# An Assessment of the Effects of an Oil Spill on Coastal Archaeological Sites in Louisiana





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#### **About the Cover**

Photograph of the Acorn Mounds site (16SB185), view to the south, showing Mounds B, C, and A (left to right).

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## **List of Abbreviations and Acronyms**

AMS Accelerator mass spectrometry

AMSL Above mean sea level

ANID Analytical identification number

ASTM American Society for Testing and Materials
BCE Before Common Era, comparable to BC

BP US BP United States

BOEM Bureau of Ocean Energy Management

CWPPRA Coastal Wetlands Planning, Protection and Restoration Act

CZMA Coastal Zone Management Act
CE Common Era, comparable to AD
CRM Cultural resource management
DOI US Department of the Interior
ESP Environmental Studies Program

ESPIS Environmental Studies Program Information System GC-CESU Gulf Coast Cooperative Ecosystems Studies Unit

GC/MS Gas chromatography-mass spectrometry

GOM Gulf of Mexico

LAPAL Louisiana Public Archaeology Lab LDA Louisiana Division of Archaeology

LDWF Louisiana Department of Wildlife and Fisheries
LUMCON Louisiana Universities Marine Consortium

MC252 Mississippi Canyon Block 252

MURR University of Missouri Research Reactor

NAA Neutron activation analysis

NOAA National Oceanic and Atmospheric Administration

NRHP National Register of Historic Places
OCD Office of Cultural Development

OCM State of Louisiana Department of Natural Resources Office of Coastal Management

OCS Outer Continental Shelf

OCSLA Outer Continental Shelf Lands Act

PI Principal Investigator

PO Project Officer

SHPO State Historic Preservation Office

TU Test unit

ULL University of Louisiana at Lafayette
USACE United States Army Corps of Engineers

USCG United States Coast Guard

XRF X-Ray fluorescence YBP Years before Present

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#### 1. Introduction

On April 20, 2010, a catastrophic incident on the *Deepwater Horizon* drilling rig in Mississippi Canyon Block 252 (MC252) in the northern Gulf of Mexico (GOM) set off a far-reaching and unprecedented environmental disaster. The MC252 wellhead blowout resulted in the largest known marine oil spill in history, releasing approximately 4.9 million barrels of crude oil that eventually impacted more than a thousand miles of shoreline along the north-central Gulf Coast (Figure 1: Jove 2015: McNutt et al. 2012: Nixon et al. 2016). Affected landforms included the wetlands of the Mississippi River delta, the largest river delta in North America, and its barrier islands, beaches, and tidal marsh zone (BOEM 2014; Michel et al. 2013). Dispersants were applied during the response to dissolve the crude oil and prevent the formation of oil slicks. The use of dispersants, both at the wellhead and by aerial spraying, was unprecedented in terms of quantity and extent (Kujawinski, et al. 2011; Seidel, et al. 2016; USCG 2011). Following the capping of the well and containment of the spill, disaster response efforts focused on shoreline cleanup and mitigating the impacts on coastal environments, marine and wetlands ecology, public health, and local economies (Aeppli, et al. 2012; Austin et al. 2014a, 2014b; Baker et al. 2016; Beyer et al. 2016; ERG 2014; Hester et al. 2016; IOM 2010; McClenachan et al. 2013; Middlebrook et al. 2012; Ortmann et al. 2012; Reddy et al. 2012; Romero et al. 2015; Schwacke et al. 2014; Silliman et al. 2012; Valentine et al. 2014).

During the oil spill response, it was immediately apparent that cultural resources were being impacted along the Gulf's shorelines and underwater, including hundreds of previously recorded historic and prehistoric archaeological sites and unknown numbers of unrecorded sites (Borrell 2010; Chin and Church 2010). Based on the precedent established by the response to the *Exxon Valdez* oil spill of 1989 (Bittner 1996; Reger et al. 2000), archaeologists were employed in Shoreline Cleanup Assessment Technique (SCAT) teams for site monitoring and surveying across the northern Gulf Coast (Cloy and Ostahowski 2015; HDR, Inc. 2011). The ensuing cleanup and environmental remediation documented the presence of oil at sites along the coasts of Louisiana, Mississippi, Alabama, and Florida. Due to the location of MC252, a majority of the affected archaeological sites are located in the Mississippi River delta of southeast Louisiana, especially along the shorelines around Chandeleur and Breton sounds, and Barataria, Timbalier, and Terrebonne bays (Michel et al. 2013).

At the time of the spill and cleanup, there was surprisingly little available information on the potential effects of oil, dispersant, or dispersed oil on archaeological sites, including immediate effects on the archaeological record and possible long-term impacts on future archaeological research. Questions were raised concerning the potential effects on cultural resources. Would crude oil infiltrate archaeological deposits on coastal sites and if so, would it contaminate artifacts and other materials? Dispersants are known to accelerate and increase the penetration of crude oil into marine sediments (Zuijdgeest and Huettel 2012), with unknown effects on the archaeological record. How might hydrocarbon contamination from an oil spill affect radiocarbon dating? While archaeologists working with SCAT teams were well informed of the possible indirect effects of the cleanup response and environmental remediation on archaeological sites, the long-term and direct effects of hydrocarbon and dispersant exposures were unknown. A lack of reliable and up-to-date information on the condition and historical significance of the affected cultural resources was also a concern, a situation that was soon systematically addressed through extensive surveys and site monitoring along the north-central Gulf Coast (Cloy and Ostahowski 2015; HDR, Inc. 2011).

There was added urgency in addressing the effects of the MC252 oil spill considering non-renewable archaeological sites may have been irreversibly impacted. Archaeological sites in the Mississippi River delta and north-central coast of the GOM provide unique yet increasingly endangered sources of information on human habitation and historical ecology spanning thousands of years, preceding the introduction of written history (Brown 1984, 2004; Jeter and Williams 1989a, 1989b; Kidder 2004a;

Gagliano 1984; Giardino 1984). The paucity of available information on many, if not most, archaeological sites in Louisiana's coastal zone precluded determination of historical significance or assessment of eligibility for listing in the National Register of Historic Places (NRHP). Only one previously recorded site in the region, Fort Livingston (16JE49), was listed on the NRHP (Cloy et al. 2013). The Louisiana State Historic Preservation Officer (SHPO) determined that additional sites were eligible for listing on the NRHP. Unknown numbers of other archaeological sites were unrecorded. It would prove difficult, if not impossible to determine potential effects without knowing more about the existing cultural resources, rapidly changing environmental conditions, archaeological integrity, and respective historical significance.

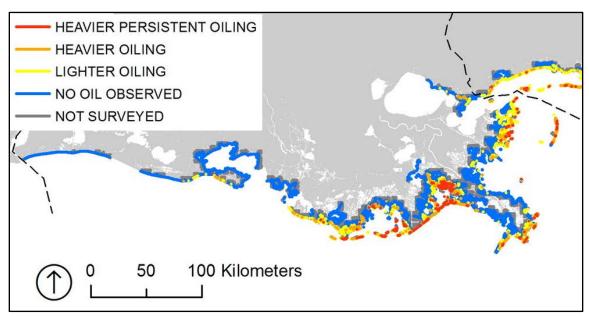


Figure 1. Impacted coastline of the MC252 oil spill.

Map of the Louisiana Coastal Zone, showing areas impacted by the MC252 oil spill. Adapted from Nixon et al. (2016).

In the wake of the *Deepwater Horizon* incident, SCAT archaeologists surveyed shoreline and near shore areas affected by the spill, documenting and monitoring the direct and indirect impacts on sites. The findings of the cultural resources investigation by HDR, Inc. (2011) for the BP Gulf Coast Restoration Organization were immediately relevant to this study and provided much of the background information (Cloy and Ostahowski 2015). More than 4,900 km (3,045 mi) of shoreline was surveyed in Louisiana. HDR, Inc. recorded 50 new sites and revisited 163 previously recorded sites in 11 coastal parishes (Cameron, Iberia, Jefferson, Lafourche, Orleans, Plaquemines, St. Bernard, St. Mary, St. Tammany, Terrebonne, and Vermilion parishes). Although four newly recorded sites and 15 previously recorded sites were recommended as being potentially eligible for listing on the NRHP (Cloy and Ostahowski 2015:7-18, 7-21), the historical significance of a majority of the sites is undetermined (70% or 149 of 213 sites). Among the sites surveyed, prehistoric components are common throughout the Mississippi River delta. Ceramic sherds produced and used by Native Americans are the most ubiquitous class of artifacts at these sites, with a majority dating from the Woodland period (500 BCE–1200 CE) and subsequent Mississippi period (1200–1700 CE). The cultural affiliation of these sites with contemporary Native Americans includes the Federally recognized Chitimacha Tribe of Louisiana.

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<sup>&</sup>lt;sup>1</sup> A problematical designation, "prehistoric" is not intended to imply lacking in historical significance and is used in this report in referring to archaeological contexts and materials that predate written documentation in the region (i.e., before the time frame of 1540 to 1700 CE).

The need for a systematic study of the effects of an oil spill on archaeological sites was underscored by the initial findings of the SCAT surveys, which recorded both the presence of oil and rapidly deteriorating shoreline conditions associated with coastal erosion, subsidence and relative sea-level rise (Burkett et al. 2003; HDR, Inc. 2011). In fact, the degradation of coastal landforms where many of the sites are located may have exacerbated the oiling of some sites. These include formerly terrestrial "wet sites" that are now intermittently or frequently subject to inundation (Jones 2014; Jones et al. 2009; Saltus and Pearson 2010). Other sites previously inundated by intermittent storm surge are now partially or entirely underwater (Ostahowski 2015, 2016). As the shoreline recedes, the potential reach of an oil spill in the intertidal zone moves inland. In short, the potential effects of an oil spill are compounded by other environmental factors.

An unknown number of either recorded or unrecorded sites in Louisiana have already been lost to coastal erosion and subsidence. Based on surveys of shoreline areas impacted by the MC252 oil spill, approximately 47% (or 77 of 163) previously recorded archaeological sites along Louisiana's coast are now mostly or partially submerged (Cloy and Ostahowski 2015:7-18; Ostahowski 2015). This is not entirely unexpected; the causes, consequences, and rate of coastal erosion in the Mississippi River delta have been the subject of concern for decades (Britsch and Dunbar 1993; Britsch and Kemp 1990; Gagliano et al. 1977, 1981, 1982; Neuman 1977a; NRC 2006; Penland et al. 1990; Walker et al. 1987). These processes cannot be accurately characterized as entirely environmental (Twilley et al. 2016). The combined effects of levee construction, channelization, canal dredging, saltwater intrusion, coastal erosion, subsidence, and subsequent shoreline retreat have been devastating. Though not caused by an oil spill, the ongoing degradation and loss of coastal wetlands is linked to long-term human modifications of the deltaic landscape through hydraulic engineering, extraction of natural resources, and industrial development (Chan and Zoback 2007; Davis 2010; Gramling 1996; Theriot 2014). The effects of an oil spill on coastal archaeological sites should, consequently, be understood in the context of anthropogenic environmental processes that have reshaped and continue to rapidly transform Louisiana's Gulf Coast (Penland et al. 1988, 2004).

The present study was the result of a request from the Louisiana Department of Culture, Recreation, and Tourism, Office of Cultural Development, Division of Archaeology, to the Bureau of Ocean Energy Management for assistance with the scientific recordation of the effects of the oil spill on cultural resources. Though the effects of oil on archaeological sites had been a focus of study in the aftermath of the *Exxon Valdez* spill of 1989 in the Arctic wilderness of Alaska (Bittner and Reger 1995; Gundlach et al. 1991; Reger et al. 2000; Yarbrough 1997), there had been no previous studies on the effects of an oil spill of this magnitude in a warm-water, deltaic environment comparable to the Mississippi River delta. There were no baseline data to comprehend the potential ramifications for the archaeological record or cultural resources. This study was conducted under the authority of the Outer Continental Shelf Lands Act (OCSLA; 43 USC 1345e) and the Consolidated Appropriations Act of 2014 (Public Law 113-76). On July 8, 2014, BOEM established a cooperative agreement with the University of Louisiana at Lafayette (ULL) Louisiana Public Archaeology Lab (LAPAL).

The principal goals of this study were to assess the effects of the MC252 oil spill on prehistoric archaeological sites on Louisiana's Gulf Coast and to provide BOEM and SHPO with information relevant to cultural resource management (CRM) planning and remediation. Among the most salient questions to be addressed in this report: Has oil and/or dispersant permeated intact archaeological deposits and interacted with artifacts and ecofacts? How might this affect analytical techniques, such as applications of radiocarbon dating and studies of absorbed residues in pottery? These and other questions underlay the development of this research and establishment of a cooperative agreement to carry it out. This research was designed to address the possible contamination of archaeological deposits and also the potential consequences for future field research, including the costs of fieldwork and subsequent analyses. Such information is essential to BOEM decision making for the GOM region under the National

Environmental Policy Act (NEPA). As a planning tool for the SHPO, it is also pertinent to compliance with the National Historic Preservation Act (NHPA) and its implementing regulations. The findings of this study are relevant to future offshore oil and gas development in the GOM region. Moreover, the results of this investigation are intended to assist BOEM with NEPA analysis, resource management, and environmental protection related to the development of oil and gas on the Outer Continental Shelf (OCS). Finally, this study should inform future responses to oil spills and serve as a baseline and reference point in mitigating the effects of oil spills on archaeological sites.

Permitting for this project required the approval of many different agencies within the State of Louisiana, and required coordination with other Federal agencies' review and approval processes. An application for a permit to conduct archaeological investigations was presented before the Louisiana Archaeological Survey and Antiquities Commission and approved on October 29, 2014, as promulgated in the Louisiana Register, Volume 20, Number 4, April 20, 1994, and as outlined in the current Archaeological Code of Louisiana. An Unmarked Human Burial Sites Permit was issued on September 26, 2014, in order to comply with §676.C of the Louisiana Unmarked Human Burial Sites Preservation Act (R.S. 8:671-681). Lastly, a review was conducted by the Department of Natural Resources Office of Coastal Management (OCM) for Coastal Zone Consistency under rules in accordance with Section 307(c) of the Federal Coastal Zone Management Act of 1972 as amended, as well as National Oceanic and Atmospheric Administration (NOAA) regulations on Federal consistency at 15 CFR §930.35. A negative consistency determination letter was issued on September 12, 2014.

The following technical report presents the rationale, objectives, methods and results of this four-year long study. The report is organized into nine sections. The goals and objectives of the research, including hypotheses and research design, are laid out in the remainder of the Introduction. Section 2 presents the environmental setting, from physiographic regions and geomorphic processes to ecological regions and soils. The culture history of the Mississippi River delta, including much of the southern Lower Mississippi Valley and north-central Gulf Coast, is summarized in Section 3, with particular attention given to the last three millennia of human occupation in the deltaic plain, or since the onset of the Early Woodland period (800 BCE). Section 4 provides a brief overview of previous archaeological investigations on Louisiana's Gulf Coast, again focusing on the delta and investigations relevant to understanding the effects of an oil spill on archaeological sites.

The research methodology is described in Section 5, beginning with the logistical issues involved in site selection and sampling. Eight archaeological sites were sampled and assessed as part of this study, including six sites where SCAT teams had previously recorded the presence of oil during the MC252 cleanup response. Two sites where oil was not previously observed were included in this study to serve as controls. Excavations and sampling of archaeological deposits were conducted at all eight sites between September of 2014 and September of 2015. Section 5 concludes with a description of the analytical and laboratory methods, including classifications, sample selection, the chemical characterization of hydrocarbons, elemental analysis, absorbed residue analysis, and radiocarbon analysis.

The findings and results of this study are presented in Section 6, beginning with an analysis of the artifact collections. The results most relevant to the stated goals and objectives of this study involved the chemical detection and characterization of hydrocarbons, elemental and absorbed pottery residue analyses, and radiocarbon dating. Based on these results, Section 7 presents a site assessment summary and considers the broader ramifications of the investigation. The major research accomplishments are reviewed, along with some of the challenges encountered along the way. The hypotheses formulated in the Introduction are evaluated in light of the results of the fieldwork and laboratory analyses. Section 7 also considers how the knowledge gained from this study might inform CRM planning, artifact and collections management, and cost estimates for conducting future research at archaeological sites affected by an oil spill. Section 8 provides a brief summary with concluding remarks. Appendices A through G

provide detailed information on the samples submitted for analysis, the various methodologies employed, and analytical results.

#### 1.1 Goals and Objectives

The goals of this research were to: (1) assess the immediate and long term effects of the 2010 MC252 oil spill on prehistoric archaeological sites on the Louisiana Gulf Coast; and (2) provide BOEM and the Louisiana Office of Cultural Development (OCD) with information relevant to CRM planning, in compliance with Federal and State legislation and accompanying regulations. The posited effects may include, but are not limited to, immediate consequences from the presence and accumulation of oil in the archaeological record. The archaeological record of Louisiana's Gulf Coast is composed of classes of artifacts such as ceramics, ecofacts, such as bone and shell, cultural features, such as earth and shell midden, and associated anthropogenic products, such as charcoal and organic residues, along with constituent soil matrices. Potential effects consequently include long-term, direct impacts on the analyses and conservation of artifacts and ecofacts, but also potential impacts on archaeological formation processes or the physical and chemical composition of constituent matrices, *in situ* preservation of information, excavation, data collection methods, and curation.

Furthermore, both immediate and long-term, *indirect* effects of an oil spill on archaeological resources may result from the oil spill response, including subsequent cleanup, application of dispersants, removal of oil and site remediation, environmental restoration, and future research costs. Dispersants used during the MC252 oil spill and during the cleanup response may interact with crude oil and enter archaeological contexts, producing unexpected and unintended results. As demonstrated in marsh environments, in some instances, site remediation and oil removal methods may prove more destructive to archaeological resources than initial accumulation. Systematic and comprehensive site assessment should inform a greater understanding of the potential adverse indirect effects of an oil spill from undertakings such as cleanup, restoration, and remediation.

The potential effects of an oil spill on archaeological sites in environments comparable to the Mississippi River delta have not been previously examined. Characterization of specific impacts as adverse, nonexistent, or relatively less detrimental should be based on a systematic study geared toward quantitative and qualitative analysis. Such analysis and characterization was a primary goal of this investigation. A second, related goal was to provide information relevant to CRM decision making and planning. This research was intended to address the needs of the Louisiana OCD, Division of Archaeology and SHPO in overseeing compliance of agencies and industry with Federal and State legislation, including NEPA, NHPA, and implementing regulations. This study supports the mission of BOEM by considering environmental impacts in the management of offshore oil and gas development in the north-central GOM region. The project goals and objectives can be outlined as follows:

- (1) Assess the effects of the MC252 oil spill on prehistoric archaeological sites on the Louisiana Gulf Coast.
  - (a) Characterize the proximate impacts on the archaeological record, including artifacts, ecofacts, cultural features, and analytic techniques.
  - (b) Evaluate long-term impacts on formation processes, data collection, analyses, conservation, and curation.
  - (c) Examine the potential for indirect impacts that may result from excavation, the cleanup response, remediation and environmental restoration.
- (2) Provide the SHPO and OCD Division of Archaeology with information relevant to CRM planning and regulatory compliance.

Based on these goals, specific research objectives and tasks were defined. The objectives included the selection of archaeological sites for inclusion in this study, fieldwork for assessment of immediate and long-term impacts from the MC252 oil spill, examination of the specific effects of oil and other contaminants on the archaeological record, and evaluation of these effects on research costs and resource management. Sites containing historic and prehistoric components were included in the study, but the primary focus was to assess the effects on sites with indigenous, Native American archaeological deposits in the region that predate written documentation. Related research tasks consisted of coordinating site access, sampling, impact assessment, oil source analysis, resource management, crew training, and artifact management. The research objectives and tasks are further discussed in the sections of this report on Research Methodology and Analytical Results.

#### 1.2 Hypotheses

The goals and objectives of this research guided the formulation of hypotheses to advance current knowledge regarding the potential effects of an oil spill on coastal archaeological sites. The presence of oil at archaeological sites was described during the cleanup response as very light, light, moderate, or heavy, with tar balls noted if present at the time of the site visit (Cloy and Ostahowski 2015: Appendix 3). Such observations about the presence (or absence) of oil are obviously non-systematic, sensory, and subjective, based on visual inspection and changing environmental conditions along shorelines. Linkage to MC252 as the source has also been largely circumstantial, based on the location and time of the site visit during the response. Oil from other sources may be present on the surface and beneath the ground at archaeological sites on the coast. A central hypothesis of this research is that chemical analyses can objectively determine and quantify the presence of oil before radiocarbon dating and archaeometric techniques. This includes chemical fingerprinting to identify possible source.

Regarding sites where SCAT teams observed oil during the cleanup response, investigators posited that oil would be most evident in more highly permeable artifacts. The evidence for oil at these sites, whether or not it is traced to the 2010 MC252 spill, was hypothesized to inversely vary according to the depth of stratified (undisturbed) deposits. Coastal sites with secondary, redeposited contexts would present the most prevalent exceptions, where reworked shoreline deposits are likely to contain oil, regardless of depth. For artifacts and samples where the presence of oil or dispersant is confirmed, investigators conjectured that sample pretreatment mitigates any adverse effects on archaeometric techniques. This research also investigated whether oil is detectable within archaeological deposits at sites where SCAT teams *did not observe oil* during the MC252 oil spill response. These hypotheses are addressed in further detail, based on the research findings and analytical results, in the Site Assessment Summary of the report.

### 2. Environmental Setting

The Mississippi River delta is a dynamic fluvial environment of Holocene age deposits (after ca. 10,000 years ago) and the outlet of the largest river drainage in North America. The Mississippi River is estimated to account for more than half of the discharge of all drainages throughout the Atlantic and Gulf Coastal Plain provinces, as well as a majority of sediment transported to the Outer Continental Shelf (OCS) (Walker and Coleman 1987:55). Though the formation and transgression of successive Mississippi River deltaic lobes date from the later Holocene, more ancient sedimentary deposits of the Pleistocene Epoch (2.6 million–10,000 years ago) can be found within the Lower Mississippi Valley in present-day northeast and southwest Louisiana (Autin 2002; Autin et al. 1991; Coleman et al. 1998; Saucier 1974, 1994a, 1994b; Saucier and Snead 1989). The Gulf Coastal Plain is characterized by a broad and nearly flat submarine shelf that was exposed during the last glacial maximum, around 21,000 years ago, with deeply dissected plains and piney hills stretching northward to the Fall Line and Piedmont Province (Goins and Caldwell 1995; Yodis and Colten 2012).

The Lower Mississippi Valley begins near the Ohio River confluence, where the Mississippi River descends into its broad alluvial floodplain. The southern Valley encompasses the alluvial floodplains, natural levees and Pleistocene terraces south of the Arkansas River, with its own distinctive riverine environment and culture history (Dye 2015). There are further environmental, geomorphological and culture historical distinctions south of the Red River confluence, from the Natchez Bluffs to the Mississippi River mouth (Kidder 2004a; Kniffen 1968). Spreading out on the north-central Gulf Coast, the Mississippi River delta lies at the southern terminus of the Valley and is the primary focus of this study.

The effects of an oil spill on archaeological sites must be understood in terms of this dynamic environmental setting, including the physiographic regions and geomorphic processes of the Mississippi River delta and north-central Gulf Coast. The overview of physiography and geomorphology includes a consideration of related geoarchaeology and chronology, including the successive formation and transgression of deltaic lobes. Issues of subsidence, coastal erosion and sea-level rise are pertinent in this regard, especially as these geomorphic and anthropogenic processes also relate to past and present human presence in the delta. The environmental setting concludes with a review of associated ecological regions and soils, with the focus again placed on the delta and contiguous coastal zone of south Louisiana (LA DNR 2010). The natural levees, bayous, fresh and saltwater marshes, estuaries, bays, barrier islands and tidal mudflats of the delta form the backdrop for the present study. In the intertidal zone where landscapes and waterscapes intermingle, the archaeological record of the past two millennia is rapidly deteriorating due to coastal erosion and subsidence.

# 2.1 Physiography, Geomorphology, and Geoarchaeology

The majority of the coastal zone of south Louisiana is a geologically recent product of the Mississippi River and its distributaries, dating from the Holocene Epoch, or after 10,000 years ago. Louisiana's coastal region can be subdivided into the Deltaic Plain on the east and Cheniere Plain on the west, both of which have been formed and transformed over the past few millennia by sediment transport and hydraulic processes of the Mississippi River. Coastal marshes extend inland from the shorelines of the southernmost parishes, with Mississippi River alluvium covering the southeastern portion of the State, between the Pleistocene terraces of southwest Louisiana and the Florida parishes north of the Pontchartrain-Maurepas Basin (Figure 2). The Mississippi Sound and Galveston Bay lie to the east and west, respectively, with the submerged yet relatively shallow continental shelf extending into the Gulf of Mexico (GOM) to the south (Gagliano 1984:9–11). The GOM OCS consists of submerged lands under U.S. Federal jurisdiction beginning three nautical miles from the Louisiana coastline.

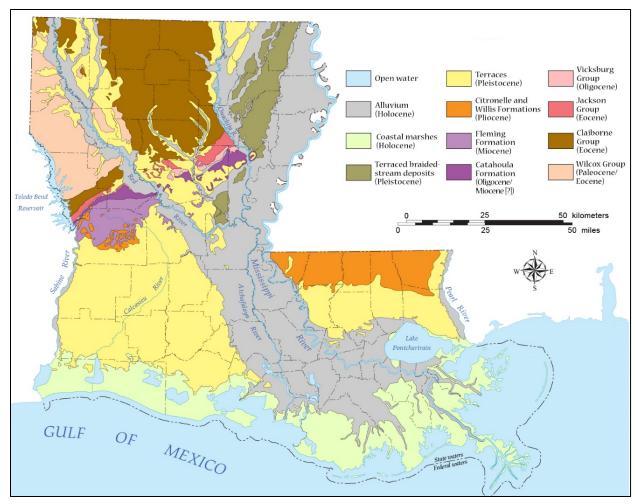


Figure 2. Geologic map of Louisiana.

(Louisiana Geological Survey 2008)

One of the largest drainage basins in the world, the Mississippi River drains an immense area greater than 1,245,000 square miles (3,224,535 km²), from the Appalachians on the east to the Rocky Mountains on the west. The lower alluvial valley of the Mississippi River is a vast and fertile floodplain that stretches from the confluence of the Mississippi and Ohio rivers to the Deltaic Plain and river mouth (USACE 2017). The lower alluvial valley in Louisiana bisects the older, Pleistocene Age Terrace Uplands of the north-central Gulf Coastal Plain, from the rolling Prairie Terrace on the southwest to the loessal Natchez bluffs and piney hills of the Florida Parishes on the east (Kniffen 1968:8–9). During the millennia since the end of the last Ice Age, the Mississippi transformed from a braided stream terrace carrying a torrent of glacial meltwaters to its present-day broad meandering channel with numerous oxbow lakes and ecologically-diverse flood basins (Blum et al. 2003). The glacial outwash deposits of abandoned valley trains such as Macon Ridge in northeast Louisiana bear witness to the tremendous geological forces that formed the present-day Lower Mississippi Valley.

During the last glacial maximum of the late Pleistocene Epoch the shoreline of present-day Louisiana was as much as 125 miles (200 km) farther south. Relative sea level rise in the northern GOM was produced by eustatic or world-wide sea-level rise due to glacial melting, deltaic subsidence, and tectonic processes (Gagliano 1984; Gagliano and Kemp 2015; Gagliano et al. 1982). Relative sea level stabilized by the end of the mid-Holocene but continues to fluctuate due to subsidence, geologic faulting, and changes in the climate (Gagliano 2005; Gagliano et al. 2003; Penland and Ramsey 1990). Coastal land loss is

consequently not new, but an increasingly anthropogenic process that is reshaping Louisiana's coastline (Barras et al. 2003, 2008; Couvillion et al. 2011; Gagliano et al. 1981; LGS 1992; Marshall 2014).

The Mississippi River formed and transformed the Deltaic Plain during the last five or six millennia of the Holocene as rising sea level stabilized. The Deltaic Plain is a complex network of natural levee ridges and crevasses, distributary channels, lakes, flood basins and ecologically diverse swamp lands, as well as freshwater, brackish and saltwater marshes. Surface elevation is generally very low, with little relief, but ranges from sea level to more than 20 feet (6 m) above mean sea level (AMSL) on the natural levees produced along the myriad river channels coursing toward the GOM (Aslan et al. 2005). Unlike the towering artificial levees constructed over the past century, the natural levees of the Mississippi River allowed for regular overbank flooding and periodic breaches or crevasses during floods. The seasonal flooding of back swamps and alluvial plain, along with episodic changes in the river's meandering course, delivered replenishing sediment and produced the many lakes and wetlands that would ultimately characterize south Louisiana as a "sportsman's paradise." The major lakes of Louisiana's Deltaic Plain include Pontchartrain, Maurepas, Lake Salvador, Lac des Allemands, and Grand Lake. As with the surrounding flood basins, oxbow lakes, and natural levees, the lakes are a byproduct of dynamic and ever changing fluvial processes (Morris 2012).

The Mississippi River has laid down millions of tons of nutrient-rich sediment over the millennia and continues to transport its muddy load to the continental shelf. Alluvial deposition, sediment loading and compaction lead to subsidence or the gradual downward movement of subsurface strata in deltaic landforms relative to sea level, which is in turn accelerated by active geologic faults that run east to west beneath the coastal wetlands of south Louisiana (Gagliano 2005; Yuill et al. 2009). Subsidence has been generally offset in the past by continued alluvial deposition from periodic flooding, channel shifting, and natural levee crevasses. More than a century of levee construction, channelization, and hydraulic engineering have generally had the opposite effect, with the Mississippi River carrying a reduced sediment load to the OCS. Though the rate of subsidence is accelerating in southeast Louisiana, Gagliano (1999, 2005) has presented evidence that geologic faults are a major cause in the submergence of coastal landforms (Gagliano et al. 2003). This suggests a surprisingly active tectonic system, rather than merely compaction or anthropogenic subsidence in coastal land loss. Coastal land loss in the deltaic plain will be magnified by eustatic sea level rise (Barras et al. 2003; CWPPRA 2017; Glick et al. 2013; NRC 2006).

Louisiana's coastline is permeated by an intricate network of bayous, rivers, interdistributary estuaries and bays that also reveal the imprint and force of the Mississippi River. Two of the best known bayous of south Louisiana, the Lafourche and the Teche, are former distributaries of the Mississippi River. Both are located within the Atchafalaya Basin west of the Mississippi, the largest freshwater cypress swamp in the U.S. The Atchafalaya River channel has formed even more recently and now carries, on average, one-third of the Mississippi River's discharge south of the Old River Control Station (Barnett 2017; Reuss 1998). Along the coastline to the south, the bayous and freshwater swamp become brackish estuaries and saltwater marsh. A complex of barrier islands with beach ridges, mud flats, and tidal passes lie beyond the saltwater marshes, estuaries, and bays of the Deltaic Plain. The barrier islands are produced and maintained by the transport of sediment along the coastline, with the continuous deposition of silt and sand in shoreline currents previously counterbalancing erosion from tidal surges and storms (NRC 2006).

The Timbaliers, Grand Isle, Isle Grande Terre, and the Chandeleurs are prominent among the barrier island chains of south Louisiana, separated from the mainland marshes by Terrebonne Bay, Caminada, and Barataria bays on the west and Chandeleur Sound north and east of the present-day bird foot delta. The barrier islands are not static, but migrate and fluctuate in size and shape due to inundation, overwash, erosion, segmentation, and consolidation. Coastal restoration and barrier island stabilization efforts must take these geomorphic processes into account (Rosati and Stone 2009; Wamsley et al. 2010). The potential for rapid transformation of low-lying barrier islands due to inundation by storm surges is exemplified by the Chandeleur Islands, which were breached and severely eroded during recent

hurricanes (Sherwood et al. 2014). In addition to reduced deposition of heavier sediments, such as sand, relative sea level rise is compounding the deleterious effects on Louisiana's rapidly eroding barrier islands (NRC 2006; USGS 2017).

Most distinctively, the Deltaic Plain is made up of former and current deltaic lobes of the Mississippi River (Figure 3). The river formed a succession of deltaic lobes through the transgression and lateral shifting of distributaries over the past seven thousand years. In relative chronological order, these are known as the Maringouin, Teche, St. Bernard, Lafourche, and modern Plaquemines sub-deltas. The newly forming Atchafalaya subdelta, as seen in the impressive deposition of sediment at Wax Lake outlet and Atchafalaya Bay, can now be added to this deltaic progression. A revised chronology for the late Holocene sub-deltas indicates a more recent progression than previously suspected (Saucier 1994a; Törnqvist et al. 1996, 2006). The St. Bernard subdelta formed after 4,000 years ago. The Lafourche subdelta was active around 1,500 years ago, followed by the modern Plaquemines subdelta since approximately 1,300 years ago (Törnqvist et al. 1996:1695). Despite concerted efforts to prevent the impending diversion of the Mississippi River into the Atchafalaya, channel aggradation south of Old River almost ensures the continuation of a roughly one to two millennia long deltaic cycle. Construction of an immense system of levees, revetments, floodways, cut offs, and canals has done little more than postpone the inevitable (Barnett 2017; Coleman et al. 1998; Roberts 1997).

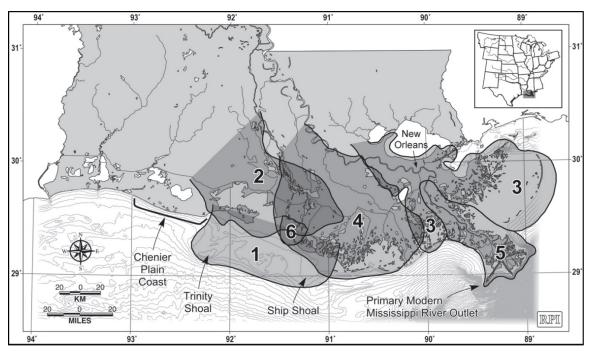


Figure 3. Deltaic lobes of the Mississippi River.

From oldest to youngest: (1) Maringouin, (2) Teche, (3) St. Bernard, (4) Lafourche, (5) Plaquemines–Balize, and (6) Atchafalaya (National Research Council 2006: Figure 2.1, page 31).

To the west of the Deltaic Plain, the Chenier Plain is also a late Holocene landform produced by the Mississippi River. "Chenier" refers to the isolated stands of oaks on relict beach ridges that are visible over vast expanses of marsh grass in southern portions of St. Mary, Iberia, Vermilion, and Cameron parishes. With surfaces of up to 12 feet (3.7 m) above mean sea level (AMSL), the cheniers stand out in the surrounding low-lying coastal landscape. The Chenier Plain is composed of fresh and saltwater marsh around the coastal bays and lakes of southwest Louisiana. From the Atchafalaya, Cote Blanche, and Vermilion bays on the east, the major lakes include the White, Grand, Calcasieu, and Sabine (Gould and McFarlan 1959; Owen 2008).

The westerly conveyance of Mississippi River sediment over the past three millennia has caused the seaward progression of the shoreline, such that more recently-formed cheniers are located closer to the present-day shore and previously-formed cheniers are found moving further inland. Archaeological components are consequently associated with the relative ages of these landforms. The formation of the Cheniere Plain succeeded millennia of postglacial sea level rise and stabilization by the end of the mid-Holocene (Gould and McFarlan 1959). Successive beach ridges formed as tidal and marine transgression reworked mudflats and estuaries. The formation and abandonment of Mississippi River distributaries continues to play an active role along the shorelines of southwest Louisiana, involving the prograding of mudflats around Vermilion Bay and Marsh Island (Owen 2008).

The salt domes are by far the most prominent geomorphic and tectonic feature of the Chenier Plain and adjoining Prairie Terrace. The salt domes have produced readily noticeable sedimentary uplift along the otherwise flat and low lying north-central coastal plain of the GOM. A northwesterly trending series of seven salt diapirs have formed the five well-known salt dome islands of southwest Louisiana, from Belle Isle on the southeast to Cote Blanche, Weeks Island, Avery Island, and Jefferson Island to the northwest. Surface elevation ranges markedly, from 75 to 170 feet (23 to 52 m) AMSL. The surrounding coastal wetlands and marsh otherwise generally range from sea level to just a few feet above sea level, with storm surges periodically inundating much of the Chenier Plain. The steep and nearly circular salt domes rise precipitously above the surrounding terrain, caused by vertical diapiric uplift and blanketed with a thin layer of eolian loess. The co-occurrence of oil and gas fields and minerals in addition to salt is not coincidental, but associated with the geomorphic processes that produced the salt domes. The salt dome islands and adjacent coastal plain are undergoing concomitant processes of erosion, gullying, colluvial deposition and subsidence, as evident in natural and anthropogenic sinkholes. In combination with regional subsidence and relative sea-level rise, the vertical uplift of salt diapirs produces visibly pronounced regional topographic relief (Autin 2002; Stern et al. 2011).

Humans are greatly influenced by, and have increasingly altered, the constituent hydrology and landforms of south Louisiana, a relationship first expressed in the mid-twentieth century through the LSU "manland" school of cultural geography and more recently, geoscience, and geoarchaeology (Mathewson and Shoemaker 2004; see Section 4). In particular, archaeologists have collaborated with geomorphologists in working to understand the complex chronological relationships between shifting human settlements and the deltaic cycle in the Mississippi River delta (Gagliano 1984; Kidder 1996; Kidder et al. 2008; McIntire 1958, 1959; Saucier 1974, 1994a; Törnqvist et al. 1996). The radiocarbon and stratigraphic dating of deltaic lobes has combined and benefitted geomorphic and archaeological studies, with evidence linking initial human occupation, use, and abandonment to the formation, progression, and abandonment of successive deltaic lobes (CEI 1977; Gagliano 1984). An underlying assumption is that changing deltaic environments and biota are directly associated with human settlement patterns and subsistence strategies.

Many of the landforms on which archaeological sites are located, such as natural levees and barrier islands, are destroyed and redeposited by the same riverine and coastal processes that create new deltaic lobes, levees and islands. Gagliano (1984) has shed light on these relationships, including the reworking and redeposition of archaeological materials (such as shell midden) on inconstant landforms such as abandoned natural levees, laterally-migrating barrier islands, and relict shorelines (Gagliano and Kemp 2015; Gagliano and van Beek 1970; Gagliano and Weinstein 1985; Gagliano et al. 1977, 1978, 1981). Subsidence, eustatic sea-level rise and tectonic processes have produced submerged yet formerly terrestrial sites and intermittently inundated "wet sites," many of which contain archaeological deposits also deeply buried by alluvial sediment (Gagliano 1984; Gagliano and Kemp 2015; Gagliano et al. 1982; Pearson et al. 1986; Saltus and Pearson 2010). Consequently, most evidence for human occupations before the Late Archaic (ca. 3000 BCE) and, more likely, the Woodland period (ca. 800 BCE) in the Mississippi River delta is either destroyed or deeply buried, with the notable exceptions of the salt domes to the west and adjoining Pleistocene uplands to the north (Section 3 of this report).

Human activities and anthropogenic changes are continuing to alter and increasingly exacerbate otherwise natural processes in the Mississippi River delta. The subsidence of deltaic landforms due to sediment deposition, compaction, and geologic faults was previously offset by alluvial deposition from periodic flooding, channel shifting and natural levee crevasses (CWPPRA 2017; Gagliano 2005). Constructed levees, stream channelization, and erosion control efforts have reduced the sediment load and transport it to the OCS, effectively starving the wetlands of sediment (Blum and Roberts 2009, 2012). The dredging of canals has increased inland saltwater intrusion and storm surges, while spoil banks impede natural drainages (Theriot 2014). Hydrocarbon extraction, including natural gas, has further contributed to the loss of wetlands through accelerated subsidence and fault reactivation (Morton et al. 2002, 2005, 2006; Walker et al. 1987). At the same time, relative sea-level rise along the Gulf Coast due to global climate change is projected to intensify erosion from tidal action and storm surge (CWPPRA 2017; Penland and Ramsey 1990; Twilley et al. 2001; USACE 2009). The ecological and archaeological effects of an oil spill are yet another variable added to this mix.

#### 2.2 Soils and Ecology

The geomorphology and hydrology of the Mississippi River delta have formed and influenced the soils and ecology of coastal Louisiana over the millennia of the Holocene, within a characteristically humid, subtropical climate since the Hypsithermal interval of the mid-Holocene (Otvos 2004; Rees 2010b:39). The soils of the coastal marshes and delta alluvium are predominantly hyperthermic and aquic. The landforms are mostly level and low-lying, at elevations ranging from below sea level to 5 feet (1.5 m) above mean sea level (AMSL) (Figure 4). The Mississippi Alluvial Plain of south Louisiana extends eastward from Vermilion Bay to the Pearl River mouth north of Lake Borgne. This section of the Gulf Coastal Plain is characterized by saturated and frequently-inundated marsh soils and southern Mississippi River alluvium, bisected by natural levees, bayous, tidal mudflats and numerous embayments. The Western Gulf Coastal Plain between Vermilion Bay and the Sabine Pass is distinguished by the low-lying shoreline, beach ridges and natural levees of the Chenier Plain, apart from the geologically more ancient salt domes of the south-central coast (NRCS 2017; Weindorf 2008). The largest salt dome islands rise more than 150 feet (46 m) above the surrounding marsh and are blanketed with eolian, Quaternary-age loess (Autin 2002).

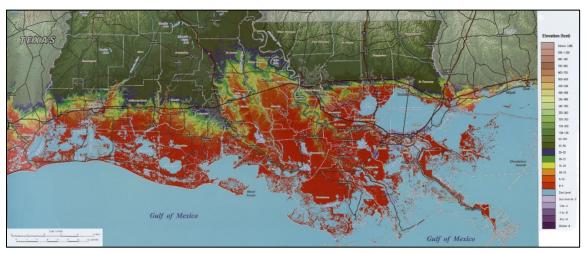


Figure 4. The low-lying elevation of Louisiana's coastal region.

Adapted from Kosovich (2008)

Soils in the study area vary widely, from clayey sediments and organic deposits in coastal marshes to poorly drained clays and silty clays of backswamps and sandy loam on natural levees and point bars.

These soils are comprised of clays, silts and fine sands deposited by the Mississippi River, with most recent Holocene age alluvium near the surface. Gulf Coast marsh soils and southern Mississippi River alluvium comprise two of the Major Land Resource Areas (MLRAs) in Louisiana (Figure 5). The majority of soils in the coastal marshes formed in organic deposits over Mississippi River alluvium. These soils tend to be composed of deep and clayey sediments, including undifferentiated Entisols and Histosols with peaty or highly organic surface layers.

The soils of the Gulf Coast marsh can be classified as Hydraquents and Haplosaprists. Hydraquents formed in clayey sediments; Haplosaprists formed in organic deposits over alluvium. Soils of the Bancker, Creole, Larose, and Scatlake series are typical of Hydraquents (Table 1). Soils of the Allemands, Clovelly, and Lafitte series are characteristic of Haplosaprists. Soils that formed in organic deposits are also present and are characterized by the Kenner and Timbalier series (Weindorf 2008:4). The soil series of the Gulf Coast marsh comprise a majority of study area, including Scatlake and Timbalier muck in eastern Mississippi River delta saltwater marshes. Bancker, Clovelly, and Lafitte muck are associated with brackish areas and Allemands, Kenner, and Larose muck are found in freshwater marshes.

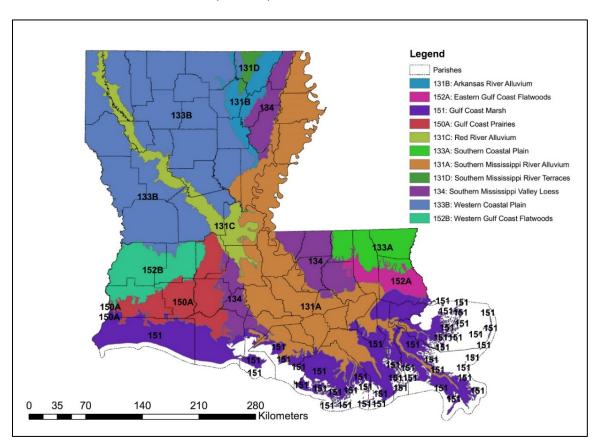


Figure 5. Major land resource areas of Louisiana.

(Weindorf 2008: Figure 6)

Southern Mississippi River alluvium is found on the natural levees, floodplains, terraces, and oxbows of the Mississippi River delta. Soils near the surface were deposited by the Mississippi River over the past few millennia of the Holocene through flooding, river crevasses, lateral channel shifting, and deltaic cycles. In comparison to Gulf Coast marsh soils, the fluvial deposits of the Mississippi River are mostly comprised of different combinations of sand, silt and clay. A majority of landforms are comprised of poorly drained loamy or clayey soils that can be classified as Alfisols, Vertisols, Inceptisols, or Entisols. Soil series of the Southern Mississippi River alluvium include Bruno, Commerce, Convent, Crevasse,

Dowling, Dundee, Robinsonville, Sharkey, Tensas, and Tunica (Table 1). The Dowling, Sharkey, and Tunica series are common throughout the alluvial plains and backswamps of the delta. The Commerce, Convent, and Robinsonville series are associated with natural levees; the Bruno and Crevasse series are found on point bars and levee splays. Soils of the Dundee and Tensas series characterize higher elevations of adjacent Pleistocene terraces (Weindorf 2008:7-8).

Table 1. Soil series classification

Soil Series MLRA*		Landscape	Phase	Classification		
Allemands	Gulf Coast Marsh	freshwater marshes	mucky peat	Clayey, smectitic, euic, hyperthermic Terric Haplosaprists		
Bancker	ker Gulf Coast Marsh brac mars		muck	Very-fine, smectitic, nonacid, hyperthermic Sodic Hydraquents		
Bruno	S. Miss. River Alluvium	floodplains	sandy loam	Sandy, mixed, thermic Typic Udifluvents		
Clovelly	Gulf Coast Marsh	brackish marshes	muck	Clayey, smectitic, euic, hyperthermic Terric Haplosaprists		
Commerce	S. Miss. River Alluvium	alluvial plains	silt loam	Fine-silty, mixed, superactive, nonacid, thermic Fluvaquentic Endoaquepts		
Convent	S. Miss. River Alluvium	floodplains	silt loam	Coarse-silty, mixed, superactive, nonacid, thermic Fluvaquentic Endoaquepts		
Creole	Gulf Coast Marsh	brackish marshes	mucky clay	Fine, smectitic, nonacid, hyperthermic Typic Hydraquents		
Crevasse S. Miss. River Alluvium		floodplains	sand	Mixed, thermic Typic Udipsamments		
		depressions and backswamps	clay	Very-fine, smectitic, nonacid, thermic Vertic Endoaquepts		
Dundee	Oundee S. Miss. River natural levees o low terraces		loam	Fine-silty, mixed, active, thermic Typic Endoaqualfs		
Kenner	Kenner Gulf Coast Marsh fresh marsh		muck	Euic, hyperthermic Fluvaquentic Haplosaprists		
Lafitte	Lafitte Gulf Coast Marsh sa		muck	Euic, hyperthermic Typic Haplosaprists		
		freshwater marshes	muck	Very-fine, smectitic, nonacid, hyperthermic Typic Hydraquents		
Robinsonville	obinsonville S. Miss. River floodplains Alluvium		very fine sandy loam	Coarse-loamy, mixed, superactive, nonacid, thermic Typic Udifluvents		
Scatlake	Gulf Coast Marsh saltwater marshes		peat	Very-fine, smectitic, nonacid, hyperthermic Sodic Hydraquents		
Sharkey	S. Miss. River Alluvium	natural levees & backswamps	clay	Very-fine, smectitic, thermic Chromic Epiaquerts		
Tensas	S. Miss. River low natural levees		silty clay	Fine, smectitic, thermic Chromic Vertic Epiaqualfs		

Soil Series	MLRA*	Landscape	Phase	Classification
Timbalier	Timbalier Gulf Coast Marsh		saltwater muck Euic, hyperthermic Ty marshes	
Tunica	S. Miss. River Alluvium	floodplains	clay	Clayey over loamy, smectitic over mixed, superactive, nonacid, thermic Vertic Epiaquepts

<sup>\*</sup>Refer to map in Figure 5 for Major Land Resource Area. Source: Weindorf (2008: Tables 1 and 2).

The biota of south Louisiana's Gulf Coastal Plain has evolved over millennia in relation to the subtropical climate and distinctive geomorphology, hydrology, and soils of the Mississippi River delta. Though a majority of the study area is classified as Deltaic Coastal Marsh, there is considerable variation between ecotones based on elevation and soils (Figure 6). Natural levee soils of Mississippi River alluvium are better drained than the clays and peat deposits of interdistributary basins. Brackish and saltwater marshes are characterized by vast stands of cordgrass, black needlerush, but coastal saltgrass, the soils of natural levees support bottomland hardwood forests and are well suited to agriculture. Stands of live oak and black mangrove, along with hackberries, palmetto, and prickly pear cactus can be found on the barrier islands, ridges, and natural levees, as well as higher elevations provided by shell midden and earthen mound sites (Daigle et al. 2006).

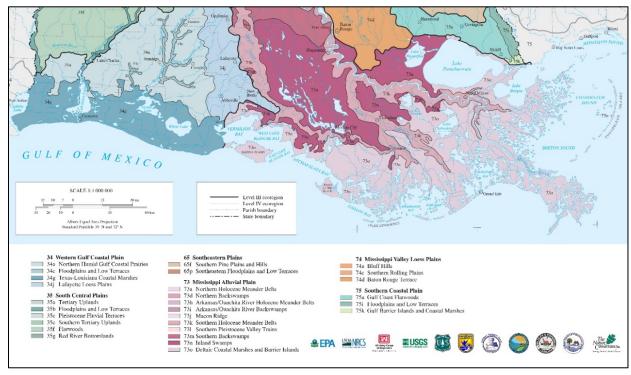


Figure 6. Ecological regions of south Louisiana.

Adapted from Daigle et al. (2006)

The Southern Holocene Meander Belt ecoregion is distinct from the Deltaic Coastal Marsh to the south. It has different species of oak and vast stands of cypress formerly found throughout the hardwood bottomland forests and freshwater swamps. Industrial logging during the late nineteenth and early twentieth centuries transformed the landscape and regional ecology (Mancil 1972). The Texas-Louisiana

Coastal Marsh ecoregion, outside of the study area and west of Vermilion Bay, is predominated by cordgrass in brackish and saltwater areas inland from the shoreline, with maidencane and sawgrass in freshwater marsh. The Chenier Plain is named for the *cheniers* or live oaks that grow on narrow ridges of relict shorelines (Daigle et al. 2006). Live oak and other less saltwater-tolerant species throughout the Deltaic Coastal Marsh have been decimated by coastal erosion and saltwater intrusion, with storm surges and relative sea level rise increasingly causing the inundation of brackish and saltwater marshes.

The marshes and waterways of south Louisiana provide habitat for numerous species of migratory and aquatic birds such as mallard (*Anas platyrhynchos*), wood duck (*Aix sponsa*), Canada geese (*Branta canadensis*), and brown pelican (*Pelecanus occidentalis*), along with bald eagle (*Haliaeetus leucocephalus*) and a profusion of smaller birds. The rivers, bayous, estuaries, and bays support diverse and prolific species of fish, amphibians, and reptiles, including red and black drum (*Sciaenops ocellatus* and *Pogonias cromis*), southern flounder (*Paralichthys lethostigma*), alligator gar (*Lepisosteus spatula*), American bullfrog (*Lithobates catesbeianus*), snapping turtle (*Chelydra serpentine*), cottonmouth snake (*Agkistrodon piscivorus*), and alligator (*Alligator mississippiensis*).

