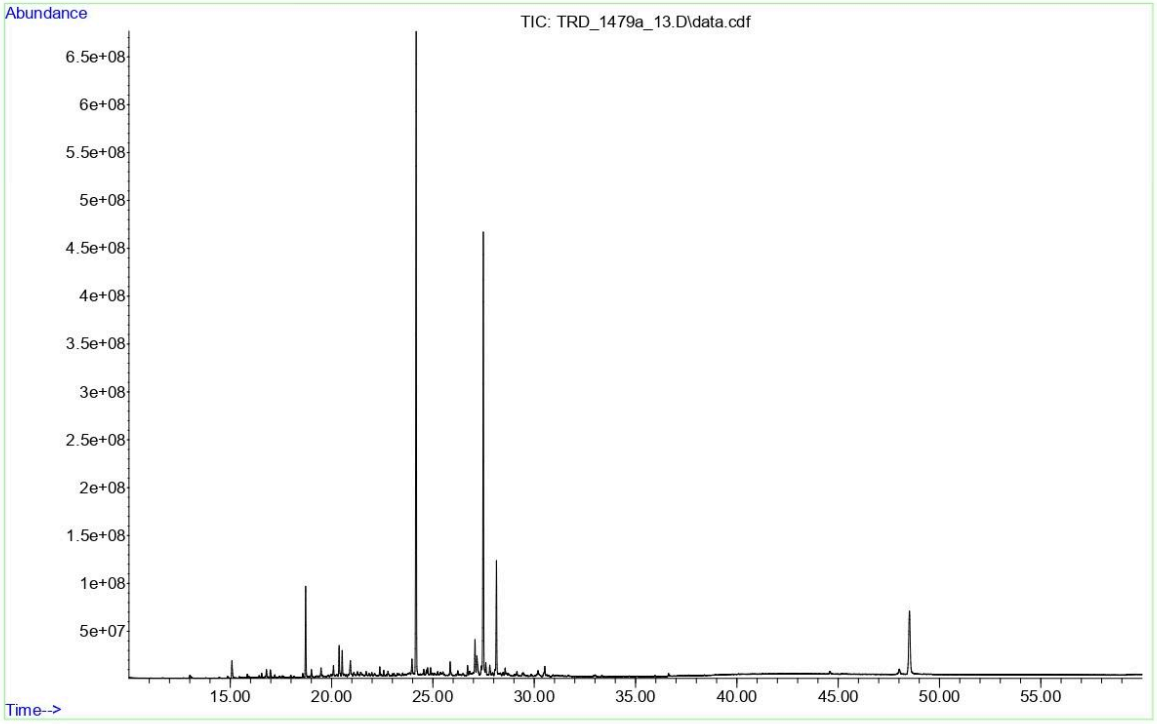
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1480a_27.D
Operator : SYSTEM
Acquired : 20221115200740+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



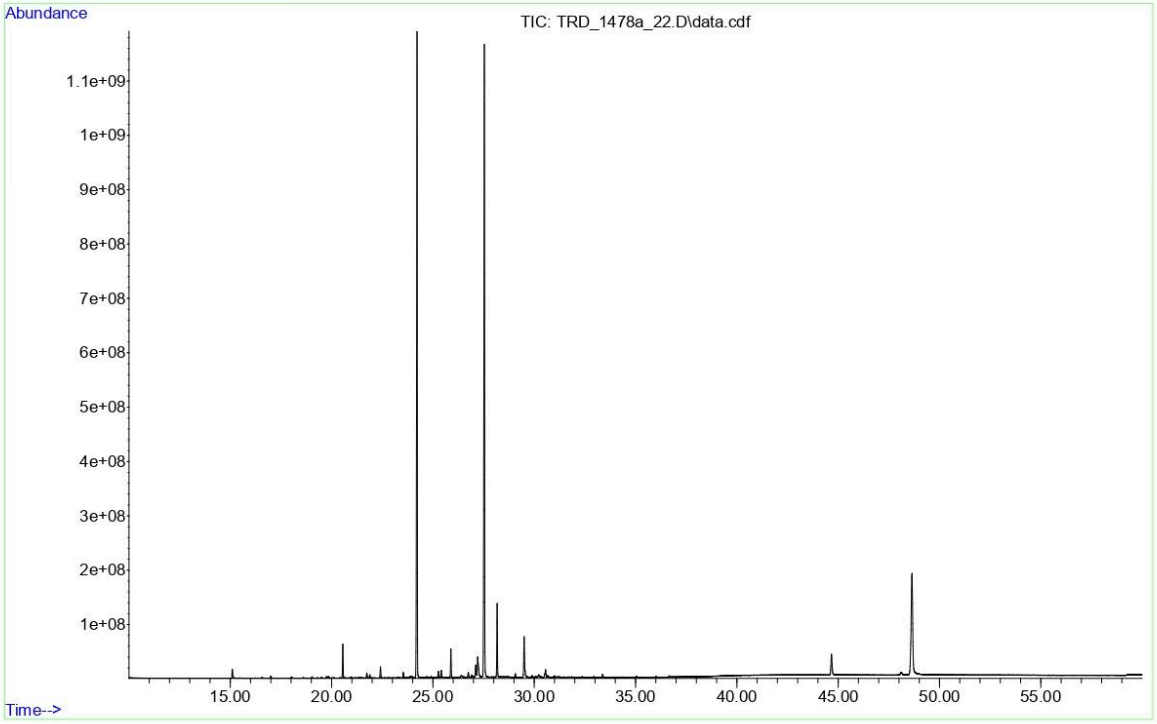
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1479a_20.D
Operator : SYSTEM
Acquired : 20221122130109+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



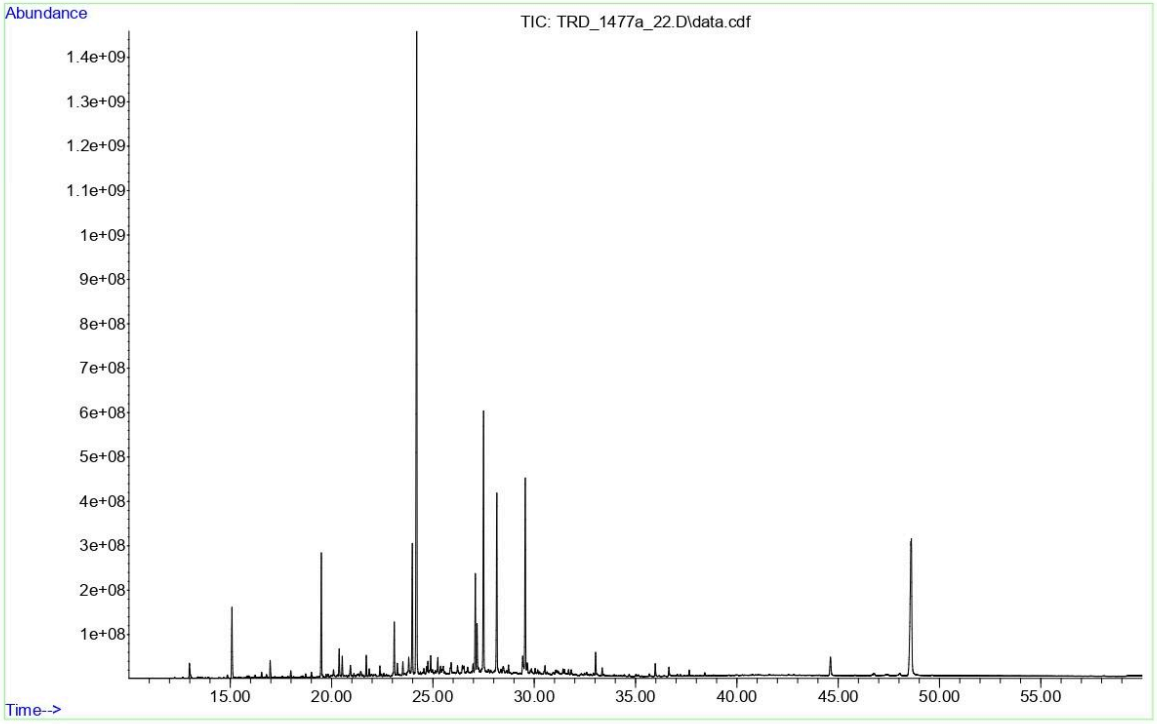
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1479a_13.D
Operator : SYSTEM
Acquired : 20221125012608+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



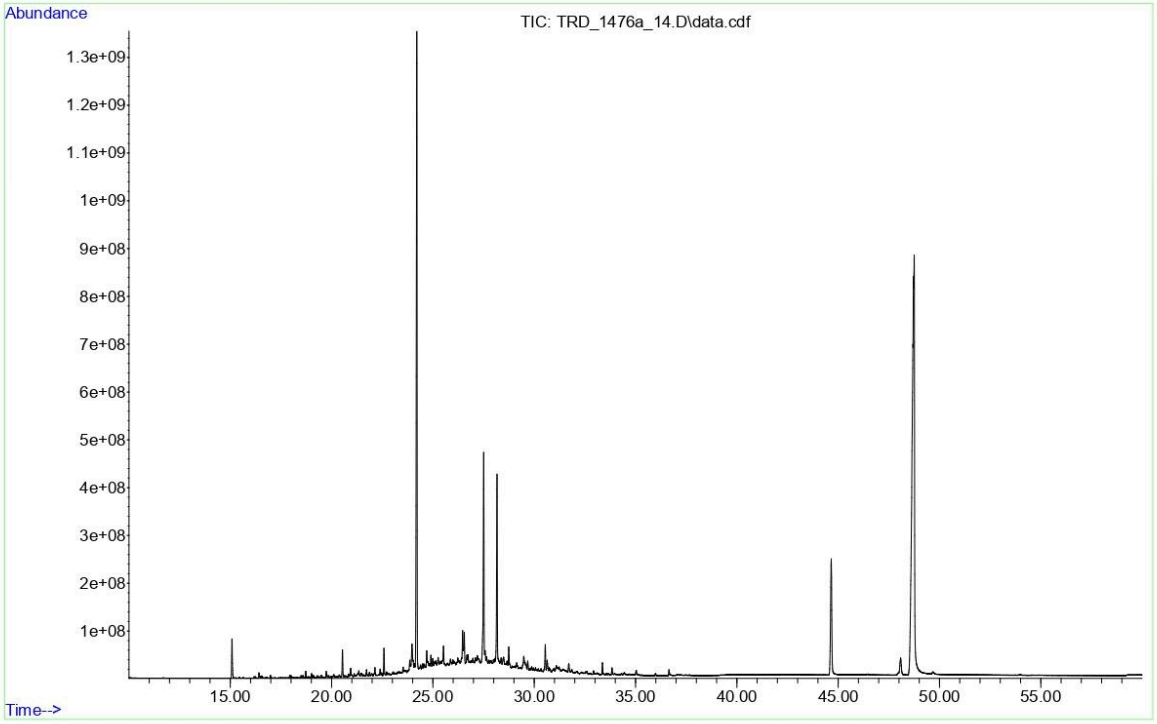
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1478a_22.D
Operator : SYSTEM
Acquired : 20221115140611+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



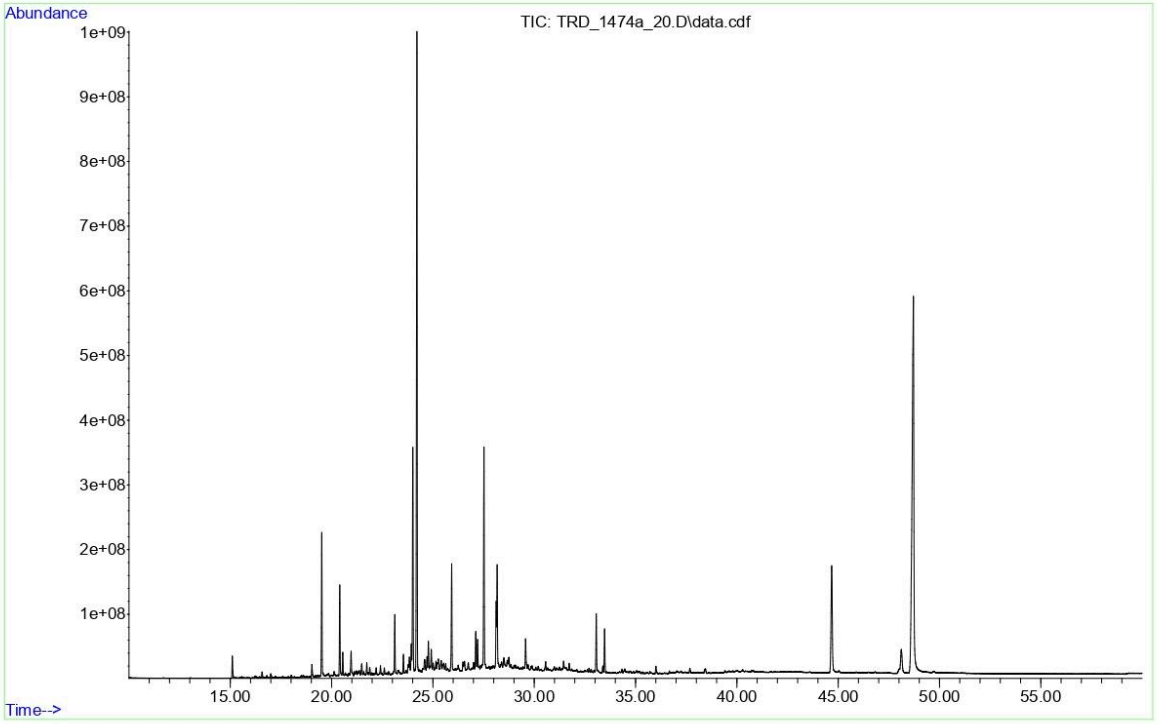
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1477a_22.D
Operator : SYSTEM
Acquired : 20221122152541+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



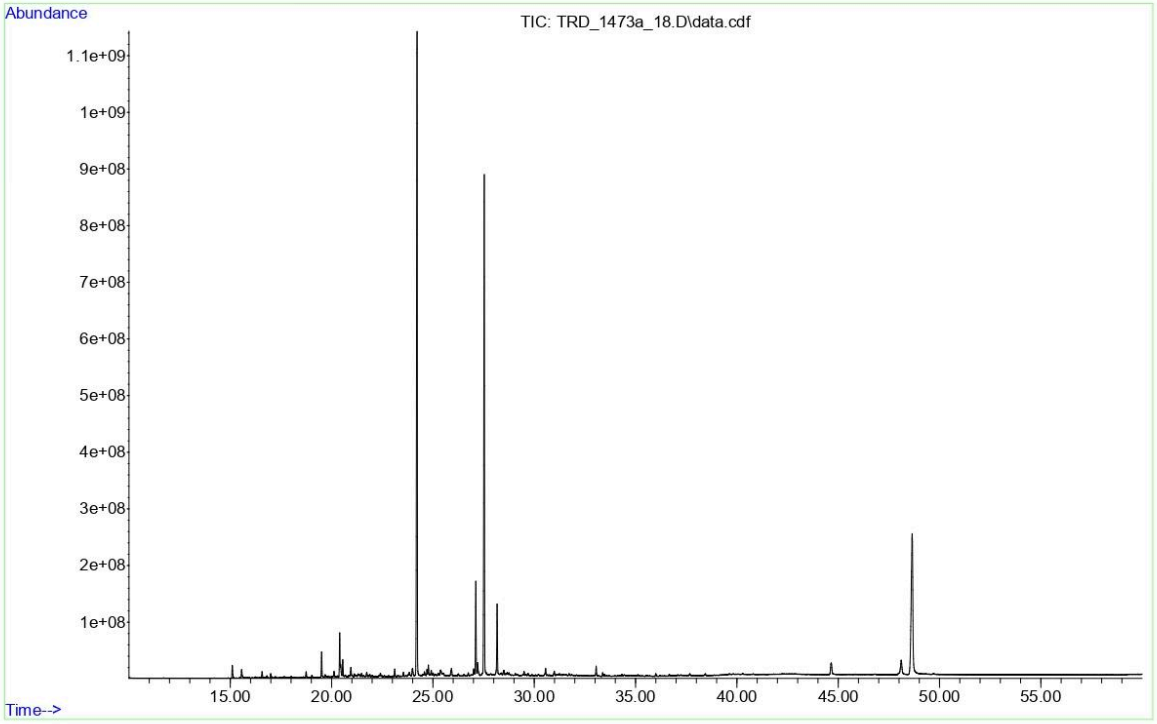
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1476a_14.D
Operator : SYSTEM
Acquired : 20221118030747+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



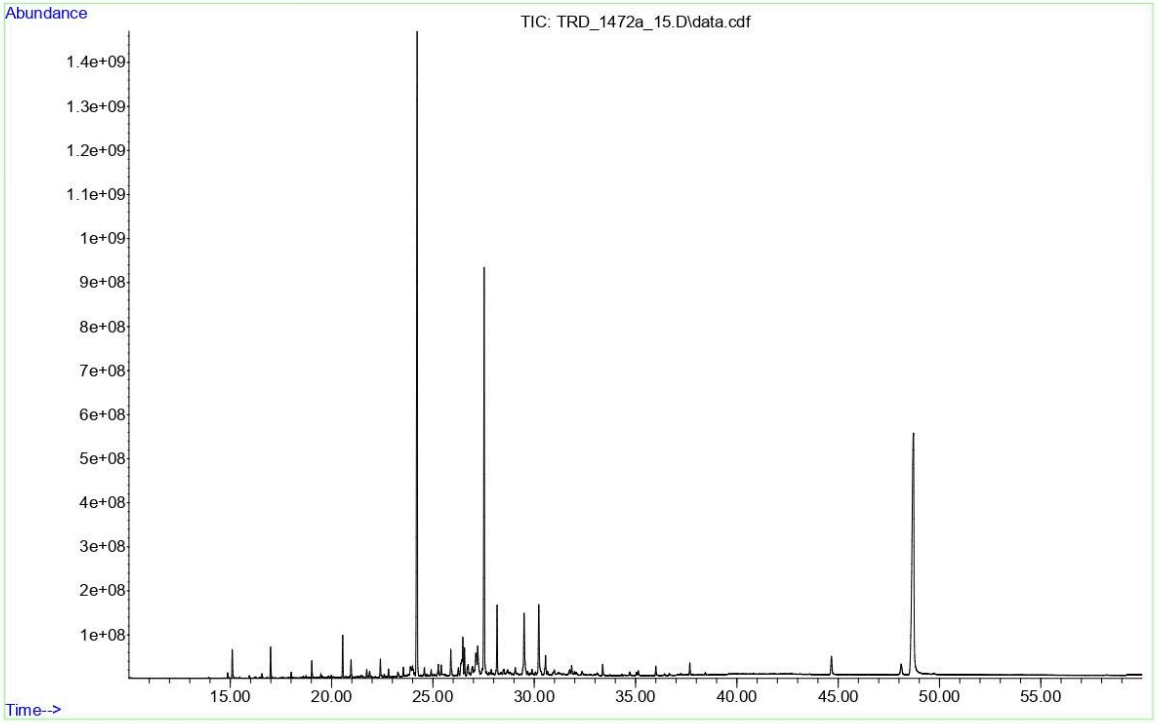
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1474a_20.D
Operator : SYSTEM
Acquired : 20221115114139+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



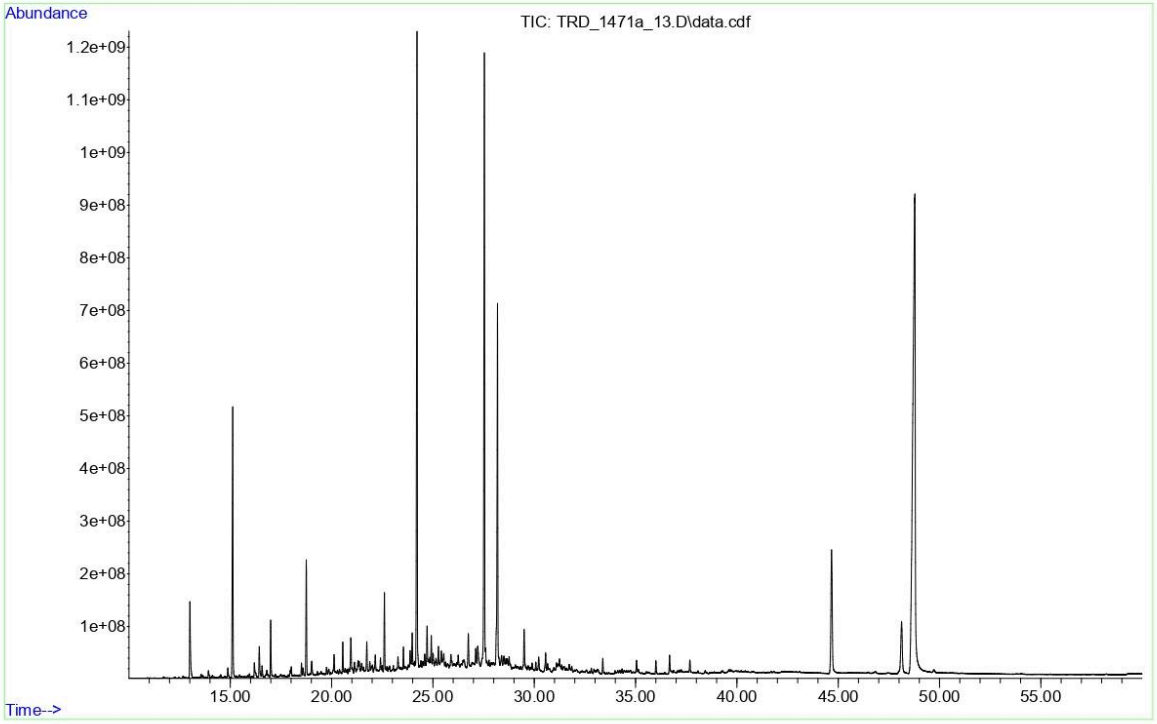
File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1473a_18.D
Operator : SYSTEM
Acquired : 20221115091709+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



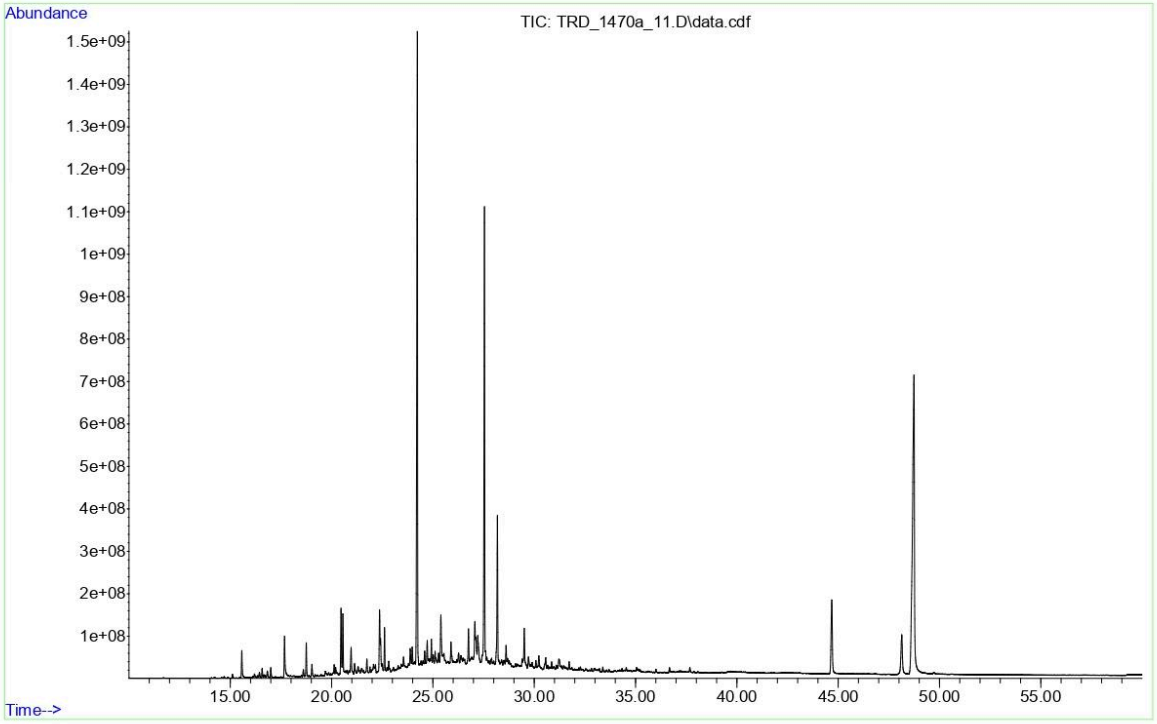
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Operator : SYSTEM
Acquired : 20221115054029+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



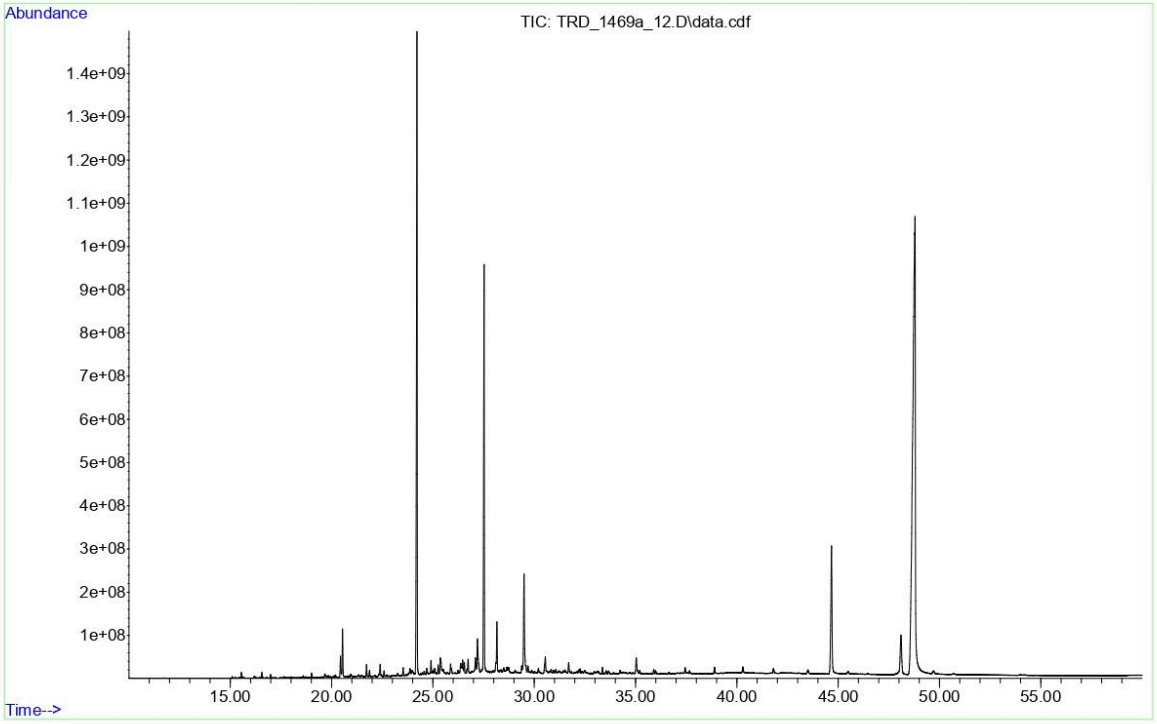
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Operator : SYSTEM
Acquired : 20221115031558+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



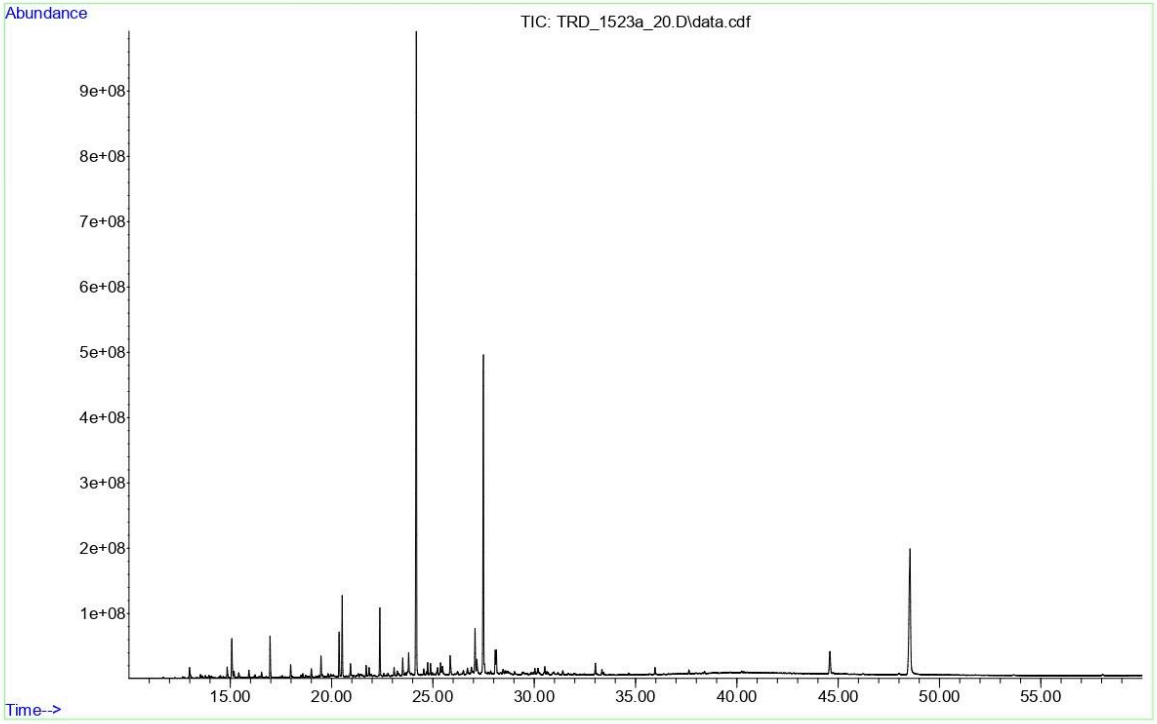
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Operator : SYSTEM
Acquired : 20221115005126+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



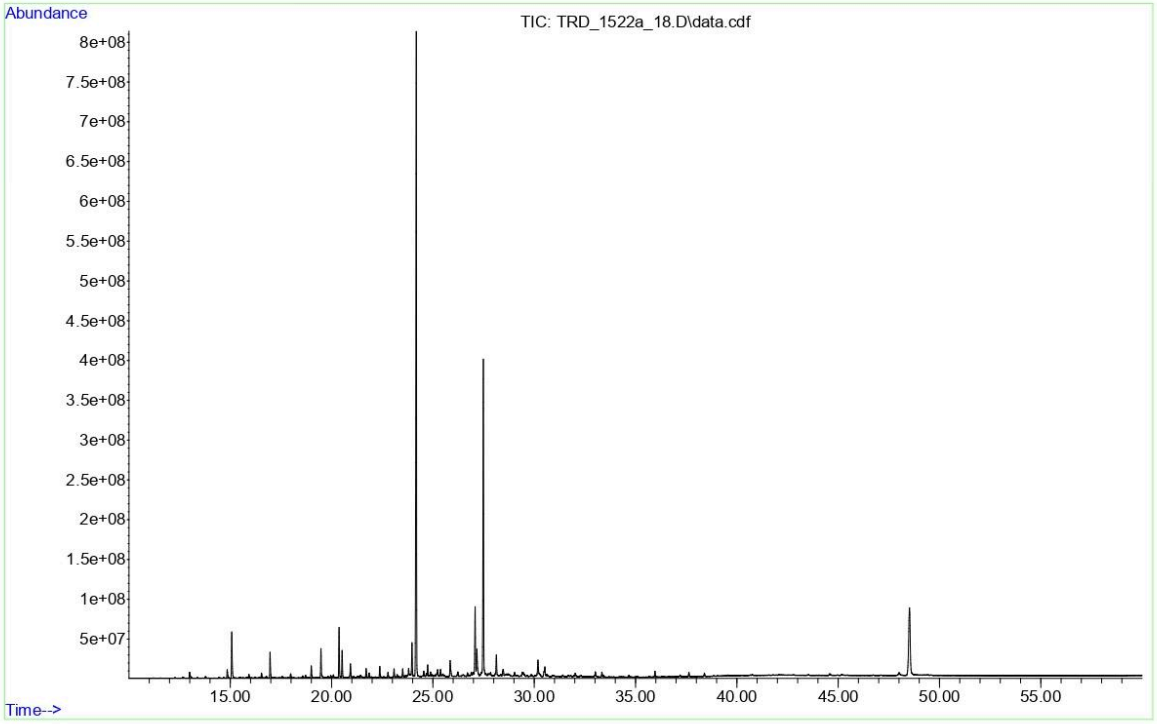
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Operator : SYSTEM
Acquired : 20221118004317+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



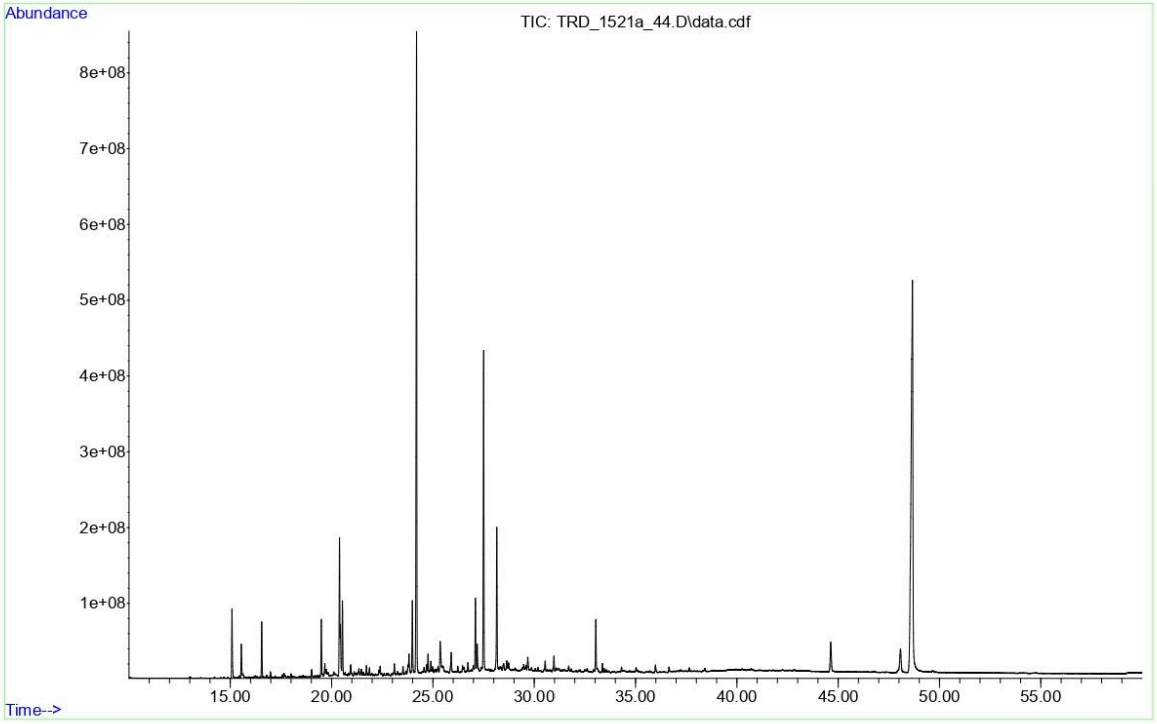
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Operator : SYSTEM
Acquired : 20221125095145+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



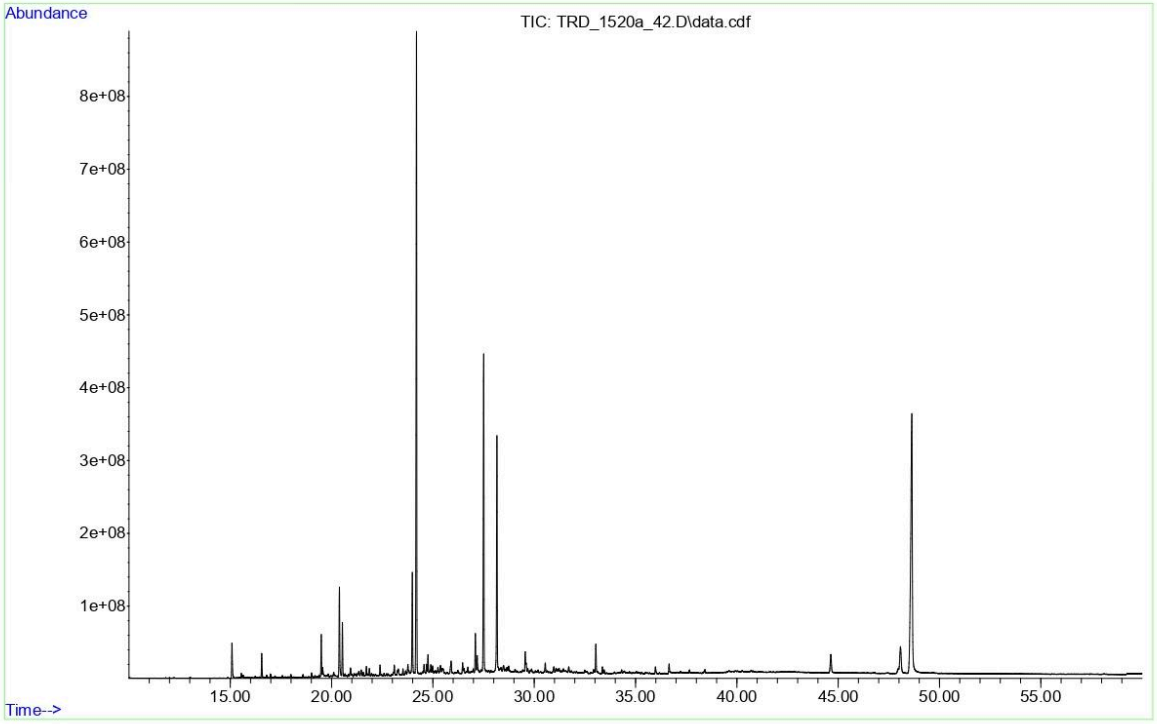
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Operator : SYSTEM
Acquired : 20221125072719+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



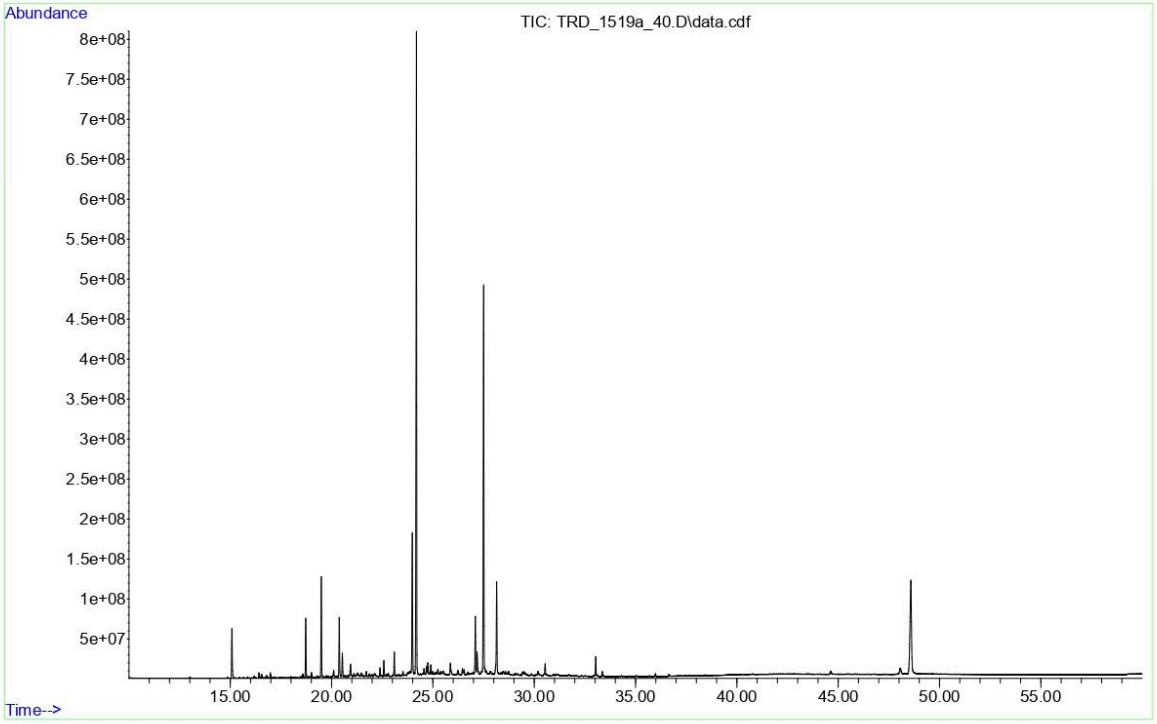
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Operator : SYSTEM
Acquired : 20221119151543+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1520a_42.D
Operator : SYSTEM
Acquired : 20221119125107+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Data by Site\Unidade21\DB5-GCMS\TRD_1519a_40.D
Operator : SYSTEM
Acquired : 20221119102635+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0

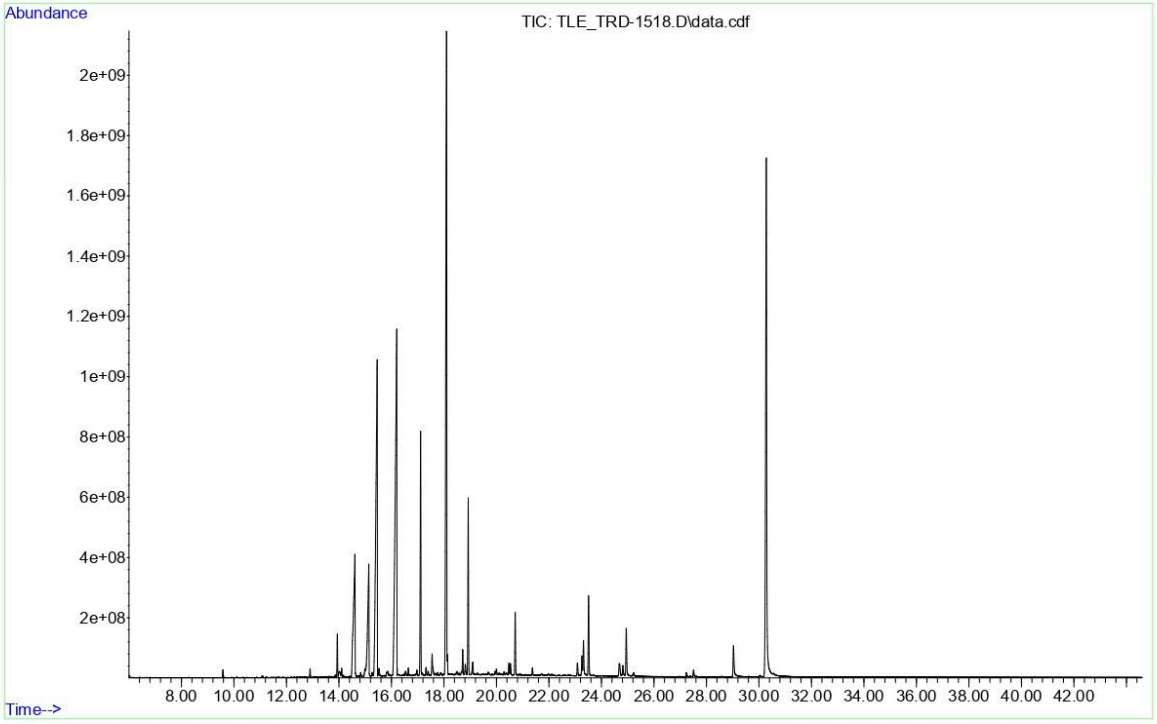


Praia Grande -Unidade 21

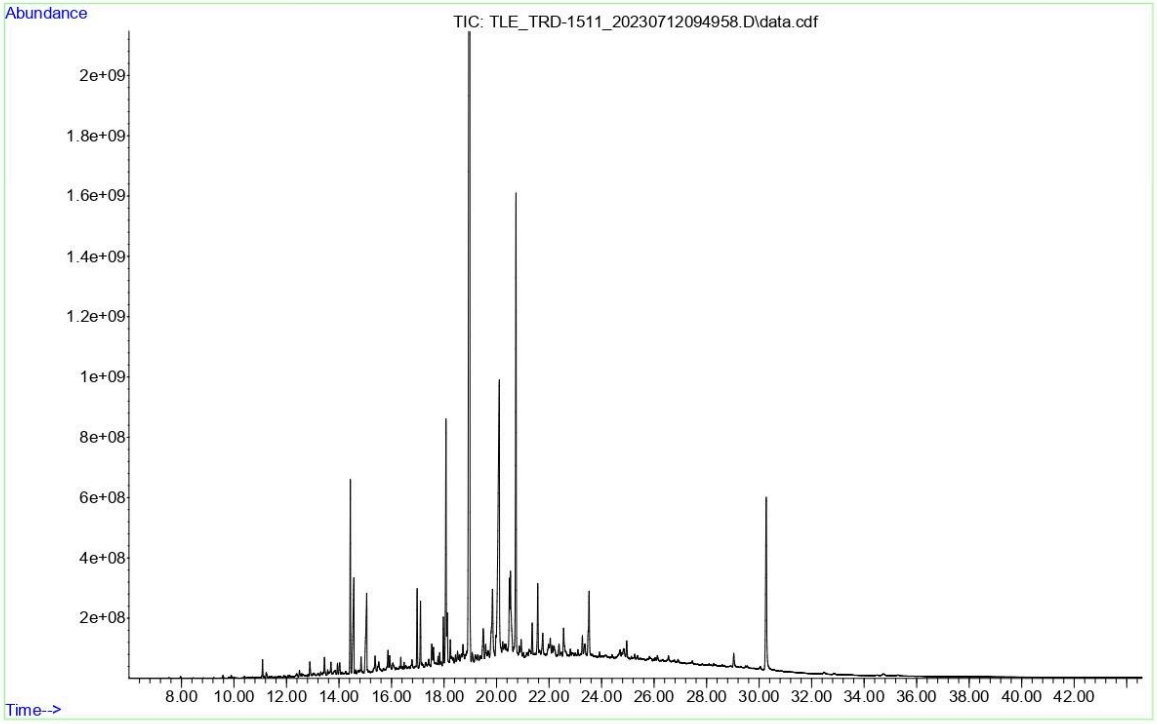
Total lipid extraction / TMS chromatograms

For Praia Grande -Unidade 21, 31 TLE files were originally generated. Of these, 16 raw chromatogram files were corrupted and could not be reopened or exported at the time of thesis revision. The retrievable TLE chromatograms are included here. Analytical notes, peak tables, and associated exported data remain available for the full set of samples.

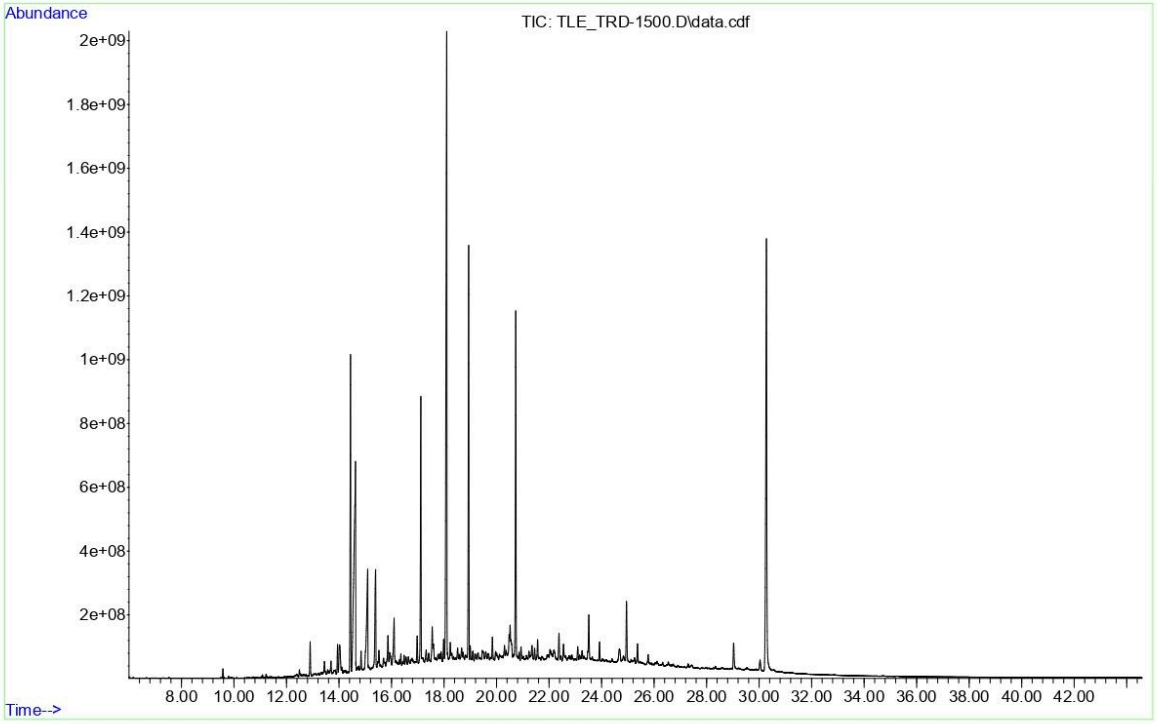
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1518
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230711193412+0000 using AcqMethod
Sample Name:
Misc Info :



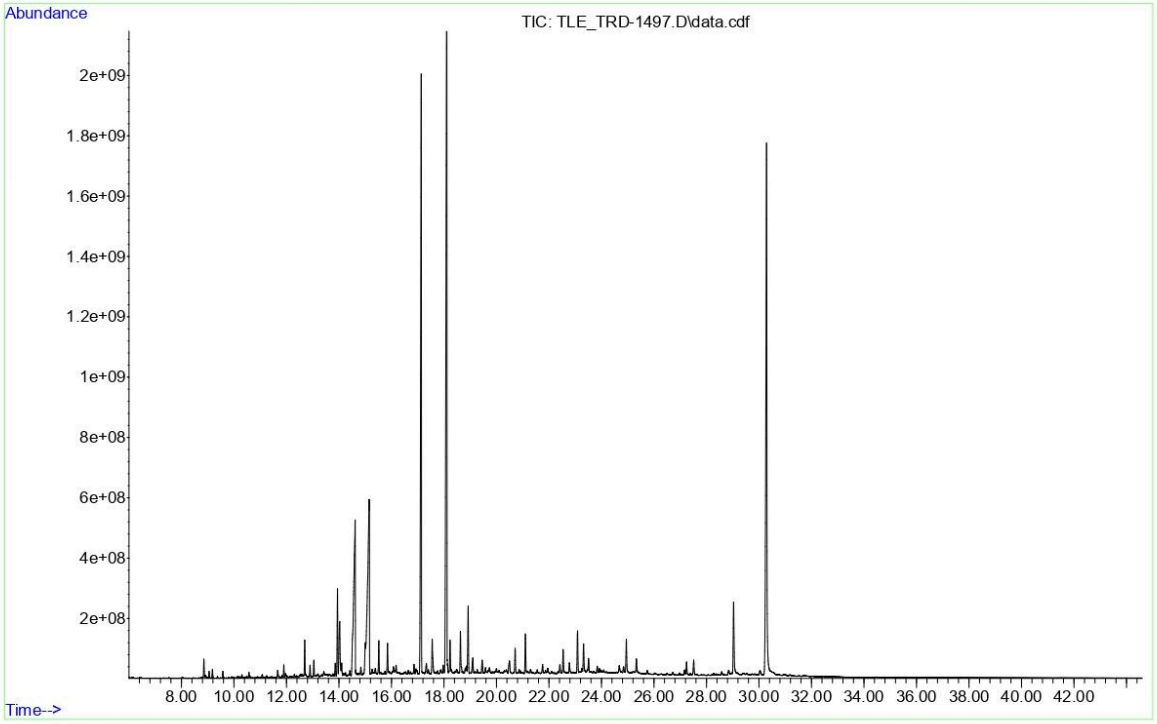
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1511
... _20230712094958.D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230712095334+0000 using AcqMethod
Sample Name:
Misc Info :



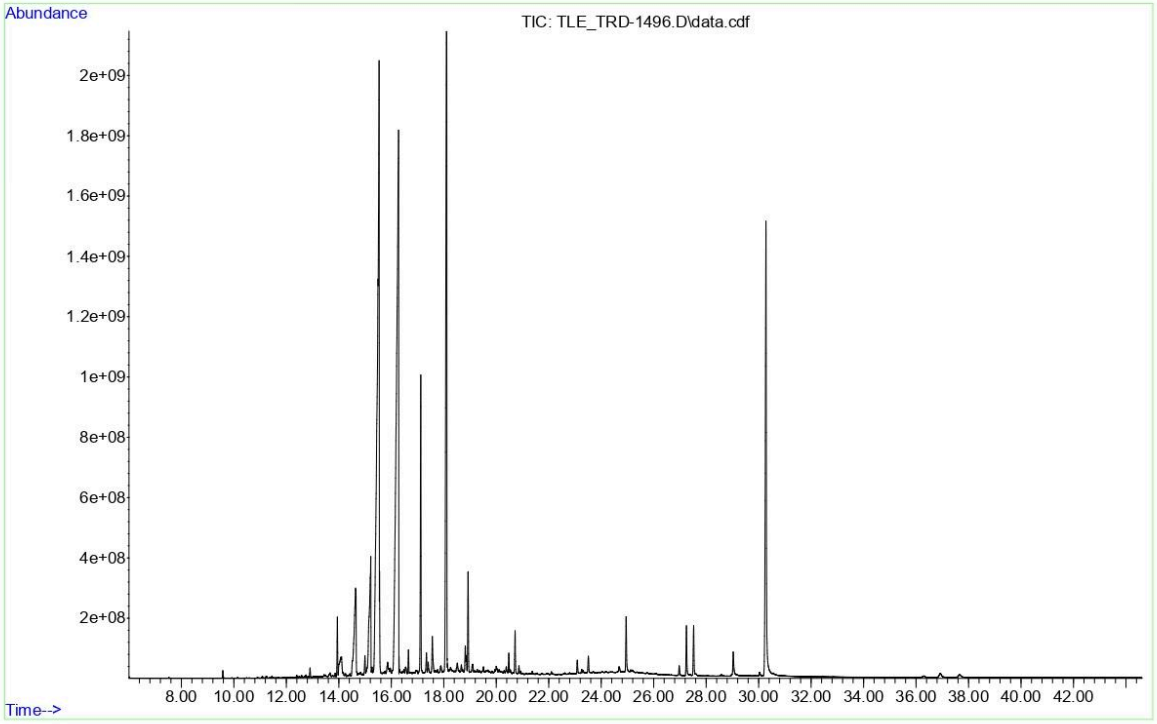
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1500
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230711221208+0000 using AcqMethod
Sample Name:
Misc Info :



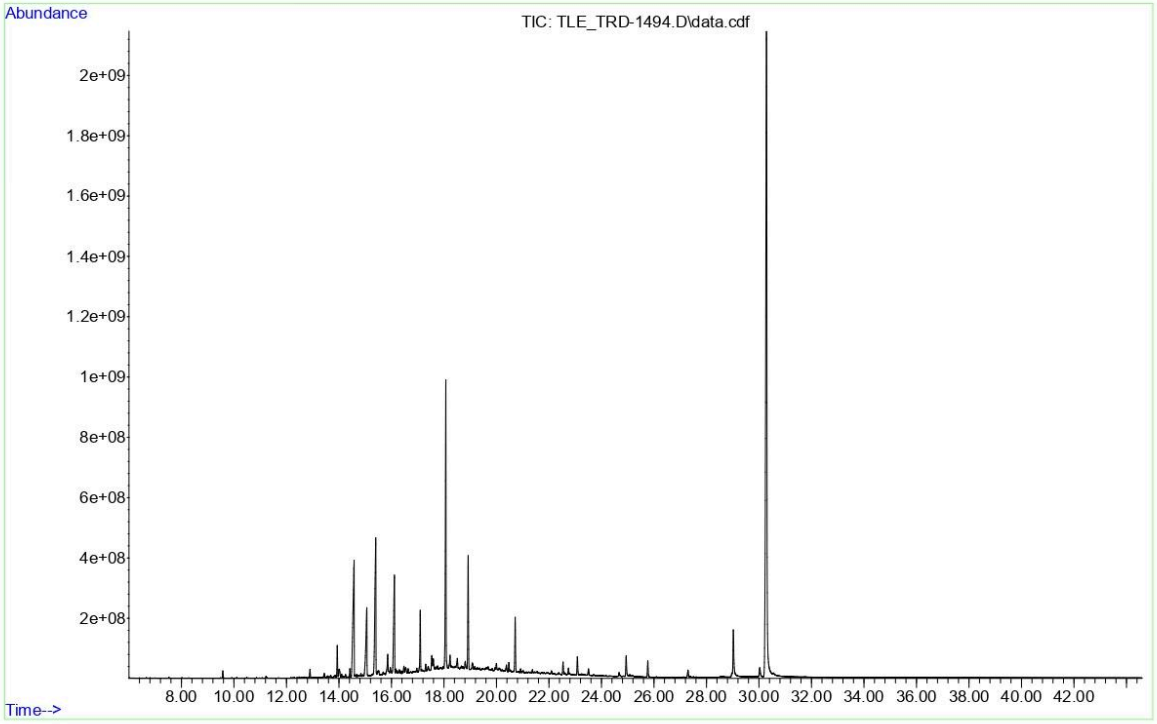
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1497
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230712063109+0000 using AcqMethod
Sample Name:
Misc Info :



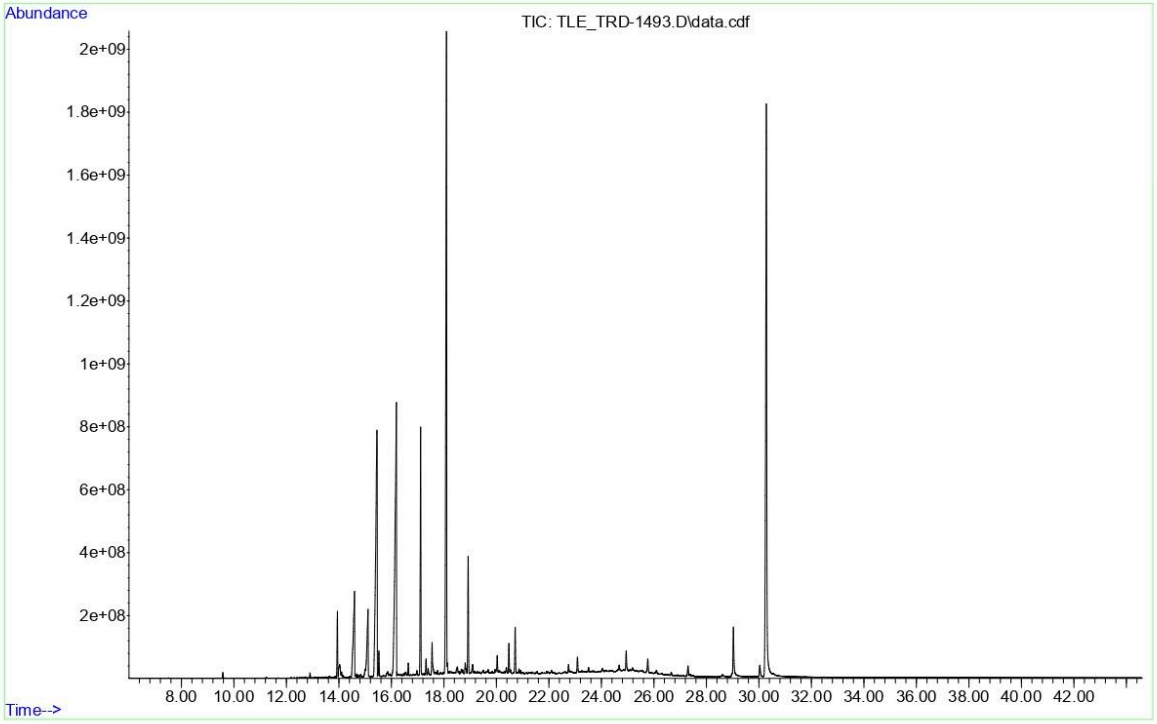
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1496
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230711184134+0000 using AcqMethod
Sample Name:
Misc Info :