Shrimp, crabs, and bivalves, such as oyster (*Crassostrea virginica*), have been particularly abundant in the Mississippi River delta, making Louisiana a major exporter of shellfish during the twentieth century. Beaver (*Castor Canadensis*), coyote (*Canis latrans*), opossum (*Didelphis virginiana*), and white-tailed deer (*Odocoileus virginianus*) are among the many species of indigenous mammal (Lowery 1974). Historically-invasive species, such as nutria (*Myocastor coypus*) and feral hogs, have disrupted local ecosystems in the delta and coastal marsh. Native species, such as black bear (*Ursus americanus*) and alligator, have at times been driven nearly to extinction due to overhunting. In addition to human predation, the loss of coastal wetlands due to reclamation, dredging, erosion, and subsidence has greatly reduced the habitat of native species, such as black bear and white-tailed deer (Barnes et al. 2015; Brasseaux and Davis 2017; NRC 2006).

### 3. Culture History

The culture history of coastal Louisiana and the Lower Mississippi Valley provides the context for investigating and understanding archaeological sites in the study area. Cultural resource investigations over the past 50 years have greatly expanded existing knowledge of the prehistory of the region. By the 1960s, archaeologists had already established the temporal framework of major cultural traditions (Ford 1951; Haag 1971; Phillips 1970). The culture-historical chronology for Louisiana and the entire southeastern U.S. is conventionally subdivided into a succession of major periods that extend back in time at least 15 millennia before the early historic era (Figure 7). Beginning with the Paleoindian period (ca. 13,000–8,000 BCE), these consist of the more than seven-millennia long Archaic period (ca. 8,000–800 BCE), the considerably shorter, yet two-millennia long Woodland period (ca. 800 BCE–1200 CE), and the comparatively brief Mississippi period (ca. 1200–1700 CE). Each of these periods are subdivided for analytical purposes into sequential early, middle, and late sub-periods, although the temporal boundaries are open to debate and vary among regions and researchers across the Southeast (Anderson and Sassaman 2012; Rees 2010a). These seemingly arbitrary subdivisions of a mostly undocumented past are associated with archaeological phases and components that reflect broad ecological, technological, and cultural changes and trends.

The following overview is drawn from published syntheses, regional reports, and previous investigations in the Lower Mississippi Valley and north-central Gulf Coast (Brown 1984, 1994; Fuller and Wiedenfeld 2015; Jeter and Williams 1989a, 1989b; Hays 1996; Kidder 2002, 2004a; McGimsey 2003a; Mann 2010, 2012; Miller et al. 2000; Neuman 1984a, 1984b; Rees, ed. 2010; Weinstein and Kelley 1992). Because of the aims of the present study, this review of culture history emphasizes current archaeological knowledge of Native American societies and communities living in the Mississippi River delta and adjacent regions during the millennia preceding 1700 CE. The onset of the historic or Colonial-American period (1700 CE–present) along the north-central Gulf Coast begins gradually but is commonly set at 1700 CE as representing a culture-historical benchmark. The commencement of systematic record keeping through written documentation, however, was preceded by nearly two centuries of intermittent and mostly unrecorded contacts between Native Americans, Europeans, and Africans. As the landforms of the delta are of relatively recent Holocene age, the emphasis is on archaeologically known cultures that post-date the Late Archaic period (after ca. 800 CE). Topics of recurring archaeological interest include material culture, such as ceramics, subsistence and settlement patterns, economic interaction or exchange, and social organization.

# 3.1 Paleoindian and Early Archaic Periods (ca. 13,000-6,000 BCE)

The culture history of the north-central GOM commences with the arrival of humans in the coastal zone. Precisely when this occurred is open to debate, but the available evidence indicates humans were in the region by at least 10,800 to 11,000 BCE, or during the Middle Paleoindian period (Rees 2010b). This time frame is now generally associated with the capacious Clovis culture, long believed to be made up of highly-mobile hunter-gatherer societies that populated the continent in close pursuit of Ice Age megafauna. Clovis culture may alternatively represent a widely shared technological adaptation focused on the use of high-quality stone for the production of the eponymous fluted biface (Anderson 2004, Anderson and Gillam 2000). The discovery of sites outside of the Lower Mississippi Valley that pre-date Clovis has pushed back the earliest arrival of people in the continent, to sometime during the Early Paleoindian period (ca. 13,000–11,000 BCE). The archaeological record of pre-Clovis cultures, for which few isolated sites have been systematically investigated, is still not well known (Goodyear 2006).

Although there are no confirmed Early Paleoindian period or pre-Clovis sites on the north-central Gulf Coast, models of early Paleoindian colonization involve migrations southward, along the margins of the Lower Mississippi Valley, or eastward along the Gulf coast (Anderson 1996; Anderson and Gillam 2000;

Anderson and Sassaman 1996). If the coastal plain served as a corridor for initial colonization of the mid-continent during the terminal Pleistocene Epoch, associated sites may have been submerged and possibly destroyed by sea-level rise (Faught 2004; Gagliano et al. 1977, 1982; Pearson et al. 1986; Stright 1986). Paleoindian sites within the alluvial valley would have been obliterated by channel shifting over the millennia or deeply buried beneath Holocene alluvium (Mann 2010:6; Rees 2010b:36).

		Period	Sub-p	oriodo	Cultures	Phases		
		Period			Cultures	West	Central	East
	1700	Historic	Ame Cold		Multicultural	Little Pecan		
	1500		Late Mississippi / Protohistoric		Mississippian		Petite Anse	Delta Natchezan / Bayou Petre
	1200	Mississippi	Middle Mississippi		Plaquemine	Bayou Chene	Burk Hill	Medora / Barataria / Early Bayou Petre
	1000				Transitional C.C.	Holly Beach	Three Bayou	St. Gabriel
<b>↑</b>	1			Coles Creek		Jeff Davis	Morgan	Bayou Ramos
H	700		Late Woodland		Coles Creek	Welsh	White Lake	Bayou Cutler
H	1			Baytown	Coastal Troyville	Roanoke	?	Des Allemands
ŀ	400	Wdld		Daylowii	Coastal Troyville	Rodiloke	r	Grand Bayou
ľ		Woodland	Middle	Marksville	Marksville	Lake Arthur	Veazey	Magnolia / Mandalay
li	0_		Woodland	iviai vəvilid		Lacassine	Jefferson Island	LaBranche
ļ	-		Early Woodland	Tchula	Tchefuncte	Grand Lake	Lafayette	Beau Mire
:ale) — -	800 BCE							Pontchartrain
- (not to scale)	1		Late Archaic	Poverty Point	Poverty Point	?	Rabbit Island	Garcia
ou) —	1700 BCE							Bayou Jasmine
1	3000 BCE					Bayou Blue	Copell	Pearl River
	6000 BCE	Archaic	Middle /	Archaic	Evans Horizon		Banana Bayou	Monte Sano Amite River
  -     →	8000 BCE		Early A	Archaic	San Patrice var. Keithville			St. Helena
	10,800 BCE		Late Pal	eoindian	San Patrice	Strohe	Vatican	Jones Creek
	11,000 BCE	Paleoindian	Middle Pa	Middle Paleoindian			Avery Island	
	13,000 BCE		Early Paleoindian		Pre-Clovis			

Figure 7. Culture historical chronology for south Louisiana.

Major periods, cultures and phases for coastal Louisiana (adapted from Rees 2010a, Fig. 1.3)

Middle Paleoindian period sites have been found on Pleistocene-age and older landforms to the west and north, such as the salt domes on the south-central Louisiana coast, the Natchez Bluffs, and Macon Ridge, a Pleistocene valley train deposit in northeast Louisiana (Gagliano 1967a; Gagliano and Gregory 1965; Hillman 1990; Saucier 1994b). The Avery Island phase on the south-central coast represents the earliest known culture in the study area, including isolated finds of Clovis and Pelican points on Avery Island, Jefferson Island, and Cote Blanche Island (Marckese 1993, 1995; Weinstein and Kelley 1992:30). Investigation of these and other sites has the potential to shed light on Paleoindian lifeways and the colonization of eastern North America (Rees 2010b).

The Late Paleoindian (10,800–8,000 BCE) and Early Archaic (ca. 8,000–6,000 BCE) periods are characterized by a series of cultural changes associated with the end of the last Ice Age, sea-level rise, stream aggradation, and the gradual onset of a more modern climate and biota. Foraging bands of the terminal Pleistocene and early Holocene are thought to have transitioned to more broad-based or logistical subsistence patterns, with seasonally-focused residential mobility (Anderson and Sassaman 2004, 2012:36–65). The Late Paleoindian period on Louisiana's coastal plain is represented by the Strohe, Vatican and Jones Creek sites, for which associated phases have been proposed but are still poorly defined. The St. Helena phase has been assigned to the Early Archaic period, based on investigation of the Hornsby Mound site (16SH21) in St. Helena Parish (Bonnin and Weinstein 1975; Fuller and Wiedenfeld 2015: 3–4, 3–5; Manuel 1979; Weinstein and Kelley 1992:32).

San Patrice culture, with its distinctive projectile points and tools, spans the Late Paleoindian-Early Archaic divide and is the earliest well represented culture in present-day Louisiana (Jennings 2008a, 2008b; Morehead and Lafitte 2014; Rees 2010b). Most of the available data, however, are from sites on the Pleistocene Terrace and piney hills of the Gulf Coastal Plain, north and northwest of Mississippi River delta. Early Archaic foragers are associated with later San Patrice components, for which corner-notched varieties of San Patrice points, such as *var. Keithville*, appear to be contemporaneous with Kirk Corner-Notched points in the interior Southeast. Gagliano (1967b) long ago noted the Early Archaic association of Kirk Serrated points east of the Mississippi River and north of the Pontchartrain-Maurepas Basin. As with San Patrice, preferences for locally available gravel cherts suggest regionally circumscribed movements of hunter-gatherers (Anderson and Smith 2003:365–366; Jennings 2008a). Though the deltaic landforms of the delta post-date the Early Holocene, the salt domes on the south-central coast and submerged levees on the OCS may contain the archaeological record of coastal foragers of the Late Paleoindian and Early Archaic periods (Gagliano 1967a; Pearson et al. 1986, 2014; Rees 2010a; Stright et al. 1999).

# 3.2 Middle and Late Archaic Periods (6000–800 BCE)

The lengthy culture history of the Middle Archaic (6,000–3,000 BCE) and Late Archaic (ca. 3,000–800 BCE) periods is comparatively better known as a result of research by a dedicated group of scholars (Gibson 1994a, 2000, 2007, 2010; Gibson and Carr, ed. 2004; Sassaman 2010; Sassaman and Anderson, ed. 1996; Saunders 2010a; Saunders et al. 1997, 2005). The commencement of the Middle Archaic period has been placed as early as 6,900 BCE for the Southeast as a whole and as late as 4,000 BCE for the Gulf Coastal Plain (Anderson and Sassaman 2012:73; Saunders 2010a; Saunders and Allen 1997; Saunders et al. 2010:13–16). The earlier date reflects the onset of the Hypsithermal, a mid-Holocene climate interval generally characterized by warmer temperatures and greater seasonal fluctuations (Anderson and Sassaman 2012:73; Schuldenrein 1996). The formation of prolific floodplain, backswamp and estuarine environments supported decreased residential mobility, population growth and cultural diversification throughout the Southeast, especially among foragers in the Lower Mississippi Valley who were increasingly focused on riverine resources. Sea-level stabilized by the end of the mid-Holocene, so Middle Archaic and earlier sites on the Gulf Coast that might be intact would be on submerged landforms (Faught 2004; Pearson et al. 2014).

The later date of 4,000 BCE for the proposed beginning of the Middle Archaic relies on the appearance of Evans projectile points and related Evans Horizon at sites in southern Arkansas, northeast Texas and Louisiana (Saunders et al. 2010:16; Saunders 2010a). This is preceded by the appearance of early side-notched and stemmed forms. The Evans point is characterized by a distinctive single set of notches on the blade above the shoulders and stem. The association of Evans with Sinner points, which resemble Kirk Stemmed and Serrated forms, suggests an earlier date of ca. 6,000 BCE for the beginning of the Middle Archaic (Anderson and Smith 2003:261–262, 284). The Middle Archaic association of Evans points is confirmed by their occurrence at earthen mound sites now known to date from as early as 3900 BCE. This has been interpreted as a cultural horizon involving technological developments, earthen mound construction and related social changes (Saunders and Allen 1997; Saunders et al. 2010:16–18).

Increased use of non-local lithics, stone bead production and baked clay objects are also associated with Middle Archaic components and correspond with the beginning of mound construction, after around 3900 BCE. (Saunders et al. 2010:18–19). The earlier construction of shell mounds in Florida and elsewhere in the Southeast, along with the appearance of stemmed projectile points, is consonant with a date of 6,000 BCE for the onset of environmental and cultural changes that came to characterize the Middle Archaic across the Southeast (Anderson and Sassaman 2012:73–79). Foremost among these changes was an increased reliance on aquatic resources and related development of both generalized and logistical subsistence strategies (Saunders et al. 2005). Ensuing restricted mobility is represented by increased numbers of residential sites and cemeteries, as well as sites with mounds (Saunders 2010a:64–65). If coastal foragers of the Middle Archaic developed more restricted residential mobility based on the prolific wetlands and estuarine resources of the Mississippi River delta, their extraction sites have been submerged as the shoreline retreated. Exceptions may be found on mid-Holocene levees, adjoining Pleistocene terraces and mound sites that may have served as nodes of ceremonial, social, and economic interaction.

Middle Archaic mounds on the southern coastal plain include Banana Bayou (16IB24) at Avery Island on the south-central coast, and the Monte Sano (16EBR17) and Hornsby (16SH21) mounds to the east (Brown 1978, 2015:131–138; Gagliano 1967a; Saunders 2010a:65–68). These are the basis for the still poorly defined Banana Bayou, Monte Sano, and Amite River phases (Fuller and Wiedenfeld 2015:3–5; Weinstein and Kelley 1992:30). More recent investigations of the LSU Campus mounds (16EBR6) have provided greater insight into Middle Archaic monument building, with a date for mound construction beginning around 3,900 BCE (Homburg 1992; Mann 2009, 2010, 2012). Investigations of Middle Archaic monumentality are consequently transforming current understanding of the social organization and ceremonialism of foraging bands in the Lower Mississippi Valley (Russo 1996; Saunders 2010b; Sassaman 2010; Sassaman and Heckenberger 2004). In addition to the challenges of understanding the origins of monument building among foraging bands, archaeologists must now come up with explanations for its apparent cessation and eventual reappearance after a thousand years (Saunders et al. 2010:19).

The Late Archaic period (3,000–800 BCE) in the Lower Mississippi Valley is often associated with the renowned yet enigmatic Poverty Point culture and site in northeast Louisiana, although in most respects these appear atypical of Late Archaic domestic economies and cultural practices in the Mississippi River delta and along the north-central Gulf Coast. Mound construction appears to have been abandoned by 2800 BCE and not taken up again for a millennium. Foraging bands maintained diversified subsistence economies focused on fishing, hunting, shellfish, and wild plant food collecting following the close of the Hypsithermal and establishment of a modern climate regime. Regional cultural diversification appears to have continued and even increased, as represented in a proliferation of stemmed and corner-notched projectile point type,s such as Ellis, Gary, Macon, Motley, and Williams. From east to west, coastal societies of the Late Archaic period are known from sites associated with the Pearl River, Copell, and

Bayou Blue phases. Sites associated with the Pearl River phase provide among the earliest and best-known evidence for human habitation in the delta (Kelley et al. 2008:16; Weinstein and Kelley 1992:33).

Poverty Point culture, which dates as early as 1,700 BCE, is in many respects distinct from the lifeways of most Late Archaic peoples on the Gulf Coast (Greenlee 2014:5–9; McGimsey 2006:13; Mann 2012:6–7; Saunders et al. 2010:20–26). The enormous earthen mounds and ridges of the Poverty Point site in northeast Louisiana were constructed in a relatively rapid series of events, by hunter-fisher-gatherers who harvested the wild plants and animals of the Lower Mississippi Valley (Gibson 2000, 2007; Ortmann 2010; Ortmann and Kidder 2010a, 2013). Coastal groups in the Mississippi River delta were invariably drawn into its social, economic and religious orbit (Gibson 2010; Spivey et al. 2015). The long-distance movement of raw materials such as exotic stone was part of this network, which included steatite vessels, hematite plummets and small amounts of early fiber-tempered and untempered pottery. In addition to projectile point types such as Delhi, Epps, Motley, and Pontchartrain, the earthen cooking balls known as Poverty Point Objects are diagnostic of far-flung Poverty Point components (Ford and Webb 1956; Gibson 1994b, 1994c; Saunders et al. 2010:22–26; Webb 1982).

Sites around Lake Pontchartrain are the basis of the Bayou Jasmine and Garcia phases, sequentially affiliated with Poverty Point, along with the related Claiborne ceremonial site on the Pearl River to the east (Blitz and Mann 2000:19-22; Gagliano and Webb 1970). The contemporaneous Rabbit Island Phase has been identified at sites along the Atchafalaya and Bayou Teche to the west (Kelley et al. 2008:17; Weinstein and Kelley 1992:34). Resource extraction sites in the Mississippi River delta would have been situated on natural levees and in strategic locations for harvesting fish and shellfish, although a majority of the recorded Late Archaic and Poverty Point related sites in southeast Louisiana are located on upland terraces adjoining the delta (Mann 2010:7). Late Archaic foragers living along the coast might have transported local resources, such as shell, to Poverty Point. Though the return voyage might have taken less than a week by canoe, the evidence from coastal sites for links with Poverty Point is less obvious. As found at the Bayou Jasmine site (16SJB2), earlier Poverty Point components in the delta were most likely deeply buried by alluvial deposition, or destroyed by channel shifting and crevasse splays (Hays and Weinstein 1999; Mann 2012:6). The end of the Late Archaic period, preceded by the cessation of Poverty Point exchange, has been associated with subsequent cultural developments and climate changes that characterize the Woodland period in the Lower Mississippi Valley and Southeast (Anderson and Sassaman 2012:107–111; Kidder 2006, 2010b).

# 3.3 Early and Middle Woodland Periods (800 BCE-400 CE)

The production and use of ceramic containers, once thought to be a cultural development signifying the commencement of the Woodland period in the Southeast, is now known to have begun much earlier (Sassaman 2004). Some of the earliest ceramics in the Lower Mississippi Valley have been found at Poverty Point, including imported St. Johns varieties (Hays and Weinstein 2004). The widespread occurrence of locally made ceramics in the Valley does not occur until centuries later, with the advent of Tchefuncte culture of the Early Woodland period (800 BCE–1 CE). Early Woodland culture of the southern Valley and Mississippi River delta is assigned to the Tchula period. In a more obvious break with Poverty Point and the Late Archaic, the acquisition of exotic items, such as non-local lithics, substantially declines and remains negligible throughout the Tchula period (Hays and Weinstein 2010). The subsequent culture historical chronology of the entire Woodland period in the Valley was assembled based largely on successive ceramic types and traditions (Brown 1998; Phillips 1970). Following the Tchefuncte culture of the Tchula period, these include the Marksville culture and period (1–400 CE), Troyville culture of the Baytown period (400–700 CE), and the Coles Creek culture and period (700–1200 CE).

Tchefuncte is the earliest well-represented culture in the Mississippi River delta, with numerous components recorded around the Pontchartrain-Maurepas Basin and landforms of older or contemporaneous age, including the St. Bernard subdelta (McIntire 1958; Mann 2010:8; Neuman 1977a; Törnqvist et al. 1996; Weinstein 1986; Weinstein and Rivet 1978). Tchefuncte culture was previously thought to date from 500 BCE. Radiocarbon dates from Bayou Jasmine and other sites have pushed the beginning of Tchefuncte back to 800 BCE and perhaps as early as 1200 BCE (Hays 1999, 2000; Hays and Weinstein 1999; Kidder 2004a). The far-flung networks of Poverty Point culture were severed during this interval, possibly associated with a climate-induced regional dispersion of populations (Kidder 2006). The resulting resettlement has been linked to the development of subsequent distinctions in material culture, most evident in ceramic traditions across the Southeast, and increased cultural diversity characterized as the development Woodland regionalism (Anderson and Sassaman 2012:107–111, 115–116). Tchefuncte ceramics are known for characteristic incised, rocker stamped, and punctated decorations on otherwise poorly formed and soft, mostly untempered clay paste (Ford and Quimby 1945; Hays 2010:8; Hays and Weinstein 1999, 2004, 2010; Mann 2010:8; Weinstein 1986; Weinstein and Rivet 1978).

As a regional expression of Early Woodland culture, hunter-fisher-gatherers in the Lower Mississippi Valley and Mississippi River delta continued the diversification and intensification of subsistence patterns, with increased harvesting of riverine, lacustrine, and coastal resources (Byrd 1994). Thick shell middens, often containing the preserved bones of aquatic fauna, are associated with Tchefuncte components at the Bayou Jasmine (16SJB2) and Tchefuncte (16ST1) sites (Heller et al. 2013:566–589). Contemporaneous Woodland societies farther upriver and in the interior Southeast developed horticultural economies based on chenopod (*Chenopodium berlandieri*), squash (*Cucurbita pepo*) and other cultigens domesticated during the Late Archaic (Gremillion 2004; Smith 2006; Smith and Yarnell 2009). There is no conclusive evidence for domesticates in Tchefuncte components in the delta (Byrd and Neuman 1978:11–13; Fritz and Kidder 1993:6–7). Residents instead relied on an abundance of wild plant foods and fauna, fishing, and harvesting copious amounts of brackish-water clams (Kidder 2004a:547). Mann (2010:8) consequently characterized coastal Tchefuncte as a "distinctive marine and aquatic adaptation."

Early Woodland monumentality is thought to have found expression at some Tchefuncte sites in the construction of low, dome-shaped earthen mounds. Though Early Woodland period mounds are known for contemporaneous components in the Lower Mississippi Valley to the north, the construction of mounds by Tchefuncte peoples has been the subject of debate (Jeter and Williams 1989a; Neuman 1984:134–135; Weinstein 1986). Ford and Quimby (1945) provided early evidence for Tchefuncte mound construction at the Lafayette Mounds site (16SM17). Recent reanalysis has confirmed the Tchula period of construction and association with Tchefuncte ceramics (Hays and Weinstein 2010:107–109; Heller et al. 2013:622–625). Though the few confirmed Tchefuncte mounds appear to represent the development of communal mortuary ceremonialism, most known Tchefuncte burials are shallow pits excavated into midden and contain no preserved grave goods (Hays and Weinstein 2010:109).

That Tchefuncte components are more numerous in the Mississippi River delta and coastal region than components from preceding periods may be due to their greater visibility and preservation on terrace edges and natural levees along bayous, lakeshores, and backwater swamps. Deltaic progression and channel shifting transformed regional ecology, providing a diversity of resources while at the same time obliterating or burying evidence for human habitation. In addition to distinctive types of pottery, such as Orleans Punctated, *var. Orleans*, Tchefuncte Incised, *var. Tchefuncte*, and Tchefuncte Stamped, *var. Tchefuncte*, Tchula period components are associated with tubular ceramic pipes, a variety of antler and bone tools, such as fishhooks, and projectile point types that persist from the Late Archaic, including Ellis, Gary, Pontchartrain, and Kent (Hays and Weinstein 2010; Kidder 2004a:545–548; Lewis 1997). Lithic artifacts were predominantly made of gravel cherts available in the drainages of Pleistocene terraces bordering the delta to the north and northwest.

Tchefuncte components in the Mississippi River delta are distinguished by the Pontchartrain phase around the Pontchartrain-Maurepas Basin and the subsequent Beau Mire phase to the west (Heller et al. 2013:24; Weinstein 1986; Weinstein and Kelley 1992:34–35; Weinstein and Rivet 1978). Four sequential phases have more recently been proposed for Tchefuncte components around Lake Pontchartrain: the Maurepas (ca. 800–600 BCE), Pontchartrain (ca. 600–400 BCE), Oak Island (ca. 400–200 BCE), and Sauvage (ca. 200–100 BCE) phases (Heller et al. 2013:590–622). The Lafayette phase characterizes Tchula period sites to the west, from the Atchafalaya Bay northward along the Pleistocene Terrace and Bayou Vermilion. The Grand Lake phase of Tchefuncte culture represents contemporaneous components along the Chenier Plain to the southwest (Hays and Weinstein 2010). Sites such as Big Oak Island (16OR6), on the south shore of Lake Pontchartrain, and Morton Shell Mound (16IB3), on Weeks Island to the west, have produced evidence for the intensive harvesting of the brackish-water shellfish *Rangia cuneata* and other aquatic resources (Byrd 1994; McGimsey 2003a; Neuman 1972; Shenkel 1974, 1984). Stable and predictable resources may have afforded coastal Tchefuncte populations with a sufficient subsistence base for steady population increases and nearly year-round residential sedentism.

The Middle Woodland period (1–400 CE) in the Lower Mississippi Valley is generally associated with the Marksville period and culture (McGimsey 2010). Although the partitioning of the Early and Middle Woodland periods is in some respects rather arbitrary, distinctive ceramic types with characteristic incising, stamping, and punctuating distinguish Marksville culture from the preceding Tchefuncte culture (Toth 1974). These include well-defined zones of punctated or stamped decorations surrounded by broad incised lines. The Middle Woodland period has been further subdivided into early and late sub-periods, with the late Middle Woodland Issaquena culture found at sites in the lower Yazoo Basin and north of Baton Rouge (Jeter and Williams 1989a:134–141; Kidder 2004a:548). Early and late Marksville phases have been defined for three regions of coastal Louisiana and are distinguished based on ceramic types and varieties. From the eastern Mississippi River delta moving westward, the early Marksville phases are LaBranche, Jefferson Island, and Lacassine. These are followed in succession, respectively, by the Magnolia and Mandalay phases, the Veazey phase, and the Lake Arthur phase (Bonnin and Weinstein 1975; Fuller and Wiedenfeld 2015:3–8; Jeter et al. 1989a:139; Kelley et al. 2008:18–19; Phillips 1970:898; Toth 1988; Weinstein and Kelley 1992:35).

The production of more refined grog tempered wares is one of the defining criteria for the later phases during the final two centuries of the Marksville period. In addition to early types such as Mabin Stamped, *var. Crooks*, later Marksville types include Churupa Punctated, *var. Thornton*; Marksville Incised, *var. Yokena*; and Marksville Stamped, *vars. Manny and Troyville* (Brown 1998:52, 58–59; Fuller and Wiedenfeld 2015:3–8, 3–9). Some archaeologists have framed these distinctions in terms of a late Marksville Issaquena culture or phase and a Coastal Marksville culture (Jeter and Williams 1989a:138–139). Based on a reanalysis of ceramics from the Morton Shell Mound, McGimsey (2003a:178–183; 2010:132) suggests the distinction between early and late designs is more problematic in the Mississippi River delta and along the coast, as both early and late Marksville ceramic types are found in Baytown period contexts centuries after the end of the Marksville period.

Marksville culture is also associated with the type site of the same name, on the Pleistocene bluff overlooking the Mississippi River floodplain in present-day Avoyelles Parish (McGimsey 2010). Once thought to be an outpost or site unit intrusion of the Middle Woodland Hopewell culture in the Midwest, the Marksville site is now understood to be a Hopewellian-influenced ceremonial precinct, with six earthen mounds enclosed within a C-shaped earthen embankment (McGimsey 2003b, 2010:121–124; Toth 1979, 1988). Middle Woodland foragers living along the coast may have had only tenuous connections to the Hopewellian-inspired ritual landscape of Marksville, but they shared a distinctive pottery tradition and mortuary ceremonialism involving the construction of low, dome-shaped mounds as sepulchers. The resumption of long-distance exchange, virtually absent during the Tchula period, is

evident from Marksville period mounds and burials that contain small amounts of exotic materials, such as copper, galena, and non-local stone (McGimsey 2010; McGimsey et al. 2000, 2005).

Major Marksville sites in coastal Louisiana include Big Oak Island (16OR6) and Veazey (16VM7/8), a multi-mound site on the Chenier Plain south of White Lake. The shell midden at Big Oak Island dates from the preceding Tchula period, but contains Marksville period burials with grave goods, including at least one copper bead. One of the 14 mounds at the Veazey site contained burials with copper and galena artifacts (McGimsey 2010:124–126). The mortuary ceremonialism and evidence for long-distance exchange at some Marksville sites suggests a multi-village or tribal organization of foraging societies influenced by Hopewellian culture of the Ohio Valley. The majority of Marksville components on the coast, however, do not have burial mounds or sufficient non-local artifacts to suggest a significant amount of long-distance trade. Lithics are predominantly made of local gravel cherts (McGimsey 2010).

Ostensibly more detached from Hopewellian ideas and influence than interior Marksville groups upriver, coastal Marksville foragers in the Mississippi River delta continued to pursue seasonally-based residential sedentism through fishing, hunting, shellfish harvesting and the gathering of wild plant foods. Unlike contemporaneous horticultural subsistence economies to the north, the dietary importance of squash (*Cucurbita pepo*), chenopod (*Chenopodium berlandieri*) and other domesticates has not been established for the Middle Woodland period along the north-central Gulf Coast. The majority of subsistence information on Marksville components comes from sites upriver, in the Tensas Basin, and at the Marksville site. Acorn and hickory were important, along with persimmon, grape, and chenopod. The localized development of Marksville culture through interaction with groups to the north would account for the regional variability expressed in its different phases (Kidder 2004a:549, 551). Under a thin veneer of Hopewellian-inspired artifact styles and mortuary ceremonialism, the subsistence patterns of coastal foragers sustained self-sufficient and relatively autonomous domestic economies and social organization that appear to have continued relatively unchanged from the Early Woodland through the Middle Woodland period.

# 3.4 Late Woodland and Mississippi Periods (400-1700 CE)

The first three centuries of the Late Woodland period (400–1200 CE) in the Lower Mississippi Valley are associated with the Baytown period (400–700 CE). The Baytown period in the lower Yazoo Basin and northern Valley was initially characterized by the waning of long-distance exchange and production of more utilitarian pottery; a time of seemingly "good gray cultures" following the Hopewellian-inspired Marksville culture (Phillips 1970; Williams 1963:297; Williams and Brain 1983:403–404). The Baytown period in the Mississippi River delta and Valley south of the Yazoo Basin is characterized by Troyville culture and its coastal variant, and Baytown culture is distinguished at sites to the north (Jeter and Williams 1989a:141–156). The defining elements of Baytown and Troyville, as well as the Middle Woodland to Late Woodland transition, have since been problematized by the addition of new data and refinement of regional ceramic chronologies (Belmont 1984; Gibson 1984; Lee 2010; McGimsey 2003a:178–183).

James Ford (1951:13) originally coined Troyville culture after the type site (16CT7) on the Black River, but cautioned that it represented an arbitrary subdivision of Marksville and the later Coles Creek culture and period (700–1200 CE). As it turns out, much of the earthen mound construction and residential deposits at the Troyville site appear to date from the end of the Baytown period (Lee 2010; Lee et al. 2011; Walker 1936). Furthermore, Marksville pottery types are found in Baytown period contexts and continue to be produced and used well after 400 CE, with some types dating as late as 800 CE. Adding to the conundrum, some Troyville types, such as Troyville Stamped, *vars. Troyville* and *Elm Ridge*, are found in Marksville contexts, predating the Baytown period (McGimsey 2003a:178–183; 2010:132). Perhaps even more so than previous cultural periods, the partitioning of the Middle and Late Woodland

now seems to be an arbitrary archaeological classification that persists out of convention and convenience.

The Baytown period and Troyville culture are consequently viewed as transitional and somewhat poorly defined, especially in the Mississippi River delta and along the north-central Gulf Coast. Baytown period components in the region have been described as "Troyville-like" or by the designation "coastal Troyville" (Jeter and Williams 1989a:152–156), referring to differences in ceramic assemblages from sites in the Lower Mississippi Valley to the north. In comparison to the preceding Marksville period, Baytown period components are more numerous in the delta and are usually found in association with Coles Creek sites (Neuman 1977:19-29). Coastal Troyville components are nevertheless the least well known of Baytown period sites in the Valley, perhaps owing to the difficulties of distinguishing it from earlier and later components (McGimsey 2003a:178–183). Following Ford (1951), a coastal Troyville-Coles Creek cultural continuum has been proposed in order to emphasize this continuity, at least in terms of ceramics (Jeter and Williams 1989a:152–153).

Along with Troyville Stamped, *vars. Troyville* and *Elm Ridge*, and later varieties of Marksville Incised and Marksville Stamped, Baytown period components are distinguished by the introduction of the red-filmed ware, Larto Red. The Grand Bayou and Des Allemands phases have been defined for southeastern Louisiana, with the latter phase (Des Allemands) apparently distinguished by an absence of later Marksville types and the introduction of incised wares, such as Coles Creek Incised, French Fork Incised, and Mazique Incised, *var. Bruly*. Baytown period components in coastal southwestern Louisiana are associated with the Roanoke phase (Fuller and Wiedenfeld 2015:3–9; Kelley et al. 2008:20). Pontchartrain Check Stamped, including *var. Canefield*, is introduced as early as the Marksville period. Pontchartrain Check Stamped, *var. Pontchartrain*, is found at coastal sites throughout the Baytown and Coles Creek periods, becoming particularly common in Coles Creek contexts (Brown 1982a, 1982b, 1984:115, 1998:63–64).

Native Americans in the Lower Mississippi Valley and coastal Louisiana were using the bow and arrow by the end of the Baytown period, as indicated by the appearance of small projectile point types, such as Alba and Scallorn (Webb 2000:14–16). This technological change had potentially far-reaching economic and political impacts beyond its implications for hunter-gatherer subsistence. In the rock-deficient environment of the Mississippi River delta, coastal foragers maintained toolkits heavily reliant on the modification of bone, as well as antler and wood (Davis et al. 1983; Kidder and Barondess 1982). Lithic sources remain mostly gravel cherts from Pleistocene landforms near the Valley. Deer, small mammals, amphibians and reptiles were hunted and trapped on the natural levees, along lakeshores and coastal marshes, and the cheniers of southwest Louisiana's coast. Subsistence patterns otherwise continued to be oriented around the seasonal exploitation of aquatic resources, including backwater and estuarine fish and migratory waterfowl, with possible year-round harvesting of shellfish (Jeter and Williams 1989a:155).

Ceramic types and varieties are the principal means of distinguishing the Coles Creek period (ca. 700-1200 CE) and its various phases, including Avoyelles Punctated, *var. Avoyelles*; Beldeau Incised, *var. Beldeau*; Coles Creek Incised, *vars. Athanasio, Coles Creek, Hardy*, and *Mott*; Evansville Punctated, *var. Wilkinson*; French Fork Incised, *vars. French Fork, Iberville*, and *Laborde*; Harrison Bayou Incised, *var. Harrison Bayou*; Mazique Incised, *vars. Kings Point, Manchac, and Mazique*; and Pontchartrain Check Stamped, *vars. Pontchartrain* and *Tiger Island* (Brown 1998; Weinstein and Kelley 1992:37). As with the preceding Troyville culture, differences among Coles Creek ceramic assemblages from sites in the Mississippi River delta south of Baton Rouge and the Lower Mississippi Valley to the north have led archaeologists to distinguish a coastal Coles Creek culture (Brown 1984; Jeter and Williams 1989a). Stylistic similarities with Weeden Island culture to the east have been noted, but not systematically examined (Brown 1984, 2004:578–580; Weinstein and Kelley 1992:37). Pontchartrain Check Stamped is an especially common decorative treatment in Coles Creek components, lending further credence to distinguishing a coastal Coles Creek culture from the Coles Creek of the interior Valley (Brown

1984:115). Coles Creek components in the delta are classified by the successive Bayou Cutler, Bayou Ramos and St. Gabriel phases. The sequential White Lake, Morgan, and Three Bayou phases have been defined for the south-central coast, while Coles Creek components in southwest Louisiana are represented by the Welsh, Jeff Davis, and Holly Beach phases (Brown 1984:97–99; Kelley et al. 2008:20–21; Weinstein and Kelley 1992:31, 37).

One of the more pronounced changes from the Middle Woodland period is the establishment of a hierarchical and demographically-nucleated settlement pattern, probably dating from the late Baytown to early Coles Creek period. This is indicated by the proliferation of ceremonial sites with platform mounds and plazas of varying sizes (Kidder 1992a, 1998, 2004b; Roe and Schilling 2010). Implicating population movements, Gibson (1996:58–59) described this demographic transformation in northeastern Louisiana as incipient urbanism. Ostensibly beginning with Troyville, these changes appear to date from the last century of the Baytown period and to continue uninterrupted into the Coles Creek period (Lee 2010; Rees and Lee 2015).

The Greenhouse site (16AV2) near Marksville dates from this transitional time and has informed current understanding of Coles Creek social organization and ceremonialism in the Lower Mississippi Valley (Ford 1951; Kidder 1998, 2002). There is evidence for communal and even elite sponsored feasts in both mound and non-mound contexts by the late Baytown period. Elongated, "bathtub-shaped" pits have been excavated at Baytown period mound sites and are thought to have been used for cooking large quantities of food at communal feasts (Brown 1984; Kidder 2004a:554). Though mounds continue to serve mortuary functions, the appropriation of formerly communal space for elite residences and ceremonies is a hallmark of Coles Creek culture (Kidder 1998). With the beginning of the Coles Creek period, access to earthen platforms becomes more restricted and associated with the residential compounds of higher ranked lineages or kin groups (Roe and Schilling 2010). The construction of conical mounds with associated mortuary ceremonialism continues, but the transformation of mound precincts from communal to private, privileged space appears to have occurred by the beginning of the Coles Creek period (Kidder 2002, 2004a:554; Rees and Lee 2015).

The population of the Mississippi River delta is thought to have increased during the Coles Creek period, as seen in the large numbers of Coles Creek sites with and without earthen mounds and shell midden (McIntire 1958; Neuman 1977a:19–29). Preceding Marksville and early Baytown period components are comparatively less well represented. This development might be ascribed to large-scale population movement, but without evidence for migrations into the region it more plausibly represents a sampling bias related to dynamic deltaic landforms. The profusion of late Baytown and Coles Creek components in the Mississippi River delta is directly linked to landforms of older or contemporaneous age. The large number of Coles Creek components in the Terrebonne Basin and along Bayou Lafourche, for example, can be attributed to the formation of the Lafourche subdelta during the Baytown period (Törnqvist et al. 1996:1695). Natural levees would have been unavailable for previous inhabitation, but river crevasses, channel shifting and deltaic progression probably obliterated the ephemeral archaeological record of earlier subsistence forays by coastal foragers.

The proliferation of Coles Creek components in the Mississippi River delta and throughout the north-central Gulf Coast is nonetheless almost certainly indicative of larger regional populations between 700 and 1200 CE. Major Coles Creek sites in the delta and along the coast include the Bayou Grande Cheniere Mounds (16PL159) in the marsh west of the Mississippi River, the Gibson Mounds (16TR5) on Bayou Black, and the Morgan Mounds (16VM9) on Pecan Island (Brown 1984; Fuller and Fuller 1987; Mann 2008; Schilling 2004, 2009). If comparisons with contemporaneous groups upriver are valid, coastal Coles Creek culture may have been made up ofnumerous small and medium-sized political-religious centers that served as administrative and ceremonial nodes for surrounding communities. Because evidence for hereditary social ranking is lacking in mortuary treatment, large mound sites such as

Bayou Grande Cheniere provide the principal evidence for hierarchy and political-religious centralization (Schilling 2009).

Early Coles Creek populations appear to have established sedentary communities in resource rich environments of the Lower Mississippi Valley without the benefit or hindrance of agriculture (Kidder 2004a:553). As during the Baytown period, there is little evidence for domesticates, long-distance trade or extra-regional connections. Instead, regionally autonomous polities may have increasingly vied for control of local territories and resources (Wells 1997). There is little evidence for warfare at sites in the Valley or Mississippi River delta, but the potential for conflicts would have increased among competing polities not engaged in trade. The localized subsistence economies of fisher-hunter-gatherers might have posed fundamental constraints to additional political and religious centralization. In contrast to the large Coles Creek mound and plaza precincts found in the Valley north of the Red River confluence, the evidence for political-religious hierarchy and consolidation of centralized polities is less pronounced in the delta and coastal region (Kidder 2004b). Along with the aforementioned differences in ceramic assemblages, this offers further support for regional variation in historical trajectories.

The final two centuries of the Coles Creek period have been characterized as "Transitional Coles Creek" and implicated with emergent Mississippian upriver in the Lower Mississippi Valley and interior Southeast (Jeter and Williams 1989a:156–159; Weinstein 1987). The designation of emergent Mississippian is somewhat problematic, however, in the Valley and along the north-central Gulf Coast (Kidder 1998). Coles Creek culture south of the Natchez Bluffs appears to seamlessly transition into Plaquemine culture, rather than Mississippian culture, by the beginning of the Mississippi period (1200–1700 CE). In the Central Mississippi Valley and interior Southeast, the Mississippi period begins two centuries earlier and is associated with the advent of Mississippian culture (Rees and Livingood 2007). Mississippian communities were demographically nucleated and organized around major political-religious centers that relied on the food surpluses provided by intensive maize agriculture, with hereditary social inequalities, distinctive types of shell-tempered ceramics, and long-distance exchange in exotic raw materials. Though Cahokia in the American Bottom of the Mississippi Valley was the largest and earliest of the Mississippian centers, its massive size and far-reaching influence make it far from typical (Pauketat 2004).

The beginning of the Mississippi period in Mississippi River delta is associated with small and mediumsized ceremonial centers, as represented by mound and plaza complexes with Plaquemine artifact assemblages, often in association with earlier Coles Creek components. Plaquemine components are typically indicated by the presence of Plaquemine Brushed, var. Plaquemine, in ceramic assemblages, along with lesser amounts of Anna Incised, vars. Anna, Australia, and Evangeline, Carter Engraved, L'eau Noire Incised and Maddox Engraved. Other ceramic types and varieties persist from Transitional Coles Creek into the Mississippi period. For example, Evansville Punctated, var. Wilkinson, and Mazique Incised, var. Manchac, are associated with both Transitional Coles Creek and Plaguemine components (Brown 1998:54; Weinstein 1987:96; Weinstein and Kelley 1992:37). Pontchartrain Check Stamped, common in Coles Creek components, also persists in Plaquemine assemblages at sites along the coast (Brown 2015:274; Giardino 1990). The Barataria phase has been identified for Plaquemine components around the Barataria Basin, and the contemporaneous Medora phase is associated with Plaquemine components upriver, around present-day Baton Rouge (Duhe 1981; Holley and DeMarcay 1977; Jeter and Williams 1989b; Phillips 1970; Quimby 1951). The Burk Hill phase is found along the central coast, in the Petite Anse region around Cote Blanche and Avery Island, and the Bayou Chene phase is represented by Plaquemine components further west along the coast (Brown 1984; Weinstein 1987).

There is little initial evidence for external contacts or long-distance exchange in exotic raw materials during the Mississippi period in the Mississippi River delta. Along with the scarcity of non-local goods, continuities in ceramic assemblages and site habitations suggest Plaquemine culture in the delta represents an indigenous development of Coles Creek (Rees and Livingood 2007). In contrast, late Coles

Creek period components in the Lower Mississippi Valley to the north, notably in the lower Yazoo Basin and northeast Louisiana, exhibit earlier evidence of external contacts with Mississippian communities. Archaeologists consequently described Plaquemine in these regions as a cultural hybridization of Coles Creek and Mississippian (Brain 1989; Williams and Brian 1983). The occurrence of extra-regional, Mississippian shell-tempered ceramics, possibly as trade goods, may represent direct or indirect contacts with Mississippian emissaries and ideas during the Transitional Coles Creek period in the Valley north of the Red River confluence (Wells and Weinstein 2007). A comparable scenario does not appear to have occurred in the delta until at least a century and a half later (Rees 2010c; Weinstein and Dumas 2008).

Following the Transitional Coles Creek St. Gabriel phase, the Bayou Petre phase marks the beginning of the Mississippi period in the eastern Mississippi River delta. Bayou Petre was initially defined based on ceramic assemblages from sites in the delta that exhibited similarities with assemblages from Mississippian components to the east, including the use of shell temper (Kniffen 1936; Knight 1984). The presence of D'Olive Incised, Moundville Incised, Mound Place Incised, and other types associated with Pensacola culture have been interpreted as evidence of expanding Mississippian influence along the coast, the diffusion of ceramic technology or ideas, exchange networks, alliances and intermarriages. The likely source of these stylistic and technological changes in ceramics has more recently been tied to the Mobile Bay area and the major Mississippian site of Bottle Creek (Brown 2004; Rees 2010c; Weinstein and Dumas 2008).

Beginning around 1400 CE, ceramic types associated with Mississippian components in the Yazoo Basin and northern Lower Mississippi Valley are also found at sites in the Mississippi River delta. These include Barton Incised, Cracker Road Incised, Owens Punctated, Walls Engraved, and Winterville Incised. Sims Place (16SC2) is one of the best known examples of a mound site in the delta with an initial Plaquemine component that also contains Mississippian ceramic types from the Valley to the north and Pensacola culture to the east (Fuller 1998, 2003; Rodning and Mehta 2017; Weinstein and Dumas 2008:212). Along with an influx of Mississippian ceramic types, there is increased evidence for interregional contacts along the coast between the thirteenth and sixteenth centuries. Carved stone discs and Mississippian iconography on ceramics have been found at sites associated with the Barataria and Medora phases (Weinstein 1987). The use of shell temper in ceramic production appears in some instances to have been adopted by Plaquemine communities in the delta, and even combined with grog and other tempering agents (Livingood 2007; Shuman 2007). Components of the Barataria phase exhibit small amounts of shell-tempered wares and types normally attributed to Mississippian culture. Consequently, there appears to be little direct correlation between shell temper and Mississippian culture, but instead considerable variation and a mixture of pottery traditions across the delta. This is what might be expected from the movement of not only pottery, but potters and their ideas about making pots.

If communities in the Mississippi River delta were relatively late in adopting the ceramics and interregional exchange networks characteristic of Mississippian culture, the reasons may be related to changes in subsistence practices. In contrast with much of the Southeast, the adoption of agriculture occurs later and remains relatively less important in the Lower Mississippi Valley, especially south of the Red River confluence. The cultivation of maize does not appear to have occurred until the last century of the Coles Creek period and even then, it was not uniformly practiced by hunter-fisher-gatherers throughout the Lower Mississippi Valley (Davis 1987; Kidder 1992b; Kidder and Fritz 1993; Listi 2007, 2010). Maize fragments from a feature at the Bayou Grande Cheniere site produced a radiocarbon date of 720 YBP (cal. 1250–1300 CE), considerably later than the Coles Creek mound construction (Mann 2008:34). There is otherwise sparse evidence for the cultivation of maize in the delta and along the coast. The timing of the adoption and relative dietary importance of maize agriculture in the delta remain important avenues for understanding a wider range of cultural changes during the Late Woodland to Mississippian transition. Though natural levees and terraces were well suited for agriculture, Plaquemine societies were proficient and complex foragers of the backswamps, fresh and saltwater marshes, bays, and

estuaries. Locally autonomous subsistence economies founded on prolific hunting, fishing, and gathering appear to have selectively incorporated the agricultural practices pursued by Mississippian societies in the Central Mississippi Valley and elsewhere in the interior Southeast.

In addition to the Bayou Petre phase in the eastern Mississippi River delta and the Bayou Chene phase of Plaquemine culture to the west, archaeologists have identified three final phases for the Mississippi River delta and south Louisiana coast beginning in the sixteenth century. These are the Delta Natchezan, Petite Anse, and Little Pecan phases. Plaquemine components representing the Delta Natchezan phase are found throughout the western delta south of the Natchez Bluffs. Originally associated with the historic Natchez. the Delta Natchezan phase is known from sites such as Bayou Goula (16IV11: Ouimby 1957). Early historic descriptions of the Natchez tribe, including the uses of residential and temple mounds, are consonant with the evidence from Plaquemine and even some earlier Coles Creek mound sites, prompting the use of ethnographic analogies (Brown 2007; Lorenz 1997; Neitzel 1983). The Delta Natchezan phase follows the Barataria and Medora phases of Plaquemine culture and extends westward from the Barataria Basin to Atchafalaya Bay, encompassing the homeland of the Chitimacha Tribe of Louisiana (Davis 1984; Giardino 1984). Diagnostic ceramic types of the Delta Natchezan phase include Fatherland Incised, vars. Fatherland and Bayou Goula, along with Plaquemine Brushed and grog tempered ceramics with small amounts of shell temper (Phillips 1970; Jeter et al. 1989b; Quimby 1957; Weinstein 1987; Weinstein and Dumas 2008). The contemporaneous late Bayou Petre phase is found as late as 1700 CE at sites in the delta to the east.

The Petite Anse phase has been identified at Avery Island, west of Delta Natchezan phase sites (Brown 1978, 2015). The Petite Anse phase was defined by shell-tempered ceramic types at Salt Mine Valley, such as Owens Punctated and Parkin Punctated. The assemblages resemble those of Mississippian components in the Lower Mississippi Valley to the north, leading Brown (1981, 1999, 2015) to suggest that Mississippians may have traveled south to Avery Island in the sixteenth century to produce salt. The lesser-known Little Pecan phase has been defined at Little Pecan Island on the coast to the west and might be associated with the historic Attakapa (Fuller and Wiedenfeld 2015:3–13; Swanton 1911). These final phases of Mississippian and Plaquemine culture represent the closing centuries of a period often regarded as protohistoric, spanning prehistory and the early historic period (Wesson and Rees 2002).

The commencement of the historic or Colonial-American period (ca. 1700–present) in the Mississippi River delta is characterized by increased arrivals of foreigners who appropriated land and resources. This was preceded, however, by nearly two centuries of intermittent and mostly unrecorded contacts between Native Americans, Europeans, and Africans. Spanish vessels sailed along the northern Gulf Coast during the sixteenth century, transporting cargo, immigrants, and enslaved people to and from the growing colony of Mexico. The chronicles of Hernando De Soto's ill-fated expedition through the interior Southeast describe the Lower Mississippi Valley as densely populated (Clayton et al. 1993). Nearly a century and a half would pass before French explorers and *coureur de bois* arrived in present-day Louisiana and established a permanent presence.

Contacts between Native Americans and others during the intervening years were likely brief and sporadic, involving limited exchanges of goods and ideas. The introduction of viruses and infectious pathogens had a more profound and long-lasting effect on indigenous populations. Disease epidemics decimated indigenous communities, eventually enabling the expansion of French and Spanish colonies. Entire regions in the Lower Mississippi Valley described as densely populated in the mid-sixteenth century appear to have been all but abandoned a century and a half later. Other historically known tribes, such as the Tunica and Natchez, economically and politically positioned themselves between the expanding British and European colonial empires (Barnett 2007; Brain 1988). Warfare, systematic violence, enslavement, and deportation accompanied episodic disease epidemics in wreaking havoc on remaining Native American communities. The vestiges from the millennia of their residences are now scattered in the rapidly deteriorating archaeological record of the Mississippi River delta and north-central

Gulf Coast. During the eighteenth and early nineteenth centuries, other Native Americans groups, such as the Choctaw and Coushatta, sought refuge in present-day Louisiana (Kniffen et al. 1987; Usner 1992). Intermarriages between indigenous peoples, French colonists, and African Americans contributed to a process of creolization, but even today many tribes assiduously maintain their identity and culture. The Chitimacha Tribe of Louisiana still reside in their homeland on Louisiana's Gulf Coast (Brightman 2004; Gregory 2004).

## 4. Previous Investigations

Previous archaeological investigations on Louisiana's Gulf Coast provide methodological background and precedent for the present study (Neuman 1977b). Although studies of cultural resources in the Mississippi River delta and coastal zone began a century ago (Collins 1927; McGimsey 1999; Neuman 1984b, 2002), the vast majority of archaeological investigations date from the past few decades. Seminal developments in cultural resource management (CRM) that began during the late 1960s accelerated and expanded the focus of archaeological research throughout the 1970s and 1980s (Neuman 1984b). There are a substantial number of archaeological investigations conducted annually in south Louisiana, the majority of which are terrestrial and underwater Phase I cultural resource surveys (LDA 2017). The numbers of recorded archaeological sites in each parish has consequently increased, along with our knowledge of regional culture history and long-term human habitation on the Gulf Coast. The following account of previous investigations begins by briefly considering some of the formative contributions to Louisiana Gulf Coast archaeology, in an overview of applied CRM studies and university-based research. The succeeding section provides a more focused examination of previous archaeological investigations related to oil spills and in particular, the MC252 *Deepwater Horizon* disaster response.

## 4.1 An Overview of Louisiana Gulf Coast Archaeology

In 1936, the Louisiana Geological Survey published what might be regarded as the first systematic study of archaeological sites and cultures of the Mississippi River delta and surrounding Gulf Coast. Kniffen's (1936) *Preliminary Report on the Indian Mounds and Middens of Plaquemines and St. Bernard Parishes* was followed two years later by his *Indian Mounds of Iberville Parish* (Kniffen 1938). The Louisiana Geological Survey published an earlier study by Kniffen's colleagues at LSU on the "submergence" of sites in Cameron Parish of coastal southwest Louisiana (Howe et al. 1935). Numerous scholars had previously examined various sites, artifacts and material culture of south Louisiana, with particular attention paid to Avery Island and the salt domes on the south-central coast (Beyer 1896, 1899; Czajkowski 1934; Joor 1895; Mercer 1895; Moore 1913; Veatch 1899; see Neuman 1977a:6–13, 1984a:6–52). In 1926, Henry Collins of the U.S. National Museum visited at least 37 sites along Louisiana's coast and performed excavations at 14 of the sites. The Smithsonian Institution published a brief article on the investigation the following year (Collins 1927). McGimsey (1999) subsequently reviewed Collins' investigations and reexamined the collections, producing a more detailed description of the sites and artifacts.

Kniffen's (1936:408–414) study stands out, however, in systematically examining different types of sites in the Mississippi River delta and using pottery collections to define the Bayou Cutler and Bayou Petre complexes. He identified earthen mounds, shell mounds, shell middens, and beach deposits. The pottery complexes Kniffen defined became known as the Bayou Cutler Phase of Coles Creek culture and the Bayou Petre Phase of Mississippian culture (Phillips 1970:920–923, 951–953; Wauchope 1947:188). Kniffen (1936:417) also broached the important issue of site locations and geomorphology of the Mississippi River and its distributaries, a topic many other scholars would pursue (Gagliano and van Beek 1970; McIntire 1954; Phillips et al 1951; Saucier 1963). Drawing on the pioneering work of Fisk (1944), Phillips, Ford, and Griffin (1951:295–306) devoted an entire section of their classic *Archaeological Survey in the Lower Mississippi Alluvial Valley* to correlating culture historical sequences and geomorphology. Combined with the pioneering studies of Ford (1935a, 1935b, 1935c, 1936; Ford and Quimby 1945), Gagliano (1963, 1964, 1967a), McIntire (1954, 1958), and Saucier (1963), Kniffen's seminal research informed subsequent culture historical syntheses and continues to influence archaeological investigations in the delta.

William McIntire (1954, 1958, 1959) performed a systematic, large-scale survey of Louisiana's coastal region, demonstrating the need for regional survey data to address the connections between shifting

landforms, geomorphology, settlement patterns and site distributions of different ages. Building on the work of Kniffen, Howe, and colleagues, McIntire (1958:8–28, 51–101) identified five types of sites. These consisted of earth mounds, shell mounds, shell middens, black-earth middens, and beach deposits. He used regional survey data to correlate the distribution of sites and culture chronology to deltaic lobe formation. He also examined the relationship between archaeological sites and coastal subsidence (McIntire 1958:24–28). Gagliano (1963, 1964, 1967a) and Saucier (1963, 1974, 1981) further established the connection between geomorphology and culture chronology for archaeological research in the Mississippi River delta and coastal zone (see also Frazier 1967; Gagliano 1984; Gagliano and Saucier 1963; Gagliano and van Beek 1970).

Phillips (1970:920–923, 949–955) relied on these and other key works (Ford and Quimby 1945; Ford and Willey 1940; Neitzel 1965; Quimby 1951, 1957) for information on site components and pottery types in his influential discussion of the distribution of archaeological phases in the Lower Mississippi Valley. Phillips' widely cited study set the course for much subsequent archaeological research in the Valley and along Louisiana's Gulf Coast, in which pottery types and *varieties* constituted the primary evidence for site components, phases, cultural diffusion and migrations (Brain 1988, 1989; Brown 1978, 1979a, 1979b, 1981; Neuman 1977a; Weinstein and Rivet 1978; Williams and Brain 1983). The Museum of Geoscience at LSU published Neuman's (1977a) *An Archaeological Assessment of Coastal Louisiana*, based in part on the author's 1972 survey of coastal sites. Along with regional survey data and a culture historical overview, Neuman (1977a:1–6; 31) touched on some of the most critical issues facing Louisiana Gulf Coast archaeology today, including site destruction.

During this formative period of archaeological research, from the 1930s and throughout the 1970s, Harvard University's Lower Mississippi Survey (LMS) and LSU's "man-land" school of cultural and historical geography were the predominant influences on culture historical investigations and geoarchaeology in the Mississippi River delta (Mathewson and Shoemaker 2004). This research culminated in cohesive culture historical syntheses (Haag 1965, 1971, 1978; Neuman 1977a, 1984a; Neuman and Hawkins 1982; Phillips 1970:14–20). With the groundwork laid, archaeologists increasingly turned to the investigation of specific cultures, sites and regions, with growing interest in cultural adaptations in the delta. Byrd's (1974, 1976a, 1976b) study of Tchefuncte subsistence patterns and Rivet's (1973) reappraisal of Tchefuncte ceramics stand out as focused studies of that singular cultural expression (see also Davis 1981; Davis and Giardino 1981; Springer 1973, 1974, 1980; Weinstein and Rivet 1978).

Archaeological investigations in the Mississippi River delta and throughout the Gulf Coast increased dramatically during the 1970s and early 1980s following the passage of Federal and State legislation (notably, the NHPA) and regulations pertaining to the management of cultural resources (36CFR800), historic properties, and the human environment (Beavers et al. 1980; Castille and Holmes 1983; CEI 1977, 1980; Davis et al. 1978; Floyd 1981; Gagliano et al. 1975, 1976, 1977, 1978, 1979, 1982; Gibson 1978, 1982; Goodwin et al. 1985, 1986; Neuman 1970, 1973, 1974a, 1974b, 1974c, 1975a; Poplin et al. 1986; Ryan and Hicks 1984; Weinstein 1980, 1984; Weinstein et al. 1978; Wiseman et al. 1979). Though the LMS and LSU continue to influence archaeological research, the advent of CRM archaeology greatly diversified, enhanced and expanded the scope of investigations in the Mississippi River delta and northcentral Gulf Coast. By comparison, there has been limited development of state university graduate programs focused on Louisiana archaeology. Neuman (2002:89-91) describes the history of Louisiana archaeology from the 1970s through the 1990s, with many of the advances in university-based archaeological research curtailed since his article was published. The most notable advances in archaeological research on Louisiana's Gulf Coast have consequently come through CRM, along with the public archaeology programs of the Louisiana Office of Cultural Development (OCD), Division of Archaeology.

The establishment of the State archaeology program in 1974 introduced archaeology to a broader constituency through public outreach, and provided for the guidance and centralized recording of archaeological investigations throughout the State (Neuman 2002:91-94). Beginning in the late 1970s and throughout the 1980s, the Anthropological Studies Series brought archaeology to the attention of the public (Brain 1977; Brown 1981; Hawkins 1989; Neuman and Hawkins 1982). The Division of Archaeology issued Louisiana's Comprehensive Archaeological Plan in 1983 with support from the National Park Service (NPS) Historic Preservation Fund (Smith et al. 1983). The comprehensive plan organized areas of potential research or themes according to regional management units, with Management Unit V encompassing the Mississippi River delta and Management Unit III extending across the Chenier Plain, prairies, and coastal marsh to the west. The plan recognized prehistoric adaptation to the changing deltas as a major research theme in Unit V and touched on the threats to cultural resources posed by coastal erosion and the ongoing development of the oil and gas industry (Smith et al. 1983:97-100). The State plan identified adverse effects of the oil and gas industry in Management Unit VI, which includes the Mississippi River, submerged archaeological sites and underwater cultural resources (Smith et al. 1983:118). Thirty-five years later, the forthcoming revised Comprehensive Archaeological Plan recognizes oil and gas development as among the challenges to site preservation (Girard et al. 2018:66).

The cultural resources database maintained by the Louisiana OCD, Division of Archaeology, has been most influential in systematizing and organizing archaeological research throughout the State. A majority of the investigations consist of CRM-focused studies (Byrd and Neuman 2010). A search of the OCD CRM bibliography database on November 7, 2017 produced 1,121 entries for the seven coastal parishes in southeast Louisiana where the present study is focused (Orleans, St. Bernard, Plaquemines, Jefferson, Lafourche, Terrebonne, and St. Mary). Though not all of these studies focus on the coast, this database represents the bulk of existing knowledge of archaeological sites in the Mississippi River delta and coastal zone. HDR, Inc. identified 131 previous investigations and 243 previously recorded cultural resources in the study area extending 125 meters (410 ft) from the shoreline affected by the MC252 oil spill (Cloy and Ostahowski 2015:4–1, 4–5, Appendix 4A).

The majority of these previous investigations are Phase I or reconnaissance level surveys conducted under the purview of CRM legislation for regulatory compliance, resulting in the identification, delineation, and recording of sites, or the revisiting of previously recorded sites. The focus of investigations has been to determine archaeological integrity and historical significance, including the potential effects of an undertaking on sites eligible for listing on the National Register of Historic Places (NRHP). Archaeological sites that are found to be historically significant under the criteria for eligibility (usually Criterion D) as a result of survey or testing (Phase II investigations) are typically avoided during the planning stages of an undertaking (Turner and Goodwin 1988; Gibson 1989; Weinstein and Pearson 1982). This is especially the case in underwater archaeology, where remote sensing technologies facilitate the identification of anomalies that can often be avoided (Saltus and Pearson 2010). The adverse effects of a proposed undertaking on sites listed or eligible for listing on the NRHP are less frequently mitigated through more intensive or larger-scale data recovery (Phase III) excavations.

Among the implications of this three-phased approach to applied archaeology in the Mississippi River delta has been the production of geographically extensive datasets within the constraints of a particular area of potential effect (APE). Early examples include surveys by Coastal Environments, Inc. for the Gulf Intracoastal Waterway (Gagliano et al. 1975) and Mississippi River Gulf Outlet (CEI 1980; Wiseman et al. 1979), Gulf South Research Institute's survey for the Louisiana Interstate Gas Corporation (Saltus et al. 1975), and Goodwin Associates' surveys for hurricane protection projects (Goodwin et al. 1985, 1986, 1991). The NPS funded regional overviews and assessments during this period (Beavers 1982; Neuman 1977c; Greene et al. 1984; Speaker et al. 1986), including a study of coastal erosion and archaeological resources on five National Wildlife Refuges in Louisiana (Garrett 1983). Neuman's (1977a:31) assessment of coastal sites was prescient in identifying coastal land loss, due to both natural and human

causes, as the greatest challenge to archaeology on Louisiana's Gulf Coast. Goodwin and Associates, Inc. subsequently provided an overview of archaeological sites in the coastal zone for the Louisiana DNR, with an assessment of the potential for buried archaeological components based on deltaic and coastal geomorphology (Goodwin et al. 1991:89–93).

Large-scale surveys and site testing have more recently been conducted in southeast Louisiana for projects such as the Mississippi River levee restoration (Somers et al 2011), hurricane risk reduction (Boyko et al. 2013), the White Ditch Diversion and marsh revitalization (Heller et al. 2011), and the Mississippi River Gulf Outlet ecosystem restoration (Kowalski et al. 2011). These and hundreds of other investigations have produced a substantial and still-growing database on site locations, components, artifact assemblages, cultural affiliations, and changing environmental conditions in the Mississippi River delta (Athens 1992; Braud 2008; CEI 2009; Goodwin et al. 1991, 2012; Hahn et al. 2012; Hinks et al. 1991, 2001; Kelley et al. 2000, 2008; Maygarden et al. 1995; Moreno et al. 2011; Pelletier et al. 2005; Perrault et al. 1994; Rawls et al. 2010; Robblee et al. 2003; Ryan et al. 2005; Saltus and Pearson 1990; Sick et al. 2003; Smith et al. 2002, 2014; Thomas et al. 1997; Weinstein et al. 1994; Weinstein and Kelley 1992; Wells et al. 2014). The results of these and other studies have contributed to a greater understanding of regional settlement patterns, artifact typologies, subsistence and culture history in the Mississippi River delta and north-central Gulf Coast.

A search of the Louisiana OCD Division of Archaeology cultural resources database on November 7, 2017 lists 2,246 previously-recorded sites in the seven easternmost coastal parishes, with hundreds more along the coast to the west. A majority of these (n=721; 32%) are located in Orleans Parish and represent the results of Phase I surveys in and around metropolitan New Orleans. St. Bernard Parish is more typical, with 194 previously recorded sites. Of these, 45 percent (n=87) are recorded as having prehistoric components and 33 percent (n=64) are listed as having historic components (LDA 2017). However, the number of sites recorded in each parish represents a fraction of the total number of extant and destroyed sites. Site recording is strongly influenced, and in many instances determined by the scope of work, area of potential effect and sampling biases of an investigation, formation processes, archaeological visibility, and cultural obtrusiveness (Rees 2011:81).

Neuman's (1975b) excavation at the Bayou Jasmine site (16SJB2) and Shenkel's (1974, 1980) work at Big Oak Island (16OR6) and Little Oak Island (16OR7) were among the first large-scale and intensive archaeological investigations focused on site testing and data recovery in the Mississippi River delta (Shenkel and Morehead 1980). More recently, Goodwin and Associates, Inc. conducted Phase III data recovery investigations at the Discovery site (16LF66) in Lafourche Parish (Miller et al. 2000) and Earth Search, Inc. performed a Phase III investigation of the Yscloskey Mounds site (16SB8/46) in St. Bernard Parish (Smith et al. 2014). There have been numerous investigations of submerged terrestrial sites and underwater cultural resources in the region, from a study of the underwater portion of the Mulatto Bayou site (16SB12) in St. Bernard Parish (Weinstein and Pearson 1982) to data recovery of the Mardi Gras Shipwreck (16GM01) on the GOM continental slope (Ford et al. 2008). Under the category of alternative mitigation, R. Christopher Goodwin & Associates, Inc. conducted an extensive reanalysis of Tchefuncte sites and collections (Heller et al. 2013), in fulfillment of a programmatic agreement for the Federal Emergency Management Agency (FEMA). Their study expands current understanding of Tchefuncte culture and demonstrates the substantial research potential of existing collections.

Previous archaeological investigations not carried out under the aegis of CRM or regulatory compliance with Federal legislation are relatively fewer in number, but have made key contributions to the understanding of past cultures and historical ecology in the Mississippi River delta. Such studies include the previously mentioned NPS-funded assessments and regional surveys (Beavers 1982; Garrett 1983; Greene et al. 1984; Neuman 1977c; Neuman and Servello 1976; Speaker et al. 1986). Unrestricted by the Section 106 process, these investigations have been designed around topical research issues, such as site chronologies and conservation, culture historical synthesis, settlement patterns and subsistence. The now

defunct LDA regional archaeology program stands out as an exemplar in this regard, with over two decades of focused research by the Southeast and Southwest Regional Archaeology programs producing a wealth of archaeological information on the Mississippi River delta and Gulf Coast (Hays 1996, 1999, 2000; McGimsey 1995, 1998, 2003a, 2004, 2006; McGimsey et al. 1999, 2000; Mann 2001, 2002, 2005, 2008, 2009, 2010, 2011, 2012; Mann and Saunders 2008; Mann et al. 2006; Palmer 2010, 2011, 2012; Russo 1992, 1993; Saunders 1993, 1994). Along with the university-based Regional Archaeology program, a series of theses and dissertations have also contributed to the archaeology of Louisiana's Gulf Coast (Brown 2012; Byrd 1974; Evans 2012; Fullen 2005; Futch 1979; Goodwin 2003; Homburg 1991; Rivet 1973; Schilling 2004; Shelley 1980; Smith 1996; Springer 1973; Stevenson 1992; Vasbinder 2005; Woodiel 1980).

A perusal of articles published since 1974 in *Louisiana Archaeology*, the Bulletin of the Louisiana Archaeological Society, highlights recurrent themes of research in the Mississippi River delta and along Louisiana's Gulf Coast. Authors have most frequently described site excavations, structure, organization and components (Bruseth 1980; Gray 2015; Homburg 1992; Jones 2014; McGimsey et al. 2005; McLain 2014; Neuman 1992; Rodning and Mehta 2017; Saunders et al. 2009; Shenkel 1974, 1982; Schilling 2009; Struchtemeyer and Eberwine 2013; Weinstein 1996; Woodiel 1993), but have also explored geomorphology, survey and remote sensing (Goodwin 2012; Neuman 1977b; Neuman and Byrd 1981; Saucier 1999). Ceramic types, seriation and stylistic change have been a recurring focus of research (Davis and Giardino 1981; Hays and Weinstein 1999; Saunders 1997; Shenkel 1974; Springer 1977), along with faunal use (Lewis 1997), bone tools (Kidder and Barondess 1982), subsistence patterns and seasonality (Byrd 1976b, 1994; Davis 1987; Delahoussaye et al. 2015; Duhe 1977; Futch 1981; Springer 1974; Webb 1982). The Tchula Period and Tchefuncte culture, in particular, are of continuing interest (Hays and Weinstein 1999; Lewis 1997; Weinstein 1995). There has been increased interest in historical archaeology, particularly urban archaeology (Jones and Gray 2015). The influence of CRM in coastal archaeology remains evident throughout, representing only a portion of the data generated by CRM.

## 4.2 Archaeology, Oil Spills, and the MC252 Response

The potential effects of an oil spill on archaeological sites were still relatively unknown in April 2010, despite the knowledge gained from an earlier oil spill. In March 1989, *Exxon Valdez* ran aground on Bligh Reef in Prince William Sound in the Gulf of Alaska, causing what was then the largest maritime oil spill. During and after the *Exxon Valdez* cleanup response, teams closely monitored archaeological sites along south-central Alaskan shorelines (Wooley and Haggarty 2013). For more than a decade, archaeologists reported on a series of studies regarding the direct and indirect impacts of the *Exxon Valdez* oil spill on cultural resources (Betts et al. 1991; Bittner and Reger 1995; McMahan 1993; Mobley et al. 1990; Reger and Corbett 1999; Reger et al. 1992, 1996, 1997, 1998, 1999, 2000; Yarbrough 1997). Cultural resource managers regarded the emergency conditions, magnitude, and scope of the *Exxon Valdez* oil spill disaster as unprecedented at the time, with only one known previous investigation of the potential impacts of an oil spill on archaeological sites (Mobley et al. 1990:9). A similar scenario would play out two decades later in the north-central Gulf of Mexico.

Among the substantive findings of the CRM response to the *Exxon Valdez* disaster was the urgent need for avoidance of adverse effects during oil spill cleanup and remediation, including impacts from shoreline treatment, looting, and vandalism (Haggarty et al. 1991:249–252; Mobley et al. 1990:197–200; Reger et al. 2000). Archaeologists and heritage resource managers consequently emphasized the need for education, agency accountability, and site stewardship programs during an oil spill response (Reger and Corbett 1999). In the instance of the *Exxon Valdez* oil spill, the adverse effects of hydrocarbon contamination on archaeological deposits were ultimately regarded as negligible in comparison to the potential for indirect site damages and disturbance caused by shoreline treatment and other cleanup activities (Bittner 1996; Mifflin and Associates 1991; Reger et al. 2000).

Of particular interest to the present study, an investigation of the effects of crude oil contamination initially suggested there would be no adverse effects on radiocarbon dating at sites in Prince William Sound where shorelines had been oiled (Reger et al. 1992). Investigators did not determine whether the radiocarbon samples had been contaminated with oil, so the findings were presented as tentative and deemed not broadly applicable beyond the four sites that were tested. In at least one sample, pretreatment appeared to indicate the absence of hydrocarbons. The authors noted that the results of their study could not conclusively address whether crude oil affects radiocarbon dating at oiled sites (Reger et al. 1992:96–98).

Of even greater relevance to the MC252 oil spill response, the *Exxon Valdez* damage assessment led to the institution of techniques and procedures for CRM planning and decision making in response to an oil spill or other environmental disaster. In 1997 the Advisory Council on Historic Preservation, National Conference of State Historic Preservation Officers, NPS, Department of the Interior, Environmental Protection Agency (EPA), and other Federal agencies signed a programmatic agreement for the protection of historic properties during emergency responses to pollution from hazardous substances (ACHP 1997). The implementation of Shoreline Cleanup Assessment Technique (SCAT) crews and inclusion of an archaeologist on SCAT teams had proved critical to the overall success of the *Exxon Valdez* CRM response (Mobley et al. 1990:4, 9). Two decades later and more than 3,000 miles to the southeast, it would inform the MC252 oil spill response (Michel et al. 2013:1).

The *Exxon Valdez* and *Deepwater Horizon* disasters were both massive oil spills, but of substantially different magnitudes and in remarkably different environments (Atlas and Hazen 2011; Sylves and Comfort 2012). *Exxon Valdez* spilled approximately 257,000 barrels (10.8 million US gallons) of crude oil into Prince William Sound, eventually reaching an estimated 1,300 miles (2,100 km) of coastline (EVOS 2017; Gundlach et al. 1991). The MC252 oil spill was more than 19 times larger in volume, discharging an estimated 4.9 million barrels (210 million U.S. gallons) of crude oil from the Macondo well into the north-central Gulf of Mexico. The MC252 spill may have initially affected a comparable extent of shoreline. However, its source at the Macondo wellhead–41 miles (66 km) offshore, at a depth of more than 5,000 feet (1,524 m)—coupled with the use of 1.8 million gallons of chemical dispersants, produced a plume of dispersant-treated oil across the northern GOM extending from Texas to Florida (Michel et al. 2013; Nixon et al. 2016:170; NRDC 2015; Stout et al. 2017; Valentine et al. 2014; Wilson et al. 2015).

Cleanup after the *Exxon Valdez* spill involved oil removal and pressure washing of rocky intertidal zones of island, inlet, and cove shorelines around Prince William Sound. The cleanup response following the MC252 oil spill also involved mechanical and hand removal techniques based on the SCAT surveys developed during the *Exxon Valdez* oil spill response (NOAA 2015). SCAT teams working in the Mississippi River delta and along the northern Gulf Coast encountered very different environmental conditions, including brackish and saltwater marshes, estuaries, low-lying mudflats and barrier island beaches, natural levees, bayous, backwater lakes, and freshwater swamps. The removal of oil and remediation of oiled shoreline proved to be more problematic in coastal wetlands because of these environmental conditions, particularly for oiled marshes (Michel and Rutherford 2014; Owens et al. 2011; Zengel and Michel 2013; Zengel et al. 2015). The cultural resources were also remarkably different, with archaeological sites located on subsided landforms increasingly impacted by coastal erosion, storm surges, and relative sea-level rise.

During the weeks and months after the *Deepwater Horizon* disaster and MC252 oil spill, archaeologists began to consider the potential effects on sites and cultural resources on the north-central Gulf Coast (Borrell 2010; O'Brien 2010). The potential impacts of the offshore oil and gas industry on archaeological sites in the northern Gulf of Mexico are routinely considered in the permit approval process, including underwater cultural resources and submerged terrestrial sites. Studies of the effects of an oil spill on cultural resources are considerably less common and, other than the *Exxon Valdez* response, no

investigation had been attempted previously at the magnitude of the MC252 oil spill. Initial response efforts focused on containment, oil spill cleanup, environmental remediation, and shoreline monitoring (NOAA ORR 2018; Owens et al. 2011). Applying the lessons learned from the *Exxon Valdez* disaster, archaeologists and tribal monitors were employed in SCAT teams to monitor and survey oiled shoreline and cleanup efforts.

Early in the MC252 oil spill response, the NPS National Center for Preservation Technology and Training (NCPTT) provided guidance to property owners, resource managers, and agency officials on materials conservation and the protection of historic structures from crude oil in marine environments (Chin 2010). The NCPTT studied the effects of oil on historic Fort Livingston on Grand Terre Island and tested methods for cleaning historic structures (Chin and Church 2010). According to their initial findings, some cleaning agents and techniques have the potential to cause additional damage. Preventative measures, such as the use of containment booms placed around structures, were recommended to avoid additional oiling (Chin and Church 2010:6–7).

The cultural resource response to the MC252 oil spill was an enormous and sustained undertaking. It was carried out in accordance with Section 106 of the NHPA, the regulation for the "Protection of Historic Properties" (36 CFR Part 800) and the 1997 Programmatic Agreement developed following the *Exxon Valdez* oil spill (Cloy and Ostahowski 2015: Table 5-1). The survey and monitoring of archaeological sites initially spanned more than 3,107 miles (5,000 km) of shoreline along the north-central Gulf Coast, from Texas to Florida. HDR, Inc. led the cultural resources response, assisted by archaeologists with Coastal Environments, Inc.; Earth Search, Inc.; Geo-Marine, Inc.; MRS Consultants, LLC; Wiregrass Archaeological Consulting, LLC; and Southeastern Archaeological Research, Inc. Tribal monitors were employed in Louisiana by United South and Eastern Tribes, Inc. (USET) and included representatives with the Chitimacha Tribe of Louisiana, the Coushatta Tribe of Louisiana, the Jena Band of Choctaw Indians, the Mississippi Band of Choctaw Indians, the Alabama-Coushatta Tribe, the Choctaw Nation of Oklahoma, the Muscogee (Creek) Nation, and the Thlopthlocco Tribal Town of Oklahoma (Cloy and Ostahowski 2015:i, 1-1, 1–11; HDR 2011:27, 37–38). Tribal monitors accompanied SCAT teams to protect cultural resources and participate in the Section 106 review process (Cloy and Ostahowski 2015:5–51; Gabler and Parker 2015).

Cultural resource investigations began in May of 2010 and involved SCAT teams working along the coasts of Alabama, Florida, Louisiana, and Mississippi. Waterborne and pedestrian surveys, shovel testing, auger and trench sampling, and tribal monitoring were coordinated with State and Federal agencies, including the SHPO in each state and the U.S. Coast Guard (USCG), the latter of which filled the role of on-scene coordinator for the overall oil spill response effort. According to Cloy and Ostahowski (2015:1-1, 5-1), the effort in Louisiana was subdivided into 52 SCAT divisions and ultimately extended across all 11 coastal parishes (Cameron, Vermilion, Iberia, St. Mary, Terrebonne, Lafourche, Jefferson, Plaquemines, St. Bernard, Orleans, and St. Tammany). HDR, Inc. (2011:iii, 1–2) issued an interim report in April of 2011, by which time SCAT crews had identified 50 previously unrecorded archaeological sites and relocated 76 previously recorded sites in Louisiana (HDR, Inc. 2011:49–50, Appendix B, Tables B.1 and B.2). Many of the sites with prehistoric components in Louisiana are readily identifiable as having shell midden or earthen mounds, while many others consist almost entirely of redeposited cultural materials (Table 2). Those sites distinguishable by scattered artifacts along the shoreline are often associated with redeposited shell hash.

SCAT teams also described the amount and degree of oiling along shorelines of barrier islands, coastal marshes and wetlands. Surveys and monitoring focused on locations with recorded sites and areas of high probability for archaeological sites. This informed recommendations for the avoidance of adverse impacts, as well as appropriate treatment and cleanup methods during the response, resulting in the development of Shoreline Treatment Recommendations and Best Management Practices for cultural resources protection (Cloy and Ostahowski 2015:1-2, 5-1, 5-2; HDR, Inc. 2011:27–28, 33, 37, 49–50). As

during the *Exxon Valdez* oil spill response, potential adverse impacts to archaeological sites included cleanup activities such as the mechanical and manual removal of oil.

Table 2. Selected sites investigated during the MC252 oil spill response

Site Number	Site Name	Components	Cultural Features	NRHP Eligibility Recommendation
16JE2	Cheniere St. Denis	Troyville, Coles Creek	Earth and shell mounds, artifact scatter	Eligible
16JE3	Bayou Cutler #1	Marksville, Troyville, Coles Creek	Shell midden, possible mound, artifact scatter	Eligible
16JE111	South of Mud Lake	Indeterminate Woodland	Shell midden	Not eligible
16JE229*	Raccoon Lake 2	Indeterminate prehistoric	Shell midden, artifact scatter	Not eligible
16LF293*	Redfish Slough	Coles Creek, Plaquemine, Mississippian	Artifact scatter	Undetermined
16OR16	East Rabbit Island 1	Tchefuncte, Marksville, historic	Artifact scatter	Undetermined
16PL7	Toncrey	Coles Creek, Plaquemine, Mississippian	Earthen mounds, shell midden, artifact scatter	Eligible
16PL8	Adams Bay	Plaquemine, Mississippian	Earthen mounds, shell midden, artifact scatter	Eligible
16PL13	Buras Mounds	Plaquemine, Mississippian	Earthen mounds, shell midden, artifact scatter	Eligible
16SB11/13	Bayou Petre	Mississippian	Shell midden, artifact scatter	Eligible
16SB24	Bayou Eloi	Tchefuncte, Mississippian	Shell midden	Eligible
16SB33	Seven Dollar Bay	Indeterminate Woodland, Middle Woodland or later	Shell midden, artifact scatter	Eligible
16SB171*	Brush Island 1	Indeterminate Woodland, Mississippian	Artifact scatter	Undetermined
16SB172*	Brush Island 2	Indeterminate Woodland	Artifact scatter	Ineligible
16SB174*	Comfort Island	Indeterminate Woodland, historic	Artifact scatter	Ineligible
16SB178*	Southern Comfort	Tchefuncte, Marksville, historic	Artifact scatter	Ineligible
16SB180	Bayou Pierre 1	Marksville, Troyville, Coles Creek	Artifact scatter	Ineligible
16SB182*	Scow Island Scatter	Marksville, Troyville, Coles Creek, historic	Artifact scatter	Ineligible
16SB185*	Acorn Mounds	Indeterminate Woodland	Earthen mounds	Eligible
16SB186*	Live Oak Bayou Mounds	Indeterminate Woodland	Earthen mounds	Eligible
16SMY17	Bayou Sale	Indeterminate Woodland, historic	Shell midden	Undetermined
16SMY150	Crawford Point	Indeterminate prehistoric, historic	Artifact scatter	Eligible

<sup>\*</sup>Site recorded during the MC252 response (Cloy and Ostahowski 2015: Appendix 4B; LDA 2017).

The recovery of stranded boom and displaced absorbent materials in marshes and along shoreline posed another hazard to archaeological resources. Archaeologists monitored recovery at archaeological sites to circumvent potential impacts from the removal of stranded boom and response-related structures (Cloy

and Ostahowski 2015:5–34–5–50; HDR, Inc. 2011:28). At the time of the Interim Report, HDR, Inc. (2011:50) described 18 of the 76 previously recorded sites and 31 of the 50 newly recorded sites as visibly oiled (Appendices C.4 and D.1). This did not include an assessment of oil at formerly terrestrial, submerged sites.

After the well was capped, the cleanup and CRM response shifted to onshore areas. The focus of the CRM investigations expanded from initial monitoring and survey for cleanup support and the protection of sites, to delineating site boundaries, determining site age and significance, and making recommendations on eligibility for listing on the NRHP within the constraints of the Phase I survey methodology (Cloy and Ostahowski 2015:ii, 5-1, 5-2, 5-44, 6-1; HDR, Inc. 2011:38). In the ensuing months and years following the initial response, archaeologists revisited sites, continued to monitor shoreline, conducted shoreline surveys, and performed additional subsurface testing. This included shovel test pits; auger test pits; and the monitoring of SCAT trenches, auger tests, and Snorkel SCAT pits. In addition to SCAT surveys, HDR, Inc. performed shoreline surveys in advance of cleanup activities (Cloy and Ostahowski 2015: i–ii, 1-6, 5-22, 5-31).

SCAT teams and HDR, Inc. archaeologists completed most of the fieldwork between May of 2010 and March of 2014, ultimately surveying 4,918 kilometers (3,055.90 miles) of shoreline in Louisiana. HDR recorded 50 new sites, 333 isolated finds of artifacts, and revisited 163 previously recorded sites in Louisiana. Most of the newly recorded sites (n=39, 80 percent) consisted of redeposited artifacts. HDR recommended four of the newly recorded sites as eligible for listing on the NRHP, with eligibility undetermined for 22 sites. Of the total 213 sites in Louisiana, 165 sites contained prehistoric site components. Of these, 118 sites consisted only of prehistoric site components. The most common prehistoric deposits were scattered ceramic sherds and redeposited or intact shell midden material. The artifacts spanned the Tchula period through the historic period (Cloy and Ostahowski 2015:1-6, 5-22, 5-31, 6-1, 6-2, 7-1, 7-5, 7-18, Tables 6-1, 6-2, 7-5, and 7-7).

HDR recorded direct adverse effects from the MC252 oil spill at only one site, during a SCAT survey at Fort Livingston (16JE49) on West Grand Terre Island, where the NRHP-listed structure was coated with oil (Cloy and Ostahowski 2015:6-431, 7-19; see also Chin 2010; Chin and Church 2010; Cloy et al. 2013). As in the wake of the *Exxon Valdez* oil spill, the potential for adverse effects was more commonly associated with response related activities, such as oil removal and shoreline cleanup. Despite the potential for direct and indirect impacts of the oil spill and cleanup response, HDR, Inc. concluded that coastal erosion and subsidence "are the greatest long-term concerns to terrestrial archaeological resources along the Louisiana coast" (Cloy and Ostahowski 2015:7-17). Though 163 previously recorded sites were revisited during the response, 77 previously recorded sites were found to have been submerged since they had last been visited an average of 23.4 years ago (Cloy and Ostahowski 2015:7-18, Table 7-6; HDR, Inc. 2011:49–50, Appendix B, Tables B.1 and B.2).

The CRM investigation conducted during the MC252 oil spill response did not address the potential effects of oil on the archaeological record, such as site formation processes, the *in situ* preservation of information, or the consequences for analytical techniques and future archaeological studies. There were no chemical analyses to detect the presence of oil on artifacts. The contamination of artifacts with oil was determined based on visual inspection, including the appearance of an oily sheen when immersed in water. Special procedures were followed in handling artifact collections from sites impacted by the oil spill. Visibly oiled artifacts were wrapped in foil and double bagged to prevent off-gassing and contamination of other artifacts. In the final analysis, artifacts thought to be contaminated with oil (n=42) were only a small fraction (0.4 percent) of the 10,496 artifacts recovered from Louisiana during the response (Cloy and Ostahowski 2015:5-68, 6-1).

Studies of historic shipwrecks have more recently shed light on the effects of an oil spill on underwater cultural resources in the GOM. An investigation of historic shipwrecks from the nineteenth and twentieth

centuries within the area impacted by the fallout from the underwater plume of dispersed oil indicate decreased biodiversity in sediment microbiomes in comparison to uneffected areas (Hamdan et al. 2018). This research drew on multiple lines of evidence, from the genetic sequencing of bacterial composition and sedimentation rates, to chemical hydrocarbon analysis. Because the presence of shipwrecks in an ecosystem increases microbiome diversity, the consequences of the MC252 oil spill are still unfolding in the chemical and biological interactions on the seafloor (Hamdan et al. 2018:12). Experimental data likewise indicate the combination of crude oil and dispersant can adversely affect the composition of biofilms on the metal hulls of shipwrecks and potentially accelerate corrosion (Salerno et al. 2018). With support provided from the BOEM Environmental Studies Program (Cooperative Agreement No. M13AC00015), these and other studies of the environmental impacts of the MC252 oil spill are beginning to shed light on the long-term effects of an oil spill on cultural resources.

## 5. Research Methodology

The eight archaeological sites that are the subject of this study are located along Louisiana's Gulf Coast, on marsh islands, and remote areas of shoreline of the Mississippi River Delta impacted by the MC 252 oil spill. The initial selection of sites for inclusion in this study was carried out through correspondence between the Principal Investigator (PI) Dr. Mark A. Rees, the Louisiana State Archaeologist Dr. Charles "Chip" McGimsey, and the BOEM Project Officer (PO) Mr. Scott Sorset. The PI and the Project Director, Mr. Samuel Huey, compiled information on various archaeological sites in the region from site records and reports of previous investigations, including the interim report on the cultural resources response performed by HDR Environmental, Operations, and Construction, Inc. (HDR 2011). Addressing the goals and objectives of this research was a primary concern that guided the overall research methodology. The following section describes how these issues and other variables influenced site selection. This is followed by overviews of the fieldwork and sampling methodology. The final two sections of the Research Methodology consist of detailed descriptions of the fieldwork conducted at each site and the various analytical methods applied in this study.

### 5.1 Site Selection, Permits, and Access

All of the sites selected for this study had been previously recorded with the Louisiana OCD, Division of Archaeology and were described as having prehistoric components. A deliberate effort was made to include sites with earthen mounds and shell midden previously reported by SCAT teams as having been impacted by the MC252 oil spill. Previous investigations and site records indicate that most archaeological deposits along the shoreline impacted by the MC252 spill have also been adversely affected by coastal erosion. Almost 80 percent of the 50 sites newly recorded during the MC252 oil spill response consisted of redeposited artifact scatters (Cloy and Ostahowski 2015:7–18). One of the goals of site selection for the present study was to assess the effects of an oil spill on intact archaeological deposits, as well as secondarily redeposited materials. By including sites with earthen mounds and shell midden, rather than only small procurement sites or diffuse scatters of artifacts, the intent was to increase the likelihood of sampling and assessing intact archaeological deposits, rather than just wave-washed artifacts in redeposited contexts.

The PI presented a list of potential sites for discussion at a post-award meeting on August 5, 2014. At this meeting, the PI also presented a Draft Action Plan to the BOEM PO, the BOEM Regional Federal Preservation Officer, the BOEM Environmental Studies Program representative James Moore, and the Louisiana State Archaeologist. The list of sites for potential assessment was shortened from 14 to 12 sites in the revised Draft Action Plan, with a minimum of eight and a maximum of 12 sites to be assessed, as stipulated in the Project Management Plan (Table 3). Sampling would be conducted at a minimum of six sites where oil had been observed during the MC252 response and a minimum of two control sites, where oil had not been previously observed.

Variables for inclusion of sites on the list involved location within the area directly impacted by the MC252 oil spill, reports of accumulations of oil on the shoreline, and relative proximity to the MC252 source. Because the absence of oil could not be reliably confirmed through observations during one or more site visits, the selection of control sites was influenced by greater distance from the MC252 source. The potential for encountering and sampling undisturbed archaeological deposits at these sites was also considered, although many of the recorded coastal sites in the region are thought to largely consist of secondary, redeposited materials. Though some indication of intact cultural deposits was a criterion in site selection, historical significance, and previous recommendations about eligibility for listing on the NRHP were not specific requirements.

Two control sites (16PL7 and 16SMY95), where oil had not been observed during the MC252 response, were dropped from the revised Draft Action Plan list. Two remaining control sites (16SB153 and 16SMY17) to the north and west of the most heavily impacted coastal areas were kept on the list, along with 10 sites previously characterized as containing varying accumulations of oil from the MC252 spill (Table 3). The participants at the post-award meeting agreed that these 12 sites constituted a provisional list for possible assessment. Four of the 12 sites in the revised Draft Action Plan are located in southern Jefferson Parish (16JE2, 16JE3, 16JE111, and 16JE229), one site is in Orleans Parish (16OR16), six sites are in eastern St. Bernard Parish (16SB153, 16SB171, 16SB172, 16SB174, 16SB178, and 16SB180), and one is in St. Mary Parish (16SMY17).

Table 3. List of sites for potential assessment in the revised Draft Action Plan

Site No.	Site Name	Components	Cultural Features	NRHP Eligibility Recommendation
16JE2	Cheniere St. Denis	Troyville, Coles Creek	Earth and shell mounds, artifact scatter	Eligible
16JE3	Bayou Cutler #1	Marksville, Troyville, Coles Creek	Shell midden, possible mound, artifact scatter	Eligible
16JE111	South of Mud Lake	Indeterminate Woodland	Shell midden	Not eligible
16JE229	Raccoon Lake 2	Indeterminate prehistoric	Shell midden, artifact scatter	Not eligible
16OR16	East Rabbit Island 1	Tchefuncte, Marksville, historic	Artifact scatter	Undetermined
16SB153*	Unnamed	Troyville, Coles Creek, Mississippian, historic	Shell midden	Eligible
16SB171	Brush Island 1	Indeterminate Woodland, Mississippian	Artifact scatter	Undetermined
16SB172	Brush Island 2	Indeterminate Woodland	Artifact scatter	Ineligible
16SB174	Comfort Island	Indeterminate Woodland, historic	Artifact scatter	Ineligible
16SB178	Southern Comfort	Tchefuncte, Marksville, historic	Artifact scatter	Ineligible
16SB180	Bayou Pierre 1	Marksville, Troyville, Coles Creek	Artifact scatter	Ineligible
16SMY17*	Bayou Sale	Indeterminate Woodland, historic	Shell midden	Undetermined

<sup>\*</sup>Control site not previously characterized as having oil present from the 2010 MC 252 spill.

It was determined that subsequent revisions to the list of sites for assessment would be made through correspondence between the BOEM PO, the State Archaeologist, and the PI. The potential reasons for additional changes included the removal of a site from the list due to lack of authorization from a property owner. Information on land ownership in site records is often outdated or incomplete and requires searching through the records of parish tax assessors. The Project Director, Mr. Samuel Huey, carried out background research on site ownership, online and at tax assessor offices in Jefferson, Orleans, and St. Bernard parishes. Even after the Project Director had obtained current contact information for all landowners, out-of-state property-owners and corporate landowners proved to be difficult to contact and slow to respond.

Rapidly changing environmental conditions along the coast were also a critical issue in site selection, with the potential for precluding sites from this study. Knowledge of site conditions is generally based on information collected when a site was recorded or last visited, and consequently may be outdated. Relocating previously recorded sites can be especially problematic in coastal wetlands that have been

heavily impacted by erosion, subsidence, and sea-level rise. It was discovered that approximately 50 percent of the sites revisited as part of the MC252 oil spill response were entirely or mostly submerged (Cloy and Ostahowski 2015:7–17). Rapidly changing coastal landforms and shorelines could thus potentially exclude a site from being assessed as part of this study.

Though the majority of the sites in this study are located on privately owned land, the State Archaeologist recommended at the post-award meeting that a State permit be requested in the event site assessment occurred on State lands. The PI consequently applied for a permit from the Louisiana Archaeological Survey and Antiquities Commission. The Louisiana OCD, Division of Archaeology, issued a Cultural Resources Investigation Permit to the PI on October 29, 2014. The permit was in effect for a period of one year. The BOEM PO confirmed that while the U.S. Army Corps of Engineers has authority over waterways, lands below the mean high water line, or at mid-tide, fall under the authority of the State of Louisiana. The possible need for a coastal use permit from the Louisiana Department of Natural Resources (DNR) was discussed during a meeting of the Louisiana Archaeological Survey and Antiquities Commission on September 9, 2014. In consultation with the BOEM PO and the State Archaeologist, Louisiana DNR subsequently exempted this study from the requirement of a coastal use permit.

Although it was not the intent of this study to excavate or otherwise disturb human remains, a preliminary visit to one of the control sites, Bayou Sale (16SMY17), revealed fragmentary and redeposited human remains on the ground surface. Human remains along the shoreline were reported in a site record update for 16SMY17. The Project Director observed human bone on the surface of the shell midden and in other areas of the site, interspersed with redeposited non-human animal bone, shell, and organic debris. There was consequently a potential at Bayou Sale, as well as other sites, for the inadvertent disturbance and collection of human bone fragments in redeposited contexts during excavation and sampling. With this in mind, the PI requested an unmarked burial permit from the State Archaeologist. The PI was issued an unmarked burial permit on September 26, 2014. The provisions of the permit stipulated that bone identified as human during fieldwork would not be collected. Excavations that encountered human remains would be terminated, backfilled, and the units relocated. Furthermore, all inadvertently collected human remains identified as such in the laboratory would be returned to the site of origin for reburial.

The post-award meeting also focused on field methods for sampling and the need for a Logistics, Fieldwork, and Sampling Plan, to be addressed in the following sections of this report. Participants discussed the possible use of existing collections from previously investigated sites. Though the date of investigation (pre- or post-MC252 oil spill), type of collection (pottery sherds or soil), and archaeological context would have to be well established, it was determined that similar controls in the collecting and processing of samples might not apply. For example, the washing of artifacts and possible inadvertent contamination of samples from sources outside of archaeological contexts posed unknown methodological problems. Whether or not existing collections might be used in this study, site selection took into account the information to be provided by previous investigations. In particular, the goal of locating intact archaeological deposits for sampling would benefit from reports on past excavations.

The list of sites for potential assessment had to be revised after two property owners refused permission to work at three sites (16OR16, 16SB171, and 16SB172). There was no response from landowners to requests to access two other sites on the list (16JE111 and 16JE229). With the agreement of the PI, the BOEM PO, and State Archaeologist, the Revised List of Sites for Assessment was presented in the Second Annual Progress Report (Rees and Huey 2016:3). With the removal of five sites from the list, sites 16LF293, 16PL8, 16SB182, 16SB185 and 16SB186 were added (Table 4). Although no oil cleanup activities took place at or near Acorn Mounds (16SB185) or Live Oak Bayou Mounds (16SB186), these sites were added to the list of sites for assessment based on reports of oiling in the region and the potential for encountering oil in undisturbed archaeological deposits (Michel et al. 2013; NOAA 2015). These are large sites with earthen mounds, yet both sites were only recently recorded during the MC252 response.

The revised list retained two sites identified earlier in Jefferson Parish (16JE2 and 16JE3), one control site in St. Bernard Parish (16SB153), one control site in St. Mary Parish (16SMY17), and three sites initially proposed in St. Bernard Parish (16SB174, 16SB178, and 16SB180). As described in the following sections, eight of these sites were investigated and sampled as part of this study.

Table 4. Revised list of sites for assessment

Site No.	Site Name	Components	Cultural Features	NRHP Eligibility Recommendation
16JE2	Cheniere St. Denis	Troyville, Coles Creek	Earth and shell mounds, artifact scatter	Eligible
16JE3	Bayou Cutler #1	Marksville, Troyville, Coles Creek	Shell midden, possible mound, artifact scatter	Eligible
16LF293 <sup>†</sup>	Redfish Slough	Coles Creek, Plaquemine, Mississippian	Artifact scatter	Undetermined
16PL8 <sup>†</sup>	Adams Bay	Plaquemine, Mississippian	Earthen mounds, shell midden, artifact scatter	Eligible
16SB153*	Unnamed	Troyville, Coles Creek, Mississippian, historic	Shell midden	Eligible
16SB174	Comfort Island	Indeterminate Woodland, historic	Artifact scatter	Ineligible
16SB178	Southern Comfort	Tchefuncte, Marksville, historic	Artifact scatter	Ineligible
16SB180	Bayou Pierre 1	Marksville, Troyville, Coles Creek	Artifact scatter	Ineligible
16SB182 <sup>†</sup>	Scow Island Scatter	Marksville, Troyville, Coles Creek, historic	Artifact scatter	Ineligible
16SB185 <sup>†</sup>	Acorn Mounds	Indeterminate Woodland	Earthen mounds	Eligible
16SB186 <sup>†</sup>	Live Oak Bayou Mounds	Indeterminate Woodland	Earthen mounds	Eligible
16SMY17*	Bayou Sale	Indeterminate Woodland, historic	Shell midden	Undetermined

<sup>\*</sup>Control site not previously characterized as having oil present from the 2010 MC 252 spill. †Site not previously included in the Draft Action Plan.

# 5.2 Site Sampling

Before fieldwork began, the PI submitted a Logistics, Fieldwork, and Sampling Plan to the BOEM PO, with descriptions of the proposed methods for conducting the fieldwork and laboratory activities. The plan focused on strategies for accomplishing the research goals and objectives, from site access and permits, to transportation, lodging, safety measures, field methods, special sampling, and laboratory methods. The following section builds on the Logistics, Fieldwork, and Sampling Plan to present an overview of logistical issues encountered, followed by a general description of the field methods used in this study.

As described in the preceding sections, the archaeological sites sampled for this study are located on marsh islands and other remote locations in the Mississippi River Delta (Figure 8). Mobilization for fieldwork consequently required arrangements for the safe transport of crew, equipment and samples to and from these locations. The Louisiana Department of Wildlife and Fisheries (LDWF) provided watercraft and pilots for this purpose. The LDWF transported the University of Louisiana Lafayette (ULL) crew to four sites during the course of this study (16SB174, 16SB178, 16SB182 and 16SB185). The Louisiana Universities Marine Consortium (LUMCON) provided for the rental of watercraft and a trailer. The Project Director completed boat-operator safety training and piloted the LUMCON vessel to four sites (16JE2, 16SB153, 16LF293 and 16SB185). Because of the quickly changing and often-unpredictable weather conditions on the coast, on several occasions fieldwork had to be prematurely

suspended and resumed when weather conditions improved. The length of time required to reach and return from sites by watercraft made these unexpected interruptions in fieldwork especially challenging.

The PI and Project Director referred to site records, site record updates, and reports of previous investigations to confirm the location and GPS coordinates of each site. Locational information was crosschecked with Google Earth and made available to the LDWF pilot before departure. When the LDWF transported the field crew and equipment to a site, the pilot and watercraft remained with the crew, or in close proximity, from the time of arrival until departure. The Project Director and PI established daily check-in times for the safe return of the field crew. Appropriate safety protocols for working in potentially hazardous conditions were developed and applied through standard operating procedures outlined in the Logistics, Fieldwork, and Sampling Plan. The Project Director received HAZMAT training and instructed field crew in the proper handling of potentially contaminated samples in the field and lab. Additional safety measures included the use of gloves, masks, and protective clothing when handling materials contaminated with hydrocarbons.



Figure 8. Locations of sites selected for assessment.

Oiled sites shown in red and control sites shown in yellow. Imagery from NASA Landsat Science.

#### 5.2.1 Field Methods

Because of the goals of this study, the field methods did not strictly conform to the routine survey, testing, or data recovery standards established by the Louisiana Division of Archaeology (LDA). The LDA developed these fieldwork standards as guidelines for regulatory compliance with the National Historic Preservation Act (NHPA) for Federal agency permitted or approved projects. By contrast, this research tested specific hypotheses. Nevertheless, the fieldwork adhered to the standards in many respects, such as the use of 0.25-inch (6.35 mm) mesh hardware cloth to screen soil matrix from shovel tests and test units. Whenever possible, information was collected that may be relevant in determining the historical significance of a site for listing in the NRHP. The field and laboratory methodology, however, were not focused on determining historical significance, archaeological integrity or NRHP eligibility, but specifically aimed at sampling for assessment of the effects of an oil spill.