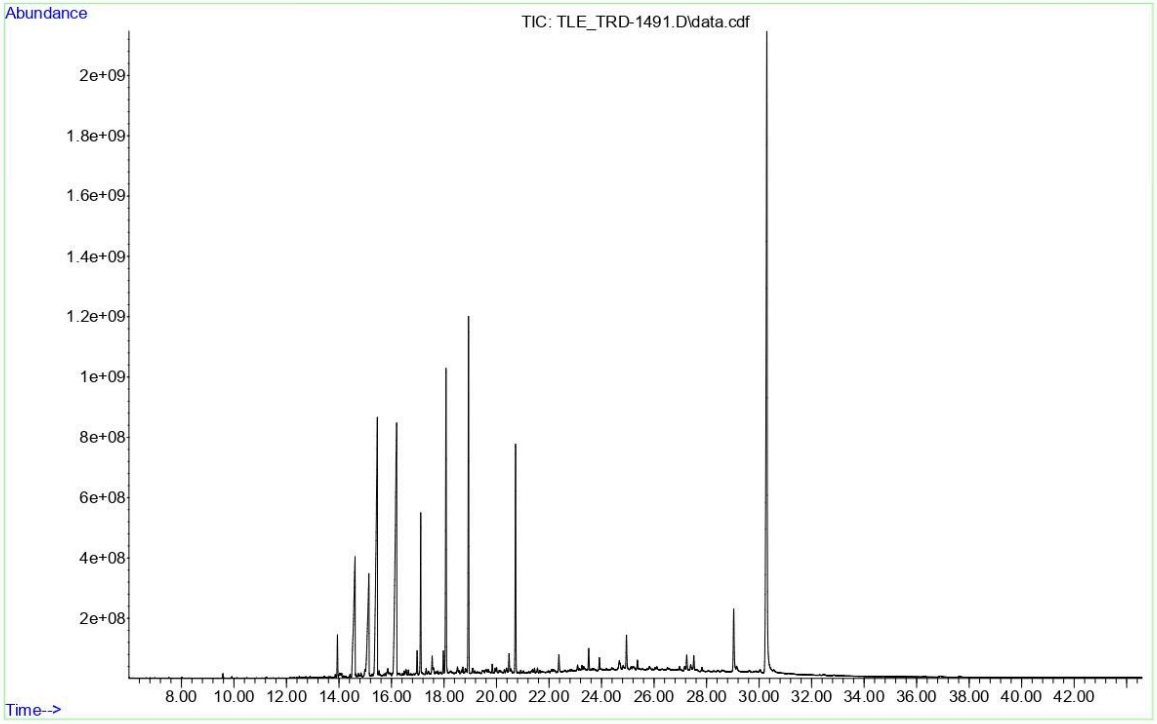
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1494
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230712022137+0000 using AcqMethod
Sample Name:
Misc Info :



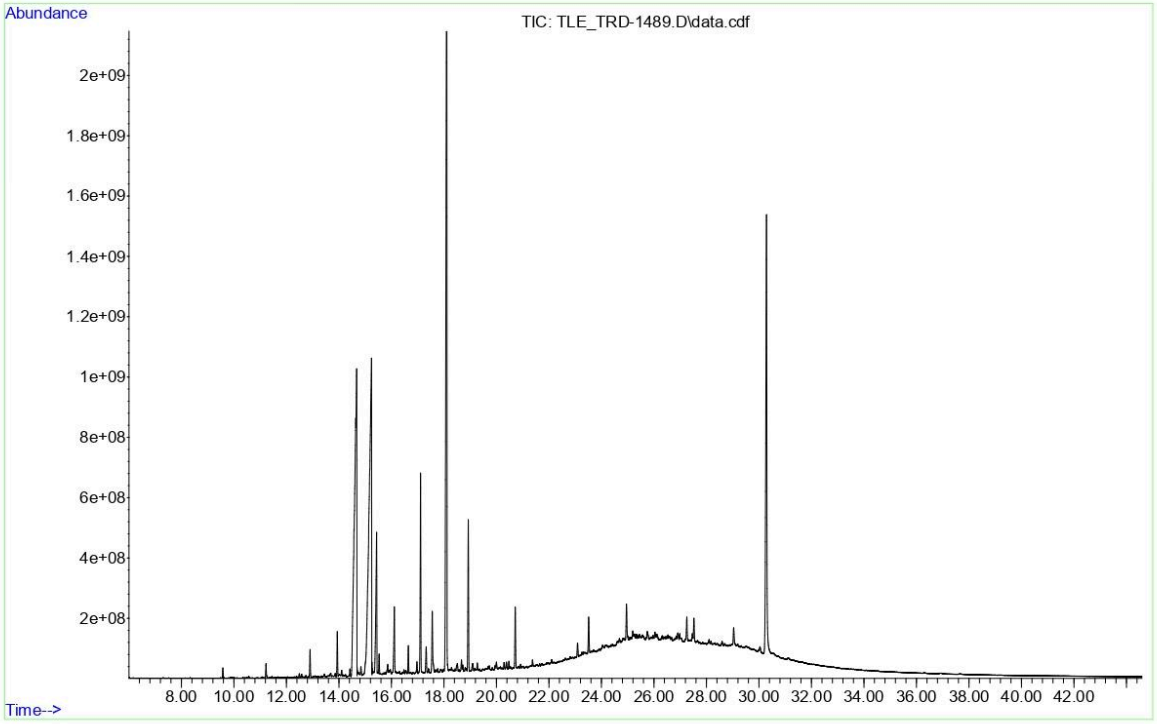
File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRD-1493
... .D
Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230712044548+0000 using AcqMethod
Sample Name:
Misc Info :



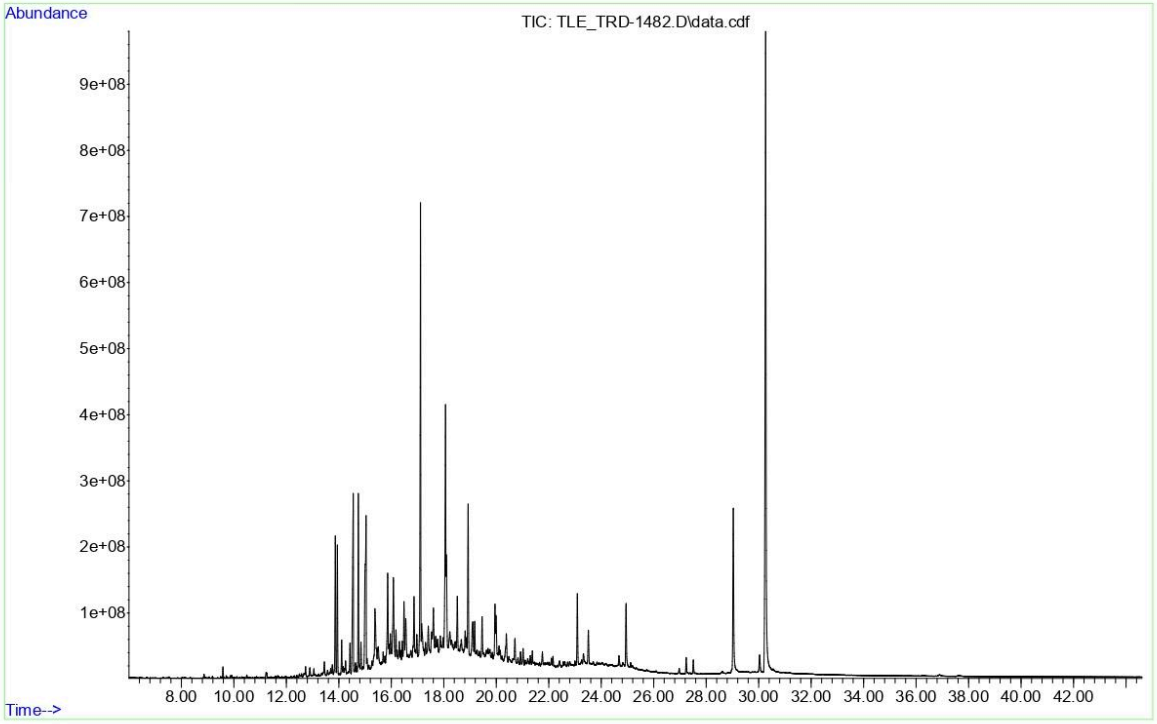
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Instrument : LCQ
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Sample Name:
Misc Info :



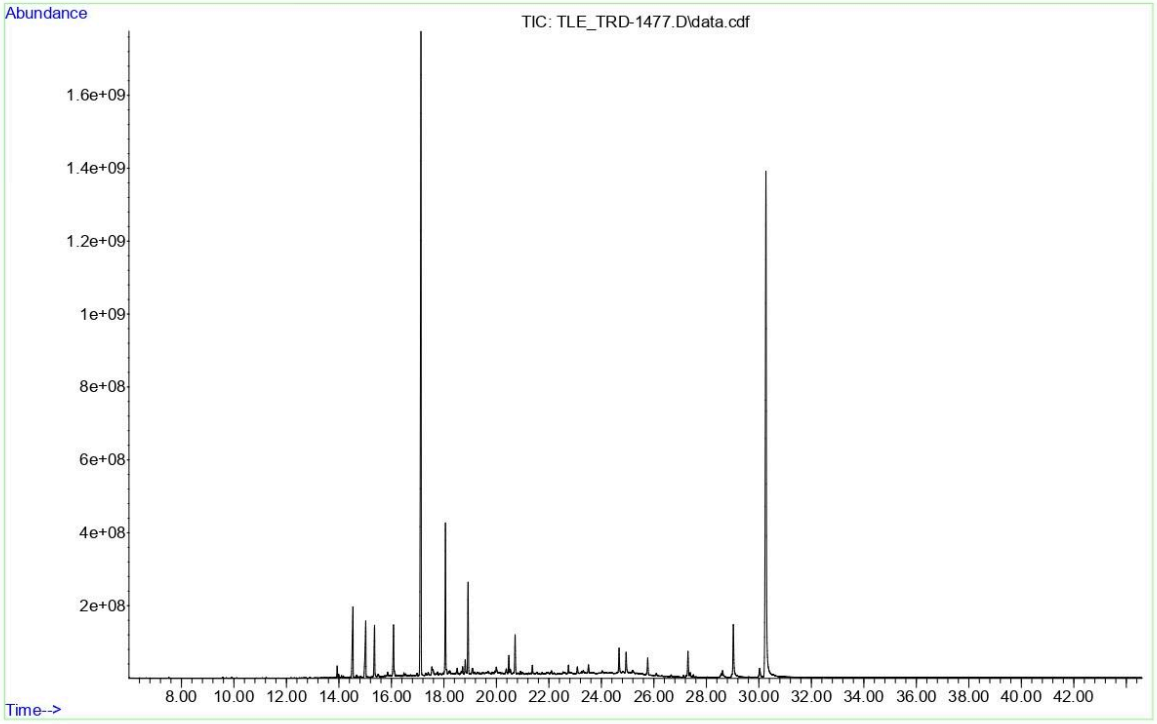
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Instrument : LCQ
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Sample Name:
Misc Info :



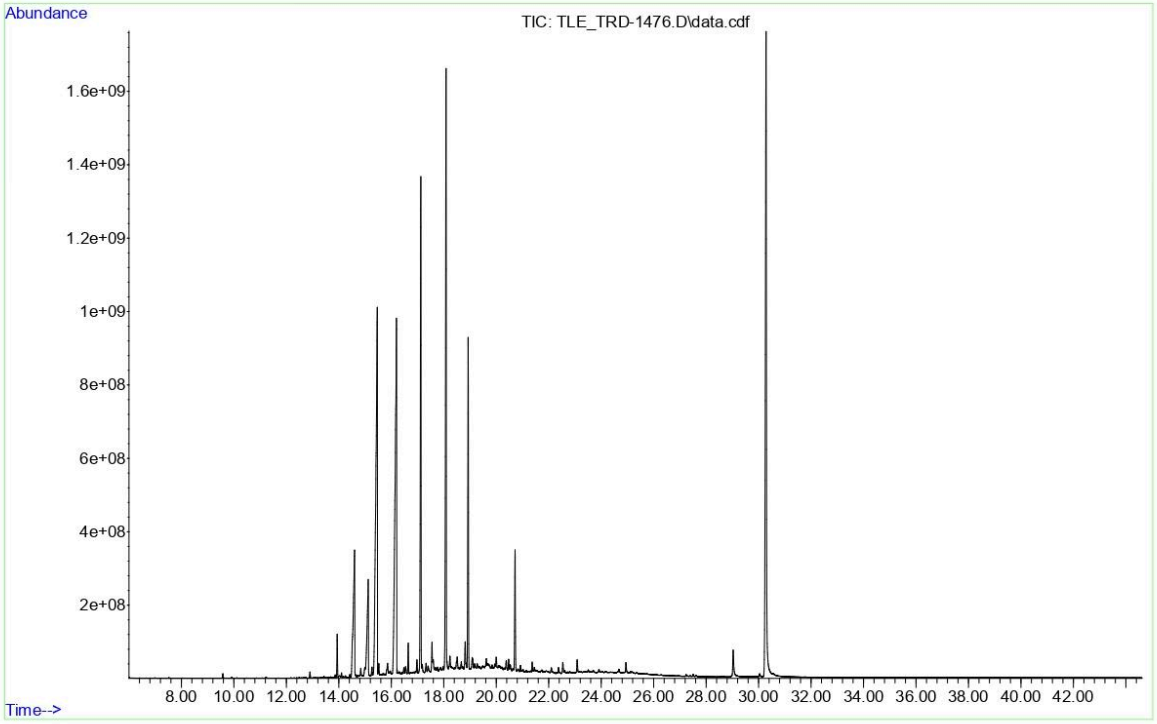
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Instrument : LCQ
Acquired : 20230711211929+0000 using AcqMethod
Sample Name:
Misc Info :



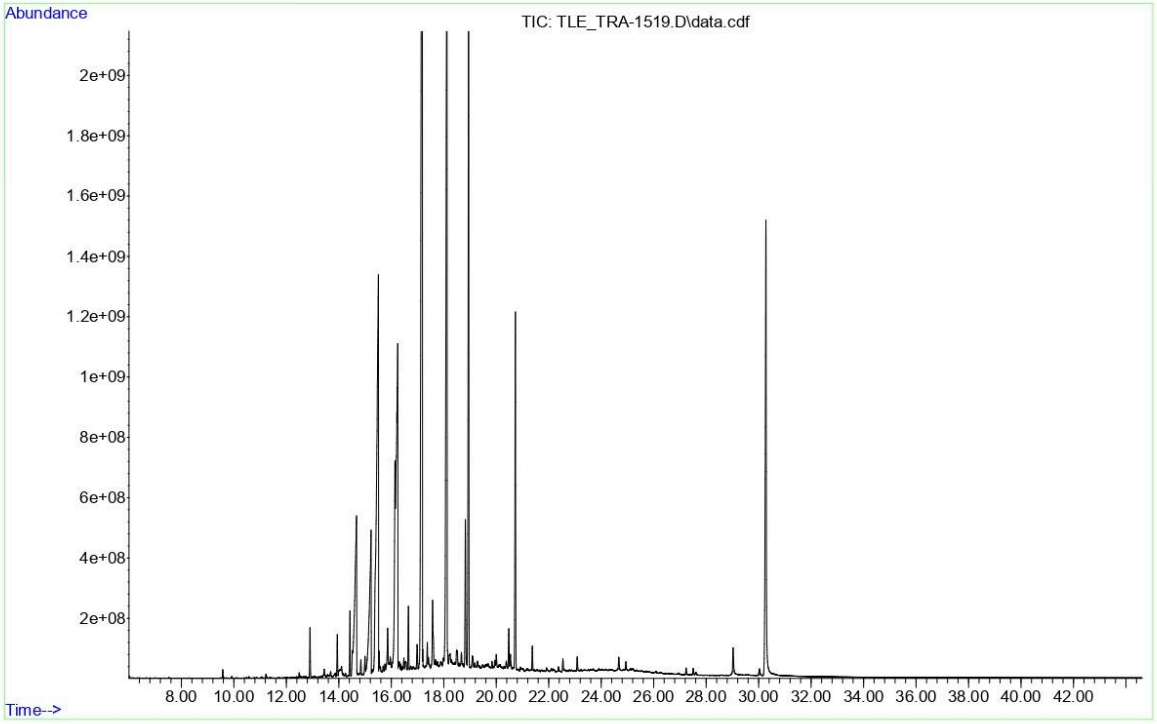
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Instrument : LCQ
Acquired : 20230711234340+0000 using AcqMethod
Sample Name:
Misc Info :



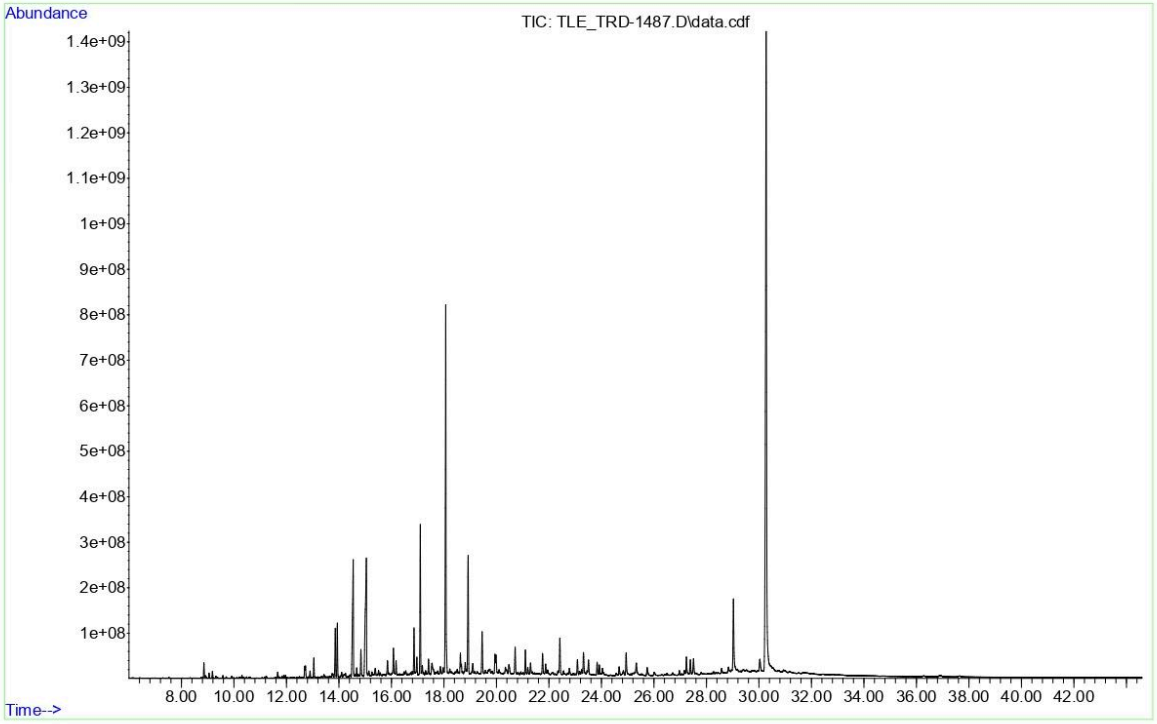
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Operator : Xcalibur_System
Instrument : LCQ
Acquired : 20230712003618+0000 using AcqMethod
Sample Name:
Misc Info :



File :G:\ADM_JULY_TLE_BIOARCH\IND20230711_ISQ_AdM_cdf\TLE_TRA-1519
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Operator : Xcalibur_System
Instrument : LCQ
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Sample Name:
Misc Info :



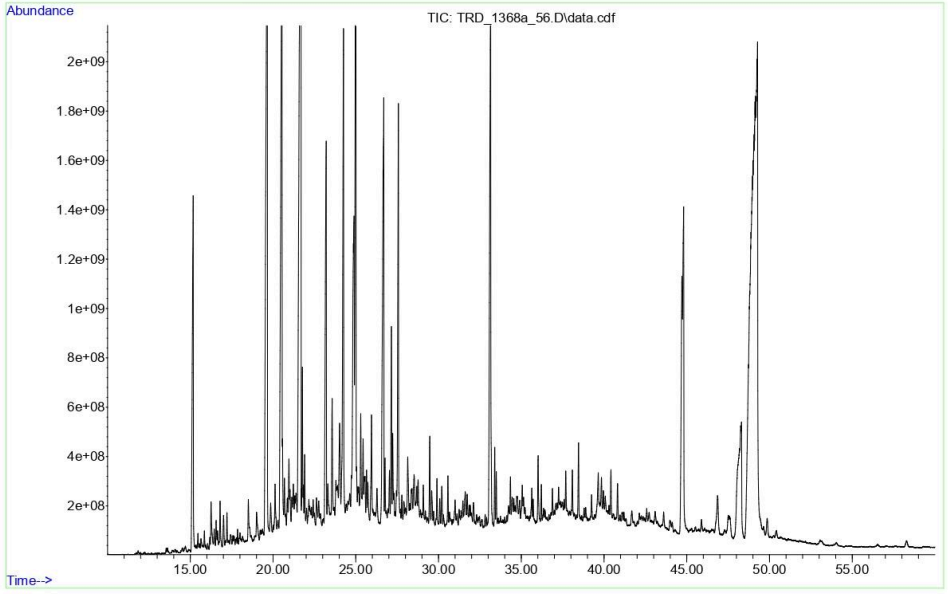
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Sample Name:
Misc Info :



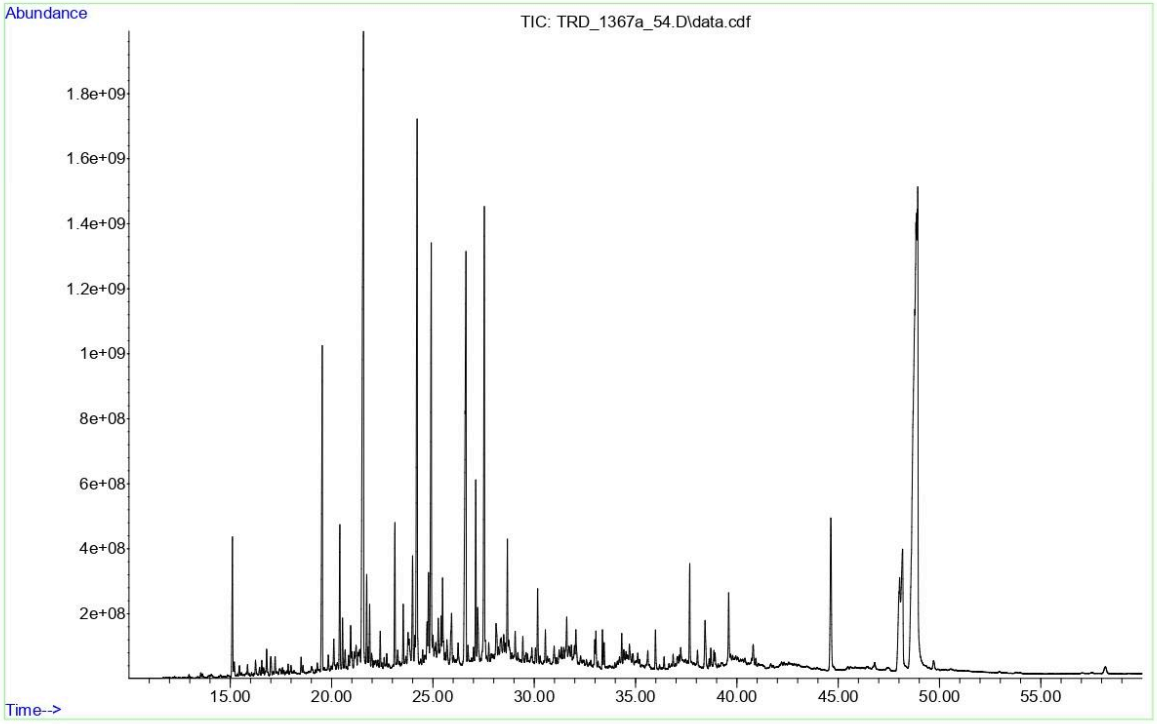
Arroio Tamarina 2

Acidified methanol extraction chromatograms

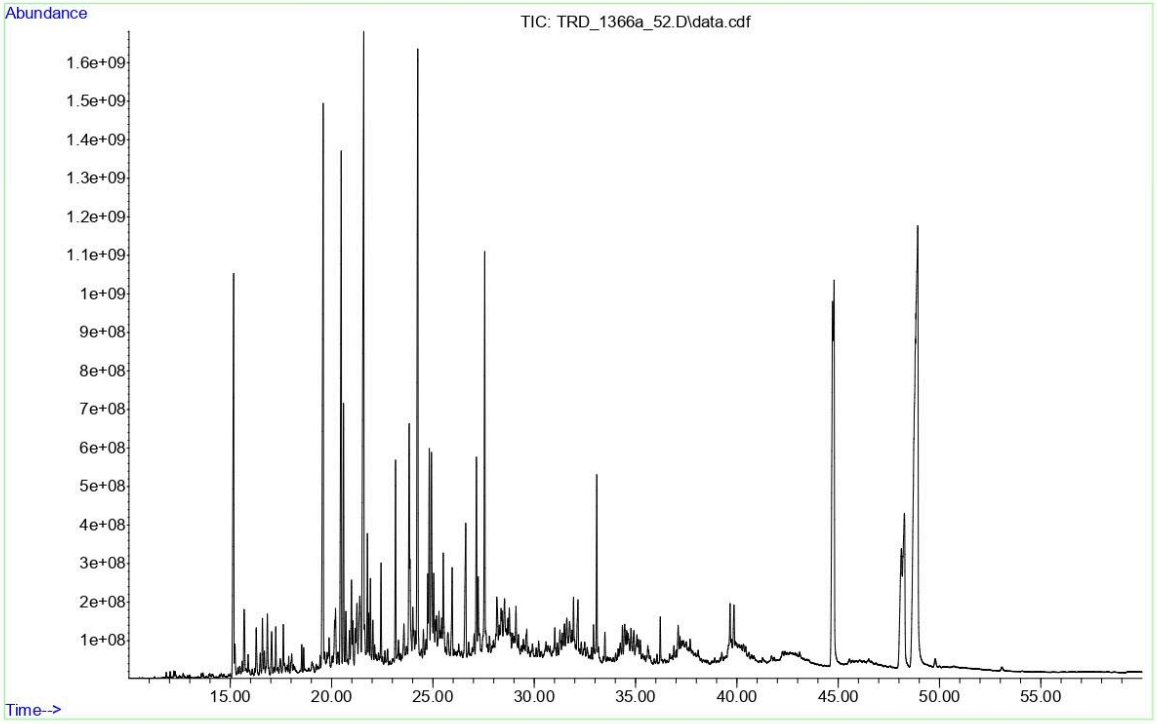
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Instrument :    LCQ
Sample Name:
Misc Info  :
Vial Number: 0
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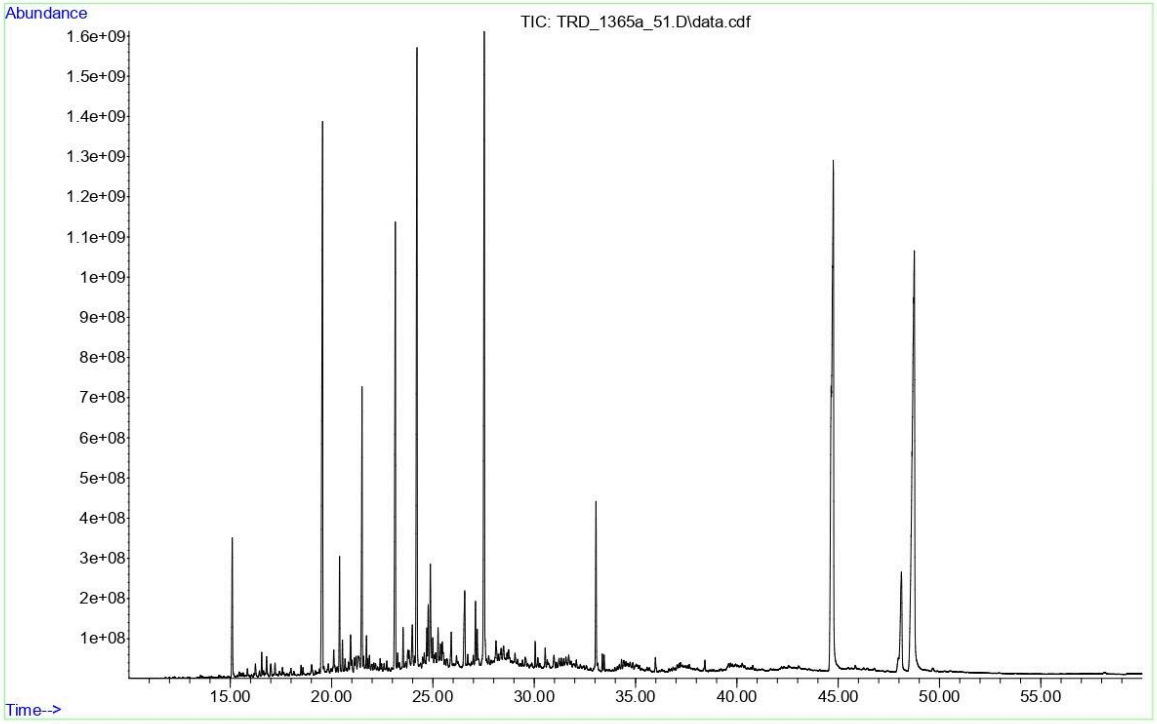
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



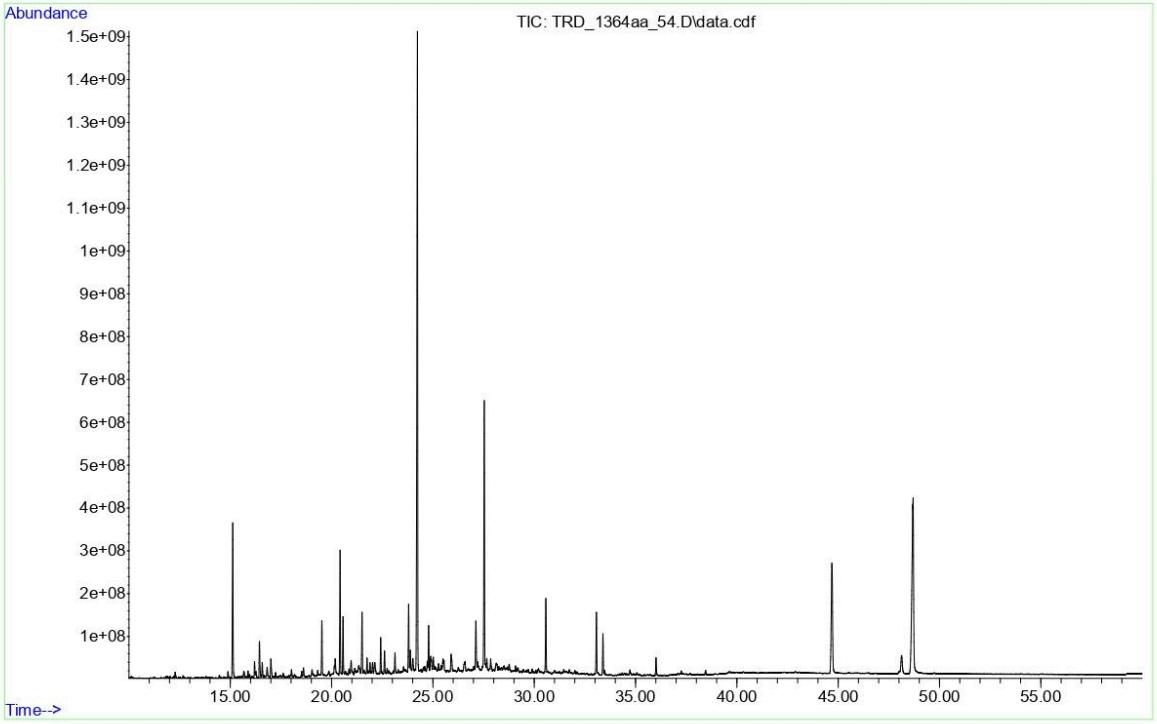
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



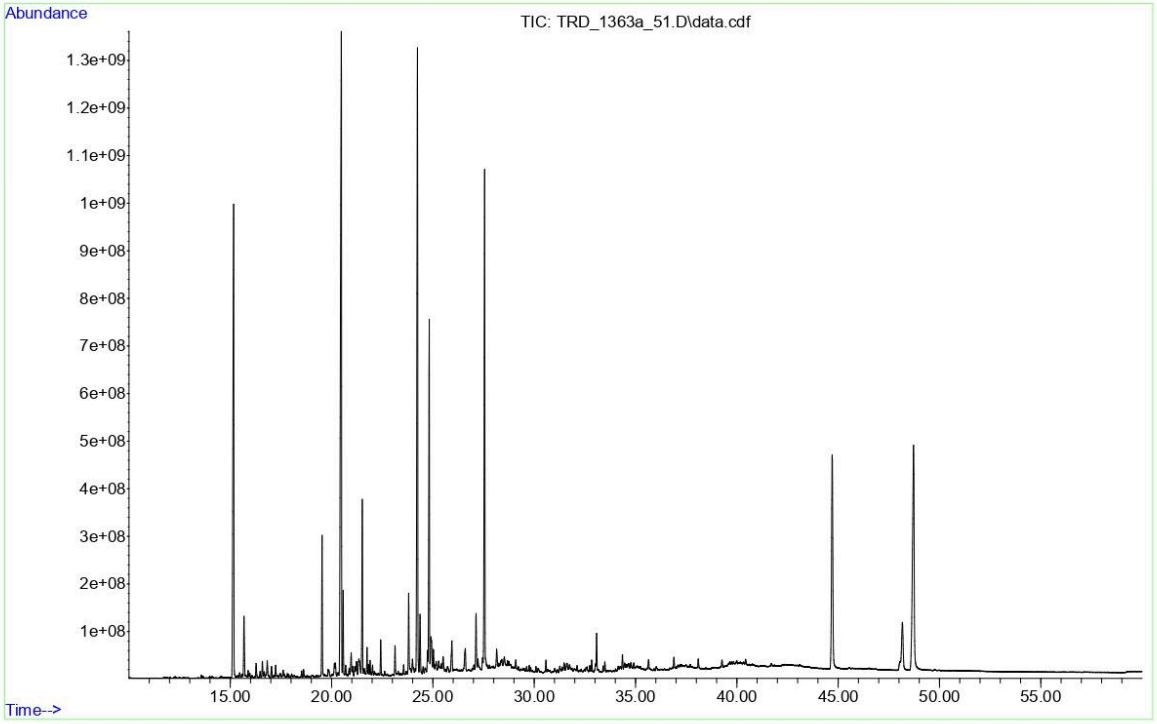
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



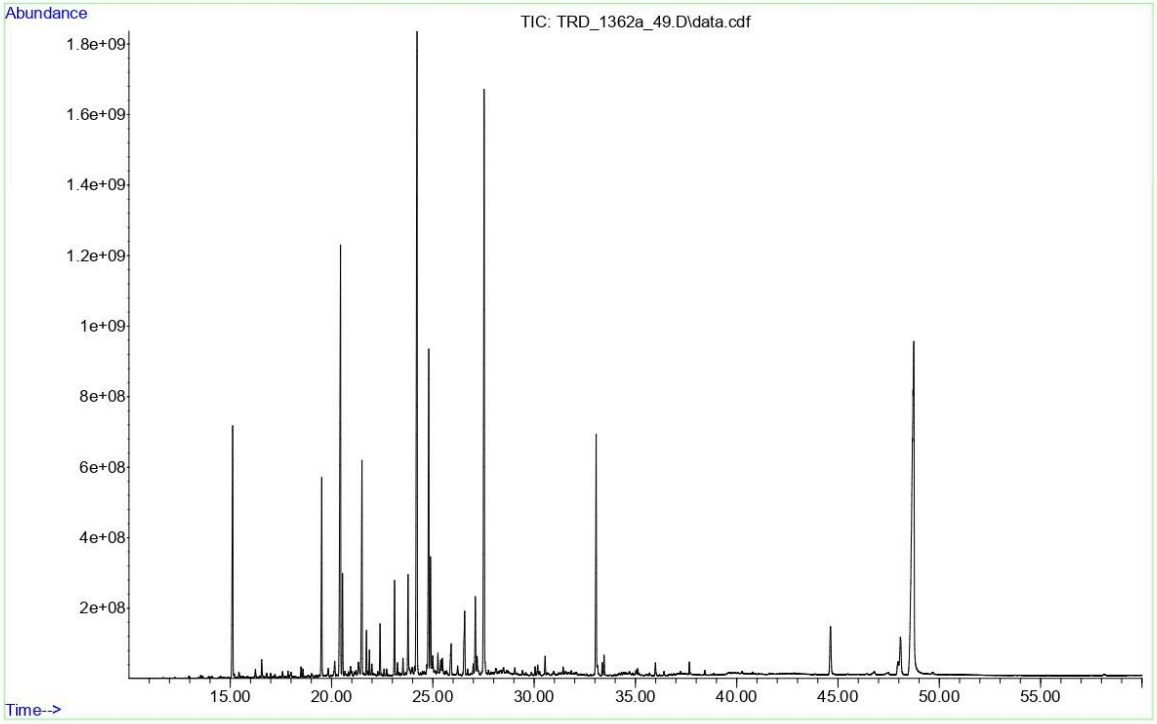
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



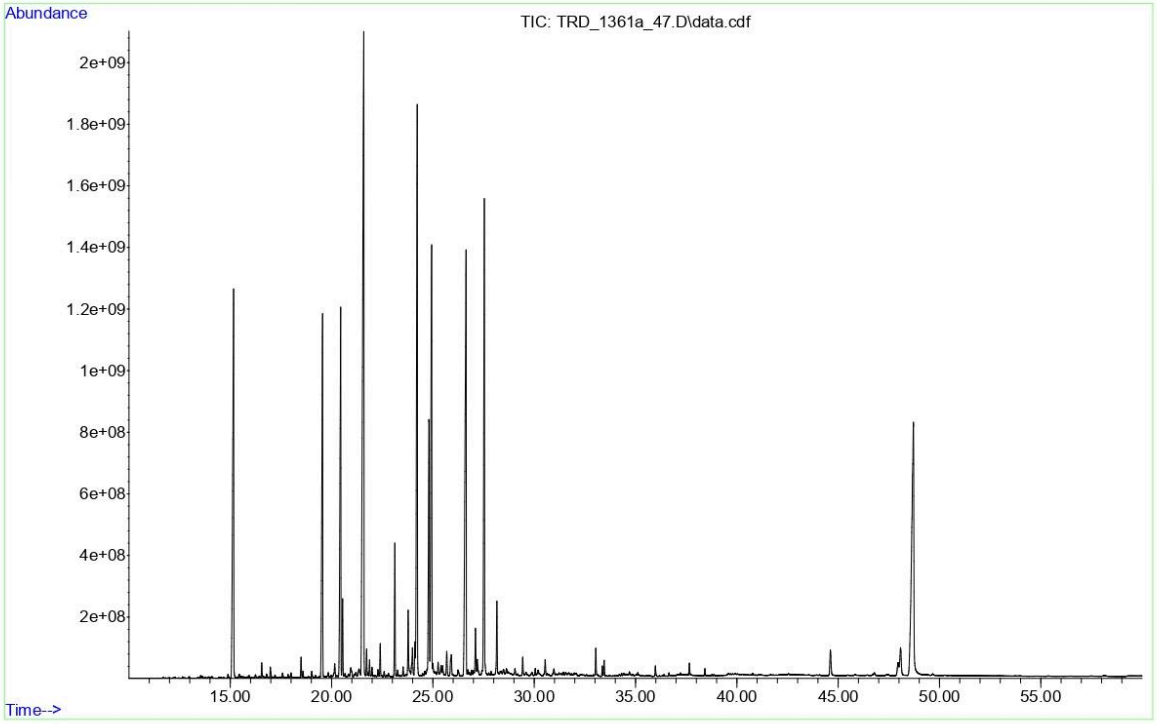
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



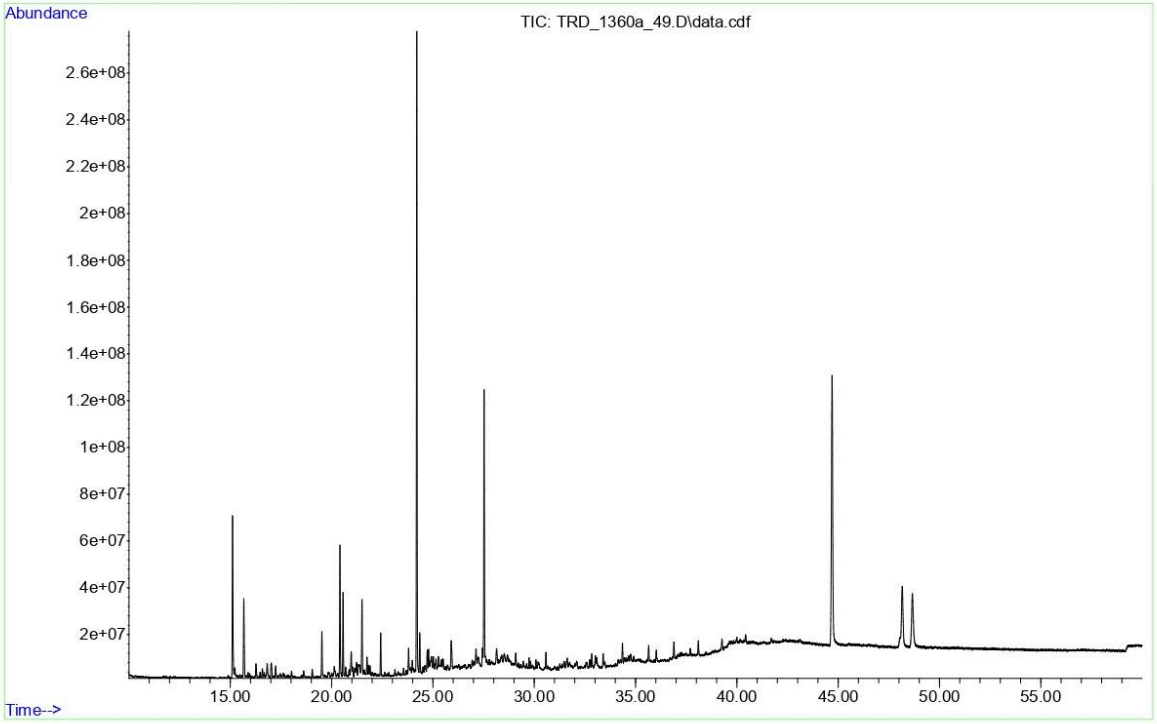
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



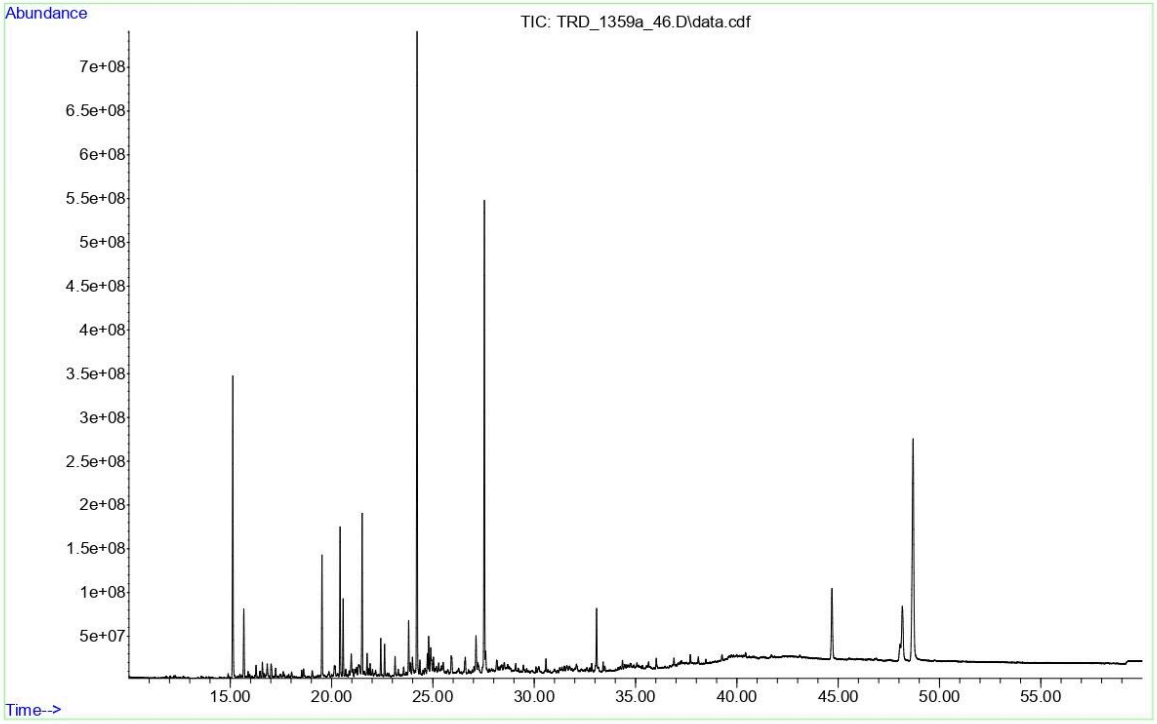
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



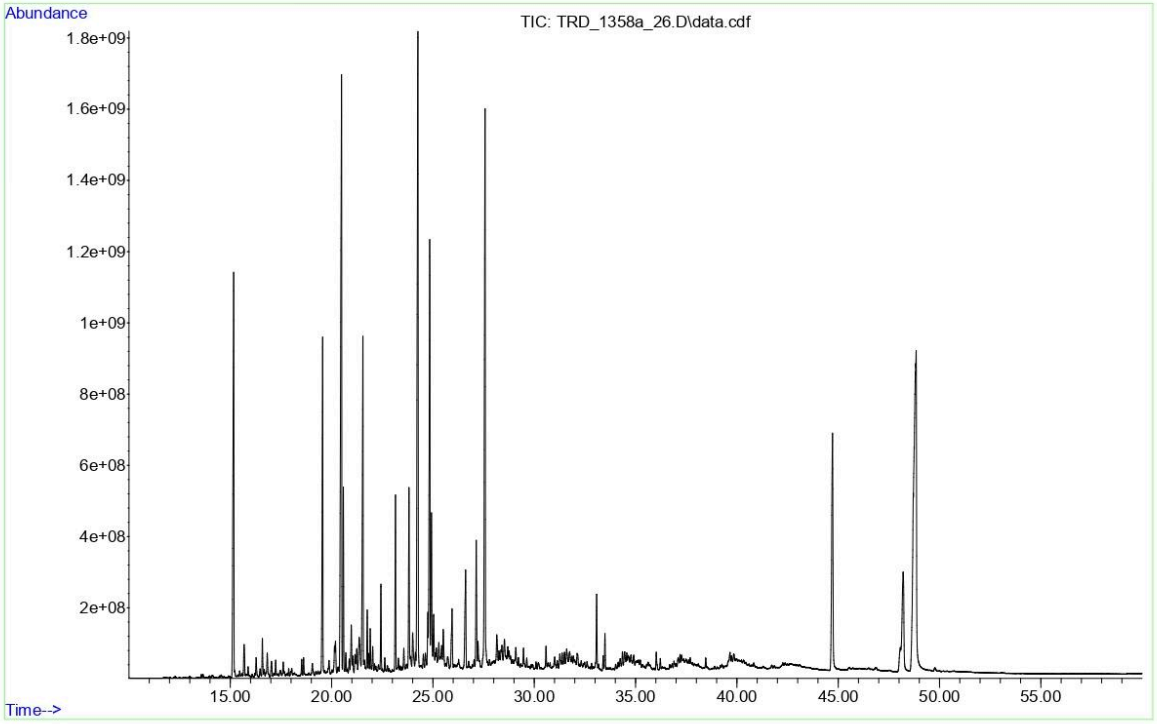
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



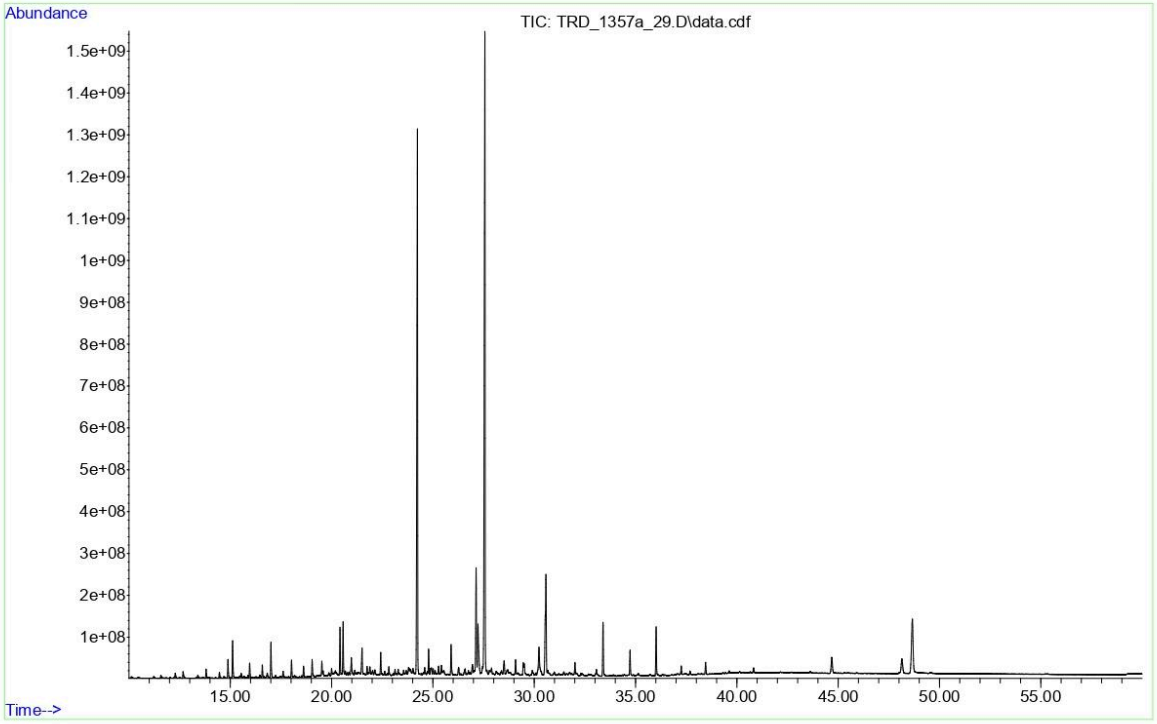
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



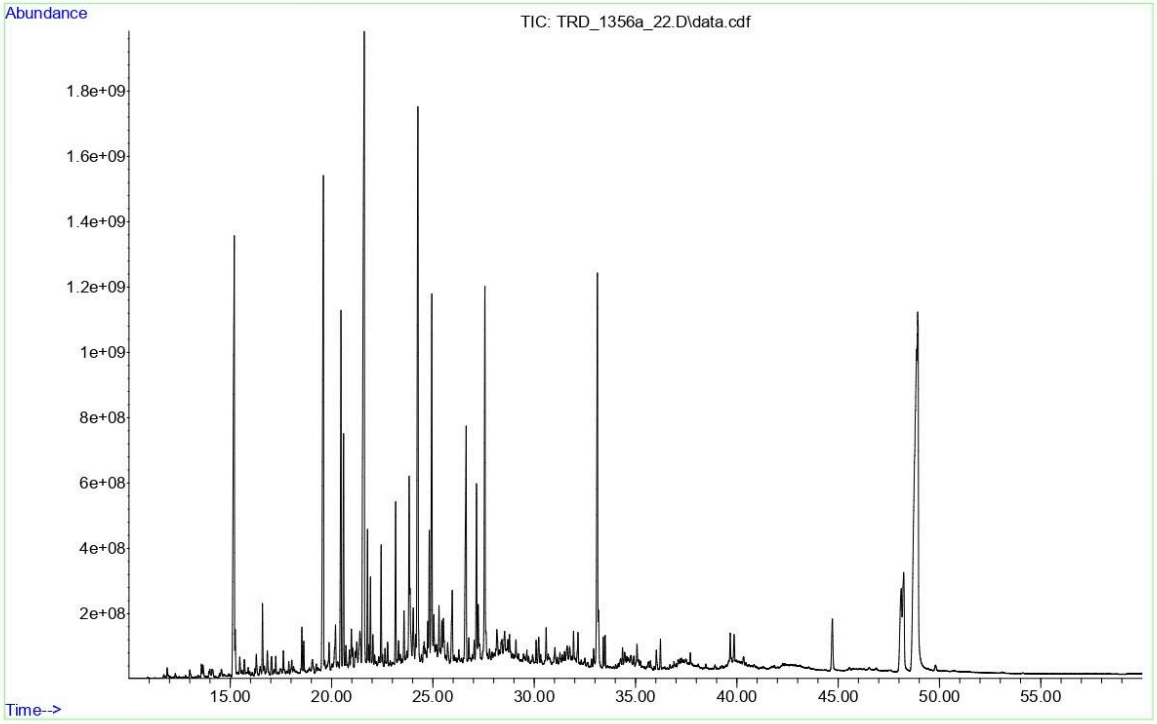
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



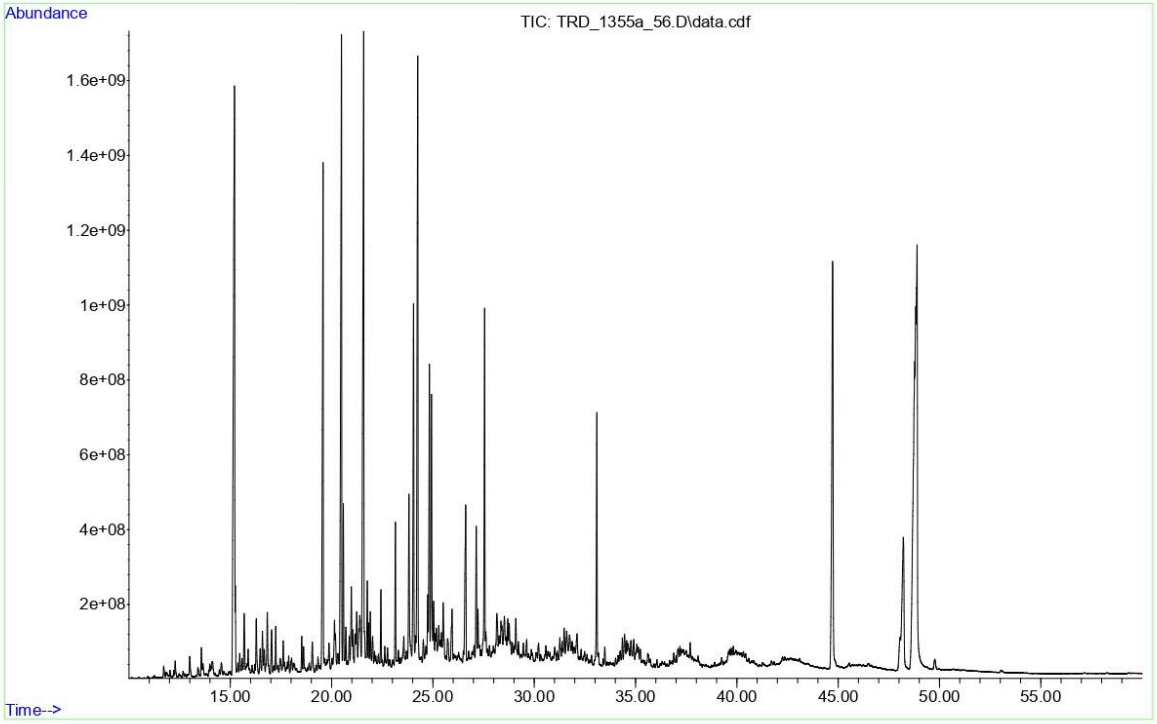
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



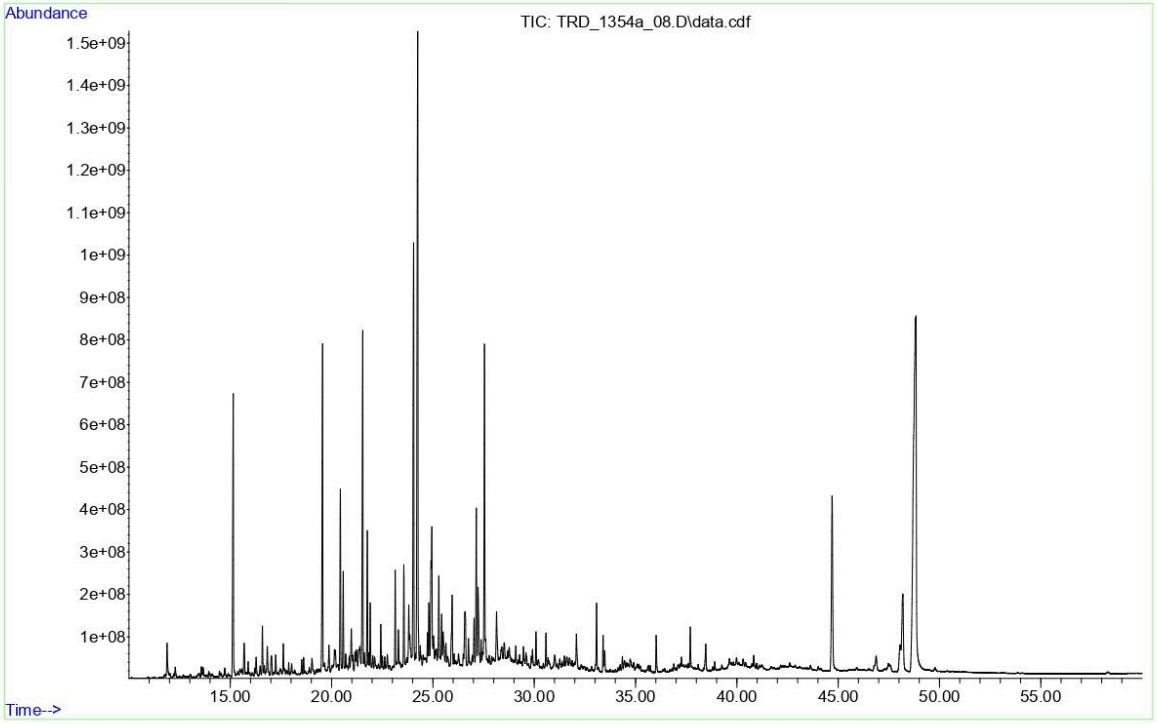
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



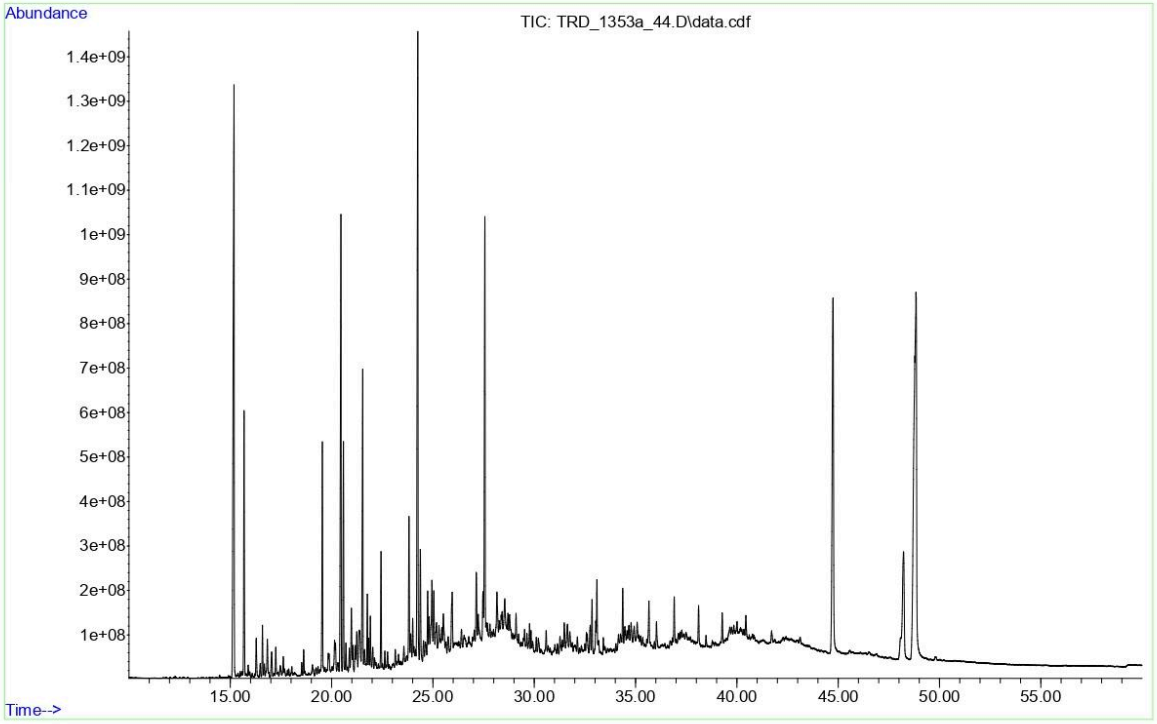
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



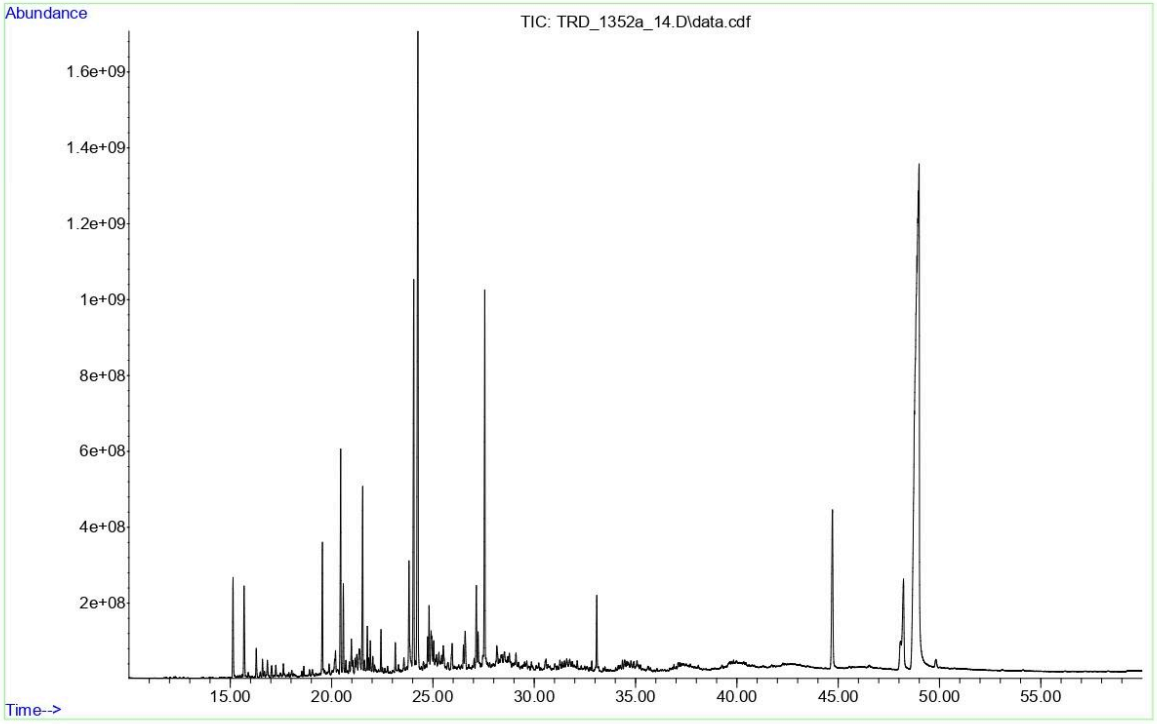
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



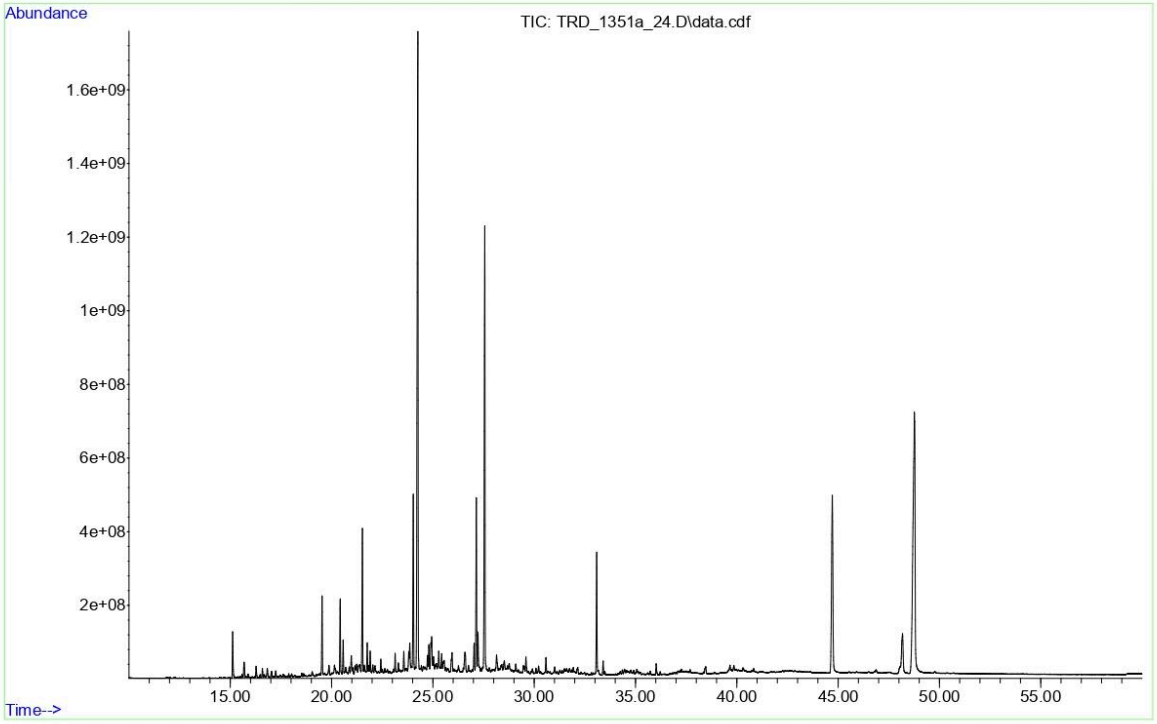
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



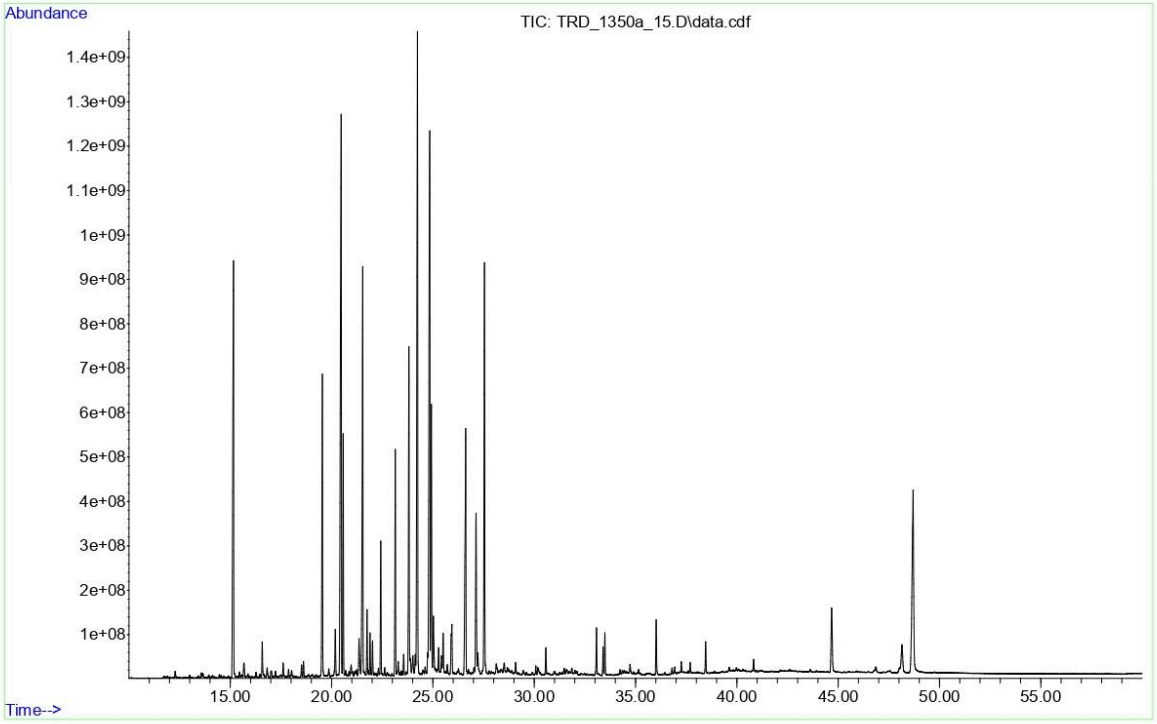
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



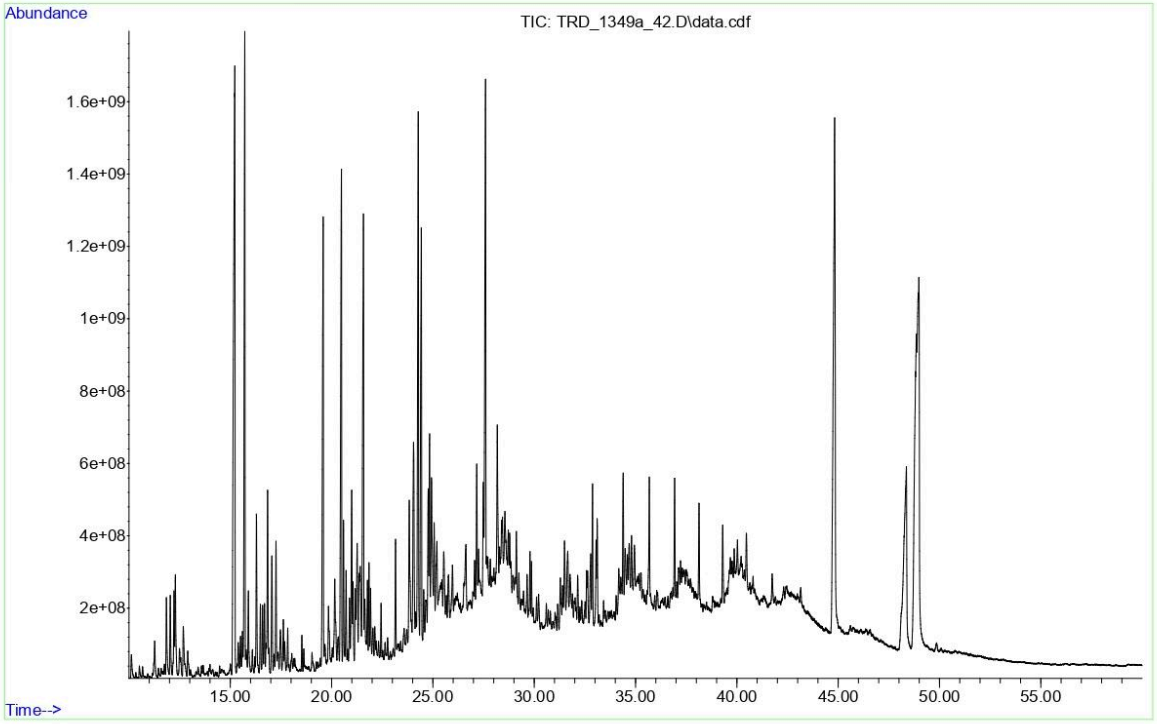
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



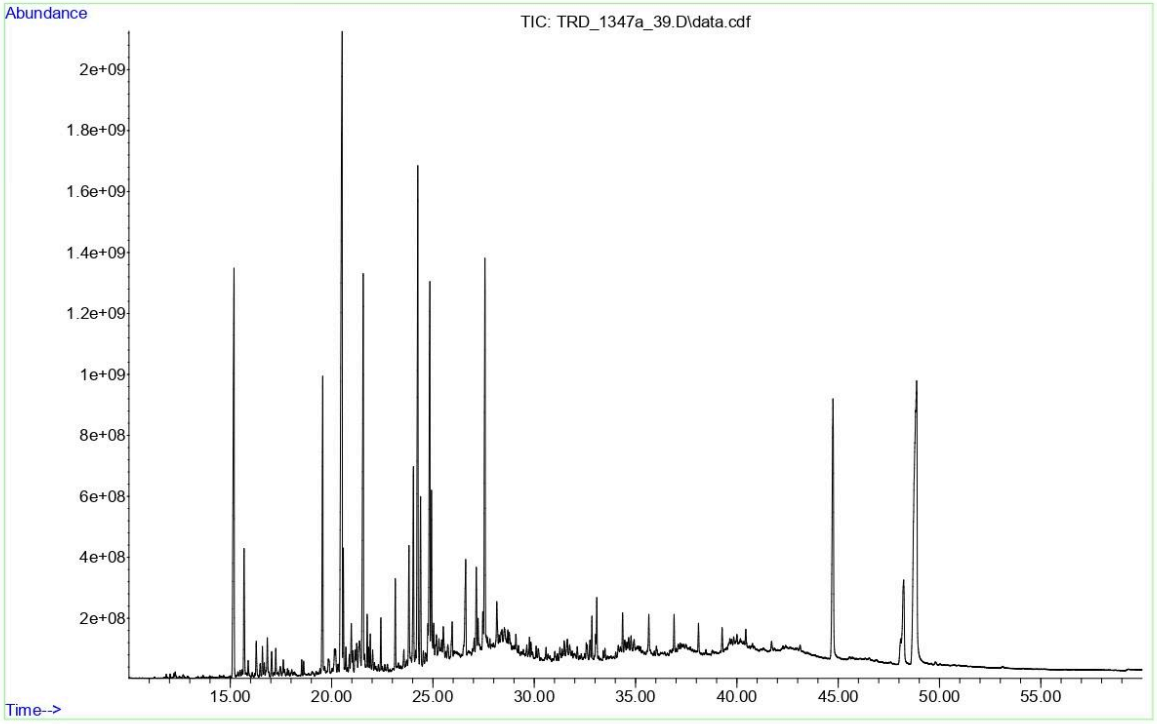
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Sample Name:
Misc Info :
Vial Number: 0



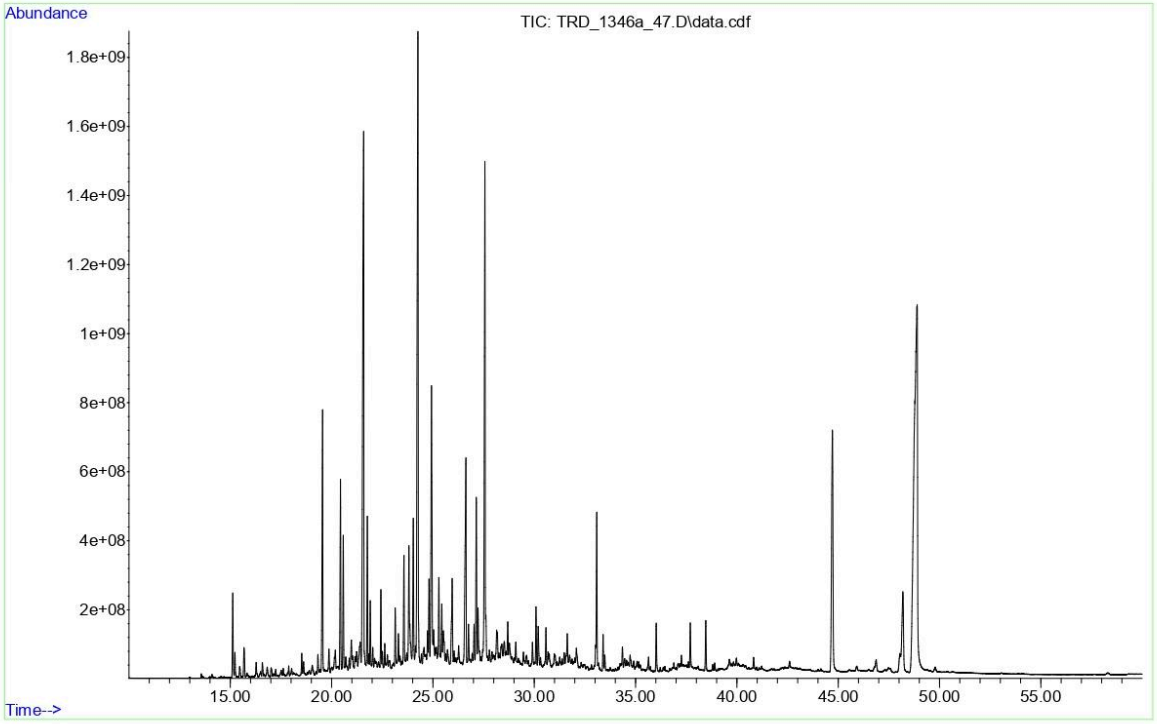
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



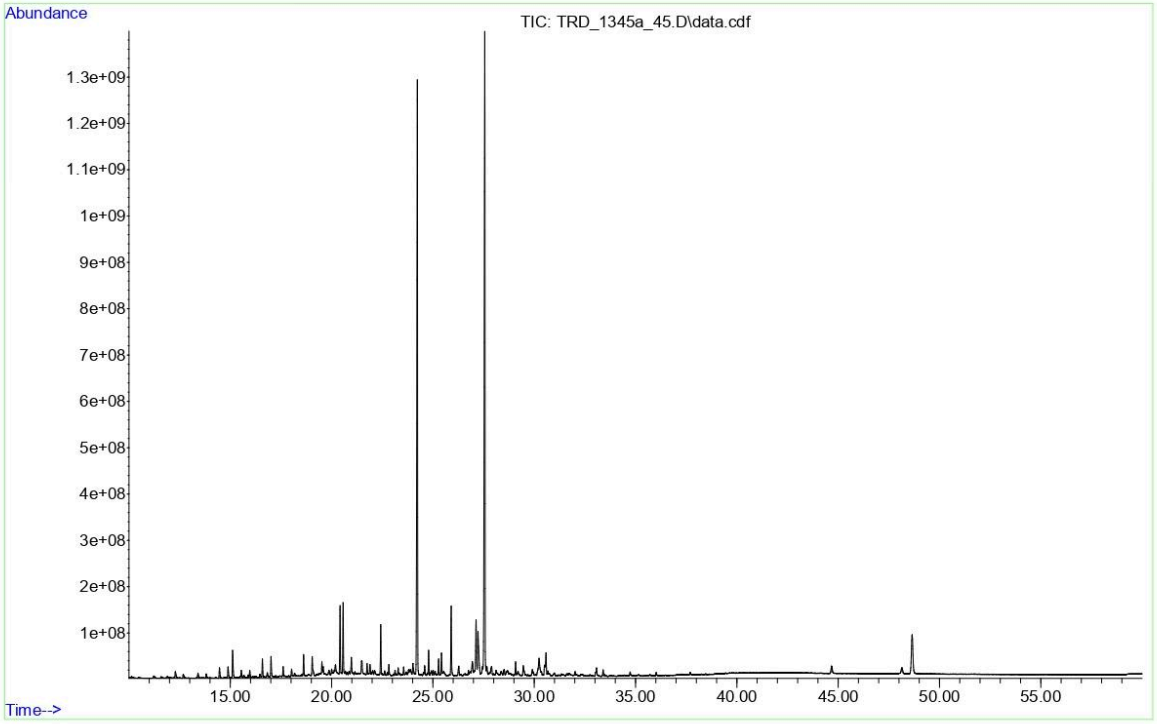
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



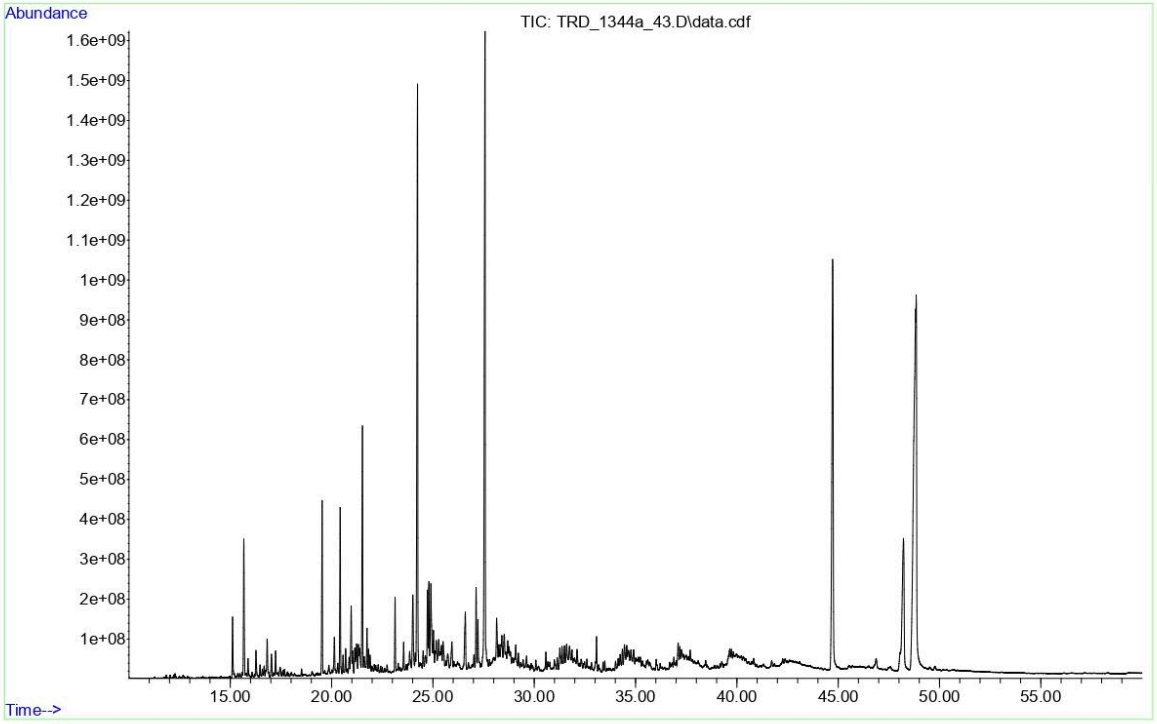
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



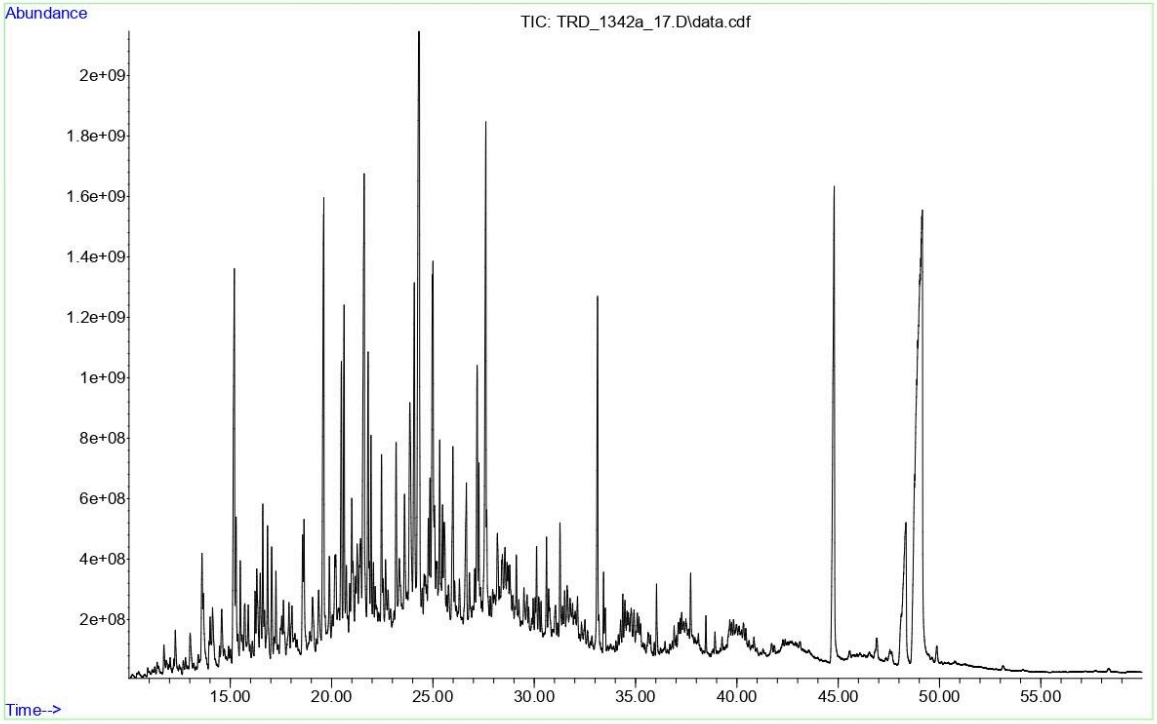
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



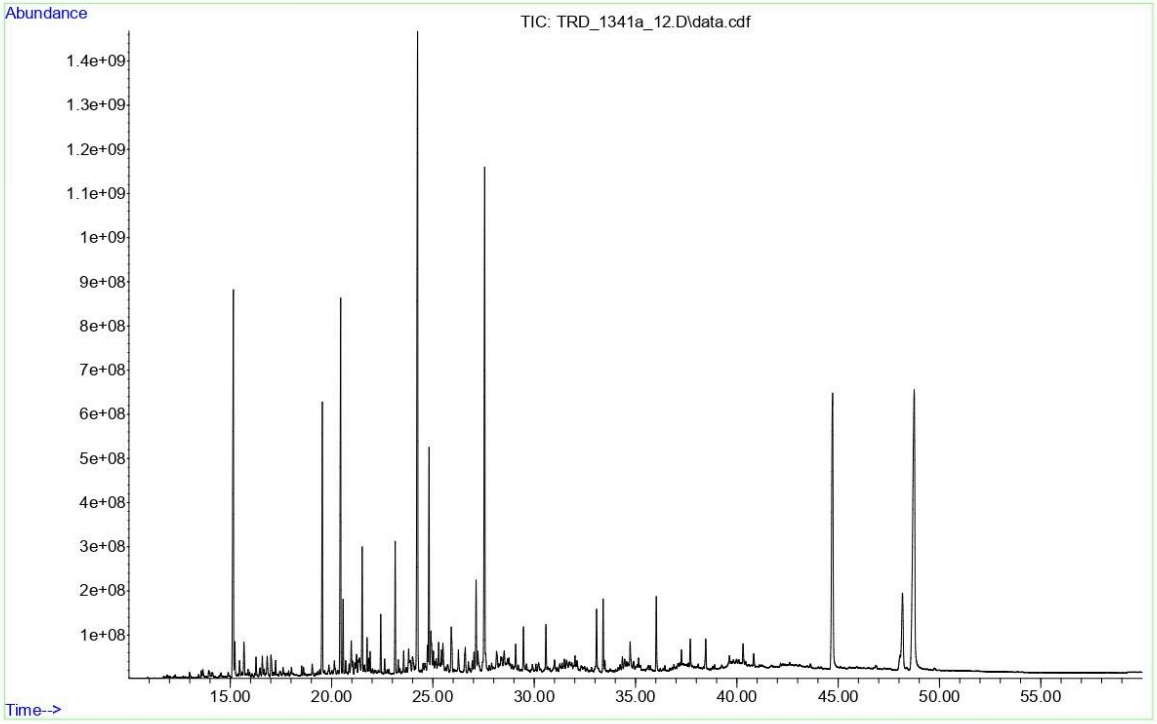
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



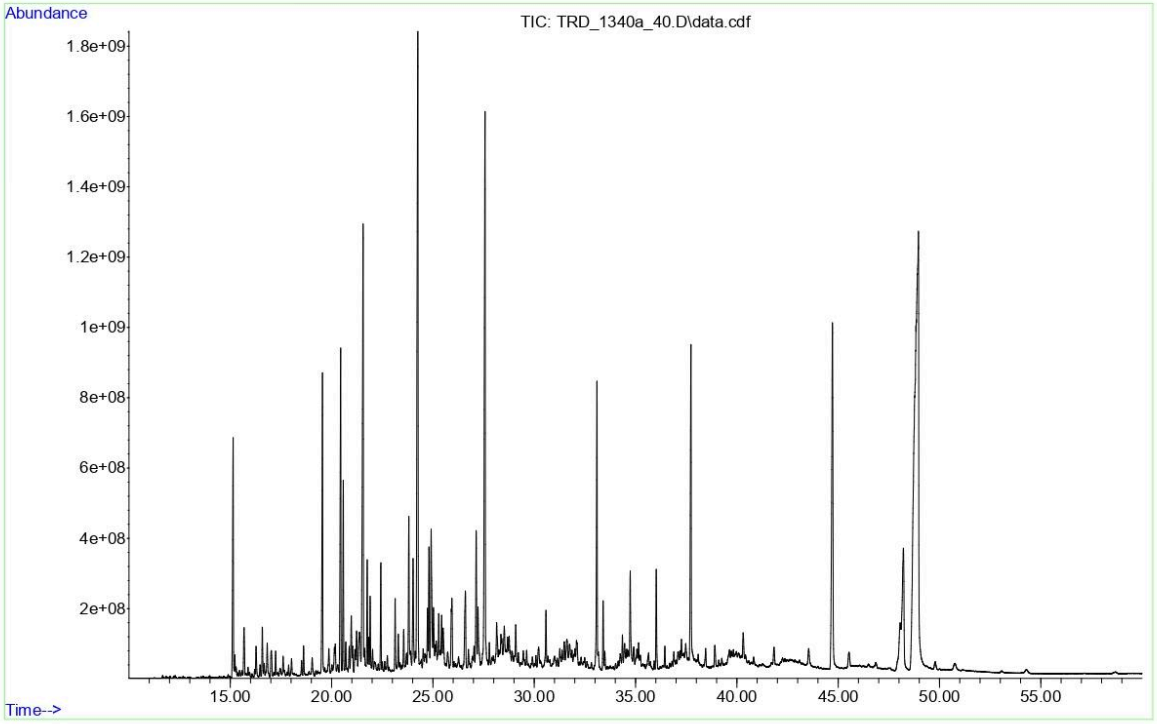
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



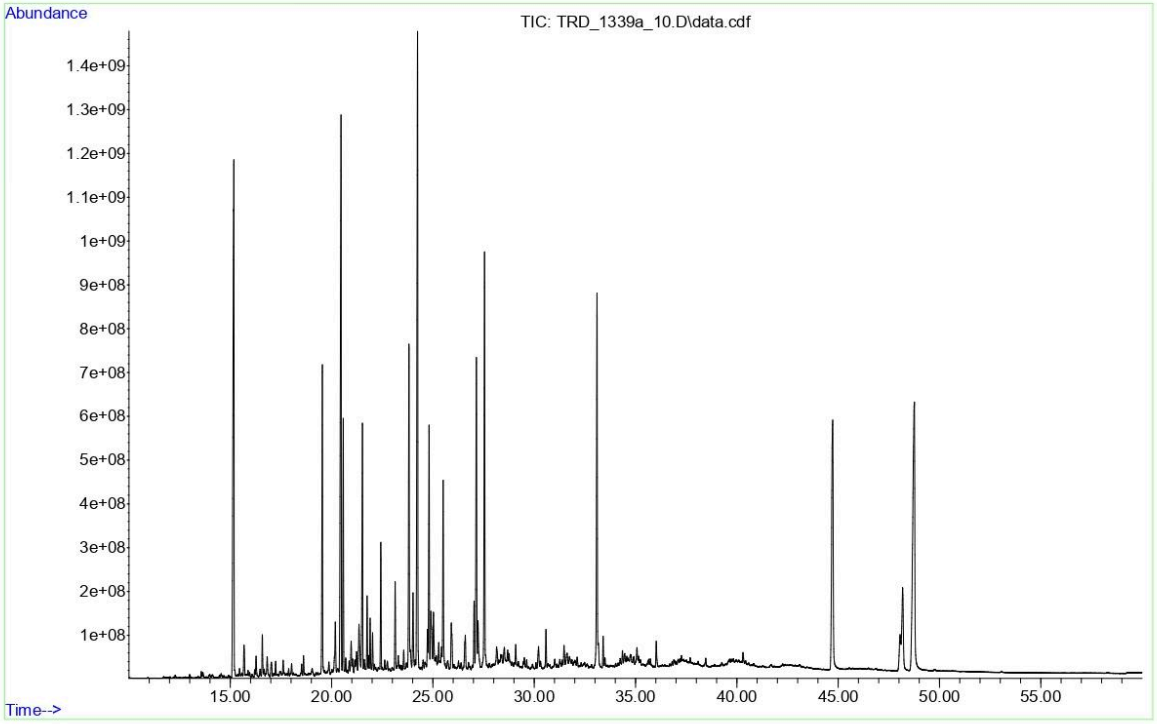
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



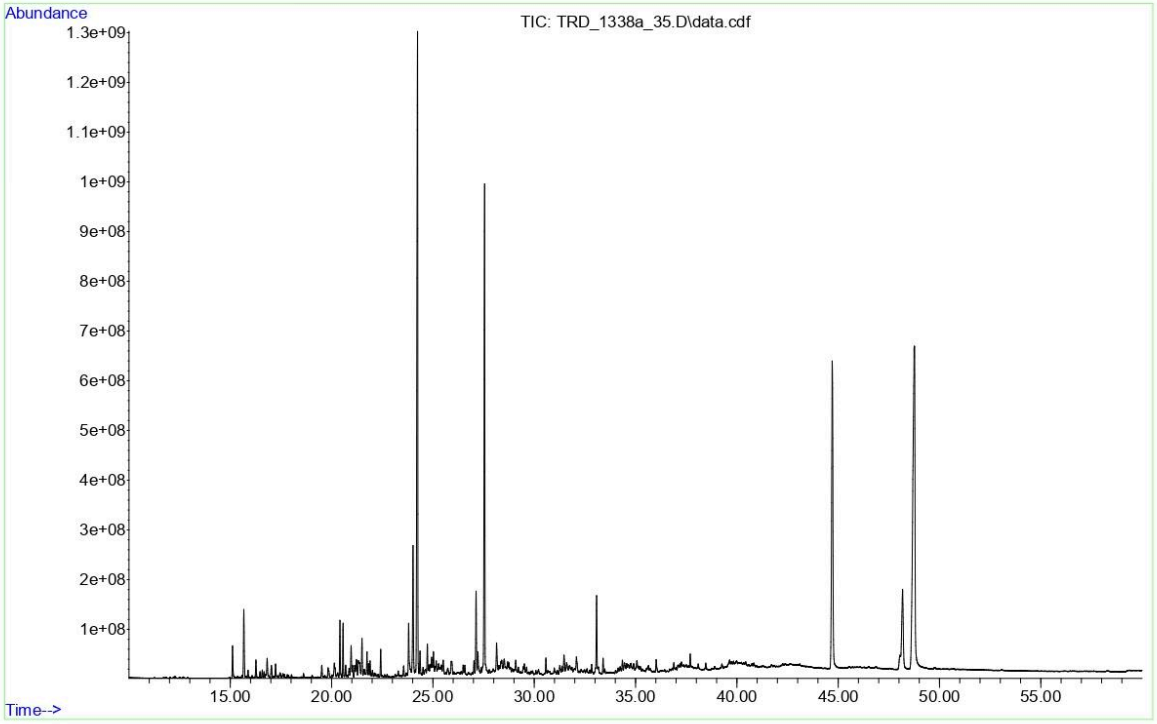
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



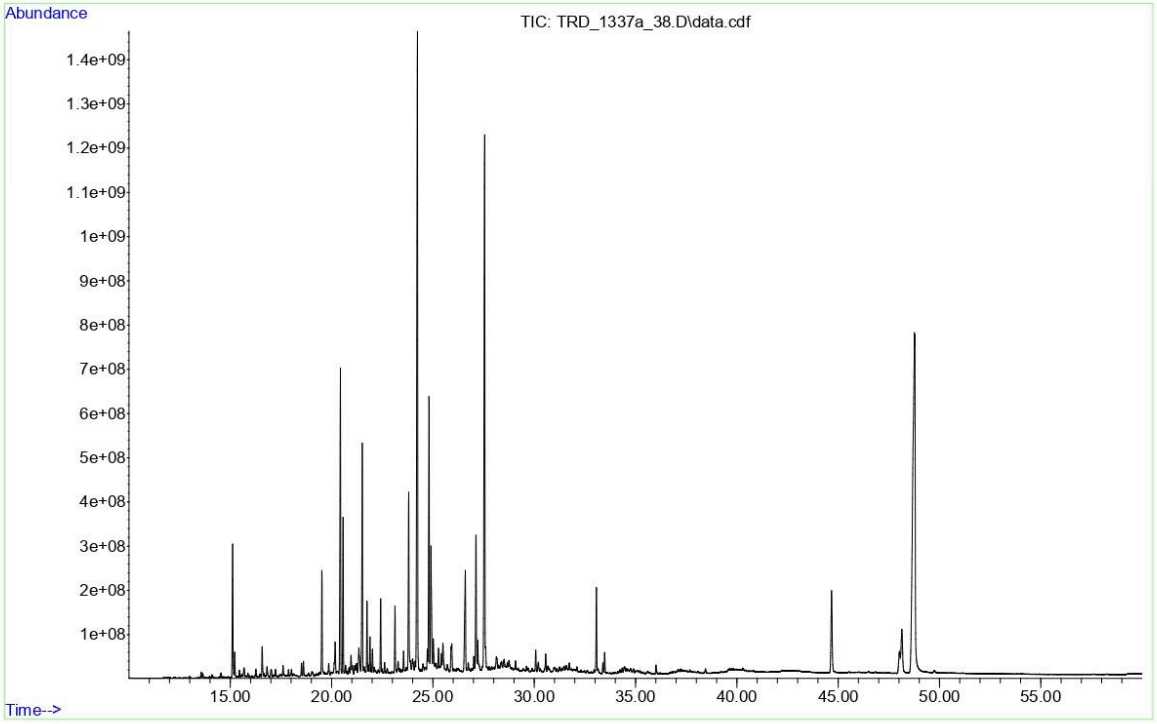
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



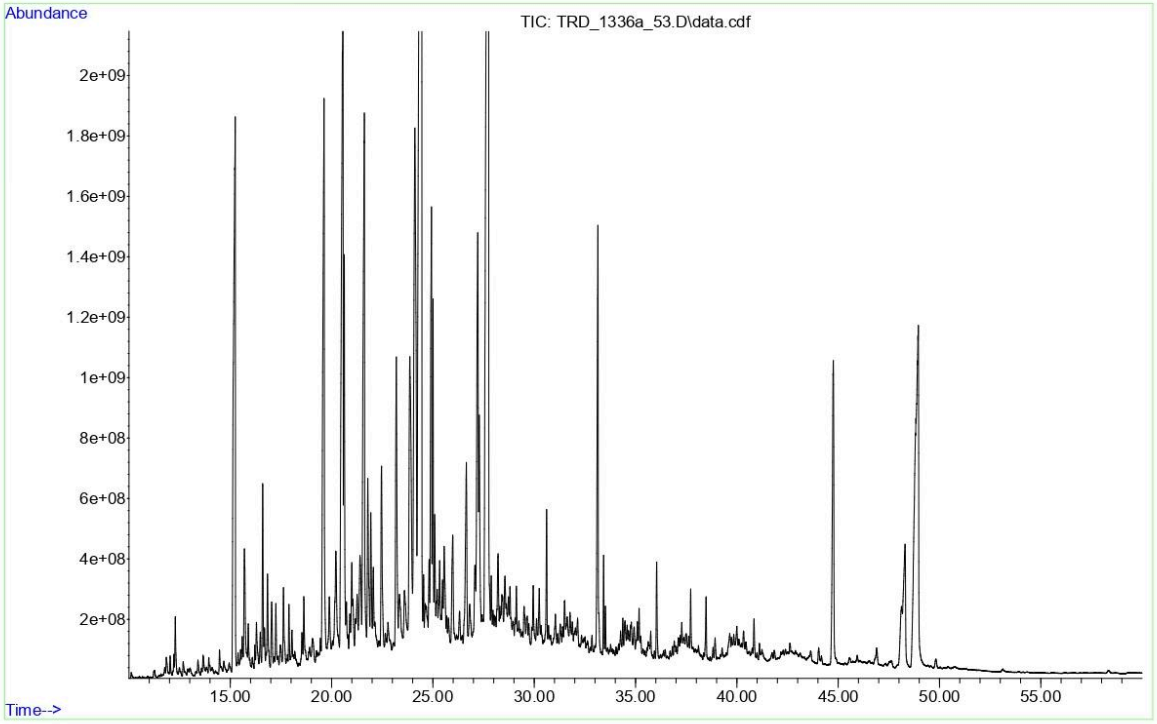
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



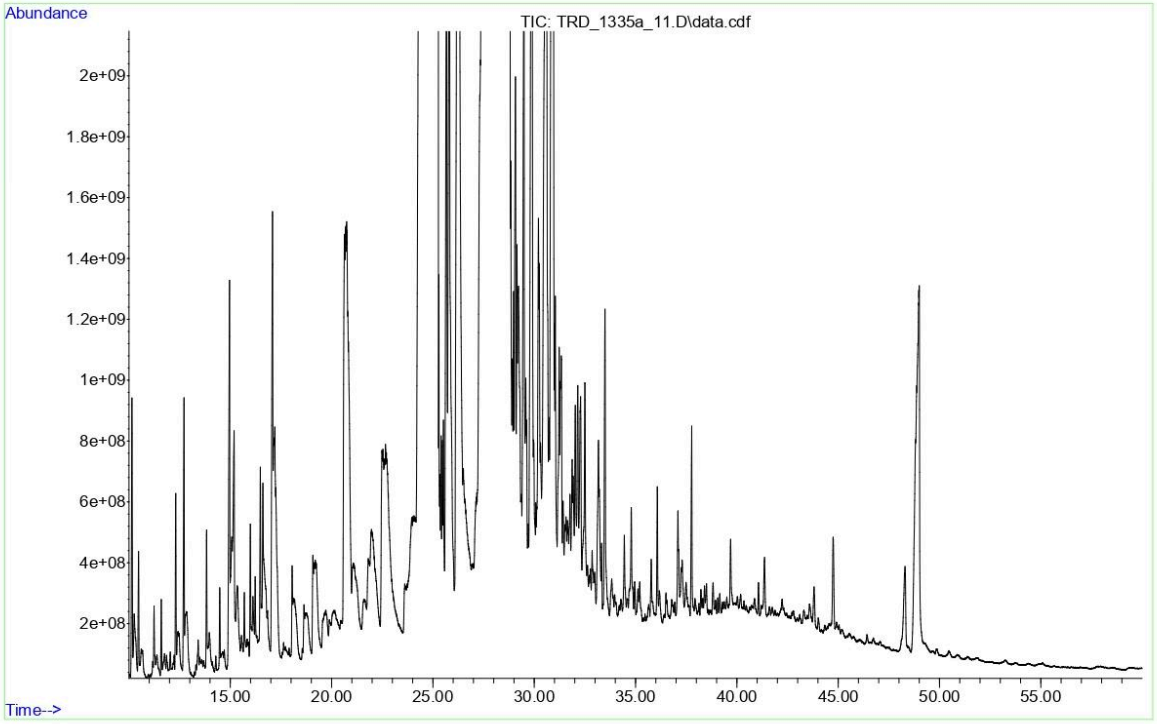
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



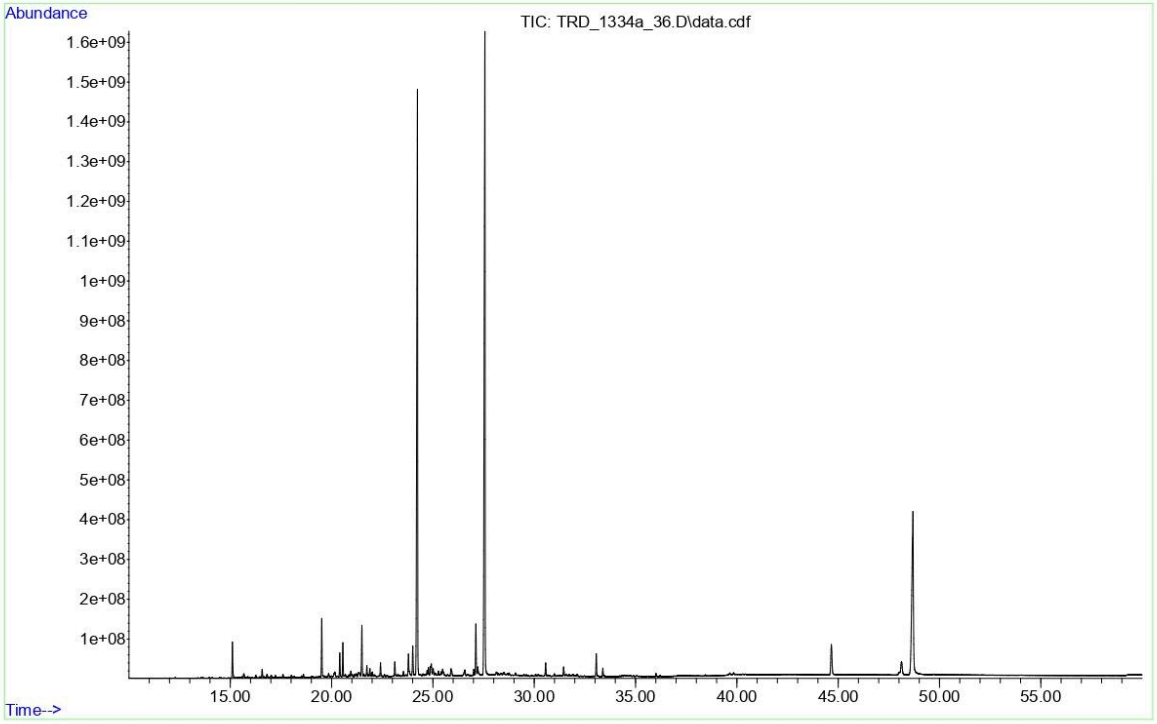
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Operator : SYSTEM
Acquired : 20221105012941+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



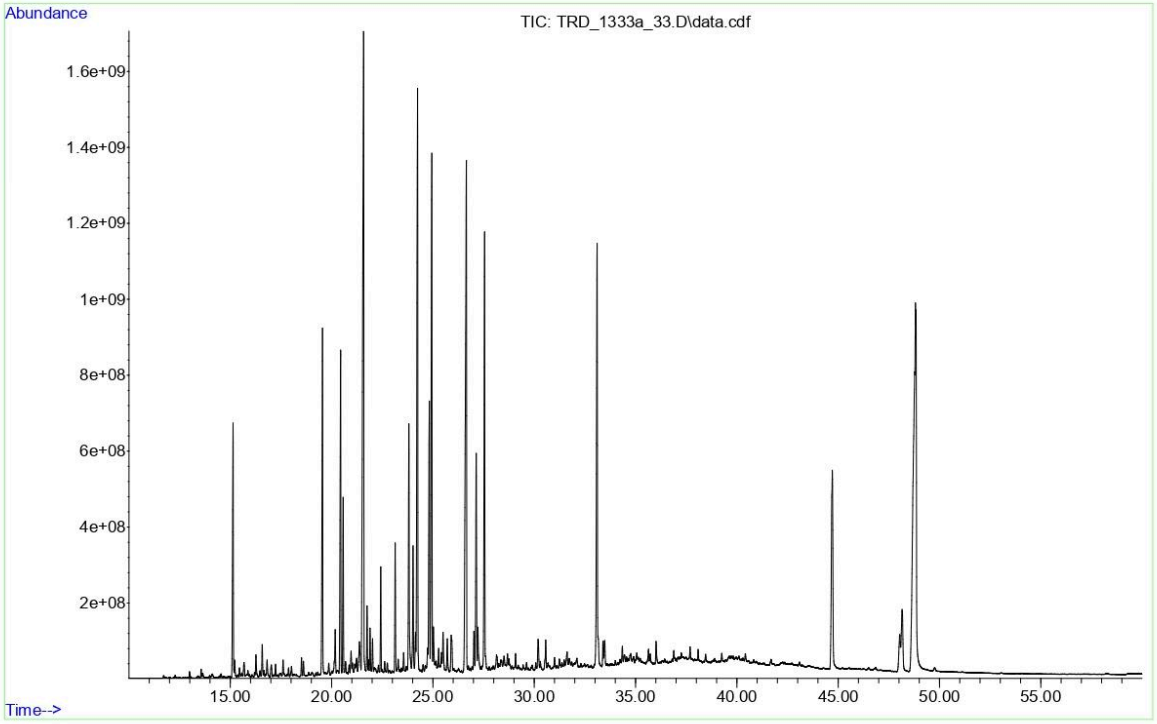
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Operator : SYSTEM
Acquired : 20221102225601+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



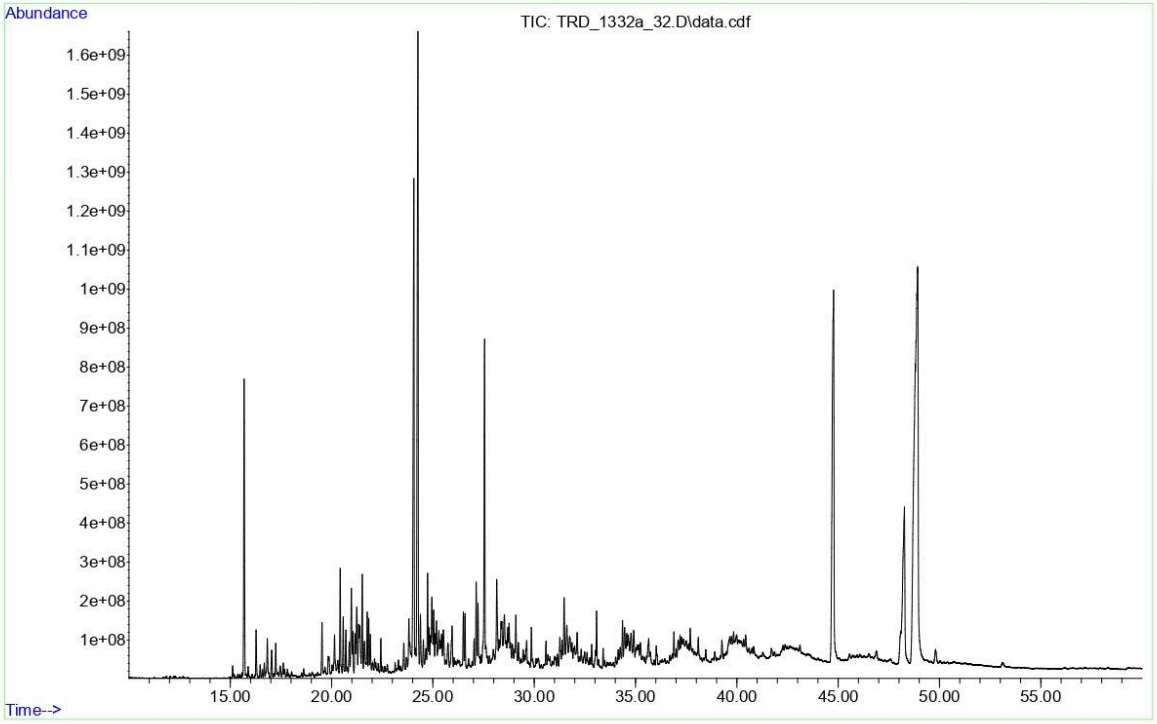
File :G:\Data by Site\ArroioTamarina\GCMS\TRD_1334a_36.D
Operator : SYSTEM
Acquired : 20221109052336+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



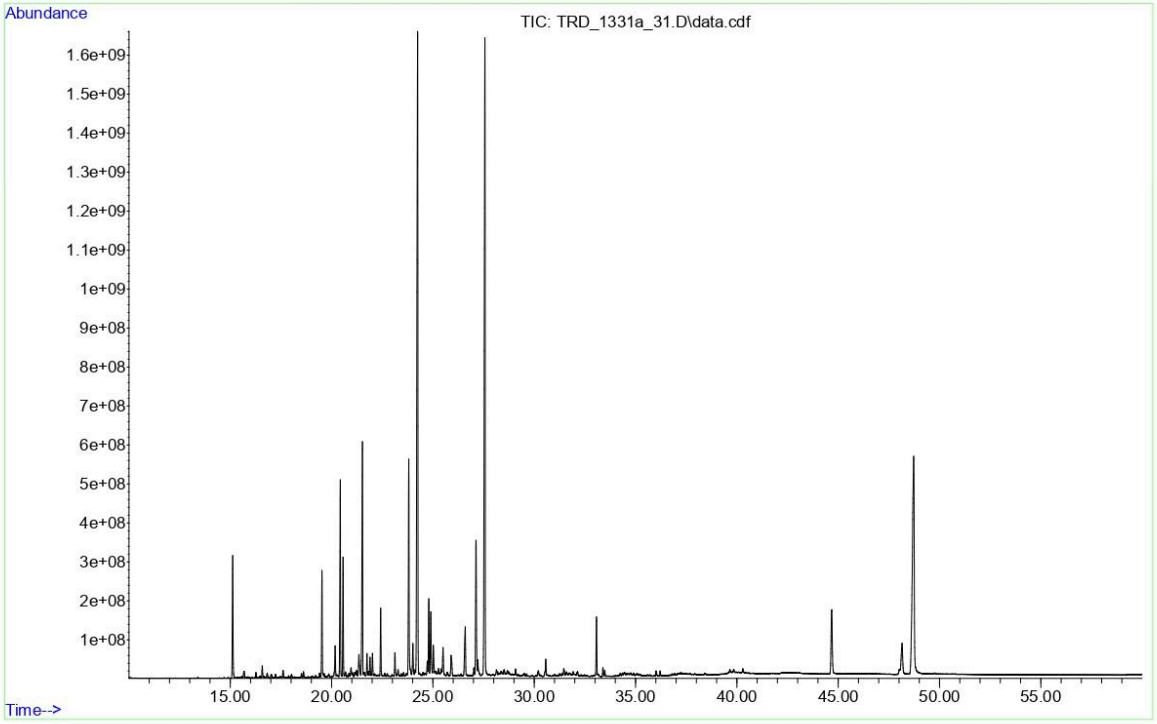
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Operator : SYSTEM
Acquired : 20221109014659+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



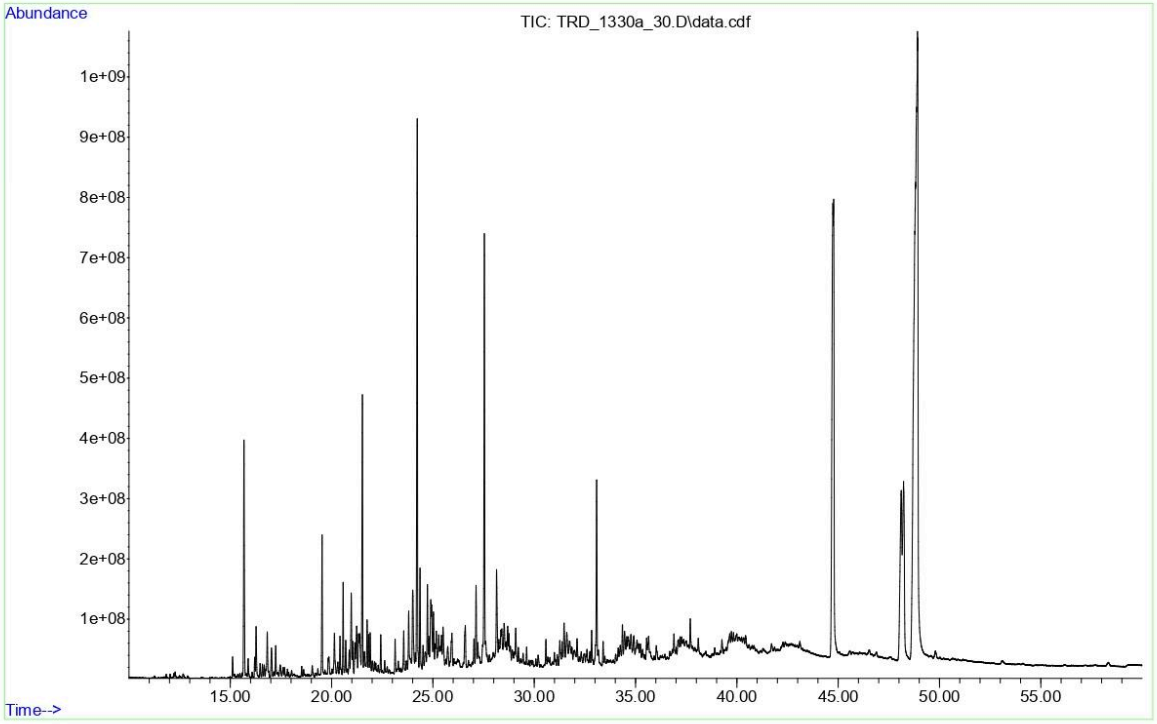
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Operator : SYSTEM
Acquired : 20221104001251+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



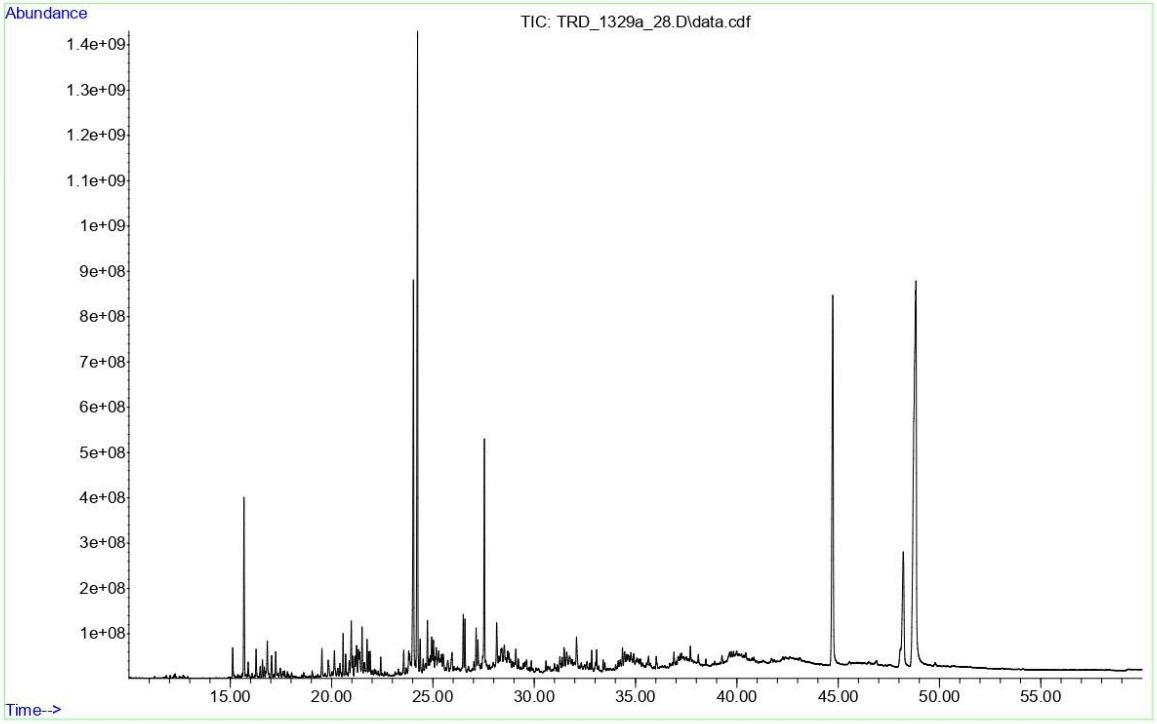
File :G:\Data by Site\ArroioTamarina\GCMS\TRD_1331a_31.D
Operator : SYSTEM
Acquired : 20221108232231+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Data by Site\ArroioTamarina\GCMS\TRD_1330a_30.D
Operator : SYSTEM
Acquired : 20221103214822+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



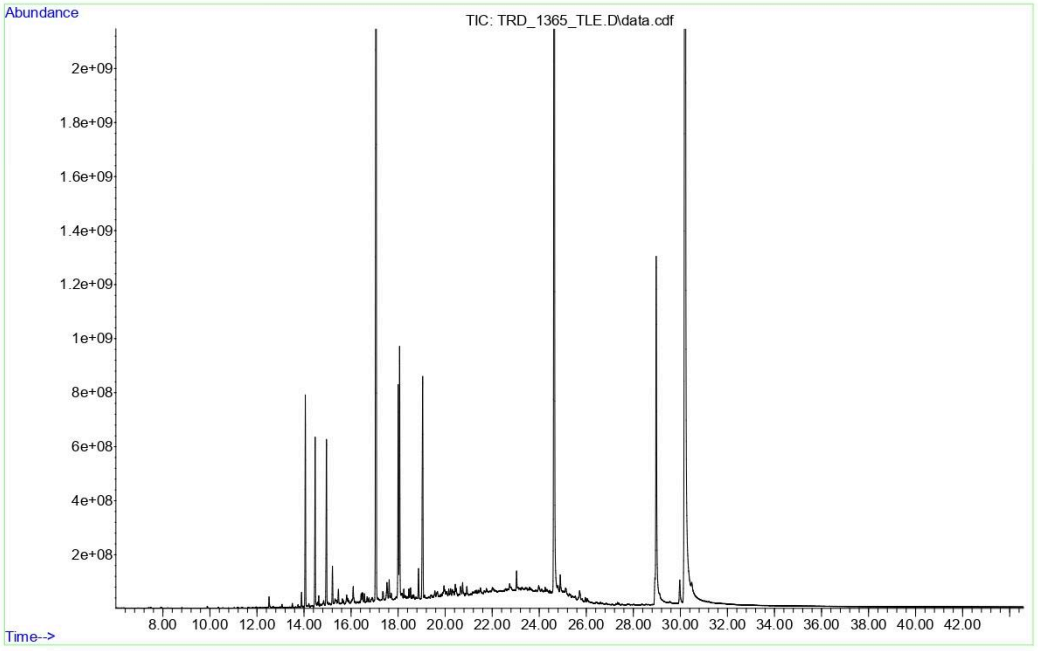
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Operator : SYSTEM
Acquired : 20221103192349+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