The field methods consisted of pedestrian surface inspection, systematic or controlled surface collection, the use of a hand-operated 4-inch (10 cm) diameter bucket auger, a 2-cm diameter hand-held soil probe, coring with a 2-inch (5 cm) diameter hammer-driven core, shovel testing, and sample collection through small-scale excavations. Site assessment generally began with visual inspection of ground surfaces, followed by the excavation of 30-cm diameter shovel tests, auger testing, and the use of a soil probe to locate subsurface cultural deposits for further investigation. A controlled surface collection was conducted if surface inspection produced evidence of diagnostic cultural materials. The surface collection of artifacts, such as pottery sherds, was generally limited to representative diagnostic materials for the purposes of component description or obtaining samples to test for the presence of oil. The Project Director used a GPS to record the provenience of surface-collected samples. Samples were placed in clean tin canisters or aluminum foil that was then placed in a cloth bag for transport inside an airtight, waterproof container.

Following the surface inspection and controlled surface collection, subsurface methods examined areas with the highest probability of containing cultural features or deposits. In areas where ground surface visibility was poor or limited, the horizontal extent and distribution of cultural deposits was examined through the excavation of 30-cm diameter shovel tests along one or more transects spaced at 5, 10, 20, or 30-meter intervals. Shovel test transects were oriented in cardinal directions or corresponding to the shoreline and landscape. The excavation of shovel tests proceeded in stratigraphic levels or observed cultural layers. All soil matrices were processed through 0.25 inch (6.35 mm) wire mesh. A 2-cm diameter soil probe was used at the bottom of some shovel tests to investigate underlying stratigraphy. A hand-operated, 4-inch (10 cm) diameter bucket auger was used to investigate the vertical extent of deposits and the potential for deeply-buried cultural deposits. Soils collected by auger were also processed through 0.25-inch (6.35 mm) wire mesh or collected in cloth sample bags. At some sites, the 2-cm diameter auger extension rod was used as a probe to locate buried shell midden. The locations of each shovel test and auger test were recorded by GPS and noted as positive or negative for cultural materials.

The shovel testing, auger testing, and probing methods were used to locate buried cultural deposits or areas with relatively higher concentrations of artifacts. The use of these methods was substantially reduced or entirely avoided at sites where good ground surface visibility provided information on the distribution of cultural materials, or where reports of previous investigations assisted in relocating subsurface deposits. The fieldwork proceeded to the hand excavation of test units once the locations of higher artifact concentrations or subsurface cultural deposits suitable for further investigation were identified through surface inspection, shovel testing, and augering. The express purpose of these test units was to collect samples of artifacts, ecofacts, and soil for further analysis. This included the sampling of intact or relatively undisturbed archaeological deposits from primary contexts, as well as the more common wave washed and redeposited cultural materials in secondary contexts. The coring consisted of horizontal cores collected from test units and 2-inch (5 cm) diameter vertical soil cores. The coring is described in the following section on Sample Methods.

Test units typically measured 50-by-50 cm. Depending on the results of each 50-by-50 cm test unit, the perimeters were expanded, as needed, into 50 cm-by-1 meter or 1-by-1 meter test units. At some sites, such as Bayou Sale (16SMY17), St. Malo (16SB153), and Scow Island Scatter (16SB182), excavation proceeded almost immediately to 1-by-1 meter units based on a surface inspection and the results of previous investigations. Excluding the shovel tests and auger tests, the excavated test units at each site comprised a combined total area of at least one square meter. The Project Director recorded the locations of all test units with a GPS and plotted these on a site map.

All test units were hand excavated, usually in 10-cm levels within cultural or natural strata, until depths at which culturally sterile subsoil was reached, or until inundation of the unit prohibited further excavation. Artifacts and ecofacts were collected from each 10-cm level in clean cloth bags, tin canisters, or aluminum foil pouches. As to be expected in low-lying coastal areas, inundation from the water table

made excavation difficult at most sites. In some instances where deeply buried cultural deposits were inundated, a hand-operated pump and/or electric sump pump was used to facilitate continued excavation. The sump pump was placed at the bottom of the excavation unit or in an adjacent shovel test, powered by a small generator. To avoid inadvertent contamination of the test unit, the generator was placed at a safe distance and if possible, downslope from the test unit. At sites with deeply-buried cultural deposits, such as St. Malo (16SB153), excavation could proceed only by removal of water with a larger, 3-inch diameter pump. Appropriate precautions were taken to avoid contamination when collecting samples and transporting the gasoline generator and pump.

The soil matrix from each test unit was ordinarily sieved through 0.25-inch (6.35 mm) wire mesh in the field. Dry screening was preferable and generally practiced to avoid inadvertent contamination from water during the processing of soil matrices. Water screening was recorded in the field notes when it became necessary due to the high clay content of the soil matrix, or to expedite fieldwork due to time constraints. After a test unit or shovel test was completed, the walls were cleaned, photographed and recorded with profile illustrations. A hand-held 2-cm diameter soil probe and 4-inch (10 cm) diameter auger were used to investigate underlying strata and to confirm whether the underlying matrix was culturally sterile. Shovel tests were usually terminated upon inundation, while the excavation of inundated test units was continued in some instances with the assistance of a water pump.

Because the fieldwork was designed to assess the effects of an oil spill, it was necessary to avoid the incidental contamination of artifacts and samples with oil or dispersants during excavation and collection. Precautionary methods were taken to avoid the introduction of oil, dispersants and other contaminants into cultural deposits from surficial or exogenous sources during sampling. This involved cleaning the floor and walls of each level with a clean trowel. Underlying cultural materials and soil matrix could then be sampled with decreased possibility of accidental contamination during excavation. Oils were not used in cleaning tools. No synthetic or plastic implements were used for excavation or the collection of samples. The use of plastic bags, plastic buckets, and petroleum based products was avoided. The following section describes the sample collection methods in more detail.

#### 5.2.2 Sampling Methods

The sampling methodology was designed to produce two categories of collections. The first were standard collections of artifacts, soil matrices, and other materials that were assigned field sample (FS) numbers. These included pottery sherds collected from the screening of matrix from a shovel test or excavation unit. Materials designated as field samples were typically recovered using standard archaeological protocols and included artifacts and ecofacts from augering, shovel testing, and test unit excavation. Field samples were collected in paper or cloth bags and labeled with provenience information. For the purposes of this study, field samples carry some unknown risk of inadvertent contamination through excavation or incidental contact with hydrocarbons, dispersants, or other chemicals. Though field samples could prove useful in the interpretation of site components and cultural deposits, these were not specifically intended for analyses involving the detection of oil or its potential effects on archaeological deposits and analytical techniques.

The second category of collections consisted of special samples (SS). These samples were assigned numbers sequentially with FS numbers in the field. In order to avoid inadvertent contamination by hydrocarbons, dispersants or other chemicals, all special samples were recovered and handled in the field and lab according to special sampling protocols. This included the use of hand tools and containers made of metal or non-plastic materials for collection, the periodic cleansing and rinsing of tools with Dawn detergent and clean water, and not using oils or other chemicals for cleaning. Most important, special samples were sealed during collection and not opened until processing and analysis in a controlled laboratory environment. These protocols were taken to ensure that collection and transport of special samples did not introduce contaminants from sources outside of the sampled archaeological context.

Special samples thus constitute a control measure, by which potential contamination during collection might be avoided, or examined in comparison to field samples collected by traditional methods. The use of such controls were intended to assist in determining whether traditional methods of excavation and sample collection are compromised at oiled sites due to the potential for secondary contamination during recovery. Distinguishing *in situ* contamination from secondary, archaeologically introduced contamination might allow researchers to determine if traditional methods of excavation and sample collection are inadequate at oiled sites, potentially requiring special sampling methods at all sites thought to be potentially impacted by an oil spill.

Special samples included individual specimen samples from test units and controlled surface collections, soil samples, column samples, and unit core samples. The majority of special samples were recovered during the excavation of test units. Specimen samples of individual artifacts and ecofacts, such as pottery, shell, and bone, were collected *in situ* during excavation, using freshly-cleaned tools within the 10-cm level of a test unit. Specimen samples were placed in aluminum foil envelopes, clean cloth bags, or previously unused tin canisters. Soil samples typically ranged from 1 to 4 liters in volume and were collected from 10-cm levels of test units and placed in clean cloth bags. Soil samples were also collected from what appeared to be cultural features. Soil samples and other special samples were placed in an airtight, waterproof container and transported to the lab for processing (Section 5.4 Analytical and Laboratory Methods).

Column samples were collected from freshly cleaned test unit walls, usually after a test unit had been completed. These consisted of vertical columns of soil matrices, measuring approximately 10-by-10 cm and excavated into an exposed and freshly cleaned test unit wall in 10 cm levels. Column samples were collected in previously unused, clean tin canisters, which were then placed in clean cloth bags and assigned "SS" numbers that recorded provenience information. These were transported to the lab in an air tight, waterproof container.

Small unit core samples were also collected from within test units. The special samples obtained from unit cores can be distinguished from site coring, or the extraction of vertical soil cores to obtain stratigraphic information from a site. The unit cores were collected from test units by driving a clean, 2.375-inch (6 cm) diameter steel tube, 12 to 18 inches (30 to 46 cm) in length, perpendicularly into a freshly-troweled unit wall, or vertically into a cleaned test unit floor. The field crew used a wooden block and hammer to drive the metal tube into a unit wall or floor. Extraction of a unit core, usually from a profile wall, typically produced a small, 6 to 8 inch (15 to 20 cm) long sample of matrix. Column samples and unit core samples were comprised of matrices comparable to the associated wall profile stratum where each was extracted.

The steel tubes used for the test unit cores were thoroughly cleaned and dried before samples were collected and were not reused. Following extraction from a unit wall, the ends of the steel tube were sealed with aluminum foil. The unit cores were labeled according to provenience, placed in cloth bags, and similarly transported to the lab in an air tight, waterproof container. These small unit cores supplemented the column samples and provided additional safeguards against inadvertent contamination during collection. Whether by this unit coring method or column sampling, a minimum of one special sample was collected from each 10 cm level of each test unit. Column samples and unit core samples were mostly soil matrices, with varying amounts of crushed and whole shell. Some column samples and unit core samples also contained artifacts and ecofacts. These were separated, sorted, and classified in the lab during the processing of samples (Section 5.4 Analytical and Laboratory Methods).

Another soil coring method was implemented in producing special samples from the Acorn Mounds site (16SB185). The deeply buried and saturated deposits encountered at this site required sampling at greater depths than could normally be reached in a test unit. The field crew collected samples from different areas of the site using a 2-inch (5 cm) diameter core specifically designed for these conditions. This coring

device is equipped with a hammer-driven handle, extensions of different lengths, a proximal cap or "core cap" that allows air to escape when the core is inserted, aluminum sample sleeves that fit within the 12-inch (30 cm) long barrel of the core, and a butterfly valve or "sludge tip" for marsh soils. The butterfly valve has a hinged door across its opening that is attached to the distal end of the core. As the core is driven into the ground, the hinged door opens and allows soil to enter into the sample sleeve of the core. Upon extraction, the butterfly valve closes and the proximal core cap creates suction, sealing the sample within the sleeve (Figure 9).

This coring technique proved effective in extracting often heavily saturated, deeply buried marsh soils on subsided landforms. Before each sample was collected, the core was cleaned and a previously unused aluminum sleeve was loaded into the barrel. After removal, both ends of the sleeve were sealed with aluminum foil and provenience information was recorded. This method ensured that material collected within the sleeve was not contaminated from sources outside of the sampled deposit. As with the other special samples, these were placed in clean cloth bags and transported to the lab in an air tight, waterproof container.



**Figure 9. Sample extraction from the hammer-driven core.** Katherine Sinitiere extracting the inner sleeve of a core sample at the Acorn Mounds site (16SB185).

#### 5.3 Fieldwork

The Project Director carried out fieldwork at eight sites selected for this study, assisted by a field crew of one or two student research assistants. Fieldwork was completed at seven sites during the first fiscal year, between September of 2014 and June of 2015 (Huey and Rees 2015). Fieldwork was completed at the eighth site during the second year, in August and September of 2015 (Rees and Huey 2016). The following sections describe the fieldwork in the order each site was visited, along with additional information on previous investigations, site conditions, and archaeological deposits.

#### 5.3.1 Bayou Sale (16SMY17)

The Bayou Sale site consists of extensive shell midden deposits located along the shore of East Cote Blanche Bay, approximately 0.36 mile (0.6 km) north of the mouth of Bayou Sale and 1.1 mile (1.8 km) southeast of the Burns Point Recreation Area and LA Route 317. The midden is situated on a remnant of a natural levee, west of Bayou Sale and on the eastern shore of the bay. Marsh grass, willow trees, and dense vegetation cover much of the midden, but shell (primarily *Rangia cuneata*) is visible on the surface under leaves and organic debris washed in by the tide. The site is eroding and an unknown percentage of the site area is submerged. Much of the site above the shoreline appears to consist of wave redeposited shell and overburden, although intact deposits are thought to lie beneath these secondary deposits in places along the shoreline and offshore (Figures 10 and 11).

William McIntire and A. D. Warren with the Lower Mississippi Survey first recorded the Bayou Sale site in 1952. Surprisingly little is known about the site sixty-six years later, despite repeated, albeit brief visits by archaeologists. Neuman (1977a:21) noted extensive pottery and bone at the site but reported "no data" on the cultural period. Ian Brown included the Bayou Sale site in his study of the *Petite Anse* region in 1979 and archaeologists with Earth Search, Inc. visited the site again in 2005 (Brown et al. 1979:65; LDA 2017; Smith et al. 2006). Brown (2015:220) observed that a majority of the pottery from the site is classified as Baytown Plain, *var. unspecified*, providing little in the way of chronological or culturally diagnostic information, other than a Woodland period association.



**Figure 10. Shell midden at 16SMY17.** View to the east at the Bayou Sale site.



**Figure 11. Shell midden at 16SMY17.** View to northwest at the Bayou Sale site.

The boundaries of the Bayou Sale site have not been sufficiently determined, but are loosely defined by cursory visual inspections and observations of shell deposits on the surface along the shoreline, rather than systematic subsurface testing. The extent of the intact and redeposited shell midden suggests the Bayou Sale site may be part of Site 16SMY95, an even more extensive shell midden 130 meters to the northwest (LDA 2017), as well as deposits of shell along the shoreline to the south of 16SMY17, noted during the present study. This would account for the differing opinions on the site's distance north of Bayou Sale (0.36 mile to one mile). It also suggests that shell midden and associated cultural deposits may extend for at least 0.6 mile (1 km) along the shore of East Cote Blanche Bay.

Archaeologists with HDR, Inc. and the LDA conducted a visual inspection of the shoreline by boat in May of 2010, as part of the MC252 oil spill response (Cloy and Ostahowski 2015:6–135). The crew did not observe oil on the shoreline at this time, although a systematic pedestrian survey was not conducted. No oil was observed at the site or along the shoreline immediately to the north or south during the MC252 response and cleanup activities were not conducted here (Cloy and Ostahowski 2015: Appendix 3, p. 17–18). The site was consequently selected as one of two control sites for this study. Investigators have repeatedly observed human remains at the site during previous site visits, but little is actually known about the possible associations of burials with prehistoric or historic components. The site's archaeological integrity and eligibility for listing on the National Register have not been adequately determined. The site is recorded as a "prehistoric cemetery," however, and avoidance of adverse impacts is recommended due to the presence of human remains (LDA 2017).

An initial site reconnaissance was conducted for this study on September 23, 2014. Bayou Sale was the only site accessed on foot, from a parking area near a radio tower northeast of the site. Based on a preliminary surface inspection, it quickly became evident that human remains were scattered across the site, mixed with organic debris and redeposited shell. Much of the fragmentary human remains appear to have eroded from the shell midden and been redeposited by tidal action. An unmarked burials permit was requested and issued on September 26, 2014, in the event human remains were inadvertently disturbed or collected during the course of this study. Shovel testing conducted during a return visit on September 26 produced bone fragments, some of which were subsequently identified as human. Bone that was

identified as human was recorded in the field but not collected. As stipulated by the unmarked burials permit, fragments of bone that were identified in the lab as human were returned to the site for reburial during a second site visit on October 6, 2014.

In total, six shovel tests were excavated on September 26 (Figure 12). Shovel Test 1 (ST 1) and ST 2 were excavated into a shell midden deposit on the bank of Cote Blanche Bay just south of the main portion of the site. A surface inspection in this area did not reveal any artifacts, so shovel tests were excavated to investigate the nature and extent of the shell deposit. The shell observed on the surface did not initially appear to be wave washed or to have undergone extensive redepositing. ST 1 was negative for cultural materials, but ST 2 contained a nutria (*Myocastor coypus*) tooth. Crushed and broken clam shell, primarily *Rangia*, was present in both shovel tests. The accumulated shell and associated stratigraphy indicate this southern portion of the site was likely redeposited from an area of the midden now submerged beneath the bay. Investigations subsequently proceeded to the main area of the site to the north.

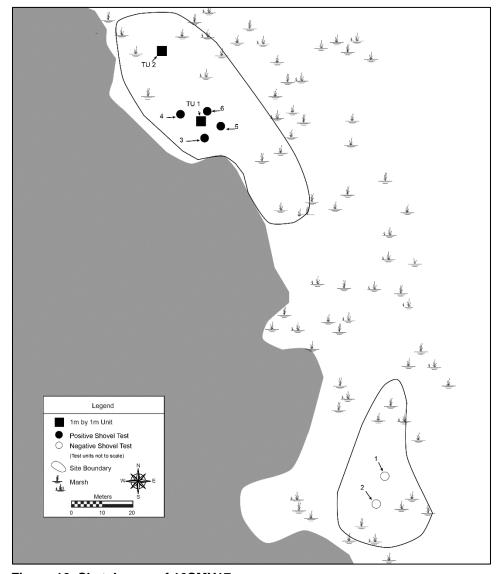


Figure 12. Sketch map of 16SMY17.

The Bayou Sale site Site boundaries were

The Bayou Sale site. Site boundaries were only loosely determined by visual inspection and are included here only as an approximation.

Four additional shovel tests were excavated along the shoreline to the north. ST 3 and ST 4 were placed closer to the shoreline; ST 5 and ST 6 were excavated on what appeared to be heaps of redeposited shell farther back from the shoreline. Stratum I in ST 3 and ST 4 consisted of a 14 to 15 cm thick layer of very dark gray (10YR 3/1) clay within crushed, broken, and whole *Rangia* shell. Stratum I may represent the remaining intact portions of shell midden at the site. Strata II and III consisted of very dark gray (10YR 3/1) clay with dark brown (7.5YR 3/3) mottles and gray (10YR 5/1) clay, void of shell. ST 5 and 6 were excavated in an area of the site where wave action and tidal surges have pushed shell and sediment eastward across the site. This has resulted in the redeposition of crushed and broken shell in heaps along the eastern and northeastern area of the site.

ST 5 and ST 6 produced crushed shell hash that confirmed the midden is redeposited and disturbed at this location. The stratigraphy in ST 3 through ST 6 suggests that tidal activity is removing material from the upper strata of the midden adjacent to the shoreline and redepositing it across the site, accumulating in heaps on the eastern fringes of the site. Human remains with minimal signs of weathering are eroding from the shoreline in areas that may still contain intact midden. Besides fragments of human remains and unidentified faunal bone, wave-washed pottery sherds, and a small quantity of glass shards were recovered from the shovel tests (see Section 6.1).

The landowner briefly restricted access to the site after September 26, 2014. The crew returned on October 6, 2014 to continue the fieldwork. Fragmentary human remains that had been inadvertently collected from shovel tests were reburied at this time at the location of ST 6. The extent of redeposited human remains across the site made it difficult to find an area suitable for the excavation of a test unit (TU) that would avoid collecting or disturbing redeposited human bone. During the third site visit, two 1-by-1 meter units were opened, but only one was excavated to a depth that allowed samples to be collected. Excavation of the first 1-by-1 meter test unit (TU 1) was halted after only a few centimeters due to a large amount of disarticulated and fragmentary human remains. These remains were left *in situ* within the test unit, which was then backfilled. The crew moved to another area of the site to excavate a second test unit.

TU 2 was placed approximately 25 meters northwest of TU 1, in a location where no human remains were visible on the surface. The placement of TU 2 was determined based on surface inspection for the presence of pottery sherds and absence of human remains, as well as a series of soil probes that indicated the presence of shell midden beneath the surface. TU 2 was excavated in four levels to a depth of approximately 35 cm below surface, at which point inundation from the water table prohibited further excavation.

Three distinct strata, including a buried A-horizon at approximately 20 cm below surface, were evident during the excavation of TU 2 (Figures 13 and 14). Stratum I was a very dark brown (10YR 2/2) clay loam within a dense concentration of crushed and broken *Rangia* shell. Stratum II was made up of mostly black (10YR 2/1) clay loam, mottled with very dark gray (10YR 3/2) clay loam and a dark brown (10YR 3/3) clay loam, with whole and broken shell. In contrast, the top of Stratum III was marked by a thick root mat that was interpreted as the surface of a buried A-horizon. This stratum was not previously encountered in the shovel tests. Stratum III consisted of very dark grayish brown (10YR 3/2) clay loam with whole and broken *Rangia* shell. Wave worn prehistoric pottery sherds, bone fragments, glass container shards, one piece of brick, ferrous metal, and a historic ceramic sherd were recovered from Strata I and II, in excavation levels 1 through 4 (see Section 6.1).

The amount of broken and crushed shell decreased at 26 cm below surface in TU 2, while the relative amount of Native American ceramic sherds appeared to increase. The root mat encountered at this depth, at the beginning of Level 4, appears to mark the surface of a buried A-horizon (Stratum III), but it is uncertain whether Stratum III represents an undisturbed context. Twelve samples were collected from TU 2 (Appendix A1), of which eight consisted of special samples. The special samples consisted of four

column samples from the East wall, one unit core sample from the North wall at a depth of 22 cm below surface, and three soil samples, from levels 1 through 3. Seven additional special samples were collected from ST 4, 5, and 6. The remaining samples from the Bayou Sale site were processed in the field by dry or wet screening through 0.25-inch mesh (Appendix A1). Samples of soil, shell and pottery were brought to the lab at ULL for processing and analysis, to be discussed in Section 6 of this report.

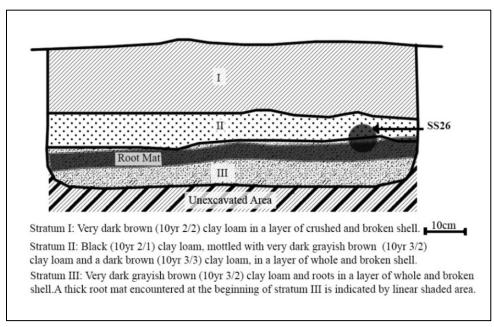


Figure 13. North wall profile of TU 2 at 16SMY17.

Bayou Sale site.

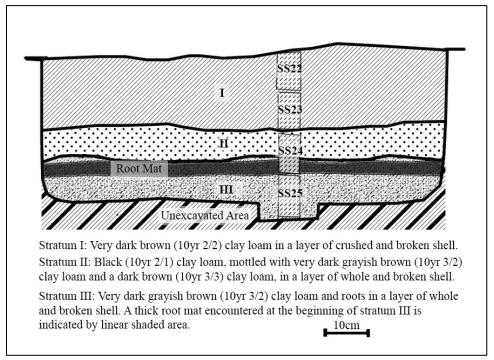


Figure 14. East wall profile of TU 2 at 16SMY17.

Bayou Sale site.

### 5.3.2 Cheniere St. Denis (16JE2)

Cheniere St. Denis is a prehistoric shell midden on the northeastern bank of Bayou St. Denis. There are two intact shell and earth mounds at the site, located in a north-south orientation to one another. The midden and mounds are situated on a partially-submerged and subsided natural levee in the Barataria drainage basin, approximately 12.5 miles (20 km) southeast of Lafitte and 15.7 miles (25 km) northeast of Galliano, Louisiana. Much of the site was overgrown with marsh grasses, scrub oaks, and palmettos at the time it was visited for this study (Figures 15 and 16). Other portions of the site are submerged and part of the shell midden, consisting primarily of *Rangia*, is eroding and being redeposited by wave action (LDA 2017).

Cheniere St. Denis has been the subject of numerous archaeological investigations since it was originally recorded in 1935 by Kniffen. It stands out in this study as one of two sites, along with Site 16SB153, that have been more intensively investigated by archaeologists (Coughlin et al. 2004:35–42). The Cheniere St. Denis site was visited in 1977 by Coastal Environments, Inc. and by R. Christopher Goodwin & Associates, Inc. in 1984 (Gagliano et al. 1979; Goodwin et al. 1985). Archaeologists with R. Christopher Goodwin & Associates, Inc. returned to the site in 2002 and performed more extensive Phase II test excavations as part of the construction of the proposed Endymion Pipeline (Coughlin et al. 2004; LDA 2017). As a result of these efforts, the prehistoric component of the site was determined to retain archaeological integrity and to possess qualities of significance under criterion D of the NRHP.



Figure 15. Photograph of Site 16JE2.

View to the north. North mound in the background and southern mound in the foreground to the right.



Figure 16. Photograph of Site 16JE2.

View to the south from northernmost mound.

Archaeologists with R. Christopher Goodwin & Associates delineated the horizontal and vertical extent of archaeological deposits through the excavation of 109 shovel tests, 28 auger tests, and seven 1-by-1 meter units in the submerged portion of the site. Their investigation yielded evidence of intact archaeological contexts in mounded areas of the site (Coughlin et al. 2004:53–58, 99). Based on the ceramics that have been recovered from the site and three radiocarbon dates obtained by Goodwin and Associates (Coughlin et al. 2004, Appendix 3), the Cheniere St. Denis site contains Late Baytown-Troyville and Coles Creek components. The shell midden and mounds appear to date from the last century of the Baytown period and first three centuries of the Coles Creek period (ca. 670 to 970 CE). The historic component is associated with a fishing camp previously located on the west side of the site.

Cheniere St. Denis was more recently visited by archaeologists with HDR, Inc. during the MC 252 oil spill response, when it was described as lightly oiled (Cloy and Ostahowski 2015:6–393, Appendix 3:37; HDR 2011:D-4). Based on previous investigations, the Cheniere St. Denis site was determined to be eligible for listing in the NRHP. Avoidance of clean-up activities was recommended in the wake of the MC252 oil spill. HDR conducted surface inspections and surface collections at the site but did not do any shovel testing or subsurface investigations (Cloy and Ostahowski 2015:6–394).

The Cheniere St. Denis site was visited for the present study on October 31 through November 2, 2014. The project director piloted a LUMCON vessel, transporting the field crew and equipment to and from the site. Information gleaned from the previous investigation by R. Christopher Goodwin & Associates, Inc. was used to relocate intact subsurface deposits, making it unnecessary to excavate additional shovel tests. A brief pedestrian survey and controlled surface collection were conducted upon reaching the site. Pottery sherds were visible on the surface primarily in two areas: in the vicinity of the northernmost mound (Area A) and the southernmost point of the site (Area B). There was no discernible evidence (visual or olfactory) of oil on the surface of the site at the time the fieldwork.

A hand-held, 2-cm diameter soil probe was used to investigate buried portions of the shell midden. This confirmed the presence of shell midden in an area between the northern and southern mounds. Coupled with information from previous investigations and the surface inspection, the soil probes were used to

guide the placement of test units along the shoreline and on mound flanks, in areas suspected to contain intact archaeological deposits. The crew excavated a total of 1.25 square meters, consisting of three 50-by-50 cm test units and one 50 cm-by-1 meter test unit (Figure 17). The large amounts of broken and whole shell in the tests units primarily consisted of *Rangia*.

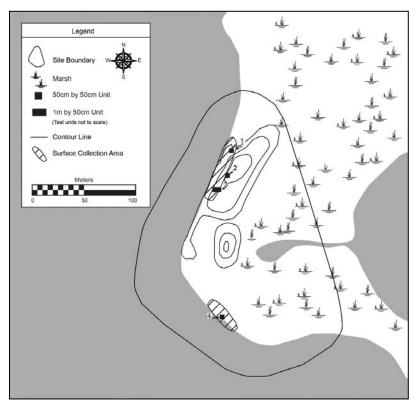


Figure 17. Sketch map of 16JE2.

Cheniere St. Denis site.

Test unit (TU) 1 was a 50-by-50 cm unit placed near the northern shoreline and excavated to a depth of 30 cm below surface, at which point the unit was inundated and the water table hindered further excavation. Two distinct strata were encountered (Figure 18). Stratum I was a black (10YR 2/1) silty clay within a dense shell hash that appeared to be redeposited. Stratum II was comprised of black (10YR 2/1) to very dark grayish brown (10YR 3/2) silty clay within a dense deposit of whole shell and minor amounts of broken shell. Artifacts recovered from TU 1 include Native American pottery sherds, faunal bone, ferrous metal, glass, and plastic (Section 6.1).

TU 2 was a 50-by-50 cm unit excavated on the west slope of the northernmost mound. It reached a depth of 70 cm below surface, at which point an auger was used to investigate deeper deposits. Excavation of TU 2 revealed four distinct strata (Figures 19 and 20). Stratum I consisted of black (10YR 2/1) silty clay and roots within a dense deposit of shell. Stratum II was distinguished from Stratum I by the relative absence of roots. Stratum III was composed of very dark gray (10YR 3/1) silty clay in a dense deposit of crushed and broken shell. Beneath this stratum, Stratum IV consisted of a layer of black (10YR 2/1) silty clay and whole shell. Strata I, II and IV appear to be consistent with the intact depositional context identified by Goodwin and Associates as Stratum I, which they correlate with the Late Baytown-Coles Creek site occupation (Coughlin et al. 2004:64).

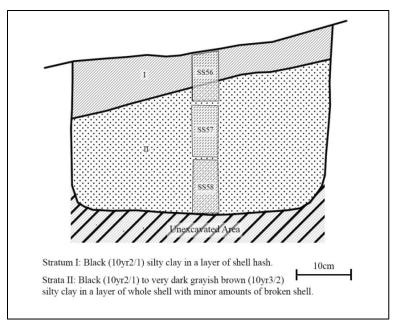
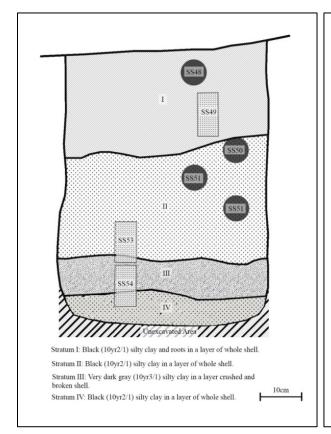


Figure 18. North wall profile of TU 1 at 16JE2.

Cheniere St. Denis site.



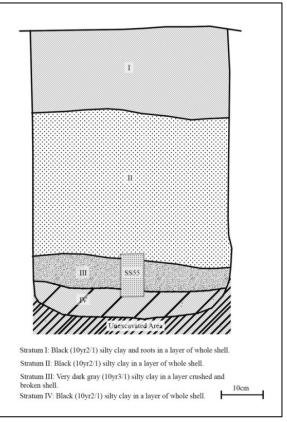


Figure 19. North (left) and East (right) wall profiles of TU 2 at 16JE2.

Cheniere St. Denis site



Figure 20. Photograph of TU 2 at 16JE2

View to the south at the Cheniere St. Denis site.

An auger test placed at the bottom of the unit reached a depth of 95 cm below surface and indicated no further changes in stratigraphy from Stratum IV. The auger test was terminated at this depth due to the dense shell deposit and inundation by the water table. Artifacts recovered from TU 2 included ceramics, lithics, and a small amount of ferrous metal fragments. Historic artifacts were limited to level 1, while pottery and fauna were collected from levels 2 through 7 (Section 6.1). The auger test placed at the bottom of the unit produced one small pottery sherd (<0.5 inch). The stratigraphy of TU 2 was relatively undisturbed based on the condition of the shell and absence of historic artifacts beneath 10 cm.

TU 3 was a 50 cm-by-1 meter unit also excavated on the western flank of the northernmost mound, south of TU 2. It revealed three distinct strata (Figure 21). Stratum I consisted of a black (10YR 2/1) silty clay within a dense deposit of whole shell. Stratum II was distinguished by a very dark gray (10YR 3/1) silty clay in a dense deposit of crushed and broken shell. Beneath this layer, Stratum III was encountered as a black (10YR 2/1) silty clay layer with whole shells. Excavation ended in this stratum, at 45 cm below surface due to inundation by the water table. Stratum III is consistent with the intact depositional context described by Goodwin and Associates as Stratum I, which dates from the Late Baytown-Coles Creek occupation of the site (Coughlin et al. 2004:64). Materials recovered from TU 3 include pottery sherds, faunal bone, a relatively larger amount of lithic artifacts, and a small amount of ferrous metal and glass. Historic artifacts were limited to levels 1 and 2; Native American pottery and lithic artifacts were recovered from levels 1 through 4 (Section 6.1).

Excavation of TU 4 near the southern shoreline revealed two distinct strata (Figure 22). Stratum I consisted of a very dark grayish brown (10YR 3/2) silty clay within a dense layer of crushed, broken, and

whole shell. Stratum II was distinguished by a black (10YR 2/1) silty clay muck within a dense deposit of whole shell. Excavation was terminated due to inundation by the tide at only 20 cm below surface. Materials recovered from levels 1 and 2 of TU 4 include Native American pottery, lithics, ferrous metal, and plastic (Section 6.1). Based on the presence and condition of historic and prehistoric artifacts, both strata in TU 4 appear to be redeposited.

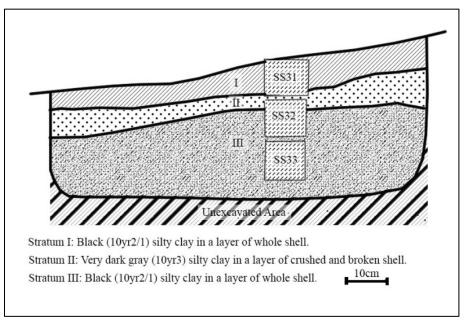


Figure 21. North wall profile of TU 3 at 16JE2.

Cheniere St. Denis site.

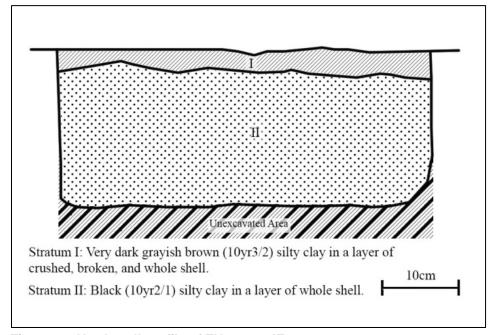


Figure 22. North wall profile of TU 4 at 16JE2.

Cheniere St. Denis site.

A total of 59 samples were collected from the Cheniere St. Denis site, including 40 special samples from the four test units. Nineteen field samples were processed by dry screening or water screening through 0.25-inch mesh. Twenty-two of the special samples were individual specimens (pottery, lithics or fauna), four were soil samples from TU 1, TU 2, and TU 4, ten were collected as column samples from TU 1, TU 2, and TU 3, and four were unit cores collected from the north wall of TU 2 (Appendix A2). All samples and cultural materials were labeled by provenience and transported back to the lab at ULL for processing. Some of the special samples were analyzed for the presence and effects of oil, to be discussed in Section 6 of this report.

## 5.3.3 Southern Comfort (16SB178)

Southern Comfort is a linear-shaped shell and artifact deposit on the south shore of Comfort Island in Chandeleur Sound. The island is in a fairly remote area of the delta, approximately 24 miles (38.8 km) east of the Breton Sound Marina in Hopedale, Louisiana. The Southern Comfort site is situated on a beach ridge along the eroded southeastern shore of the island. The site consists of wave-washed artifacts, redeposited shell and shell hash (Figures 23 and 24). It was described as oiled by archaeologists with HDR, Inc. during the MC252 oil spill response (Cloy and Ostahowski 2015:6-766; HDR 2011:D-59).

The Southern Comfort site was initially recorded when HDR archaeologists visited Comfort Island and observed oil on the southern shoreline (HDR 2011:D-59). The presence of oil along the shore was described as moderate (Cloy and Ostahowski 2015, Appendix 3:59). The site was visited 14 times between August of 2010 and October of 2012. A total of 30 shovel test pits were excavated across the site. All of these shovel tests were void of cultural materials. Artifacts recovered from the surface included 88 Native American ceramic sherds, associated with the Marksville and Tchula periods, in addition to an historic component (LDA 2017). The site was described as disturbed and consisting entirely of secondary beach deposits from Site 16SB136, a now-submerged site located off the southeastern shore of Comfort Island. Given its disturbed condition and lack of archaeological integrity, HDR archaeologists recommended the site as ineligible for inclusion on the NRHP (Cloy and Ostahowski 2015:6-767, 6-768, 6-770, 6-771, Table 6-2).

Fieldwork for the present study was scheduled for three days, beginning on January 5, 2015, but a wind advisory and severe weather prevented the crew from returning to the site on January 7. A return trip was made on January 29, 2015 to complete the excavations and site sampling. The LDWF provided a boat and pilot to transport the ULL crew and equipment to the site. On the first site visit, the surface was inspected while walking along the southern shoreline. Historic artifacts and modern debris were visible on the surface. Oil was also visible on the surface and within excavation units.

Two auger tests and a series of soil probes were conducted in hopes of locating an intact or redeposited midden, although these were unproductive. The auger tests were inundated with water at a shallow depth (22 to 26 cm) and terminated in silty clay, void of artifacts, and with little shell. The first auger test was placed where the presence of an oily sheen and tar balls had been observed on the surface. This auger test produced a small amount of subsurface shell hash.

A systematic surface inspection resulted in designating two areas for surface collection (Figure 25). Area A comprised the western portion of the site. An oily sheen was observed along the shoreline within Area A. Area B was limited to the central and east-central portions of the site as delineated in the site record. An oily sheen was present on a portion of the shoreline in Area B. The easternmost portion of the site as defined by HDR archaeologists was inspected, but no artifacts were collected and no shovel tests or test

units were excavated in this area. The present investigation instead mostly focused on Area A, where the surface inspection revealed oil and wave-worn artifacts on the surface.



Figure 23. Photograph of 16SB178.

Facing west, showing crew on the shoreline at the Southern Comfort site.



Figure 24. Photograph of ST 1 at 16SB178.

Facing east along the shoreline at the Southern Comfort site.

In total, eight shovel tests were excavated in hopes of locating intact cultural deposits beneath the surface. Two of these shovel tests (ST 3 and ST 6) produced artifacts, although ST 2 was terminated at a depth of only 18 cm due to the unexpected presence of baby diamondback terrapins. A layer of redeposited shell hash was encountered in all of the shovel tests except for ST 2 and ST 8. A brown substance was encountered in ST 3 that was thought to indicate the presence of oil, although it lacked any commonly associated odor. Two strata were recorded in the shovel tests along the shoreline. Stratum I was a redeposited layer of very dark-gray (10YR 3/1) to dark grayish-brown (10YR 4/2) sandy-clay loam and dense shell hash that ranged in depth from 0 to 60 cm below surface. Stratum II was a dark gray (10YR 4/1) to dark grayish-brown (10YR 4/2) clay muck. The recovery of artifacts and identification of the layer of shell hash in the shovel tests informed the placement of test units.

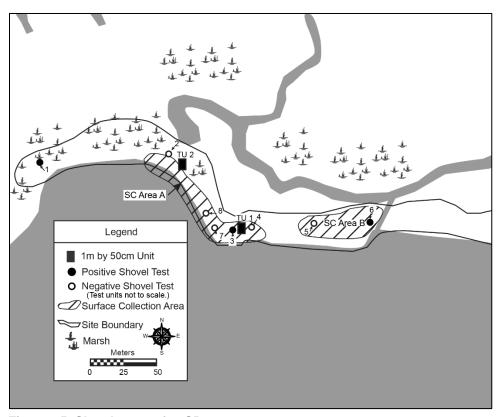


Figure 25. Sketch map of 16SB178.

The Southern Comfort site.

Two 50 cm-by-1 meter units were excavated at the Southern Comfort site, comprising a total of 1 square meter. TU 1 was excavated to a depth of 40 cm below surface until inundation prohibited further excavation (Figure 26). Despite having been placed near a positive shovel test, only a few very small sherds were recovered from this test unit (Section 6.1). Four distinct strata were recorded (Figure 27). Stratum I was characterized as a dense shell hash deposit with roots, very dark gray (10YR 3/1) sandy clay loam, dark brown (10YR 3/3) sandy clay, and black (10YR 2/1) organic inclusions. Stratum II was distinguished by a very dark brown (10YR 3/3) sandy clay loam mottled with dark drown (7.5YR 3/4) silty loam within a moderately-dense deposit of shell hash. Stratum III was void of shell and consisted of a very dark gray (10YR 3/1) silty clay mottled with black (10YR 2/1) silty clay inclusions. Stratum IV was a very dark grayish brown (10YR 3/2) clay loam layer that was void of shell and contained a large

amount of roots. The root mat encountered in Stratum IV was interpreted as a buried A horizon of unknown age.

A second 50 cm-by-1 meter unit, TU 2, was excavated to a depth of 35 cm below surface, until water inundated the unit and excavation was terminated. TU 2 revealed six stratigraphic layers (Figure 28). Stratum I was characterized as a very dark gray (10YR 3/1) silty clay loam within a dense shell hash deposit. Stratum II was distinguished by a dramatic decrease in shell and a dark gray (10YR 4/1) sandy clay loam. Stratum III was void of shell and consisted of dark grayish brown (10YR 4/2) clay loam with roots and decaying organic matter. Stratum IV was a dark gray (10YR 4/1) clay loam void of shell that contained a moderate amount of roots. Stratum V was a dark grayish brown (10YR 4/2) clay loam with roots and decaying organic matter without shell. Stratum VI was also void of shell and characterized as a dark grayish brown (10YR 4/2) loamy clay with dark gray (10YR 4/1) silty and sandy clay mottles.



Figure 26. Photograph of TU 1 at 16SB178.

The Southern Comfort site.

Before the excavation of TU 2, a Baytown Plain, *var. unspecified* pottery sherd had been collected from the surface (top of level 1). A few other Baytown Plain pottery sherds were recovered within Stratum I, between 5 and 10 cm below surface. Except for these artifacts, no other prehistoric materials were encountered during the excavation of TU 2. As reported by archaeologists with HDR, Inc., the present study confirmed that a majority of the site consists of redeposited materials on the shoreline, which may originate from the now-submerged shell midden (Site 16SB136) to the southeast. Artifacts were recovered during the present study from beneath the surface in shovel tests and test units.

The presence of a root mat in Strata IV of TU 1 and roots in Strata IV and V of TU 2 appear to represent buried surfaces. No artifacts were associated with these strata and the subsurface recovery of artifacts was limited to redeposited contexts. Forty-one samples were collected from Southern Comfort, including 19 special samples (Appendix A3). The special samples consisted of seven column samples from TU 1 and TU 2, one unit core sample from TU 1, six soil samples from TU 1, TU 2, and the surface, and five specimen samples, including pottery sherds that appeared to be oiled. Special samples were submitted to the Department of Environmental Sciences (DES) lab at LSU and the University of Missouri Research Reactor (MURR) Archaeometry Lab for analysis; the results are reported in Section 6.

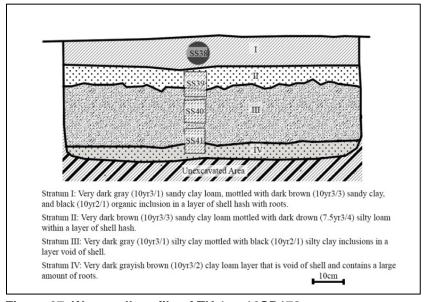


Figure 27. West wall profile of TU 1 at 16SB178.

The Southern Comfort site.

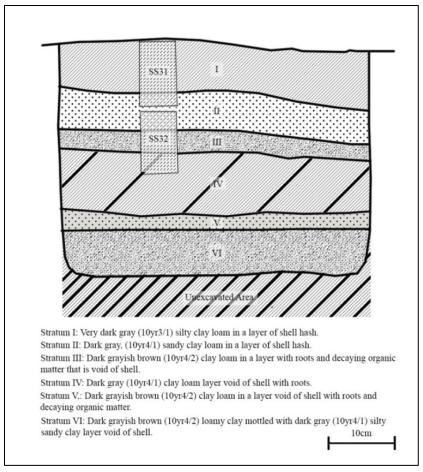


Figure 28. North wall profile of TU 2 at 16SB178.

The Southern Comfort site.

#### 5.3.4 Comfort Island (16SB174)

Like the Southern Comfort site to the south, the Comfort Island site is a linear-shaped beach deposit concentrated in at least two locations. This site is located on the north-facing shore of Comfort Island, approximately 100 meters east of Old Henry Bayou. The Comfort Island site is situated on a marsh island fringe in Chandeleur Sound, approximately 24 miles (38.8 km) east of the Breton Sound Marina in Hopedale, Louisiana. The site consists of wave-washed and secondarily deposited artifacts, oyster shell, and shell hash overlying a thick humic layer, with partially decayed and more recent organic debris. Ground cover primarily consisting of marsh grasses allowed for moderate to good surface visibility (Figures 29 and 30).

The Comfort Island site was also recorded during the MC252 oil spill response, when archaeologists with HDR, Inc. visited the island and observed oil along the northern shoreline (Cloy and Ostahowski 2015:6-756; HDR 2011:D-58). The presence of oil was described as moderate to very light (Cloy and Ostahowski 2015, Appendix 3:59). The site was initially visited in July of 2010 and subsequently revisited five times until October of 2012. Wave washed pottery sherds and historic debris were found on the surface among redeposited shell hash in two areas of the site. Three of the seven shovel tests excavated by HDR archaeologists produced cultural materials and bone fragments. The ceramics from the surface and shovel tests include Baytown plain, *var. unspecified*, and unidentified sand tempered sherds, indicating a Woodland and possibly Marksville period or later component (Cloy and Ostahowski 2015:6-758; LDA

2017). HDR archaeologists surmised that the Comfort Island site represents a redeposition of materials from a nearby and eroded site offshore, most likely Site 16SB135. Because of the lack of depositional integrity and disturbed condition of the cultural deposits, they recommended the site to be ineligible for inclusion on the NRHP (Cloy and Ostahowski 2015:6-759, Table 6-2).



Figure 29. Photograph of 16SB174.

From Area B facing west to Area A at the Comfort Island site.



Figure 30. Photograph of 16SB174.

From Area A facing north at the Comfort Island site, showing TU 1 at the lower right.

The LDWF transported the ULL crew and equipment to the Comfort Island site following the completion of fieldwork at the Southern Comfort site. The ULL crew did not detect any oil on the surface or during excavations at the Comfort Island site on January 29 and 30 of 2015. Fieldwork consisted of a controlled surface collection, one shovel test and the excavation of two 50 cm-by-1 meter units (Figure 31). The surface collection was separated into Areas A and B, which correspond with Locus A and Locus B in the HDR report and site record (Cloy and Ostahowski 2015:6-759). While a majority of prehistoric materials from Comfort Island and other sites in the region consist of pottery sherds, a single Kent projectile point, made of heat-treated gravel chert, was notably collected from Area B (Section 6.1).

The presence and depth of redeposited shell midden along the shoreline were assessed with a 2-cm diameter soil probe. Though oil was not detected by sight or smell, a varying amount of redeposited shell hash was observed in the soil probes, ostensibly from the eroded midden offshore. One shovel test was excavated in Area A. Although it was negative for artifacts, a dense shell hash was recorded in the upper two strata. More important, this shovel test revealed a layer of shell hash overlying live *Spartina* marsh grass and marsh muck, confirming that the shell had been recently deposited.

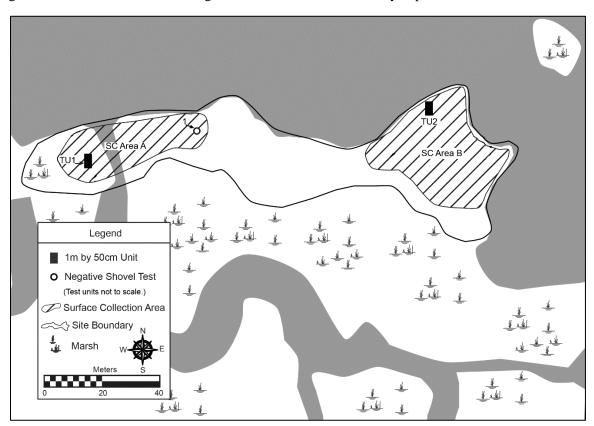


Figure 31. Sketch map of 16SB174.

Comfort Island site.

Placement of the test units was based on the results of surface inspection and the use of the soil probe to examine underlying stratigraphy. TU 1 was placed in Area A at the western end of the site, on the shoreline of a small cove that may provide some protection from wind and wave action. Excavation of TU 1 revealed three distinct strata (Figure 32). Stratum I was characterized by a very dense shell hash deposit lacking soil and roots. Stratum II was distinguished by a very dark grayish brown (10YR 3/2) clay loam within a deposit of dense shell hash, broken shell, and whole shell. Stratum III was void of shell and consisted of black (10YR 2/1) clay muck mottled with very dark gray (10YR 3/1) clay. Excavation of TU 1 ended at approximately 30 cm below surface, at the bottom of level 3, with the inundation of the unit.

Artifacts recovered from TU 1 included clear container glass, pieces of rubber, and pottery sherds (Section 6.1). The historic artifacts were recovered from levels 2 and 3, indicating disturbance or relatively recent redeposition.

TU 2 was placed at the eastern end of the site, two meters north of where the Kent projectile point was recovered. Excavation of TU 2 revealed four strata (Figures 33, 34 and 35). Stratum I was a very dense shell hash deposit without any discernible soil or roots. Stratum II was distinguished by a very dark gray (10YR 3/1) clay loam and roots within a dense deposit of crushed and broken shell. Stratum III was a very dark gray (10YR 3/1) clay loam in a dense deposit of broken and crushed shell.

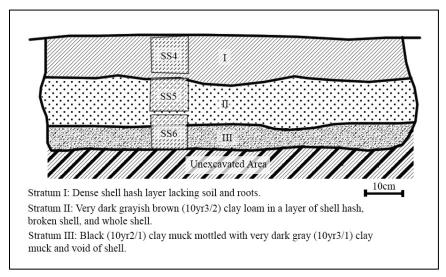


Figure 32. East wall profile of TU 1 at 16SB174.

Comfort Island site.

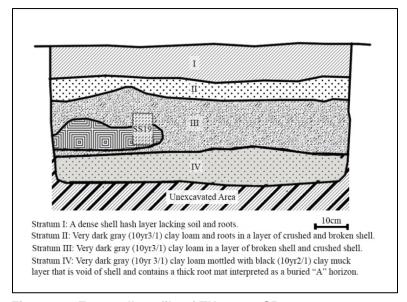


Figure 33. East wall profile of TU 2 at 16SB174.

Comfort Island site.

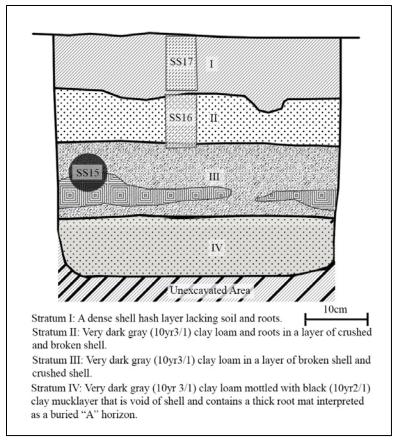


Figure 34. North wall profile of TU 2 at 16SB174.

Comfort Island site.

Stratum IV in TU 2 was void of shell and distinguished by a thick root mat that was interpreted as the top of a buried A horizon. Stratum IV consisted of very dark gray (10YR 3/1) clay loam and black (10YR 2/1) clay muck. Because it was void of artifacts, Stratum IV appeared to be a culturally sterile A horizon on which shell and cultural materials had been redeposited. Excavation of TU 2 was terminated at approximately 42 cm below surface.