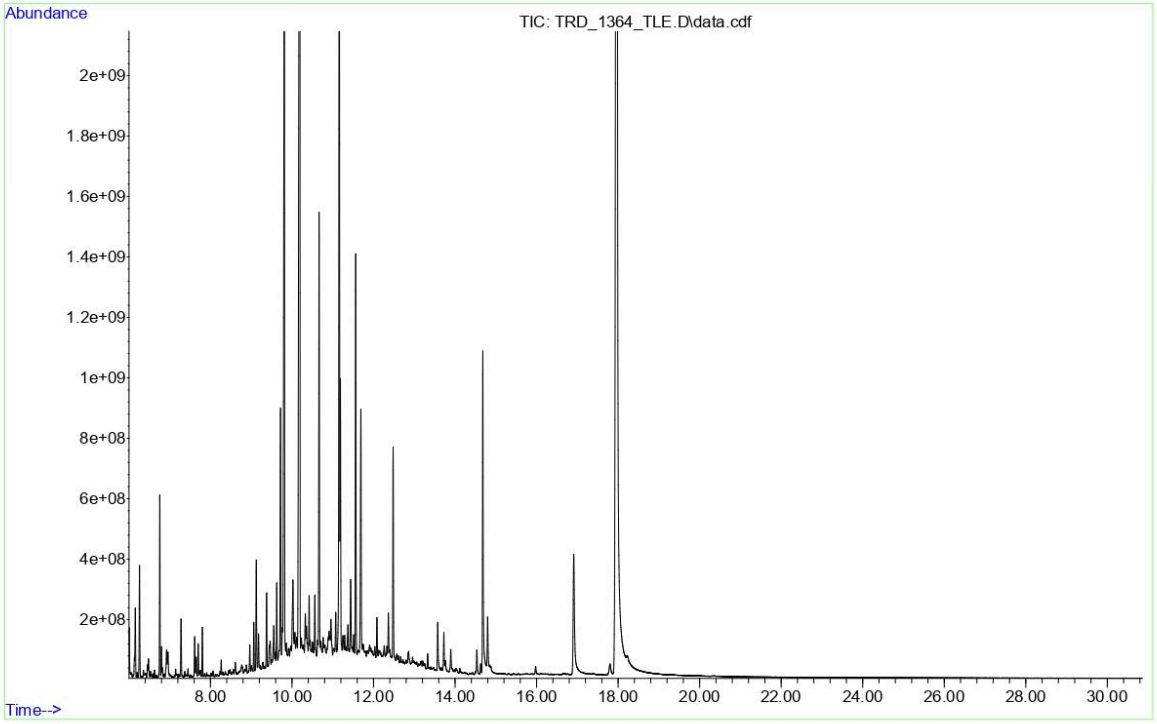
Arroio Tamarina 2

Total lipid extraction / TMS chromatograms

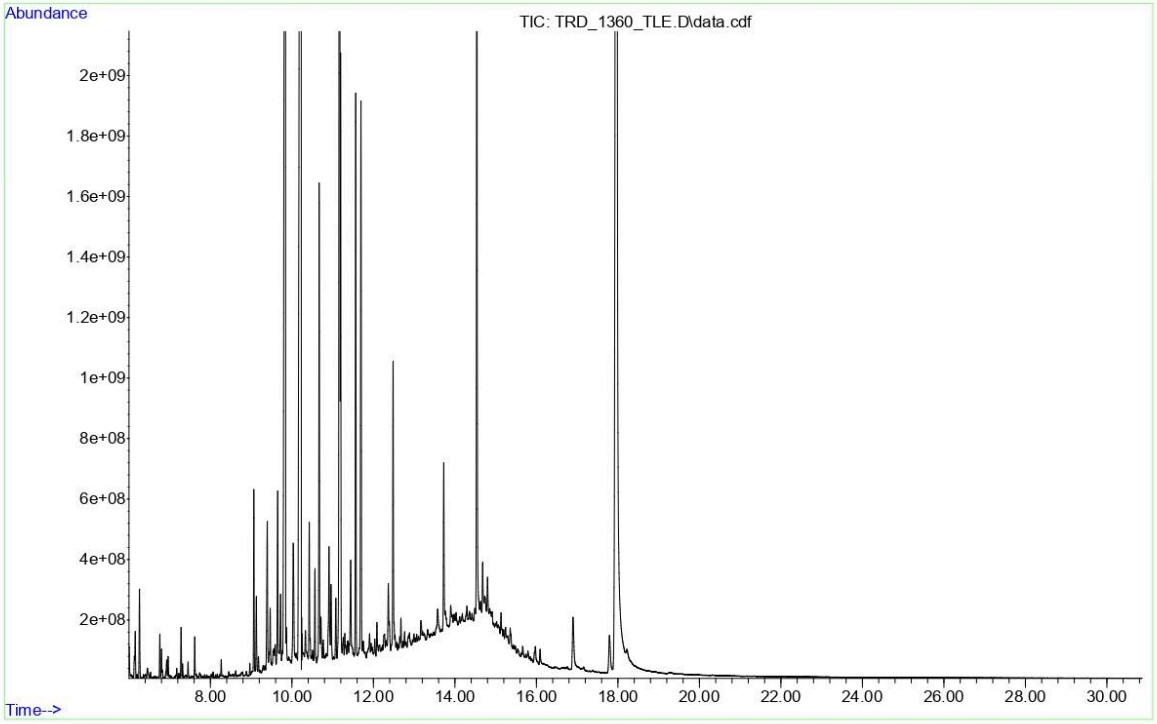
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File       :G:\Solvent Extraction\TRD_1365_TLE.D
Operator   : Xcalibur_System
Acquired   : 20231011205237+0000 using AcqMethod
Instrument :    LCQ
Sample Name:
Misc Info  :
Vial Number: 0
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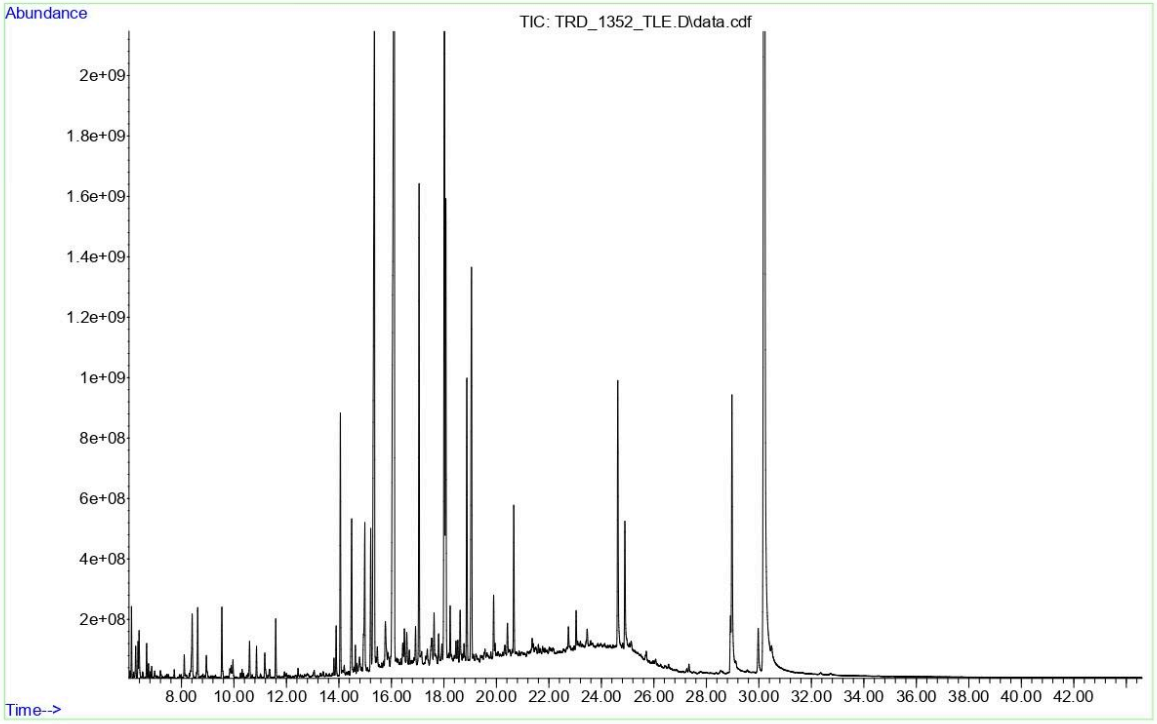
File :G:\Solvent Extraction\TRD_1364_TLE.D
Operator : Xcalibur_System
Acquired : 20231012042732+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



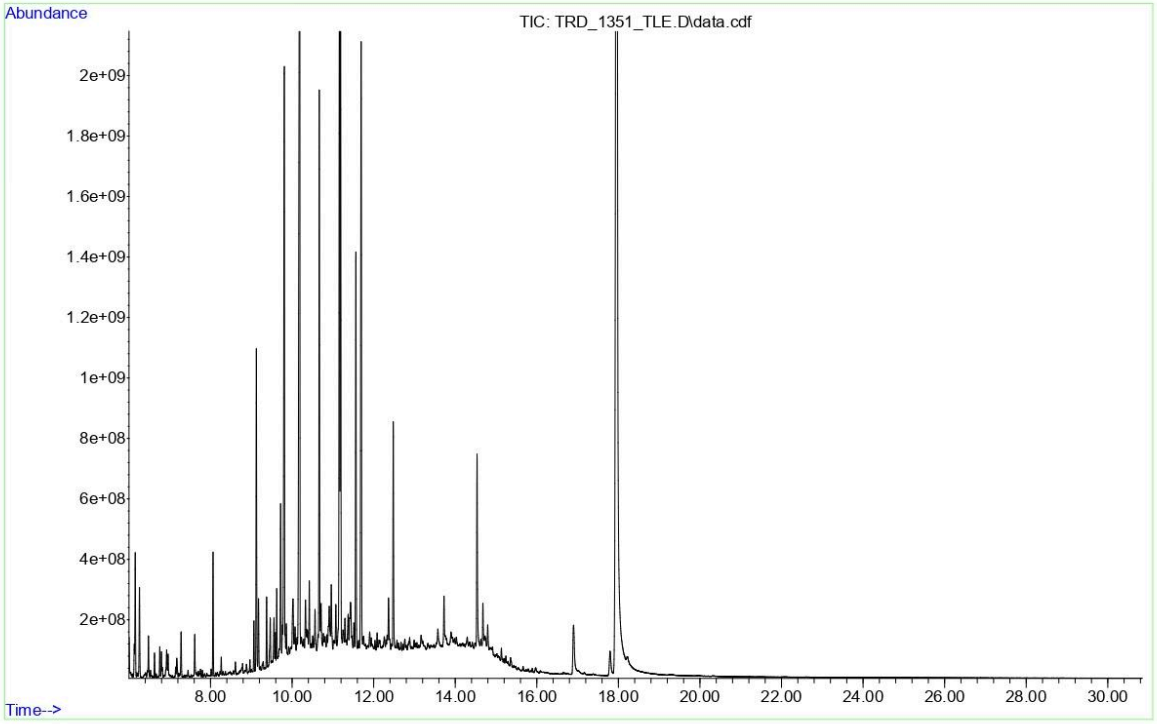
File :G:\Solvent Extraction\TRD_1360_TLE.D
Operator : Xcalibur_System
Acquired : 20231012015305+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



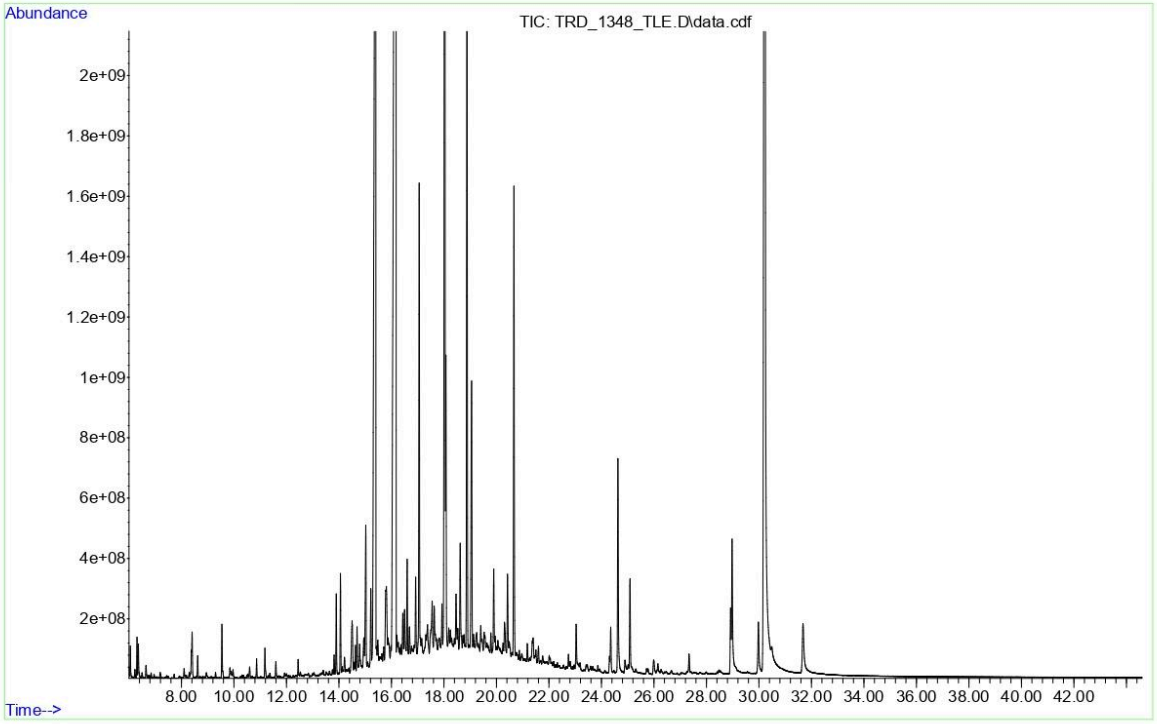
File :G:\Solvent Extraction\TRD_1352_TLE.D
Operator : Xcalibur_System
Acquired : 20231011214459+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



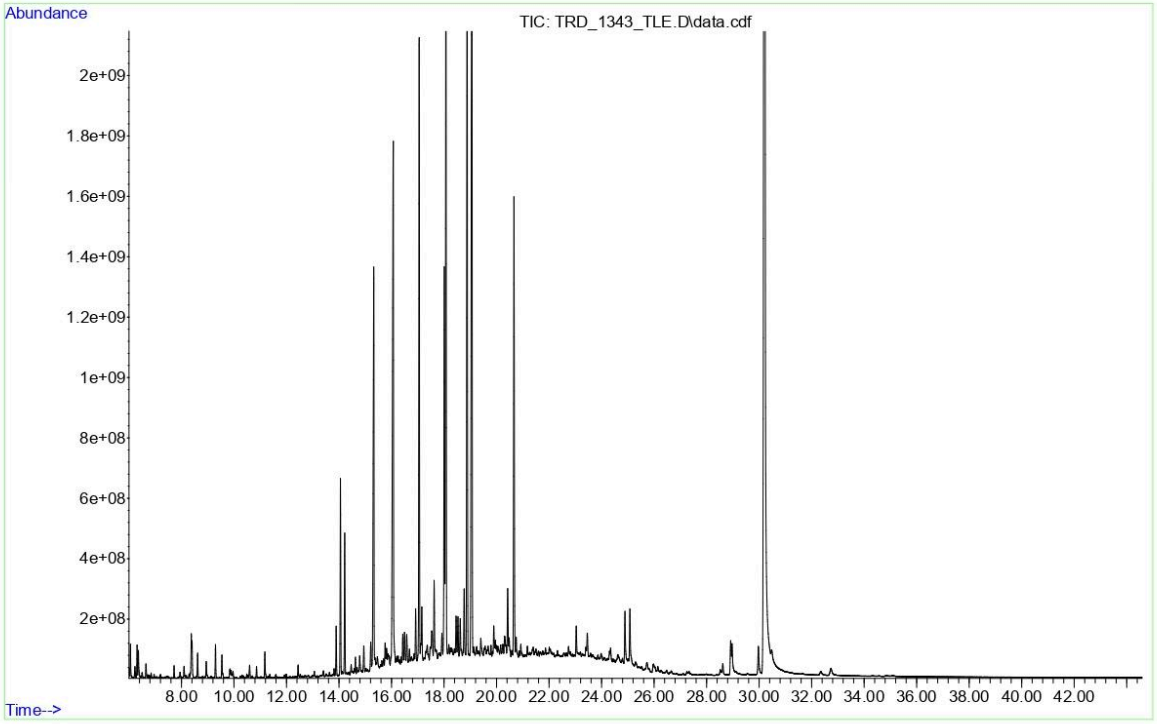
File :G:\Solvent Extraction\TRD_1351_TLE.D
Operator : Xcalibur_System
Acquired : 20231012023141+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



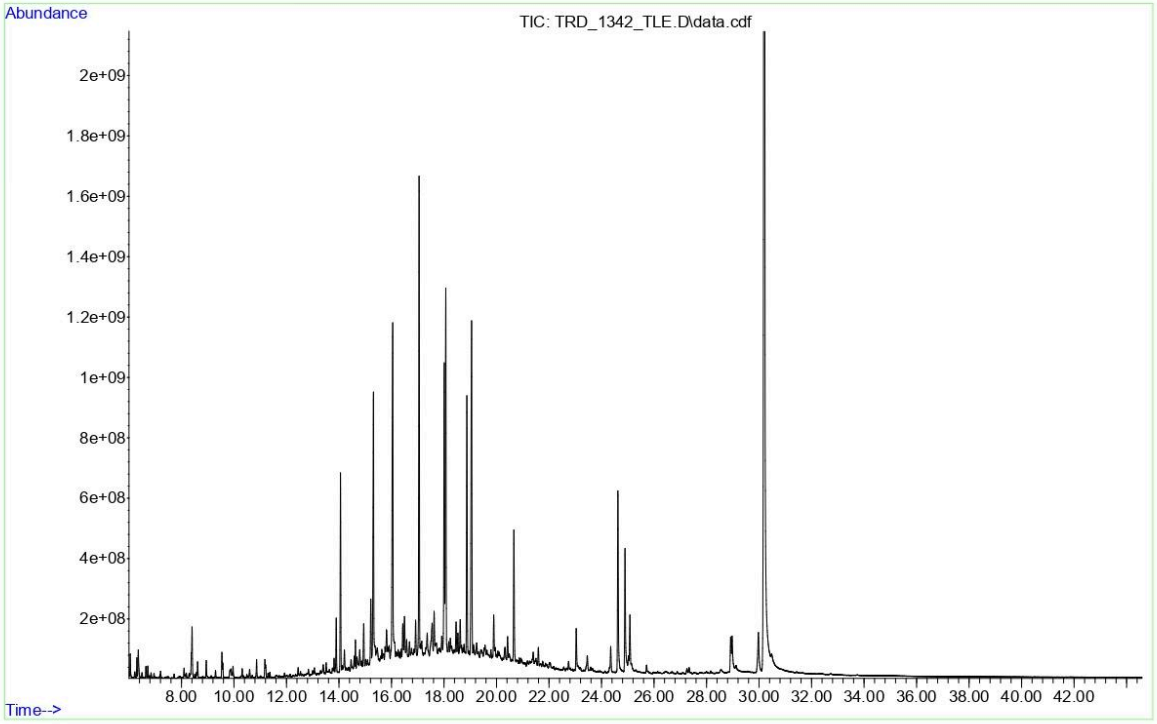
File :G:\Solvent Extraction\TRD_1348_TLE.D
Operator : Xcalibur_System
Acquired : 20231011155226+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



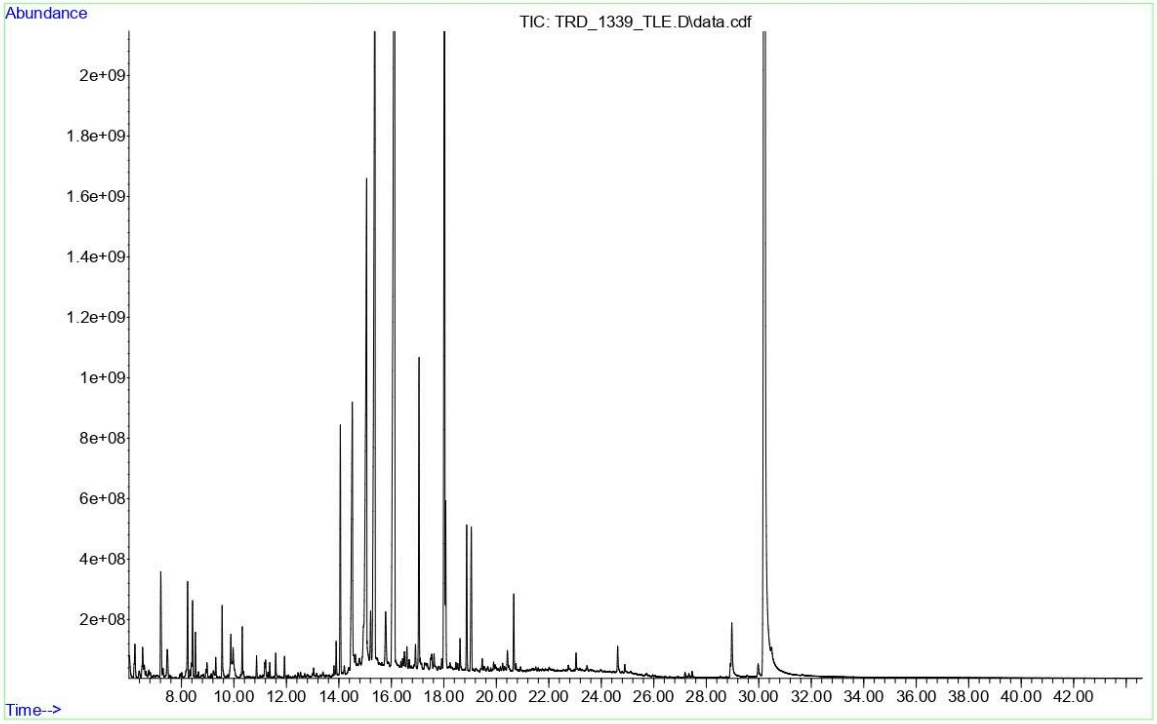
File :G:\Solvent Extraction\TRD_1343_TLE.D
Operator : Xcalibur_System
Acquired : 20231011223722+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



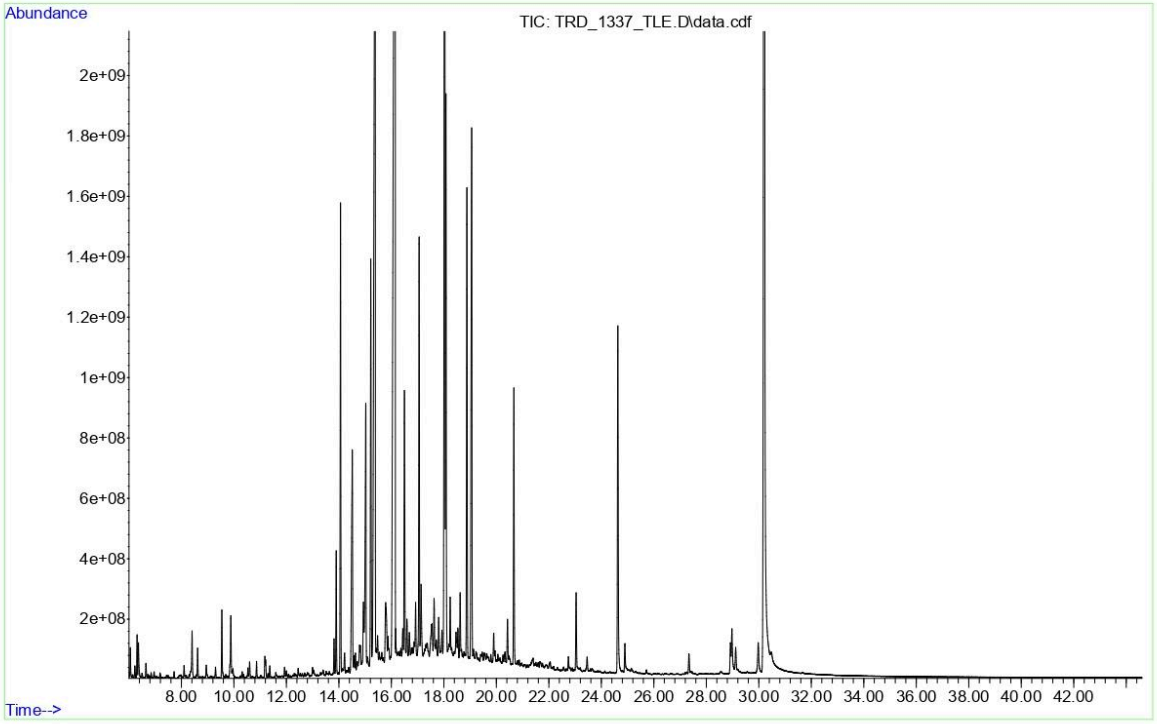
File :G:\Solvent Extraction\TRD_1342_TLE.D
Operator : Xcalibur_System
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



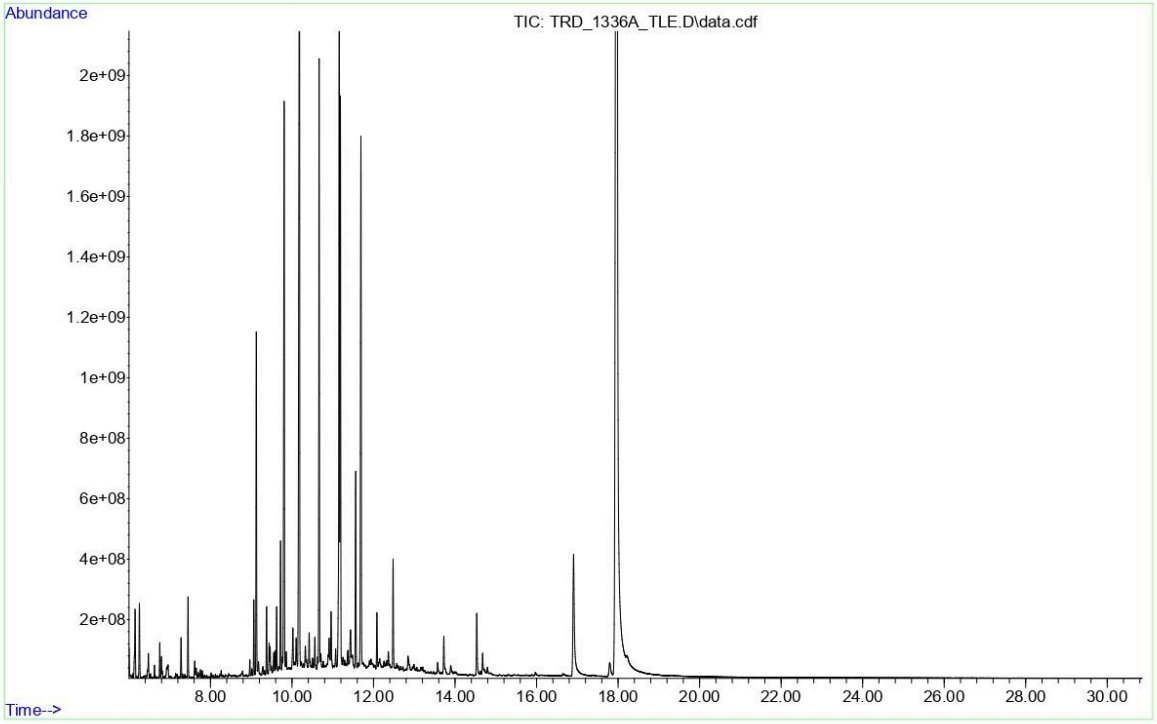
File :G:\Solvent Extraction\TRD_1339_TLE.D
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Acquired : 20231011182931+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



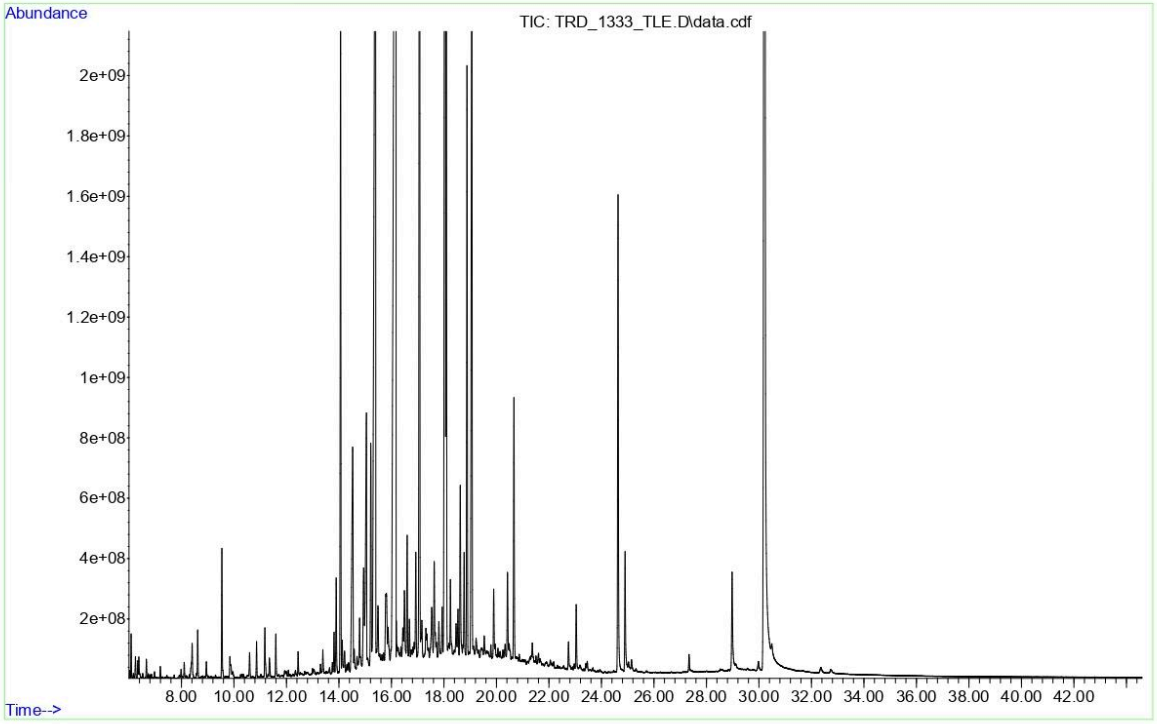
File :G:\Solvent Extraction\TRD_1337_TLE.D
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



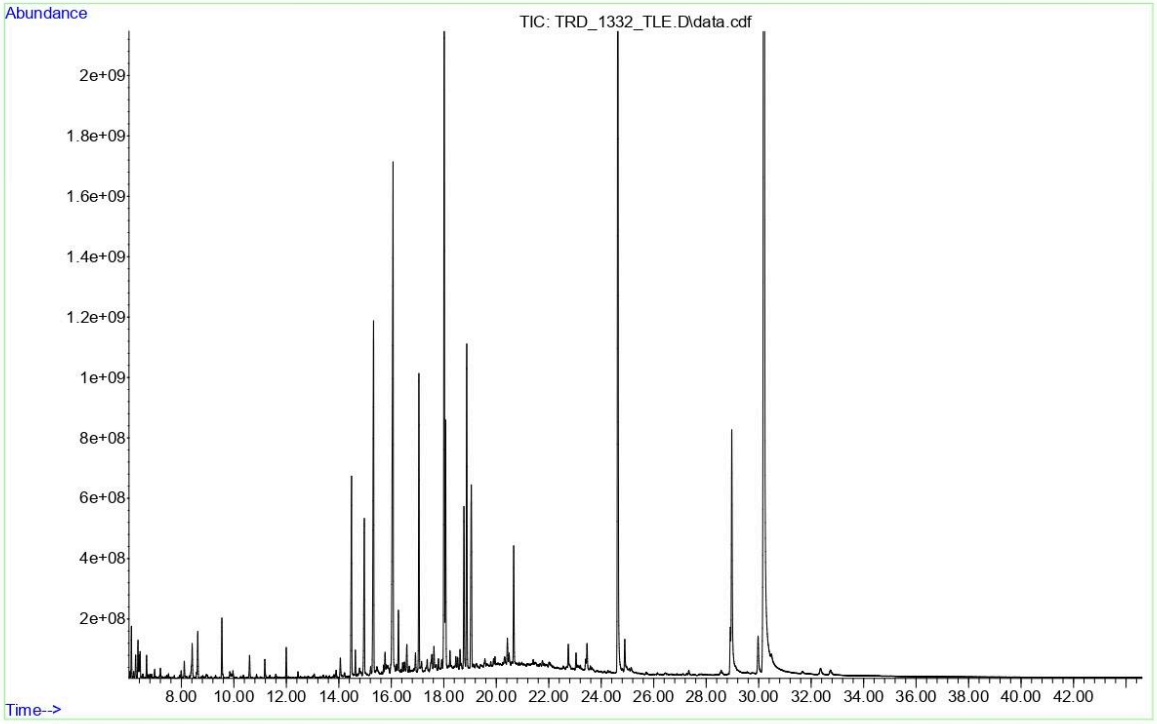
File :G:\Solvent Extraction\TRD_1336A_TLE.D
Operator : Xcalibur_System
Acquired : 20231012034856+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Solvent Extraction\TRD_1333_TLE.D
Operator : Xcalibur_System
Acquired : 20231011200018+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Solvent Extraction\TRD_1332_TLE.D
Operator : Xcalibur_System
Acquired : 20231011164446+0000 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0

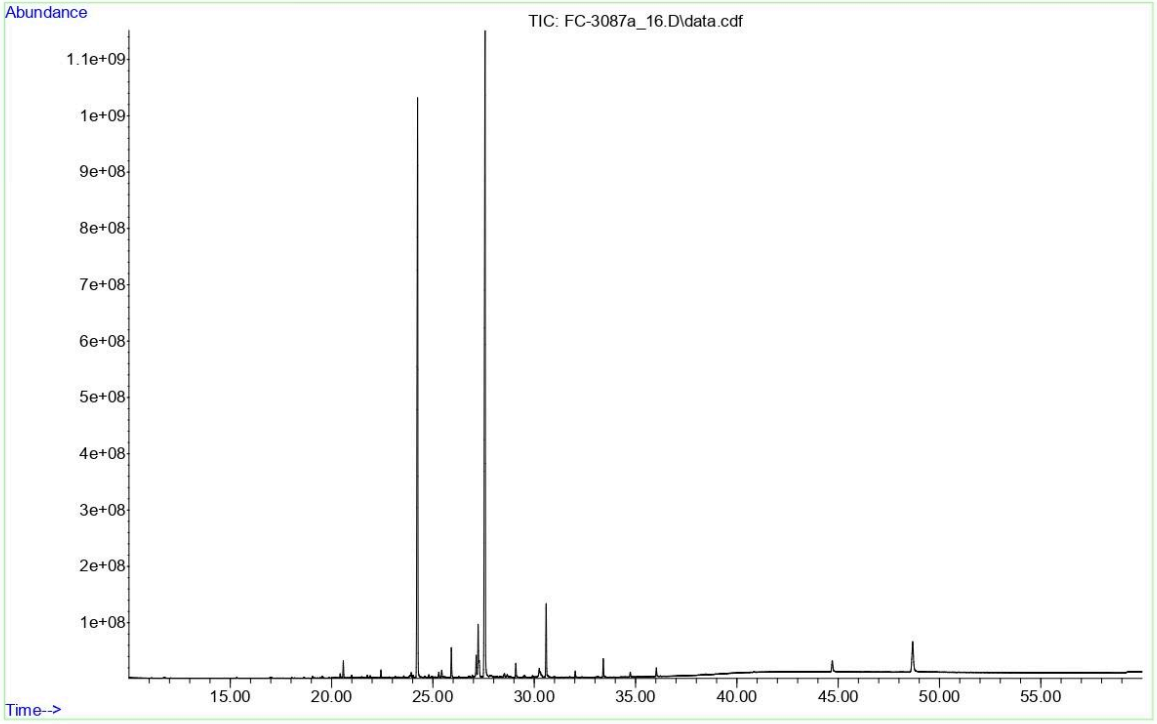


Foz do Cubatão

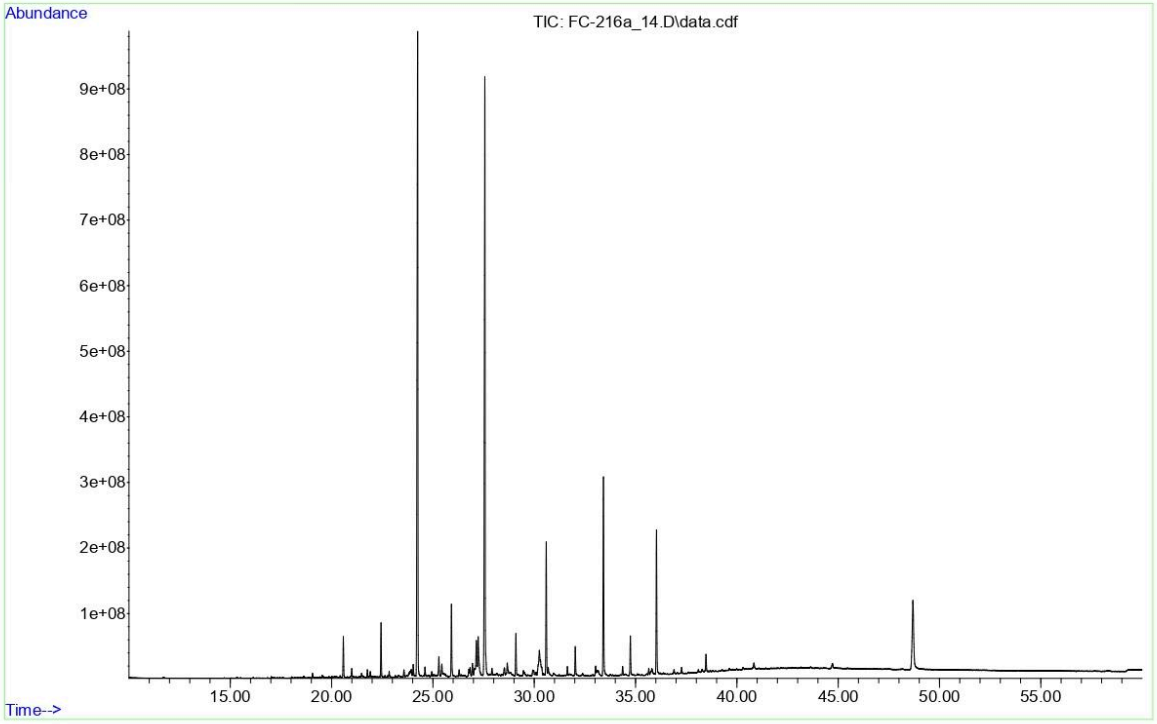
Acidified methanol extraction chromatograms

For Foz do Cubatão, TLE chromatogram files were originally generated; however, the raw chromatogram files were corrupted and could not be reopened or exported at the time of thesis revision. Analytical notes, peak tables and associated exported data remain available for the full set of samples.

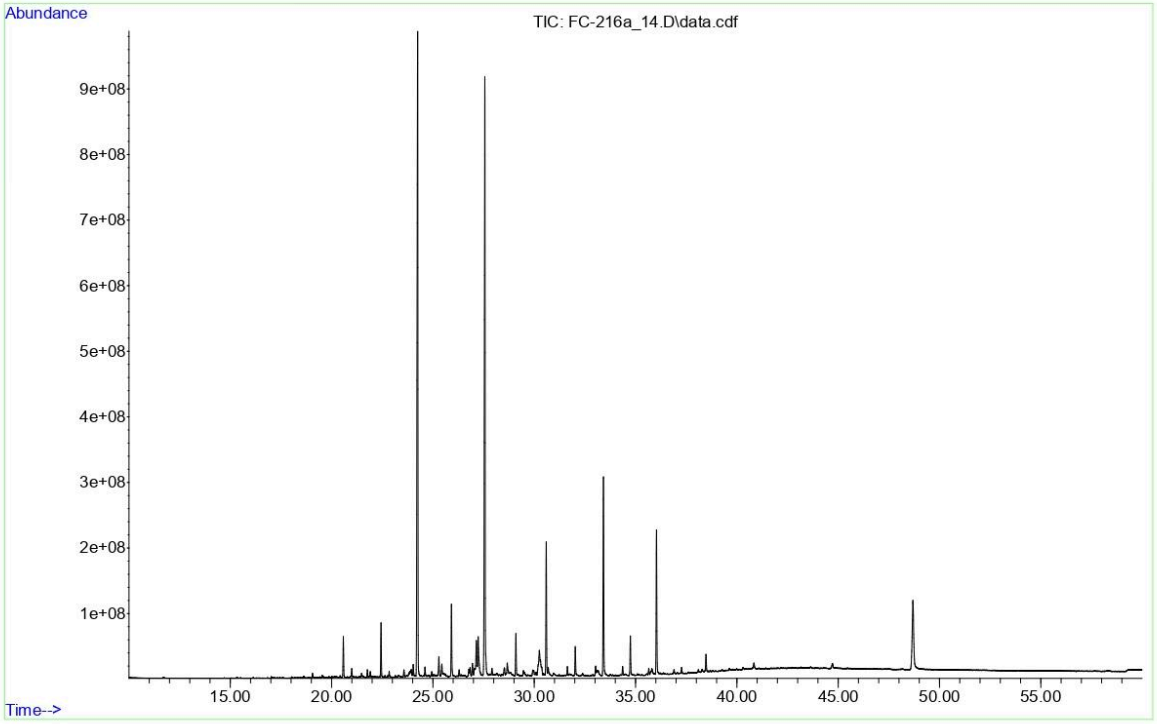
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erator   : SYSTEM
quired   : 20221028071122+0100 using AcqMethod
strument :    LCQ
mple Name:
sc Info  :
al Number: 0
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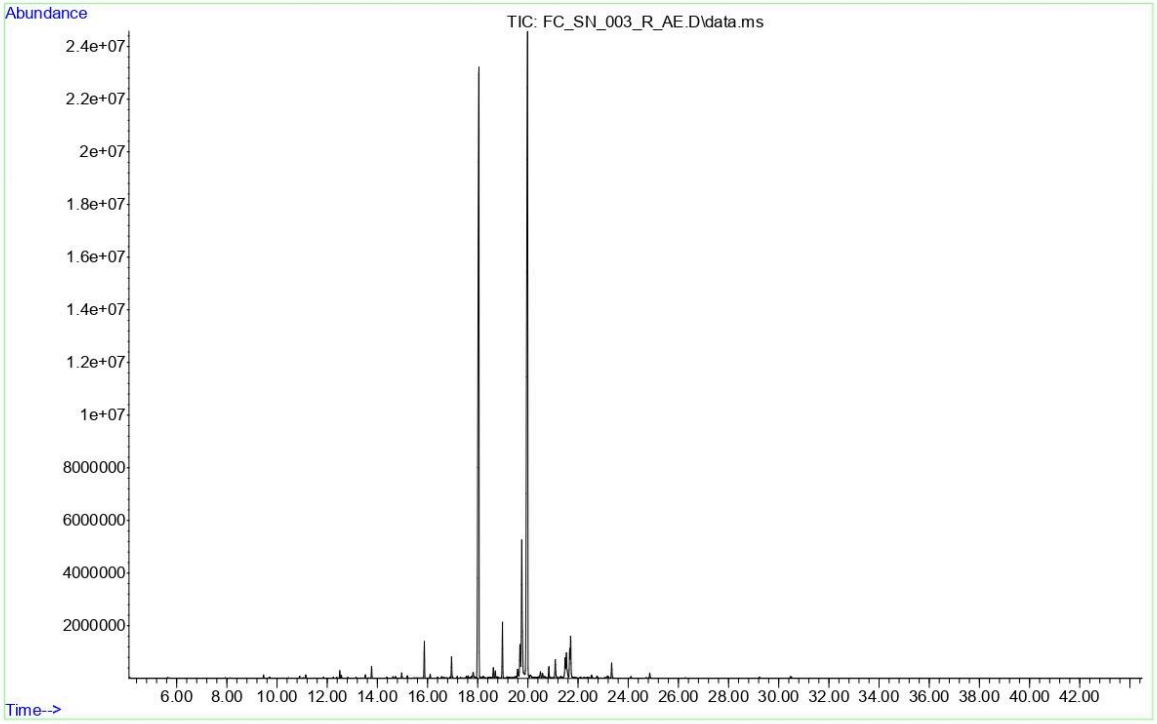
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



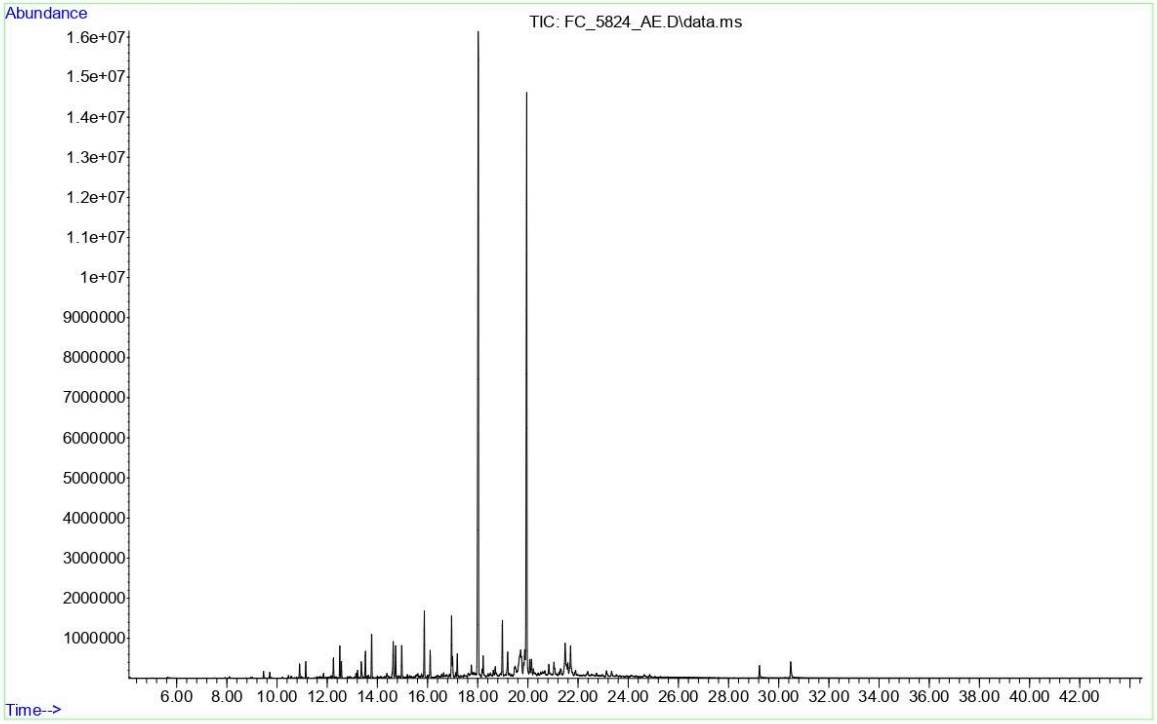
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Operator : SYSTEM
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



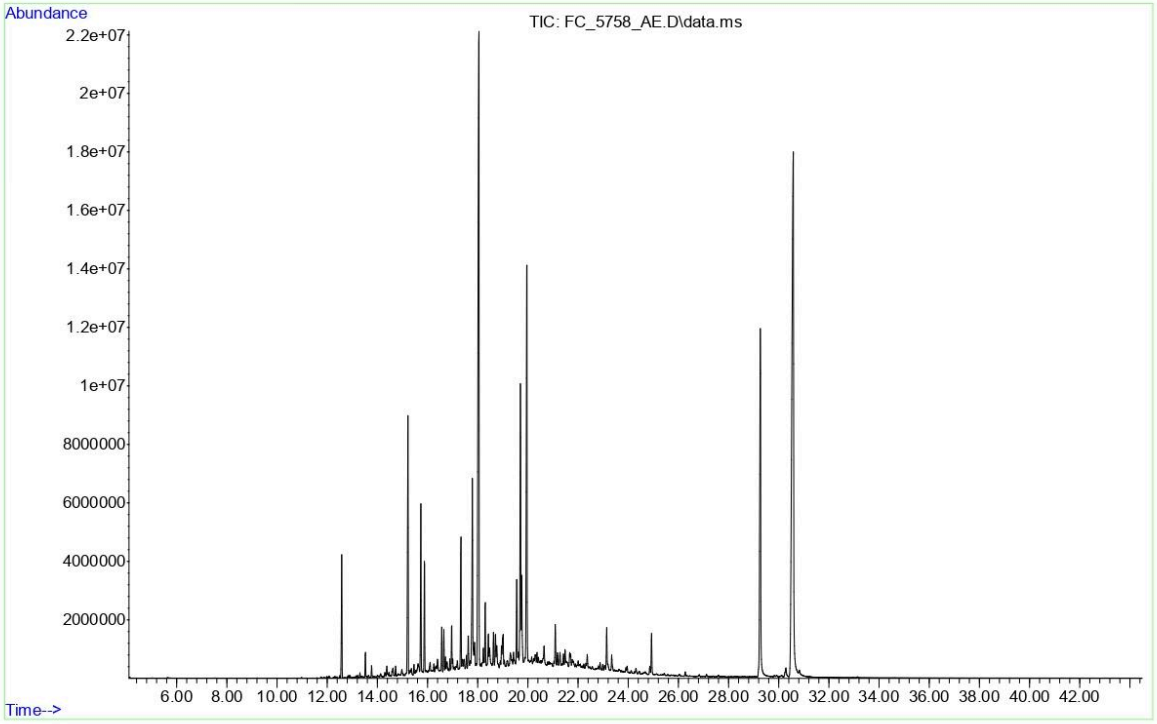
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Operator : hmt
Acquired : 06 Mar 2021 23:50 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_SN_003_R_MA030321
Misc Info :
Vial Number: 35



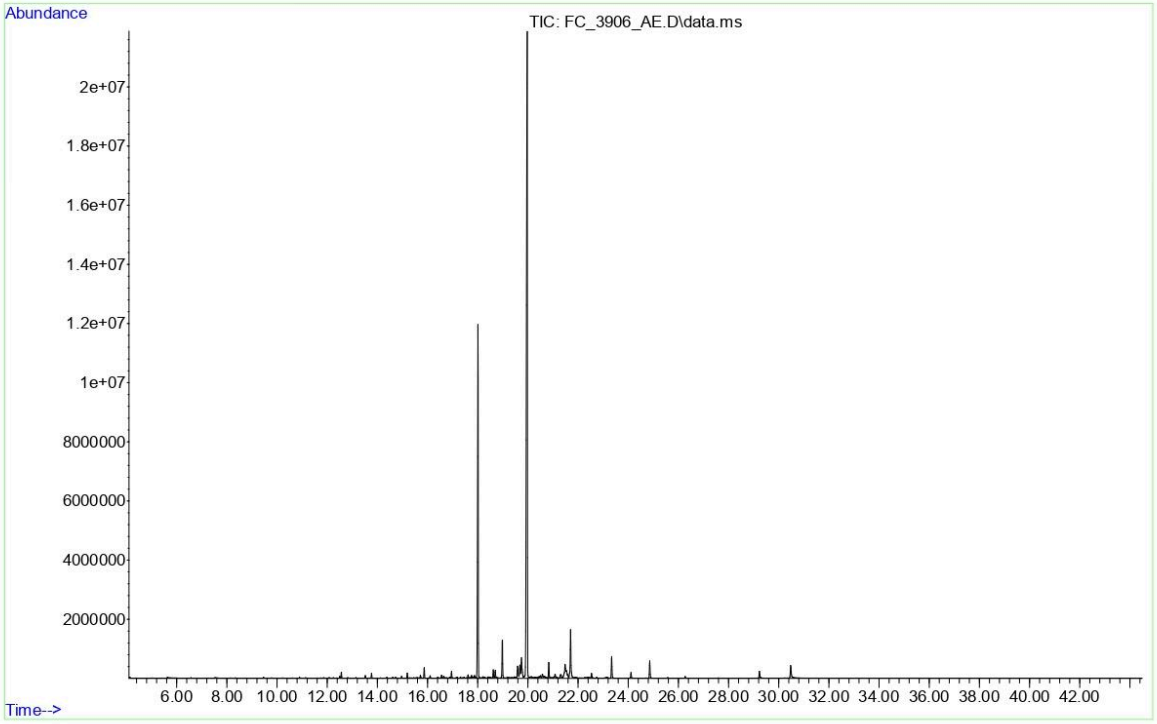
File :G:\Data by Site\FozDoCubatao\GCMS\FC_5824_AE.D
Operator : hmt
Acquired : 07 Mar 2021 00:41 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_5824_MA030321
Misc Info :
Vial Number: 36



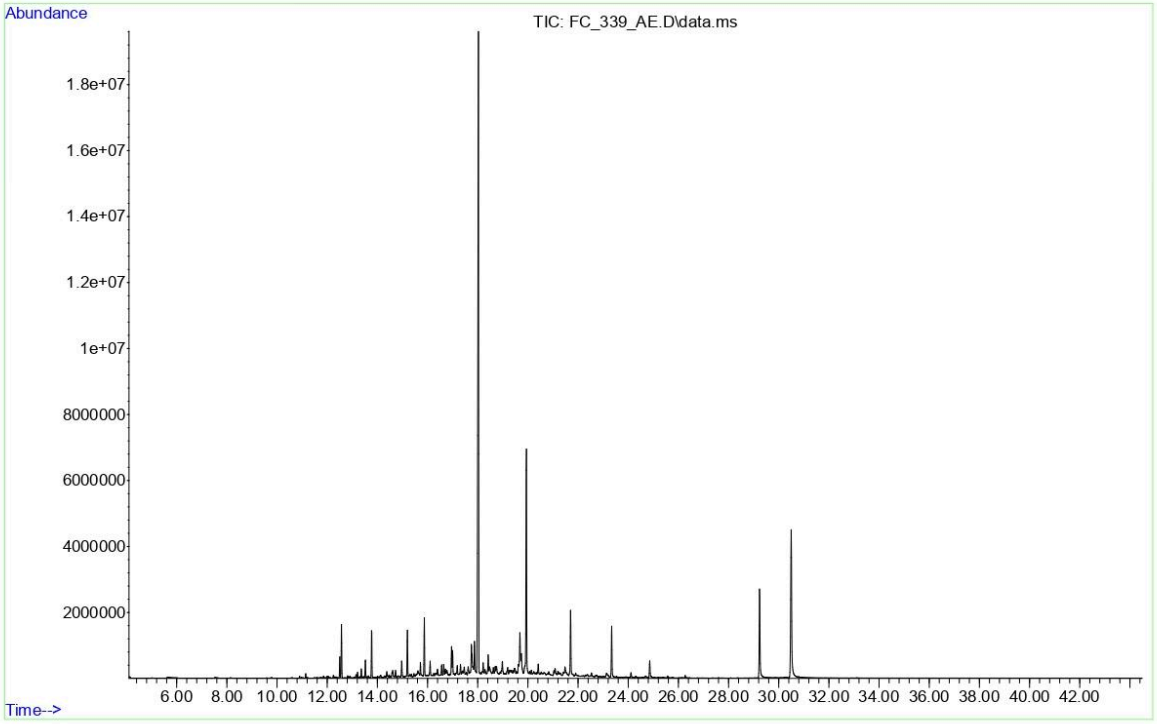
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Operator : hmt
Acquired : 07 Mar 2021 06:37 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_5758_MA030321
Misc Info :
Vial Number: 41



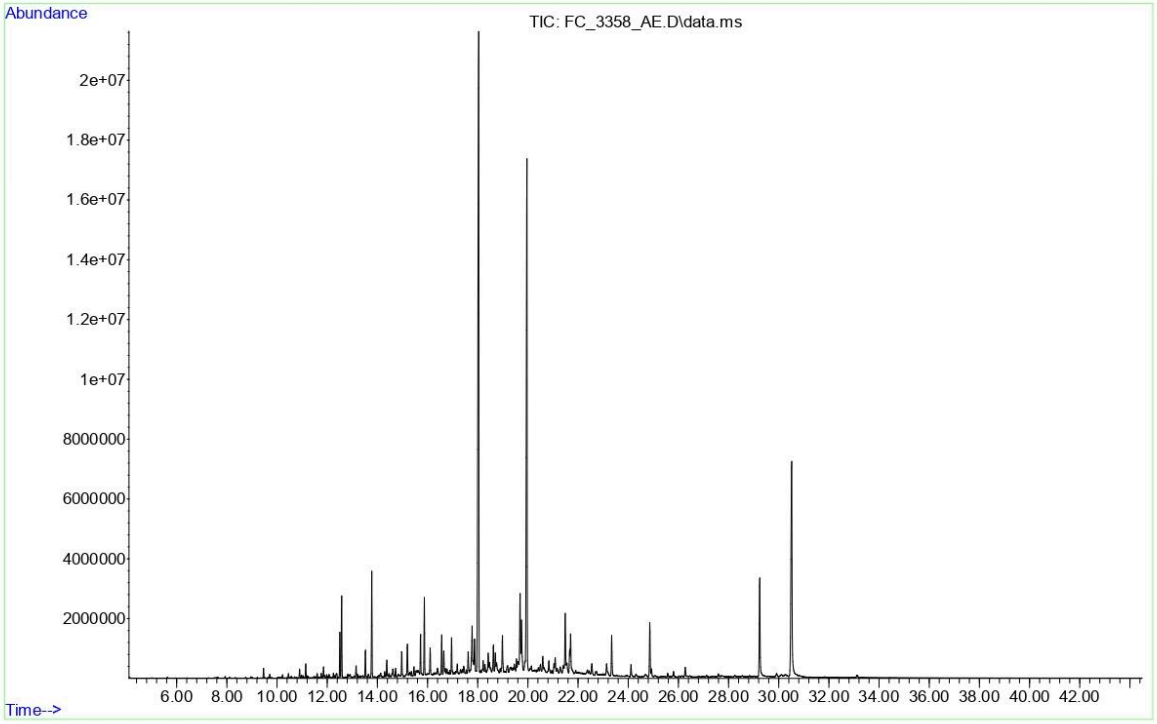
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Operator : hmt
Acquired : 07 Mar 2021 04:04 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_3906_MA030321
Misc Info :
Vial Number: 39



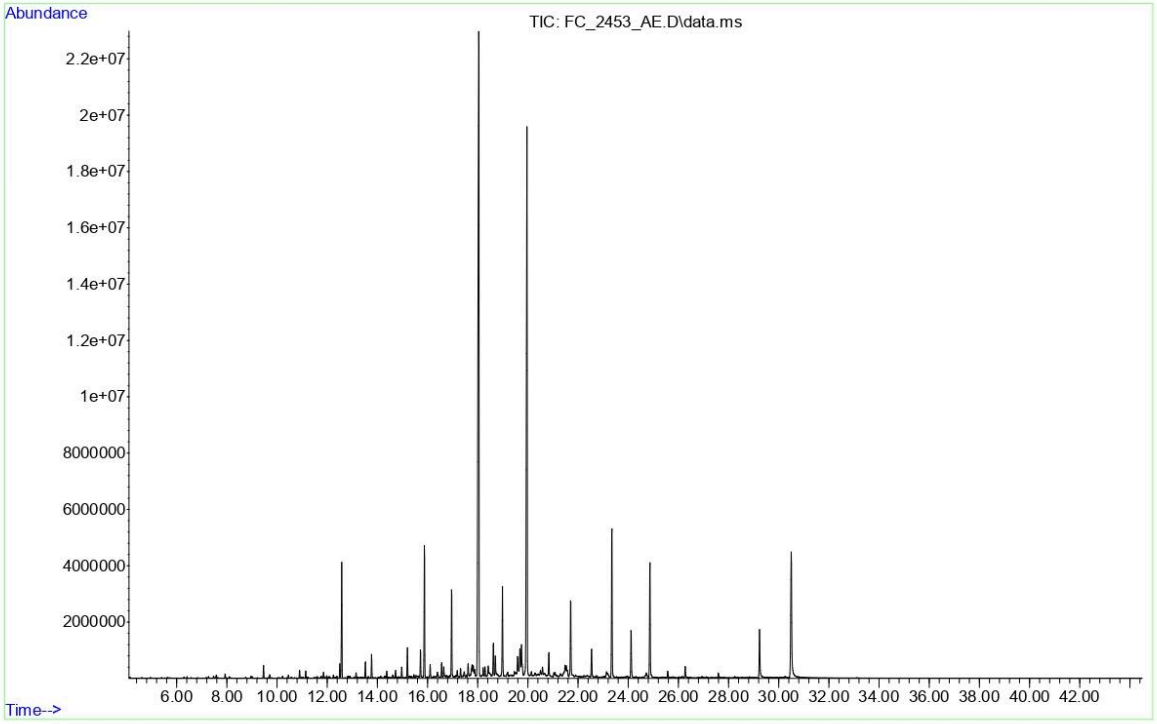
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Operator : hmt
Acquired : 07 Mar 2021 04:55 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_339_MA030321
Misc Info :
Vial Number: 40



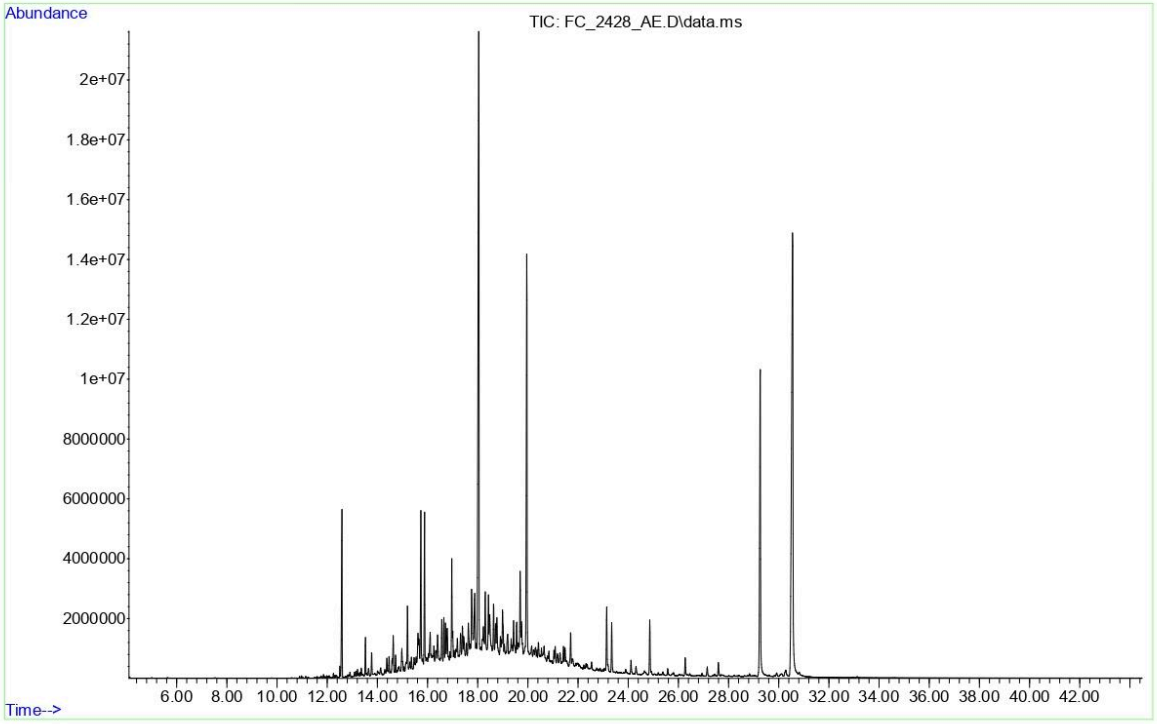
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Operator : hmt
Acquired : 07 Mar 2021 03:14 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_3358_MA030321
Misc Info :
Vial Number: 38



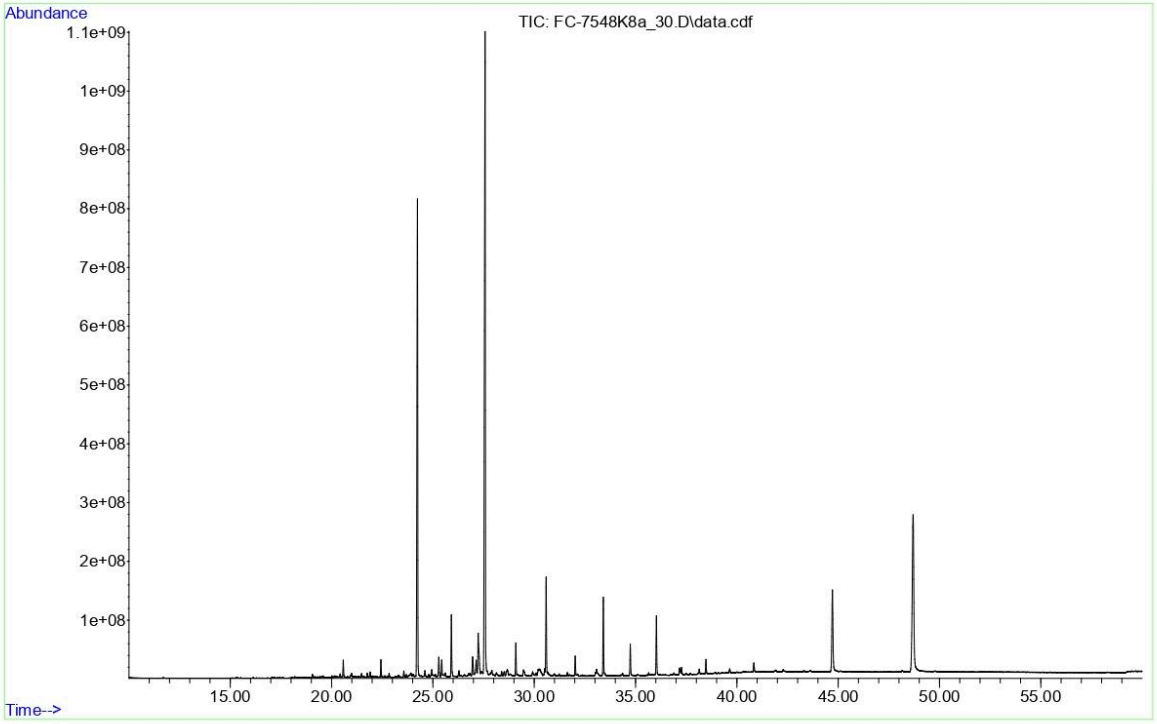
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Operator : hmt
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Instrument : MSD
Sample Name: FC_2453_MA030321
Misc Info :
Vial Number: 37



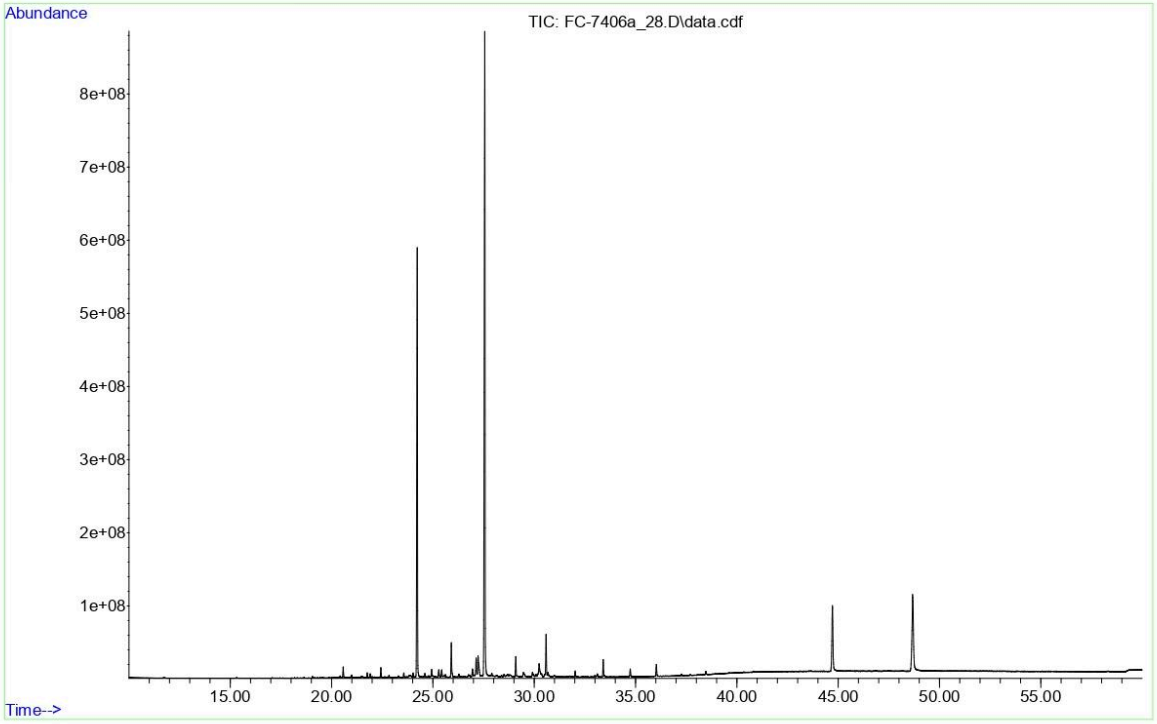
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Operator : hmt
Acquired : 07 Mar 2021 07:28 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC_2428_MA030321
Misc Info :
Vial Number: 42



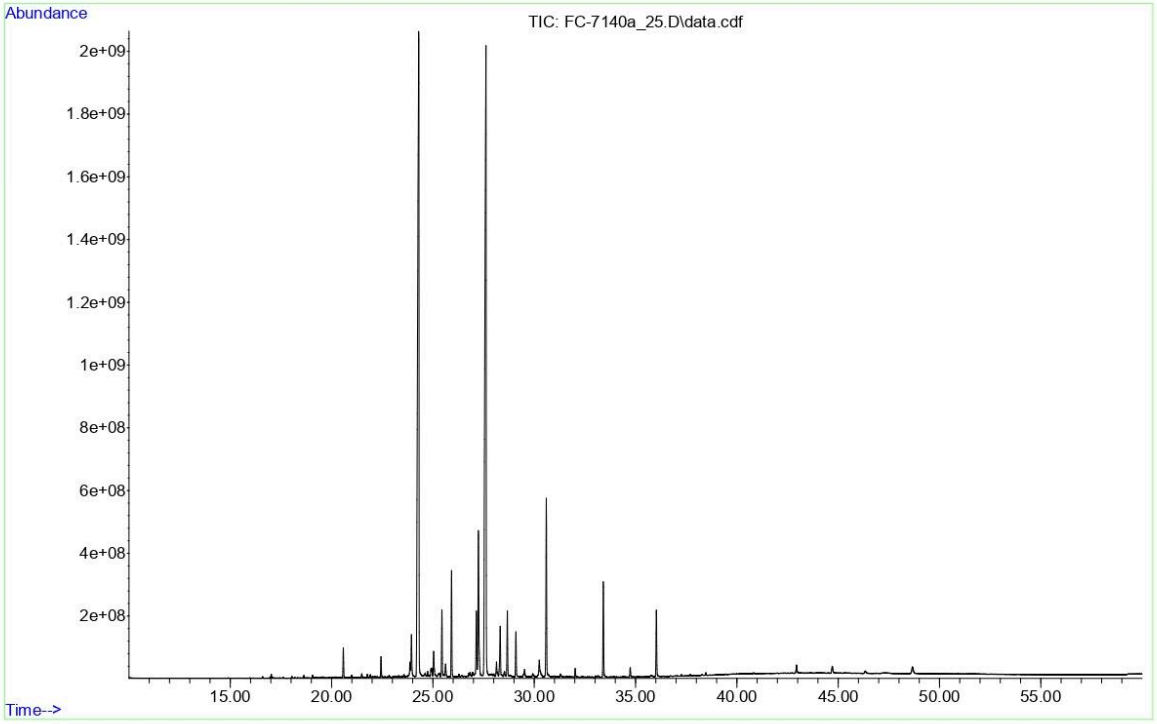
File :G:\Data by Site\FozDoCubatao\GCMS\FC-7548K8a_30.D
Operator : SYSTEM
Acquired : 20221029000220+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



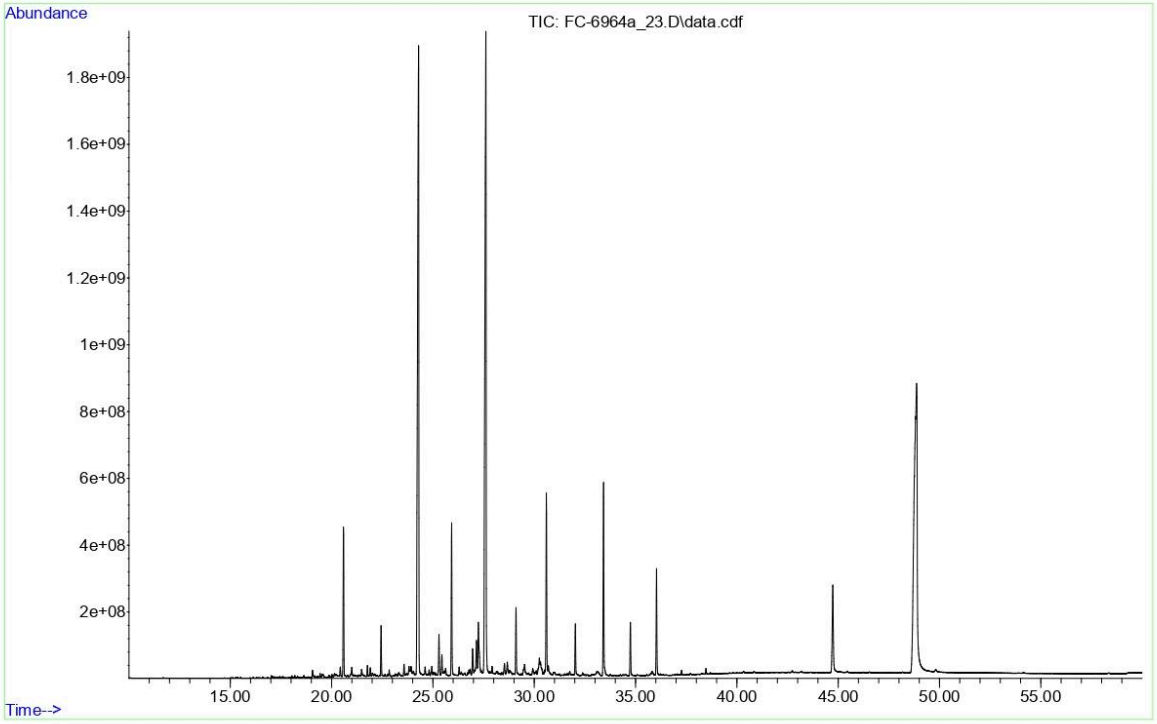
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Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



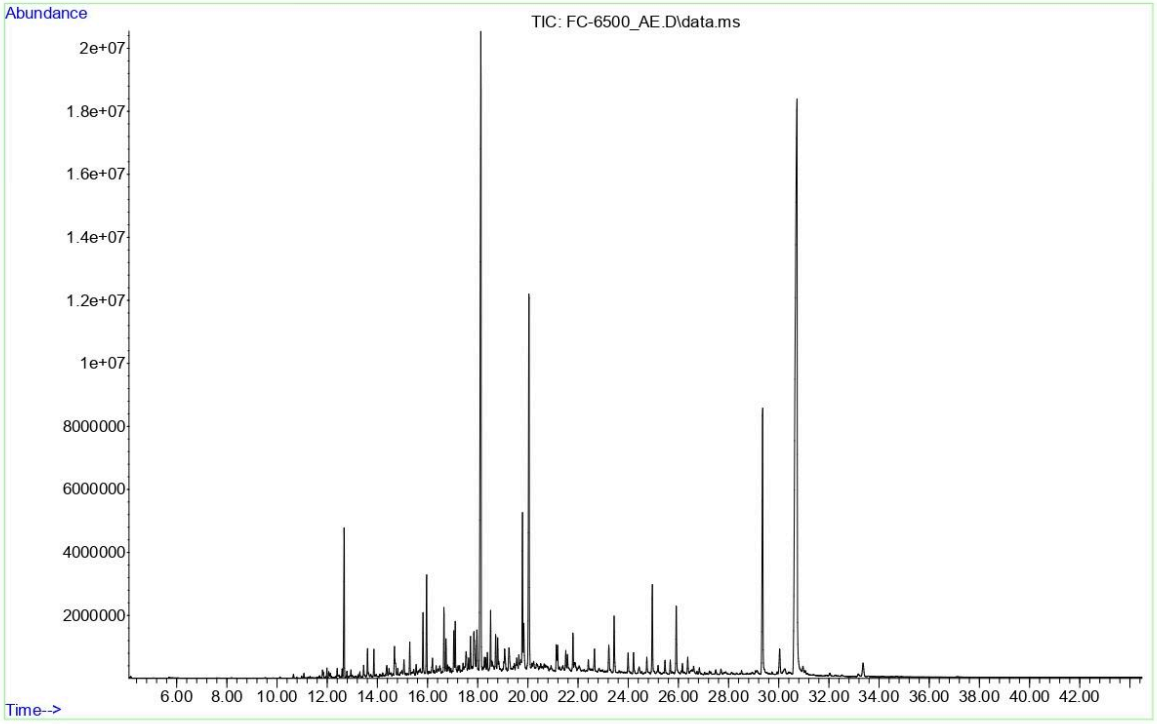
File :G:\Data by Site\FozDoCubatao\GCMS\FC-7140a_25.D
Operator : SYSTEM
Acquired : 20221028180117+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



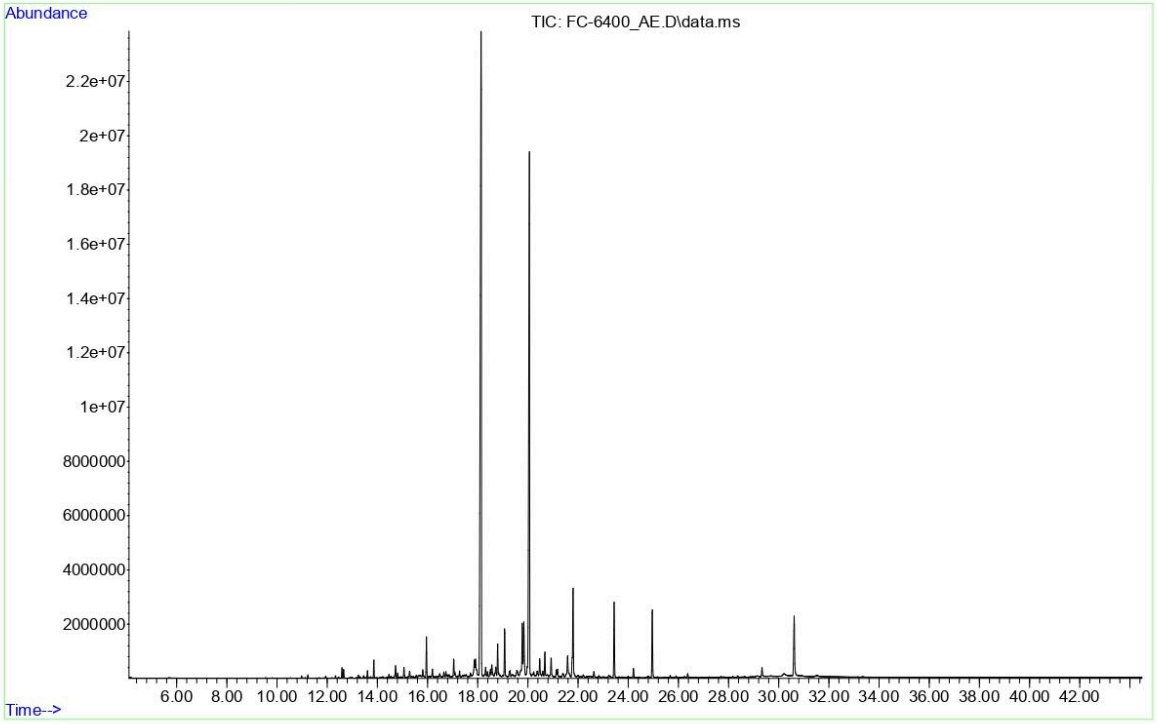
File :G:\Data by Site\FozDoCubatao\GCMS\FC-6964a_23.D
Operator : SYSTEM
Acquired : 20221028153650+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



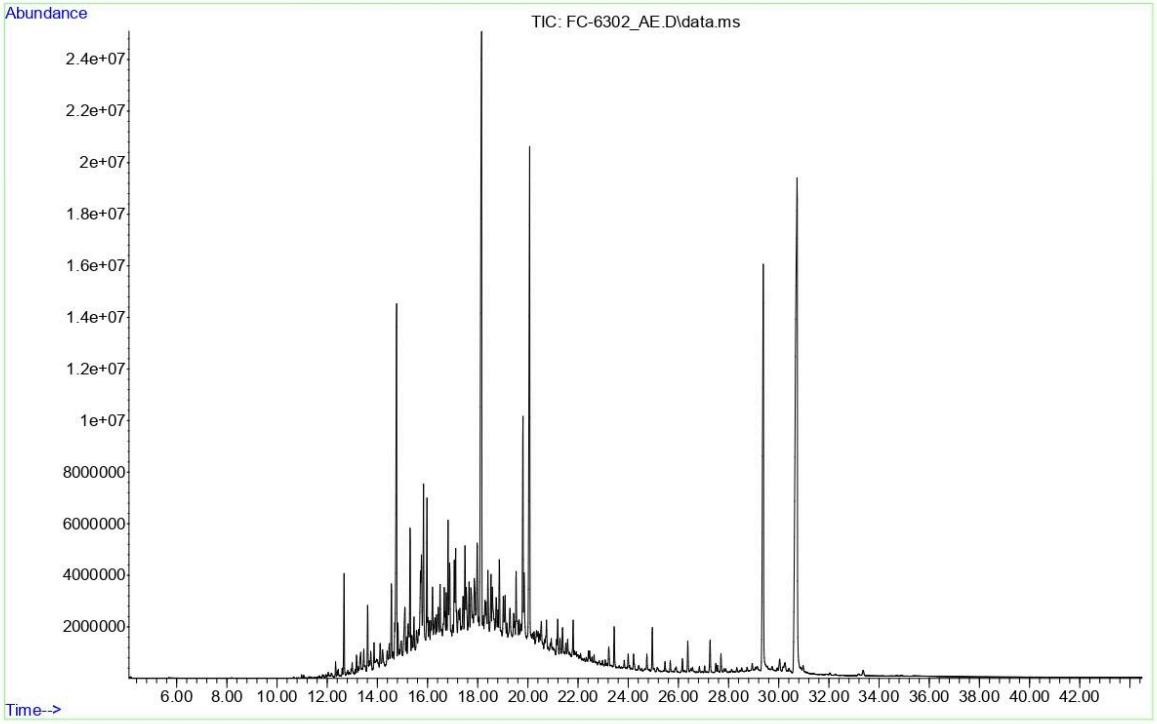
File :G:\Data by Site\FozDoCubatao\GCMS\FC-6500_AE.D
Operator : JL
Acquired : 25 Sep 2021 20:00 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-6500_MA200921_AE
Misc Info :
Vial Number: 38



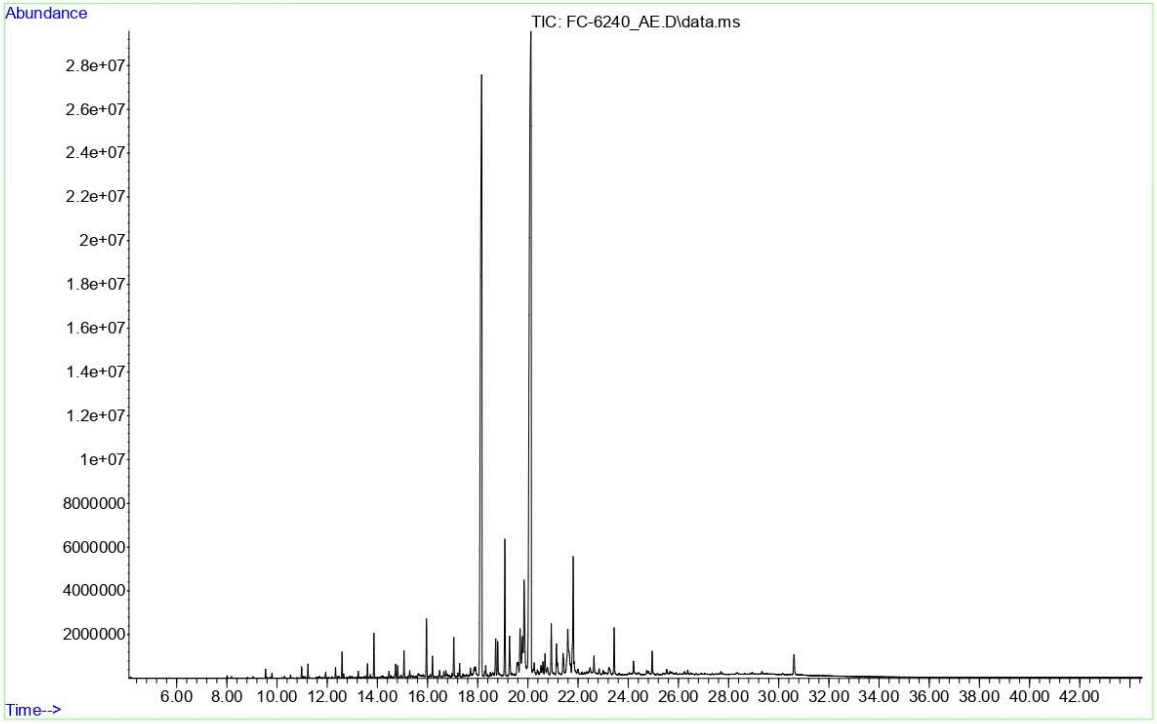
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Operator : JL
Acquired : 25 Sep 2021 19:09 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-6400_MA200921_AE
Misc Info :
Vial Number: 37



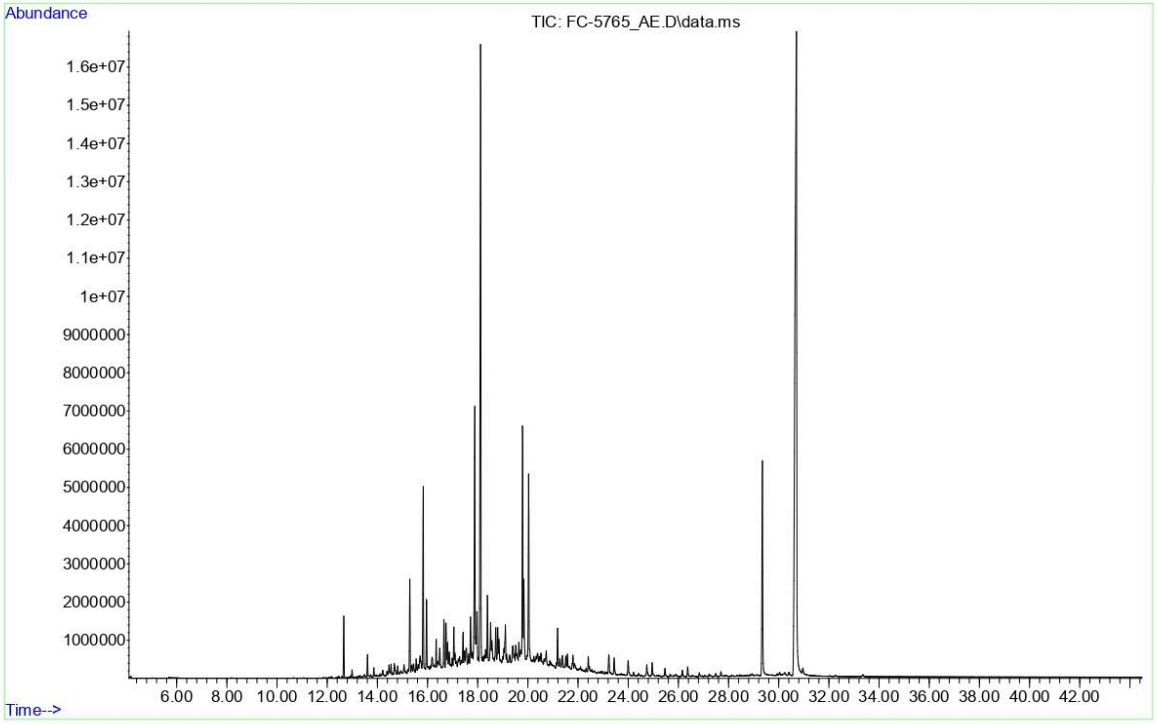
File :G:\Data by Site\FozDoCubatao\GCMS\FC-6302_AE.D
Operator : JL
Acquired : 25 Sep 2021 16:35 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-6302_MA200921_AE
Misc Info :
Vial Number: 35



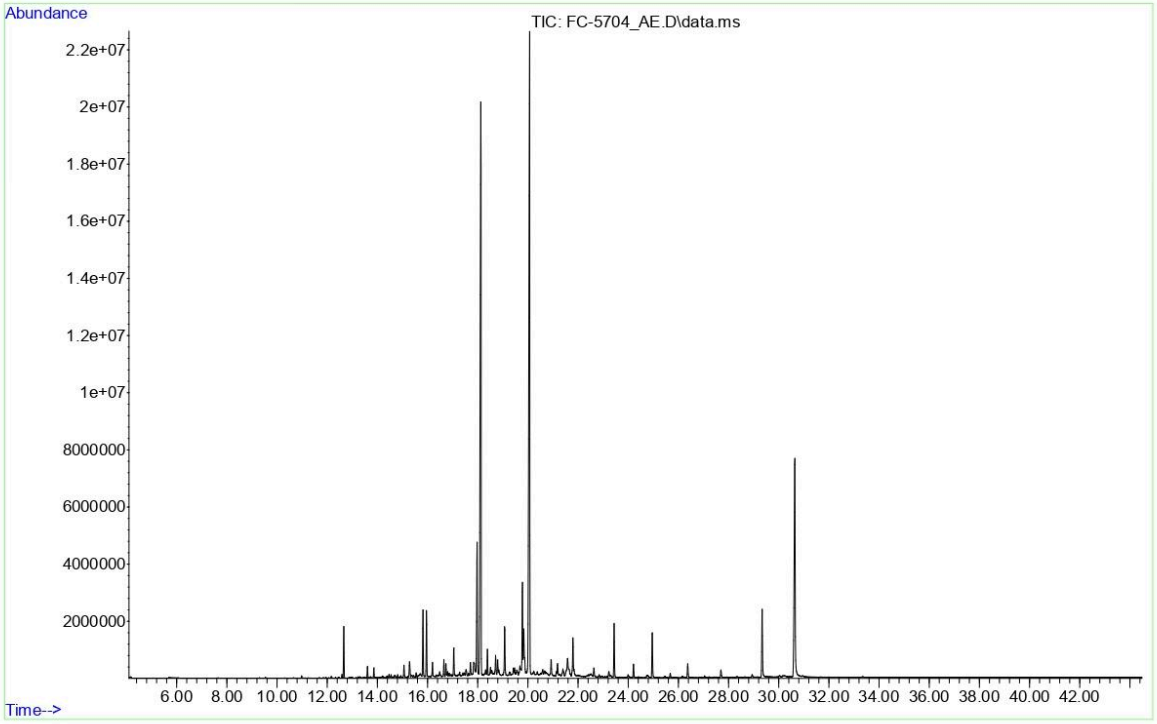
File :G:\Data by Site\FozDoCubatao\GCMS\FC-6240_AE.D
Operator : JL
Acquired : 25 Sep 2021 17:26 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-6240_MA200921_AE
Misc Info :
Vial Number: 36



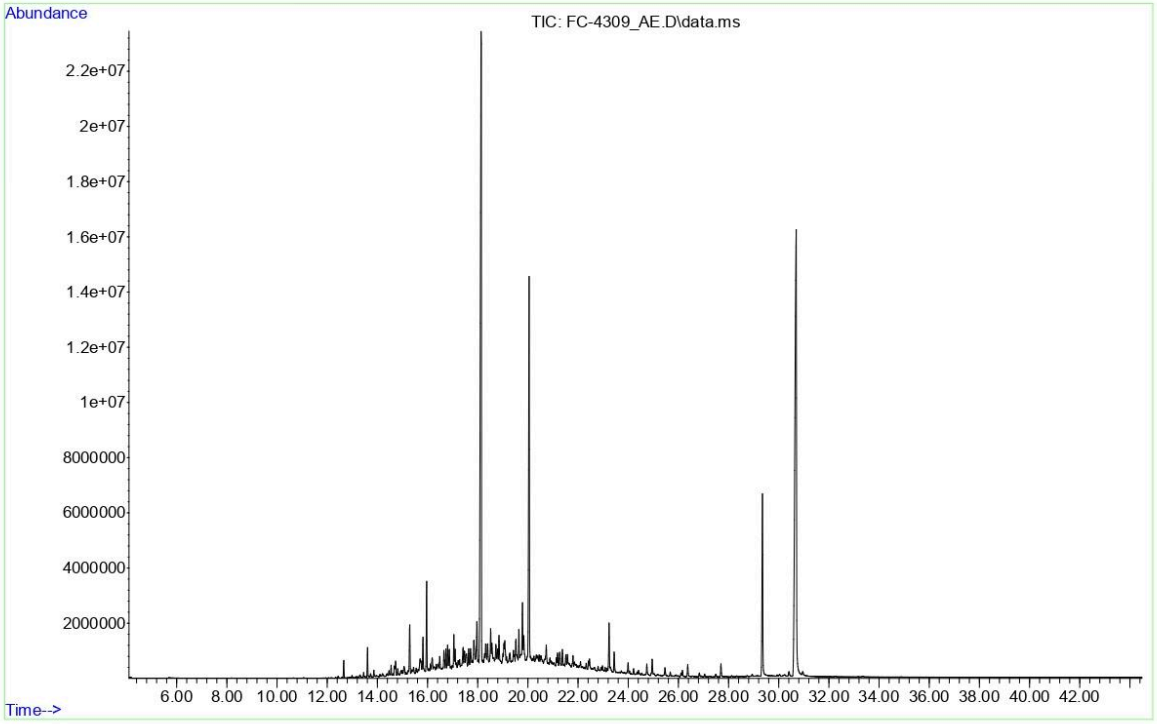
File :G:\Data by Site\FozDoCubatao\GCMS\FC-5765_AE.D
Operator : JL
Acquired : 25 Sep 2021 15:44 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-5765_MA200921_AE
Misc Info :
Vial Number: 34



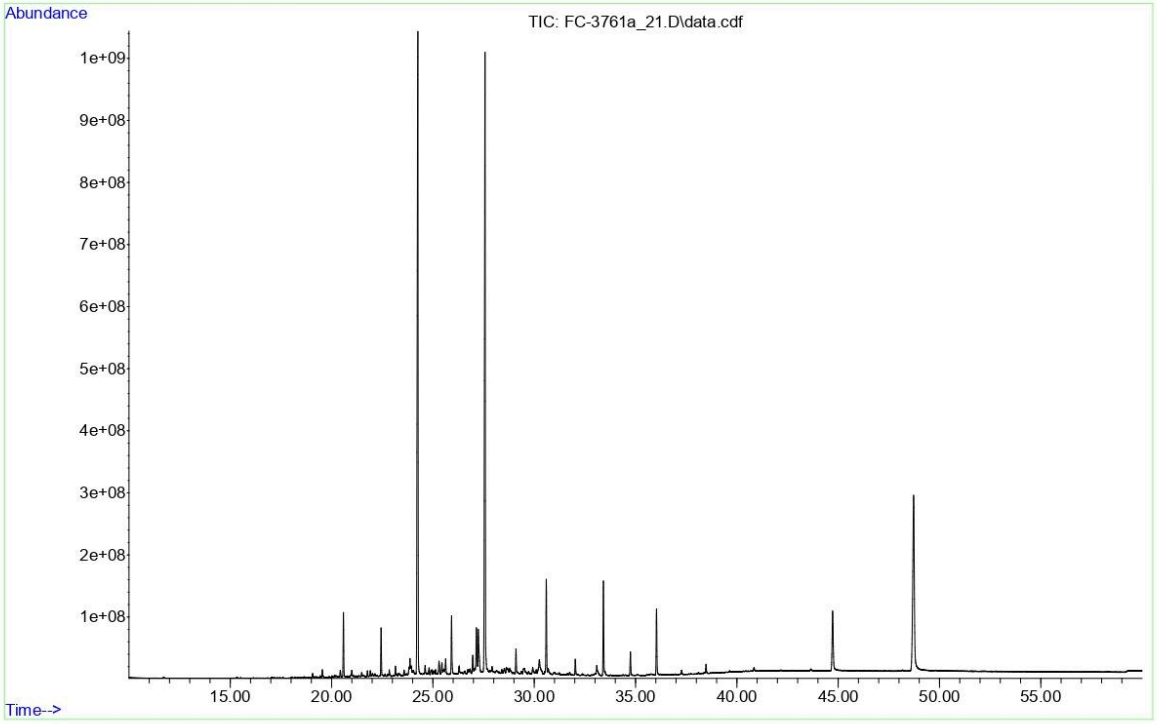
File :G:\Data by Site\FozDoCubatao\GCMS\FC-5704_AE.D
Operator : JL
Acquired : 25 Sep 2021 14:53 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-5704_MA200921_AE
Misc Info :
Vial Number: 33



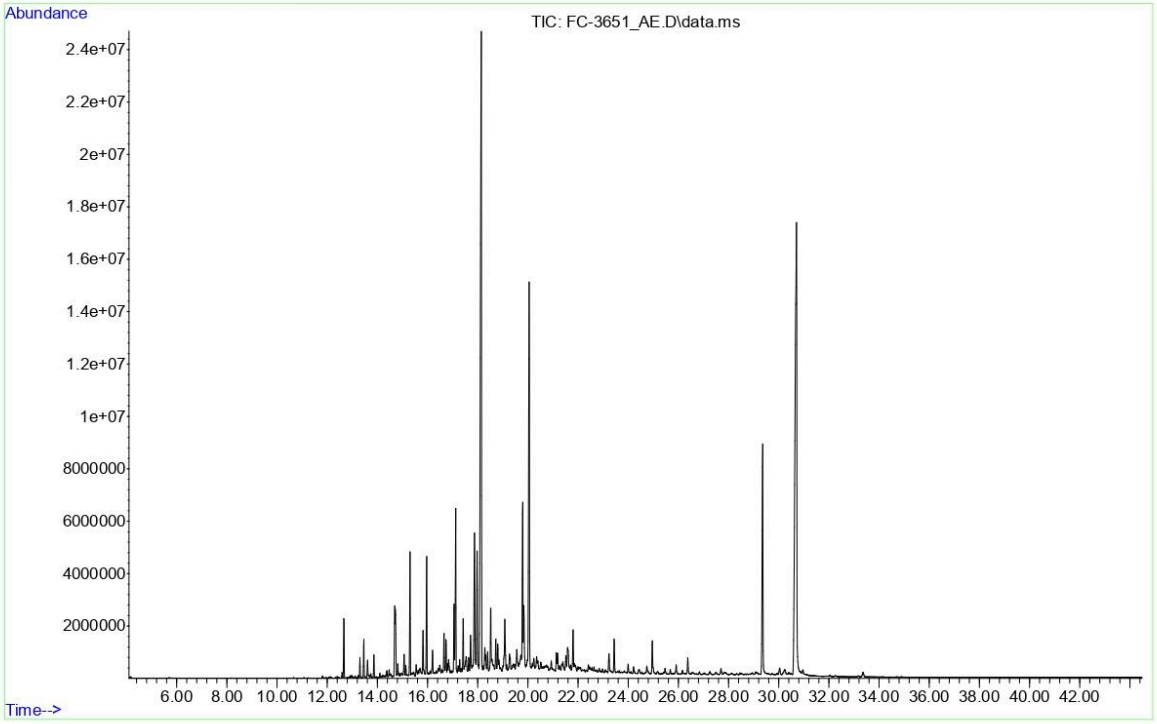
File :G:\Data by Site\FozDoCubatao\GCMS\FC-4309_AE.D
Operator : JL
Acquired : 25 Sep 2021 13:10 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-4309_MA200921_AE
Misc Info :
Vial Number: 32



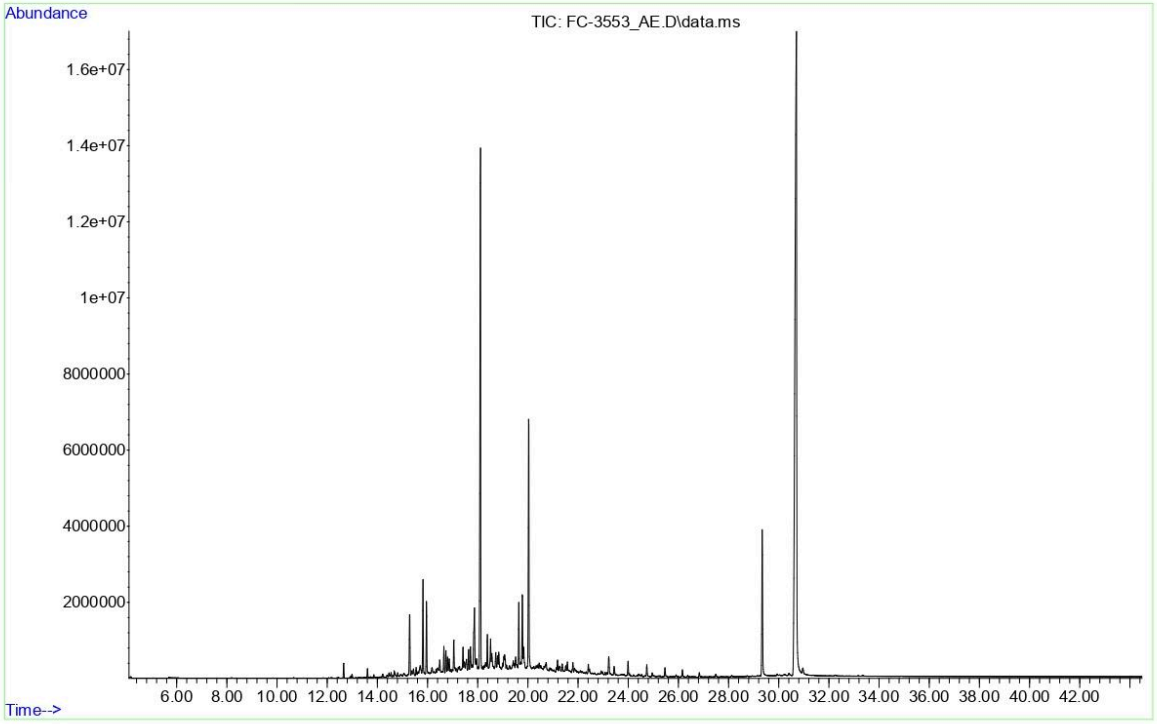
File :G:\Data by Site\FozDoCubatao\GCMS\FC-3761a_21.D
Operator : SYSTEM
Acquired : 20221028131227+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



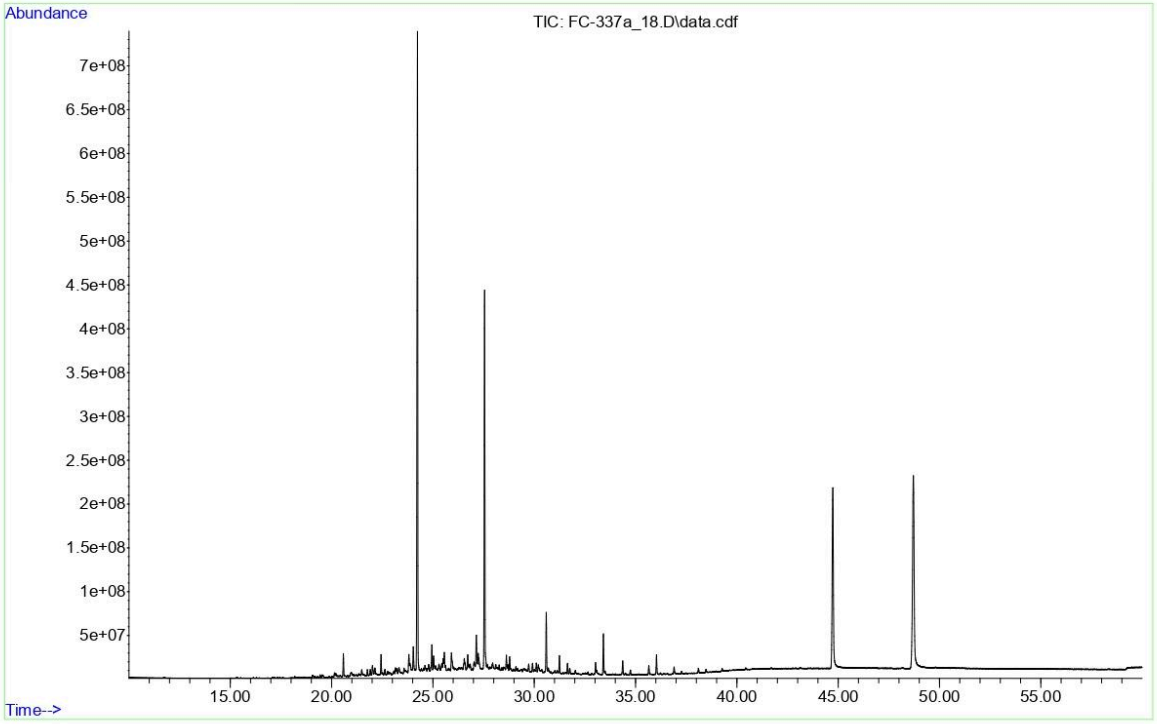
File :G:\Data by Site\FozDoCubatao\GCMS\FC-3651_AE.D
Operator : JL
Acquired : 25 Sep 2021 12:19 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-3651_MA200921_AE
Misc Info :
Vial Number: 31



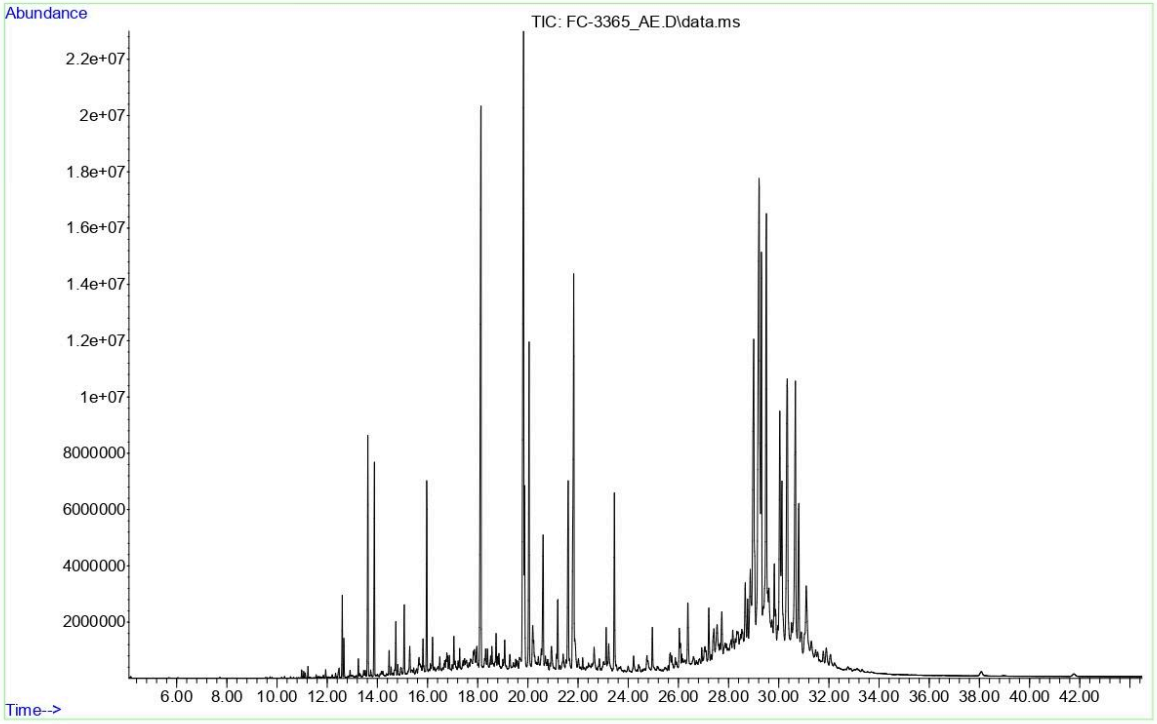
File :G:\Data by Site\FozDoCubatao\GCMS\FC-3553_AE.D
Operator : JL
Acquired : 25 Sep 2021 11:28 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-3553_MA200921_AE
Misc Info :
Vial Number: 30



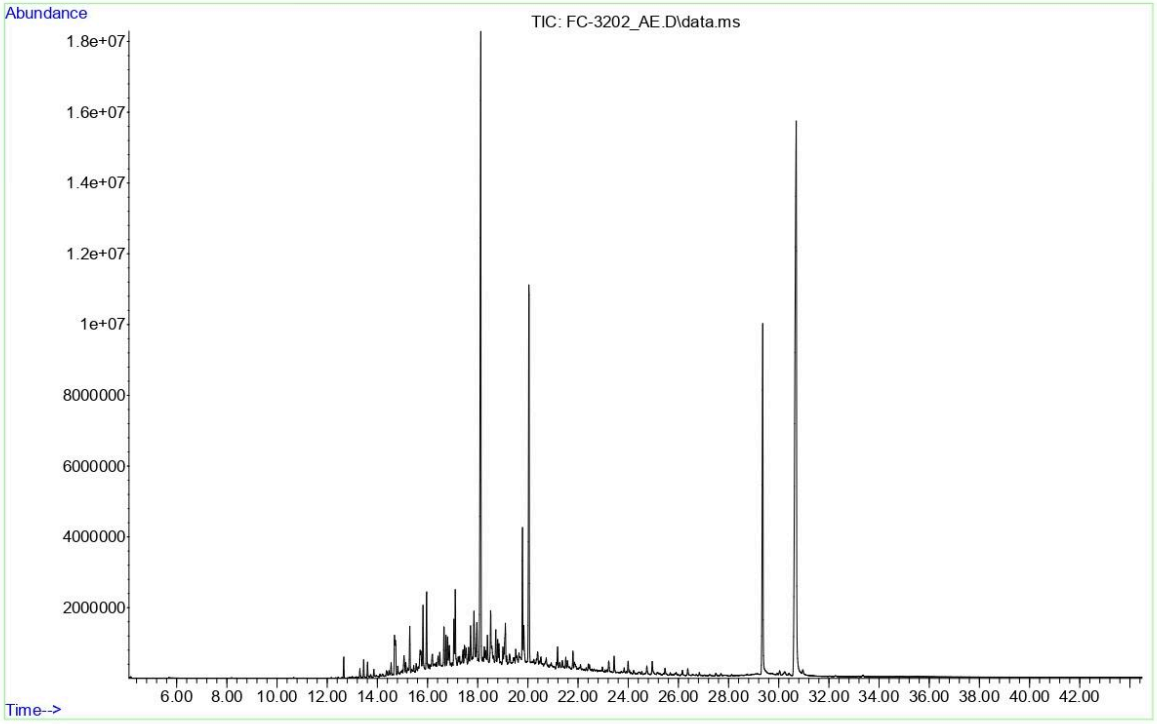
File :G:\Data by Site\FozDoCubatao\GCMS\FC-337a_18.D
Operator : SYSTEM
Acquired : 20221028093549+0100 using AcqMethod
Instrument : LCQ
Sample Name:
Misc Info :
Vial Number: 0



File :G:\Data by Site\FozDoCubatao\GCMS\FC-3365_AE.D
Operator : JL
Acquired : 25 Sep 2021 10:37 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-3365_MA200921_AE
Misc Info :
Vial Number: 29



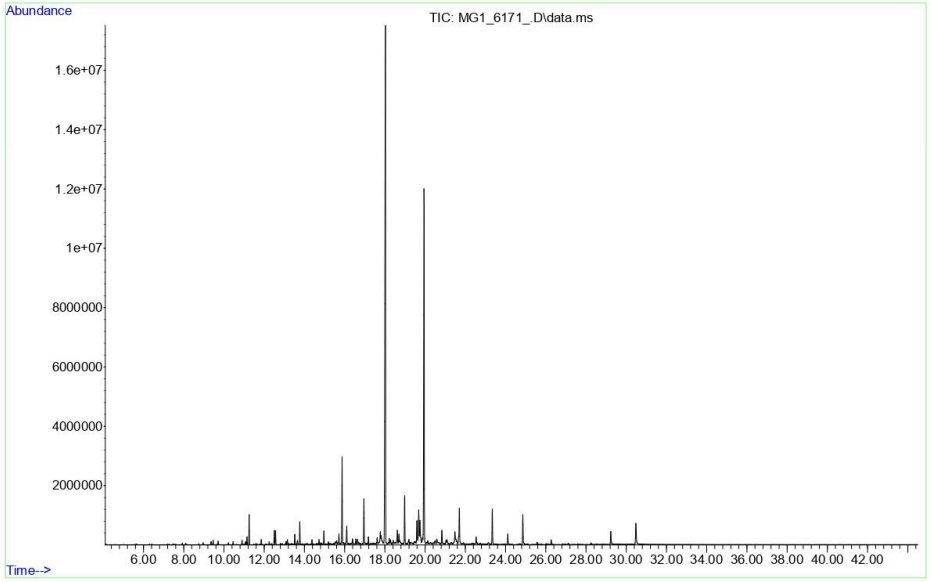
File :G:\Data by Site\FozDoCubatao\GCMS\FC-3202_AE.D
Operator : JL
Acquired : 25 Sep 2021 08:54 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: FC-3202_MA200921_AE
Misc Info :
Vial Number: 28



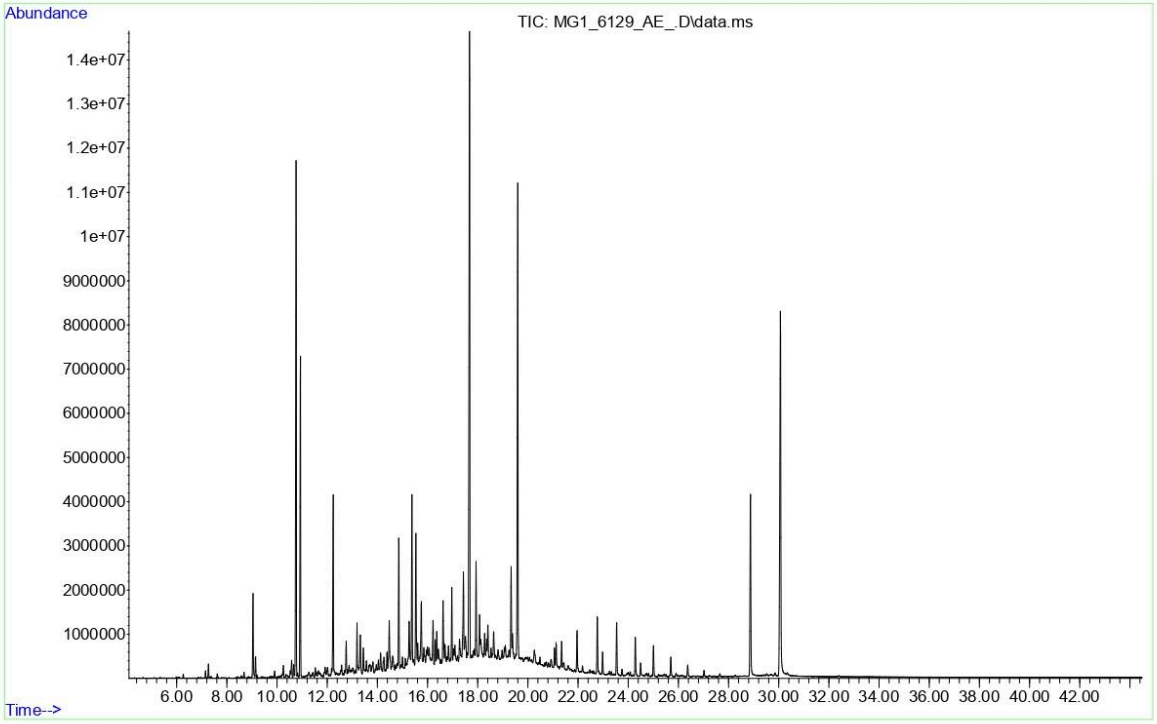
Morro Grande

Acidified methanol extraction chromatograms

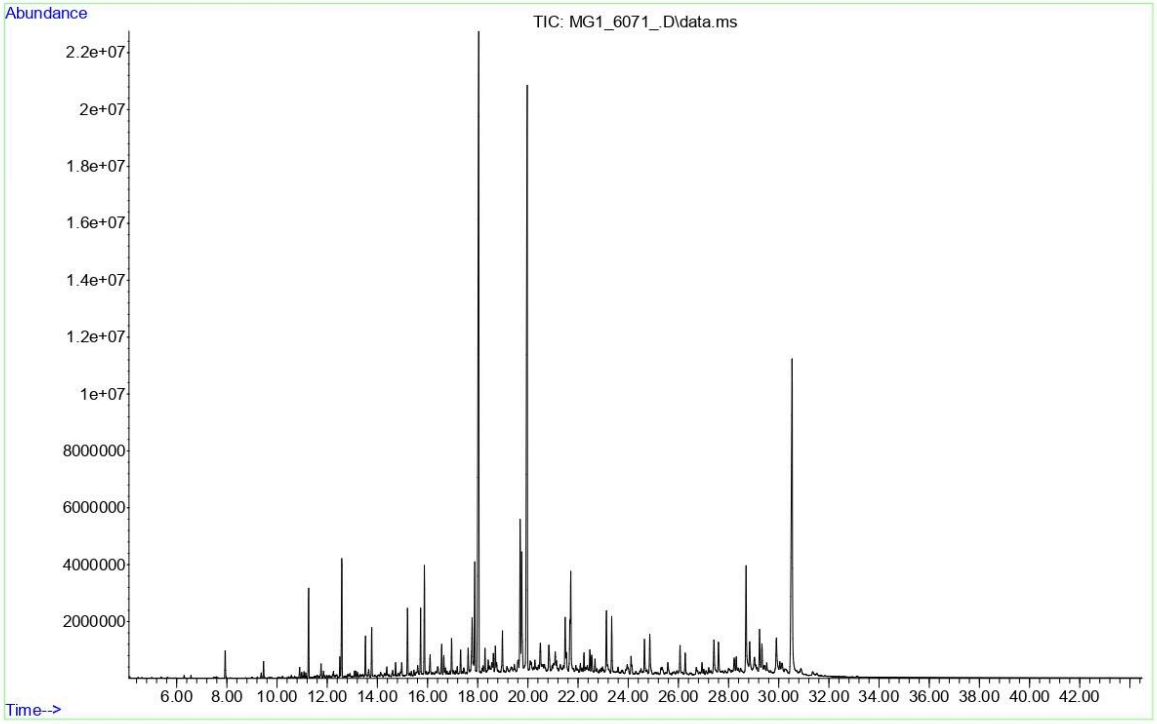
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File       :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6171_.D
Operator   : hmt
Acquired   : 07 Mar 2021  10:52     using AcqMethod DB5MS_CDS_BIOL.M
Instrument :    MSD
Sample Name: MG1_6171_MA030321
Misc Info  :
Vial Number: 45
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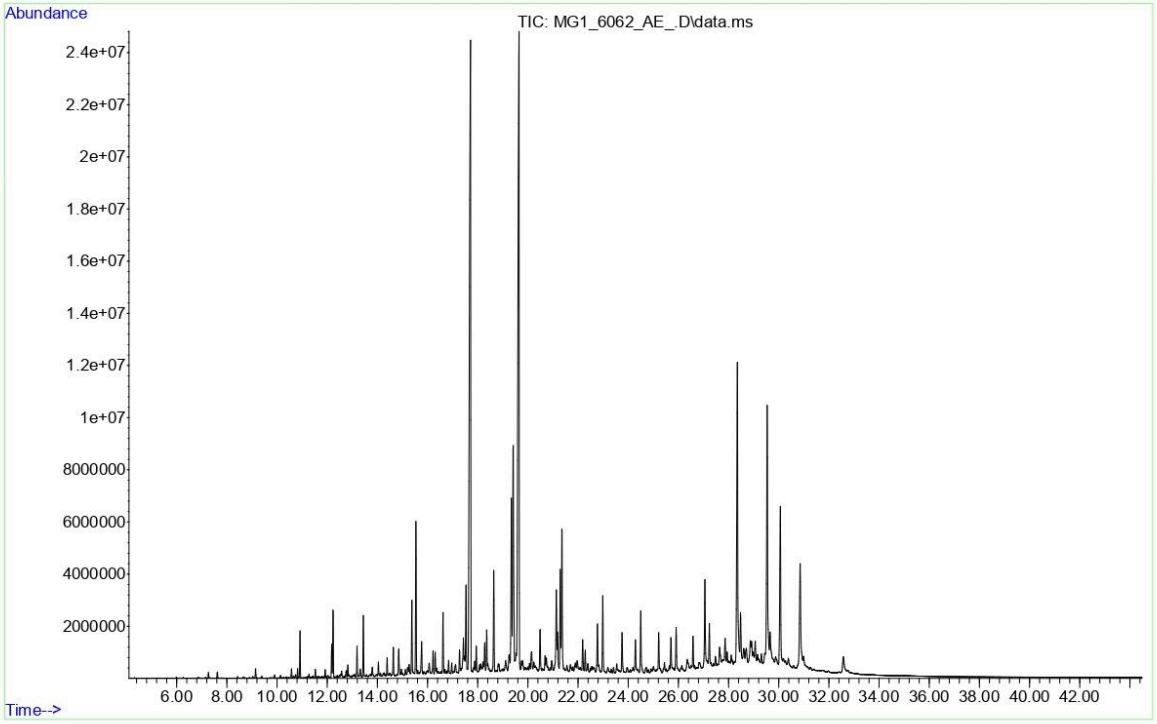
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6129_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 12:59 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6129_AE_MA030621
Misc Info :
Vial Number: 16