Nineteen samples were collected from the Comfort Island site, including 12 special samples (Appendix A4). The special samples consist of three column samples from TU 1 and four column samples from TU 2, two soil samples from each unit, and a unit core sample from TU 2. Some of these samples were selected for chemical and elemental analyses to detect the presence of oil, as discussed in Section 6.



Figure 35. Plan view of TU 2 at 16SB174.

Comfort Island site.

#### 5.3.5 Site 16SB153

This unnamed site is, for the most part, submerged beneath Lake Borgne (Figure 36). Local residents refer to both this site and a nearby site (16SB47) immediately to the west as St. Malo. Site 16SB153 is situated along the southeastern shore of Lake Borgne, approximately 360 meters southwest of the mouth of Bayou St. Malo. Linear shell midden deposits, primarily *Rangia*, are visible along the shoreline. Two landward extensions of subsurface shell are extant beneath the marsh muck. SCAT teams did not report oiling of the shoreline in the vicinity of the site, which has already been impacted by subsidence (HDR 2011; Landry 2010). As such, Site 16SB153 was included in this study as the easternmost of two control sites.

Site 16SB153 was previously investigated by Goodwin & Associates, Inc. and, more recently, by Coastal Environments, Inc. (Labadia et al. 2007; Weinstein et al. 2012). The site was found to extend for nearly

600 meters along the shoreline and to contain two areas of deeply buried, intact shell midden along the lakeshore. A third area of intact shell midden lies offshore beneath the lakebed.

Troyville, Coles Creek, and Mississippian components are noted in the site record, along with subsequent historic occupations. Archaeologists with Coastal Environments, Inc. found evidence of a historic component that might be associated with the Filipino fishing village of St. Malo. Artifacts from the site also indicate an earlier historic component possibly associated with the *cimarrones*, a name used for groups of runaway slaves (Weinstein et al. 2012:30, 31). Their excavations revealed that portions of the midden were redeposited by storm surge, but that significant portions remain intact beneath the lakebed and shoreline. The pottery and radiocarbon dates obtained by Coastal Environments confirmed Baytown, late Coles Creek, and Mississippian components. Although now deeply buried, the site retains archaeological integrity and merits eligibility for inclusion on the NRHP (Weinstein et al. 2012:187–188).

The ULL crew conducted fieldwork at Site 16SB153 over three days, from April 7 through April 9, 2015. The Project Director piloted a LUMCON vessel to the site. Shovel tests were not excavated because previous investigations provided sufficient information on the locations of intact subsurface deposits. Fieldwork by the ULL crew consisted of a brief surface inspection, auger tests, soil probes, and the excavation of one 1-by-1 meter test unit (Figure 37). A sample of wave-worn pottery was collected from the exposed midden surface approximately 15 meters southwest of the test unit. Site stratigraphy was investigated with a bucket auger, which allowed the crew to locate the buried shell midden. The auger tests were quickly inundated by the water table, so a 160 cm section of auger pole was used as a probe to detect the buried midden. Shell midden was recorded at varying depths by six of seven probes, two of which were placed in the bottom of auger tests.



Figure 36. Photograph of location of TU 1 at 16SB153.

Facing east, showing the shoreline, unnamed site on Lake Borgne.

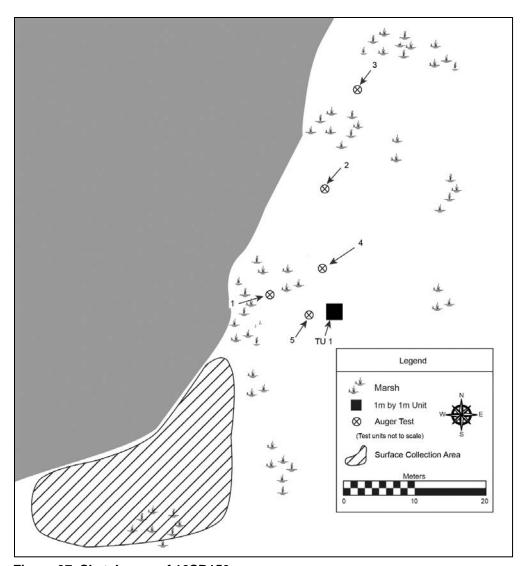


Figure 37. Sketch map of 16SB153.

Unnamed site on Lake Borgne.

Placement of the test unit was based on the results of the auger tests, soil probes, and surface inspection, but primarily informed by previous work conducted by Coastal Environments, (Weinstein et al. 2012). TU 1 was placed in an area of relatively higher elevation, where the buried shell midden was determined to lie within 80 cm of the surface. The test unit was excavated to 145 cm below surface and a total of 10 strata were recorded (Figures 38, 39 and 40). The upper 75 cm of overburden was not screened, because Coastal Environments had previously determined that it consists of recently deposited, culturally-sterile layers. Most of the shell midden deposit was screened through 0.25-inch (6.35 mm) mesh. An attempt was made to use 0.125-inch (3.2 mm) mesh in the field, but this was stopped due to the amount of clay in the soil matrix and time constraints.

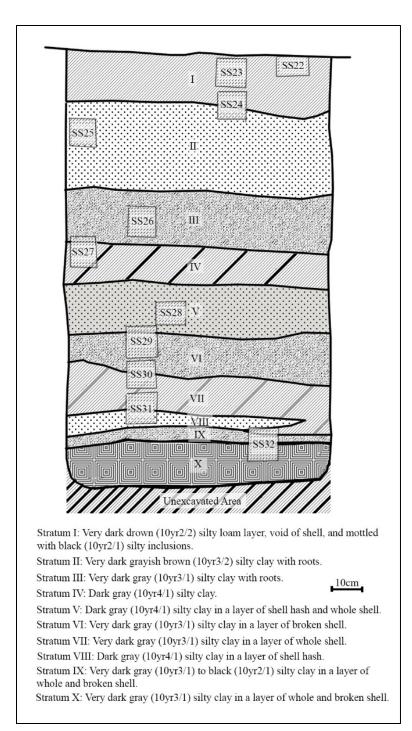


Figure 38. North wall profile of TU 1 at 16SB153.

Unnamed site on Lake Borgne.

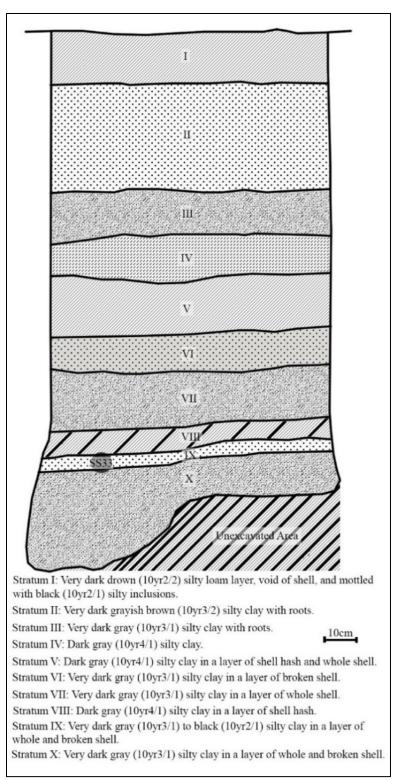


Figure 39. West wall profile of TU 1 at 16SB153.

Unnamed site on Lake Borgne.

The upper four strata of the test unit consisted of overburden. Stratum I was characterized as a layer of humus void of shell, with very dark brown (10YR 2/2) silt loam with black (10YR 2/1) mottling. Stratum

II also lacked shell and was made up of a very dark grayish brown (10YR 3/2) silty clay with roots. Stratum III was a very dark gray (10YR 3/1) silty clay, with fewer roots than Stratum II, but also void of shell. Although the distinction between strata III and IV was somewhat obscured by mottling, Stratum IV was a dark gray (10YR 4/1) silty clay that directly overlaid the shell midden.



Figure 40. Photograph of TU 1 at 16SB153.

Taken at the base of Level 5, unnamed site on Lake Borgne.

Strata V through X comprised the artifact-producing shell midden deposit. Stratum V was composed of dark gray (10YR 4/1) silty clay within a deposit of shell hash and whole shell. Stratum VI contained very dark gray (10YR 3/1) silty clay and dense broken shell deposit. In Stratum VII, a whole *Rangia* shell deposit with very dark gray (10YR 3/1) silty clay was encountered. Stratum VIII was a relatively thin layer not discernible in the east wall profile. It was composed of dark gray (10YR 4/1) silty clay within a dense deposit of shell hash. Stratum IX was a thin layer of very dark gray (10YR 3/1) to black (10YR 2/1) silty clay within a deposit of whole and broken *Rangia* shell. The final stratum (X), consisted of very dark gray (10YR 3/1) silty clay within a deposit of whole and broken *Rangia*. Excavation was terminated at 145 cm below surface. Although the use of a water pump with a 4½ inch diameter outlet had allowed excavation to this depth, increased inundation of the unit from the water table impeded further excavation.

Test unit 1 at Site 16SB153 stands out from the other sites assessed by this study in terms of the relatively large amount of shell tempered pottery (Section 6.1). A total of 33 samples were collected from the site (Appendix A5). Sixteen were field samples of surface collected materials or matrix from TU 1 that was water screened in the field through 0.25-inch (6.35 mm) mesh. Of the 17 special samples, 11 were column samples from TU 1, one was extracted as a unit core sample from TU 1, and five were soil samples processed in the lab through 0.25 inch and 0.0625-inch mesh screen. The 11 column samples were recovered from the TU 1 north wall profile. Six of these were collected from the overburden and five were from the underlying shell midden deposit. The objective was to collect samples from the overburden

as well as the underlying midden that could be tested for the presence of oil. Special samples were processed in the lab at ULL and subsamples were submitted for analyses to determine the presence of oil.

# 5.3.6 Scow Island Scatter (16SB182)

Scow Island Scatter is a linear-shaped beach deposit of *Rangia* and oyster shell on the eastern shore of Scow Island (Figure 41). The site is situated on the fringe of a marsh island in Chandeleur Sound, south of Drum Bay, approximately 15 miles (24 km) east of the Breton Sound Marina in Hopedale, Louisiana. Archaeologists with HDR, Inc. recorded the site in 2010 during the MC 252 oil spill response. Oil was observed at the site and conditions along the shoreline were characterized at that time as having light to very light amounts of oil. SCAT teams excavated an exploratory trench that was monitored by archaeologists, but it produced no artifacts (Cloy and Ostahowski 2015, Appendix 3:59; HDR 2011:D-62; LDA 2017).



Figure 41. Photograph of 16SB182.

Facing north, along the shoreline at the Scow Island Scatter site.

HDR, Inc. archaeologists revisited the site twice in 2011 and performed additional surface collections and subsurface investigations. They excavated nine shovel tests, all of which were negative for cultural materials (Cloy and Ostahowski 2015:6-782; LDA 2017). Of the 27 Native American pottery sherds collected from the surface during these site visits, the majority (n=16) were Baytown Plain, *var. unspecified*, with five identified as *var. Troyville*. Also collected from the surface were six sherds identified as Marksville Incised, *vars. Yokena* and *unspecified*; Marksville Stamped, *var. Troyville*; French Fork Incised, *var. Laborde*; Weeden Island Incised, *var. unspecified*; and an unidentified sand tempered sherd. The pottery is representative of Marksville, Baytown-Troyville, and possibly Coles Creek components, along with an historic component represented by a single stoneware sherd (Cloy and Ostahowski 2015:6-783). Because most of the sherds were wave worn and the shovel tests were all negative, the site was described as secondarily-redeposited shell and artifacts from an unidentified, eroded site offshore in Chandeleur Sound. Because of its disturbed condition and apparent lack of archaeological

integrity, Scow Island Scatter was recommended as ineligible for inclusion on the NRHP (Cloy and Ostahowski 2015:6-785; LDA 2017).

The LDWF transported the ULL crew and equipment to the Scow Island Scatter site on June 2 and 3, 2015. Fieldwork for the present study consisted of a systematic surface collection, six shovel tests, two auger tests, and the excavation of two 1-by-1 meter units. The surface collection was separated into areas A, B, and C; areas B and C had relatively higher frequencies of artifacts. Ground cover during the site visit was primarily *Spartina* marsh grass, allowing for moderate to good surface visibility in some areas (Figure 42).



Figure 42. Auger testing in ST 2 at 16SB182.

Facing northwest, showing the project director at the Scow Island Scatter site.

Shovel tests were excavated in each of the three areas, and an area of slightly higher elevation further from the shore, southwest of Area A (Figure 43). Four of the shovel tests were positive for cultural materials. ST 2, 4, 5, and 6 yielded a few Baytown Plain, *var. unspecified* pottery sherds and ST 4 produced a fragment of ferrous metal. Except for ST 1, varying accumulations of redeposited, broken shell, and shell hash were noted in each of the shovel tests. An auger test was placed at the bottom of ST 2 to investigate the possibility of deeply buried deposits. The auger test went to a depth of 120 cm below surface, but produced no artifacts, buried midden, or evidence of cultural deposits.

Placement of the two 1-by-1 meter test units was based on the results of the surface collection, shovel tests, and topography. The test units were excavated in areas B and C due to relatively higher artifact frequencies in these areas, the absence of linear shell heaps formed by wave action, and ground cover that suggested less recent disturbance. The excavation of TU 1 revealed four strata (Figures 44 and 45). Stratum I was comprised of a grayish brown (10YR 5/2) sandy loam within a dense shell hash deposit with roots. Stratum II was characterized by dark gray (10YR 4/1) silty clay loam heavily mottled with dark grayish brown (10YR 4/2) silty clay in a very dense deposit of broken shell and roots. Stratum III contained dark grayish brown (10YR 4/2) sandy clay loam with very dark grayish brown (10YR 3/2) silty clay mottles in a moderately dense deposit of whole and broken shell. The amount of roots increased in

Stratum IV, which consisted of a dark gray (10YR 4/1) to gray (10YR 5/1) clay loam that was void of artifacts and shell. Excavation of TU 1 ended in this stratum at 30 cm below surface with inundation of the unit. Artifacts recovered from TU 1 include Native American pottery sherds and pieces of container glass (Section 6.1).

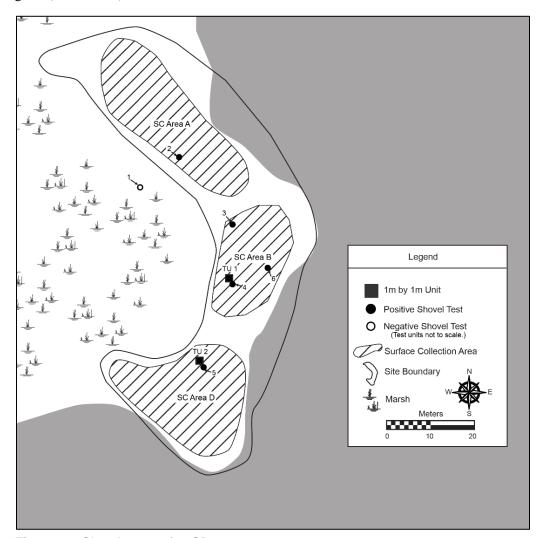


Figure 43. Sketch map of 16SB182.

Scow Island Scatter site.

An auger was placed in the floor of TU 1 to a depth of 180 cm below surface without producing evidence of buried archaeological deposits. Sediment from the auger test consisted of dark grayish brown (10YR 4/2) to gray (10YR 5/1) clay loam from approximately 30 cm to 80 cm below surface. A change in stratigraphy was apparent between 80 and 100 cm below surface, consisting of a heavy-mottled layer of black (10YR 2/1) clay muck and very dark grayish brown (10YR 3/1) silty clay. Although the remaining stratigraphy was difficult to interpret due to inundation, neither shell midden nor artifacts were encountered to a depth of 180 cm.

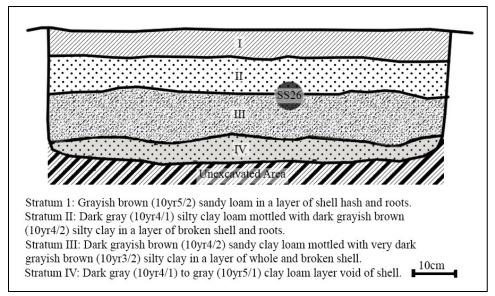


Figure 44. North wall profile of TU 1 at 16SB182.

Scow Island Scatter site.

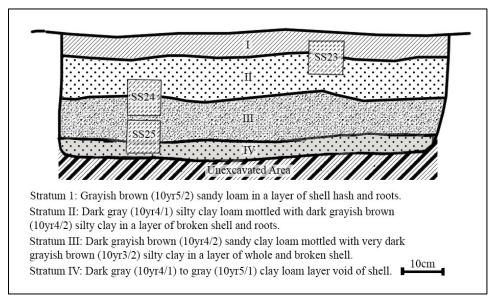
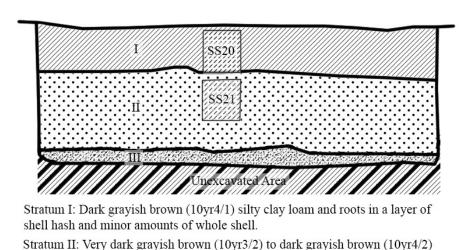


Figure 45. West wall profile of TU 1 at 16SB182.

Scow Island Scatter site.

The excavation of TU 2 revealed three strata and terminated in a sterile stratum (Figures 46, 47, and 48). Stratum I contained a dark grayish brown (10YR 4/1) silty clay loam and roots in a deposit of shell hash with small amounts of whole shell. Stratum II was a very dark grayish brown (10YR 3/2) to dark grayish brown (10YR 4/2) sandy clay loam with roots in a deposit of broken shell with lesser amounts of shell hash and whole shell. Baytown Plain, *var. unspecified* pottery sherds and container glass fragments were recovered from Strata I and II (Section 6.1). At 19 cm below surface, a black (10YR 2/1) silty clay with associated burned wood was encountered in the northeast quadrant of the unit. A fragment of burned wood, a pottery sherd, and unit core sample were obtained from the black silty clay.



sandy clay loam and roots in a layer of broken shell, shell hash, and whole shell. Stratum III: Dark gray (10yr4/1) silty clay mottled with gray (10yr5/1) silty clay,

Stratum III: Dark gray (10yr4/1) silty clay mottled with gray (10yr5/1) silty clay, dark brown (7.5yr3/4) sandy clay, and black (10yr2/1) clay inclusions. \_ 10cm \_

Figure 46. North wall profile of TU 2 at 16SB182.

Scow Island Scatter site.

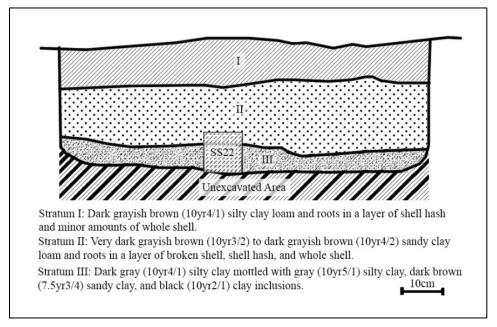


Figure 47. West wall profile of TU 2 at 16SB182.

Scow Island Scatter site.

Stratum III in TU 2 was void of shell and consisted of dark gray (10YR 4/1) silty clay with gray (10YR 5/1) silty clay mottles and dark brown (7.5YR 3/4) sandy clay and black (10YR 2/1) clay inclusions. Stratum III contained roots and was initially thought to lack both artifacts and shell. A crab trap was encountered at the base of level 3, however, protruding from the south wall and indicating a disturbed context for levels 1 through 3. The excavation of TU 2 ended at a depth of 30 cm below surface.



Figure 48. Photograph of TU 2 at 16SB182.

Scow Island Scatter site.

The combined results of shovel testing, auger testing, and test unit excavation at Scow Island Scatter confirmed that the shell midden is redeposited. It extends to 40 cm below surface at its deepest point. The artifacts collected from shovel tests and test units were found in disturbed contexts, but can provide information on site components and the potential impacts of oil. A total of 12 special samples were collected from TU 1 and TU 2 at Scow Island Scatter. These include four specimen samples of pottery sherds and charcoal, six column samples from TU 1 and TU 2, and two unit cores from TU 1 and TU 2 (Appendix A6).

#### 5.3.7 Redfish Slough (16LF293)

Redfish Slough is a shell midden and associated artifact scatter located on the southwest end of Philo Brice Island in Timbalier Bay, approximately nine miles (14.5 km) northwest of Port Fourchon. Archaeologists with HDR, Inc. recorded the site in 2011 as part of the MC 252 oil spill response. Exposed shell midden is visible at the surface in shoreline areas of the site. Other portions of the midden are covered with vegetation consisting of *Spartina* marsh grass, black mangrove, and saltwort, obscuring ground surface visibility in some areas (Figures 49 and 50). The presence of oil was described as moderate at the Redfish Slough site and along the shoreline to the north and south. SCAT crews reported heavier oiling of the shoreline and islands of Timbalier Bay to the south (Cloy and Ostahowski 2015:6-373, 6-375, Appendix 3:29; LDA 2017).

Surface inspection of the Redfish Slough site by HDR, Inc. produced a collection of wave-washed pottery sherds and faunal bone. Two distinct concentrations of materials were identified during their surface inspection. The larger concentration of artifacts and bone was delineated in the northwestern portion of the site and a smaller concentration was located just to the north. The pottery sherds were mostly Baytown Plain, including *var. Addis, var. Cataouatche*, and *var. unspecified*, with smaller amounts of

Coles Creek Incised *var. unspecified*; Evansville Punctated, *var. Rhinehart*; Bell Plain, *var. unspecified*; and Mississippi Plain, *var. unspecified*. The ceramic types indicate Transitional Coles Creek, Plaquemine, and Mississippian components. The bone included elements of white-tailed deer (*Odocoileus virginianus*) and other fauna (Cloy and Ostahowski 2015:6-374; LDA 2017).



Figure 49. Photograph of Area C at 16LF293.

Facing south, along the shoreline at the Redfish Slough site.



Figure 50. Photograph of Area B at 16LF293.

Facing north at the Redfish Slough site.

Archaeologists with HDR, Inc. excavated eight shovel tests, of which five produced cultural materials, including 17 sherds of Baytown Plain, *var. unspecified*. All of the artifacts from the shovel tests were recovered from redeposited shell hash. HDR determined that Redfish Slough is the product of redeposited materials from an undocumented, apparently now submerged, site in the vicinity. Although disturbance was noted in the shovel tests, their investigation did not fully assess the potential for deeply-buried, intact deposits. The eligibility of Redfish Slough for inclusion on the NRHP was consequently undetermined (Cloy and Ostahowski 2015:6-375, Table 6-2).

The ULL Project Director piloted a LUMCON vessel to the Redfish Slough on June 8 and 9, 2015. The fieldwork was interrupted by severe weather on the second day, so the crew made a return visit on June 30. The fieldwork consisted of a systematic surface collection, the excavation of nine shovel tests, and two 1 m-by-50 cm test units (Figure 51). The surface collection was divided into three areas that were designated A, B, and C. Areas A and B corresponded to the artifact concentrations previously noted by archaeologist with HDR., and Area C was located to the south, along the southernmost shoreline of the island. Artifacts collected from each area were labeled according to provenience. Pottery sherds and faunal bone were collected from Areas A and B, but Area C yielded only a small amount of pottery and no fauna. The selection of areas for excavation, though informed by the surface inspection, was constrained by large numbers of nesting birds that were present during the fieldwork.

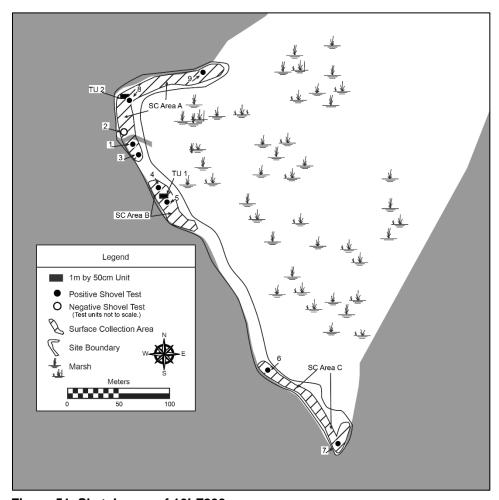


Figure 51. Sketch map of 16LF293.

The Redfish Slough site.

The shovel tests' locations were selected based on the results of the surface collection and topography. Shovel tests 1 through 3 were excavated in Area A; shovel tests 4 and 5 were excavated in Area B; and shovel tests 6 and 7 were conducted in Area C. Two additional shovel tests (ST 8 and 9) were excavated in Area A. Of the nine tests, eight produced artifacts and/or faunal materials and only ST 2 was negative. Varying amounts of broken shell and redeposited shell hash were recorded in each of the shovel tests. Test unit locations were determined by the results of shovel testing, the surface collection, and topography. TU 1 was excavated in Area B, adjacent to ST 5 (Figure 52). The placement of TU 1 beside ST 5 allowed the shovel test to be used as a well for a sump pump in order to drain the test unit during excavation. A second test unit was initially laid out in Area A, but a sudden thunderstorm interrupted the fieldwork and forced the crew to evacuate the island before excavation began. The second 1 m-by-50 cm unit (TU 2) was excavated at this location in Area A during a return site visit, on June 30.

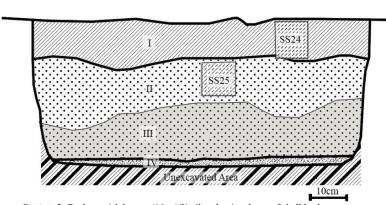


Figure 52. Photograph of TU 1 at 16LF293.

The Redfish Slough site.

Four strata were recorded in TU 1 (Figures 53 and 54). Stratum I was characterized as dark grayish brown (10YR 4/2) silty clay within a loose deposit of shell hash. Minor amounts of broken and whole shell were encountered near the base of Stratum I. Stratum II was very dark grayish brown (10YR 3/2) clay loam with dark grayish brown (10YR 4/2) silty clay mottles, black (10YR 2/1) silty clay inclusions, and broken and whole shell. The distinction between strata II and III was difficult to discern because of similarities in composition and shell hash that caused the profile walls to crumble.

In comparison to Stratum II, Stratum III lacked the prevalent black (10YR 2/1) inclusions. Stratum III was a very dark grayish brown (10YR 3/2) clay loam within a deposit of crushed, broken and whole shell. Roots and marsh grass were visible in Stratum III. Stratum IV was a very dark grayish brown (10YR 3/2) clay loam with black (10YR 2/1) clay mottles, marsh grass and abundant roots, but lacking shell. Stratum IV was consequently interpreted as a recently buried A horizon. The excavation of TU 1 terminated in Stratum IV at a depth of approximately 45 cm below surface in the southeastern half of the unit. Cultural materials, including Native American pottery sherds and historic artifacts, such as pieces of container glass, were collected from each level of TU 1 (Section 6.1).



Stratum I: Dark grayish brown (10y 4/2) silty clay in a layer of shell hash.

Stratum II: Very dark grayish brown (10YR 3/2) clay loam with dark grayish brown (10YR 4/2) silty clay mottles, black (10YR 2/1) silty clay inclusions, and broken and whole shell.

Stratum III: Very dark grayish brown ( $10 \mathrm{YR}\ 3/2$ ) clay loam in a layer of crushed, broken, and whole shell.

Stratum IV: Very dark grayish brown ( $10YR\ 3/2$ ) clay loam with black ( $10YR\ 2/1$ ) clay mottles, marsh grass and roots, but lacking shell.

Figure 53. North wall profile of TU 1 at 16LF293.

The Redfish Slough site.

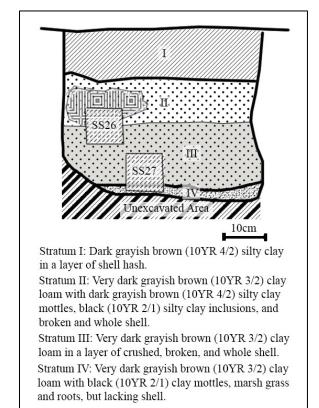


Figure 54. East wall profile of TU 1 at 16LF293.

The Redfish Slough site.

The LAPAL crew returned to the site on June 30 and excavated two additional shovel tests (ST 8 and 9) and a second 1 m-by-50 cm unit (TU 2) in Area A. ST 8 allowed for the placement of a bilge pump during the excavation of TU 2. As in TU 1, TU 2 also revealed four distinct strata. At the bottom of Level 1 and throughout Level 2 the excavation revealed a layer of roots, shell hash, and decaying organic material in a dark brown (10YR 3/3) silty clay (Stratum II). Stratum I consisted nearly entirely of redeposited shell hash (Figure 55). Stratum III was made up of dense shell hash within a very dark grayish brown (10YR 3/2) silty clay. A piece of container glass was collected from Level 4 in Stratum III, indicating recent redeposition with the layer of shell hash (Section 6.1).

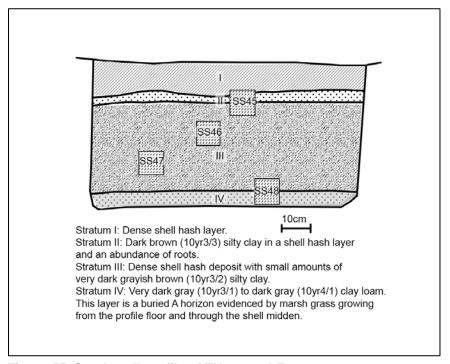


Figure 55. South wall profile of TU 2 at 16LF293.

The Redfish Slough site.

Stratum IV in TU 2 appeared to be a recently buried A horizon, as indicated by marsh grass found at the base of Level 4 and throughout Level 5. Fragments of iron wire were recovered from Level 5. The soil consisted of a very dark gray (10YR 3/1) to dark gray (10YR 4/1) clay loam. TU 2 terminated at 50 cm below surface. An auger test was placed at the bottom of TU 2 to a total depth of 210 cm below surface (160 cm beneath the unit floor) without encountering additional artifacts or shell midden. Twenty special samples were collected from TU 2 and the previously excavated TU 1. These included ten column samples, one unit core, three soil samples, and six samples of pottery and faunal bone (Appendix A7). These samples were transported to the lab at ULL for processing and analysis.

# 5.3.8 Acorn Mounds (16SB185)

Acorn Mounds is located on an eroded marsh island in a remote area of St. Bernard Parish, west of Chandeleur Sound and south of Drum Bay. Keelboat Pass is located on the eastern side of the island. There are three earthen mounds arranged in a triangular configuration at the site. The mounds appear to define the boundaries of a centrally located plaza. An unnamed channel that crosses a portion of the island appears to demarcate the northern site boundary. This relict channel may be an abandoned tributary of Bayou La Loutre. The triangular configuration of the mounds, with the bayou demarcating the hypotenuse, suggests this waterway was present when the mounds were constructed. The mounds appear

today as three relatively low crests, rising less than one meter above the surrounding marsh grass. The mounds are visible at a distance due to groves of wax myrtle (*Myrica cerifera*) on the summits (Figure 56).



Figure 56. Mounds at 16SB185.

View to the south, Mounds B, C, and A (left to right) at the Acorn Mounds site.

Although the earthen mounds are visible from the surrounding marsh, the site was only recently recorded during the MC252 oil spill response. An 1845 General Land Office Survey plat map and a later U.S. Coastal Survey map of 1858 to 1859 show Indian mounds at this location. William McIntire (1958), however, did not include this site in his archaeological survey of the Mississippi Delta. In fact, the site appears to have escaped the attention of archaeologists for the remainder of the twentieth century until a site record was completed as a result of the MC252 oil spill response in 2011 (LDA 2017). Surprisingly, Acorn Mounds is not the only mound site in the region to have gone unrecorded for so long. Live Oak Bayou Mounds (16SB186), located just 3.5 miles (5.6 km) across Drum Bay to the north, was also first recorded in 2011 (Cloy and Ostahowski 2015:924, 929).

Though the layout of the mounds and plaza at the Acorn Mounds site is easily discerned, the size and boundaries of the site have yet to be adequately determined. Moving clockwise from the westernmost mound, the midpoints of Mounds A and B are approximately 103.5 meters apart, Mounds B and C are approximately 64 meters apart, and approximately 58 meters separate Mounds C and A. The entire mound and plaza precinct, then, is about 1 hectare (2.5 ac) in size, with the plaza between the mounds encompassing approximately 0.25 hectare (0.6 ac). The entire site was recorded as covering a slightly larger area, or approximately 1.35 hectare (3.3 ac) (Cloy and Ostahowski 2015:922; LDA 2017). This was not informed by subsurface investigations, however, so the actual area that was formerly inhabited beyond the mound and plaza complex may have been considerably larger.

The landform where the Acorn Mounds site is located has subsided and overburden would have buried any former living surfaces. The size of the surrounding marsh island has also been drastically reduced due to shoreline erosion. Any cultural deposits potentially located around the mounds, in the plaza or outside of the mound-and-plaza precinct have most likely subsided with the surrounding landform and are now deeply buried beneath marsh sediment. Using the bayou to demarcate the northern site boundary and the

locations of surface collected artifacts on the shoreline of Keelboat Pass to represent the possible eastern limits, the site may actually cover 4.5 hectares (11 ac) or more. Systematic augering or deep coring might be the most efficient and only feasible method of investigating the size and internal structure of the site.

The extent of oil from the MC252 spill at Acorn Mounds was described as heavy to moderate along the shoreline to the east and light to very light along the shoreline to the north and south (Cloy and Ostahowski 2015, Appendix 3:59). When archaeologists with HDR, Inc. initially visited the site, they collected pottery sherds from the surface along Keelboat Pass. Subsequent visits produced a total of 35 artifacts, bones of fauna, and a human tibia from the shoreline to the east and southeast, approximately 120 to 650 meters from the mound and plaza precinct (Cloy and Ostahowski 2015:6-803). A pedestrian inspection of the mounds produced no artifacts, although ground surface visibility was very limited. The HDR crew excavated one shovel test into the summit of each mound. The stratigraphy revealed accumulations of natural marsh soils over a lighter colored soil interpreted as possible mound fill. There were no cultural materials from any of the shovel tests, which were excavated to only 50 cm below surface before filling with water. Subsidence and deltaic sedimentation have almost entirely buried the mounds and surrounding landscape, so the absence of artifacts was not unusual.

The surface collected artifacts from the shoreline to the east and southeast of Acorn Mounds only indirectly suggest a Woodland period cultural affiliation for the site or other sites in the area. Pottery from the shoreline included Marksville Stamped, *var. Troyville*; Wakulla Check Stamped, *var. unspecified*; and Baytown Plain, *var. Marksville* and *var. unspecified*. Cloy and Ostahowski (2015:6-804) suggest a possible Marksville affiliation, around the time the La Loutre lobe of the St. Bernard delta complex might have been initially inhabited (Kesel 2008; Törnqvist 1996). Some Marksville types, including *var. Troyville*, are known to date into the first century of the Baytown period, so a relatively later Troyville affiliation seems just as plausible, around the time the La Loutre lobe was abandoned and no longer active (Cloy and Ostahowski 2015:6-804). The association of the mound and plaza precinct with artifacts collected from the shoreline, 120 to 650 meters away, remains to be established. The general layout and size of the mound and plaza complex is generally characteristic of Coles Creek mound sites in the Lower Mississippi Valley that date from approximately 700 CE (Roe and Schilling 2010).

The fieldwork at Acorn Mounds was conducted in two site visits, on August 4 and 5 of 2015, followed by a return visit on September 6 and 7 of 2015. The LDWF provided transportation on the first two days. The Project Director piloted a LUMCON vessel on the return visit in September. During the initial site visit, equipment and gear were transported by canoe from the landing area at Keelboat Pass to the mounds and plaza south of the bayou, a distance of more than 250 meters. On the second visit, the LDWF canoe was not available, so the crew carried the equipment and gear across the marsh island along the south bank of the bayou. When the crew first arrived at the site in August of 2015, they observed oil in the form of tar balls on the shoreline where the bayou enters Keelboat Pass, approximately 175 meters east of Mound B (Figures 57, 58 and 59). Samples of this oil were collected from the surface.

A total of seven shovel tests and two test units were excavated at Acorn Mounds during the two site visits, along with five auger tests and seven soil cores (Figure 60). During the first site visit, the crew excavated six shovel tests along the shoreline east of the mounds, where oil was observed on the surface. A seventh shovel test was placed north of Mound A. None of the shovel tests yielded cultural materials, although several were excavated as deep as 80 cm beneath surface. A dense layer of oyster shell was encountered in ST 2, 3, and 6 at approximately 60 to 80 cm beneath surface. A 4-inch (10 cm) diameter bucket auger was used to investigate these deeply-buried deposits near ST 6 and in an area along the bayou, north of the mounds. The heavily saturated condition of the soils made it difficult to collect samples or examine stratigraphy with the bucket auger.



Figure 57. Oil on the shoreline at 16SB185.

Facing west, east of the Acorn Mounds site.



Figure 58. Shoreline east of 16SB185.

Facing northwest, research assistant is standing near ST 2 and 3.



**Figure 59. Bayou north of 16SB185.** View to the west, Mound A at the Acorn Mounds site on the left.

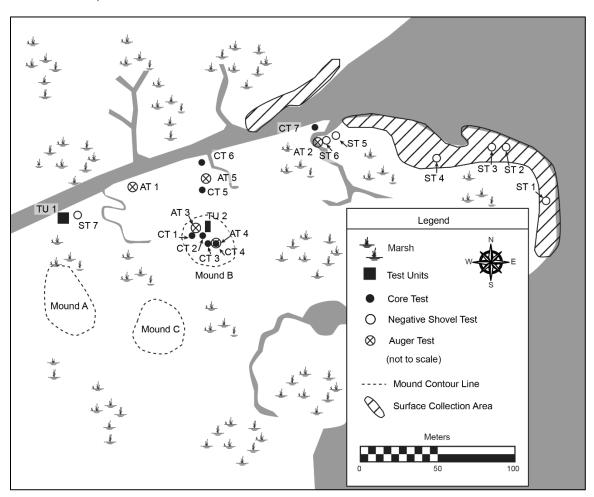


Figure 60. Sketch map of 16SB185.

Acorn Mounds site.

Dense deposits of oyster shell further obstructed auger testing. This deeply-buried layer of oyster shell was encountered along the south bank of the bayou north of the mounds, at depths of more than one meter beneath the surface. An auger rod extension served as a probe to investigate the areal extent of the shell deposit. The soil probes, auger tests, and negative shovel tests suggested that cultural deposits contemporaneous with the construction and use of the mounds are most likely subsided and deeply buried beneath alluvium and marsh sediments. Cultural deposits may be buried beneath more than 1.5 meters of overburden in the area north of the mounds and along the bayou. This would account for the absence of artifacts on the surface in this area. Additional testing at greater depths is required to substantiate this hypothesis.

TU 1 was excavated approximately 30 meters north of Mound A and just south of the bayou in order to investigate buried deposits of oyster shell and potentially associated cultural deposits in this area. Based on previous investigations at similarly laid out mound sites in south Louisiana, shell midden and residential midden contemporaneous with mound construction are commonly located just outside of the mound-and-plaza precinct and adjacent to a nearby body of water (Rees 2007:90-91; Roe and Schilling 2010:160). Located approximately 300 meters west of the shoreline where oil was observed, TU 1 was also intended to investigate whether tidal action had transported oil along the bayou and into the northwestern portion of the site.

TU 1 was begun as a 50 cm-by-1 meter unit and was expanded to the south into a 1-by-1 m unit (Figures 61 and 62). It was excavated to a depth of 145 cm below surface, through layers of alluvium that had accumulated during the historic period, into a dense deposit of oyster shell that was void of artifacts. Increased inundation of the unit prevented excavation beneath the layer of oyster shell. The shell appeared to be a natural accumulation, possibly dating from the historic period. It may have formed as an oyster bed or as a product of historic oyster harvesting that silted over. Storm surges moving across the marsh island may have alternatively deposited this layer of shell along the south bank of the bayou. A combination of subsidence and deltaic sedimentation would have subsequently buried the dense concentration of oyster shell.

The upper 82 cm of overburden in TU 1 was excavated without screening, although small pieces of blue plastic were noted. ST 7 had indicated that the overburden was void of artifacts in this area and probing had revealed the presence of the buried layer of shell. This dense concentration of shell was encountered at 85 to 92 cm below surface in TU 1. The overburden was made up of four strata of silty clay and clay loam that were somewhat difficult to distinguish. These ranged from a very dark grayish brown (10YR 3/2) silty clay with strong brown (7.5YR 4/6) clay loam mottles in Stratum I, to a more homogeneous, very dark gray (10YR 3/1) silt clay loam in Stratum IV. The dense layer of oyster shell was in a matrix of very dark gray (10YR 3/1) to dark gray (10YR 4/1) clay muck. This layer, Stratum V, continued to a depth of 145 cm below surface in TU 1. The excavation was terminated at this depth due to inundation of the unit, despite the use of a pump. No artifacts were recovered from Stratum V, although several small pieces of plastic were found in the upper strata of TU 1 (Section 6.1).

A 1-by-0.5 m test unit (TU 2) was excavated into the northern flank of Mound B during the second visit to Acorn Mounds. The purpose of this unit was to investigate potentially intact mound deposits and to collect samples to determine if oil was present. TU 2 was excavated to a depth of 20 cm below surface, into a very dark grayish brown (10YR 3/2) to dark grayish brown (10YR 4/2) saturated clay loam (Figure 63). The excavation of TU 2 was terminated when severe weather and a small craft advisory forced the crew to quickly leave the island. The unit was backfilled but two soil samples were collected from the south half of levels 2 and 3.

A special soil coring technique was applied at Acorn Mounds because of the deeply buried condition of the deposits (Figure 64). A 2-inch diameter, hammer-driven core with a butterfly valve and internal aluminum sleeve was used to collect samples from depths that could not be easily accessed by test unit

excavation (Section 5.2.2). This proved to be a more effective method of sampling saturated soils in subsided and deeply-buried contexts. Seven cores were collected during the second site visit. Cores 1 through 4 were extracted from Mound B. Cores 5 through 7 were from areas north and northeast of Mound B (Section 6.2).

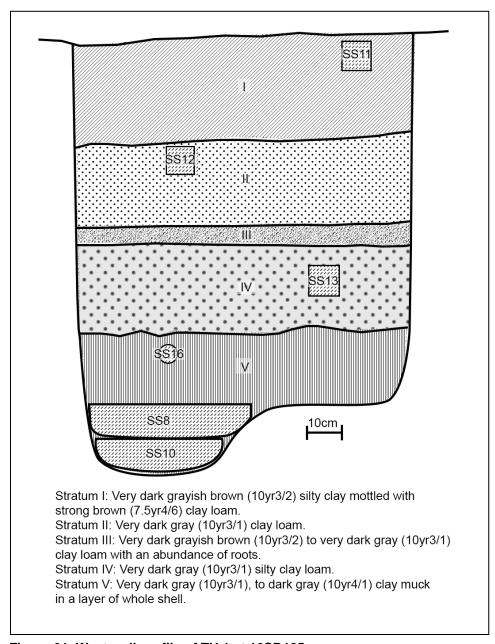


Figure 61. West wall profile of TU 1 at 16SB185.

Acorn Mounds site.

The fieldwork at Acorn Mounds resulted in the collection of a total of 36 special samples, including 17 samples from the seven core tests, each sealed in 12-inch sections of 2-inch diameter aluminum sleeve (Appendix A8). Two soil samples were collected from the surface of the shoreline of Keelboat Pass and a third soil sample was collected from 20 cm below surface in ST 2. Five samples of soil and shell were recovered from auger tests along the bayou and shoreline. Two soil samples were collected from TU 2

and TU 1 produced three soil samples, four column samples and two unit core samples. The analysis of samples from Acorn Mounds and other sites is discussed in the Analytical Results (Section 6).



Figure 62. TU 1 at 16SB185.

At 92 cm below surface, Acorn Mounds site.

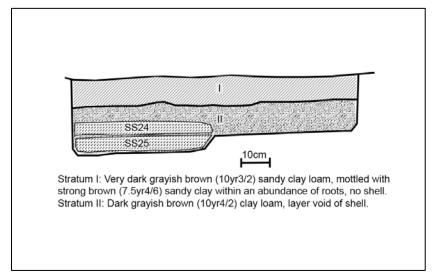


Figure 63. East wall profile of TU 2 at 16SB185.

Acorn Mounds site.



**Figure 64. Coring on Mound B at 16SB185.**Project Director Samuel Huey using the hammer driven core at Acorn Mounds site.

# 5.4 Analytical and Laboratory Methods

The Project Director transported all samples and collections to the Louisiana Public Archaeology Lab at ULL for processing and preparation for analysis. The standard procedure for processing of collections was to avoid washing all artifacts and ecofacts, whether special samples or collected by standard field techniques. After they were visually inspected, the soil samples, column samples, and unit core samples were subsampled or subdivided, as needed, on metal trays or through clean geological sieves. Sample matrices were initially sorted through 0.25-inch (6.35 mm) wire mesh or 4.75 mm geological sieve in the lab. Subsamples of artifacts, ecofacts and matrix extracted by this procedure were then sorted, identified, tallied and weighed. The remaining matrices from some special samples, such as bulk soil samples, were then water-screened through 0.0625-inch (1.6 mm) wire mesh or 1.0 mm geological sieve.

Metal tools and trays were used for the processing of all samples. The tools and trays were regularly cleaned in between uses with Dawn detergent and clean water. When necessary for identification and analysis, a natural-filament (non-synthetic) brush was used to remove sediment or matrix adhering to artifacts or ecofacts. Field samples were repackaged in cloth or paper bags and stored in acid-free boxes on metal shelving. Special samples were repackaged in aluminum foil envelopes and cloth bags, or kept in their original packaging. Field inventory forms, catalog records, sample logs, photo logs, shovel test and test unit records, and site sketch maps were organized in a binder for each site. The methods for selecting individual samples for analysis are described in the following section, followed by the methods

used for the chemical characterization of hydrocarbons, elemental analysis, absorbed residue analysis and radiocarbon dating.

## 5.4.1 Classification and Sample Selection

All artifacts and ecofacts were sorted and classified by major categories of material. These included Native American ceramics, lithics, faunal bone, and shell. As described in the results section of this report, pottery sherds made and used by Native Americans were by far the most common class of artifact recovered from the sampled sites. The classification and analysis of pottery involved the identification of paste or fabric, temper or inclusions, surface treatment, decoration, and whenever possible, vessel form (Ortner et al. 1993:68, 76, 127; Rice 1987:4-5). Diagnostic pottery types were identified using the typevariety system developed for the Lower Mississippi Valley (Brown 1998; McGimsey 2003a; Phillips 1970; Weinstein 2000; Williams and Brain 1983). Native American pottery sherds were classified and cataloged according to type-variety by provenience, described, counted and weighed. Very small sherd fragments, or "sherdlets," too small for identification of type (<0.5inch) were counted, weighed and classified as indeterminate type.

As expected at coastal sites in the delta, relatively small amounts of lithic artifacts were collected. Lithics were classified, described, and cataloged according to material, such as gravel chert, and diagnostic types for the Lower Mississippi Valley and Gulf Coast (McGahey 2000; Webb 2001). Thermal alteration, the presence of cortex and stage of modification were also noted. Historic artifacts, such as glass, ceramic, and metal were classified, described, and cataloged by material and type. Glass shards were relatively common and were described by color and shape. Container glass included all curved glass shards that appeared to be pieces of bottles, jars, or other containers. Flat glass included what might be pieces of windowpane or flat portions of large containers. Historic ceramics were relatively less common and were also categorized according to standard types (Noël Hume 1976, 2001). Metal artifacts were classified by material, such as iron, and categorized whenever possible by type or functional category.

A. James Delahoussaye at ULL analyzed samples of faunal bone from each of the eight sites for the identification of element and taxon. Faunal analysis involved recording the number of identified specimens (NISP) and bone weight by provenience for each taxon, along with evidence of burning, other modification, and whole or fragmented size. Bone not identifiable by taxon or element due to fragmentary condition was classified as unidentified bird, unidentified fish, unidentified mammal, unidentified reptile, or unidentified specimen. Taxon and element were also recorded for samples of fauna submitted for radiocarbon analysis. Appendix G presents the results of the faunal analysis.

Soil matrix, with and without varying amounts of whole, broken, and crushed mollusk shell comprised the largest volume of samples from all of the assessed sites. Two of the most ubiquitous types of bivalve mollusk shell in the region, *Rangia cuneata* and *Crassostrea virginica* (Eastern oyster), were commonly encountered. Samples of mollusk shell were collected when present in test units and shovel tests. Whole, broken, and crushed *Rangia* and oyster shell were collected in soil samples, column samples and unit core samples, but have not been individually catalogued or identified by taxon. An important distinction was made between whole bivalve shells and highly fragmented shell hash, providing evidence of redeposition by shoreline erosion and tidal action. Special samples containing soil matrix and shell were stored in sealed containers in a refrigerator, rather than room temperature or open-air environment, to retard potential off-gassing of contaminated samples. Once ceramics and other artifacts were sorted and removed, special samples were returned to refrigeration.

The PI and Project Director developed a system for analytical identification (ANID) in sorting and extracting specimens and sub-samples for further analysis. The ANID system allowed researchers to record provenience and track individual samples throughout the analytical process, while concealing information from analysts that might otherwise introduce bias to the interpretation of results. Each ANID

is 6 to 7 characters, consisting of three or four letters followed by three numbers. The first two letters identify the parish in which the site is located. The third letter designates a specific site, in the order visited by the Project Director and crew. The fourth letter distinguishes different specimens collected from the same provenience. Last, the numbers at the end of the ANID represent the unique SS or FS number assigned to that sample in the field, which is associated with a specific provenience. The PI and Project Director provided analysts with the ANID and sample description, but usually provided no other information on context. No information was provided on the potential for sample contamination by oil, dispersant or other chemicals, unless specifically requested as needed by the analyst.

As previously discussed, the potential effects of oil were examined through the chemical characterization of hydrocarbons, elemental analysis, absorbed residue analysis, and radiocarbon dating, including sample pretreatment. Because the different analytical techniques were in many respects complementary, the PI and Project Director established seven criteria in selecting samples for further analysis (Table 5). The seven criteria were: (1) type of material to be analyzed, (2) preliminary physical inspection, (3) quantification of the sample, (4) matrix or associated deposits, (5) depth of the sample in relation to confirmed or suspected hydrocarbon contamination, (6) sample provenience, and (7) any previous results of other analyses, particularly in determining the potential presence and source of hydrocarbons.

Selection of the type of material (Criteria 1) was dependent to some degree on the specific analytical technique. Gas chromatography-mass spectrometry (GC/MS) has been successfully used to determine the presence of hydrocarbons and fingerprint oil to its source in samples of soil from marsh environments (Iqbal et al. 2008; Mendelssohn et al. 2012). Compositional analyses and trace-element analysis techniques, such as neutron activation analysis (NAA) and X-ray fluorescence (XRF), have been applied to artifact classes such as ceramics and lithics in determining geographic source (Glascock 2008; Glascock and Neff 2003; Steponaitis et al. 1996). Samples of pottery from archaeological contexts can be analyzed for absorbed residues related to use and taphonomic processes (Barker et al. 2012; Reber 2012, 2013; Reber et al. 2010). Radiocarbon dating is based on the measurement of Carbon-14, a radioactive isotope of carbon found in organic materials such as charcoal, wood and bone (Beta Analytic, Inc. 2017).