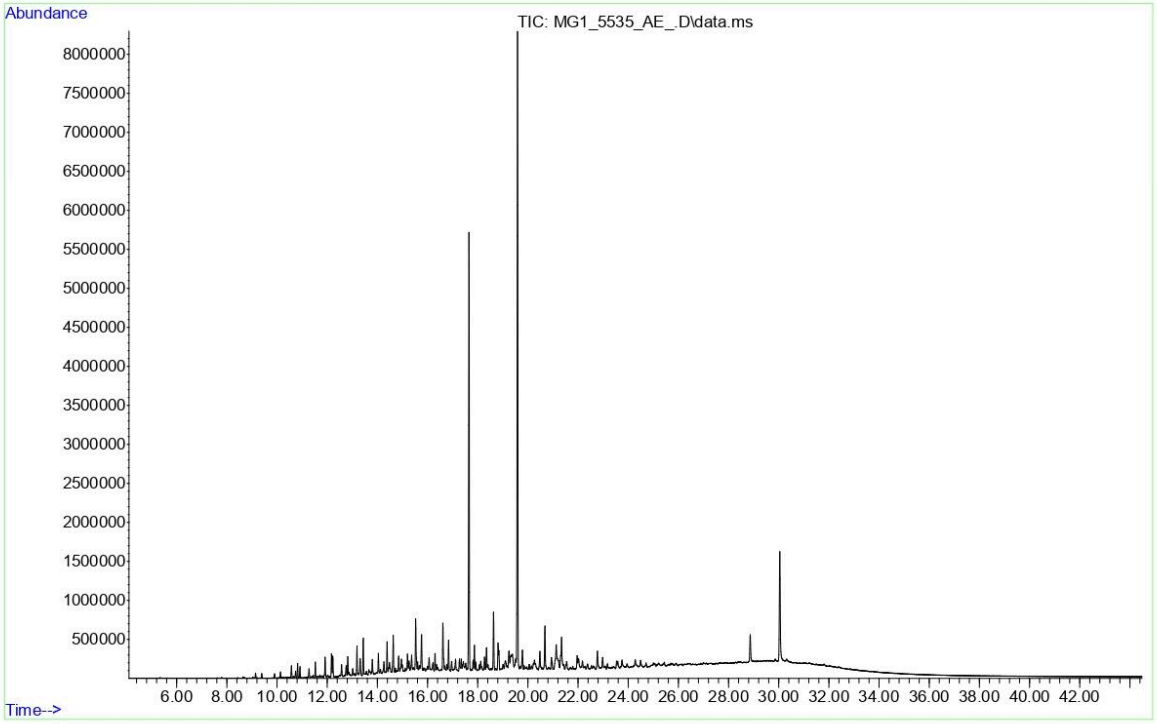
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6071_.D
Operator : hmt
Acquired : 07 Mar 2021 08:19 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6071_MA030321
Misc Info :
Vial Number: 43



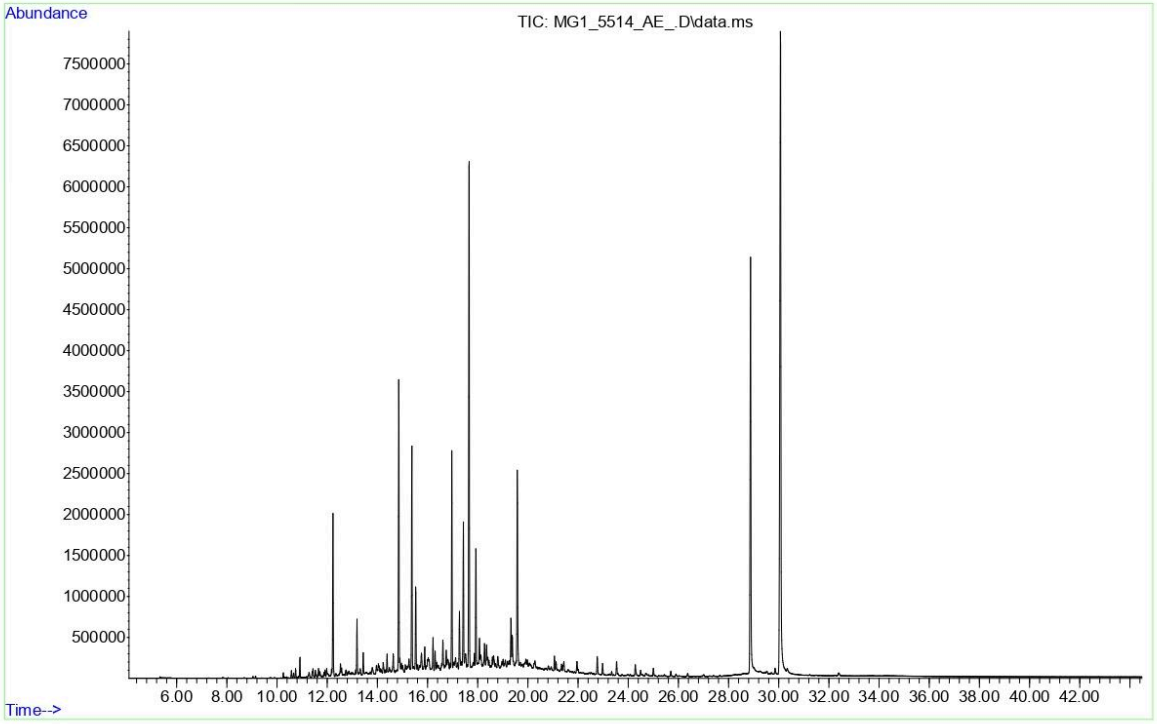
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6062_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 11:35 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6062_AE_MA030621
Misc Info :
Vial Number: 15



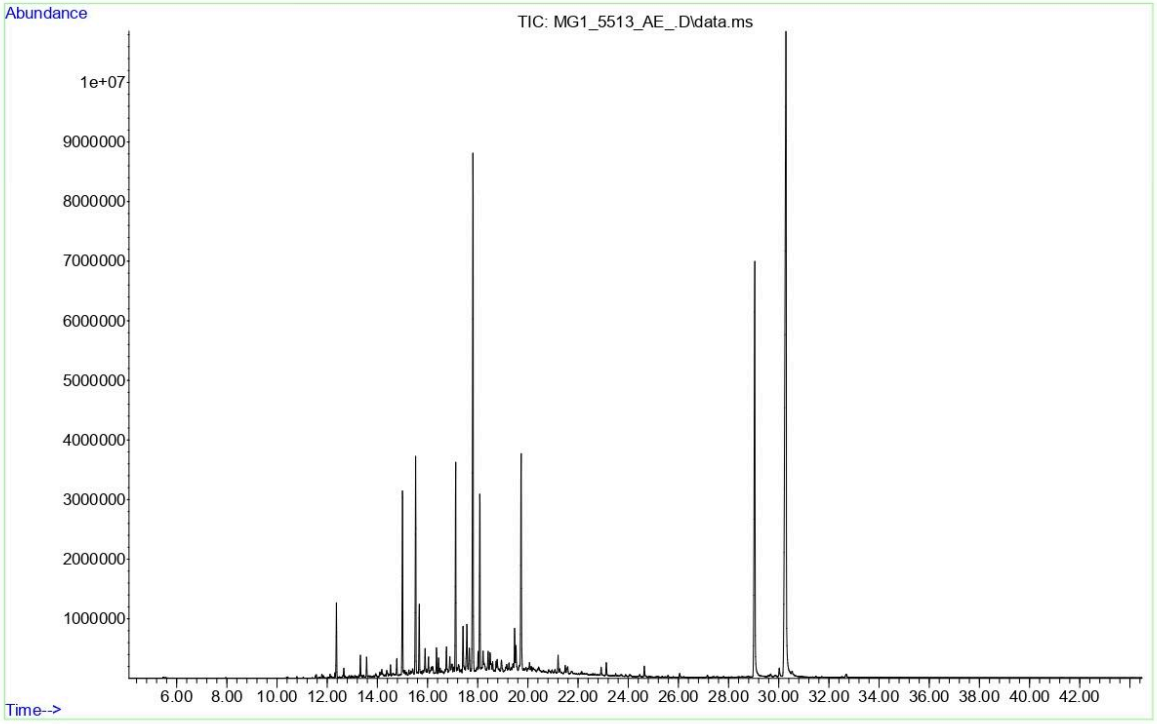
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5535_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 09:52 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5535_AE_MA030621
Misc Info :
Vial Number: 14



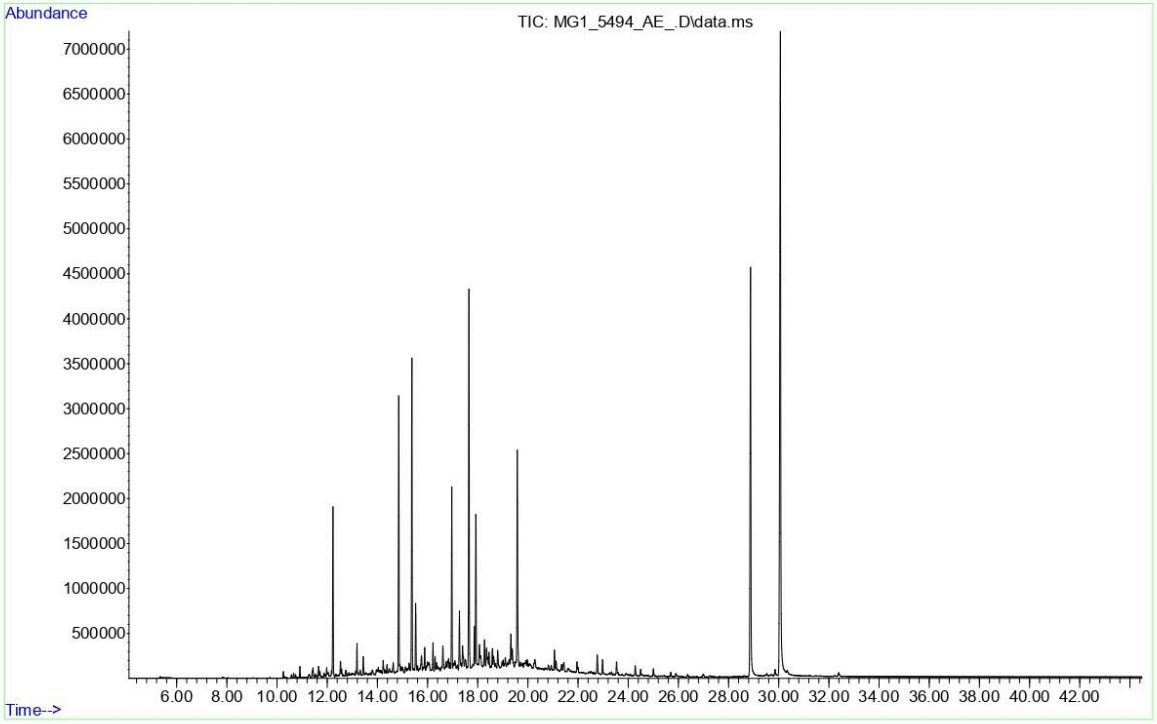
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5514_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 09:01 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5514_AE_MA030621
Misc Info :
Vial Number: 13



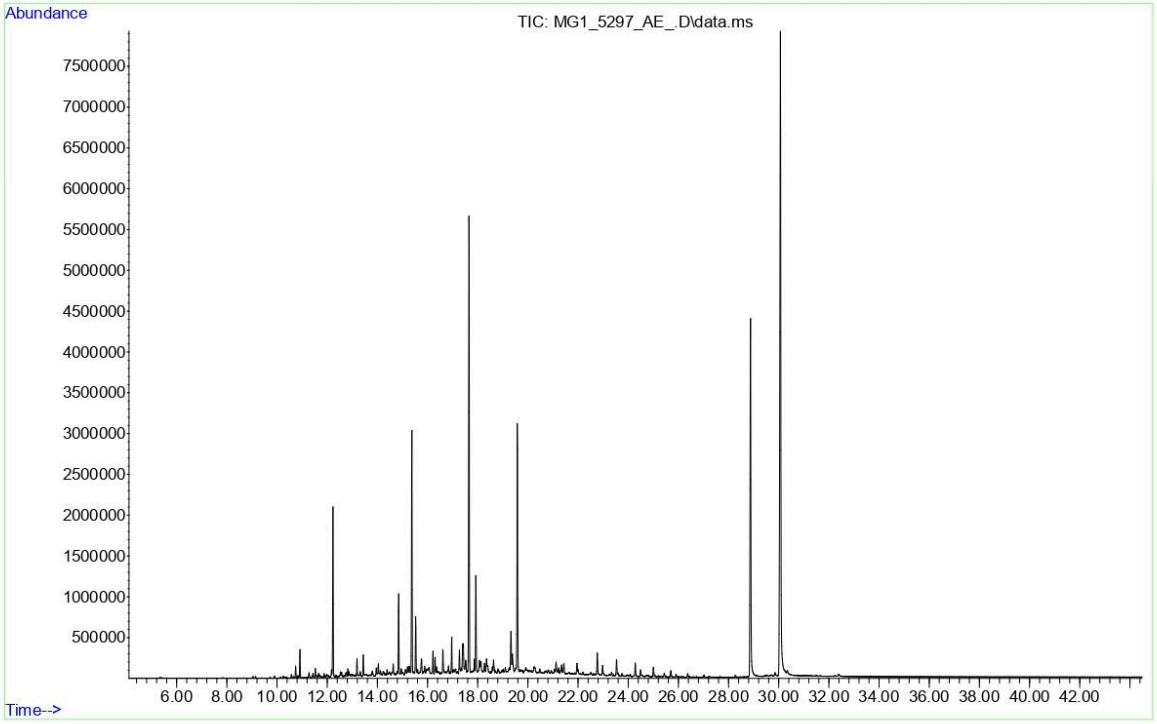
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5513_AE_.D
Operator : hmt
Acquired : 13 May 2021 09:40 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5513_AE_MA12_260421
Misc Info :
Vial Number: 33



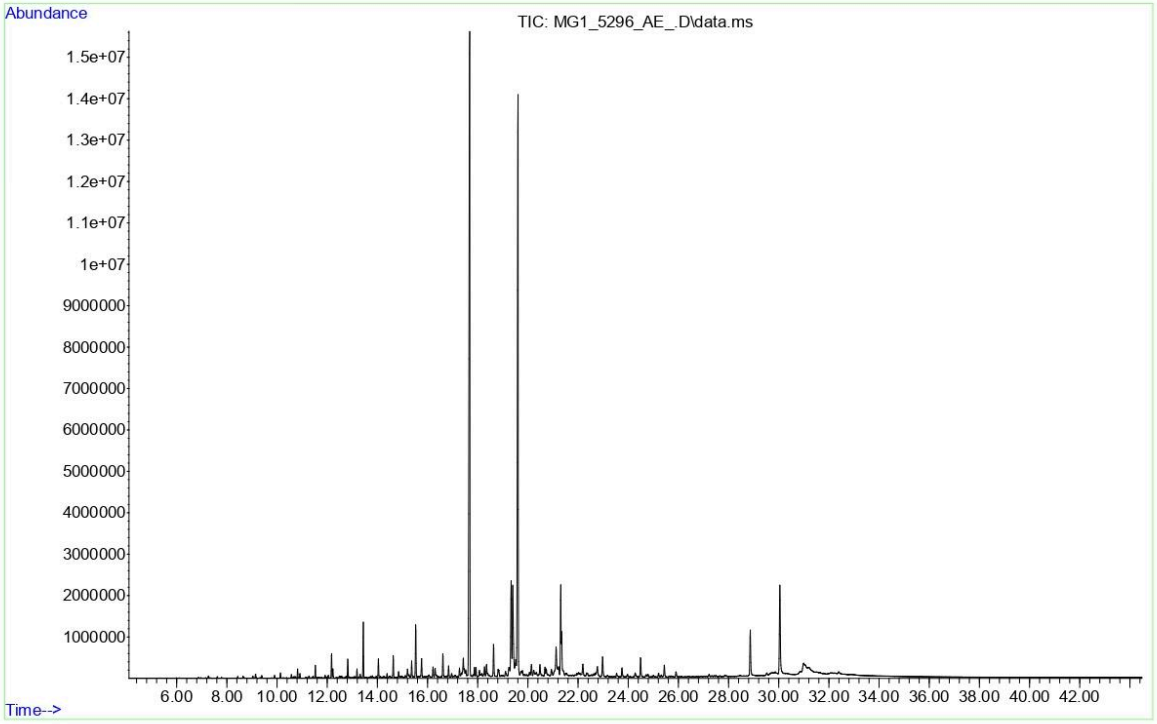
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5494_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 08:09 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5494_AE_MA030621
Misc Info :
Vial Number: 12



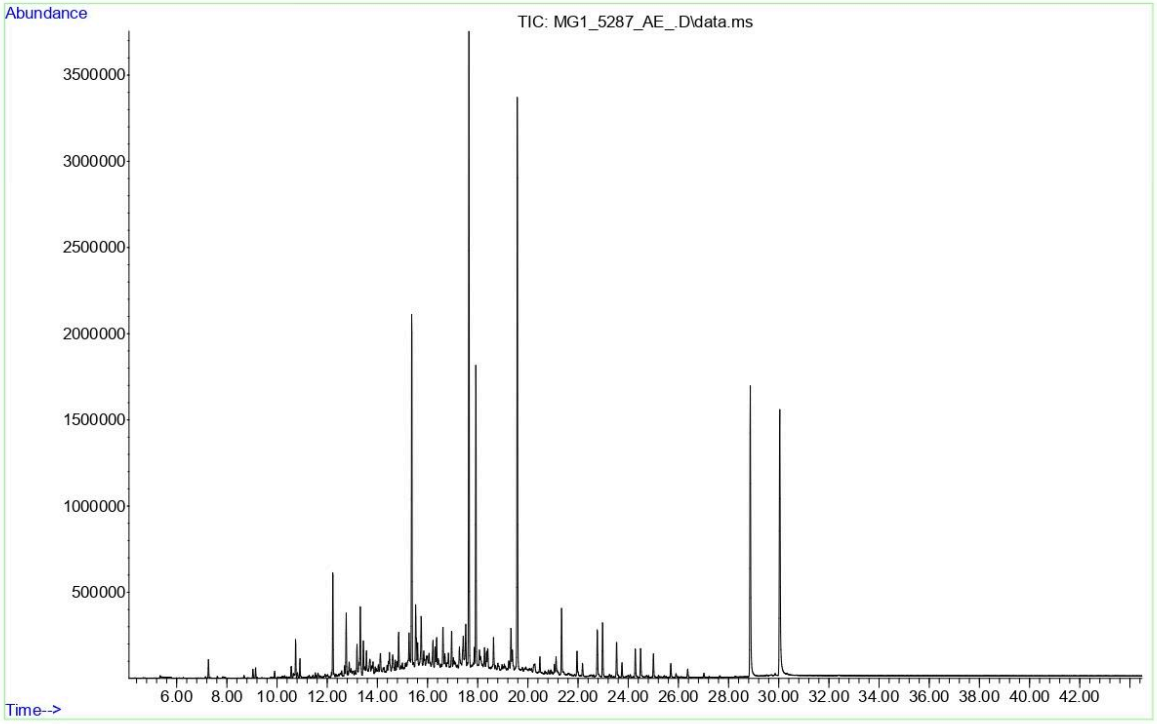
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5297_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 07:18 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5297_AE_MA030621
Misc Info :
Vial Number: 11



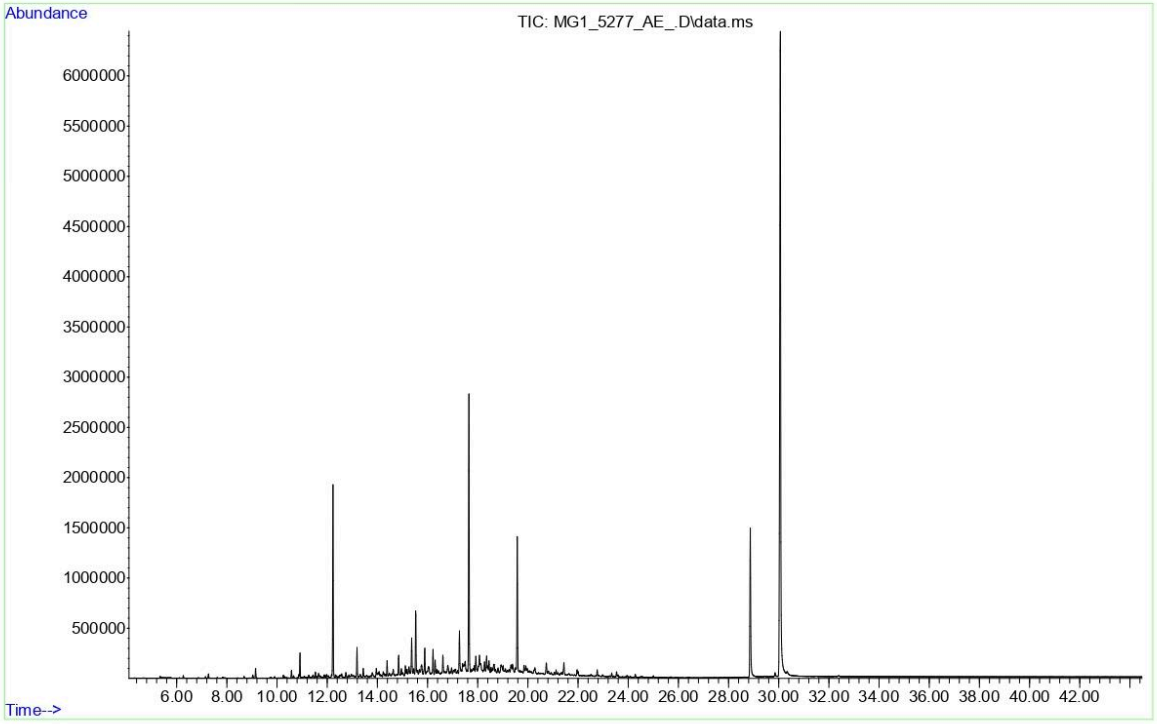
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5296_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 05:34 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5296_AE_MA030621
Misc Info :
Vial Number: 10



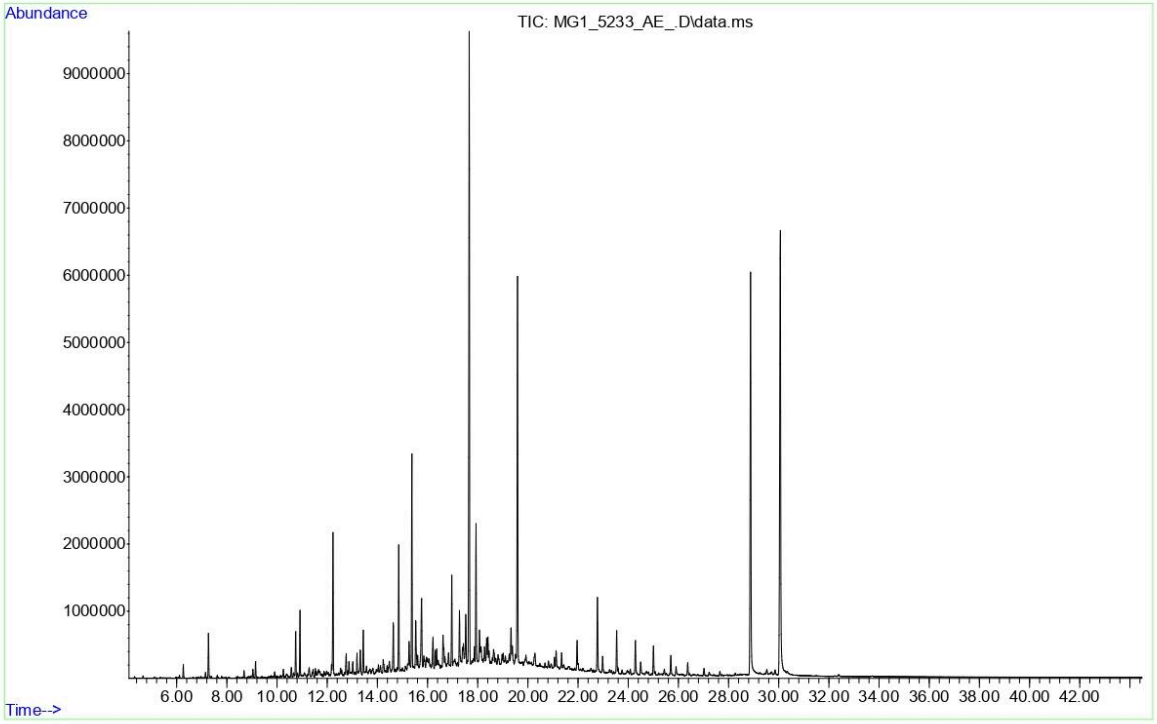
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5287_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 04:43 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5287_AE_MA030621
Misc Info :
Vial Number: 9



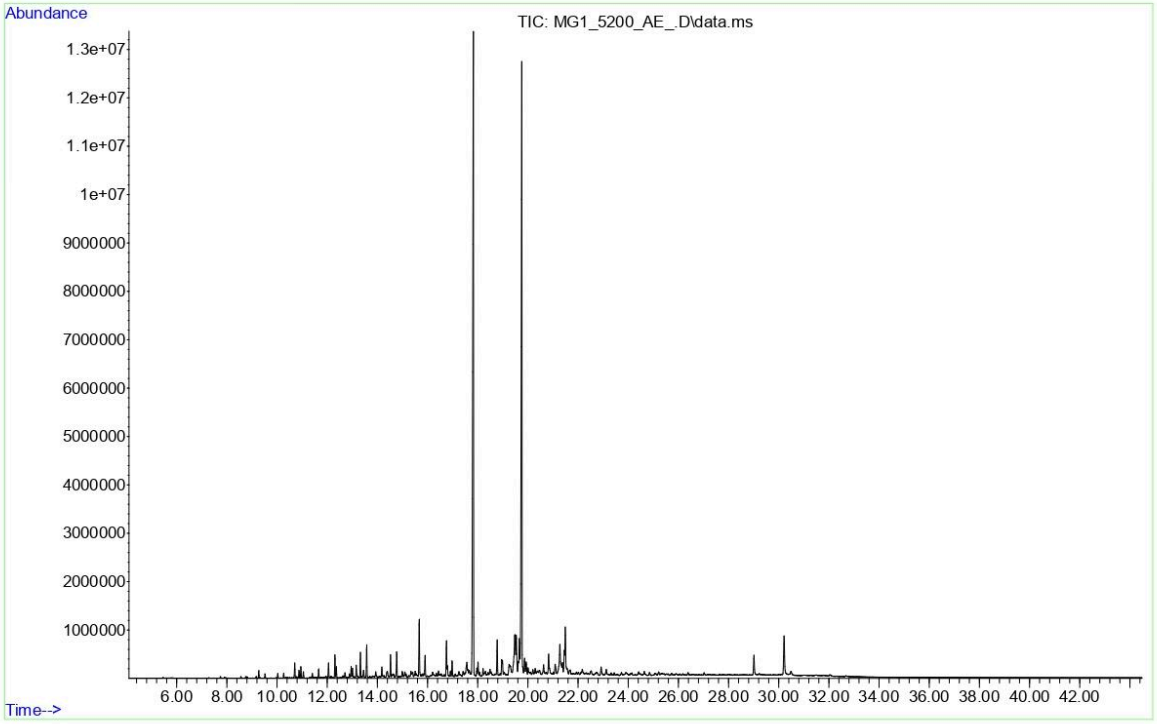
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5277_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 03:51 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5277_AE_MA030621
Misc Info :
Vial Number: 8



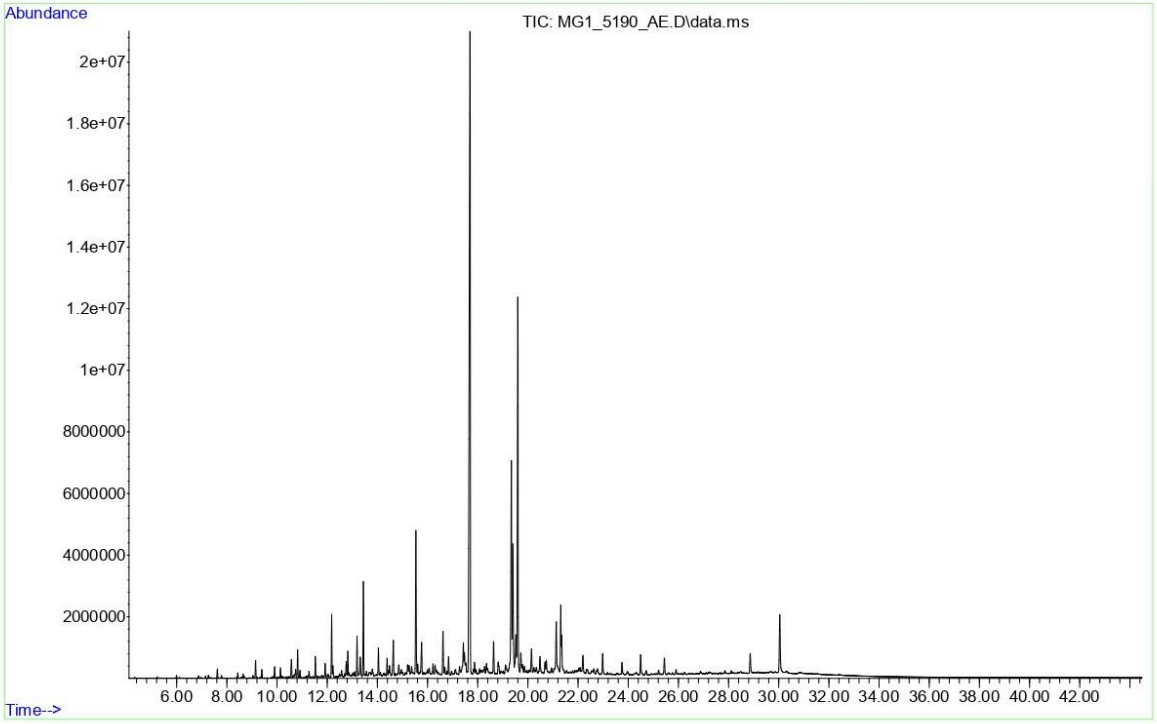
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5233_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 02:59 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5233_AE_MA030621
Misc Info :
Vial Number: 7



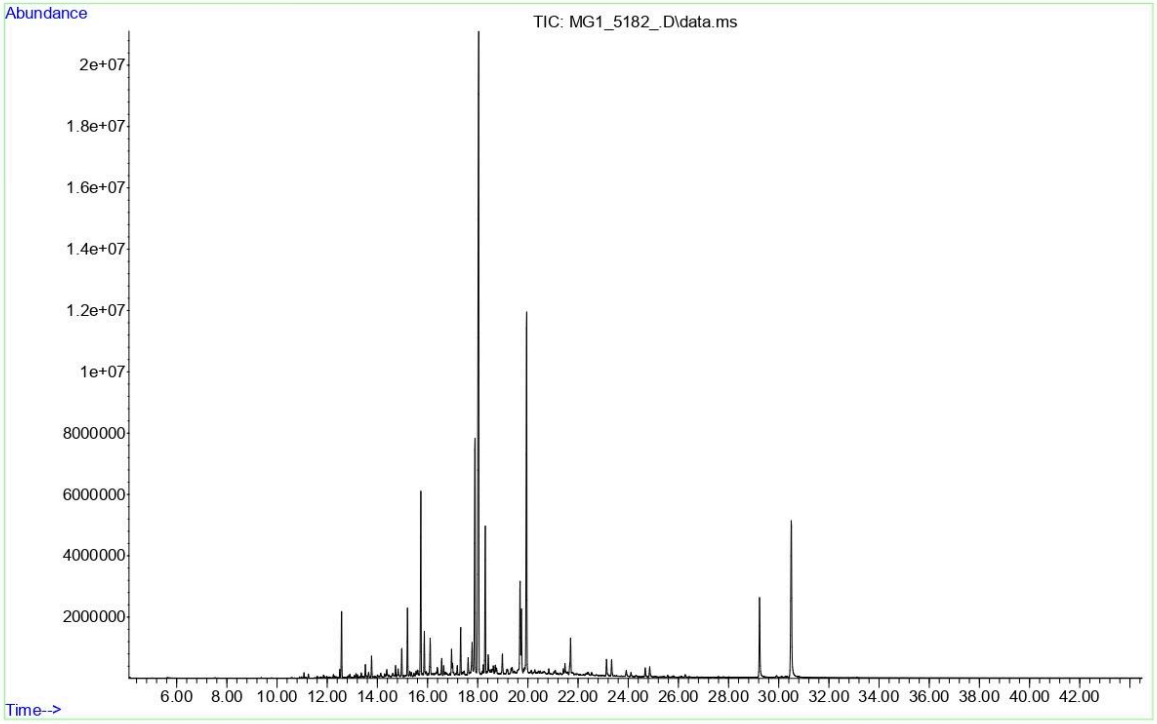
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5200_AE_.D
Operator : hmt
Acquired : 13 May 2021 01:13 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5200_AE_MA12_260421
Misc Info :
Vial Number: 32



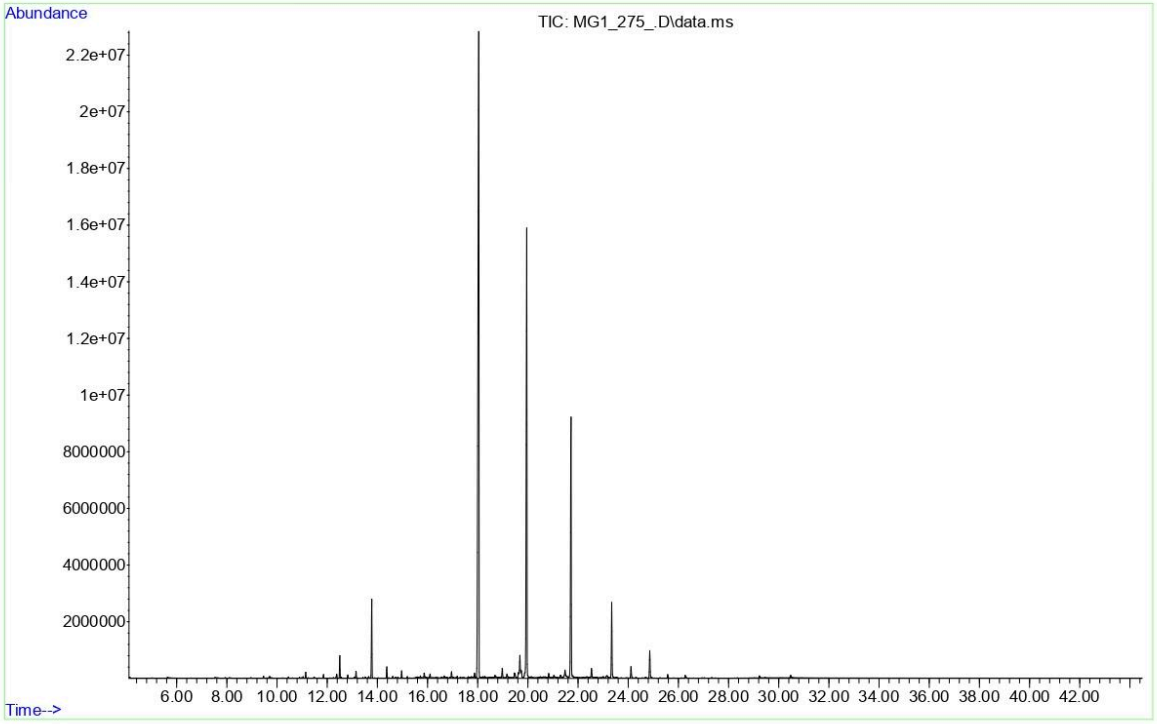
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5190_AE.D
Operator : hmt
Acquired : 08 Jun 2021 01:16 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5190_AE_MA030621
Misc Info :
Vial Number: 6



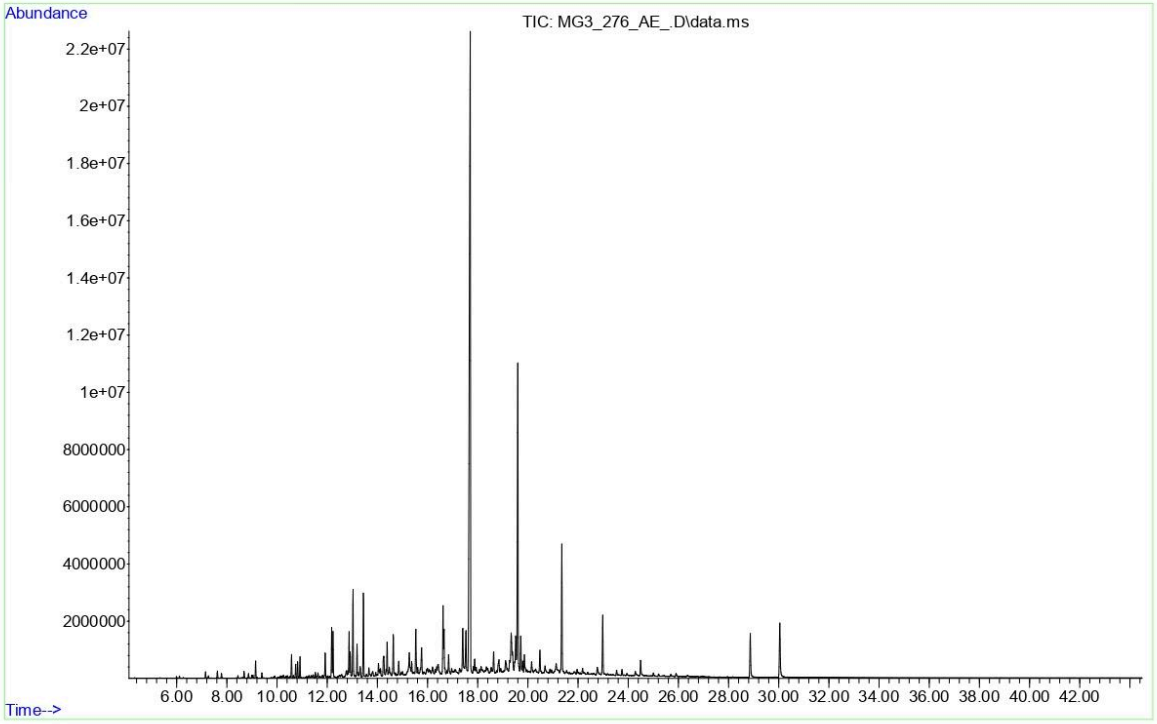
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_5182_.D
Operator : hmt
Acquired : 07 Mar 2021 09:10 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_5182_MA030321
Misc Info :
Vial Number: 44



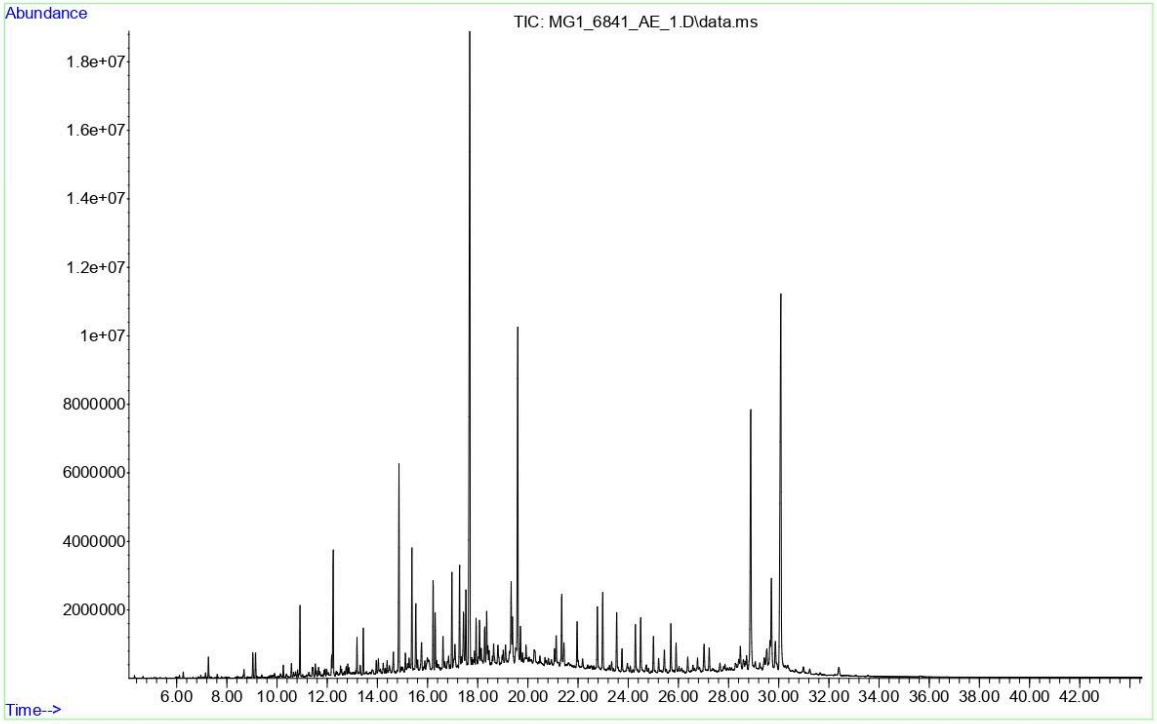
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_275_.D
Operator : hmt
Acquired : 07 Mar 2021 11:43 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_275_R_MA030321
Misc Info :
Vial Number: 46



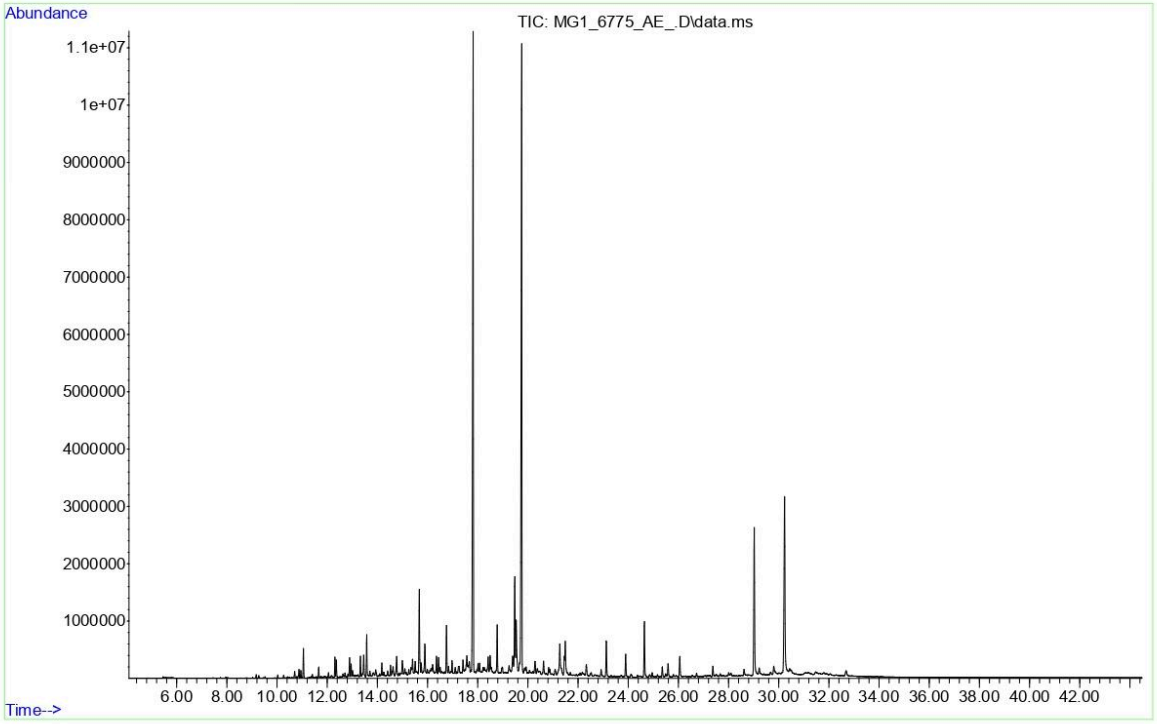
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG3_276_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 00:24 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG3_276_AE_MA030621
Misc Info :
Vial Number: 5



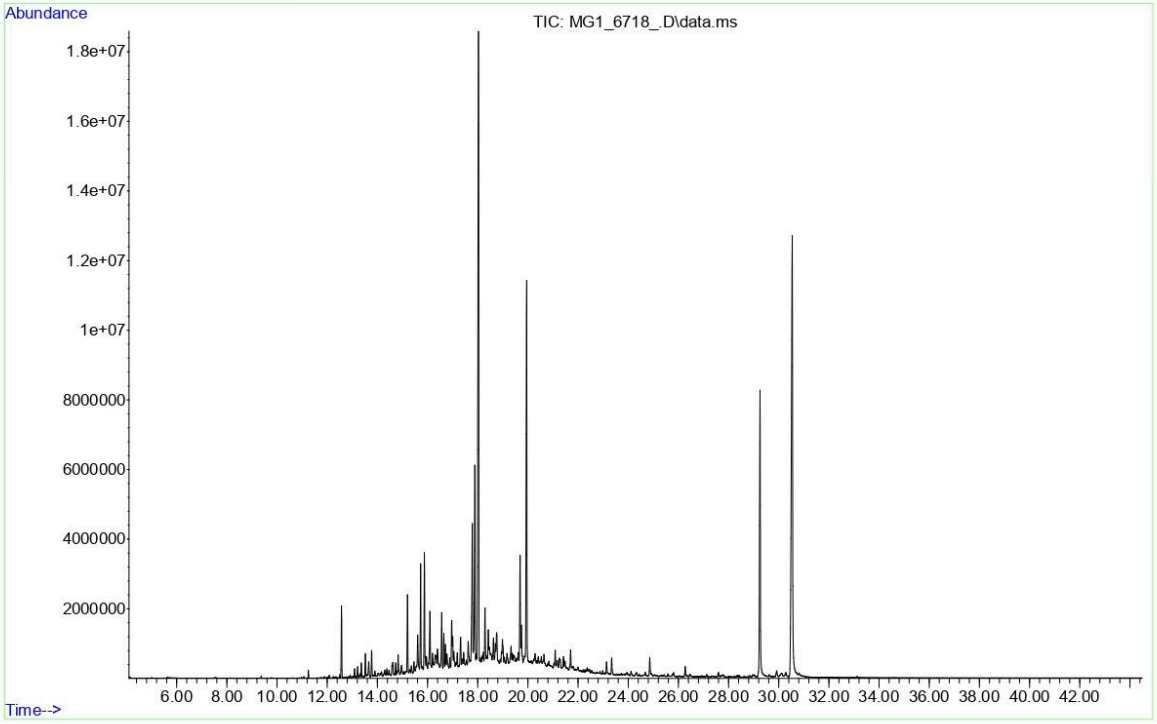
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6841_AE_1.D
Operator : hmt
Acquired : 08 Jun 2021 22:07 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6841_AE_MA030621
Misc Info :
Vial Number: 24



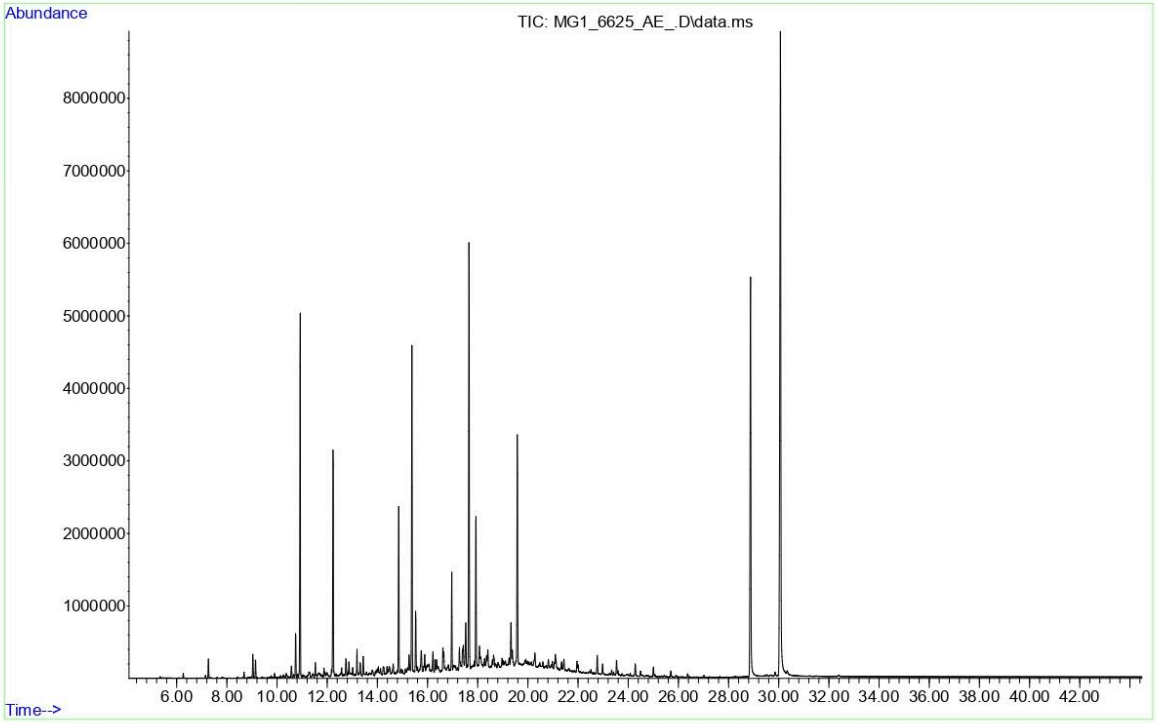
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6775_AE_.D
Operator : hmt
Acquired : 13 May 2021 14:48 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6775_AE_MA12_260421
Misc Info :
Vial Number: 38



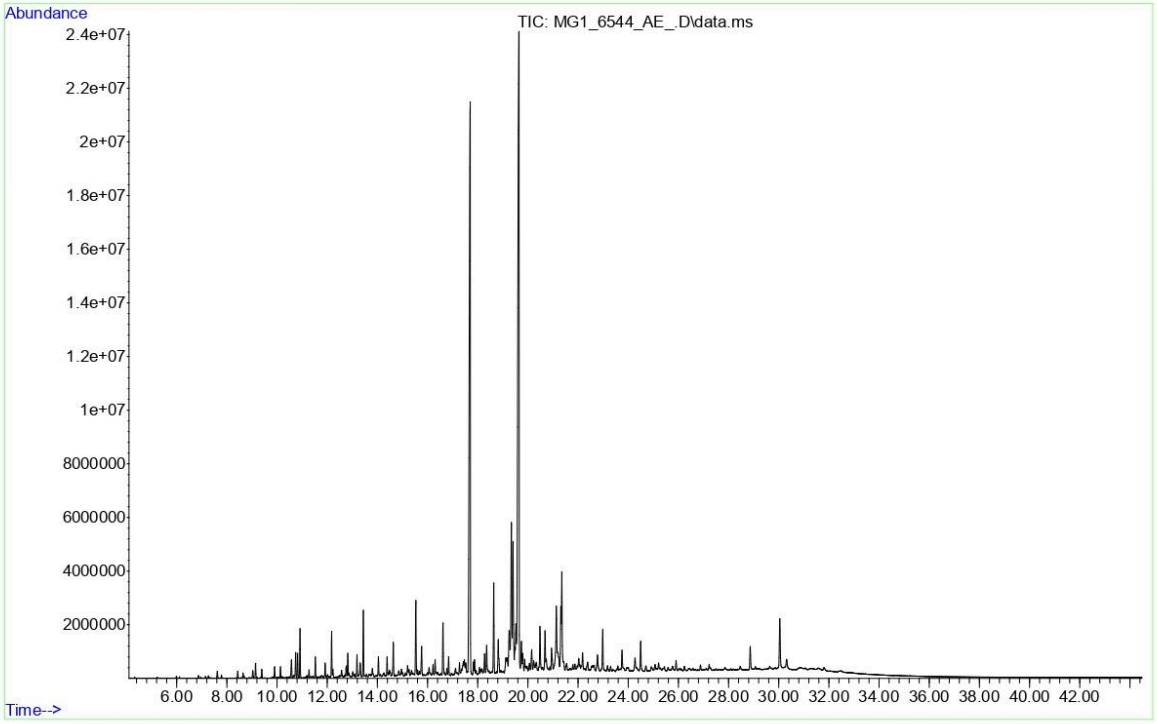
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6718_.D
Operator : hmt
Acquired : 07 Mar 2021 12:34 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6718_MA030321
Misc Info :
Vial Number: 47



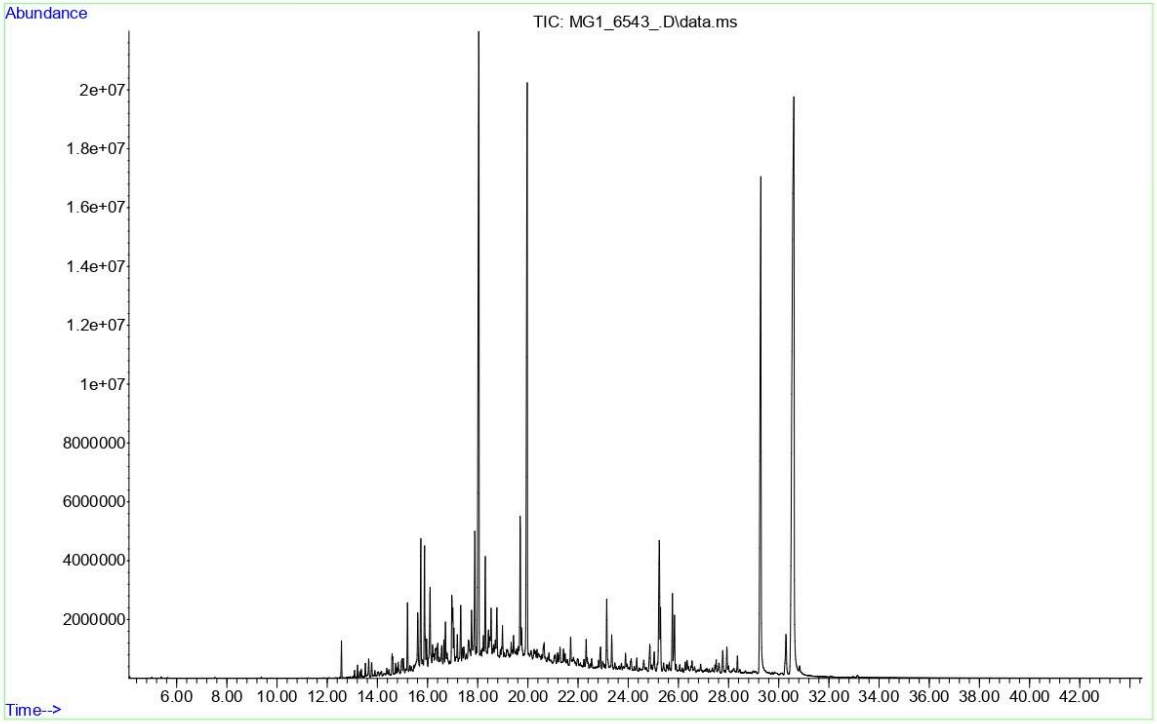
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6625_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 21:16 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6625_AE_MA030621
Misc Info :
Vial Number: 23



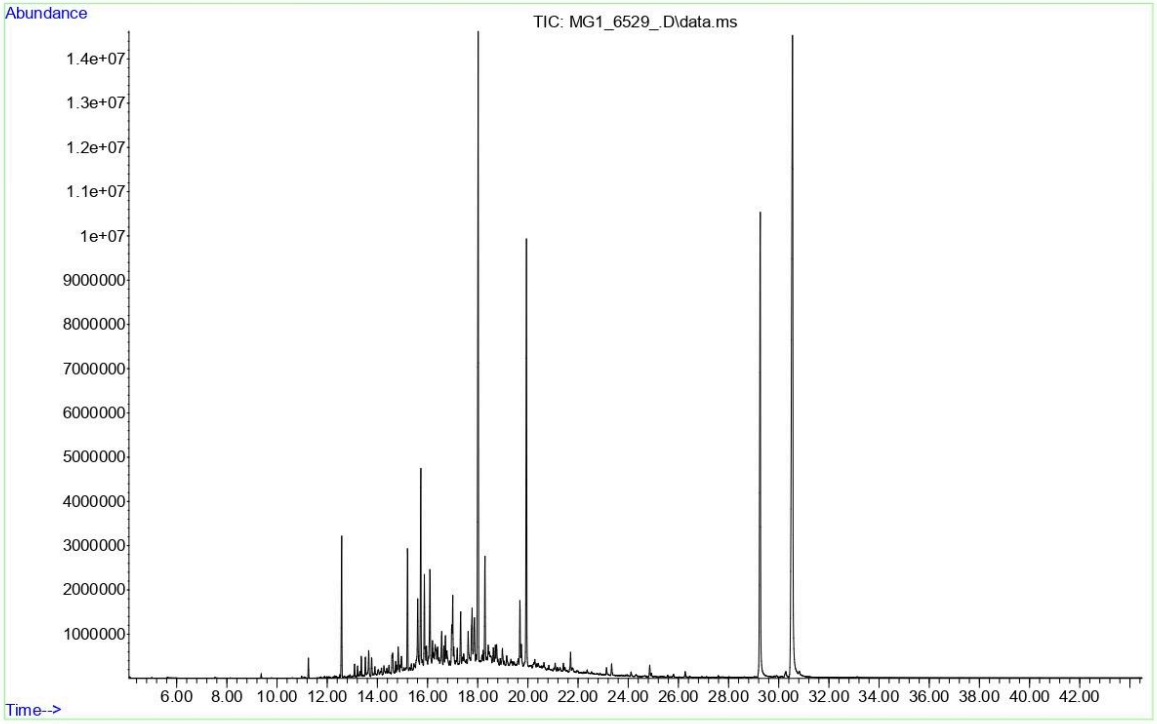
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6544_AE_.D
Operator : hmt
Acquired : 08 Jun 2021 19:33 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6544_AE_MA030621
Misc Info :
Vial Number: 22



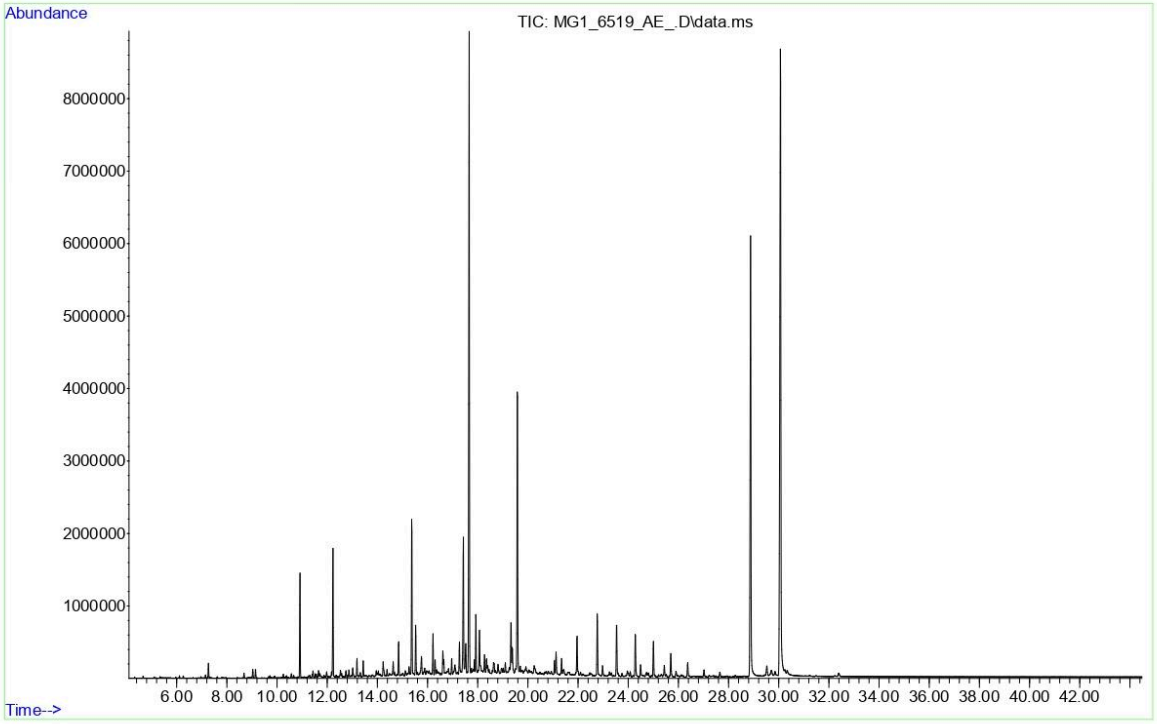
File :G:\Data by Site\MorroGrande\DB5-GCMS\MG1_6543_.D
Operator : hmt
Acquired : 07 Mar 2021 15:58 using AcqMethod DB5MS_CDS_BIOL.M
Instrument : MSD
Sample Name: MG1_6543_MA030321
Misc Info :
Vial Number: 50



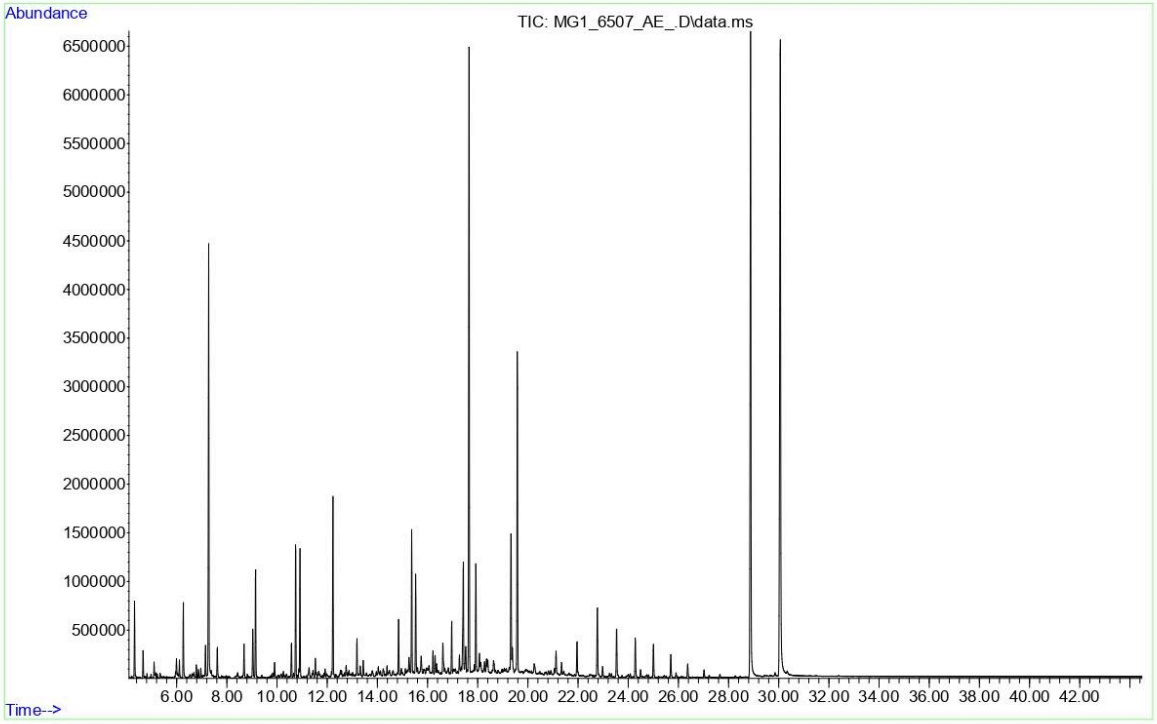
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Instrument : MSD
Sample Name: MG1_6529_MA030321
Misc Info :
Vial Number: 48



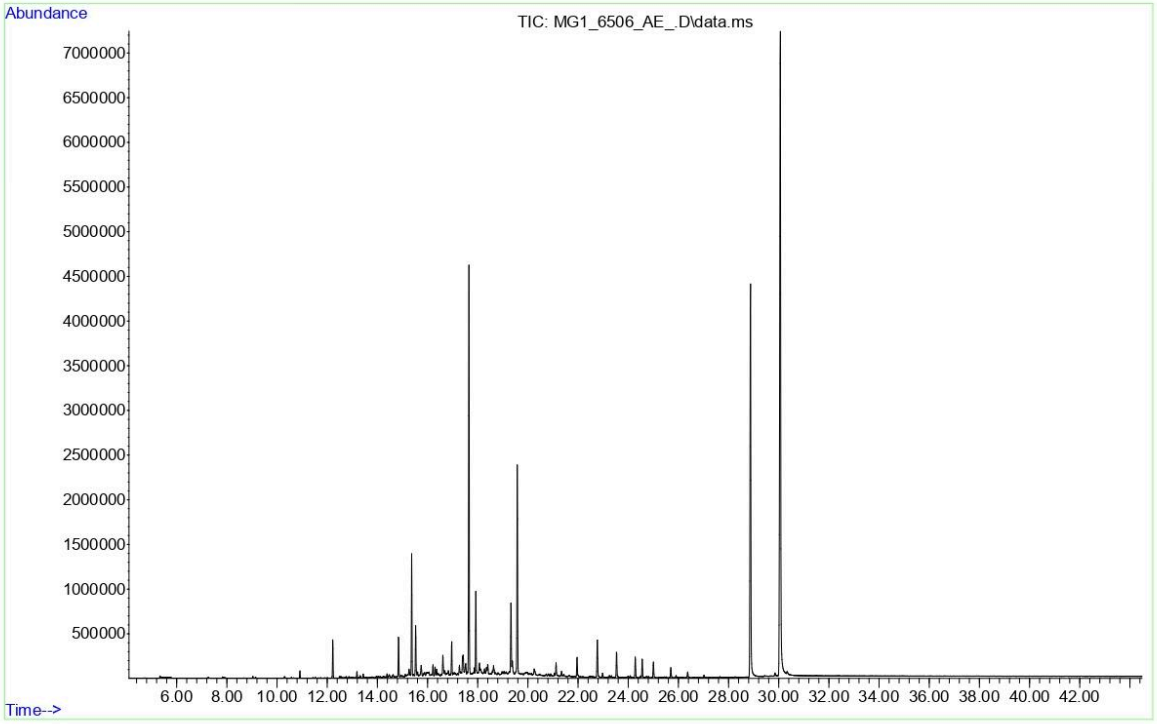
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Instrument : MSD
Sample Name: MG1_6519_AE_MA030621
Misc Info :
Vial Number: 21



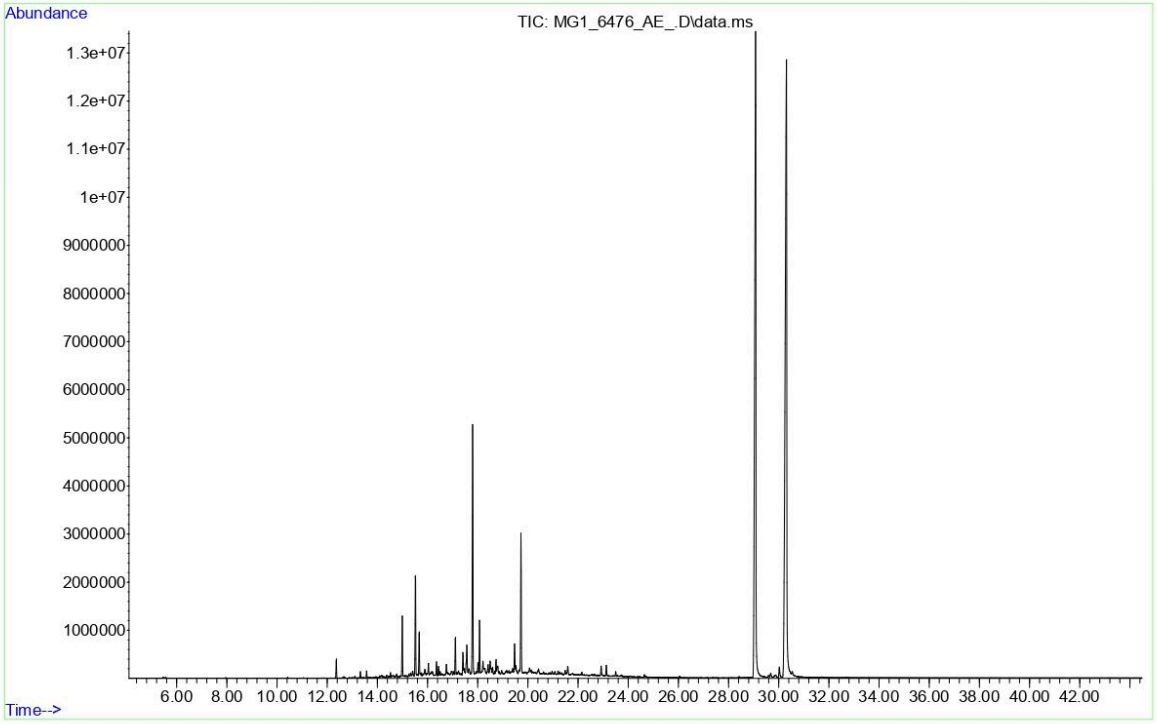
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Misc Info :
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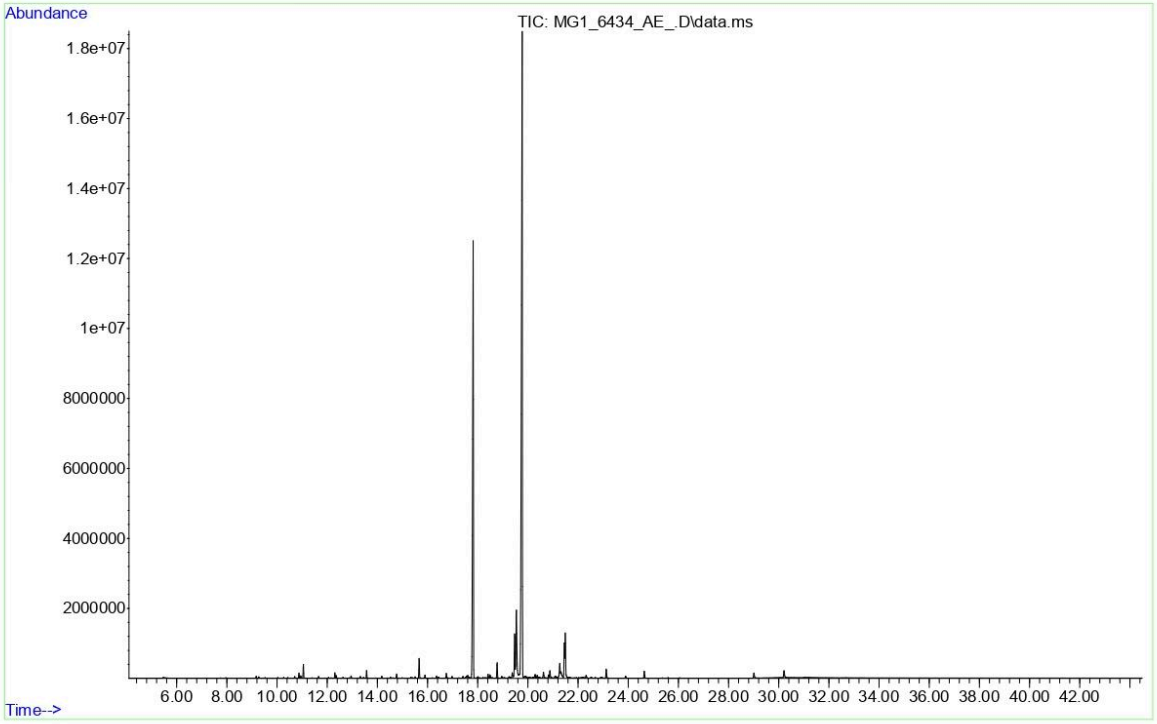
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Instrument : MSD
Sample Name: MG1_6506_AE_MA030621
Misc Info :
Vial Number: 19



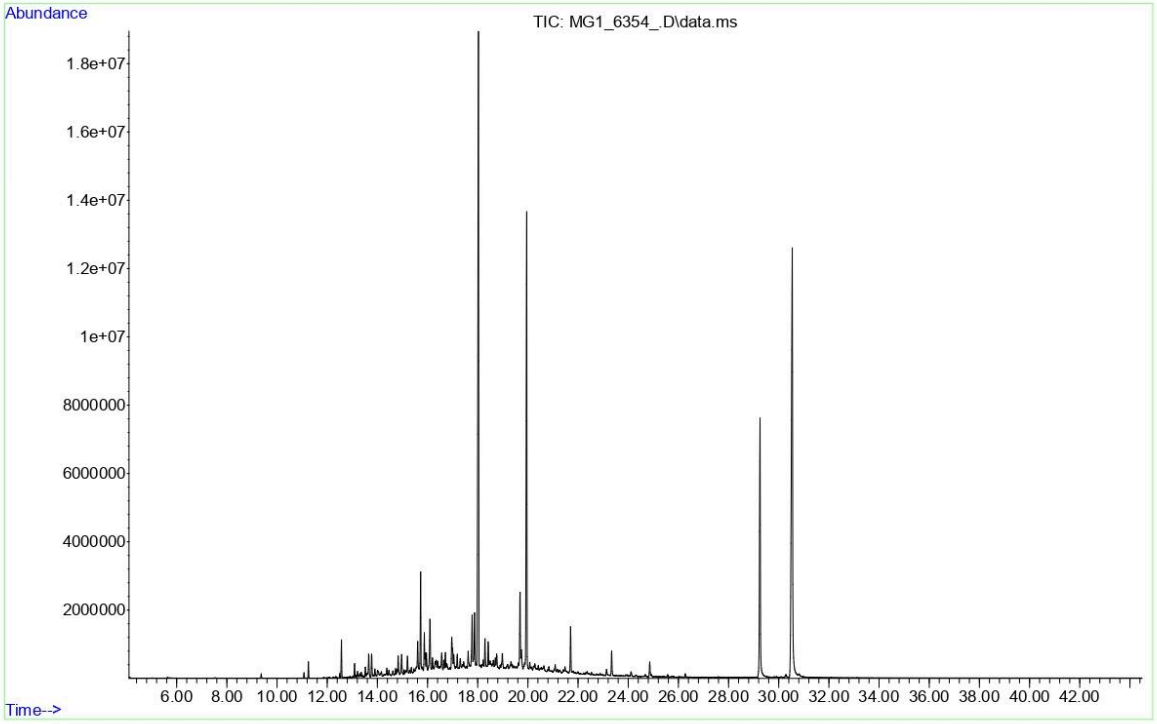
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Instrument : MSD
Sample Name: MG1_6476_AE_MA12_260421
Misc Info :
Vial Number: 37



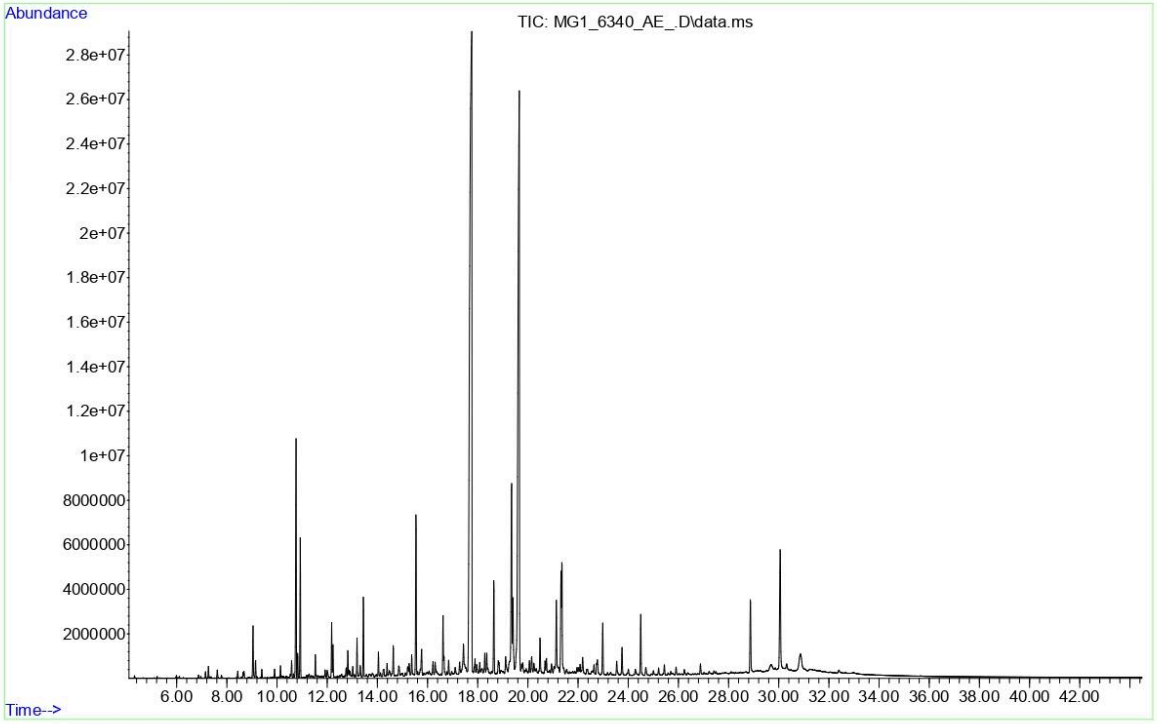
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Instrument : MSD
Sample Name: MG1_6434_AE_MA12_260421
Misc Info :
Vial Number: 36



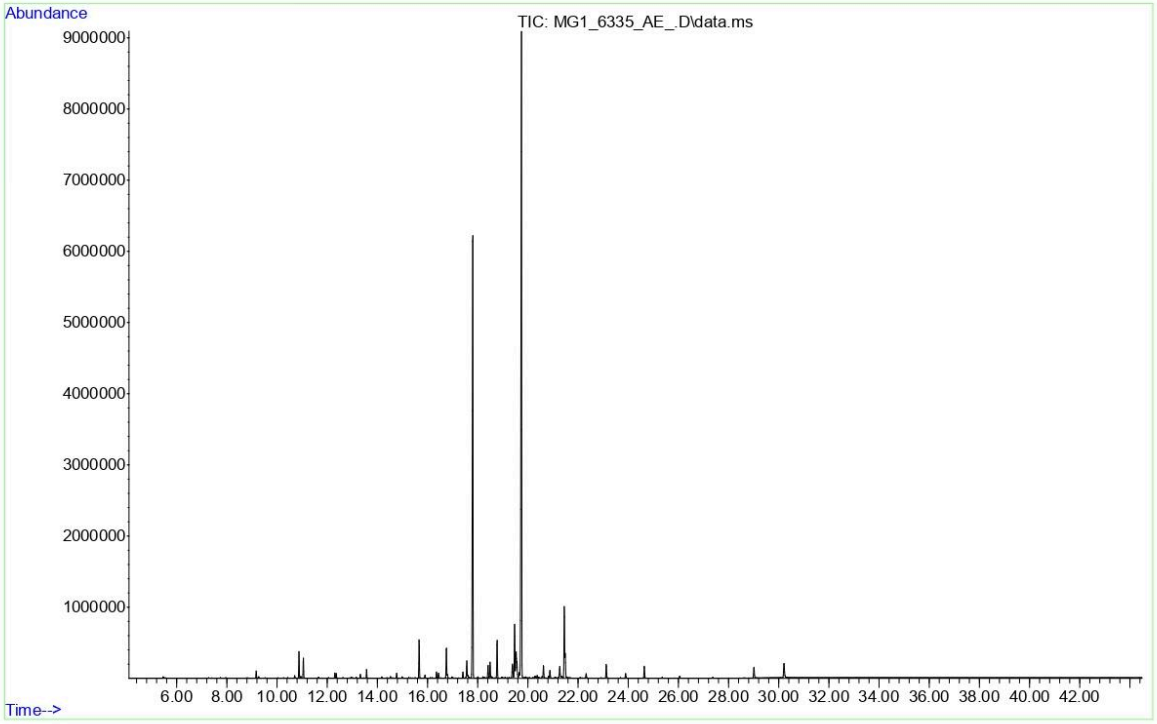
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Misc Info :
Vial Number: 49



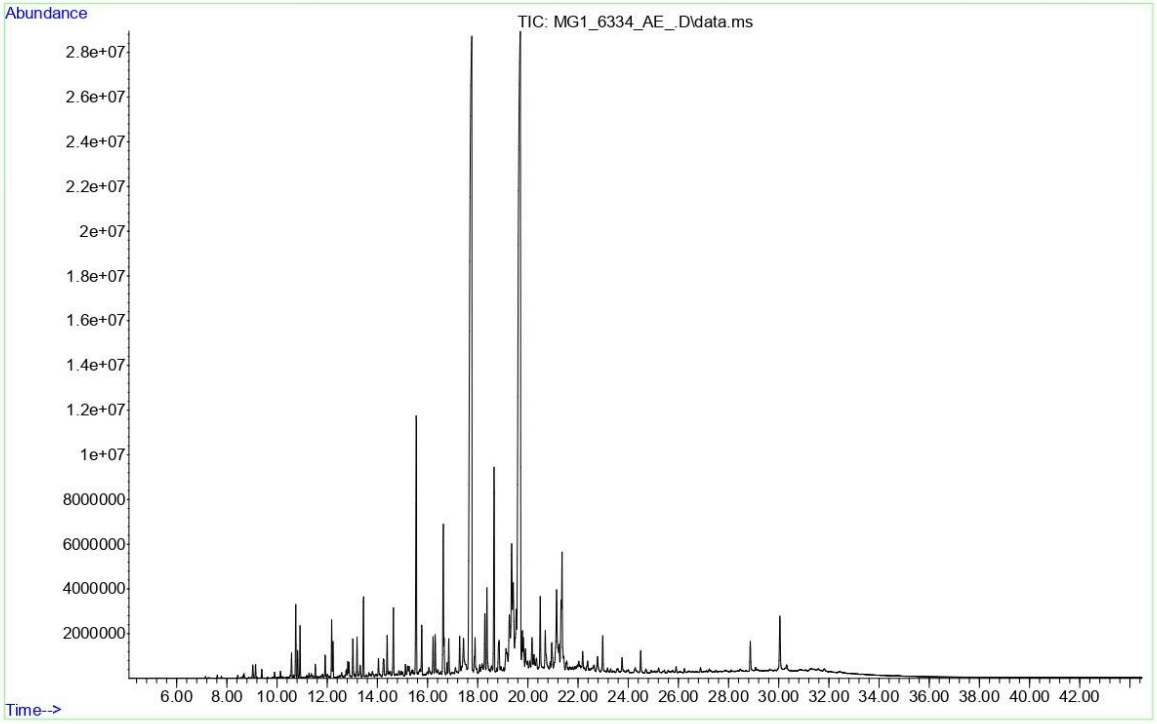
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Sample Name: MG1_6340_AE_MA030621
Misc Info :
Vial Number: 18



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Sample Name: MG1_6335_AE_MA12_260421
Misc Info :
Vial Number: 35



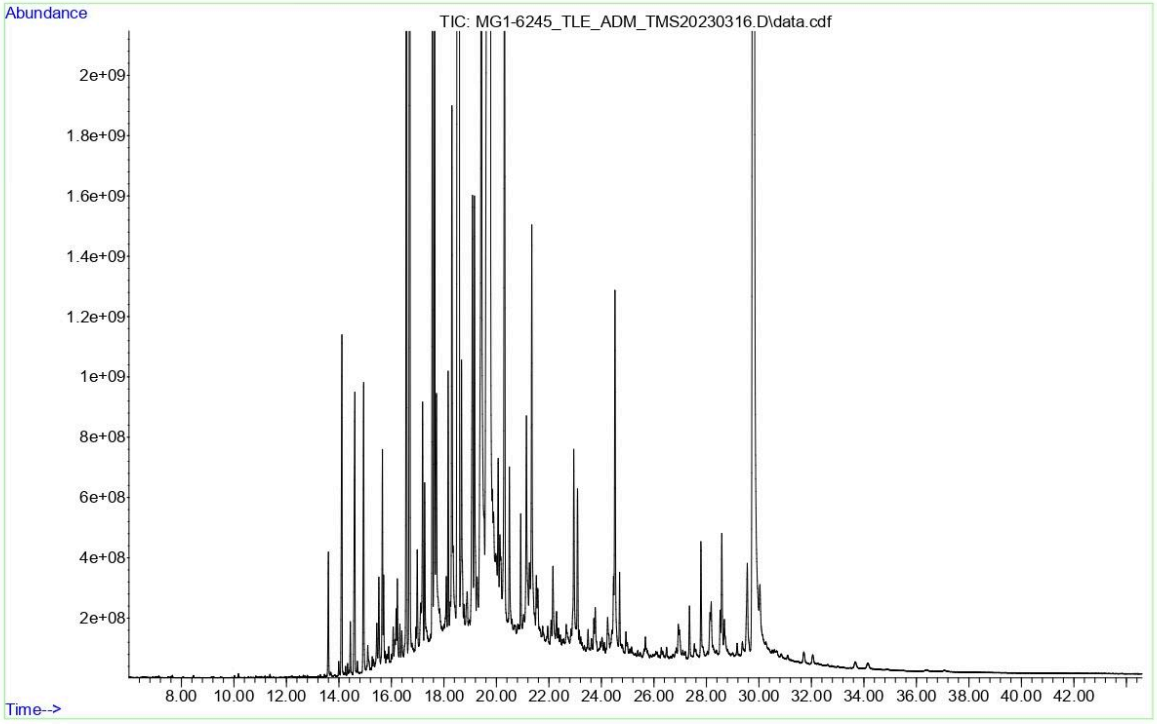
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Misc Info :
Vial Number: 17



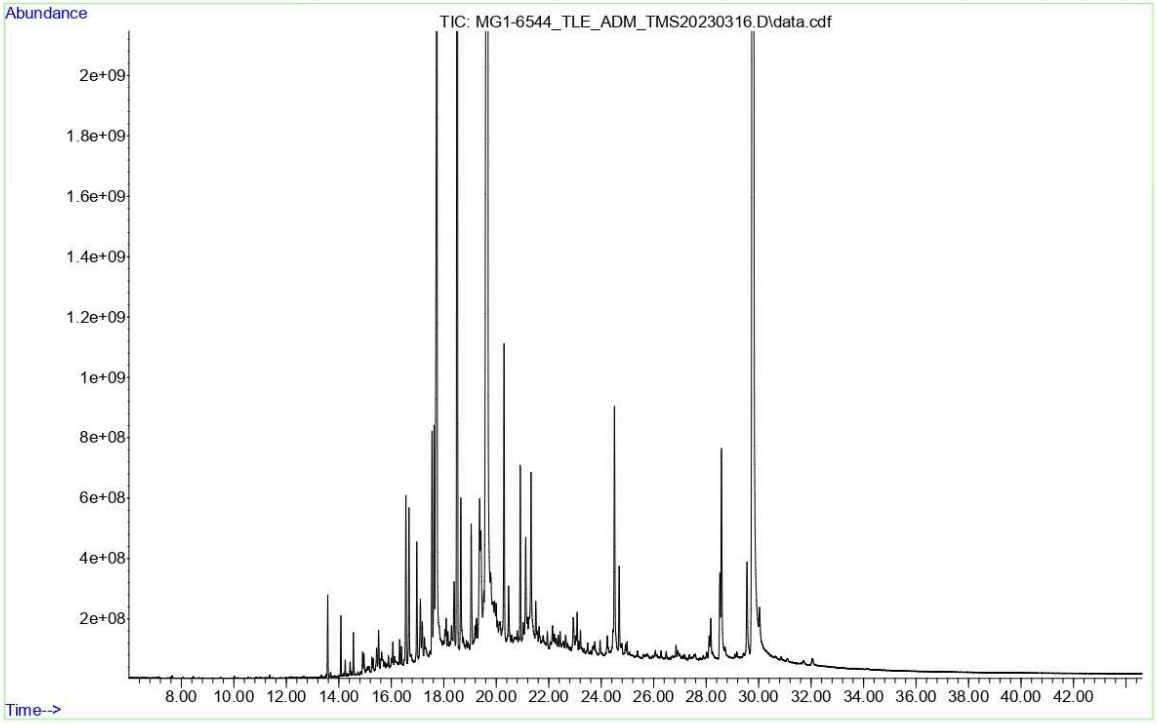
Morro Grande

Total lipid extraction / TMS chromatograms

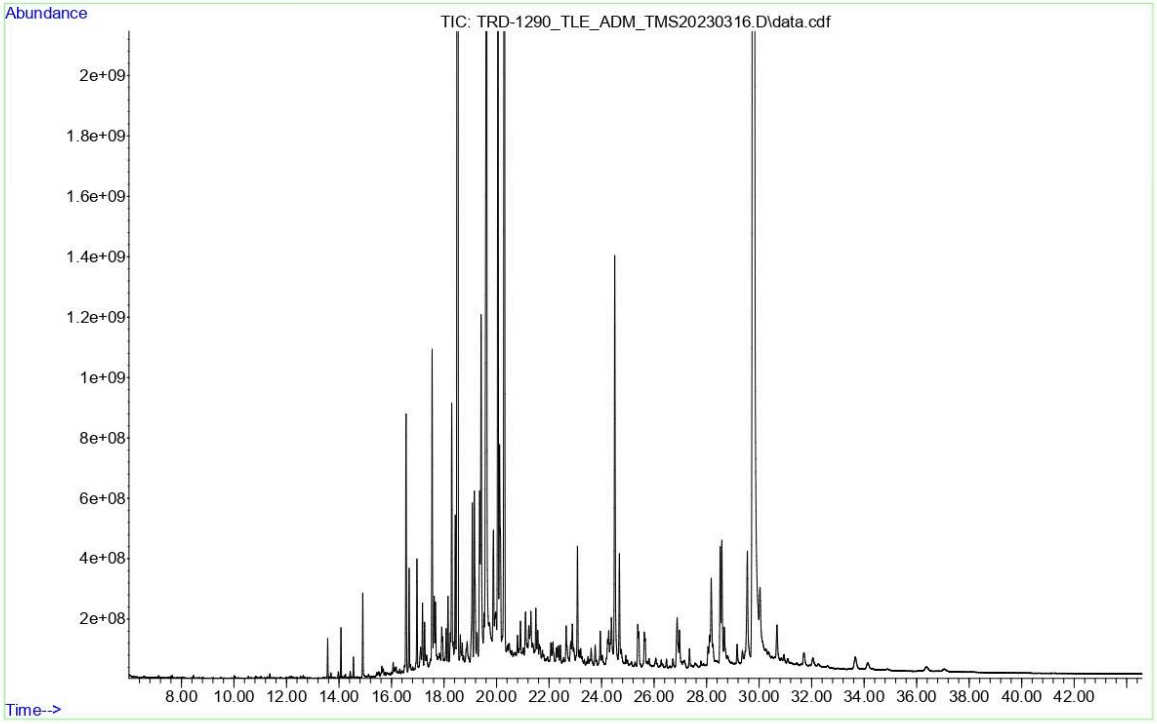
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Instrument : LCQ
Acquired : 20230317154823+0000 using AcqMethod
Sample Name:
Misc Info :



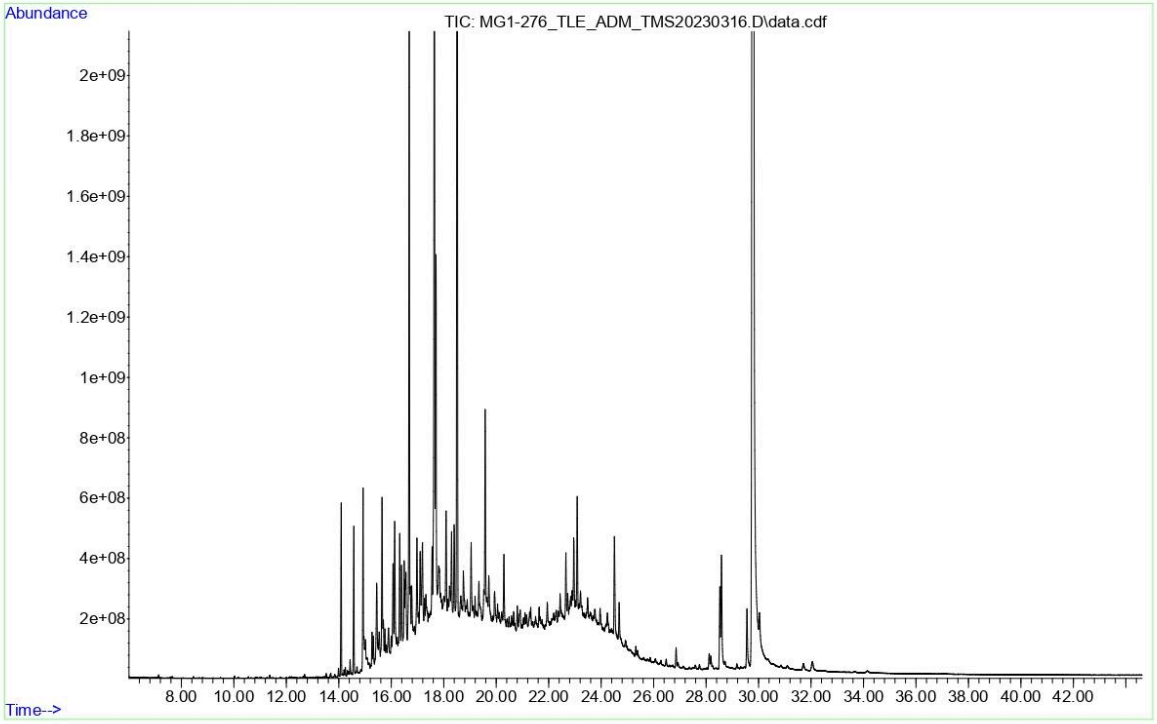
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Sample Name:
Misc Info :



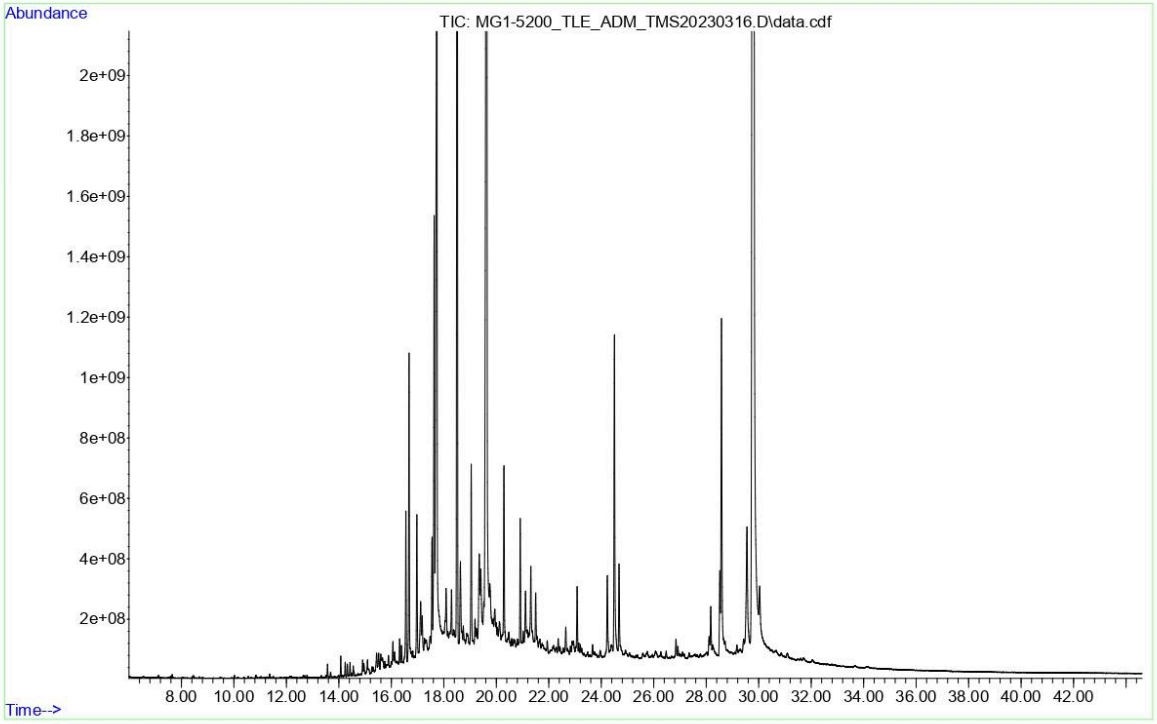
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Sample Name:
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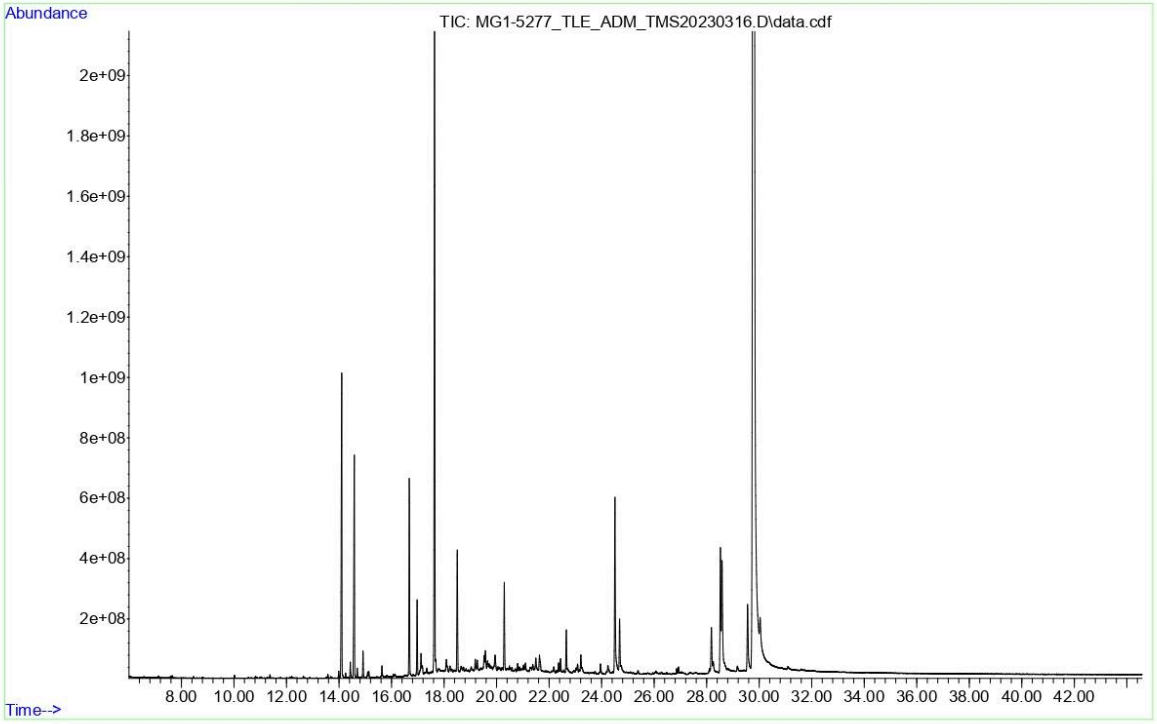
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Sample Name:
Misc Info :



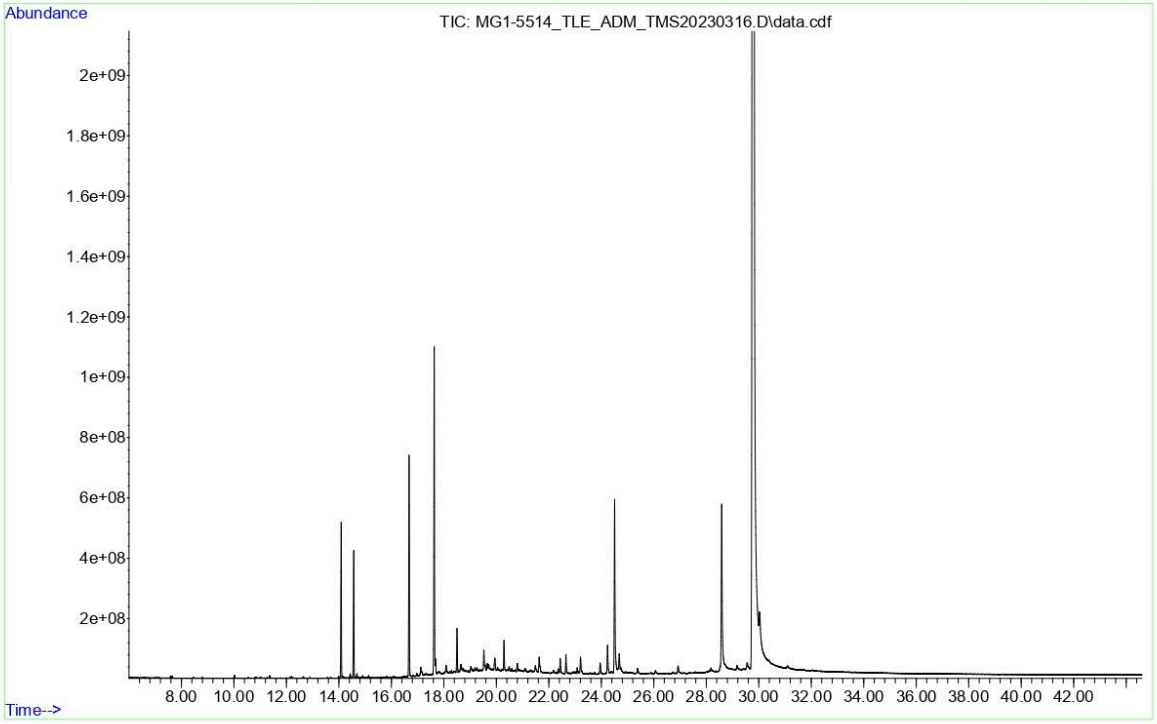
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Sample Name:
Misc Info :



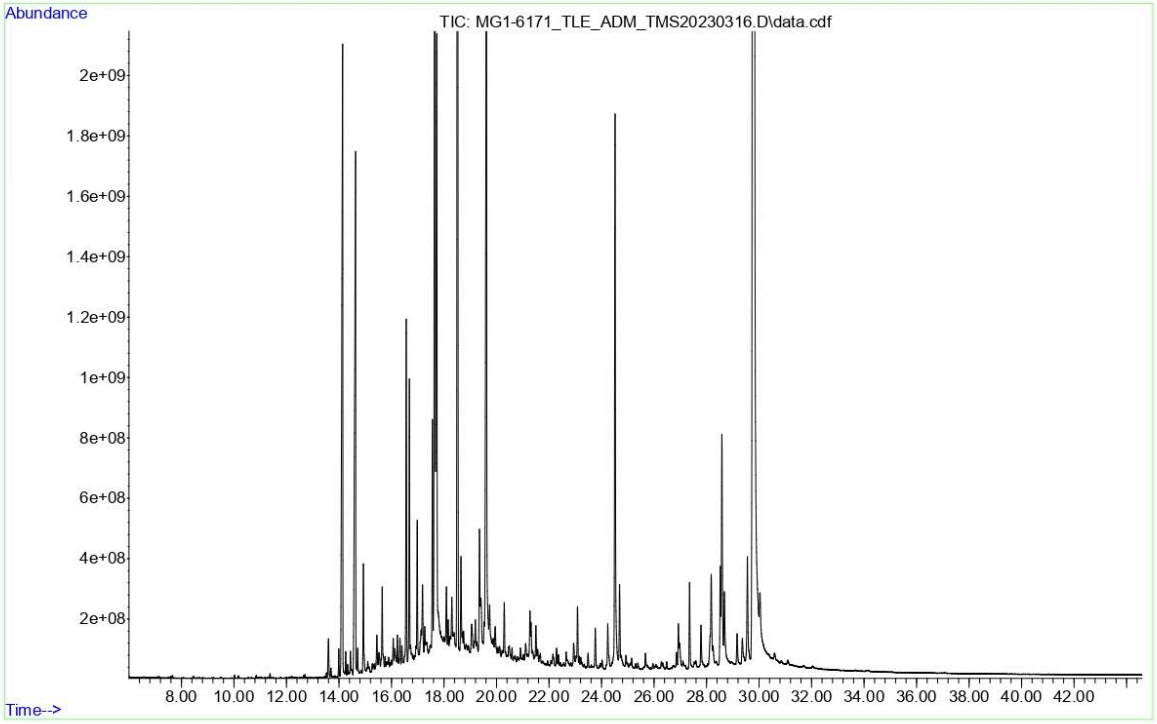
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Sample Name:
Misc Info :



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Acquired : 20230318000230+0000 using AcqMethod
Sample Name:
Misc Info :



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Acquired : 20230317164036+0000 using AcqMethod
Sample Name:
Misc Info :



APPENDIX 3 - Modern references experiments for APAA C_{18} AND C_{20}

This appendix reports the modern reference experiments used to support the interpretation of APAA-C18 E/H and APAA-C20/C18 values. The table includes the reference material, experimental treatment, relevant APAA values and the units used for reporting compound abundance or concentration where applicable. Because some heated plant reference samples also produced measurable APAA-C20/C18 values, this dataset is used to evaluate the behaviour of the threshold rather than to treat it as a definitive aquatic/non-aquatic boundary.

APAA-C18

Resource	APAA_C18 A	APAA_C18 B	APAA_C18 C	APAA_C18 D	APAA_C18 E	APAA_C18 F	APAA_C18 G	APAA_C18 H	APAA_C18 I	TotC18	APAA C18 E/H ratio
Manioc Flour	72	157	194	327	829	899	370	791	205	3844	1
Manioc Flour	111	220	234	440	1059	1183	464	392	293	4396	2.7
Beans	15127	40742	39204	39204	269759	487626	306118	320079	172371	1690230	0.8
Beans	27746	69341	72928	62121	446037	845430	547064	576987	318083	2965737	0.8

Beans	17484	42206	48095	48245	350821	518856	275732	271201	161862	1734502	1.3
Beans	10676	25445	26243	50554	197706	303078	158370	164192	95534	1031798	1.2
Indica Rice	4552	8716	11718	20836	85733	88912	21679	12962	15784	270892	6.6
Indica Rice	4687	9057	12121	21389	89651	93835	23179	13776	16945	284640	6.5

APAA C20

Resource	APAA C20 8	APAA C20 7	APAA C20 6	APAA C20 5	APAA C20 4	APAA C20 3	APAA C20 2	APAA C20 total	APAA C20/C18 ratio
Manioc Flour	12	79	12	12	12	84	95	306	0.08
Manioc Flour	1376	17	17	17	144	144	146	1861	0.42
Beans	75	75	128	162	149	1235	399	2223	0
Beans	112	123	116	179	154	224	293	1201	0
Beans	856	708	523	518	875	762	2407	6649	0

Beans	1542	1525	981	1006	1743	1175	3950	11922	0.01
Indica Rice	713	594	603	636	1047	728	3612	7933	0.03
Indica Rice	369	562	585	585	585	589	2119	5394	0.02

APPENDIX 4 - Organic residue analysis of ceramics from Patos Lagoon - CASA 2 - CHAPTER 6

Appendix 4 reports the sample-level organic residue analysis results for the Casa 2 ceramic assemblage discussed in Chapter 6. The principal interpreted dataset is based on acidified methanol extraction (AE), including lipid concentration, fatty acid distributions, APAA ratios, isoprenoid compounds and compound-specific isotope values where available. Six samples were also analysed by TLE/TMS as a complementary extraction strategy; however, the available TLE/TMS chromatograms were affected by laboratory hexane contamination. For this reason, TLE/TMS outputs are retained here for transparency as part of the analytical record, but no TLE/TMS-derived MAG, DAG, TAG, sterol or plant-wax assignments were used for source-specific interpretation.

Compounds marked with an asterisk **(*)** are reported as possible or tentative assignments only. They are retained for transparency but are not treated as diagnostic biomarkers unless supported by additional evidence, including lipid concentration, chromatographic profile, APAA/DB23 indicators, isoprenoids and/or compound-specific isotope values. - Targeted DB-23/SIM screening corresponds to the AQUASIM.m method used to monitor selected plant and aquatic-related compounds, including APAAs, TMTD, pristanic acid and phytanic acid. Where available, these outputs are reported in the relevant columns below.

To improve legibility, the Casa 2 organic residue dataset is divided into 3 complementary tables instead of being presented as a single wide table. Table **4A** reports sample information, lipid recovery, the main AE molecular results and the targeted DB-23/SIM compounds detected for the retained extracts. Table **4B** reports APAA and isoprenoid proxy data, CSIA results, TLE/TMS status. Together, the tables provide the full sample-level analytical record used to support Chapter 6. Table **4C** reports the six records from the initial Casa 2

ORA dataset that were not retained for AE-based molecular or isotopic interpretation because they produced low-yield or non-interpretable lipid signals during initial screening.

SFA: saturated fatty acids; **DC:** dicarboxylic acids; **UFA:** unsaturated fatty acids; **br:** branched fatty acids; **pris:** pristanic acid; **phy:** phytanic acid;

APPENDIX 4A: Sample information, lipid recovery, AE molecular summary and DB-23/SIM compounds detected

TRD_ID	Sample_ID	Site	Method	Fragment	Decoration	Thickness (mm)	Concentration (µg/g)	Yield class	AE molecular compounds/classes retained*	DB-23/SIM targeted compounds detected	P/S ratio	C12:0 %	C14:0 %
TRD_687	C2-5773	Casa 2	AE, TLE (tms)	body	smoothed	6.99	39.20	Moderate	SFA: C13-C24, C16/C18 dom.; UFA; DC*; BrFA*; phenol/phorbol-like*	phytanic acid; C18 APAAs; C20 APAAs	0.80	0.11	2.82
TRD_688	C2-427	Casa 2	AE	body	smoothed	8.50	12.21	Low >5	SFA: C12-C22, C16/C18 dom.; UFA; DC*; BrFA*; hydroxy/sterol-like*	phytanic acid	1.50	1.10	2.20

TRD_689	C2-1325	Casa2	AE	body	smoothed	6.10	30.95	Moderate	SFA: C12, C14-C22, C16/C18 dom.; UFA; DC*; BrFA*; phenol/phorbol-like*	phytanic acid	1.00	0.47	1.32
TRD_690	C2-798	Casa2	AE	body	smoothed	9.40	20.26	Moderate	SFA: C12-C22, C16/C18 dom.; UFA; DC*; BrFA*	phytanic acid	1.10	0.61	2.99
TRD_691	C2-869	Casa2	AE	body	smoothed	11.51	26.71	Moderate	SFA: C12-C24, C16/C18 dom.; UFA; DC*; BrFA*; hydroxy/sterol-like*	phytanic acid	1.20	4.23	7.81
TRD_692	C2-355	Casa2	AE	body	smoothed	9.40	24.71	Moderate	SFA: C12-C22, C16/C18 dom.; UFA; DC*; BrFA*	phytanic acid	1.20	2.21	8.77
TRD_693	C2-3065	Casa2	AE, TLE (tms)	rim	smoothed	8.80	79.07	High	SFA: C12-C20, C16/C18 dom.; UFA; DC*; BrFA*; phenol/phorbol-like*	phytanic acid; C18 APAAs	0.60	0.53	2.22

TRD_694	C2-473	Casa 2	AE, TLE (tms)	rim	smoothed (decoration on rim)	6.63	5.22	Low >5	SFA: C14-C20, C16/C18 present; UFA; DC*; BrFA*	phytanic acid	0.60	0.01	0.67
TRD_696	C2-499	Casa 2	AE, TLE (tms)	rim	smoothed	5.49	31.91	Moderate	SFA: C12, C14-C24, C16/C18 dom.; UFA; DC*; BrFA*; phenol/phorbol-like*	phytanic acid	1.00	0.41	1.94
TRD_697	C2-5666	Casa 2	AE	rim	smoothed	4.45	46.97	Moderate	SFA: C12-C24, C16/C18 dom.; UFA; DC*; BrFA*; hydroxy/sterol-like*; phenol/phorbol-like*	phytanic acid	0.90	1.21	2.60
TRD_698A-B	C2-6606	Casa 2	AE, TLE (tms)	body	smoothed	7.11	12.51	Low >5	SFA: C14-C24, C16/C18 present; UFA; DC*; BrFA*	phytanic acid	1.10	0.03	2.19
TRD_699A-C	C2-3661	Casa 2	AE	body	smoothed	6.23	24.62	Moderate	SFA: C14-C22, C16/C18 dom.; UFA; BrFA*; hydroxy/sterol-like*	phytanic acid	0.90	0.15	1.45

TRD_700	C2-485	Casa 2	AE, TLE (tms)	base	smoothed (dark paint)	5.77	175.40	High	SFA: C14-C20, C16/C18 dom.; UFA; BrFA*	phytanic acid	0.70	0.48	5.12
TRD_702A-B	C2-3453	Casa 2	AE	rim	smoothed/ slightly brushed	5.64	72.48	High	SFA: C12-C22, C16/C18 present; UFA; DC*; BrFA*; hydroxy/sterol-like*	phytanic acid; C18 APAAs; C20 APAAs	6.90	1.62	3.03

APPENDIX 4B: Targeted biomarkers, CSIA, TLE/TMS

TRD_ID	Sample_ID	Site	APAA C18 E/H DB23	APAA C20/C18 DB23	Proxy summary	SRR%	δ13C16:0 (‰)	δ13C18:0 (‰)	Δ13C (C18:0 -C16:0)	TLE/TMS
TRD_687	C2-5773	Casa 2	1.00	0.31	Aquatic-compatible proxy	55.7	-21.1	-22.7	-1.6	Performed;background-contaminated; transparency only; not used
TRD_688	C2-427	Casa 2			No APAA proxy	52.0	-29.7	-31.0	-1.3	Not analysed
TRD_689	C2-1325	Casa 2			No APAA proxy	56.5	-30.3	-31.1	-0.8	Not analysed
TRD_690	C2-798	Casa 2			No APAA proxy	52.6	-30.3	-30.9	-0.7	Not analysed
TRD_691	C2-869	Casa 2			No APAA proxy	48.9	-30.0	-30.5	-0.5	Not analysed
TRD_692	C2-355	Casa 2			No APAA proxy	45.6	-29.8	-29.8	0.0	Not analysed
TRD_693	C2-3065	Casa 2	4.20		Plant-processing proxy	47.8	-24.6	-26.8	-2.3	Performed;background-contaminated; transparency only; not used
TRD_694	C2-473	Casa 2			No APAA proxy	51.0	-29.2	-29.5	-0.3	Performed;background-contaminated; transparency only; not used

TRD_696	C2-499	Casa 2			No APAA proxy	54.4	-31.2	-31.0	0.2	Performed;background-contaminated; transparency only; not used
TRD_697	C2-5666	Casa 2			No APAA proxy	57.6	-31.3	-31.7	-0.4	Not analysed
TRD_698 A-B	C2-6606	Casa 2			No APAA proxy	43.4	-28.0	-28.0	0.0	Performed;background-contaminated; transparency only; not used
TRD_699 A-C	C2-3661	Casa 2			No APAA proxy	50.6	-31.5	-31.6	-0.1	Not analysed
TRD_700	C2-485	Casa 2			No APAA proxy	50.1	-31.4	-23.9	7.5	Performed;background-contaminated; transparency only; not used
TRD_702 A-B	C2-3453	Casa 2	1.16	0.17	Aquatic-compatible proxy	37.4	-31.2	-32.0	-0.7	Not analysed

APPENDIX 4C: Casa 2 records not retained for AE-based interpretation

Records from the initial CASA 2 ORA dataset that were not retained for AE-based molecular or isotopic interpretation. They are included here to document low-yield, non-interpretable or contamination-affected results as part of the analytical record.

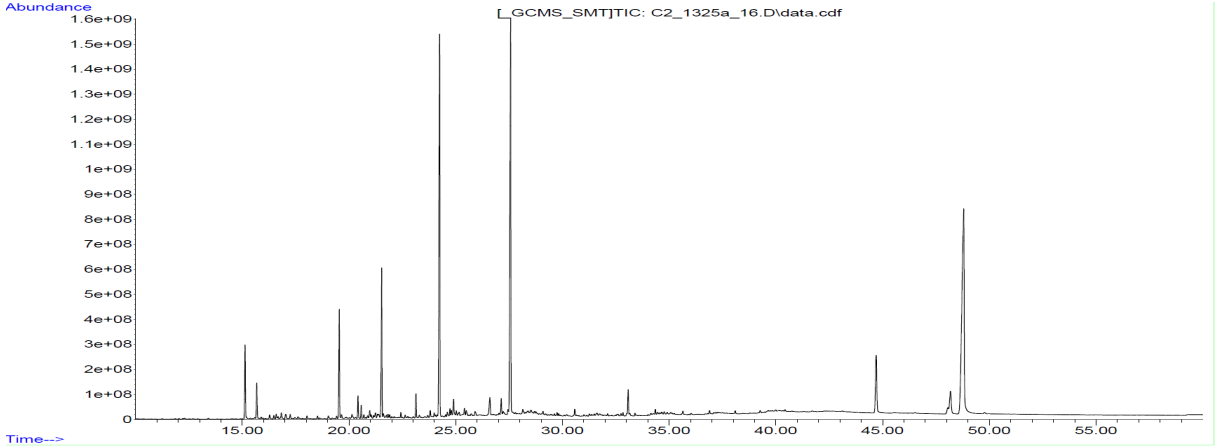
Initial record ID	Sample ID	Site	Final AE status	Reason / analytical note
TRD_692D	C2-6477	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.
TRD_692G	C2-1248	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.
TRD_696	C2-861	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.

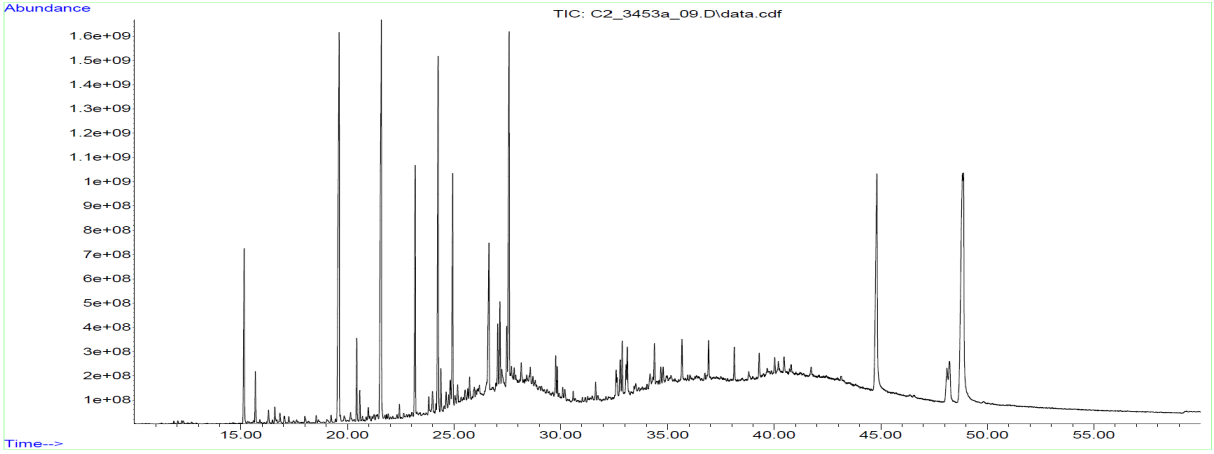
TRD_701A-C	C2-1387	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.
TRD_703A-C	C2-1268	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.
TRD_704A-C	C2-3460	Casa 2	Not retained	AE extract not retained for molecular or isotopic interpretation because it produced a low-yield or non-interpretable lipid signal during initial screening.

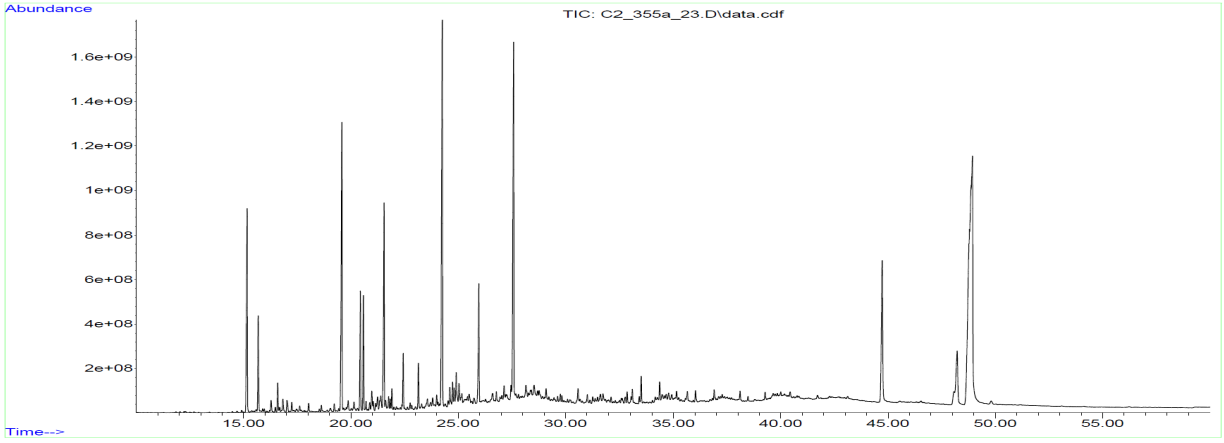
APPENDIX 5 - CASA 2 - AE chromatograms used for molecular interpretation and TLE/TMS chromatograms retained as analytical documentation

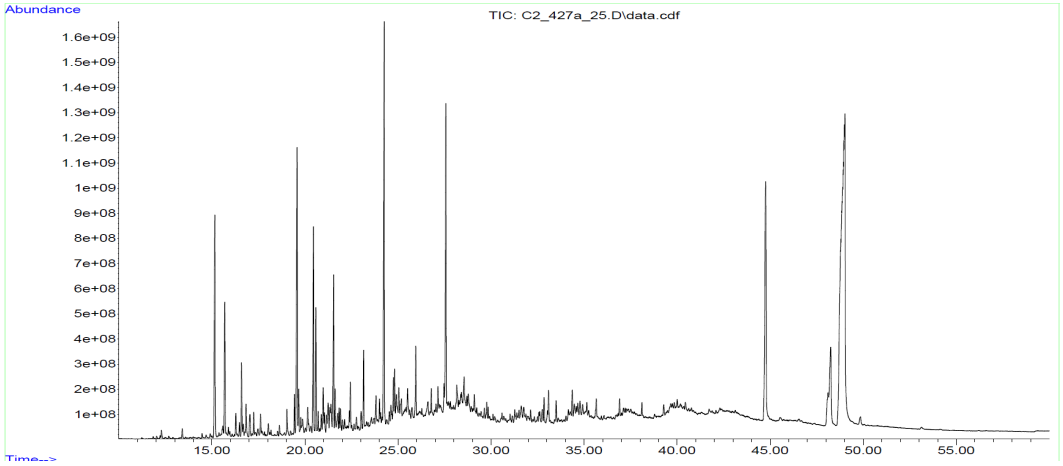
This appendix presents the available chromatographic outputs from the Casa 2 ceramic assemblage discussed in Chapter 6. Acidified methanol extraction (AE) chromatograms are presented first, as these form the principal dataset used for molecular interpretation. The available TLE/TMS chromatograms are presented separately as analytical documentation only. Because the TLE/TMS traces were affected by laboratory hexane contamination, they were not used for source-specific interpretation. No TLE/TMS-derived MAG, DAG, TAG, sterol or plant-wax assignments were used in the main interpretation of the Casa 2 assemblage.