Because hydrocarbons, dispersants, and other contaminants may interact with various materials in different ways, it was essential to establish if there was any evidence for sample contamination (Criteria 2). Preliminary techniques for determining the presence of oil on artifacts, ecofacts, and soil samples include inspection in the field and lab. Observable evidence includes an odor of petroleum and visual identification of oil or an oily sheen when submerged in water. As with tactile inspection, these techniques are generally subjective and provide no reliable indication of the absence of hydrocarbons, the source of the oil, chemical composition, quantification, or relative measure of contamination, including the presence of trace amounts of oil, or period of time since exposure to hydrocarbons. Visual, olfactory, and tactile inspection of samples in the field and lab are only as a preliminary step in determining the presence of oil. Because trace amounts of hydrocarbons may not be perceptible, determination of the *absence* and absolute quantification or relative amount of oil must ultimately rely on chemical analyses.

Table 5. Criteria guiding sample selection

	Chemical Characterization of Hydrocarbons	Elemental Analysis	Absorbed Residues	Radiocarbon
1. Material	Sediment, ecofacts	Ceramics, lithics	Ceramics	Wood charcoal, shell, and/or fauna
2. Inspection	Odor, color, and texture	Odor, appearance, evidence of residue	Odor, appearance, evidence of residue	Odor, appearance, evidence of residue
3. Quantification	Minimum sample size of 10 -20 g dry	Minimum sample size requirement of 1 square inch for ceramics	Minimum sample size requirement	Minimum sample size requirements for AMS dating; sample to be subdivided into halves
4. Matrix	Hydrocarbon contamination to be determined; presence or absence of artifacts or ecofacts in matrix	In situ deposits preferred; matrix suspected of, or tested positive for, hydrocarbon contamination	In situ deposits preferred; matrix suspected of, or tested positive for, hydrocarbon; hydrocarbon contamination to be determined	In situ deposits preferred; matrix suspected of, or tested positive for, hydrocarbon contamination
5. Depth	Samples from upper and lower strata	From levels suspected to be, or tested positive for, hydrocarbon contamination	Hydrocarbon contamination to be determined	From levels suspected to be, or tested positive for, hydrocarbon contamination
6. Provenience	Different proveniences at each site	Different proveniences at more than one site testing positive for hydrocarbons; control sample required	Different proveniences at more than one site testing positive for hydrocarbons	Different proveniences at more than one site testing positive for hydrocarbons
7. Prior results	Additional samples selected based on results of prior analyses	DES analysis indicates contamination of sampled context	DES analysis indicates contamination of sampled context	DES analysis indicates contamination of sampled context

Sample selection is also dependent on quantification (Criteria 3), because analysts have required and preferred minimal sample sizes. Though the preferred sample size for determining the presence and source of hydrocarbon by GC/MS ranges from 10 to 20 grams of dry soil, the minimum requirements for Accelerator Mass Spectrometry (AMS) dating of organic samples is considerably smaller. The constituent matrix from which a sample is extracted or associated is also relevant to this study (Criteria 4). For example, the selection of pottery samples should take into account the *in situ* recovery of sherds in association with a soil matrix that tested positive for hydrocarbon contamination, compared to surface contexts or association with a soil matrix that tested negative for hydrocarbons. The depth and provenience of samples are important for similar reasons (Criteria 5). Determining the presence or absence of hydrocarbons in samples from the upper and lower strata of a test unit may shed light on the permeation of oil into archaeological contexts and potential effect on site formation processes. Analysis

of soil samples from stratigraphic columns will assist in determining if hydrocarbons have vertically infiltrated archaeological deposits and, if so, to what depth.

To examine the effects of oil on intact (primary) archaeological deposits, as well as secondary, redeposited materials, it is also necessary to know the provenience of each sample (Criteria 6). Any previous results of GC/MS analysis in determining the presence or absence of hydrocarbons were important in selecting samples for additional analysis. As criteria 4 through 6 depended to some extent on either the suspected or confirmed presence of oil, analysis of samples for the chemical characterization of hydrocarbons became a priority for subsequent decisions regarding sample selection (Criteria 7). Budgetary constraints for the various analytical methods represented an eighth consideration not listed here, but inevitably taken into account in determining the number of samples to be submitted and deciding whether to submit additional samples.

# 5.4.2 Chemical Analysis

Because of the limitations and subjective nature of visual, olfactory, and tactile inspection, the chemical analysis of samples by GC/MS is the most thorough, reliable, and efficient method for determining the presence or absence of crude oil. GC/MS can also provide information on the chemical composition of contaminants, quantification or relative measure of contamination, and geographic source of hydrocarbons through oil source fingerprinting. Edward Overton and research associates in the DES laboratory at LSU have developed the capability to chemically characterize oil in order to identify the likely source of origin, including oil that originated from the MC252 oil spill (Iqbal et al. 2008; Mendelssohn et al. 2012).

Previous investigations have addressed the presence of hydrocarbons and dispersants in sediments on the Gulf Coast (Zuijdgeest and Huette 2012), but there has been no previous study of the presence and potential effects of oil and dispersants in archaeological deposits at coastal sites. The weathering of oil residues results in a loss of chemical information for definitive fingerprinting as to source. Chemical analysis by GC/MS can determine whether or not hydrocarbons from archaeological contexts can be traced to source (Edward Overton, personal communication March 14, 2014). This includes samples from intact (primary) and deeply-buried archaeological contexts, as well as secondary, redeposited contexts. Chemical analysis of special samples obtained from test unit wall profiles and floors might determine if hydrocarbons have permeated vertically into archaeological deposits and if so, to what depths.

Special samples made up of soil matrix, often with variable amounts of shell fragments, were selected for this analysis. Collection procedures adhered to the previously described methods in order to avoid inadvertent contamination outside of the specific archaeological contexts. The selection of samples for testing was guided by the criteria outlined in the previous section. Negative results for the presence of oil were regarded as just as important in guiding the selection of additional samples for GC/MS and other analyses. The potential identification of oil with no known connection to MC252 was likewise important, because it would suggest the presence of hydrocarbons in the archaeological record from other sources.

Samples submitted for chemical analysis for the presence of hydrocarbons were typically subdivided in the laboratory into subsamples. Clean metal tools were used to separate shell, artifacts, organic materials and soil matrix. Artifacts and subsamples were then assigned individual ANID and set aside for further analyses depending on the results of the initial GC/MS procedure. To avoid delays and potentially detrimental temperature fluctuations, the Project Director delivered samples to the DES Lab at LSU in Baton Rouge by automobile. Twenty-eight samples from all eight sites were submitted to the DES laboratory for analysis by GC/MS (Appendix B).

The presence and quantity of oil were determined by concentrations of petroleum hydrocarbon analytes (Table 6; Appendix C). This allowed for the qualitative and quantitative oil source fingerprinting of

samples. As described by Meyer et al. (2017), petrogenic compounds include a range of "parent aromatic hydrocarbons" with four groups of oil biomarkers. "These oil biomarkers are routinely used for oil source fingerprinting and include the triterpanes (including hopanes), diasteranes and regular steranes,  $14\beta(H)$ -steranes, and the triaromatic steroids" (Appendix C; Meyer et al. 2017). Once a sample was received by the DES lab, the extraction procedure involved sub-sampling of homogenized soil samples to produce concentrated extracts. Chemical characterization of samples was then conducted by a GC/MS method specifically developed for detecting and quantifying compounds commonly associated with oil spills (Appendix C; Meyer et al. 2017).

The extraction procedure, analytical standards, instrumental analysis, and data processing methodologies are described in detail in Appendix C. Meyer et al. (2017) analyzed all of the samples that were submitted as part of this study to determine whether any of the oil contained in the samples was a positive match with oil from the MC252 spill. This was accomplished by three separate oil source fingerprinting techniques. Oil biomarkers of the sample profiles were compared to the MC252 source through qualitative comparison, diagnostic biomarker ratio analysis and chemometrics. These techniques are further described in Appendix C. The results of the chemical characterization and oil source fingerprinting of samples are presented in Section 6.3, and supporting data in Appendix C.

Table 6. Petroleum hydrocarbon analytes targeted in the chemical characterization of oil

Anthracene	Fluoranthene	C-1 Phenanthrenes/Anthracenes
Benz[a]anthracene	Fluorene	C-2 Phenanthrenes/Anthracenes
Benzo[a]pyrene	C-1 Fluorenes	C-3 Phenanthrenes/Anthracenes
Benzo[b]fluorene	C-2 Fluorenes	C-4 Phenanthrenes/Anthracenes
Benzo[e]pyrene	C-3 Fluorenes	Pyrene
Benzo[g,h,i]perylene	Indeno[1,2,3-cd]pyrene	C-1 Fluoranthenes/Pyrenes
Benzo[k]fluorene	Naphthalene	C-2 Fluoranthenes/Pyrenes
Chrysene	C-1 Naphthalenes	C-3 Fluoranthenes/Pyrenes
C-1 Chrysenes	C-2 Naphthalenes	C-4 Fluoranthenes/Pyrenes
C-2 Chrysenes	C-3 Naphthalenes	Saturate Hydrocarbons:
C-3 Chrysenes	C-4 Naphthalenes	nC <sub>10</sub> -nC <sub>35</sub>
C-4 Chrysenes	Naphthobenzothiophene (NBT)	Oil Biomarkers:
Dibenz[a,h]anthracene	C-1 NBTs	Triterpanes (m/z 191)
Dibenzothiophene (DBT)	C-2 NBTs	Diasteranes & Regular Steranes (m/z 217)
C-1 DBTs	C-3 NBTs	14β(H) Steranes ( <i>m/z</i> 218)
C-2 DBTs	Perylene	Triaromatic Steroids (m/z 231)
C-3 DBTs	Phenanthrene	

#### 5.4.3 Elemental Analysis

Trace-element analysis of artifacts such as pottery and lithics has proven useful in provenance studies for determination of geographic source (Glascock and Neff 2003). Among the analytical techniques that have been used are instrumental neutron activation analysis (INAA or NAA), X-ray fluorescence (XRF), inductively coupled plasma-mass spectrometry (ICP-MS), and laser-ablation ICP-MS (LA-ICP-MS). NAA is an extraordinarily sensitive, accurate, and reliable technique that is especially useful for

quantitative and qualitative multi-element analysis of samples (Glascock 2008). Due to its dependability, NAA is also suitable as a complementary technique in the application of other methods of compositional analysis (University of Missouri Research Reactor Archaeometry Laboratory 2017, accessed online).

The potential effects of hydrocarbon contamination on elemental analyses of pottery sherds, lithics, and other archaeological samples has not been previously investigated and so is not well understood. Pottery sherds and lithic artifacts from sites where oil is present might be washed in warm water and detergent to remove any residual oil from the surface without affecting the results of elemental analysis. Mechanical removal of the surfaces of pottery sherds with a burring tool might further reduce the potential effects of oil on trace element analysis (Glascock, personal communication, January 25, 2014). However, the possible penetration of hydrocarbons into pottery samples has not been examined, so potential adverse effects on techniques such as NAA and ICP-MS are unknown (Boulanger et al. 2013).

The PI consulted Michael Glascock of the Archaeometry Lab at the University of Missouri Research Reactor (MURR) in formulating a series of research questions pertaining to the potential effects of oil on elemental analysis. First, does the presence of oil affect sample preparation and if so, how? If pretreatment such as washing in a cleansing agent to remove oil is necessary, then pre-testing for the presence of oil in archaeological deposits may be necessary for sites impacted by an oil spill. Second, if oil has infiltrated artifacts such as pottery sherds, will scanning of cross-sections by LA-ICP-MS reveal elements associated with crude oil, such as sulfur (S), vanadium (V), and nickel (Ni)? Elevated levels of elements known to occur in crude oil might indicate other, unknown adverse impacts to archaeological deposits (Glascock, personal communication, January 25, 2014). Third, if oil is present, does the amount or depth of penetration into an artifact impede overall compositional analysis or interpretation, even with pretreatment?

Last, the comparative effectiveness of ICP-MS and NAA in analyzing samples in which oil is present has the potential to affect future research costs. LA-ICP-MS is thought to be preferable to NAA at sites impacted by an oil spill, because it is potentially more sensitive in detecting V and Ni, along with additional elements that might be present in low amounts from oil residues. However, ICP-MS is more labor intensive and is generally more expensive (Glascock, personal communication, January 25, 2014).

Five samples of pottery sherds from three sites were shipped overnight in sealed containers to the MURR Archaeometry Laboratory (Appendix B). The MURR Archaeometry Lab recommended a minimum sample size of one square inch for ceramic sherd analysis by NAA or LA-ICP-MS. One gram was the recommended minimum sample size for lithic artifacts. All of the samples were grog-tempered sherds, selected based on the previously outlined criteria for sample selection. Four of the pottery samples were collected from contexts that tested positive for oil at 16SB174 and 16SB178 (ANID SBDA011, SBDB011, SBD012, and SBC035). The fifth was from an intact archaeological context in a test unit at control site 16SB153 (ANID SBE017).

All of the sherds were prepared for NAA by using a dremel tool to remove exposed surface areas and extract a 1 cm<sup>2</sup> sample. Sherds were not prewashed or otherwise pretreated before analysis. The paste samples were ground into a powder, dried, and subdivided. The two analytical subsamples were irradiated according to MURR standard operating procedures (Glascock and Neff 2003). Samples to be analyzed by LA-ICP-MS were similarly prepared and mounted on glass slides (Appendix D). The sampling methods, analytical procedures and results are described in Appendix D and the Analytical Results section (6.4) of this report. Lithic artifacts were not included in the elemental analysis, because so few lithics were recovered. None were from archaeological contexts that tested positive for oil.

# 5.4.4 Absorbed Residue Analysis

The analyses of absorbed residues in pottery sherds from archaeological sites provide an independent line of evidence in studies of subsistence and foodways that may complement or fill in for archaeobotanical and zooarchaeological analyses. For example, analysis of pottery sherds from Late Woodland and early Mississippi period sites in the Mississippi Valley has identified residues from plants (Reber and Evershed 2006). Plant residues absorbed into pottery vessels during cooking and food preparation can be contrasted with those of animals, such as deer or fish, which impart distinctly different chemical residues. Relative amounts of plant and animal residues can be compared in different samples from various sites in reconstructing the diet of a community or region (Heron and Evershed 1993; Reber 2012, 2013; Reber et al. 2010; Reber and Kerr 2013).

The analysis of organic residues in pottery sherds involves the extraction of lipids that were absorbed within the ceramic matrix during vessel use or introduced in archaeological contexts subsequent to deposition. GC/MS is the preferred method for analyzing extracted lipids since it can identify a wide range of compounds and distinguish a complex mixture of compounds typically found in pottery residues from archaeological contexts (Reber and Hart 2008:129). Both the extraction and analysis of residues can be problematic, with different techniques and procedures based on experimental archaeological chemistry (Barker et al. 2012). There have been no previous investigations of the potential effects of hydrocarbon contamination on the analysis of absorbed residues in ceramics.

GC/MS can identify biomarkers for petroleum in pottery residues from archaeological sites, as well as the dispersants used in oil spill cleanup and response. Several issues might accordingly be addressed regarding the possible absorption of hydrocarbons and dispersants. The presence of oil biomarkers and/or dispersants in absorbed residues may be correlated with samples of pottery from coastal sites known to have been impacted by an oil spill. Conversely, the absence of oil biomarkers and/or dispersants in absorbed residues may correlate with samples of pottery from sites not known to have been impacted by an oil spill. The analysis of absorbed residues in potsherds provides an independent source of data for comparison with GC/MS analyses of soil samples and may indicate the overall extent of the impacts of oil spills and dispersant use on coastal sites.

Absorbed residue analysis can identify the relative amounts of oil and dispersant biomarkers in potsherds, in contrast to compounds such as fatty acids typically found in pottery sherds from archaeological sites. The potentially disruptive effects of oil and dispersants on the analysis of preexisting absorbed residues are unknown and must be addressed, along with the potential for misinterpreting oil or dispersant contaminated residues (Reber, personal communication July 20, 2010 and January 24, 2014). Oil and dispersants may interact with absorbed residues in archaeological deposits and accelerate or otherwise affect post-depositional processes such as hydrolysis, oxidation, or microbial decomposition (Appendix E; Reber 2016). Last, oil and/or dispersant contamination may affect the successful application or overall costs of absorbed residue analysis.

Seventeen pottery sherds from seven sites were selected for absorbed residue analysis using the previously described collection procedures and criteria for sample selection (Appendix B). Eleven samples were sent to Dr. Eleanora Reber at the Archaeological Residue Laboratory, University of North Carolina at Wilmington in September of 2015. Six additional samples were submitted in January of 2016. The initial batch of 11 samples included four sherds from Cheniere St. Denis (JEB029, JEB022, JEB043, and JEB059), one from Comfort Island (SBD010), two from Southern Comfort (SBC028 and SBC029), two from Scow Island Scatter (SBF007 and SBF017), and two from Redfish Slough (LFG034 and LFG018). Sample selection was based on availability and minimum sample size requirements in contexts thought to contain oil. The previous results of chemical characterization of soil samples were particularly relevant, and allowed for the selection of samples from excavated contexts known to contain oil.

The second batch of six samples was selected based on the results of oil detection by the DES Lab and associations with previously submitted samples. An effort was also made to investigate the vertical and horizontal extent of hydrocarbon contamination at sites determined to contain oil. This second group included two sherds from Southern Comfort (SBC037 and SBCA037), and additional samples from Cheniere St. Denis (JEB025) and Redfish Slough (LFG047). A single sample was selected from each of the two control sites, Bayou Sale (SYA018) and the unnamed site near Bayou St. Malo (SBE015), in order to assess the potential for oil in absorbed pottery residues at sites not otherwise known to have been impacted by an oil spill. The samples were shipped overnight in sealed containers to the UNCW Archaeological Residue Laboratory. The ANID system tracked provenience information while concealing this information during the analysis.

Standard pretreatment involved the removal of surface impurities from the pottery sherds, which were then crushed in a mortar and pestle. Absorbed residues were extracted using standard protocols and methodology for the analysis of absorbed residues (Evershed et al. 1990). Reber (2016) describes the application of GC/MS for absorbed residue analysis. The results of this analysis, including the effects of oil and dispersant contamination on the interpretation of absorbed lipids, are presented in Appendix E and discussed in the Analytical Results section (6.5) of this report. Approximate degree of contamination was determined by quantifying total lipid and biomarkers of contamination to sherd weight (g). Among the important findings, Reber (2016) identified biomarkers for both oil and Corexit (9500 and 9527), the dispersants used in the MC252 oil spill cleanup response.

# 5.4.5 Radiometric Analysis

The potential effect of hydrocarbon contamination on radiocarbon dating was one of the first issues raised in the weeks and months following the MC252 oil spill. Taylor (1987) and more recently, Taylor and Bar-Yosef (2014) provide general introductions to radiocarbon dating in archaeology, including the basic principles, methodologies and application of techniques such as accelerator mass spectrometry (AMS). Standard radiocarbon and AMS dating can provide a radiocarbon age in years before present (YBP) for a sample of organic material, such as charcoal, by measuring the relative amount of carbon-14, an unstable and radioactive isotope of carbon, in relation to the stable isotopes of carbon (Bowman 1990; Taylor 1998; Taylor and Bar-Yosef 2014). AMS is highly accurate and generally requires small sample sizes, since it is based on the detection of relative numbers of carbon atoms (Beta Analytic 2017; Beukens 1992; Tuniz et al. 1998). Previous investigation of the potential effects of oil on radiocarbon dating of archaeological deposits following the *Exxon Valdez* oil spill did not discern any adverse effects on radiocarbon dating. The results were not considered to be applicable to sites outside of the region, however, much less the northern Gulf of Mexico or an oil spill of the magnitude of MC252 (Reger et al. 1992).

The issue of contamination has, in fact, been systematically addressed in the radiocarbon dating literature (Hedges 1992; Taylor 1987) and archaeologists take routine precautions to avoid introducing contaminants during sample collection and storage. The collection guidelines provided by Beta Analytic Laboratories (2017) specifically state that "hydrocarbons, glue, biocides, polyethylene glycol, or polyvinylacetate must not come in contact with samples for radiocarbon dating." The introduction of hydrocarbons from an oil spill into archaeological deposits has the potential to alter the 14C/12C ratio within a sample (Hedges 1992:166). Without pretreatment, the presence of such contaminants might otherwise preclude samples from radiocarbon analysis or produce erroneous results. Physical and chemical pretreatments can remove impurities and possible contaminants prior to radiocarbon analysis (Hedges 1992:165–166; Taylor 1987:39–41). Understanding the potential effects of hydrocarbon contamination on radiocarbon dating may thus hinge on pretreatment.

Following the MC252 oil spill, the PI corresponded with Darden Hood at Beta Analytic, Inc. about the challenges of radiocarbon dating materials from sites affected by an oil spill. Solvent extraction is the

recommended pretreatment technique for samples known or suspected to be contaminated with hydrocarbons. This pretreatment technique has proven successful on samples with fossil carbon contamination (Reger et al. 1992). Pretreatment by solvent extraction works well, but is limited to AMS analysis and is generally more expensive than standard pretreatment techniques. Previous knowledge of contamination may be crucial, particularly when dealing with a limited number of samples. Determining the presence of oil through sensory observation is subjective and unreliable, especially with the small samples typically submitted for AMS (ASTM 2006; Dijs et al. 2006). Furthermore, the potential effects of oil and dispersants on the reservoir effect and 14C depletion in AMS analysis of carbonate samples have not been addressed (personal correspondence with Darden Hood, June 4 and July 19, 2010).

The staff of Beta Analytic Laboratory, Inc. conducted radiocarbon dating by AMS to assess the potential effects of oil contamination on pretreatment processes, radiocarbon dating methods and results. The Project Director and PI submitted fifteen samples from ten different proveniences at seven sites (Table 7; Appendix B). Sample selection was based on the previously described collection procedures and criteria, including the earlier detection of crude oil in associated archaeological contexts. Due to the disturbed condition of many coastal sites in the Mississippi River delta, identifying suitable materials for radiocarbon dating from intact, primary contexts proved even more challenging than finding samples where GC/MS had detected oil in associated soil matrices. Six of the 15 samples were collected from what were interpreted as undisturbed archaeological contexts. Two were from the TU 3, Level 2 (25–35 cm below surface) at Cheniere St. Denis (JEB024 and JEBA024), two were from the west wall of TU 1 (134-139 cm below surface) at Site 16SB153 (SBE033 and SBEA0332), and two were from a core extracted from Mound B at Acorn Mounds (16SB185) at depths of 195–199 cm and 210–216 cm below surface (SBH034 and SBHA034). The other samples were collected from excavation units in redeposited contexts at Redfish Slough (16LF293), Comfort Island (16SB174), Scow Island Scatter (16SB182), and Bayou Sale (16SMY17).

Before the fieldwork, the Project Director and PI conferred with the Beta Analytic staff about the proper handling, shipping, and analysis of samples. Samples for radiocarbon dating were shipped overnight in sealed containers to Beta Analytic Laboratory. The ANID system was used to track sample provenience, while information on the presence or absence of oil was not provided (Table 7). Three of the 15 samples were included as supplemental material (SYAB021, SYAC021, and JEBA024). When there was sufficient material, samples were subdivided to allow for both standard pretreatment and solvent extraction techniques on subsamples from the same archaeological context. Radiocarbon analysis was cancelled following pretreatment of three samples of bone (LFG038, LFG020, and SBH034) that failed to yield separable collagen. One sample (SBF010) from TU 1 at Scow Island Scatter (16SB182) was pretreated only by solvent extraction. Eight samples were of sufficient size to permit subdivision and application of both standard pretreatment and solvent extraction.

As described in the results (Section 6.6), radiocarbon ages were obtained on 17 subsamples, which included 8 pairs of subsamples. Initial comparisons of the radiocarbon ages of subsamples did not reveal statistically significant differences between solvent extraction and standard pretreatment techniques. At issue was whether contexts that had tested positive for oil had resulted in the hydrocarbon contamination of associated radiocarbon samples. The PO, PI, and Project Director designed two experiments to further examine the effects of crude oil contamination on the pretreatment of samples from the control sites, where crude oil from MC252 was absent in archaeological contexts.

Table 7. Samples submitted for radiocarbon dating

Site & Sample	ANID	Provenience	Material	n	g
16JE2-24	JEB024 <sup>1</sup>	TU3 L2 at 25-35 cm, field sample	Unid. mammal bone	1	3.5
16JE2-24	JEBA024 <sup>1,3</sup>	TU3 L2 at 25-35 cm, field sample	Unid. mammal bone	1	1.9
16LF293-20	LFG020	TU1 at 16 cm, specimen sample	Ondatra Zibethicus, femur	1	2.1
16LF293-38	LFG038 <sup>1</sup>	TU2 at 36 cm, specimen sample	Mississippiensis, jugal	1	16.5
16SB153-33	SBE033	TU1 at 134-139 cm W wall	Unid. bone fragments	6	4.7
16SB153-33	SBEA033 <sup>2</sup>	TU1 at 134-139 cm W wall	Unid. bone fragment	1	5.5
16SB174-12	SBDA012 <sup>1</sup>	TU2 L2 at 15-20 cm, sediment	Wood charcoal	13	0.3
16SB182-10	SBF010	TU1 L3 at 20-30 cm, field sample	Unid. bone fragment	1	1.1
16SB182-16	SBF016	TU2 L2 at 19cm, E half	Wood charcoal	1	0.4
16SB185-34	SBH034	Mound B, CT4 at 195-199 cm	Unid. bone fragments	8	1.0
16SB185-34	SBHA034	Mound B, CT4 at 210-216 cm	Wood charcoal	8	0.1
16SMY17-21	SYA021	TU2 L4 at 30-35 cm, field sample	Unid. turtle bone	1	1.8
16SMY17-21	SYAB021 <sup>3</sup>	TU2 L4 at 30-35 cm, field sample	Unid. turtle bone	2	2.1
16SMY17-21	SYAA021 <sup>2</sup>	TU2 L4 at 30-35 cm, field sample	Unid. turtle bone	1	3.3
16SMY17-21	SYAC021 <sup>2,3</sup>	TU2 L4 at 30-35 cm, field sample	Unid. turtle bone	2	4.3

<sup>&</sup>lt;sup>1</sup>Samples collected from units reported by DES as oiled. <sup>2</sup>Samples intentionally contaminated with MC252 oil as part of a controlled, comparative study. <sup>3</sup>Sample submitted as supplemental material (JEBA024 to supplement JEB024, SYAB021 to supplement SYA021, and SYAC021 to supplement SYA021).

At Site 16SB153, a sample of unidentified non-human bone fragments (16SB153-33) was collected from the west wall of TU 1 at 134 to 139 cm below surface. Soil matrix from a column sample from the first level (0–10 cm) of this unit had previously tested negative for the presence of oil. Sample 16SB153-33 was divided into two subsamples (SBE033 and SBEA033). The second of these subsamples (SBEA033) was intentionally contaminated with crude oil from the shoreline of the Southern Comfort site (16SB178), where oil was detected in a nearby soil sample (16SB178-26) and determined to be a possible match for MC252 oil (SBC026). The oil was introduced by hand onto the surface of the second subsample (SBEA033), while the first subsample remained uncontaminated. These subsamples were then each subdivided, producing four subsamples. Two subsamples were processed by solvent extraction (one contaminated and one uncontaminated) and two received standard pretreatment (one contaminated and one uncontaminated).

The potential effects of hydrocarbon contamination were also explored by introducing oil with seawater. A sample of unidentified turtle bone (16SMY17-21) from TU 2, Level 4 (30-35 cm below surface) at Bayou Sale (16SMY17) was subdivided into four subsamples. Two subsamples (SYAA021 and SYAC021) were intentionally contaminated with a mixture of oil from the Southern Comfort site and

seawater from Bayou Sale Bay. These two subsamples were placed in glass vials and maintained at room temperature for one week. The other two subsamples were left uncontaminated. One contaminated and one uncontaminated subsample were pretreated by solvent extraction. The other subsamples received standard pretreatment. The results of the radiocarbon analysis and the experiments are presented in the following section (6.6) of this report.

# 6. Analytical Results

The objectives and hypotheses formulated for this study guided the analysis of samples collected from the assessment of eight sites, in order to assess the direct and indirect effects of an oil spill. The analytical results also provided supplementary information on sites, such as chronology, cultural affiliation, archaeological integrity, and the context of associated samples. Cultural materials collected from the surface and excavation units at sites consist of Native American ceramic sherds, lithic artifacts, faunal bone, and historical artifacts, such as glass. The samples collected from excavation units included column and unit core samples. These consisted mostly of soil matrices with different amounts of mollusk shell and broken shell hash.

Shell in association with midden was ubiquitous at many sites, with *Rangia cuneate* most abundantly represented, followed by oyster (*Crassostrea virginica*). Columns samples, unit cores, and soil samples contained varying amounts of whole, broken, and crushed mollusk shell. An important distinction was made during excavation between whole bivalve shells and highly fragmented shell hash, providing evidence of redeposition by shoreline erosion and tidal action. Faunal bone was also recovered from test units and the surface of shorelines, providing additional contextual information on the sites. Appendix G presents the results of the zooarchaeological analysis.

The following section presents the results of the analyses, beginning with the major classes of artifacts that were recovered from each of the assessed sites. This is followed by a discussion of the soil cores collected from the Acorn Mounds site. Specialists who collaborated in this study examined samples for the chemical detection and characterization of hydrocarbons, the potential effects of oil on elemental analyses, absorbed residues in ceramics, and radiocarbon dating. Appendix B presents a list of all samples submitted to specialists for analysis. Sections 6.3 through 6.6 present the results of their analyses. The reports of their findings are included in Appendices C through G.

#### 6.1 Artifacts

All artifacts and ecofacts were sorted and classified by major categories of material. These included ceramics, lithics, faunal bone, mollusk shell, glass, metal, and plastic. As expected, the majority of the artifacts from all sites were ceramic sherds manufactured and used by Native Americans. These consist of sherds from unglazed, non-vitrified pottery vessels fired at relatively low temperatures. Classification involved the identification of paste, inclusions or temper, surface treatment, decoration, and when possible, vessel form (Ortner et al. 1993:68, 76, 127; Rice 1987:4–5). The use of the existing type-variety system developed for the Lower Mississippi Valley allowed for the identification of diagnostic pottery types (Brown 1998; Phillips 1970; Weinstein 2000; Williams and Brain 1983). Previous investigations in Louisiana's coastal region provide detailed descriptive overviews of pottery types and varieties as a means of addressing culture chronology and interaction (Giardino 1990; Kidder 1995; McGimsey 2003a; Miller et al. 2000; Weinstein et al. 2012:149–178).

Pottery sherds and samples containing sherds were sorted but not washed, making the identification of type and variety often difficult. The majority of the sherds from all of the sites were undecorated plain wares. Grog-tempered plain wares were generally classified as Baytown Plain *variety unspecified*. Distinguishing *varieties* of Baytown Plain, such as *Addis, Bayou Des Oies, Little Tiger*, and *Percy Creek*, depends to some degree on determining relative percentages of grog as a tempering agent (Ryan 2004:91–97; Weinstein et al 2012:162–167). Identification of specific varieties of Baytown Plain was problematic due to the small size of many of the sherds, the often wave-worn condition of the surfaces, and the procedure of not cleaning artifacts by washing. In instances where sherds were smaller than 0.5 inch (1.3 cm) in diameter or too small for reliable identification as to type, the sherds were sorted, identified by

temper, counted and weighed. Many of these were classified as indeterminate grog tempered or indeterminate sherdlets.

The condition of pottery sherds also provided contextual information, as seen in the large numbers of wave-worn sherds that are associated redeposited, secondary contexts. Historic artifacts, particularly pieces of glass containers, were relatively common on the surface at many sites. With few exceptions, the historic artifacts represent modern, incidental intrusions of refuse likely deposited by wave action. Only two sites, 16JE2 and 16SB153, have recorded historic components. The presence of historic artifacts in excavated contexts at these and other sites often provided an indication of recent deposition or disturbed contexts.

In contrast, there were relatively small amounts of lithic artifacts recovered from the assessed sites. There were no lithic artifacts recovered from five of the sites. Only the Bayou Sale (16SMY17), Cheniere St. Denis (16JE2), and Southern Comfort (16SB178) sites produced lithics and the majority (n=28; 90%) came from Cheniere St. Denis. The total number of lithics (n=31) from all sites may be partly due to the small sample size, but is also associated with the absence of local stone sources for tools. This is characteristic of sites on the north-central Gulf Coast, where Native Americans commonly used bone and shell for tools (Davis et al. 1983; Weinstein 2012:178). The following discussion is organized by site and major classes of material in the order each site was assessed.

### 6.1.1 Bayou Sale (16SMY17)

As at most of the sites assessed by this study, a majority of the artifacts collected from Bayou Sale consisted of Native American ceramic sherds (Table 8). As noted by previous investigators (Brown 2015:22), the Native American ceramic assemblage was dominated by Baytown Plain sherds. A majority of these can probably be classified as *var. Addis*, which is described as a grog-tempered plain ware with occasional inclusions of organic materials and sometimes bone (Williams and Brain 1983:92). The distinctions between varieties of Baytown Plain, such as *Bayou Des Oies*, *Little Tiger*, and *Percy Creek* appear to lie in relative percentages of grog (Ryan 2004:91–97; Weinstein et al 2012:162–167). Identification of specific varieties of Baytown Plain from Bayou Sale and other sites assessed by this study was often problematic due to the small size of the sherds, their frequently wave-worn condition, and the practice of not cleaning artifacts by washing. Grog-tempered plain wares were consequently classified as Baytown Plain *var. unspecified*. These included grog-tempered sherds with variable amounts of sand in the pastes.

Only two decorated sherds in the ceramic assemblage from Bayou Sale stand out: a single Coles Creek Incised, *var. Stoner* rim sherd from the surface and a grog-tempered sherd with a single, rectangular punctate from TU 2 (Figure 65). Both sherds are wave worn, indicating their redeposition from the shell midden at Bayou Sale. Only two lithic artifacts were recovered from the site: a small piece of ground sandstone was recovered from ST 6 and one small piece of heat-treated gravel chert was collected from TU 2, level 3.

In addition to the wave worn condition of the pottery sherds, the shovel tests and test units at the Bayou Sale site produced evidence of shell midden redeposited by tidal action, as seen in the large amounts of broken shell and shell hash. The relatively small amount of historic artifacts from Bayou Sale provides further indication of the excavated contexts of the Native American ceramic assemblage and associated cultural deposits. Glass shards from bottles or other containers were the most common historic artifact (n=12; 67%). These were recovered from three of the shovel tests and levels 2, 3, and 4 of TU 2. The glass container fragments probably represent modern refuse washed in and redeposited in the site with the Native American pottery sherds, shell, and bone. A single brick fragment from TU 2, Level 2, may represent a historic component at the site or perhaps a site nearby, as the site and surrounding landscape have not been systematically surveyed. Additional investigations are needed to delineate the boundaries

of the Bayou Sale site and to assess the site components, and the vertical and horizontal extent of the shell midden. The two pieces of clear, flat glass from ST 5 are probably from a large container and are not a windowpane or structural debris.



Figure 65. Ceramics from Bayou Sale (16SMY17)

Coles Creek Incised, *var. Stoner* (left) from the surface and grog-tempered sherd with single rectangular punctate from TU 2, Level 2.

Table 8. Artifacts from Bayou Sale (16SMY17)

	ST 3		S1	Г4	ST 5		ST 6		TU 1		TU 2		
	Surface	L1	L1	L2	L1	L2	L1	L2	L1	L1	L2	L3	L4
NATIVE CERAMICS													
Baytown Plain, var. unspecified	13	2			5	3	4	1	3	10	4	15	9
Coles Creek Incised, var. Stoner	1												
Indet. grog-temp. punctated											1		
Indet. sherdlets (<0.5")			2				2			8	20	32	5
LITHICS													
Chert, HT shatter												1	
Sandstone, ground							1						
HISTORIC													
Brick, fragment											1		
Ceramic, porcelain											1		
Glass, container fragments			1				2	1			3	1	2
Glass, flat					2								
Metal													
Aluminum, corroded												2	
Iron, rusted												2	

Stratum III in TU 2 was interpreted as a buried A-horizon, consisting of a very dark grayish brown (10YR 3/2) clay loam with whole and broken shell beneath a root mat. While the Baytown Plain sherds from Level 4 in this stratum appear to be only moderately wave worn, this level also contained two shards of container glass. As described in the following sections, one Baytown Plain pottery sherd from Level 1 in TU 2 was analyzed for absorbed residue and a column sample from Level 4 of TU 2 was analyzed for the presence of oil. As all of the excavated contexts appear to have been redeposited, further investigations are also needed at the Bayou Sale site to locate and examine potentially intact cultural deposits. These may be deeply buried terrestrial contexts or submerged offshore.

# 6.1.2 Cheniere St. Denis (16JE2)

The Cheniere St. Denis site was the subject of intensive Phase II test excavations by R. Christopher Goodwin & Associates, Inc. (Coughlin et al. 2004). Their investigation produced evidence of intact cultural deposits associated with the Late Baytown and Coles Creek periods. Pottery sherds collected from the surface for the present study consisted of Baytown Plain, *var. unspecified*, and three sherds of Coles Creek Incised, *var. Phillips* (Figures 66 and 67). The latter date was from the Baytown period. The fieldwork for this investigation involved the excavation of three 50-by-50 cm test units and one 50 cm-by-1 meter test unit (Section 5.3.2). The first three test units (TU 1-3) were placed on the west flank of the northernmost mound, and TU 4 was excavated near the shoreline to the south.



Figure 66. Grog-tempered rim sherds from Cheniere St. Denis (16JE2).

Baytown Plain, var. unspecified (top row) and Coles Creek Incised, var. Phillips (bottom row) from Area B surface.

The artifacts recovered from the four test units reflect the depositional contexts at the site. Though only two levels could be excavated in TU 4 before it was inundated, the two strata that were revealed are interpreted as redeposited by wave action. These strata contained pieces of plastic and ferrous metal fragments, as well as wave-worn Baytown Plain, *var. unspecified* sherds (Table 9). TU 1 was excavated near the northern shoreline and likewise produced artifacts from disturbed and redeposited contexts, including a large amount of rusted iron fragments and an aluminum can tab from Level 3. The fragments of rusted iron in TU 1 may represent the remains of a crab trap and as such, are part of the historic component of the Cheniere St. Denis site. Goodwin and Associates determined that the historic component lacked archaeological integrity (Coughlin et al. 2004:101–102, 127).

Table 9. Artifacts from Cheniere St. Denis (16JE2)

	Surfa	ace	_	TU 1		TU 2								TU 3				TU 4	
	Α	В	L1	L2	L3	L1	L2	L3	L4	L5	L6	L7	L9	L1	L2	L3	L4	L1	L2
NATIVE CERAMICS																			
Avoyelles Punctated, var. Avoyelles								3											
Avoyelles Punctated, var. unspec.						1													
Baytown Plain, var. unspecified	28	9	4	6	4	13	9	19	18	32	8	5		5	17	24	120	3	4
Coles Creek Incised, var. Phillips		3																	
Coles Creek Incised, var. unspec.					1														
Evansville Punctated, var. unspec.								1											
French Fork Incised, var. unspec.							1												
Indet. grog-temp. incised								1											
Indet. grog-temp. punctated						1													
Indet. sherdlets (<0.5")				2	3	11	13	12	16	14	2	12	1		3	9	150	5	5
LITHICS																			
Chert, core		2														1	1		
Chert, primary flake	1									1		1			5				
Chert, tertiary flake																3			1
Chert, fire cracked										1		1							
Chert, shatter	2																1		
Chert, pebble		1		1						2								1	
Quartz, pebble					2														
HISTORIC																			
Glass, container				1	2														
Glass, flat														1					
Metal																			
Aluminum, can tab					1														
Iron, fragments			10	416	29	4									4			2	9
Plastic, indet.					2													4	

Artifacts from the lower levels of TU 2 and TU 3 represent intact depositional contexts previously identified during the Phase II site testing. Goodwin and Associates identified Stratum I as correlating with the Late Baytown-Coles Creek occupation and described it as a layer of whole *Rangia* shell in association with a black silt loam or gray clay on terrestrial and submerged portions of the site, respectively (Coughlin et al. 2004:64, 109–110). The absence of historic artifacts beneath Level 1 in TU 2 and beneath Level 2 in TU 3 is consistent with an interpretation of the stratigraphy as representing cultural deposition coeval with the Late Baytown-Coles Creek site occupation and construction of the mounds. The artifacts

from these test units are mostly Baytown Plain, *var. unspecified* sherds (n=291). Four sherds of Avoyelles Punctated (*var. Avoyelles* and *var. unspecified*) and two small sherds of Evansville Punctated and French Fork Incised (*vars. unspecified*) from TU 2 are also consistent with previous findings (Figures 68 and 69). Among the samples from Site 16JE2 submitted for absorbed residue analysis were four grog-tempered pottery sherds from TU 2 and TU 3 (Section 6.5).



Figure 67. Grog-tempered sherds from Cheniere St. Denis (16JE2).

Baytown Plain, var. unspecified body sherds from Area B surface.



Figure 68. Ceramics from TU 2 at Cheniere St. Denis (16JE2).

Baytown Plain, *var. unspecified* rim (top), Avoyelles Punctated, *var. unspecified* (bottom left) and unidentified punctate sherd (bottom center) from Level 1 and French Fork Incised, *var. unspecified* (bottom right) from Level 2.



Figure 69. Ceramics from TU 2 at Cheniere St. Denis (16JE2).

Evansville Punctated, var. unspecified (left), and three Avoyelles Punctated, var. Avoyelles sherds (right) from Level 3.

The Cheniere St. Denis site produced, by far, the largest number of lithic artifacts of any site in this study. A relatively large number of lithic artifacts (n=668) was also recovered from Cheniere St. Denis during Phase II testing, including several bipolar pebble cores from Stratum I (Coughlin et al. 2004:68, 102–109). Excluding seven unmodified chert and quartz pebbles, the present study recovered 21 lithic artifacts from excavated contexts and the surface. A majority of these (n=12; 57%) were primary or tertiary chert flakes and just less than half of these showed evidence of thermal alteration (n=5; 42%). Two chert pebble cores were collected from the surface of Area B, one of which was thermally altered (Figure 70). One chert pebble core fragment was recovered from Level 3 in TU 3 and another was collected as a specimen sample from TU 3, Level 4.



Figure 70. Lithic artifacts from Cheniere St. Denis (16JE2).

Chert pebble cores from Area B surface (first and second from left) and TU 3, Levels 3 and 4 (third and fourth from left, respectively).

The chert artifacts from the Cheniere St. Denis site are a brownish yellow to dark yellowish brown gravel chert, similar to the gravels found within the Pleistocene and Holocene stream sediments of central Louisiana and north of Lake Pontchartrain (Heinrich 1987:175). As local lithic sources are absent in the delta, the relatively large amount of lithic artifacts from Cheniere St. Denis has been tied to the political and economic role of mound center residents who may have controlled regional trade (Coughlin et al. 2004:109). Technological and ecological variables may have also been involved, such as the adoption of the bow and arrow, or changing subsistence patterns during the Late Baytown-Coles Creek transition. These and other possible explanations depend to a large degree on the identification of lithic sources as the raw material for stone tools.

### 6.1.3 Southern Comfort (16SB178)

The artifacts from the Southern Comfort site were mostly Native American pottery sherds collected from the surface (n=74; 80%). Of these, 95 percent (n=70) were wave-worn, grog-tempered sherds provisionally classified as Baytown Plain, *var. unspecified* (Table 10). The few diagnostic types were identified as Marksville Incised, *var. unspecified* and Mabin Stamped, *var. Mabin* (Figures 71 and 72). Ceramics recovered from the site during the oil spill response likewise included Baytown Plain, *var. unspecified*; Mabin Stamped, *var. Mabin*; and Marksville Incised, *var. unspecified* (Cloy and Ostahowski 2015:6-768), leading investigators to infer a Marksville period occupation, along with an earlier Tchefuncte component. Historic artifacts collected by the present study consist of three pieces of glass, one annular-decorated whiteware sherd and a few pieces of ferrous metal.

**Table 10. Artifacts from Southern Comfort (16SB178)** 

	Surf	face	ST 3	ST 6	TU 1	TU 2
	Α	В	L2	L1	L1	L1
NATIVE CERAMICS						
Baytown Plain, var. unspecified	41	29	1	1		4
Marksville Incised, var. unspec.	1	2				
Mabin Stamped, <i>var. Mabin</i>	1					
Indet. sherdlets (<0.5")					4	1
HISTORIC						
Ceramic, whiteware, annular		1				
Glass, container fragments		2			1	
Metal, iron fragments					1	2

Previous investigators found artifacts to be limited to the surface at the Southern Comfort site, leading them to conclude that it lacked archaeological integrity and was the result of secondary deposition from shoreline erosion (Cloy and Ostahowski 2015:6-771). The excavation of eight shovel tests and two 50 cm-by-1 meter units during the present study produced only 15 artifacts. Four of these were pieces of glass container or metal. The remainder were Baytown Plain, *var. unspecified* sherds (n=6) or very small grog-tempered sherds (n=5). Except for ST 3, subsurface recovery was limited to the first level. TU 1 and TU 2 were excavated to 40 cm and 35 cm, respectively, before the test units were inundated. Though the Southern Comfort site does appear to lack depositional integrity, further analysis of the artifacts can nonetheless provide a valuable source of information, including cultural chronology in the delta and absorbed residues in ceramics (Section 6.5).



Figure 71. Ceramics from Area A at Southern Comfort (16SB178).

Mabin Stamped, *var. Mabin* (left) and Marksville Incised, *var. unspecified* (right).



Figure 72. Ceramics from Area B at Southern Comfort (16SB178). Marksville Incised, *var. unspec*ified.

# **6.1.4 Comfort Island (16SB174)**

As at the Southern Comfort site (16SB178), the nearby Comfort Island site produced relatively few artifacts. These were predominantly from the surface and in redeposited, secondary contexts (Table 11).

The excavation of two 50 cm-by-1 meter units produced 18 Baytown Plain, *var. unspecified* sherds, one sand-tempered sherd and 75 small grog-tempered sherds. A majority (n=58; 74%) of the 74 pottery sherds from TU 2 came from levels 2 and 3, but most of these (n=62; 84%) were very small (<0.5") fragments of grog-tempered pottery that can be described as sherdlets of indeterminate type.

Table 11. Artifacts from Comfort Island (16SB174)

	Surf	ace		TU 1			TU 2	
	Α	В	L1	L2	L3	L1	L2	L3
NATIVE CERAMICS								
Baytown Plain, var. unspecified	3	4	3	3		8	3	1
Indet. grog-tempered incised	1	1						
Indet. sand tempered	1		1					
Indet. sherdlets (<0.5")		1		2	11	8	18	36
LITHICS								
Chert, Kent Projectile Point		1						
HISTORIC								
Ceramic								
Coarse earthenware		1						
Whiteware								1
Glass, container fragments				1				
Metal, iron fragments								1
Rubber fragments					2			

Level 3 in TU 2 also produced a single historic whiteware sherd and one small fragment of ferrous metal. The lowest stratum in both test units (Stratum III in TU 1; Stratum IV in TU 2) consisted of a very dark gray (10YR 3/1) clay loam and black (10YR 2/1) clay muck that was devoid of shell. This may represent an A-horizon buried beneath redeposited shell. However, the artifacts were associated with the layers of redeposited shell and were not found in the lowest stratum. Like the Southern Comfort site on the opposite shore of Comfort Island, the cultural materials from Site 16SB174 were redeposited by shoreline erosion. The Native American pottery from the Comfort Island site is visibly wave worn. Incised lines can be made out on two small grog-tempered sherds from the surface, but there are no diagnostic types other than Baytown Plain, *var. unspecified*.

Of particular interest from the Comfort Island site is the only diagnostic lithic artifact recovered from the assessment of eight sites. A stemmed projectile point was collected from the surface in Area B. This dart point is made of a dark yellowish brown, heat-treated gravel chert (Figure 73). It measures 44 mm in length and 22.5 mm in width. The triangular blade, slightly-obtuse shoulders and rectangular stem are characteristic of the Kent type, which is known to date as early as the Late Archaic period but is also commonly associated with Tchula and Marksville period contexts (Hays and Weinstein 2010:104; McGimsey 2010:127; Webb 2000:10–11). Besides offering additional evidence for a Middle Woodland period occupation apparently destroyed by shoreline erosion, this little dart point exemplifies a radically different ecological niche from 1,600 or more years ago.



Figure 73. Kent point from Comfort Island (16SB178).

Heat-treated gravel chert (both sides).

#### 6.1.5 Site 16SB153

Site 16SB153 is one of the better-known sites included in this study. Previous investigations by Coastal Environments, Inc. informed the present study (Weinstein et al. 2012), including the placement of a single 1-by-1 meter test unit in order to obtain samples for analysis. As the easternmost of the two control sites in this investigation, Site 16SB153 was also the only site not assessed during the oil spill response. Coastal Environments recorded deeply buried cultural deposits in terrestrial and submerged portions of the site on the south shore of Lake Borgne. In some areas, these cultural deposits were in excess of 1.5 meters thick. Investigators suggested part of the buried shell midden was redeposited by ancient storm surges or hurricanes. The prehistoric occupation dates principally from the Mississippi period (Bayou Petre phase), with a possible late Coles Creek period component (Weinstein et al. 2012:187–188).

Though limited in scope by comparison, the present study confirms the principal conclusions made by investigators with Coastal Environments that Site 16SB153 consists of a Mississippian component deeply buried by reworked shell midden. The Native American ceramic assemblage from TU 1 is not especially large (n=104) and one-third (n=34; 33%) consists of very small (<0.5 inch) sherdlets. Most of these contain variable amounts of shell temper. A majority of the 112 sherds from the site, including eight collected from the surface, are provisionally classified as Mississippi Plain, *variety unspecified* (n=43; 39%). Two Mississippi Plain rim sherds with strap handles were collected form the surface in Area B (Figure 74). Coastal Environments identified only two shell-tempered sherds from 16SB153 as Mississippi Plain, *var. Pocahontas*, describing these as "tan to light gray" with a "compact paste and a matte surface finish" (Weinstein et al. 2012:175). Based on these criteria, some of the Mississippi Plain sherds from TU 1 and the surface might be classified under *var. Pocahontas*. As with the grog-tempered sherds from other sites, the small size of many sherds made it difficult to designate types beyond classification of temper.

A significant portion of the ceramic assemblage from Site 16SB153 consists of small shell-tempered or combined grog and shell tempered sherds of an indeterminate type (n=30; 27%; Table 12). The combined use of shell and grog as a tempering agent occurs in late Mississippi period Plaquemine components in the delta (Livingood 2007; Shuman 2007). Some of the shell-tempered sherds described here as indeterminate type have a fine sandy paste. Based on whether the shell tempering is fine or coarse, these might be classified as Graveline Plain, *var. Proctor Point*, or Guillory Plain, *var. St. Bernard* (Weinstein et al. 2012:171). But further complicating the issue of typology, some of the grog-tempered sherds with a minor amount of shell temper also have a fine sandy paste.

Table 12. Artifacts from Site 16SB153

	Sur	face			Τl	J 1				TU	1 SW	1/4	
	A	В	L2	L3	L4	L5	L6	L7	90-110 cm	110-128 cm	128-138 cm	138-158 cm	158-178 cm
NATIVE CERAMICS													
Bell Plain, var. unspecified	1												
Coles Creek Incised, var. unspec.	1												
Mound Place Incised, <i>var.</i> unspec.							1				1		
Mississippi Plain, <i>var. unspecified</i>	4	2					11	4	12	2	3		5
Indet. shell tempered			5	2	7	6	3					4	
Indet. grog and shell tempered			_	3				1					
Indet. sherdlets (<0.5")			1	10	6			5	3	5	4		



Figure 74. Ceramic sherds from Site 16SB153.

Top Row: Coles Creek Incised, *var. unspecified*, from Area A (left) and Mississippi Plain rims with strap handles from Area B. Bottom Row: Mound Place Incised *var. unspecified* from TU 1.