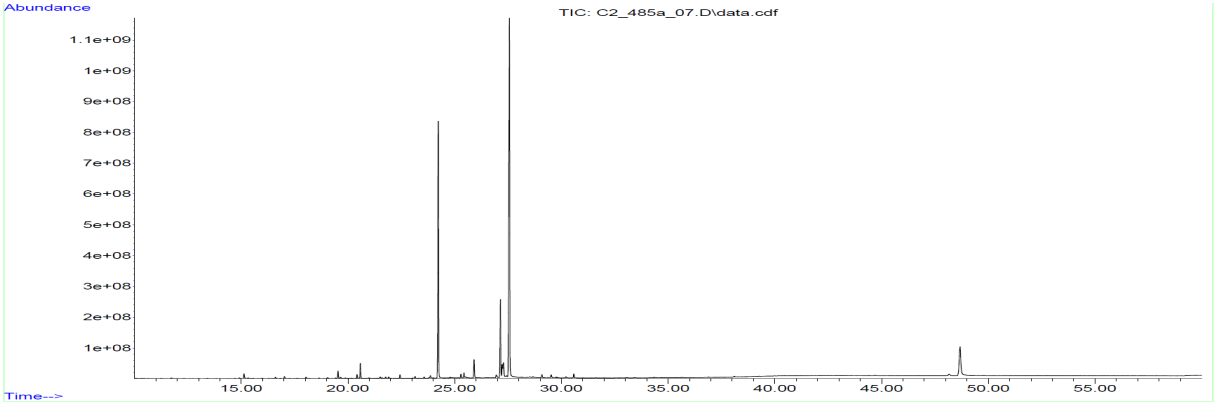
CASA 2 - Acid Extraction Chromatograms

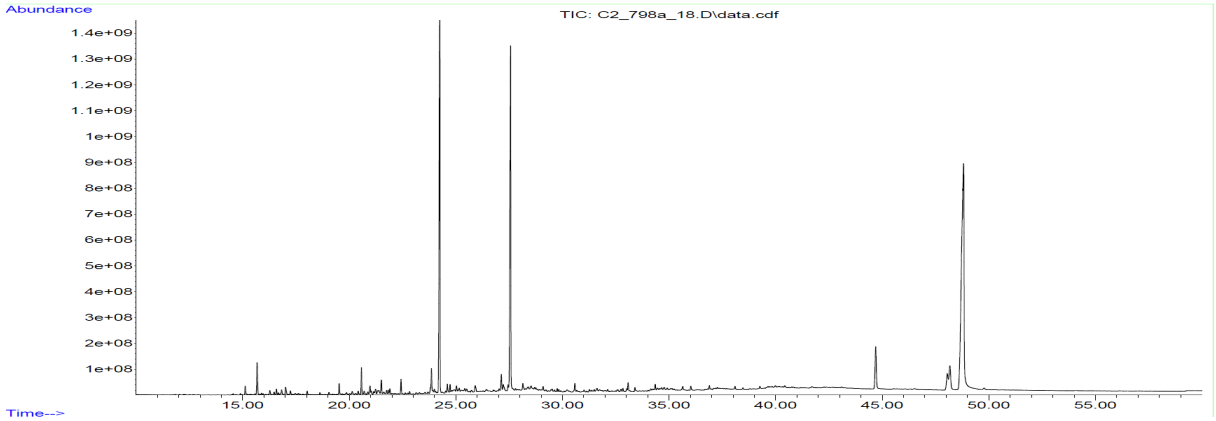


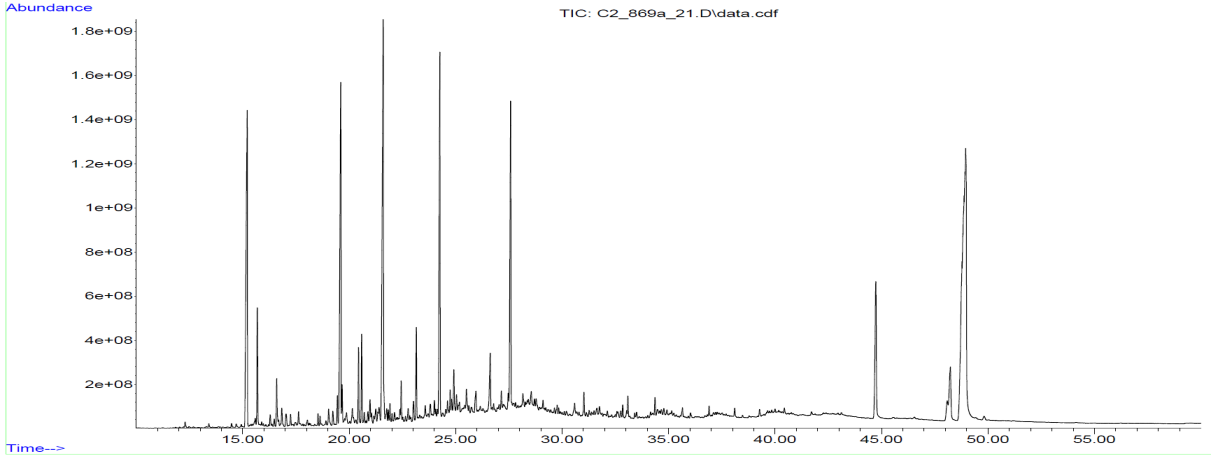

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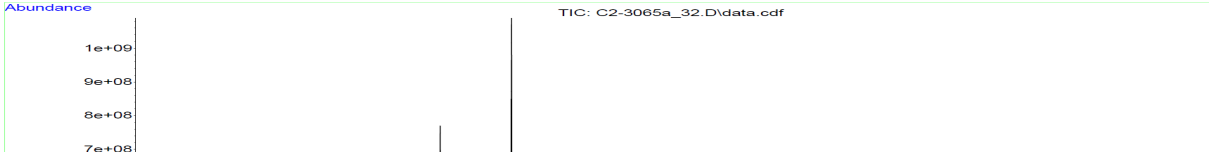

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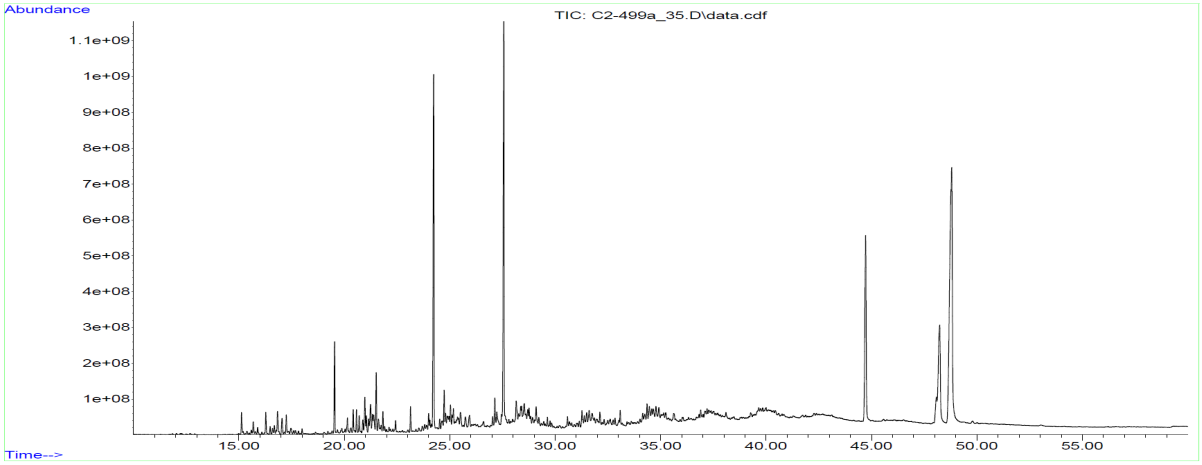

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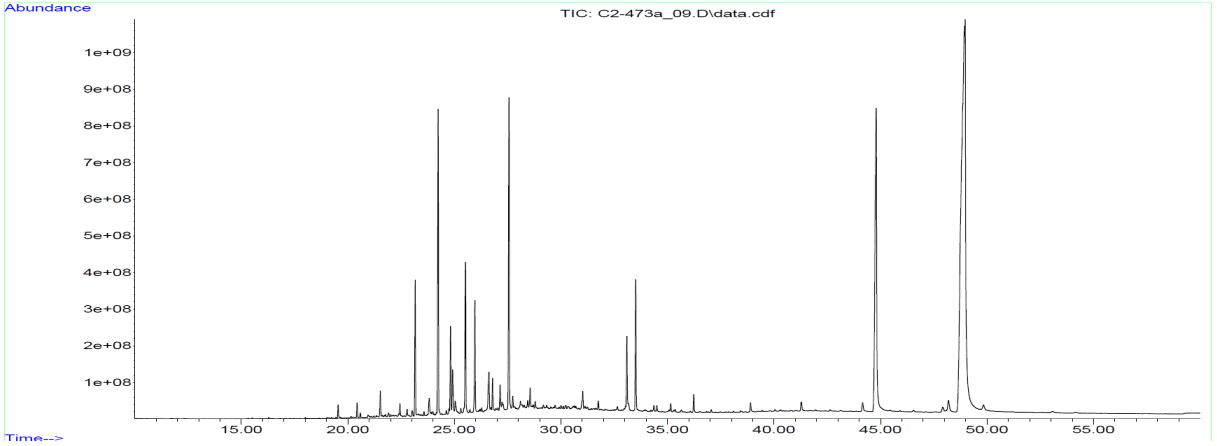

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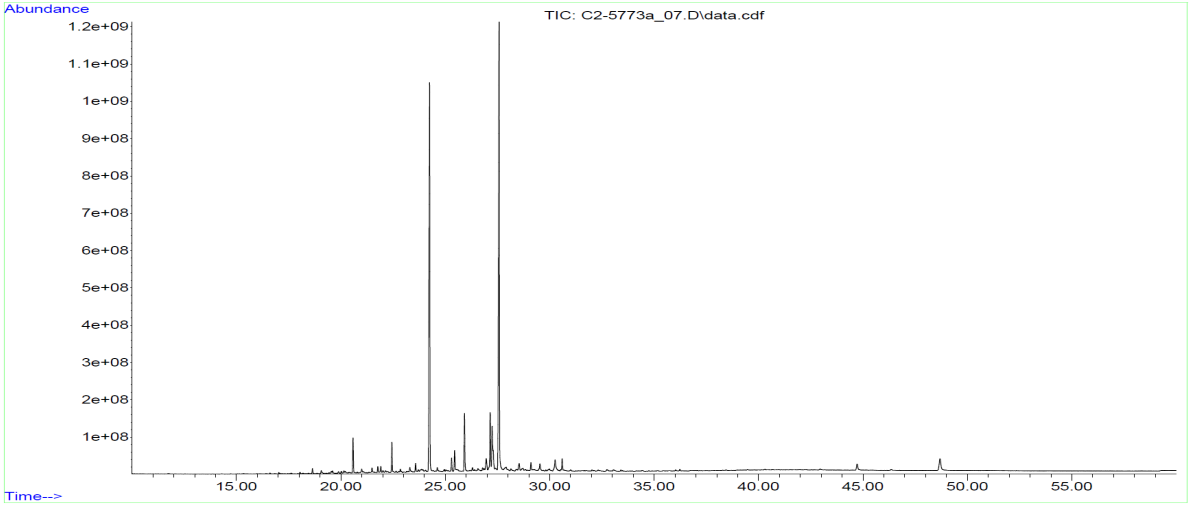

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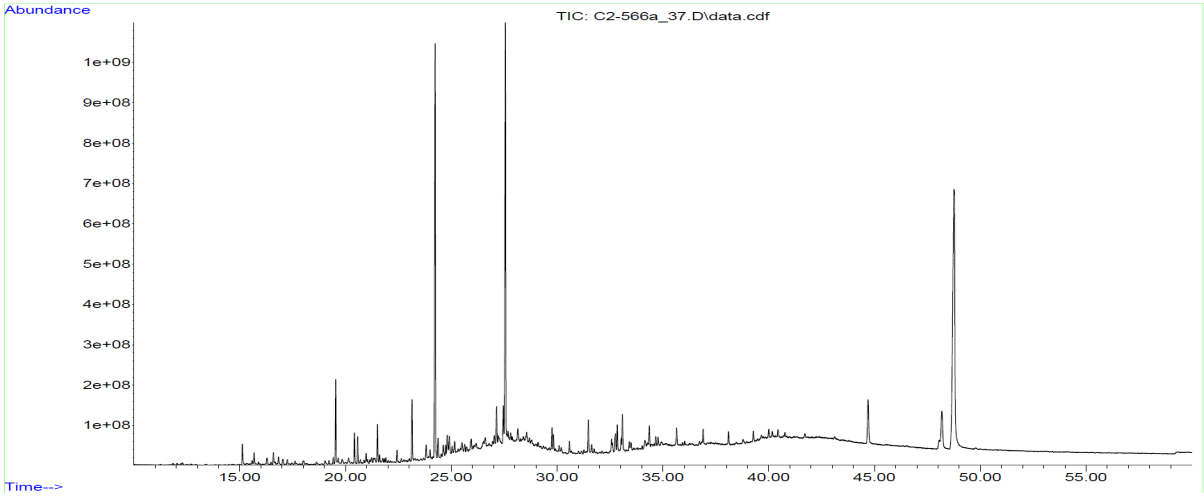

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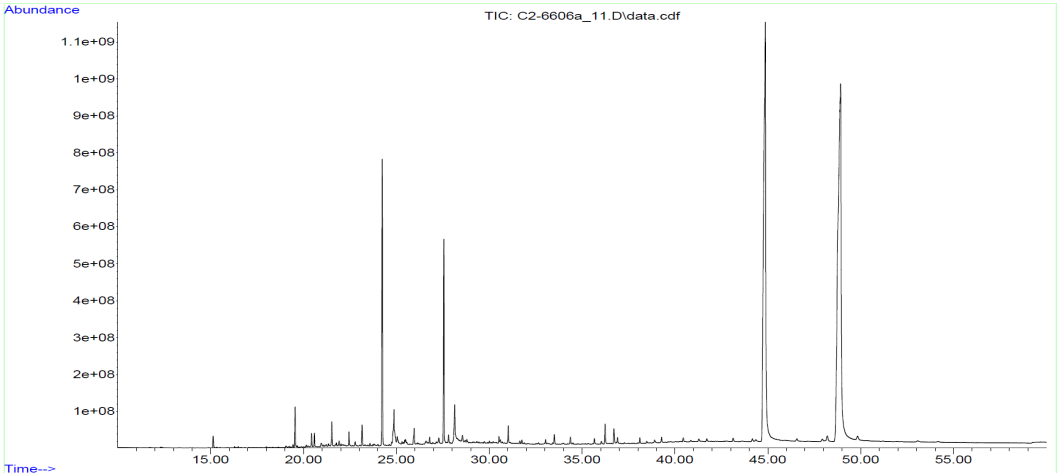

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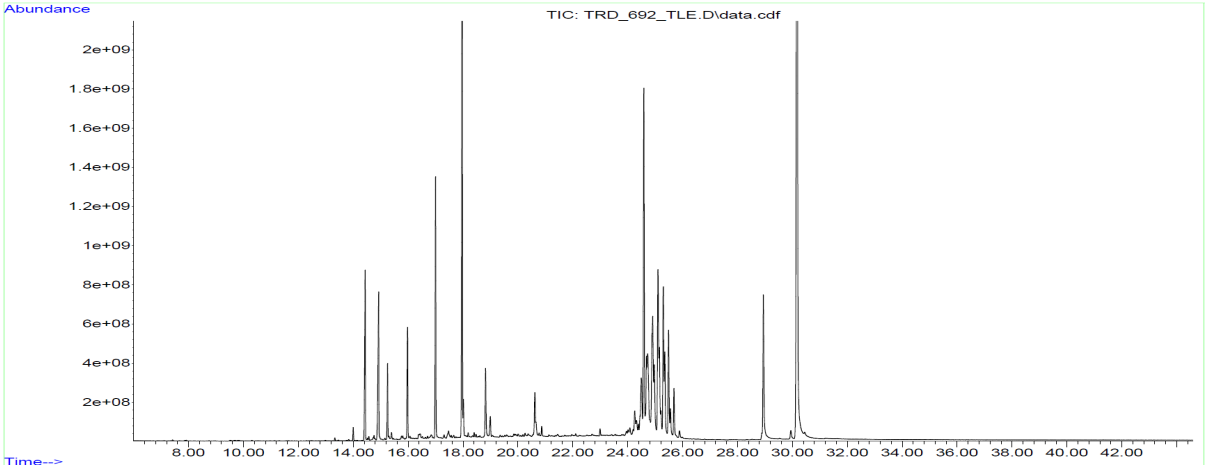

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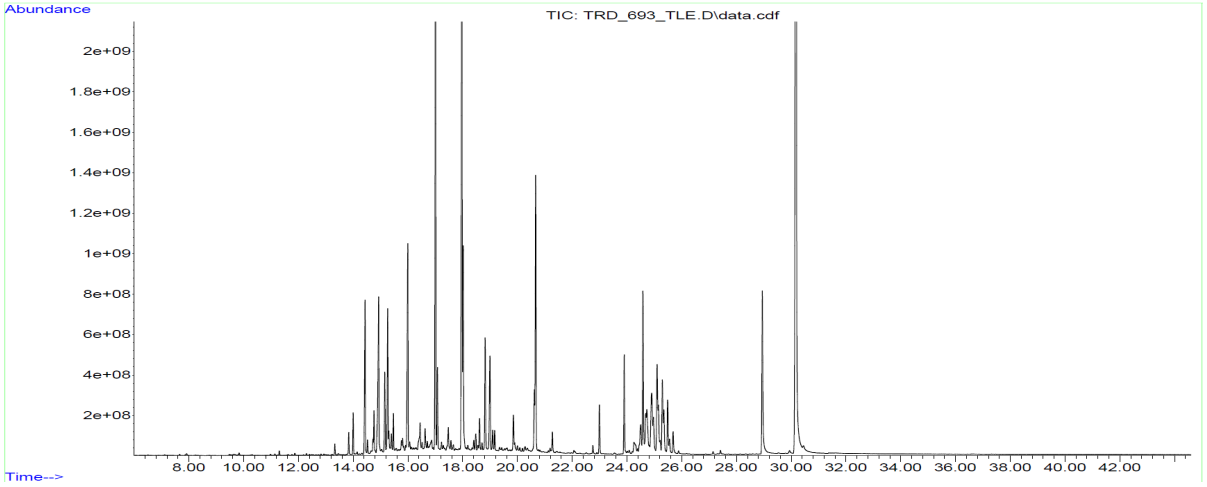

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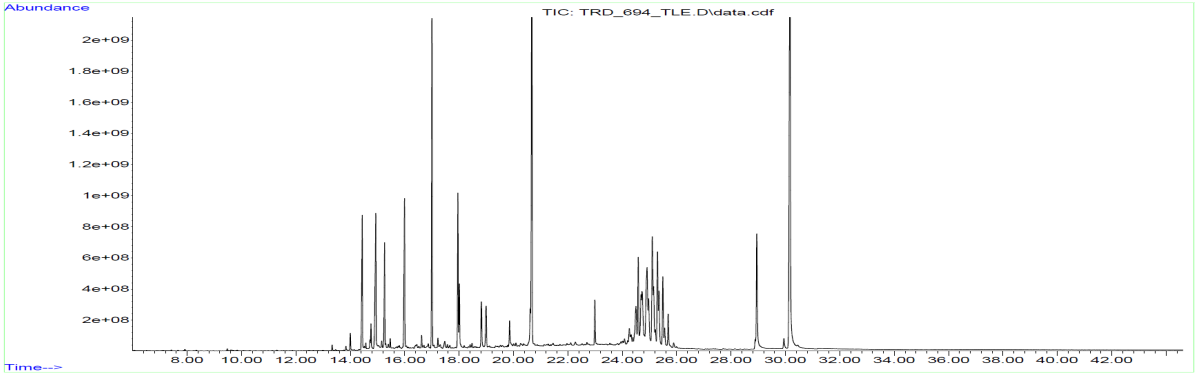
CASA 2 -TLE/TMS chromatograms retained as analytical documentation only

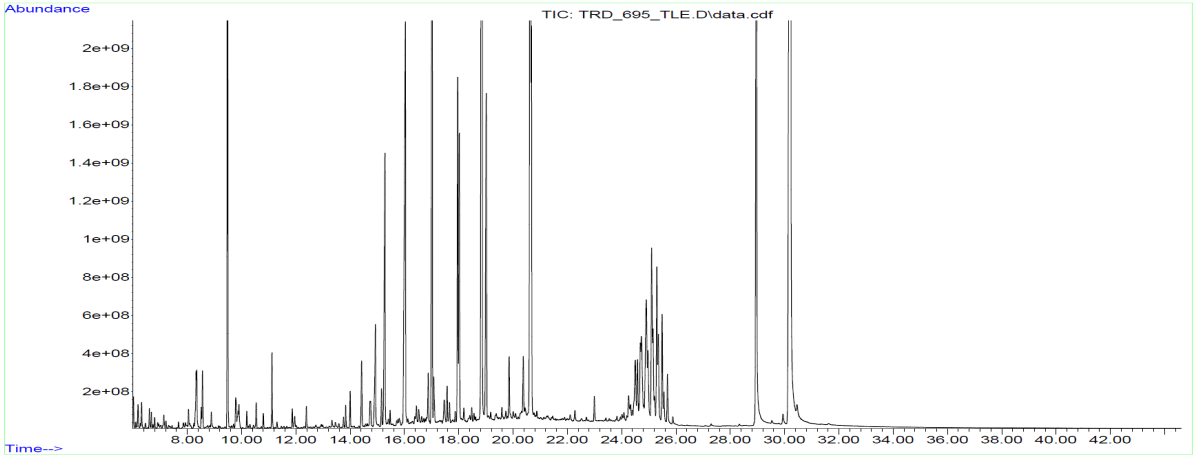
These chromatograms document the laboratory hexane contamination pattern observed in the TLE/TMS outputs. It is included to clarify why the Casa 2 TLE/TMS chromatograms were retained as analytical documentation but excluded from source-specific interpretation.

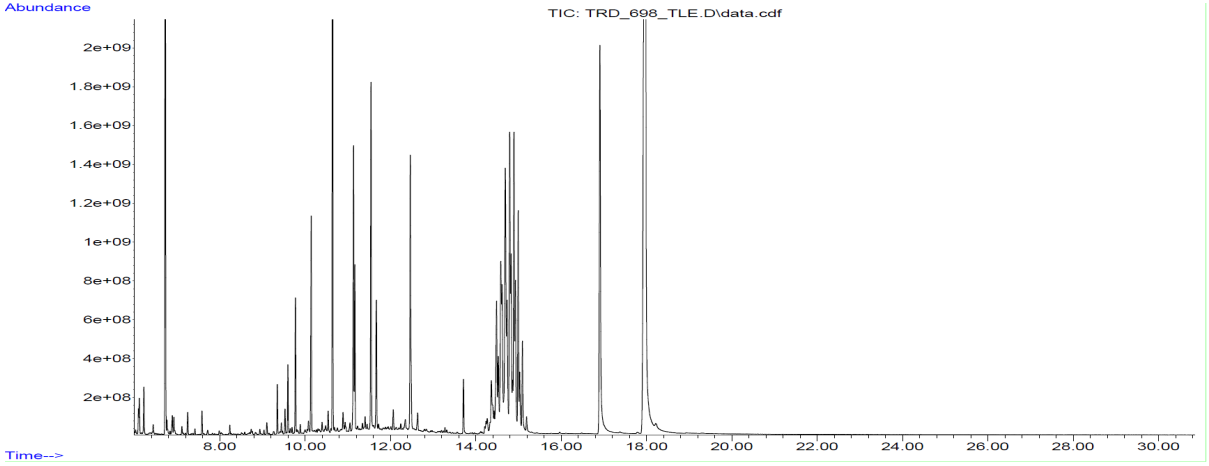


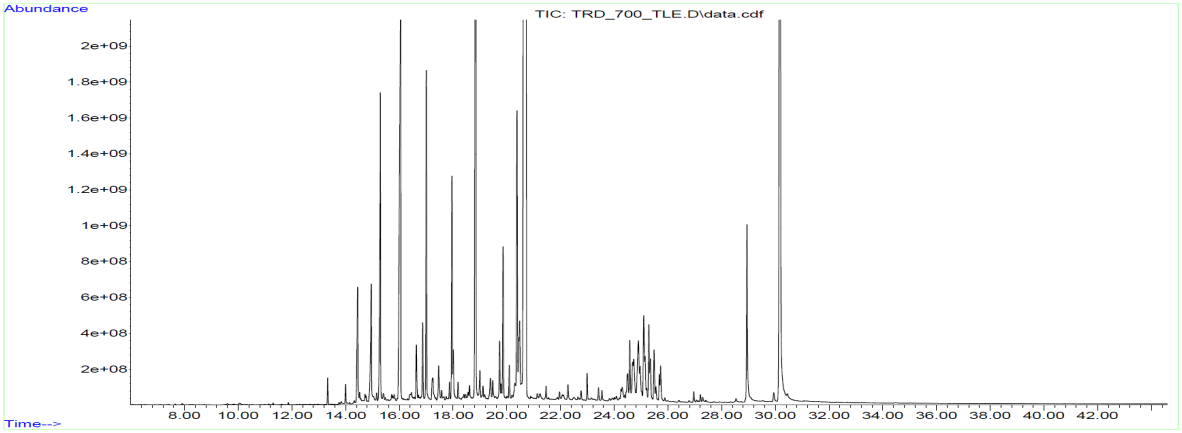
Laboratory hexane contamination


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4e+08
2e+08
Time-->
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Abundance
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2e+08
Time-->
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Abundance
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1.2e+09
1e+09
8e+08
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4e+08
2e+08
8.00 10.00 12.00 14.00 16.00 18.00 20.00 22.00 24.00 26.00 28.00 30.00 32.00 34.00 36.00 38.00 40.00 42.00
Time-->


Abundance
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1e+09
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6e+08
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2e+08
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Time-->


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Time-->

APPENDIX 6 - Stable isotope analysis of faunal and human collagen from Santa Catarina (Babitonga Bay) and Rio Grande do Sul (Patos Lagoon) - Chapter 7

Appendix 6 reports the faunal and human collagen stable isotope results used in Chapter 7. Taxonomic identifications are reported at the most specific level available for each specimen; where identification to species level was not possible, broader taxonomic categories are retained. Empty cells indicate that the information was not available in the original archaeological or laboratory records. Bone element information is included where recorded, because skeletal element selection can affect dietary interpretation through differences in tissue turnover and preservation.

Region	Site	Taxonomic class / group	Taxonomic identification	Common name / group	Environment	%C	%N	C:N	$\delta^{13}C$	$\delta^{15}N$	Reference
Santa Catarina	Unidade21	Mammal	*Dasypodidae*	Armadillo	Terrestrial	14.75	5.12	3.36	-21.9	6.79	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.86	14.84	3.29	-17.58	12.11	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.32	14.72	3.27	-14.71	9.28	This study

Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.65	15.02	3.23	-15.32	9.25	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	40.7	14	3.39	-16.11	11.28	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.22	15.24	3.15	-18.86	10.03	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	40.51	14.05	3.36	-15.93	9.29	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	39.86	14.22	3.27	-16.06	9.3	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.96	15.03	3.26	-15.1	10.25	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	41.11	14.89	3.22	-19.29	10.83	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	42.93	14.67	3.41	-19.51	11.44	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	42.1	14	3.5	-19.1	11.2	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	43.7	15.21	3.35	-19.24	9.7	This study
Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	43.96	14.54	3.53	-19.99	10.98	This study

Santa Catarina	Unidade21	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	44.1	14.76	3.48	-19.09	8.72	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	41.32	14.59	3.3	-23.54	6.25	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	34.13	12	3.32	-9.69	4.76	This study
Santa Catarina	Morro Grande 1	Mammal	*Probable Bovidae*	Cattle	Terrestrial	25.24	8.88	3.31	-11.3	4.95	This study
Santa Catarina	Morro Grande 1	Mammal	*Suinae (Tayassuidae)*	Wild pig	Terrestrial	40.08	13.61	3.43	-23.65	1.32	This study
Santa Catarina	Morro Grande 1	Mammal	*Suinae (Tayassuidae)*	Wild pig	Terrestrial	37.4	13.28	3.28	-22.65	1.43	This study
Santa Catarina	Morro Grande 1	Mammal	*Suinae (Tayassuidae)*	Wild pig	Terrestrial	39.7	13.9	3.3	-22.7	1.7	This study
Santa Catarina	Morro Grande 1	Mammal	*Suinae*	Domestic pig	Terrestrial	41.5	14.7	3.3	-18.3	11.3	This study
Santa Catarina	Morro Grande 1	Mammal	*Suinae (Tayassuidae)*	Wild pig	Terrestrial	41.6	14.3	3.4	-22.7	1.7	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	41.5	14.7	3.3	-22.7	5.5	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	38.1	12.9	3.4	-23.2	5.1	This study

Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	42.7	14.1	3.5	-24.7	5.2	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	43.4	15.3	3.3	-23	5.4	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	43.2	15.5	3.2	-23.4	5.5	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	39.8	13.3	3.5	-23.6	5.1	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	32.75	11.55	3.3	-23.6	5.32	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	42.9	14.6	3.4	-23.6	5.1	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	42.3	14.4	3.4	-23.3	6.2	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	43.7	15.1	3.4	-23.5	5.6	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	40.2	13.9	3.4	-9.3	4.9	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	41.21	14.29	3.36	-9.29	5.26	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	41.9	13.5	3.6	-15	6.4	This study

Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	40.1	13.7	3.4	-10.5	6	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	36.6	12.4	3.5	-12	4.9	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	41.2	14.1	3.4	-15.3	6.5	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	43.03	13.95	3.6	-13.31	5.67	This study
Santa Catarina	Morro Grande 1	Mammal	*Cervidae*	Deer	Terrestrial	42.6	14.8	3.4	-23.2	5.7	This study
Santa Catarina	Morro Grande 1	Mammal	*Bovidae*	Cattle	Terrestrial	43.7	13.8	3.7	-16.8	6.7	This study
Santa Catarina	Morro Grande 1	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	43.05	14.51	3.47	-23.45	7.2	This study
Santa Catarina	Morro Grande 1	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	43.8	15.1	3.4	-22.9	6.4	This study
Santa Catarina	Morro Grande 1	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	42	14.8	3.3	-21.1	5.6	This study
Santa Catarina	Morro Grande 1	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	43.3	14.8	3.4	-20.5	5.6	This study
Santa Catarina	Morro Grande 1	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	42.5	15	3.3	-21.2	6.2	This study

Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	37.7	12.7	3.5	-12.6	12.1	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	40.4	13.7	3.4	-11.2	12.9	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	40.3	14.4	3.3	-11	11.9	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	41.7	14.5	3.4	-11.6	12.9	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	40.13	14.39	3.23	-11.29	11.84	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	40.4	14.1	3.3	-13	11.9	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	37.1	13	3.3	-11.2	11.9	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	39.1	13.5	3.4	-11.5	13.3	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	41.15	14.08	3.43	-11.91	13.19	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	37.5	12.9	3.4	-11.6	13.1	This study
Santa Catarina	Morro Grande 1	Fish	*Pogonias courbina*	Marine fish	Marine/Brackish	40.3	14.1	3.3	-11.6	12.45	This study

Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	15.85	5.45	3.39	-13.24	6.27	This study
Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	38.61	13.94	3.23	-10.29	6.02	This study
Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	40.69	14.59	3.25	-11.53	5.93	This study
Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	18.49	6.64	3.25	-11.73	5.56	This study
Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	42.15	14.81	3.32	-11.2	6.54	This study
Santa Catarina	Unidade21	Mammal	*Bovidae*	Cattle	Terrestrial	42.67	15.54	3.2	-13.97	5.82	This study
Santa Catarina	Unidade21	Mammal	*Suinae*	Domestic pig	Terrestrial	39.62	13.78	3.35	-19.1	8.09	This study
Santa Catarina	Unidade21	Mammal	*Dasypodidae*	Armadillo	Terrestrial	41.69	14.76	3.29	-20.44	7.04	This study
Santa Catarina	Unidade21	Mammal	*Dasypodidae*	Armadillo	Terrestrial	41	14.51	3.3	-19.46	9.34	This study
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.16	14.64	3.2	-10.6	15	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	41.28	14.59	3.3	-12.45	12.28	Fossile et al., 2024

Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	41.94	15.4	3.18	-10.73	14.54	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.9	14.92	3.2	-10.75	15.26	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.55	15.08	3.13	-10.69	14.62	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.56	14.12	3.35	-11.72	14.27	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.69	14.66	3.24	-11.23	15.05	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	41.2	15.04	3.19	-10.59	14.42	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	39.88	14.49	3.21	-11.96	13.24	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	41.3	14.5	3.3	-10.7	13.4	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	39	13.1	3.5	-12.1	14.1	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	42.4	15.2	3.3	-12.4	12.6	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	39	14.4	3.2	-12.8	12.5	Fossile et al., 2024

Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	39.13	14.09	3.24	-12.24	13.05	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.33	14.64	3.21	-13.11	12.2	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.2	14.36	3.26	-11.68	13.93	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Micropogonias furnieri*	Marine fish	Marine/Brackish	40.02	14.39	3.24	-13.03	12.07	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	39.5	14.6	3.2	-12.2	12.7	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	36.58	13.74	3.11	-11.09	12.4	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	35.82	12.33	3.39	-11.69	12.8	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	38.69	13.95	3.23	-12.71	12.54	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.61	14.77	3.29	-11.19	12.87	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	40.19	14.33	3.27	-11.06	13.37	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.81	13.63	3.58	-11.49	12.87	Fossile et al., 2024

Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	38.84	13.94	3.25	-12.2	12.61	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	42.17	15.1	3.26	-10.39	12.92	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	40.36	14.04	3.35	-11.19	13.29	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.51	14.56	3.32	-11.88	12.99	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.08	14.7	3.26	-11.09	13	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.23	14.64	3.28	-11.75	12.97	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	40.47	14.76	3.2	-11.43	12.28	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.76	14.3	3.41	-12.83	12.85	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	36.51	13.62	3.13	-11.45	12.42	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	39.93	15.06	3.09	-10.82	13.02	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.19	14.53	3.31	-10.68	11.94	Fossile et al., 2024

Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	41.3	14.79	3.26	-12.09	12.67	Fossile et al., 2024
Santa Catarina	Unidade21	Fish	*Conodon nobilis*	Marine fish	Marine/Brackish	38.32	14.09	3.17	-11.61	12.49	Fossile et al., 2024
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	39,9	14,2	3,3	-15.5	15.6	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	42,7	14,8	3,4	-13.88	13.98	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	42,2	14,6	3,4	-17.4	13.7	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	39,8	14,1	3,3	-14.26	13.52	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	41,1	14,8	3,2	-12.1	13.1	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	40,2	14,6	3,2	-13.9	13.2	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	43,9	15,7	3,3	-13	13.4	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	42,5	13,8	3,6	-14.2	12.6	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	41	13,73	3,48	-13.1	12.6	This study

Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	42,5	13,9	3,6	-15.07	8.33	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	41,62	14,04	3,46	-16	11.2	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	41,6	13,6	3,6	-13.1	13.9	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	41,3	13,7	3,5	-17.8	10.1	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	40,5	13,6	3,5	-13	12.8	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	36,2	12,8	3,3	-16.6	8.6	This study
Rio Grande do Sul	Igreja da Matriz	Human	*Homo sapiens*	Human	Terrestrial	39,8	13,4	3,5	-14.61	10.76	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	42,51	14,41	3,44	-15.8	12.9	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	42,9	14,2	3,5	-16.7	15.7	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	41,6	13,6	3,6	-16.8	14.4	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	42,5	14,5	3,4	-16.2	14.8	This study

Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	42,3	14,3	3,5	-16.1	14.5	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	42,4	15	3,3	-14.6	14.4	This study
Santa Catarina	Igreja Matriz SFS	Human	*Homo sapiens*	Human	Terrestrial	41,13	13,96	3,44	-16.5	13.8	This study
Rio Grande do Sul	Casa 8	Fish	*Pogonias courbina*	Black drum	Terrestrial	10.6	0.34	3,2	-11.3	11.2	This study
Rio Grande do Sul	Casa 8	Fish	*Pogonias courbina*	Black drum	Terrestrial	11.2	0.39	3,3	-13.5	10.9	This study
Rio Grande do Sul	Casa 8	Mammal	*Bos taurus*	Cattle	Terrestrial	11.8	0.2	3,2	-14.2	6	This study
Rio Grande do Sul	Casa 8	Mammal	*Bos taurus*	Cattle	Terrestrial	12.1	0.19	3,3	-12.6	8.2	This study
Rio Grande do Sul	Casa 8	Mammal	*Bos taurus*	Cattle	Terrestrial	14.35	0.22	3,35	-8.6	4.5	This study
Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	42.95	14.98	3.3	-10.6	6.2	This study
Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	43.7	15.4	3.3	-9.1	5.8	This study
Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	43.3	15.3	3.3	-10.5	8	This study

Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	42.05	14.12	3,4	-10.5	6.1	This study
Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	41	14.2	3,4	-11.3	6	This study
Rio Grande do Sul	Igreja da Matriz	Mammal	*Bos taurus*	Cattle	Terrestrial	42	14.2	3.4	-10.1	5.4	This study
Rio Grande do Sul	Casa 8	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	14.4	0.28	3,3	-8.3	7.9	This study
Rio Grande do Sul	Casa 8	Aves	*Gallus gallus domesticus*	Chicken	Terrestrial	14.9	0.28	3,3	-9.2	7.2	This study
Rio Grande do Sul	Casa 8	Mammal	*Sus scrofa*	Domestic pig	Terrestrial	33.20	12.25	3.2	-10.5	12.6	This study
Rio Grande do Sul	Casa 8	Mammal	*Didelphis sp.*	Opossum	Terrestrial	42.6	15.4	3.2	-10.6	10.5	This study
Rio Grande do Sul	Casa 8	Mammal	*Canis Lupus*	Dog	Terrestrial	42.1	15.0	3.3	-14.7	10.2	This study

APPENDIX 7 - Modern references for CSIA of fatty acids

Category	Common name	*Taxa*	Sample type	Location	$\Delta^{13}C_{(18:0-16:0)}$	$\delta^{13}C_{16:0}$	$\delta^{13}C_{18:0}$	Reference	DOI
C3 plant	Algarrobo	*Prosopis alba Girseb.*	Pods	Argentina	1.1	-34.00	-32.90	Lantos et al. 2015 JAS	https://doi.org/10.1016/j.jas.2014.12.022
C3 plant	Alubia bean	*Phaseolus vulgaris L.*	Seeds	Argentina	1.2	-35.00	-33.80	Lantos et al. 2015 JAS	https://doi.org/10.1016/j.jas.2014.12.022
C3 plant	Acorn	*Quercus sp.*	Tissue	Japan	-0.4	-34.00	-34.40	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
C3 plant	Acorn	*Quercus sp.*	Tissue	Japan	-0.9	-32.70	-33.60	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
C3 plant	Acorn	*Quercus sp.*	Tissue	Japan	-0.8	-35.10	-35.90	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
C3 plant	Acorn	*Quercus sp.*	Tissue	Japan	-1.5	-33.10	-34.60	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
C3 plant	Acorn	*Quercus sp.*	Tissue	Japan	-1.9	-32.10	-34.00	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
C3 plant	Broadbean	*Vicia faba*	bean	Argentina	1.0	-32.40	-31.40	March et al., 2013	https://doi.org/10.1515/9781789

									201574-020
C3 plant	Broadbean	*Vicia faba*	bean	Argentina	1.3	-32.60	-31.30	March et al., 2013	https://doi.org/10.1515/9781789201574-020
Ruminant	Cattle	*Bos taurus*	Soft tissue	Switzerland	-1.5	-27.10	-28.60	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Ruminant	Cattle	*Bos taurus*	Soft tissue	Switzerland	-0.5	-27.80	-28.30	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Ruminant	Cattle	*Bos taurus*	Soft tissue	Switzerland	0.1	-31.40	-31.30	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Ruminant	Cattle	*Bos taurus*	Soft tissue	Switzerland	-0.2	-30.00	-30.20	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Wild ruminant	Deer	*Cervus sp.*	Skin	Switzerland	-0.9	-24.00	-24.90	Spangenberg et al. 2010	https://doi.org/10.1016/j.jas.2010.02.003
Wild ruminant	Hokkaido deer	*Cervus sp.*	Experimental sherd	Japan	0.0	-28.10	-28.10	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Wild ruminant	Hokkaido deer	*Cervus sp.*	Experimental sherd	Japan	-2.3	-30.40	-32.70	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Ruminant	Llama	*Lama glama L.*	Fresh fat	Argentina	-3.0	-23.70	-26.70	Lantos et al. 2015 JAS	https://doi.org/10.1016/j.jas.2014.12.022

Ruminant	Llama	*Lama glama L.*	Jerkey fat	Argentina	-3.0	-27.50	-30.50	Lantos et al. 2015 JAS	https://doi.org/10.1016/j.jas.2014.12.022
C3 plant	Manioc	*Manihot esculenta*	root	? (buy in France)	1.7	-37.60	-35.90	March et al., 2013	https://doi.org/10.1515/9781789201574-020
C3 plant	Oat	*Avena sativa*	grain	France	0.5	-34.20	-33.70	March et al., 2013	https://doi.org/10.1515/9781789201574-020
C3 plant	Oat	*Avena sativa*	grain	France	-0.2	-33.70	-33.90	March et al., 2013	https://doi.org/10.1515/9781789201574-020
C3 plant	Peanut	*Arachis hypogaea*	dry oilseeds	China	-0.7	-26.40	-27.10	Guo et al. 2010	DOI:10.1007/s11746-010-1564-3
C3 plant	Pine nut	*Pinus pinea*	dry oilseeds	China	-1.3	-24.90	-26.20	Guo et al. 2010	DOI:10.1007/s11746-010-1564-3
C3 plant	Potato	*Solanum tuberosum*	dryed tuber	Argentina	-0.2	-34.20	-34.40	March et al., 2013	https://doi.org/10.1515/9781789201574-020
C3 plant	Pumpkin	*Cucurbita*	dry oilseeds	China	-0.7	-26.80	-27.50	Guo et al. 2010	DOI:10.1007/s11746-010-1564-3
C3 plant	Pumpkin	*Cucurbita*	dry oilseeds	China	-0.4	-27.10	-27.50	Guo et al. 2010	DOI:10.1007/s11746-

									010-1564-3
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-1.4	-28.30	-29.70	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-1.7	-28.80	-30.50	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-0.3	-29.40	-29.70	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-3.3	-28.80	-32.10	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf

Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-5.2	-28.00	-33.20	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-3.1	-30.70	-33.80	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Adipose	UK	-2.8	-30.80	-33.60	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Wild ruminant	Red deer	*Cervus elaphus*	Tissue	Switzerland	-2.1	-31.00	-33.10	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Wild ruminant	Red deer	*Cervus elaphus*	Tissue	UK	-1.7	-31.50	-33.20	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Wild ruminant	Red deer	*Cervus elaphus*	Experimental sherd	UK	-2.2	-31.40	-33.60	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113

Wild ruminant	Red deer	*Cervus elaphus*	tissue	France, Provence	-4.8	-28.30	-33.10	Drieu et al., 2021a	https://doi.org/10.1016/j.jasrep.2020.102682
Wild ruminant	Red deer adipose	*Cervus elaphus*		Germany	-1.2	-24.90	-26.10	Spiteri 2012	https://etheses.whiterose.ac.uk/id/eprint/3391/
Wild ruminant	Roe deer adipose	*Capreolus capreolus*		Germany	-6.0	-24.50	-30.50	Spiteri 2012	https://etheses.whiterose.ac.uk/id/eprint/3391/
Wild ruminant	Sika deer	*Cervus nippon*	Experimental sherd	Japan	-1.6	-27.80	-29.40	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Wild ruminant	Sika deer	*Cervus nippon*	Experimental sherd	Japan	-3.5	-28.10	-31.60	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Wild ruminant	Sika deer (Nagano)	*Cervus nippon*	Tissue	Japan	-3.2	-31.90	-35.10	Lucquin et al. 2016	https://doi.org/10.1073/pnas.1522908113
Maize	Maize	*Zea mays L.*	Kernel	Chile	-0.3	-17.20	-17.50	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Maize	Maize	*Zea mays L.*	Kernel	Chile	0.6	-17.60	-17.00	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Maize	Maize	*Zea mays L.*	Kernel	Chile	-0.4	-17.40	-17.80	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Maize	Maize	*Zea mays L.*	Kernel	Chile	-0.5	-17.20	-17.70	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58

Maize	Maize	*Zea mays L.*	Kernel	Chile	0.2	-16.60	-16.40	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Maize	Maize	*Zea mays L.*	Kernel	Amazon, Brazil	-0.2	-17.40	-17.60	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Maize	Maize	*Zea mays L.*	Kernel	Amazon, Brazil	0.2	-16.10	-15.90	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Maize	Maize	*Zea mays L.*	Kernel	Amazon, Brazil	0.7	-16.50	-15.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Maize	Maize	*Zea mays L.*	Kernel	Amazon, Brazil	0.5	-15.20	-14.70	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Maize	Maize	*Zea mays L.*	Kernel	Amazon, Brazil	-1.2	-16.00	-17.20	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Maize	Maize	*Zea mays L.*	Kernel	Chile	-0.7	-18.10	-18.80	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
C3 plant	Quinoa	*Chenopodium quinoa*	Seed	Chile Araucanía Region	0.9	-31.30	-30.40	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
C3 plant	Chilean hazelnut	*Gevuina avellana*	Seed	Chile	1.3	-26.90	-25.60	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Ruminant	Llama	*Lama glama L.*	Fresh fat	Argentina	-3.0	-23.70	-26.70	Lantos et al. 2015 JAS	https://doi.org/10.101

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Ruminant	Llama	*Lama glama L.*	Jerkey fat	Argentina	-3.0	-27.50	-30.50	Lantos et al. 2015 JAS	https://doi.org/10.1016/j.jas.2014.12.022
Wild non-ruminant	Armadillo	Didelphis sp	Bone	Amazon, Brazil	0.9	-23.20	-22.32	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Porcine	Pig	*Sus scrofa*	Bone	Amazon, Brazil	-0.4	-23.95	-24.38	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Porcine	Pig	*Sus scrofa*	Bone	Amazon, Brazil	1.4	-22.27	-20.88	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
C3 plant	Madi	*Madia sativa*	Seed	Chile	-0.6	-31.50	-32.10	Cabrera-Montalvo et al., 2024	doi:10.1017/laq.2023.58
Marine	Almeja blanca rayada	*Eurhomalea exalbida*	Bone	South America	-0.5	-20.20	-20.70	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Almeja blanca rayada	*Eurhomalea exalbida*	Bone	South America	-0.7	-20.70	-21.40	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Almeja blanca rayada	*Eurhomalea exalbida*	Bone	South America	0.0	-20.20	-20.20	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5

Marine	Argentine hake	*Merluccious hubbsi*	Bone	South America	0.9	-23.40	-22.50	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Argentine hake	*Merluccious hubbsi*	Bone	South America	0.7	-23.50	-22.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Cod	*Merluccius Merluccius*		UK	-2.5	-22.30	-24.80	Craig et al. 2011	https://doi.org/10.1073/pnas.1107202108
Marine	Cod	*Merluccius Merluccius*		UK	-0.4	-21.60	-22.00	Craig et al. 2011	https://doi.org/10.1073/pnas.1107202108
Marine	European eel	*Anguilla anguilla*		Japan	0.4	-24.80	-24.40	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	Cod	*Merluccius Merluccius*		UK	2.4	-23.50	-21.10	Craig et al. 2011	https://doi.org/10.1073/pnas.1107202108
Marine	Pilot Whale	*Globicephala macrorhynchus*		UK	-0.2	-25.40	-25.60	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	Black drum	*Pogonias cromis*	Bone	UK	-0.6	-24.10	-24.70	Colonese et al. 2015	https://doi.org/10.1002/rcm.7144
Marine	Black drum	*Pogonias cromis*	Bone	South America	1.5	-24.70	-23.20	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5

Marine	Black drum	*Pogonias cromis*	Bone	South America	2.5	-25.50	-23.00	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Blue mussel	*Mytilus edulis*	Bone	South America	1.4	-22.00	-20.60	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Blue mussel	*Mytilus edulis*	Bone	South America	0.8	-21.50	-20.70	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Blue mussel	*Mytilus edulis*	Bone	South America	1.6	-22.20	-20.60	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Catfish	*Diplomystes viedmensis*	Bone	South America	-0.6	-19.50	-20.10	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Catfish	*Diplomystes viedmensis*	Bone	South America	-0.5	-19.30	-19.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Cetacea	*Cetacea*	Bonw	UK	-1.7	-24.20	-25.90	Colonese et al. 2015	https://doi.org/10.1002/rcm.7144
Marine	Seashell	*Babylonia sp.*		UK	2.7	-18.50	-15.80	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011

Marine				UK	0.6	-21.90	-21.30	Craig et al. 2013	DOI: 10.1038/nature12109
Marine				UK	-1.6	-18.90	-20.50	Craig et al. 2011	https://doi.org/10.1073/pnas.1107202108
Marine				UK	0.3	-22.40	-22.10	Craig et al. 2013	DOI: 10.1038/nature12109
Marine				UK	0.2	-22.70	-22.50	Craig et al. 2013	DOI: 10.1038/nature12109
Marine	Limpet	*Nacella magellanica*	Bone	South America	0.7	-23.40	-22.70	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Limpet	*Nacella magellanica*	Bone	South America	0.0	-24.00	-24.00	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Seashell (Ruditapes)	*Venerupis philippinarum?*		Japan	-0.2	-23.60	-23.80	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	Ribbed mussel	*Aulacomya atra*	Bone	South America	-0.5	-19.90	-20.40	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Ribbed mussel	*Aulacomya atra*	Bone	South America	-0.1	-20.10	-20.20	Admiraal et al., 2023	https://doi.org/10.1038/s41598-

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Marine	rock cod	*Eleginops maclovinus*	Bone	South America	-0.2	-20.70	-20.90	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine				UK	0.6	-23.70	-23.10	Craig et al. 2013	DOI: 10.1038/nature12109
Marine	Harbour seal	*phoca vitulina*	Bone	South America	-0.3	-22.30	-22.60	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Harbour seal	*phoca vitulina*	Bone	South America	-3.3	-22.50	-25.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Short fin pilot whale	*Globicephala macrorhynchus*		Japan	0.5	-23.60	-23.10	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	Tuna	*Thunnus sp.*		Japan	1.0	-25.00	-24.00	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	Pilot Whale			Japan	-0.4	-22.90	-23.30	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine	silversides	*Odontesthes sp.*	Bone	South America	-0.4	-21.10	-21.50	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5

Marine	silversides	*Odontesthes sp.*	Bone	South America	0.1	-21.10	-21.00	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	silversides	*Odontesthes sp.*	Bone	South America	0.4	-24.20	-23.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	South American sea lion	*Otaria flavensis*	Bone	South America	-0.1	-19.60	-19.70	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Marine	Pilot Whale	*Globicephala*		Japan	0.4	-25.30	-24.90	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Marine				UK	0.4	-21.80	-21.40	Craig et al. 2013	DOI: 10.1038/nature12109
C3 plant	tortuous mesquite	*Prosopis flexuosa*	APAA exp tissue	Argentina	-0.5	-28.60	-29.10	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
C3 plant	Chañar	*Geoffroea decorticans*	APAA exp tissue	Argentina	0.2	-28.30	-28.10	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
C3 plant	Piquillin	*Condalia microphylla*	APAA exp tissue	Argentina	0.5	-27.30	-26.80	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5

C3 plant	Piquillin	*Condalia microphylla*	APAA exp tissue	Argentina	0.1	-27.70	-27.60	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
C3 plant	Chañar	*Geoffroea decorticans*	APAA exp tissue	Argentina	-0.1	-31.30	-31.40	Admiraal et al., 2023	https://doi.org/10.1038/s41598-023-42662-5
Porcine	Pig	*Sus scrofa*	Adipose	UK	0.8	-24.80	-24.00	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.6	-26.40	-24.80	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.7	-26.50	-24.80	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.1	-25.50	-24.40	Dudd 1999	https://research-infor

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Porcine	Pig	*Sus scrofa*	Adipose	UK	1.1	-26.00	-24.90	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.3	-25.10	-23.80	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.3	-25.70	-24.40	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Adipose	UK	1.3	-25.40	-24.10	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/

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Porcine	Pig	*Sus scrofa*	Adipose	UK	-0.9	-24.40	-25.30	Dudd 1999	https://research-information.bris.ac.uk/ws/portalfiles/portal/34490556/DX213544.pdf
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	0.9	-24.70	-23.80	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	1.1	-24.69	-23.60	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	0.6	-24.38	-23.80	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	0.8	-24.28	-23.50	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	1.2	-24.10	-22.90	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Kazakhstan	1.1	-23.89	-22.80	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig	*Sus scrofa*	Soft tissue	Switzerland	1.3	-28.67	-27.40	Spangenberg et al. 2006	https://doi.org/10.101

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Porcine	Pig	*Sus scrofa*	Soft tissue	Switzerland	-0.9	-25.62	-26.50	Spangenberg et al. 2006	https://doi.org/10.1016/j.jas.2005.05.013
Porcine	Pig blood	*Sus scrofa*	Blood	Kazakhstan	-2.9	-28.39	-31.29	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Porcine	Pig blood	*Sus scrofa*	Blood	Kazakhstan	-1.3	-28.79	-30.09	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Ruminant	Sheep	*Ovis aries*	Bone	Kazakhstan	-1.8	-28.19	-29.99	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Ruminant	Sheep	*Ovis aries*	Bone	Kazakhstan	-1.8	-30.39	-32.19	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Ruminant	Cow	*Bos taurus*	Ruminant Adipose	Kazakhstan	-2.1	-30.19	-32.29	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Ruminant	Cow	*Bos taurus*	Ruminant Adipose	Kazakhstan	-2.0	-30.49	-32.49	Outram et al. 2009	https://doi.org/10.1016/j.jas.2012.02.009
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-5.5	-26.36	-31.81	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-4.6	-25.94	-30.53	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5

Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-3.2	-26.57	-29.80	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-3.2	-26.28	-29.50	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-4.3	-26.65	-30.97	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-4.3	-26.95	-31.24	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-3.6	-27.25	-30.88	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-4.5	-27.32	-31.79	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-3.6	-27.97	-31.59	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Dairy Ruminant C3/C4	Cow	*Bos taurus*	Ruminant Milk	UK	-3.5	-28.28	-31.82	Roffet-Salque, M. et al. (2017b).	DOI:10.1007/s12520-016-0357-5
Wild non-ruminant	Bear	*Ursus arctos arctos*	Bone	Alaska	-1.3	-26.3	-27.6	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824

Wild non-ruminant	Bear	*Ursus arctos arctos*	Bone	Alaska	-0.7	-27.1	-27.8	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824
Wild non-ruminant	Bear	*Ursus arctos arctos*	Bone	Alaska	-0.2	-26.8	-27.0	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824
Wild non-ruminant	Bear	*Ursus arctos arctos*	Bone	Alaska	-0.8	-29.0	-29.8	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824
Wild non-ruminant	Tortoise	*Chelonoidis chilensis*	Bone	Alaska	0.5	-26.3	-25.8	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824
Wild non-ruminant	Rhea	*Rhea sp.*	Bone	Alaska	0.1	-27.9	-27.8	Admiraal et al., 2023a	https://doi.org/10.1016/j.jas.2023.105824
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Argentina	2.5	-26.9	-24.37	March et al., 2013	https://doi.org/10.1515/9781789201574-020
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Argentina	1.0	-26.6	-25.54	March et al., 2013	https://doi.org/10.1515/9781789201574-020

Wild non-ruminant	Green pheasant	Phasianus versicolor	Bone	UK	-0.06	-28.3	-28.32	Craig et al. 2013	DOI: 10.1038/nature12109
Wild non-ruminant	Raccoon dog	Nyctereutes procyonoides	Bone	UK	0.18	-28.33	-28.15	Craig et al. 2013	DOI: 10.1038/nature12109
Wild non-ruminant	Raccoon dog	Nyctereutes procyonoides	Bone	UK	-0.8	-25.88	-26.68	Craig et al. 2013	DOI: 10.1038/nature12109
Wild non-ruminant	Wild boar	Sus scrofa	Bone	UK	-0.44	-26.49	-26.93	Craig et al. 2013	DOI: 10.1038/nature12109
Wild non-ruminant	Wild boar	Sus scrofa	Bone	UK	0.43	-28.10	-27.67	Craig et al. 2013	DOI: 10.1038/nature12109
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	1.12	-27.42	-26.30	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	-0.74	-28.13	-28.87	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	-0.15	-27.75	-27.90	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	-1.43	-27.8	-29.22	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	-0.08	-29.5	-29.57	Lucquin et al. 2016	https://doi.org/10.101

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Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	-0.56	-28.9	-29.50	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Wild non-ruminant	Wild boar	Sus scrofa	Bone	Japan	0.85	-27.4	-26.53	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Deer	Cervus elaphus		France	-3.3	-28.7	-32.00	Bondetti et al. 2021	https://doi.org/10.1111/arcm.12631
Ruminant	Deer	Cervus elaphus		France	-6.1	-24.3	-30.40	Drieu et al., 2021a	https://doi.org/10.1016/j.jasrep.2020.102682
Ruminant	Guanaco	Lama guanicoe		South America	-2.9	-27.8	-30.70	Admiraal et al. 2023	https://doi.org/10.1038/s41598-023-42662-5
Ruminant	Guanaco	Lama guanicoe		South America	-3.2	-27.7	-30.90	Admiraal et al. 2023	https://doi.org/10.1038/s41598-023-42662-5
Ruminant	Guanaco	Lama guanicoe		South America	-2.5	-26.5	-29.00	Admiraal et al. 2023	https://doi.org/10.1038/s41598-023-42662-5
Ruminant	Hokkaido deer	Cervus sp.		Japan	0	-28.1	-28.10	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011

Ruminant	Hokkaido deer	Cervus sp.		Japan	-2.3	-30.4	-32.70	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Red deer	Cervus elaphus		Japan	-1.7	-31.5	-33.20	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Red deer	Cervus elaphus		Japan	-2.2	-31.4	-33.60	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Red deer	Cervus elaphus		Japan	-4.8	-28.3	-33.10	Drieu et al., 2021a	https://doi.org/10.1016/j.jasrep.2020.102682
Ruminant	Roe deer	Capreolus capreolus		France	-1.2	-31.2	-32.40	Bondetti et al. 2021	https://doi.org/10.1111/arcm.12631
Ruminant	Sika deer	Cervus nippon		Japan	-1.6	-27.8	-29.40	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Sika deer	Cervus nippon		Japan	-3.5	-28.1	-31.60	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011
Ruminant	Sika deer (Nagano)	Cervus nippon		Japan	-3.2	-31.9	-35.10	Lucquin et al. 2016	https://doi.org/10.1016/j.tetlet.2016.01.011