Two sherds of Mound Place Incised, *var. unspecified*, were recovered from TU 1, in Level 6 and from 128 cm to 138 cm below surface in the southwest quarter of the unit. These might be classified as *var. St. Malo*, a provisional variety described as a shell-tempered ware similar to Graveline, but with two or more

parallel, horizontal incised lines on the exterior of bowls (Weinstein et al. 2012:175). The two sherds from TU 1, however, are too small to identify vessel form. The only other diagnostic pottery types from the site are one sherd of Bell Plain (*var. unspecified*) and Coles Creek Incised (*var. unspecified*) from the surface in Area A.

Historic artifacts are conspicuously absent from the TU 1 artifact assemblage. This is further indication of undisturbed cultural deposits dating from the Mississippi period (Bayou Petre phase), deeply buried beneath reworked shell midden. One shell-tempered sherd from Level 6 in TU 1 was submitted for absorbed residue analysis and another sherd from Level 7 was sent to MURR for analysis by ICP-MS. The latter was a grog-tempered sherd with a minor amount of shell temper and fine sandy paste.

### 6.1.6 Scow Island Scatter (16SB182)

The majority of the Native American ceramic assemblage from Scow Island Scatter consists of Baytown Plain, *var. unspecified* sherds (n=45; 80%). A single sherd each of Coles Creek Incised, *vars. Stoner* and *unspecified*, and Marksville Incised, *vars. Marksville, Sunflower*, and *Yokena* were recovered from the surface in Areas B and C (Table 13; Figures 75 and 76). Four sherds of Marksville Incised, *var. unspecified*, were surface collected from Areas A and B. A similar ceramic assemblage was found at the site during the oil spill assessment, including Baytown Plain, *var. unspecified* and Marksville Incised, *vars. Yokena* and *unspecified* (Cloy and Ostahowski 2015:6-783). The ceramic assemblage from beneath the surface of Scow Island Scatter is otherwise nondiagnostic and in redeposited contexts.

HDR, Inc. described Scow Island Scatter as a secondary beach deposit lacking subsurface archaeological integrity, with no subsurface cultural materials (Cloy and Ostahowski 2015:6-785). The present study recovered Baytown Plain sherds from four of six shovel tests and two 1-by-1 meter test units. These were mostly distributed in the upper levels, with a few sherds from level 3 (20-30 cm) in TU 1 and TU 2. Glass shards were recovered from Level 2 in TU 1 and Level 1 in TU 2.

Table 13. Artifacts from Scow Island Scatter (16SB182)

	Sı	urfac	е	ST 2	ST 4	ST 5	ST 6		TU 1			TU 2	
	Α	В	U	L1	L2	L2	L2	L1	L2	L3	L1	L2	L3
NATIVE CERAMICS													
Baytown Plain, <i>var. unspecified</i>	10	12	5	1	1	4	2	3	2	1		2	2
Coles Creek Incised, var. Stoner			1										
Coles Creek Incised, var. unspec.		1											
Marksville Incised, var. Marksville			1										
Marksville Incised, var. Sunflower			1										
Marksville Incised, var. Yokena			1										
Marksville Incised, var. unspec.	2	2											
Indet. sand tempered											1		
Indet. sherdlets (<0.5")										1			
HISTORIC													
Glass, container fragments									3		2		
Metal, iron fragments					1								



Figure 75. Ceramic sherds from Area B at Scow Island Scatter (16SB182).

Coles Creek Incised, var. unspecified (upper left); Marksville Incised, var. unspecified (upper right and below).



Figure 76. Ceramic sherds from Area C at Scow Island Scatter (16SB182).

Coles Creek Incised, *var. Stoner* (upper left); Marksville Incised, *var. Yokena* (upper middle); Marksville Incised, *var. Sunflower* (upper right); Marksville Incised, *var. Marksville* (lower left).

At the bottom of both test units, a layer of dark gray (10YR 4/1) silty clay with gray (10YR 5/1) silty clay mottles (Stratum III) was void of the *Rangia* shell hash found in the upper strata. Part of a metal crab trap

was found in the south wall of TU 2 at the base of Level 3, however, indicating a disturbed context. The findings of this study support the assessment by HDR, Inc. that Scow Island Scatter is a secondary beach deposit that lacks subsurface archaeological integrity. Nonetheless, twelve special samples were collected from the two test units and materials from seven of these were the subject of further analysis.

### 6.1.7 Redfish Slough (16LF293)

Redfish Slough is another beach deposit of wave-worn pottery and shell recorded by HDR archaeologists as consisting of redeposited materials from an undocumented, now submerged site. The ceramic assemblage collected by HDR consists mostly of Baytown Plain (*varieties Addis, Cataouatche* and *unspecified*), along with Bell Plain, *var. unspecified*; Coles Creek Incised, *var. unspecified*; Evansville Punctated, *var. Rhinehart*; and Mississippi Plain, *var. unspecified* (Cloy and Ostahowski 2015:6-375). The ceramic assemblage recovered from the surface and subsurface during the present study is consistent with Coles Creek and Mississippian components (Table 14).

Table 14. Artifacts from Redfish Slough (16LF293)

	S	urfac	е		1	Sh	ovel	Test				TL	J 1			1	TU 2		
	Α	В	С	1	3	4	5	7	8	9	L1	L2	L3	L4	L1	L2	L3	L4	L5
NATIVE CERAMICS																			
Avoyelles Punctated, var. Avoyelles		1																	
Baytown Plain, <i>var.</i> unspecified	9	11	4	4		1	2		11	8	6	2	1	4	2	2	2	1	3
Bell Plain, var. unspecified			3																
Coles Creek Incised, <i>var. Blakely</i>			1																
Evansville Punctated, <i>var.</i> unspec.			1																
Leland Incised, var. unspecified		2																	
Indet. grog-temp		1							2										
Indet. shell-temp. incised									1										
Indet. grog and shell tempered									1			1		1			2		1
Indet. sherdlets (<0.5")						1	4		1				1			3	5		
HISTORIC																			
Glass, container				1	1		1	1	2		1		3	2				1	
Metal																			
Iron, wire fragments				1					1			3		1	2		1		2
Tin, fragment														1					
Plastic, Indet.														2					
Rubber, indet.														2					

The ceramic assemblage consists mostly of Baytown Plain, *var. unspecified* sherds (n=73), along with minor amounts of Avoyelles Punctated, *var. Avoyelles* (n=1); Bell Plain, *var. unspecified* (n=3); Coles Creek Incised, *var. Blakely* (n=1); Evansville Punctated, *var. unspecified* (n=1); and Leland Incised, *var. unspecified* (n=2). With the exception of one small incised shell-tempered sherd of indeterminate type, all of the decorated sherds were collected from the surface in Areas A, B, and C (Figures 77 and 78). Subsurface recovery of ceramics otherwise consisted entirely of Baytown Plain and small grog-tempered, or grog and shell tempered sherds.



Figure 77. Ceramic sherds from Area B at Redfish Slough (16LF293).

Leland Incised, *var. unspecified* (left and center), and Avoyelles Punctated, *var. Avoyelles* (right).



Figure 78. Ceramic sherds from Area C at Redfish Slough (16LF293).

Coles Creek Incised, *var. Blakely* (left); Evansville Punctated, *var. unspecified* (center); Bell Plain, *var. unspecified* (right). The white substance on the surface of two sherds is bird excrement.

Seven of nine shovel tests produced artifacts, although six of these included historic artifacts such as container glass. Along with successive strata that contained varying amounts of broken *Rangia* shell and shell hash, the distribution of historic artifacts in the two 1 m-by-50 cm test units confirms the presence of disturbed contexts. Historic artifacts such as container glass and pieces of plastic, rubber and metal were found in every level. A layer of very dark grayish brown (10YR 3/2) to dark gray (10YR 4/1) clay loam was found at the bottom of both test units (Stratum IV). This stratum may represent a buried A-horizon, as it was devoid of shell and contained marsh grass and roots.

While Baytown Plain, *var. unspecified*, and grog and shell-tempered sherds are associated with Stratum IV, the stratum also produced fragments of glass containers, wire, plastic, and rubber. An auger test was placed at the bottom of TU 2 to examine the potential for more deeply buried cultural deposits, to a total depth of 210 cm below surface. The auger test did not produce any evidence of cultural deposits or shell midden. The data supported the assessment that Redfish Slough lacks undisturbed subsurface cultural deposits. Nonetheless, seventeen special samples were collected from Redfish Slough; nine of these were submitted for chemical, radiometric and absorbed residue analyses.

### 6.1.8 Acorn Mounds (16SB185)

Acorn Mounds remains somewhat enigmatic due to the virtual absence of cultural materials that can be reliably associated with the site. As discussed earlier, archaeologists with HRD, Inc. collected only a handful of Native American ceramics from the shoreline east and southeast of the mounds. The present study similarly resulted in very few artifacts, including two Baytown Plain, *var. unspecified* sherds from the shoreline more than 120 meters east of Mound B. The paucity of artifacts is undoubtedly because, in part, the surface has greatly subsided and has been covered with marsh sediment since the site was occupied. All seven of the shovel tests were negative, despite the fact that several were excavated to 80 cm below surface.

The 1-by-1 m test unit (TU 1) placed 30 meters north of Mound A revealed evidence of a dense layer of oyster shell to a depth of at least 145 cm below surface. Though it was nearly void of artifacts, several small pieces of plastic were recovered from more than 95 cm below surface (Table 15). The dense shell deposit may be a natural accumulation dating from the historic period, possibly produced by storm surges or associated with historic oyster harvesting. The off-mound areas of the site are thus appear to be deeply buried by 1.5 meters or more of marsh sediment and accumulated shell deposits. Shovel tests excavated into the mound summits by archaeologists with HDR, Inc. were negative for cultural materials. During the present study, a 1-by-0.5 m test unit (TU 2) excavated into the northern flank of Mound B produced only one small (<0.5 inch) grog-tempered sherd with sand in the paste. As described in the following section, coring produced additional information on the Acorn Mounds site.

**Table 15. Artifacts from Acorn Mounds (16SB185)** 

	Surface	TU 1	TL	12
	Area A	>95 cm	L2	L3
NATIVE CERAMICS				
Baytown Plain, var. unspecified	2			
Grog-tempered, with sand (<0.5")				1
HISTORIC				
Plastic, indet. fragments		3	1	

# 6.2 Soil Cores from Acorn Mounds (16SB185)

The ULL crew used a hammer-driven soil coring technique at the Acorn Mounds site to collect samples and assess deeply buried cultural deposits. This was necessary because of subsidence, but it was also an effective means of assessing the presence of oil and collecting information on mound construction. Acorn Mounds was the only site where vertical core samples were collected using the hammer-driven 2-inch (5 cm) diameter core with butterfly valve for marsh soils. The coring methodology is described in the section on sampling methods (Section 5.2.2). Vertical site coring is distinguished from unit core samples, which were typically 6 to 8 inch (15 to 20 cm) deep samples of matrix collected from within excavation unit walls in 2.375-inch (6 cm) diameter steel tube. The following results of site coring are confined to stratigraphic analysis and interpretation based on core samples collected in sealed, 12-inch (30 cm) long aluminum sleeves. Additional analytical results obtained from the core samples are presented in subsequent sections, including radiocarbon dating and the chemical detection and characterization of hydrocarbons.

The crew conducted seven core tests (CT) at the Acorn Mounds site. CT 1 through CT 4 were placed on the summit or north flank of Mound B, while CT 5 and CT 6 were located north of Mound B. CT 5 was midway between Mound B and the unnamed channel that bisects the northern portion of the island. CT 6 was just south of the channel. CT 7 was on the channel shoreline to the east, just west of Keelboat Pass. The core tests were positioned to assess the potential movement of oil into the site from the shoreline of Keelboat Pass. The crew obtained a series of core samples (CS) from each core test. As described in Section 5.2.2, each core sample was sealed with aluminum foil within a 12-inch (30 cm) long aluminum sleeve and transported to the lab for further analysis. CT 1, CT 3, CT 4 and CT 6 each produced one core sample. CT 2 produced five core samples and CT 5 and CT 7 each produced four core samples (Table 16; Appendix A.8).

Table 16. Core samples from Acorn Mounds (16SB185)

CT-CS	SS No.	Depth (cm)	Munsell Color, Texture and Description
1-1	27	0–30	Very dark grayish brown (10YR 3/2) clay loam with roots & organic material (Scatlake marsh alluvium)
2-1	28	0–28	Very dark grayish brown (10YR 3/2) clay loam with roots & organic material (Scatlake marsh alluvium)
2-2	29	75–105	75–95 cm: very dark gray (10YR 3/1) silty clay with charcoal; 95–105 cm: gray (10YR 5/1) clay loam with dark gray (10YR 4/1) & very dark gray (10YR 3/1) mottles, flecks of charcoal & <i>Rangia</i> shell
2-3	30	125–152	125–145 cm: gray (10YR 6/1) silt loam with gray (10YR 5/1) & dark gray (10YR 4/1) laminated silt; 145–148 cm: <i>Rangia</i> shell in dark gray (10YR 4/1) to very dark gray (10YR 3/1) silty clay; 148–152 cm: dark gray (10YR 4/1) silty clay with very dark gray (10YR 3/1) & black (10YR 2/1) mottling, charcoal & shell flecks
2-4	31	152–180	152–170 cm: gray (10YR 5/1) silty clay with dark gray (10YR 4/1) laminated silt; 170–178 cm: <i>Rangia</i> shell in gray (10YR 5/1) silty clay with dark gray (10YR 4/1) mottles; 178–180 cm: gray (10YR 5/1) silty clay with dark gray (10YR 4/1) mottling, charcoal & shell flecks
2-5	32	210–239	Homogenous gray (10YR 5/1) silty clay with dark gray (10YR 4/1) laminated silt (Scatlake marsh alluvium)

CT-CS	SS No.	Depth (cm)	Munsell Color, Texture and Description			
3-1	33	0–30	0–10 cm: grayish brown (10YR 5/2) silt loam with organic material & root mat; 10–30 cm: grayish brown (10YR 5/2) silty clay loam (Scatlake marsh alluvium)			
4-1	34	180–216	180–195 cm: light gray (10YR 7/1) silty clay; 195–199 cm: light gray (10YR 7/1) silty clay with gray (10YR 6/1) mottles, unidentified bone fragments & one <i>Rangia</i> shell; 199–210 cm: gray (10YR 6/1) silty clay; 210-216 cm: gray (10YR 6/1) silty clay with <i>Rangia</i> shell & charcoal flecks			
5-1	35	0–33	Gray (10YR 5/1) silt loam with organic material & root mat (Scatlake marsh alluvium)			
5-2	36	114–141	Very dark gray (10YR 3/1) silty clay (Scatlake marsh alluvium)			
5-3	37	195–226	Very dark gray (10YR 3/1) silty clay (Scatlake marsh alluvium)			
5-4	38	225–340	Very dark gray (10YR 3/1) silty clay with gray (10YR 5/1) silt (Scatlake marsh alluvium)			
6-1	39	0–30	0–25 cm: dark gray (10YR 4/1) silt loam with gray (10YR 5/1 and 10YR 6/1) laminated silt, organic material and root mat; 25-30 cm: dark gray (10YR 4/1) silt loam with gray (10YR 5/1 and 10YR 6/1) and light gray (10YR 7/1) mottles (Scatlake marsh alluvium)			
7-1	40	0–30	Dark gray (10YR 4/1) silt loam with organic material & root mat (Scatlake marsh alluvium)			
7-2	41	93–123	93–103 cm: very dark gray (10YR 3/1) silty clay with organic material & roots; 103–123 cm: very dark gray (10YR 3/1) to black (10YR 2/1) silty clay (Scatlake marsh alluvium)			
7-3	42	161–192	Very dark gray (10YR 3/1) silty clay (Scatlake marsh alluvium)			
7-4	43	280–310	Gray (10YR 5/1) silty clay (Scatlake marsh alluvium)			

Core Test 1, Sample 1 (CT 1-1, SS 27), was collected on the north flank of Mound B to a depth of 30 cm below surface. The soil was a very dark grayish brown (10YR 3/2) clay loam with roots and organic inclusions. It appeared to be a naturally deposited marsh alluvium comparable to the upper stratum described in the shovel tests at the Acorn Mounds site during the oil spill assessment (Cloy and Ostahowski 2015:6-802). There was no discernible mottling characteristic of mound fill. CT 1 ended prematurely due to inclement weather without the collection of additional samples. A sub-sample from CT 1-1 (SBH027) was analyzed for the presence of hydrocarbons, as described in the next section.

Core Test 2 was located east of CT 1 and reached a depth of 239 cm below surface (Figure 79). Both CT 1 and CT 2 were near Auger Test 3, which provided stratigraphic information for the collection of core samples. The first core sample (CS 1) from CT 2 was a very dark grayish brown (10YR 3/2) clay loam with roots and organic material to a depth of 28 cm, similar to the alluvium found in CT 1-1. AT 3 had produced evidence of mottling thought to be possible mound fill clay at approximately 82 to 112 cm below surface. This stratum was targeted for collection in Core Test 2, Sample 2. CT 2-2 was a very dark gray (10YR 3/1) silty clay with charcoal from 75 to 95 cm, underlain by gray (10YR 5/1) clay loam, with dark gray (10YR 4/1) and very dark gray (10YR 3/1) mottles, flecks of charcoal, and *Rangia* shell from 95 to 105 cm (Figure 80). The layers within CT 2-2 appear to be mound fill. These strata may alternatively represent a domestic midden from 95 to 105 cm, covered by a mound construction episode.

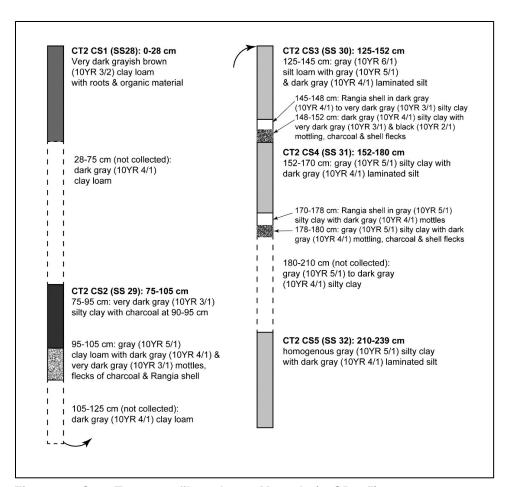


Figure 79. Core Test 2 profile at Acorn Mounds (16SB185).

Special samples 28-32 extracted from the north flank of Mound B.



Figure 80. Core Test 2, Core Sample 2 from Acorn Mounds (16SB185).

Special sample 29 extracted from the north flank of Mound B, with upper portion on the left.

Core Test 2, Sample 3, is gray (10YR 6/1) silt loam with gray (10YR 5/1) and dark gray (10YR 4/1) laminated silt from 125 to 145 cm below surface (Figure 81). This is underlain, from 145 to 148 cm, by a deposit of broken and whole *Rangia* shell in dark gray (10YR 4/1) to very dark gray (10YR 3/1) silty clay. Beneath this lies a layer of dark gray (10YR 4/1) silty clay, with very dark gray (10YR 3/1) and black (10YR 2/1) mottling that contains charcoal and shell flecks to 152 cm. The laminated silt in the upper portion of CT 2-3 (125 to 145 cm) appears to have been caused by a tidal or flood deposit, but the lower strata (145 to 152 cm) appear to be cultural deposits containing *Rangia* and charcoal.



Figure 81. Core Test 2, Core Sample 3 from Acorn Mounds (16SB185).

Special sample 30 extracted from the north flank of Mound B, with upper portion on the left.

Core Test 2, Sample 4, presents a stratigraphic sequence comparable to CT 2-3 (Figure 82). A layer of gray (10YR 5/1) silty clay with dark gray (10YR 4/1) laminated silt is underlain by a deposit of broken and whole *Rangia* shell in gray (10YR 5/1) silty clay with dark gray (10YR 4/1) mottles. Beneath this is a layer of gray (10YR 5/1) silty clay with dark gray (10YR 4/1) mottling, charcoal and shell flecks. As in Core Test 2, Sample 3, the upper portion of CT 2-4 (152 to 170 cm) appears to be a natural marsh alluvium, as indicated by the laminated silt. The strata beneath this, from 170 to 180 cm, appear to be cultural deposits containing *Rangia* and charcoal flecks. In contrast, Core Test 2, Sample 5, is a relatively homogenous, gray (10YR 5/1) silty clay with dark gray (10YR 4/1) laminated silt from 210 to 239 cm. This stratum is consistent with a naturally deposited Scatlake marsh alluvium (Trahan et al. 1989:52).



Figure 82. Core Test 2, Core Sample 4 from Acorn Mounds (16SB185).

Special sample 31 extracted from the north flank of Mound B, with upper portion on the left.

The crew collected two additional core samples, CT 3-1 and CT 4-1, from the summit of Mound B. These were both located southeast of Core Test 2. Core Test 3-1 is a grayish brown (10YR 5/2) silt loam with a root mat and organic material to a depth of 10 cm below surface. Beneath this, from 10 to 30 cm, is a grayish brown (10YR 5/2) silty clay loam, consistent with a naturally occurring marsh alluvium. CT 4-1 was collected at the bottom of Auger Test 4, from a depth of 180 to 216 cm below surface. The upper layer (180 to 199 cm) is a light gray (10YR 7/1) silty clay, with gray (10YR 6/1) mottles, bone fragments of an unidentified taxon and one *Rangia* shell beneath 195 cm. The fragments of bone were extracted from the core and submitted for radiocarbon analysis (Section 6.6). The gray mottling in light gray silty clay may represent initial stages of mound fill. A gray (10YR 6/1) silty clay lies beneath this, from 199 to 210 cm. This is underlain by a layer of gray (10YR 6/1) silty clay with *Rangia* shell and charcoal flecks from 210 to 216 cm. The shell and charcoal flecks in the lower stratum of CT 4-1 may represent cultural deposits on a pre-mound surface. A sample of the charcoal was sent for radiocarbon analysis (Section 6.6).

Nine additional core samples were collected from Core Tests 5, 6, and 7, to the north and east of Mound B. In contrast to the stratigraphy encountered within Mound B, these cores revealed strata characteristic

of the natural Scatlake marsh alluvium prevalent in the area (Trahan et al. 1989:52). Core Test 5 produced four samples, with gray (10YR 5/1) silt loam containing organic material in the upper stratum and very dark gray (10YR 3/1) silty clay in the lower stratum, to a depth of 340 cm below surface. There was no evidence of cultural deposits in CT 5, 6 or 7. Samples from CT 5-1 and CT 6-1 were analyzed for the presence of oil.

# 6.3 Detection and Characterization of Hydrocarbons

The chemical detection of oil in archaeological deposits supports a principal goal of this study—to assess the effects of an oil spill on archaeological sites along the Gulf Coast. As described in Appendix C and the methodology section of this report, the DES Lab at LSU analyzed 28 samples from eight sites in order to detect the presence and possible source of hydrocarbons (Table 17). Sample selection was based on the previously outlined criteria, including the potential for detecting crude oil in intact as well as redeposited archaeological contexts. Twenty-four of the samples consisted of soil matrix. These samples weighed between 44 to 426.8 grams prior to drying in the DES lab. Most of these soil samples contained inclusions, such as *Rangia* shell hash. Artifacts such as pottery sherds were removed during processing in the ULL lab. Two samples consisted only of *Rangia* (ANID JEB021 and JEB026) from Cheniere St. Denis (16JE2). Soil matrix and residue adhering to the shells were extracted in the DES lab. Two other samples were tar balls (SBH002 and SBH004) from Acorn Mounds (16SB185).

A detailed description of chemical characterization by GC/MS and fingerprinting for a possible match with MC252 oil are presented in Appendix C. The results are summarized in Table 17 by site and sample number, ANID, and provenience. Crude oil was detected in 12 of the 28 samples (42.9 percent). Not surprisingly, the two samples (SBE023 and SYA025) submitted from the two control sites (16SB153 and 16SMY17) tested negative for the presence of oil. These samples consisted of soil matrix from test units in relatively shallow, redeposited contexts. Despite the evidence for modern tidal redeposition, both samples produced negative results for hydrocarbons. Excluding the two samples from the control sites, some amount of oil was detected in 12 of 26 samples (46%) from six sites where oil was observed on the surface. In seven of the 12 samples from five sites that tested positive, the oil was too weathered for fingerprinting, or was inconclusive for MC252 as the possible source. Three samples (LFG045, SBDA015 and SBC026) from three sites (16LF293, 16SB174, and 16SB178) were positive for crude oil and a possible match for MC252 oil. Two samples (SBH002 and SBH004) produced evidence for hydrocarbons with a definitive match for MC252 oil. Both of these samples were from the Acorn Mounds site (16SB185).

The largest number of samples analyzed for the presence and possible source of oil were obtained from Cheniere St. Denis (n=6) and Acorn Mounds (n=6). Greater numbers of samples were selected for analysis from these sites based on the archaeological integrity of the deposits, to examine whether oil could be detected in undisturbed archaeological contexts. In contrast, recently redeposited contexts containing wave worn artifacts and shell hash were presumed to have a higher probability of testing positive for MC252 oil, particularly at sites where oil was observed on the surface during this study or during the oil spill response.

The DES lab detected oil in two of six samples from the Cheniere St. Denis site (JEB021 and JEB026). Both of the positive samples consisted of soil matrix and residue extracted from the concave surfaces of *Rangia* shells collected from levels 2 and 3 of TU 3. This test unit was excavated into the western slope of the northernmost mound at Cheniere St. Denis. Although hydrocarbons were present in both samples, the oil in the first sample (JEB021) was too weathered for chemical fingerprinting. The other sample (JEB026) was an inconclusive match for MC252. It is unknown whether the oil in these samples originated from the MC252 spill or another source.

Table 17. Results of GC/MS and oil source fingerprinting

Site-Sample No.	ANID	DES ID No.	Provenience	Material	g	Results
16JE2-21	JEB021	2015191-02	TU3 L2, 17-25 cm	3 <i>Rangia</i> shells	146.06	Oil detected; too weathered for fingerprinting
16JE2-26	JEB026	2016022-03	TU3 W half, L3, at 35cm	4 <i>Rangia</i> shells	88.27	Oil detected; inconclusive for MC252 oil
16JE2-33	JEB033	2014322-02	TU3 N wall, 20-30 cm	Soil	108.50	Negative
16JE2-35	JEB035	2014322-03	TU4 L1, 2-8 cm	Soil	111.42	Negative
16JE2-53	JEB053	2014322-04	TU2 N wall, 40-50 cm	Soil	120.42	Negative
16JE2-55	JEB055	2016022-09	TU2 E wall, L6-7, 55-65 cm	Soil	131.72	Negative
16LF293-40	LFGA040	2016022-01	TU2 L3, E half, 22-30 cm	Soil	56.92	Negative
16LF293-24	LFG024	2015230-03	TU1 N wall, L1, 0-10 cm	Soil	118.48	Negative
16LF293-45	LFG045	2015230-04	TU2 S wall, L2, 10-20 cm	Soil	152.75	Positive; possible match for MC252 oil
16LF293-48	LFG048	2016022-02	TU2 S wall, L5, 40-50 cm	Soil	241.50	Negative
16SB153-23	SBE023	2015230-08	TU1, N wall, 0-10 cm	Soil	136.05	Negative
16SB174-15	SBDA015	2016022-05	TU2 N wall, L3, 20-27 cm	Soil	142.98	Positive; possible match for MC252 oil
16SB174-6	SBD006	2015049-03	TU1 E wall, L3, 20-30 cm,	Soil	191.22	Negative
16SB174-16	SBD016	2015049-04	TU2 N wall, L2, 10-20 cm	Soil	114.75	Oil detected; too weathered for fingerprinting
16SB178-33	SBC033	2016022-04	TU2 N wall, L3, 20-30 cm	Soil	208.30	Negative
16SB178-26	SBC026	2015191-01	Surface near TU2	Soil	73.17	Positive; possible match for MC252 oil
16SB178-31	SBC031	2015049-01	TU2 N wall, L1, 0-10 cm	Soil	208.30	Oil detected; too weathered for fingerprinting
16SB178-32	SBC032	2015049-02	TU2 N wall, L2, 10-20 cm	Soil	170.36	Negative
16SB182-18	SBF018	2016022-10	TU2, E half, 19-34 cm	Soil	426.82	Negative
16SB182-21	SBF021	2015230-02	TU2 N wall, L2, 10-20 cm	Soil	100.40	Oil detected; too weathered for fingerprinting
16SB182-23	SBF023	2015230-01	TU1 W wall, L1, 0-10 cm	Soil	44.04	Oil detected; too weathered for fingerprinting
16SB185-27	SBH027	2016022-06	CT1, CS 1, 0-30 cm	Soil	247.25	Oil detected; too weathered for fingerprinting
16SB185-35	SBH035	2016022-07	CT5, CS 1, 0-33 cm	Soil	228.20	Negative
16SB185-39	SBH039	2016022-08	CT6, CS 1, 0-30 cm	Soil	233.47	Negative
16SB185-2	SBH002	2015230-05	Surface, Area A	Tar ball	44.50	Positive match for MC252
16SB185-4	SBH004	2015230-06	ST2 N wall, L1, at 20 cm	Tar ball	13.80	Positive match for MC252
16SB185-11	SBH011	2015230-07	TU1 W wall, 0-10 cm	Soil	180.50	Negative
16SMY17-25	SYA025	2014322-01	TU2, E wall, 30-38 cm	Soil	218.45	Negative

The first sample from Cheniere St. Denis that tested positive for oil (JEB021) consisted of soil matrix and residue adhering to three *Rangia* shells from Level 2 in TU 3, at a depth of 17 to 25 cm below surface. This sample came from the lower portion of Stratum II, which consisted mostly of crushed and broken shell. The condition of the shell and several small fragments of iron from Level 2 indicate a redeposited context potentially associated with the historic component of the site. The second sample that tested

positive for oil (JEB026) consisted of matrix and residue adhering to four *Rangia* shells in the west half of TU 3, at 35 cm below surface. These were obtained from Stratum III, a layer of whole *Rangia* shells and black (10YR 2/1) silty clay. As previously noted, Stratum III is thought to represent an intact depositional context associated with the Late Baytown-Coles Creek occupation of the site (Section 5.3.2). Stratum III represents intact mound fill.

The water table inundated TU 3 with the rising tide during excavation, so it is unknown whether the oil detected in these two samples was introduced with seawater during excavation or at some previous time. The four other samples from the Cheniere St. Denis site returned negative results for crude oil. This included soil from a column sample (16JE2-33; JEB033) collected from the north wall of TU 3 at 20 to 30 cm below surface, a depth clearly associated with Stratum III and the Late Baytown-Coles Creek component. That oil was not present in this sample suggests the infiltration of oil into the other two samples (JEB021 and JEB026) may have occurred at an earlier time. If oil contaminated these samples during excavation through saturation from the water table, all three samples should test positive for oil. Furthermore, oil was detected in soil matrix and residue adhering to the surfaces of *Rangia* shell. Incidental contamination by seawater during excavation seems unlikely, but cannot be ruled out.

The other three samples from the Cheniere St. Denis site that tested negative for oil came from excavated contexts in TU 2 and TU 4. TU 2 was also placed on the western slope of the northernmost mound. The TU 2 samples (JEB053 and JEB055) consisted of soil and shell matrix obtained from the north and east walls, within strata II and III, respectively. Both of these represent undisturbed mound contexts, but at greater depths than the samples from TU 3. The TU 4 sample (JEB035) consisted of soil from Level 1 (2 to 8 cm), near the southern shoreline at Cheniere St. Denis. The excavation of TU 4 was terminated at only 20 cm below surface due to inundation by the rising tide. If oil was contaminating excavation units through the water table or rising tide, sample 16JE2-35 (JEB035) should have tested positive.

The two samples from the Acorn Mounds site that produced positive matches for MC252 were tar balls (SBH002 and SBH004), collected from the surface of the shoreline and 20 cm below surface in ST 2, approximately 140 meters east of the mound and plaza precinct. Because the site boundaries have not been adequately determined by subsurface testing, it is unknown whether the samples that tested positive for MC252 were from within the inhabited area of the site. The shoreline near ST 2 is nonetheless the area closest to the mounds that has so far produced the majority of artifacts. The tar balls from the surface and ST 2 are both recently deposited contexts.

Four additional samples from the Acorn Mounds site were examined for the potential movement of oil into the site by tidal action or storm surge, along the waterway north of the mound and plaza precinct. A column sample (SBH011) from the west wall and first level of TU 1, north of Mound A, did not yield evidence for oil. The excavation of TU 1 revealed that any cultural deposits in this area along the bayou would be deeply buried. Because the sample from Level 1 tested negative for oil, no additional samples from TU 1 were submitted for chemical analysis. Two core samples from the Acorn Mounds site also tested negative for oil. One of these was collected from Core Test 6, Core Sample 1 (SBH039), within 30 cm of the surface, just south of the bayou and approximately 40 meters north of Mound B. The other sample was from Core Test 5, Core Sample 1 (SBH035), within 33 cm of the surface and approximately 20 meters north of Mound B.

Oil was detected in Core Test 1, Core Sample 1 (SBH027) from the northwest slope of Mound B, at a depth of 0 to 30 cm below surface. Because the oil was too weathered for chemical fingerprinting, a possible match with MC252 could not be ascertained. Although there is no evidence that the soil matrix in CT 1, CS 1 was redeposited, it may represent accretional marsh sediments from tidal and erosional processes. Though a source other than MC252 cannot be ruled out, the presence of oil in a core sample from Mound B appears to confirm the horizontal movement of crude oil into the interior of the site. The oil may have been transported by storm surge from the shoreline of Keelboat Pass, where the presence of

MC252 oil has been confirmed. Because the source could not be confirmed, it may plausibly represent another, unidentified release of oil in the vicinity of Mound B.

Samples from four other sites tested positive for oil (16LF293, 16SB174, 16SB178, and 16SB182). Except for the samples from Scow Island Scatter (16SB182), these produced possible matches for MC252 oil. All of the samples that produced evidence of oil are thought to be from disturbed or redeposited archaeological contexts. Four samples from the Redfish Slough site (16LF293) were tested. These were column and soil samples from TU 1 and TU 2. The only sample from Redfish Slough that tested positive for oil (LFG045) was soil matrix in a column sample from the south wall of TU 2, at a depth of 10 to 20 cm below surface. A redeposited context is indicated by shell hash and a few scattered historic artifacts such as container glass fragments in lower levels of this test unit.

Two of three samples from the Comfort Island site (16SB174) produced evidence of oil from excavated contexts. The first was a column sample from the north wall of TU 2 at a depth of 10 to 20 cm below surface (SBD016). The oil detected in this sample was too weathered for chemical fingerprinting. The second sample (SBDA015) was from a unit core in the north wall of TU 2, at a depth of 20 to 27 cm below surface. The oil in this sample was a possible match for MC252. Though the test unit stratigraphy and artifact assemblage indicate disturbed, secondary contexts for these samples, the chemical analysis confirms the accumulation of oil along with artifacts within reworked midden deposits.

Four samples from the Southern Comfort site (16SB178) were analyzed for the presence of oil and two of these tested positive. One of these was a soil sample collected from the surface near TU 2, which produced a possible match for MC252 oil (SBC026). The other was a column sample from Level 1 in the north wall of TU 2 (SBC031). Though the possible source of the oil in the column sample could not be determined by chemical fingerprinting, it consisted of soil matrix in a redeposited context. Two additional samples from levels 2 and 3 in the north wall of TU 2 tested negative for oil, suggesting that oil at the Southern Comfort site may have been limited to the surface and upper 10 cm of redeposited shell midden at the time of the fieldwork.

Three samples from Scow Island Scatter (16SB182) were analyzed for the presence of oil. These were samples of soil matrix from excavated contexts in TU 1 and TU 2. Oil was detected in two samples, but in both instances it was too weathered for fingerprinting as to its potential source. One was a column sample from Level 1 (0–10 cm) in the west wall of TU 1 (SBF023). The other was a column sample from Level 2 (10–20 cm) in the north wall of TU 2 (SBF021). Both are associated with redeposited shell midden that lacks archaeological integrity. The sample that tested negative for oil was from a unit core at a depth of 19 to 34 cm below surface, indicating oil was not present at this depth in TU 2.

Although the samples that tested positive for oil from the Redfish Slough, Comfort Island, Southern Comfort, and Scow Island Scatter sites were from secondary contexts, the results of the chemical analysis confirm the presence of crude oil in redeposited shell midden at depths of 10 to 27 cm below surface. The potential effects of oil on coastal archaeological sites are consequently occurring in combination with the processes of site submergence, shoreline erosion and the redeposition of cultural materials. The chemical analysis of samples from the Acorn Mounds and Cheniere St. Denis sites indicates that oil is not limited to redeposited archaeological contexts. The oil from a spill may contaminate intact archaeological contexts through normal wave action, storm surge or the water table. The results of this chemical analysis informed selection of samples for other analytical techniques, including the elemental analysis of ceramics.

# 6.4 Elemental Analysis

The MURR Archaeometry Laboratory conducted elemental analyses for this study by neutron activation analysis (NAA) and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS). These

techniques examined the potential effects of oil on trace element analysis and associated provenance studies, including pretreatment, analytical techniques, and overall cost estimation. The MURR Archaeometry Lab requested a minimum sample size of approximately one square inch for each ceramic specimen to be analyzed by NAA or LA-ICP-MS. One gram was the minimum recommended sample size for lithic artifacts. Only a small number of lithics (n=31) were recovered from three of the eight sites that were sampled, as described in Section 6.1. No lithics were submitted for elemental analysis, as most of the lithic material weighed less than one gram and was not recovered from contexts where the presence of oil was chemically detected.

Five ceramic samples were sent to the MURR Archaeometry Laboratory for application of NAA and LA-ICP-MS (Table 18). The samples were packaged in aluminum foil envelopes that were in turn placed in cloth bags and shipped overnight in cardboard boxes. The first sample was a one grog-tempered pottery sherd (SBC035) collected from Level 1 in TU 2 at the Southern Comfort site (16SB178), from a redeposited context which GC/MS analysis determined to be contaminated with oil. Three other samples were grog-tempered pottery sherds (SBDA011, SBDB011, and SBD012) collected from Level 2 in TU 2 at the Comfort Island site (16SB174). Oil was independently detected in the surrounding soil matrix. The fifth sample was a grog-tempered sherd (SBE017) collected from Level 7 (135–145 cm) in TU 1 at the control site on Lake Borgne (16SB153).

Table 18 presents the results of the elemental analyses of the five ceramic samples. The methods and results are described in more detail in Appendix D. Application of NAA on sample SBC035 collected from an oiled context in TU 2 at Southern Comfort indicated elevated levels of Arsenic, possibly due to oil, but no increased concentrations in elements known to be present in crude oil, such as Nickel or Vanadium. The results of LA-ICP-MS on the same sample were inconclusive. Trace-element analysis of the three sherds from oiled contexts at Comfort Island (SBDA011, SBDB011, and SBD012) produced negative results for elevated concentrations of elements known to be present in crude oil (Appendix D).

Table 18. Samples analyzed by the MURR Archaeometry Lab

Site & Sample	ANID	Provenience	Material	g	Results
16SB178-35	SBC035	TU2 L 1, 5-10 cm	1 grog-tempered sherd from oiled context	10.75	Elevated levels of Arsenic
16SB174-11	SBDA011	TU2 L 2, 10-20 cm	1 grog-tempered sherd from oiled context	1.24	No effect
16SB174-11	SBDB011	TU2 L 2, 10-20 cm	1 grog-tempered sherd from oiled context	3.88	No effect
16SB174-12	SBD012	TU2 L 2, 15-20 cm	1 grog-tempered sherd in a soil sample from oiled context	5.75	No effect
16SB153-17	SBE017	TU1 L 7, 135-145 cm	1 grog-tempered sherd from a control site	7.90	No effect

Based on this analysis, Glascock concluded that oil contamination would not hinder applications of NAA or LA-ICP-MS on ceramic sherds. According to the findings of the MURR Archaeometry Lab, related provenance studies on the possible sources of ceramic materials and manufacture will not be effected. Based on the results of the elemental analyses of the ceramic samples, the Archaeometry Lab also reported that the presence of oil should not impede elemental analyses of lithics. Standard pretreatment techniques such as artifact washing and removal of potentially oiled surfaces with a burring tool are nonetheless recommended in contexts suspected to be contaminated with oil. Though applications of NAA and LA-ICP-MS for elemental analysis of ceramics do not appear to be hindered by the presence of oil, experimental studies might examine the use of these techniques in detecting levels of hydrocarbon

absorption in different kinds of archaeological materials. In combination with techniques such as GC/MS, this might provide an independent line of evidence for contamination of archaeological contexts by an oil spill.

## 6.5 Absorbed Residue Analysis

The detection and analysis of absorbed plant and animal residues in pottery sherds can provide evidence of past subsistence practices and foodways (Reber and Evershed 2006; Reber and Hart 2008). Residue analysis by GC/MS involves the extraction of lipids and other compounds absorbed within the ceramic matrix during vessel use or from the depositional environment, as part of site formation processes (as summarized in the Research Methodology section [5.4.4] and described in Appendix E of this report). For the purposes of this study, the analysis of absorbed residues was designed to also examine the potential presence of hydrocarbons in ceramics, and the possible effects of hydrocarbon contamination on the analysis of residues from plant and animal sources.

Eleanora Reber at the Archaeological Residue Laboratory (ARL), University of North Carolina Wilmington, analyzed 17 pottery sherds from seven sites for evidence of absorbed residues. The results of the chemical characterization of soil samples were among the most relevant criteria in selecting samples for absorbed residue analysis. The ANID tracking system kept this information confidential during the absorbed residue analysis. Reber (2016) describes the pretreatment of samples, extraction procedures, and application of GC/MS for absorbed residue studies in Appendix E of this report. In addition to lipids from animals and plants, Reber (2016) was able to identify biomarkers for crude oil in the pottery residues, as well as Corexit 9500 and Corexit 9527, the dispersants used in the MC252 oil spill cleanup (Table 19). Absorbed residue analysis of ceramics, then, provides evidence of possible contamination independent from the chemical characterization of hydrocarbons in soil samples. The following discussion begins with the results of absorbed residue analysis as it pertains to the presence of oil, dispersants, and other contaminants, in relation to the sampled archaeological contexts. The possible effects of hydrocarbon contamination on analytical techniques are considered in light of these results.

The absorbed residue analysis provides evidence for some level of oil, dispersant or other contaminants in 10 of the 17 sherds (58.8%) submitted for analysis. The biomarkers for oil and/or dispersant were identified in more than half (52.9%) of the samples, with both oil and dispersant identified in two sherds, some amount of oil without dispersant in three sherds, and dispersant without oil in four sherds. Two sherds exhibited compounds consonant with other contaminants, such as hand cream or surfactant (JEB043), or DEET and fragrance (SYA018), despite the special collection procedures and precautionary methods taken in the field and lab. Perhaps not coincidentally, some of the chemicals contained in Corexit 9500 and Corexit 9527, such as Sorbitan and tri-(9Z)-9-octadecenoate, are also found in skin cream and insect spray (Graham et al. 2016). Distinguishing dispersants from other contaminants in absorbed residues may consequently be problematic. The samples were otherwise free of bug spray, sunscreen, and other contaminants.

Among the preliminary issues to be addressed is the possible correlation of oil or dispersant in absorbed residues of pottery samples from sites known from independent lines of evidence to have been impacted by an oil spill. Oil and/or dispersant were detected in samples from three of the five sites where SCAT teams recorded the presence of oil during the MC252 response (16JE2, 16SB178 and 16LF293). Only three samples were analyzed from two other sites where oil was observed (16SB174 and 16SB182) and no pottery sherds were submitted for this analysis from Acorn Mounds (16SB185). The absence of oil and/or dispersant in absorbed pottery residues from sites 16SB174 and 16SB182 may be a result of the small sample size (n=3).

Table 19. Results of absorbed pottery residue analysis

Site-Sample No.	ANID	RL No.	Provenience	Description	g	Contaminants	Interpretation
	72			2000	9		
16JE2-29	JEB029	332	TU3 L4 at 39cm, east half	Baytown Plain	12.09	None	Not interpretable; insufficient residue
16JE2-22	JEB022	336	TU3 L2 at 17- 25cm	Baytown Plain	13.7	Small amount of oil	Difficult to interpret; meat and plant lipids
16JE2-59	JEB059	339	TU4 L2 at 11- 18cm	Baytown Plain	7.11	None	Not interpretable; insufficient residue
16JE2-25	JEB025	349	TU3 L3 at 30- 35cm	Baytown Plain	16.10	Dispersant	Not easily interpretable; small amount of mixed plant and animal-based lipids
16JE2-43	JEB043	334	TU2 L7 at 65- 70cm	Grog tempered	9.80	Hand cream or surfactant	Difficult to interpret; primarily plant lipids with a small amount of fish and/or shellfish
16SB178-28	SBC028	342	Surface, near TU1	Grog tempered	11.64	None	Difficult to interpret; algal lipids, with plants lipids
16SB178-37	SBC037	347	TU2 L1 at surface	Grog tempered	18.70	Oil and dispersant	Mixture of lipids from animals and terrestrial plants
16SB178-37	SBCA037	346	TU2 L1 at surface	Baytown Plain	13.10	None	Shellfish, plant lipids, and perhaps meat
16SB178-29	SBC029	333	Surface, near TU2	Baytown Plain	31.08	Oil	Not interpretable due to oil and dispersant contamination
16SB174-10	SBD010	340	TU2 L1 at 0- 10cm	Grog tempered, burnished	5.47	None	Mixture of plant and meat
16SB182-7	SBF007	335	TU1 L1 at 5- 10cm	Grog tempered	9.33	None	Difficult to interpret; primarily plant and/or fish, with animal lipids
16SB182-17	SBF017	341	TU2 L2 at 19 cm, east half	Baytown Plain	14.90	None	Not interpretable
16LF293-34	LFG034	338	TU2 L2 at 10- 20cm	Baytown Plain	4.88	Dispersant	Primarily terrestrial plants, with small amount of animal lipids
16LF293-18	LFG018	337	TU1 at 10cm	Baytown Plain	4.86	Oil	Primarily terrestrial plant, with small amount of animal lipids
16LF293-47	LFG047	348	TU2 L4 at 30- 40cm, south wall, column sample	Grog tempered	4.89	Dispersant	Difficult to interpret, probably a mixture of animal and plants, possibly including marine algal lipids
16SB153-15	SBE015	343	TU1 L6 at 125- 135cm	Shell tempered	9.19	Oil and dispersant	Mixture of animal and terrestrial plants
16SMY17-18	SYA018	345	TU2 L1 at 0- 10cm	Baytown Plain	8.93	Dispersant, DEET and fragrance	Mixture of meat and terrestrial plants

The biomarkers for oil and dispersant, or dispersant without oil, were unexpectedly detected in one sample from each of the control sites. A shell-tempered sherd (SBE015) from TU 1, Level 6 (125-135 cm below surface) at Site 16SB153 on Lake Borgne was found to contain both oil and dispersant. A Baytown Plain sherd (SYA018) from TU 2, Level 1, at Bayou Sale (16SMY17) contained dispersant, as well as DEET and fragrance. The presence of oil and dispersant in the sherd from Site 16SB153 is notable, in that

it was recovered from a deeply buried, intact context presumed to be uncontaminated by crude oil. In fact, the results of GC/MS on a soil sample from Level 1 in TU 1 were negative for oil. The sherd from Bayou Sale was from a redeposited context, so the presence of dispersant might be due to its extensive use during the MC252 oil spill response (Graham et al. 2016; Place et al. 2016; Wilson et al 2015).

Of particular interest for this study is the potential correlation of oil and/or dispersant contamination in the absorbed residues of pottery samples from archaeological contexts independently determined by chemical characterization to contain oil. Comparing the results of the absorbed residue analysis (Table 19) with the results of GC/MS analysis of soil matrices (Table 17), several inferences can be drawn. Of the nine sherds found to contain biomarkers for oil and/or dispersant, six (67%) were from associated strata or nearby soil matrices that tested positive for oil. Four of these were possible matches for MC252 oil. The three other sherds found to contain oil and/or dispersant were from archaeological contexts that tested negative for oil, including two sherds that contained both oil and dispersant (SBE015) or oil alone (LFG018). The third sample was from the Bayou Sale control site. As previously mentioned, dispersant was identified in a pottery sherd from TU 2, Level 1, at Bayou Sale. A soil sample beneath this, from the East wall of TU 2 (30–38 cm below surface) was negative for the presence of oil. This provides evidence for a correlation between oiled archaeological contexts and ceramic absorption of oil and/or dispersant; it also indicates the presence of oil in ceramic residues from contexts that otherwise appear to be uncontaminated.

The relative ubiquity of dispersant is also apparent in the absorbed residues of pottery sherds, having been identified with (n=2) or without (n=4) the presence of oil biomarkers in six samples. That dispersant was identified in pottery samples from both control sites, in contexts where overlying or underlying soil matrices tested negative for oil, might be due to the extensive use of dispersants during the MC252 oil spill response. The sample from TU 2 at Bayou Sale that contained dispersant was from a relatively shallow excavated context (Level 1). However, the shell-tempered sherd (SBE015) from Site 16SB153 that contained biomarkers for both oil and dispersant was collected from Level 6 (125 to 135 cm below surface) of TU 1. Although appropriate precautionary measures were followed during collection and processing, these findings cannot rule out inadvertent contamination during excavation. The results otherwise indicate the infiltration and absorption of oil and dispersant into a pottery sherd in a deeply-buried archaeological context. Regardless, the MC252 oil spill response did not involve control site 16SB153 on Lake Borgne. Oil was not observed at the site, nor was the oil in the absorbed residue chemically matched with the MC252 source.

In contrast, the absence of oil biomarkers and/or dispersant in absorbed residues does not closely correlate with archaeological contexts that otherwise tested negative for oil. Of the eight sherds that produced no evidence for oil and/or dispersants in absorbed residues, five were from archaeological contexts in which the soil matrices tested positive for oil (JEB029, SBCA037, SBD010, SBF007, and SBF017). The absence of oil and/or dispersant in absorbed residues should consequently not be interpreted to mean that oil is not present in associated archaeological contexts. In one instance, a pottery sample (SBD010) from Comfort Island that was described as not contaminated came from a test unit and level (TU 2, Level 1) in which a sample of sediment from the underlying level (TU 2, Level 2) tested positive for oil (SBD016). Hydrocarbons were not detected as having been absorbed into the sherd from TU 2, Level 1, despite the presence of oil in the soil matrix from TU 2, Level 2.

Only three sherds that produced no evidence of oil or dispersant (JEB059, JEB043, and SBC028) came from archaeological contexts that either tested negative for oil, or for which comparative data are lacking. Though the presence of biomarkers for oil and/or dispersant as absorbed residues in pottery sherds is correlated with archaeological contexts in which soil matrices tested positive for oil, the inverse relationship is less apparent. The absence of oil and/or dispersants in absorbed pottery residues is not correlated with archaeological contexts that tested negative for oil. Any further conclusions regarding the correlation of oil and/or dispersant contamination of pottery samples and surrounding soil matrices are constrained by the sample size. That two-thirds (66.7%; n=6) of the nine sherds with biomarkers for oil

and/or dispersant were from archaeological contexts in which the soil matrix tested positive for oil is a reflection of the sample selection criteria. It also confirms that oil, as well as dispersants used in the oil spill cleanup, can become part of the archaeological formation processes at coastal sites. Oil and dispersant are absorbed and can be detected in pottery sherds when present in archaeological deposits.

The presence of oil and/or dispersant as absorbed residues in pottery sherds from five sites, including the two control sites, is also an indication of the widespread extent of oil and dispersant at archaeological sites along Louisiana's Gulf Coast. Though the absorption of oil and/or dispersant in ceramic matrices may be part of the formation processes at coastal sites, these findings raise additional questions about oil and dispersant interaction in depositional environments and the geographic extent of contamination along the Gulf Coast.

A final issue involves the possible effects of oil and/or dispersant contamination on the analysis and interpretation of absorbed plant and animal residues in pottery sherds. Reber (2016) found that the presence of oil or dispersant in pottery residues can cause a loss of potential information on other absorbed residues. According to Reber, this is due in part to similarities in biomarkers for absorbed residues and contaminants. Distinguishing dispersant from other contaminants can be problematic, because the same chemicals commonly occur in products such as skin cream, cosmetics, and insect spray (Graham et al. 2016).

Reber calculated approximate percentage of contamination by quantifying total lipid and total biomarkers of contaminants per gram of sherd. Three sherds from excavated contexts at Cheniere St. Denis (JEB025) and Redfish Slough (LFG018 and LFG034) were found to be lightly contaminated (less than 1%), while contaminates in the sherd from the control site 16SB153 (SBE015) were light to moderate (at least 1%). Two sherds from Southern Comfort (SBC037) and Bayou Sale (SYA018) were moderately contaminated (at least 2%) and one sherd from Redfish Slough (LFG047) was moderately to severely contaminated (at least 6%).

Degree of contamination is not clearly associated with the relative depth of archaeological deposits, although this may be a reflection of the small sample size. The effect of a marine oil spill on surficial and shallow, redeposited materials should involve relatively higher levels of contamination than in deeply buried archaeological contexts. Based on these results, however, measures of contamination were relatively greater in sherds from deeper contexts at Site 16SB153 (SBE015) and Redfish Slough (LFG047) than from the upper levels of test units at Redfish Slough (LFG034 and LFG018). The lightly to moderately contaminated shell-tempered sherd (SBE015) from control site 16SB153 was collected from Level 6 (125 to 135 cm below surface) in TU 1.

Though these results are surprising, the analysis is constrained by the paucity of comparative data on percentage of contamination in disparate environmental conditions at different sites. As the majority of the sampled contexts are redeposited and the sample size is small, it is difficult to draw further conclusions about relative degree of contamination and archaeological context. Nonetheless, the application of dispersants does not necessarily protect surficial or deeply buried archaeological deposits from contamination following an oil spill. At least one study has shown that dispersant significantly accelerates and increases the depth of crude oil penetration into saturated sands, where degradation may be inhibited (Zuijdgeest and Huettel 2012). Similar experimental studies might produce comparative data by examining the separate and combined effects of oil and dispersant on different archaeological materials in the silts and clays of marsh and deltaic environments.

Reber's analysis indicates the effects of oil and/or dispersant on absorbed residue analysis hinges on relative amounts of contamination. An interpretation of absorbed residues associated with ceramic container use or function was possible for all of the sherds in which contamination was light, light to moderate, or moderate. The difficulty of interpreting absorbed animal and plant residues increased,

however, with relative percentage of contamination. One sherd collected from the surface at the Southern Comfort site (16SB178) was so severely contaminated with oil and dispersant that absorbed residues were uninterpretable (SBC029). An associated soil sample from the surface (SBC026) tested positive for oil and was a possible match for MC252. Though contamination with oil or dispersant presented difficulties in the interpretation of absorbed residues, the greatest difficulty and amount of information loss occurred in sherds contaminated with both oil and dispersant (Reber 2016; Appendix E). An oil spill and the use of dispersants in a cleanup response can adversely affect *in situ* preservation of information at coastal sites.

## 6.6 Radiometric Analysis

Radiocarbon dating by AMS yielded results for a total of 17 samples, which included eight pairs of subsamples (Table 20; Appendix F). As described in the methodology section, assessing the potential effects of oil on the pretreatment processes was a primary objective, along with the potential effects on radiocarbon dating results. A subdivided sample of faunal bone (JEB024 and JEBA024) from an unidentified mammal in Level 2 of TU 3 at Cheniere St. Denis (16JE2) produced uncalibrated conventional radiocarbon ages of  $870 \pm 30$  and  $780 \pm 30$  YBP, respectively, for subsamples processed by solvent extraction (Beta-421684) and standard pretreatment (Beta-421683).

Applications of GC/MS independently identified oil in the matrix (JEB021) and pottery residues (JEB022) in this context, although the oil was too weathered for fingerprinting as to its source. Oil was also chemically detected in the soil (JEB026), and dispersant in pottery residue (JEB025) from the underlying Level 3 in TU 3. The archaeological context of the radiocarbon sample appears to be undisturbed within the western flank of the northernmost mound at Cheniere St. Denis. The 2-sigma calibrated results (with 95.4% probability) significantly overlap (cal 1150 to 1225 and 1215 to 1280 CE) and generally concur with a late Coles Creek component based on diagnostic ceramic types.

Comparable results were produced in the radiocarbon analysis of subsamples from Comfort Island (16SB174). A sample of wood charcoal (SBDA012) was obtained from a redeposited soil matrix that tested positive for oil (SBD016) in Level 2 (15-20 cm) of TU 2 at Comfort Island. A soil sample (SBDA015) from the underlying level also tested positive for oil and is a possible match for MC252. Pretreatment of the charcoal by solvent extraction produced an uncalibrated conventional radiocarbon age of  $1230 \pm 30$  and 2-sigma calibrated calendar age of 685 to 885 CE (Beta-421682; with 95.4% probability). The subsample that received standard pretreatment produced a slightly later, uncalibrated conventional radiocarbon age of  $1170 \pm 30$  and 2-sigma calibrated calendar age of 770 to 905 and 920 to 965 CE (Beta-421681).

One of two samples from Scow Island Scatter (16SB182), an unidentified bone fragment (SBF010), received pretreatment by solvent extraction and produced a modern date. Although recovered from Level 3 (20-30 cm) in TU 1, it was in a recently redeposited context. The other sample from Scow Island Scatter was wood charcoal (SBF016) collected from a redeposited context in Level 2 of TU 2. The subsample pretreated by solvent extraction produced an uncalibrated conventional radiocarbon age of  $1020 \pm 30$  and 2-sigma calibrated calendar age of 980 to 1035 CE (Beta-421678). The subsample that received standard pretreatment produced a slightly later uncalibrated conventional radiocarbon age of  $970 \pm 30$  and 2-sigma calibrated calendar age of 1015 to 1155 CE (Beta-421677). As with the samples from the Cheniere St. Denis and Comfort Island sites, Beta Analytic identified no statistically significant differences in the radiocarbon dating results that might be associated pretreatment methods. Although the samples from Comfort Island and Scow Island Scatter were from disturbed contexts, it is worth noting that four of the five dates are consonant with late Coles Creek components at these sites.

Table 20. Results of radiocarbon dating

Site	ANID	Beta ID	Provenience	Pretreat	Uncal. Conv. 14C Age <sup>†</sup> (B.P.)	⁵13C	2-sigma Calibrated
16JE2	JEB024 & JEBA024 <sup>1</sup>	421684	TU3 L2 at 25- 35 cm	Solvent	870 ± 30	- 22.5	Cal 1050 to 1085 CE (Cal 900 to 865 YBP) and Cal 1125 to 1140 CE (Cal 825 to 810 YBP) and Cal 1150 to 1225 CE (Cal 800 to 725 YBP)
		421683	TU3 L2 at 25- 35 cm	Standard	780 ± 30	NA	Cal 1215 to 1280 CE (Cal 735 to 670 YBP)
16LF293	LFG020	421672 & 421671	TU1 at 16 cm	Failed collagen extraction	Cancelled		NA
16LF293	LFG038 <sup>1</sup>	421670 & 421669	TU2 at 36 cm	Failed collagen extraction	Cancelled		NA
16SB153 SBE03	SBE033	421662	TU1 at 134- 139 cm W wall	Solvent	450 ± 30	-13.0	Cal 1420 to 1465 CE (Cal 530 to 485 YBP)
		421661	TU1 at 134- 139 cm W wall	Standard	500 ± 30	-13.6	Cal 1405 to 1445 CE (Cal 545 to 505 YBP)
16SB153 SB	SBEA033 <sup>2</sup>	421664	TU1 at 134- 139 cm W wall	Solvent	590 ± 30	-13.6	Cal 1295 to 1370 CE (Cal 655 to 580 YBP) and Cal 1380 to 1415 CE (Cal 570 to 535 YBP)
		421663	TU1 at 134- 139 cm W wall	Standard	500 ± 30	-12.8	Cal 1405 to 1445 CE (Cal 545 to 505 YBP)
16SB174 SB	SBDA012 <sup>1</sup>	421682	TU2 L2 at 15- 20cm	Solvent	1230 ± 30	-26.7	Cal 685 to 885 CE (Cal 1265 to 1065 YBP)
		421681	TU2 L2 at 15- 20cm	Standard	1170 ± 30	-23.9	Cal 770 to 905 CE (Cal 1180 to 1045 YBP) and Cal 920 to 965 CE (Cal 1030 to 985 YBP)
16SB182	SB182 SBF010		TU1 L3 at 20- 30cm	Solvent	125.3 +/- 0.3 pMC	-15.1	Historic/Modern
		421679	TU1 L3 at 20- 30cm	Standard	Cancelled		NA
16SB182	SBF016	421678	TU2 L2 at 19cm, E half	Solvent	1020 ± 30	-26.0	Cal 980 to 1035 CE (Cal 970 to 915 YBP)
		421677	TU2 L2 at 19cm, E half	Standard	970 ± 30	-25.7	Cal 1015 to 1155 CE (Cal 935 to 795 YBP)
16SB185	SBH034	421676 & 421675	Mound B, CT4 at 195-199cm	Failed collagen extraction	Cancelled		NA
16SB185	SBHA034	421674	Mound B, CT4 at 210-216cm	Solvent	1120 ± 30	-24.1	Cal 780 to 785 CE (Cal 1170 to 1165 YBP) and Cal 880 to 990 CE (Cal 1070 to 960 YBP)
		421673	Mound B, CT4 at 210-216cm	Standard	1030 ± 30	-25.4	Cal 975 to 1030 CE (Cal 975 to 920 YBP)
16SMY17	SYA021 & SYAB021	421666	TU2 L4 at 30- 35 cm	Solvent	1290 ± 30	-17.0	Cal 660 to 770 CE (Cal 1290 to 1180 YBP)
		421665	TU2 L4 at 30- 35 cm	Standard	1380 ± 30	-17.7	Cal 620 to 670 CE (Cal 1330 to 1280 YBP)

Site	ANID	Beta ID	Provenience	Pretreat	Uncal. Conv. 14C Age <sup>†</sup> (B.P.)	⁵13C	2-sigma Calibrated
16SMY17	SYAA021 & SYAC021 <sup>2</sup>	421668	TU2 L4 at 30- 35 cm	Solvent	1340 ± 30	-18.4	Cal 650 to 690 CE (Cal 1300 to 1260 YBP) and Cal 750 to 760 CE (Cal 1200 to 1190 YBP)
		421667	TU2 L4 at 30- 35 cm	Standard	2130 ± 30	-18.7	Cal 345 to 320 BCE (Cal 2295 to 2270 YBP) and Cal 205 to 85 BCE (Cal 2155 to 2035 YBP) and Cal 75 to 55 BCE (Cal 2025 to 2005 YBP)

<sup>&</sup>lt;sup>1</sup>Samples collected from units reported by DES as oiled. <sup>2</sup>Samples intentionally contaminated with MC252 oil as part of a controlled, comparative study. <sup>†</sup>Uncalibrated <sup>14</sup>C ages with 1-sigma (σ) standard error.

As with the investigation of Cheniere St. Denis, the Acorn Mounds site (16SB185) was of particular interest in terms of the potential for examining the effects of oil in undisturbed archaeological contexts. As previously described, site coring was used to sample deeply buried deposits at Acorn Mounds. Radiocarbon assays were run on two samples, both from Core Test 4 in Mound B. The first sample consisted of bone fragments of an unidentified taxon (SBH034) from 195 to 199 cm below surface in what may represent initial stages of mound construction. The sample was subdivided and processed by solvent extraction and standard pretreatment, but collagen extraction failed and radiocarbon analysis was cancelled. The second sample consisted of wood charcoal fragments (SBHA034) from a matrix of silty clay with *Rangia* shell at a depth of 210 to 216 cm, in what may be a pre-mound surface. The subsample pretreated by solvent extraction produced an uncalibrated conventional radiocarbon age of 1120  $\pm$  30 and 2-sigma calibrated calendar age of 780 to 785 and 880 to 990 CE (Beta-421674). The subsample that received standard pretreatment produced an uncalibrated conventional radiocarbon age of 1030  $\pm$  30 and 2-sigma calibrated calendar age of 975 to 1030 CE (Beta-421673).

The radiocarbon ages obtained on wood charcoal from 210 to 216 cm below the surface of the Mound B summit may indicate a *terminus post quem* of 780 CE for mound construction, the earlier of the 2-sigma calibrated calendar ages. A radiocarbon age, unfortunately, was not obtained on the sample of bone from the overlying stratum. Whether construction of Mound B began soon after this, or if the wood charcoal is associated with soils deposited during an initial construction episode, it is evident that Mound B most likely dates from the Coles Creek period (700–1200 CE). Oil is not expected to have permeated into soils and cultural deposits at this depth, approximately 200 meters west of the shoreline where oil was observed. As previously discussed, however, oil was detected in a sample at a depth of 0 to 30 cm below surface (SBH027) from the nearby Core Test 1 on Mound B. The potential for hydrocarbon contamination lends further credence to the older radiocarbon age obtained on the sample pretreated with solvent extraction.

Overall, there were no statistically significant differences in the AMS results obtained for subsamples processed by solvent extraction and standard pretreatment techniques, whether or not there was evidence of oil in the associated or surrounding matrix. The 2-sigma calibrated calendar ages generally overlapped, although the dates for subsamples that received standard pretreatment from Cheniere St. Denis (Beta-421683), Comfort Island (Beta-421681) and Scow Island Scatter (Beta-421677) were all somewhat later or more recent in age than the subsamples pretreated with solvent extraction. This was also true for the sample from Core Test 4 in Mound B at Acorn Mounds (Beta-421673) not chemically tested for oil. Standard pretreatment appears to result in somewhat later or more recent calendar ages than solvent extraction, whether oil is present in associated contexts. In each instance, however, the presence of oil was determined only for associated contexts, and not the individual samples that were pretreated and radiocarbon dated by AMS. The presence (or absence) of oil was not confirmed for each radiocarbon sample.

Experiments conducted on samples from the control sites indicate that crude oil can adversely affect the results of AMS, as described in Section 5.4. Pretreatment techniques may or may not resolve these effects, depending on the amount or severity of contamination. Oil was intentionally introduced into samples of non-human bone fragments from the two control sites (16SB153 and 16SMY17) in order to more closely examine the potential effects of crude oil contamination on pretreatment and radiocarbon dating results. The oil came from a soil sample (16SB178-26) on the shoreline of the Southern Comfort site that was determined to be a possible match for MC252 (SBC026).

A sample of unidentified mammal bone fragments (16SB153-33) from Site 16SB153 (134 to 139 cm below surface in TU 1) was divided into two subsamples (SBE033 and SBEA033). One of these subsamples (SBEA033) was contaminated by smudging oil onto the surface of the bone, but the other subsample was left uncontaminated. These subsamples were then each subdivided, producing four subsamples. Two subsamples (one contaminated and one uncontaminated) were processed by solvent extraction (Beta-421664 and Beta-421662) and the other two subsamples (one contaminated and one uncontaminated) received standard pretreatment (Beta-421663 and Beta-421661).

The radiocarbon ages of the four subsamples from Site 16SB153 are uniform, suggesting that regardless of pretreatment technique, the manual introduction of oil onto the surfaces of bone does not adversely affect AMS results. The uncontaminated subsamples (SBE033) produced uncalibrated conventional radiocarbon ages of  $450 \pm 30$  YBP (solvent extraction) and  $500 \pm 30$  YBP (standard pretreatment), with respective 2-sigma calibrated calendar ages of 1420 to 1465 CE (Beta-421662) and 1405 to 1445 CE (Beta-421661). The crude oil contaminated subsamples (SBEA033) produced uncalibrated conventional radiocarbon ages of  $590 \pm 30$  YBP (Beta-421664, solvent extraction) and  $500 \pm 30$  (Beta-421663, standard pretreatment), with respective 2-sigma calibrated calendar ages within the ranges obtained for the uncontaminated subsamples processed by solvent extraction and standard pretreatment. Oil that was smudged onto the surface of subsamples appears to have been effectively removed by both pretreatment techniques. Three of four radiocarbon assays indicate a date of 1405 to 1465 CE for this deeply buried and intact archaeological context at Site 16SB153. This concurs with the diagnostic ceramic types that indicate a Mississippi period component.

A second experiment involved the preparation of a mixture of oil from the Southern Comfort site (SBC026) and saltwater from Bayou Sale Bay. A sample of unidentified turtle bone (16SMY17-21) from TU 2, Level 4 (30-35 cm below surface) at the Bayou Sale site (16SMY17) was subdivided into four subsamples. Two subsamples (SYAA021 and SYAC021) were placed in glass vials containing oil and saltwater. These subsamples were maintained at room temperature for one week. The other two subsamples (SYA021 and SYAB021) were uncontaminated and stored in clean containers. One contaminated and one uncontaminated subsample were pretreated by solvent extraction (SYAA021 and SYA021). The other two subsamples (SYAC021 and SYAB021) received standard pretreatment. The uncontaminated subsamples (SYA021 and SYAB021) that were processed by solvent extraction and standard pretreatment produced uncalibrated conventional radiocarbon ages of 1290 ± 30 YBP (Beta-421666) and  $1380 \pm 30$  YBP (Beta-421665), with 2-sigma calibrated calendar ages of 660 to 770 CE and 620 to 670 CE. The contaminated subsample that was pretreated by solvent extraction (SYAA02) produced an uncalibrated conventional radiocarbon age of 1340 ± 30 YBP and 2-sigma calibrated calendar age of 650 to 690 and 750 to 760 CE (Beta-421668). This generally corresponds with the AMS results for both uncontaminated subsamples and concurs with diagnostic ceramics that indicate an early Coles Creek component.

In contrast, the contaminated subsample from Bayou Sale that received standard pretreatment produced an uncalibrated conventional radiocarbon age of  $2130 \pm 30$  YBP and 2-sigma calibrated calendar ages of 345 to 320 BCE, 205 to 85 BCE, and 75 to 55 BCE (Beta-421667). The uncalibrated conventional radiocarbon age of the contaminated subsample that received standard pretreatment was 750 to 840 years earlier than contaminated subsamples processed by solvent extraction and

uncontaminated subsamples processed by solvent extraction and standard pretreatment. The disparate results are attributable to standard pretreatment of the contaminated subsample, because the portion of the same subsample pretreated by solvent extraction produced results similar to the uncontaminated subsamples. Standard pretreatment failed to mitigate the effects of hydrocarbon contamination and produced a significantly older radiocarbon age for the sample of bone immersed in a mixture of oil and seawater. Pretreatment by solvent extraction notably mitigated these effects (SYAA02).

The experimental data from Bayou Sale indicate that standard pretreatment may not remove oil from samples with high levels of contamination, resulting in substantially skewed <sup>14</sup>C dates. Crude oil can interact with site formation processes and affect the *in situ* preservation of information. Pretreatment by solvent extraction can correct for the effects of oil contamination. By comparison, the AMS results from Site 16SB153 indicate that either pretreatment technique may correct for the effects of moderate or light contamination. Differences in the amount or severity of contamination may be difficult to determine without chemical analysis, including whether samples came into direct contact with crude oil or if oil has penetrated surfaces in submerged conditions. The type of material to be radiocarbon dated represents another variable not controlled for in the experiments, which only examined bone fragments. In selecting radiocarbon samples from sites where oil is present or suspected to be present, the application of appropriate pretreatment will be critical.

# Site Assessment Summary

The MC252 oil spill of 2010 was an unprecedented environmental disaster with far reaching consequences along the north-central Gulf Coast (Freudenburg and Gramling 2010; Joye 2015; Nixon et al. 2016). The Mississippi River delta and marshes of south Louisiana were heavily impacted and a major focus of the cleanup response. The full effects of the oil spill on underwater and terrestrial cultural resources are only now being systematically described (Hamdan et al. 2018; Salerno et al. 2018). The present study has aimed to address many of the questions raised in the aftermath of the oil spill. The goals and objectives involved examining the effects on the archaeological record, including cultural materials and analytical techniques. From a management perspective, any lessons learned from the MC252 oil spill should inform future responses to an oil spill along the Gulf Coast (Freudenburg et al. 2009). The results of this research should also inform CRM decision making and planning, in support of the Louisiana OCD, Division of Archaeology, and SHPO efforts to oversee compliance with Federal and State legislation, including NEPA, NHPA, and implementing regulations.

The following section reviews the results of this study in relation to the stated goals and objectives. The achievement of major tasks identified in the Project Management Plan provide the requisite information for an assessment of the effects of the MC252 oil spill on coastal archaeological sites, including an evaluation of the hypotheses set forth in the Introduction. The findings of this study indicate the effects of an oil spill range from site formation processes and a potential loss of information to carrying out fieldwork and applications of archaeometric techniques. The Site Assessment Summary concludes by examining how an oil spill effects resource management, with cost estimates for archaeological research and recommendations for CRM planning and decision-making.

# 7.1 Review of Accomplished Research

The Project Management Plan identified seven major tasks integral to achieving the goals and objectives of this research. The major tasks involved coordinating site access, site sampling, oil source analysis, impact assessment, resource management, crew training, and artifact management. The results in accomplishing five of these major tasks are reviewed below, with an emphasis on impact assessment. Artifact and resource management are considered separately, after an evaluation of the hypotheses formulated at the beginning of this study.

#### 7.1.1 Site Access and Crew Training

The PI and Project Director coordinated site access in preparation for this study by contacting landowners to arrange for fieldwork. This involved requesting permission from private landowners to collect samples and seeking State permits to work on public lands. The preliminary list of sites for assessment was shortened from 14 to 12 sites as a result of the post-award meeting on August 5, 2014, as isdescribed in Section 5.1. Site selection was aimed at including sites with prehistoric components, where SCAT teams observed oil on the shoreline during the MC252 oil spill response, but that also contained intact archaeological deposits. The revised List of Sites for Assessment consisted of 12 previously recorded coastal sites with prehistoric components, including two control sites where SCAT teams had not recorded oil. The likelihood for intact archaeological deposits at a particular site was based on a review of previous investigations, as well as the presence of extensive cultural deposits such as earthen mounds and/or midden.

Receiving authorization from property owners, particularly out-of-state and corporate landowners, posed initial difficulties in arranging for site access. The lack of response from two landowners and denial of permission from two others required further revisions to the list of sites for potential assessment. The PI also sought appropriate authorizations from State agencies and commissions. The Louisiana

Archaeological Survey and Antiquities Commission issued a permit to conduct archaeological investigations on State lands on October 29, 2014. The Unmarked Human Burial Sites Board issued a permit on September 26, 2014 in compliance with the Louisiana Unmarked Human Burial Sites Preservation Act (R.S. 8:671–681). The Unmarked Human Burial Sites Permit provided for the inadvertent recovery of human remains, with the stipulation that any remains identified as human were to be left *in situ* or returned for reburial at the site of origin. The Department of Natural Resources, Office of Coastal Management for Coastal Zone Consistency, determined that a Coastal Zone Use permit would not be required for the proposed research, in accordance with the Federal Coastal Zone Management Act of 1972 (Section 307c, of as amended).

Revisions were made to the List of Sites for Potential Assessment through agreement among the PI, BOEM PO, and Louisiana State Archaeologist. Revisions were presented in the revised Draft Action Plan and Second Annual Progress Report (Rees and Huey 2016:3). The revised List of Sites for Potential Assessment included 12 coastal sites with prehistoric components previously recorded with the Louisiana Division of Archaeology. SCAT teams recorded different amounts of oil on the shorelines at ten of these sites during the MC252 oil-spill cleanup response (16JE2, 16JE3, 16LF293, 16PL8, 16SB174, 16SB178, 16SB180, 16SB182, 16SB185, 16SB186). Two of the sites were included as controls (16SB153 and 16SMY17), where SCAT teams had not observed oil during the MC252 response. As nearly all of the sites are located in fairly remote areas of the Mississippi River delta, access was provided by LDWF-piloted watercraft and a LUMCON vessel piloted by the Project Director.

All research personnel received training for fieldwork involving hazardous materials and potentially contaminated samples. Appropriate safety protocols for working in potentially hazardous conditions were developed and applied through standard operating procedures outlined in the Logistics, Fieldwork and Sampling Plan. The Project Director received HAZMAT training and instructed field crew and lab assistants in the proper handling of potentially contaminated samples in the field and lab. The presence of crude oil was based on visual and olfactory inspection of shorelines, ground surfaces, excavation units and artifact collections. Additional safety measures included the use of gloves, protective clothing, masks, and ventilators when handling materials contaminated with hydrocarbons. Instances when there was no detectible odor or appearance of oil did not indicate the absence of hydrocarbons, so special sampling procedures were meticulously followed, regardless of the perceived conditions of the sites and samples.

## 7.1.2 Site Sampling

The Project Director and crew conducted fieldwork at eight sites between September of 2014 and September of 2015, beginning with Bayou Sale (16SMY17), the control site in St. Mary Parish. The seven other sites were assessed in the following order: Cheniere St. Denis (16JE2), Southern Comfort (16SB178), Comfort Island (16SB174), control site 16SB153 (unnamed), Scow Island Scatter (16SB182), Redfish Slough (16LF293), and Acorn Mounds (16SB185). Four of these sites (16SB178, 16SB174, 16SB182, and 16SB185) are clustered on the eroded remnants of marsh islands in eastern St. Bernard Parish, west of Chandeleur Sound. The Cheniere St. Denis site is located on Bayou St. Denis in the Barataria basin, west of the modern Plaquemines—Balize delta. The Redfish Slough site is located to the southwest, on Philo Brice Island in Timbalier Bay. The control sites (16SMY17 and 16SB153) are located at the western and northeastern peripheries of the study area, on the shore of East Cote Blanche Bay and the south shore of Lake Borgne.

The ULL crew excavated test units totaling a minimum of one square meter at each of these sites, in addition to systematic surface collections, hand-operated probes, cores and auger tests. The crew recorded stratigraphic profiles and proveniences of samples collected from each site. The field methodology focused on sample collection for analysis and assessment of the effects of an oil spill. The development and use of special sampling protocols avoided the contamination of samples with oil during fieldwork and minimized the potential for the inadvertent introduction of hydrocarbons, dispersants or other chemicals.

This included the use of clean metal hand tools, packaging and containers made of non-plastic materials for collection, the periodic cleansing and rinsing of tools with Dawn detergent and clean water, and not using oils or other chemicals for cleaning.

Special samples included individual specimens of pottery, artifacts and ecofacts, soil samples, column samples, unit cores, and soil cores. A total of 171 special samples were collected from all eight sites, with an average of 21 special samples per site (Appendix A). Special samples were sealed in aluminum foil, clean metal containers and clean cloth bags, recorded by provenience, placed in an air tight, waterproof container and transported to the lab for processing and analysis (Section 5). Investigators took extraordinary measures in each instance to avoid the inadvertent introduction of oil from surface sediments and other sources outside of the cultural deposits and excavated contexts.

Though the sampling of undisturbed archaeological deposits was a research priority, the majority of the assessed sites consist of mostly redeposited, wave-washed shoreline accumulations of artifacts and ecofacts. This is common in the Mississippi River delta, where coastal erosion, subsidence, and anthropogenic alterations of the landscape affect or determine site formation processes (Cloy and Ostahowski 2015:7-17; Neuman 1977a). The field investigations procured samples from intact archaeological deposits at three sites (16JE2, 16SB153 and 16SB185). Along with Bayou Sale (16SMY17), these sites consist of cultural deposits, including shell midden and earthen mounds, being worn away and redeposited by coastal erosion, or buried and submerged by subsidence and relative sealevel rise. The Redfish Slough site (16LF293) may contain intact deposits that are deeply buried or submerged, but these were not encountered during fieldwork for the present study. As reported by Cloy and Ostahowski (2015), the three other assessed sites (16SB174, 16SB178, and 16SB182) may consist entirely of beach accumulations of redeposited cultural materials and shell midden from now submerged and eroded sites offshore. The majority of samples from all sites collected as part of this study consist of sediments, artifacts and ecofacts from redeposited, secondary contexts.

#### 7.1.3 Oil Source Analysis

Oil source analysis was essential to the goals and objectives of this investigation. It was accomplished through the chemical characterization and fingerprinting of samples by a GC/MS method developed specifically for detecting and quantifying compounds commonly associated with oil spills (Appendix C; Meyer et al. 2017). This is especially important, given the limitations and subjective nature of visual, olfactory, and tactile inspection in determining the presence or absence of crude oil. The LSU DES laboratory analyzed 28 samples from all eight sites to determine the presence, quantity, or absence of oil by concentrations of petroleum hydrocarbon analytes. The samples consisted of soil matrix with variable amounts of shell fragments from redeposited and intact archaeological contexts. A priority was placed on selecting samples from sites where oil was previously observed and where there was a greater likelihood of encountering intact archaeological deposits. Twelve of the 28 samples (43%) produced chemical evidence of oil. The DES lab detected oil in samples from all six sites where oil had been observed during the oil spill response. Oil was not detected in soil samples from the two control sites (16SB153 and 16SMY17), but only one sample was analyzed from each of these sites.

Petrogenic compounds provided biomarkers for oil source fingerprinting in determining the source and potential association with MC252 (Appendix C; Meyer et al. 2017). Oil biomarkers of sample profiles were compared to the MC252 source through qualitative comparison, diagnostic biomarker ratio analysis and chemometrics. Two samples from Site 16SB185 were a match for MC252 oil. Three samples from three other sites (16LF293, 16SB174 and 16SB178) were possible matches for MC252. The oil was too weathered in seven of the 12 samples (58%) to determine the source or was inconclusive for MC252. The majority of the samples that tested positive for oil (n=7; 58%) were excavated from secondary contexts. Only two of the samples (17%) in which oil was present were collected from the surface, including a sample from the shoreline east of Acorn Mounds (16SB185) that was a positive match for MC252.

Three of the samples in which oil was detected, but was too weathered for fingerprinting or inconclusive for MC252, came from intact archaeological deposits at two sites (16JE2 and 16SB185). The two samples that contained oil (JEB021 and JEB026) from the Cheniere St. Denis site (16JE2) consisted of soil matrix and residue from the surfaces of *Rangia* shells collected from levels 2 and 3 of TU 3 in the western slope of the northernmost mound. Oil was present in three samples from the Acorn Mounds site (16SB185), including a core sample (CT 1, CS 1; SBH027) from the northwest slope of Mound B at a depth of 0 to 30 cm below surface. The associated stratum appears to be an accretional marsh sediment from an otherwise intact archaeological context. Mound B lies approximately 140 meters west of the shoreline where two samples produced a positive match for MC252 oil. As the oil from the core sample on Mound B was too weathered for chemical fingerprinting, its source cannot be definitively attributed to the MC252 oil spill.

#### 7.1.4 Impact Assessment

The assessment of potential impacts to coastal archaeological sites from an oil spill included the effects on radiocarbon dating, elemental analyses, and the analysis of absorbed residues in pottery. Although previous studies have addressed the potential for contaminants in radiocarbon studies (D'Elia et al. 2007), the present study was concerned specifically with the potential effects of an oil spill on analytical techniques in the application of AMS. Radiocarbon dating of samples from the assessed sites complemented analyses of diagnostic artifacts in providing information on site chronology and regional culture history. The primary focus, however, was on determining the possible effects of hydrocarbon contamination on pretreatment techniques for radiocarbon dating. Sampling criteria developed for this study guided the selection of samples for radiometric analysis. The pertinent criteria included selection of samples from intact archaeological contexts in which chemical analysis had already detected hydrocarbons. The samples were subdivided and processed by standard pretreatment or solvent extraction in order to examine the potential effects of hydrocarbon contamination on the results of radiocarbon dating.

Radiocarbon analysis by AMS yielded results for a total of 17 subsamples from six of the eight assessed sites (Section 6.6). The initial results showed no significant differences in radiocarbon assays that might be associated with different pretreatment methods of subsamples, regardless of whether or not oil had been chemically detected in associated strata or archaeological deposits. Standard pretreatment prior to application of AMS produced slightly later, uncalibrated conventional radiocarbon ages on samples from oiled contexts at the Cheniere St. Denis (16JE2), Comfort Island (16SB174), Scow Island Scatter (16SB182), and Acorn Mounds (16SB185) sites. The 2-sigma calibrated results overlapped for subsamples that received solvent extraction and standard pretreatment. Four of the five radiocarbon dates from the Comfort Island and Scow Island Scatter sites are consistent with late Coles Creek components indicated by diagnostic ceramics, despite the disturbed contexts from which the samples were obtained. As is discussed in the section on Resource Management, this points to the potential research value of sites that may be mostly or entirely shoreline accumulations of redeposited cultural materials. Radiocarbon dates on samples from intact archaeological deposits at Cheniere St. Denis, Acorn Mounds, and Site 16SB153 are consonant with Coles Creek period (16JE2 and 16SB185) and Mississippi period (16SB153) site occupations, including a terminus post quem of 780 CE for the construction of Mound B at Acorn Mounds.

Hydrocarbon contamination was uncertain for the previously described samples, as oil was chemically detected only in the associated strata or surrounding matrices. Experimental data were produced that indicate crude oil can affect the results of AMS dating, but that pretreatment of samples by solvent extraction mitigates the adverse impacts. In the first experiment, oil determined to be a possible match for MC252 from the shoreline of the Southern Comfort site (16SB178-26) was smudged onto the surfaces of unidentified bone fragments collected from TU 1 (134 to 139 cm below surface) at Site 16SB153 (16SB153-33). One contaminated subsample was processed by solvent extraction (Beta- 421664) and the

other oil-smudged sample received standard pretreatment (Beta-421663). Likewise, one uncontaminated subsample was processed by solvent extraction (Beta-421662) and the other received standard pretreatment (Beta-421661).

Regardless of pretreatment technique, the results of AMS concur with the diagnostic ceramic evidence for deeply buried cultural deposits in TU 1 at Site 16SB153 dating from the Mississippi period. Three of the four respective 2-sigma calibrated calendar ages overlapped between 1405 and 1465 CE, with the crude oil contaminated subsample treated by solvent extraction (Beta-421664) producing slightly earlier 2-sigma calibrated calendar ages of 1295 to 1370 CE (Cal 655 to 580 YBP) and 1380 to 1415 CE (Cal 570 to 535 YBP). Although this may be a result of sampling error, it also corresponds with the previously described samples for which standard pretreatment prior to application of AMS produced slightly later, uncalibrated conventional radiocarbon ages. The results of the first experiment on subsamples from control site 16SB153 suggest both solvent extraction and standard pretreatment mitigate the effects of crude oil contamination, at least when it is present on the surfaces of bone.

The second experiment examined the potential effects of more severe or prolonged hydrocarbon contamination in AMS dating by exposing a subsample of unidentified turtle bone (16SMY17-21) from TU 2 (Level 4) at the Bayou Sale (16SMY17) control site to a mixture of oil from the Southern Comfort site and saltwater from Bayou Sale Bay. After immersion in a mixture of oil and seawater for one week, the contaminated subsample pretreated by solvent extraction (Beta-421668) produced a 2-sigma calibrated calendar age that coincides with the 2-sigma calibrated calendar ages derived for uncontaminated subsamples processed by solvent extraction and standard pretreatment (Beta-421666 and Beta-421665). Calendar ages ranging between 620 and 770 CE generally concur with diagnostic ceramic evidence for an early Coles Creek component at the site. However, the contaminated subsample that received standard pretreatment produced significantly earlier 2-sigma calibrated calendar ages of 345 to 320 BCE, 205 to 85 BCE, and 75 to 55 BCE (Beta-421667). This is 750 to 840 years earlier than the AMS dates for contaminated subsamples processed by solvent extraction and uncontaminated subsamples processed by solvent extraction and standard pretreatment.

The erroneous AMS date on the intentionally contaminated subsample from Bayou Sale is attributable to crude oil within the sample and the failure of standard pretreatment to correct for hydrocarbon contamination. The experimental evidence indicates that standard pretreatment does not remove oil from samples with more severe contamination or prolonged exposure to crude oil in seawater, resulting in substantially earlier radiocarbon results. Standard pretreatment measures, however, may negate the adverse effects of short-term or moderate exposure to crude oil. The absorption of hydrocarbons into porous materials, such as bone, may be amplified by immersion in seawater at coastal sites. As the level or amount of contamination may be difficult to discern for individual samples of different materials without independent chemical analysis, pretreatment by solvent extraction is advisable for all radiocarbon samples from coastal sites where oil is present or suspected to be present. Additional experimental data might be collected on the vectors and rates of hydrocarbon transmission into different kinds of archaeological materials under different conditions, with and without dispersants.

Assessment of the potential effects of oil on archaeometric techniques also involved trace element analysis. The MURR Archaeometry Laboratory examined the potential effects of oil in applications of neutron activation analysis (NAA) and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) for associated provenance studies. The MURR Archaeometry Lab analyzed five grog-tempered pottery sherds by NAA and LA-ICP-MS. One specimen was from a redeposited context at the Southern Comfort site (TU 2, Level 1) in which chemical analysis had detected oil. Three other samples were from oiled contexts at the Comfort Island site (TU 2, Level 2). The fifth sample was from control site 16SB153 on Lake Borgne (TU 1, Level 7). Trace element analysis indicated no increased concentrations of elements known to be present in crude oil, such as nickel or vanadium. Only one sample (SBC035), the sherd from the Southern Comfort site, resulted in elevated levels of arsenic, possibly due to oil (Appendix

D). The results consequently indicate hydrocarbon contamination does not adversely affect elemental analysis of pottery sherds by NAA or LA-ICP-MS. Standard pretreatment techniques such as washing and removal of potentially oiled surfaces with a burring tool appear to mitigate the presence of oil. The same should apply to trace element analysis of lithics. The presence of oil should not adversely affect related provenance studies of ceramics or lithics.

In contrast, the presence of oil and dispersant does adversely affect the analysis of absorbed pottery residues by GC/MS. The Archaeological Residue Laboratory at UNCW analyzed 17 samples of pottery sherds from seven sites. Reber was able to identify biomarkers for crude oil in the pottery residues, as well as Corexit 9500 and Corexit 9527, the dispersants used in the MC252 oil spill cleanup (Appendix E). The absorbed residue analysis detected oil and/or dispersant in nine of the 17 samples (53%). Oil and/or dispersant were detected in sherds from three of five sampled sites where SCAT teams recorded the presence of oil during the MC252 response (16JE2, 16SB178, and 16LF293). The biomarkers for oil and dispersant, and dispersant without oil, were also detected in one sample from each of the control sites. Both oil and dispersant were present in the absorbed residues in two sherds, some amount of oil without dispersant was detected in three sherds, and dispersant without oil was present in four sherds.

Six of the nine sherds (67%) with biomarkers for oil and/or dispersant were in association with strata or near soil matrices that independently tested positive for oil, indicating a correlation between oiled archaeological contexts and the contamination of pottery sherds. Four of these contexts were possible matches for MC252 oil. The two sherds with biomarkers for both oil and dispersant, as well as one sherd in which only oil was detected, came from contexts that tested negative for oil. This indicates the presence of oil and dispersant in pottery residues from contexts that otherwise appear to be uncontaminated by an oil spill.

The extensive use of dispersants during the MC252 oil spill response is reflected in the absorbed residues of pottery sherds, with dispersant identified with (n=2) or without (n=4) oil biomarkers in six samples. Samples from both control sites contained dispersant in absorbed residues, even though overlying or underlying soil matrices tested negative for oil. One sherd from TU 2 (Level 1) at Bayou Sale contained dispersant. Biomarkers for both oil and dispersant were detected in a shell-tempered sherd from an intact, deeply buried context at Site 16SB153 on Lake Borgne (TU 1, Level 6). Inadvertent contamination during excavation and collection is unlikely, because the crew followed special sampling protocols as precautionary measures.

Reber also examined the effects of oil and dispersant contamination on the analysis and interpretation of absorbed plant and animal residues in pottery sherds. By quantifying total lipid and total biomarkers of contaminants per gram of sherd, she was able to calculate percentage of contamination as light (less than 1%), light to moderate (at least 1%), moderate (at least 2%) and moderate to severe (at least 6%). Relative percentage of contamination is not clearly associated with the depth of archaeological deposits, although this may be a reflection of the small sample size. Oil and dispersant contamination are associated with a loss of information, with higher percentages of contamination causing increased difficulties in interpreting absorbed animal and plant residues. Moderate to severe contamination impedes the interpretation of absorbed pottery residues, with the greatest difficulties and information loss associated with combined oil and dispersant contamination (Appendix E).

The preceding impact assessment indicates an oil spill can adversely affect the results of radiocarbon dating and the analysis of absorbed residues in pottery. Oil and the dispersants used in a cleanup response can infiltrate archaeological deposits and interact as site formation processes, adversely affecting *in situ* site preservation and resulting in a potential loss of information. This study did not assess the potential effects of hydrocarbons or dispersants on other archaeometric techniques, such as X-ray fluorescence (XRF), thermoluminescence dating (TL), isotope analysis (Bentley 2006; Nehlich and Richards 2009; Privat et al. 2007) or DNA analysis for genetic studies of human populations and plant domestication

(Schurr 2004; Zeder et al. 2006). The present study was focused primarily on collecting data from affected coastal sites through field research. Experimental studies might be designed to address the potential effects of contaminants on these and other techniques in the analysis of archaeological materials, as well as appropriate pretreatment methods. Based on the results of this study, pretreatment measures are likely to mitigate the adverse effects on techniques involving elemental analyses, such as XRF, or when oil and dispersant are present in lower amounts. Pretreatments may be less successful when contaminants are present at higher levels, accelerate material degradation or otherwise impair the interpretation of results.

## 7.2 Evaluation of Hypotheses and Results

The Introduction presented nine hypotheses to generate a better understanding of the effects of an oil spill on coastal archaeological sites. These hypotheses were framed as general, predictive statements about the presence and potential effects of oil in archaeological contexts, for which data could be collected and analyzed. The evidence in support of these statements can now be considered, along with the need for reevaluation or restatement. Admittedly, any inferences that might be drawn are limited by the sample sizes and lack of comparable data from intact archaeological contexts at coastal sites. Nonetheless, the presently available data support or provide confirmation for two hypotheses (H3 and H5). As anticipated, the available evidence provides only partial support for four statements (H4, H6, H7 and H8). There is insufficient data to address the three other hypotheses (H1, H2, and H9).

H1: The presence of oil will be *most* evident in permeable artifacts such as ceramic sherds and ecofacts, such as bone, and *least* evident in comparatively impermeable artifacts such as lithics.

The results of absorbed pottery residue analysis indicate that oil, as well as dispersant, are present in ceramic sherds in archaeological contexts. Two-thirds (66.7%) of the pottery sherds with evidence for oil and/or dispersant in absorbed residues were from archaeological contexts in which the soil matrix tested positive for oil. Oil and the dispersants used in the oil spill cleanup are absorbed and detectible within pottery sherds when present in archaeological deposits. Experimental data in the pretreatment of samples for radiocarbon analysis likewise indicate that bone is also susceptible to the absorption of hydrocarbons, specifically when exposed to a mixture of oil and seawater. There is otherwise insufficient data from this study to test the relative permeability of artifacts and the presence of oil. Due to the scarcity of lithic material from oiled contexts, trace element analysis did not examine stone artifacts for the effects of oil. Furthermore, oil was not detected in the NAA or LA-ICP-MS analysis of ceramics and was not anticipated to pose difficulties in elemental analyses of lithics. Though experimental studies with different materials might fully address issues of permeability, absorption, and contamination, of pertinence for this study was the presence of oil in artifacts within archaeological contexts. The first part of H1 can therefore be restated as confirmed. The presence of oil is evident in permeable artifacts, such as ceramic sherds, and ecofacts, such as bone, at sites affected by an oil spill.

H2: Evidence of oil on artifacts, ecofacts and within cultural deposits will inversely vary according to the depth of stratified (undisturbed) deposits.

There are insufficient data to support or assess this statement, as the majority of the encountered archaeological contexts consisted of cultural materials redeposited on shorelines and so few sites produced evidence of well-stratified, intact cultural deposits. This study recorded stratified archaeological deposits at three sites (16JE2, 16SB153, and 16SB185), with a potential for intact, deeply buried and submerged deposits at two other sites (16SMY17 and 16LF293). The three other assessed sites (16SB174, 16SB178 and 16SB182) appear to consist of accumulations of redeposited cultural materials and shell midden from now submerged and eroded sites offshore (Cloy and Ostahowski 2015). There is, then, a lack of comparative data to assess whether the presence of oil decreases with the increased depth of

stratified deposits, or increases near the surface. There is evidence of oil both on the surface and in deeply stratified deposits.

Oil was detected on the shoreline east of Acorn Mounds (16SB185) and in relatively shallow deposits (ST 2, 20 cm; CT 1, 0–30 cm). The Cheniere St. Denis site (16JE2) produced evidence of oil in undisturbed cultural deposits in TU 3 (17–35 cm below surface), and samples from greater depths and closer to the surface in other units tested negative for oil (TU 2, 40–65 cm; TU 4, 2–8 cm). The absorbed residue analysis detected oil and dispersant in pottery samples from the same depth in TU 3 (17–35 cm below surface) at Cheniere St. Denis, but a sample of pottery from a greater depth (TU 3, 39 cm) produced no evidence of oil or dispersant. However, a pottery sample from a deeply stratified deposit at control site 16SB153 (TU 1, 125–135 cm) unexpectedly contained both oil and dispersant. Though the data provide limited confirmation of oil within stratified cultural deposits, there is insufficient comparative evidence from intact deposits to address possible correlations with depth.

H3: Evidence of oil at archaeological sites can be traced to the 2010 MC252 oil spill, but will also inversely vary according to the depth of stratified (undisturbed) deposits.

The available evidence generally supports the preceding statement. Two samples from a shovel test and the surface of the shoreline east of Acorn Mounds (16SB185) produced evidence of oil that matched the MC252 source. The boundaries of the Acorn Mounds site have yet to be determined by subsurface sampling, but surface collections along the shoreline have so far produced nearly all of the cultural materials associated with the site. Ten additional samples from five other sites tested positive for oil, although only three of these from redeposited and surface contexts at Redfish Slough (16LF293), Comfort Island (16SB174) and Southern Comfort (16SB178) were possible matches for MC252. The remaining samples were either inconclusive for MC252 or too weathered to determine the source. Oil at archaeological sites can be traced to the 2010 MC252 oil spill, but MC252 oil is less distinguishable in archaeological deposits at greater depths. The oil identified in samples from excavated contexts was generally too weathered to identify a source. It is unknown whether this is due to degradation in subsurface archaeological contexts or simply the passage of time.

H4: Evidence for the presence of oil will not significantly vary according to depth in secondary, redeposited contexts.

There is some support for the preceding statement, since most of the samples that produced evidence of oil were from secondary, redeposited contexts. Oil was present in samples from the surface and in reworked shoreline contexts at Redfish Slough (16LF293), Comfort Island (16SB174), Southern Comfort (16SB178), Scow Island Scatter (16SB182), and Acorn Mounds (16SB185). Samples that contained oil in association with redeposited cultural materials ranged in depth from the surface to 27 cm below surface. Though samples from shallower and deeper contexts tested negative for oil at these sites, the presence of oil in association with redeposited cultural materials did not appreciably vary in relation to depth.

H5: Evidence for the presence of oil at archaeological sites can be established through chemical analysis prior to radiocarbon dating and archaeometric techniques, such as neutron activation analysis (NAA) and absorbed residue analysis.

The results of this study confirm that chemical analysis of soil samples by GC/MS can determine the presence of oil before radiocarbon dating and the application of archaeometric techniques. This premise was one of the major criteria in the selection of samples for radiocarbon dating, trace element analysis, and absorbed pottery residue analysis. The DES lab at LSU detected chemical evidence for oil in 12 of 28 samples (43%). The lab detected oil in samples from all six sites where SCAT teams had observed oil during the oil spill response. The preceding section on impact assessment examined the potential consequences. Hydrocarbon contamination of archaeological contexts may necessitate pretreatment of

samples by solvent extraction before radiocarbon dating (see H6), but it has no discernible effect on trace element analysis of ceramics by NAA or LA-ICP-MS. Absorbed pottery residue analysis can be adversely affected by hydrocarbon contamination, especially in combination with dispersants used in an oil spill cleanup response. The GC/MS technique used for absorbed residue analysis indicates a correlation between oiled archaeological contexts and the contamination of pottery sherds. Six of nine sherds (67%) with evidence for oil and/or dispersant were in association with strata or collected near soil matrices that independently tested positive for oil. Three sherds with biomarkers for oil and/or dispersant were collected from archaeological contexts that tested negative for oil, indicating that the presence of oil cannot always be determined by chemical analysis before archaeometric analysis.

H6: Pretreatment for oil and other contaminants in archaeological samples will mitigate any adverse effects on radiocarbon dating and archaeometric techniques, such as NAA and absorbed residue analysis.

There is evidence in support of the preceding statement for radiocarbon dating. The results of AMS dating of samples from contexts that tested positive for oil indicate no adverse effects, regardless of pretreatment technique. However, experimental data indicate that standard pretreatment of samples with higher levels of contamination may result in erroneous and substantially older radiocarbon ages. Pretreatment of samples by solvent extraction before applications of AMS effectively mitigate the adverse effects of hydrocarbon contamination. This is also true for some archaeometric techniques, such as NAA and LA-ICP-MS. The presence of oil does not appear to affect the elemental analysis of pottery sherds, and ostensibly other material such as lithics. Standard pretreatment of artifacts by washing and removal of exterior surfaces with a burring tool appear to mitigate any adverse effects. The samples analyzed by NAA and LA-ICP-MS were associated with archaeological contexts that tested positive for oil, but the samples were not individually tested. Experimental studies might consequently address degree of hydrocarbon contamination and the most effective pretreatment techniques for different materials in trace element analysis.

In contrast, pretreatments for oil and other contaminants may not be available to mitigate the adverse effects on absorbed residue analysis. This may be due, in part, to similarities in the biomarkers for absorbed residues, dispersants and other contaminants. Potsherds selected for absorbed residue analysis are typically not washed. Pretreatment to cleanse potsherds of hydrocarbons and other contaminants may effectively dissolve or remove traces of lipids or other biomarkers absorbed into the ceramic during vessel use. Controlled experiments might also address possible pretreatment techniques for different kinds and amounts of contaminants in absorbed pottery residue analysis. Nevertheless, the present study suggests pretreatment will not mitigate the adverse effects of oil and dispersant in absorbed residue analysis, especially when samples are moderately to severely contaminated with both oil and dispersant. The hypothesis is consequently only partly supported by the evidence. Pretreatment for oil and other contaminants can mitigate the adverse effects on radiocarbon dating and some archaeometric techniques.

H7: The time requirements and costs of data collection, analyses, conservation, and curation will increase in proportion to evidence for the presence of oil at archaeological sites.

The evidence presented to this point supports the preceding statement. Crude oil is composed of volatile compounds that present known health hazards and remain toxic for extended periods of time (Chin 2011:2-3). As described in the Research Methodology, fieldwork at oiled sites requires greater time and monetary investments in terms of appropriate health safety protocols and equipment such as respirators and gloves for conducting fieldwork in hazardous environments. The same applies to laboratory activities involving the analysis, conservation, and curation of collections. Because such measures would otherwise be unnecessary, the time and cost requirements increase with the presence of crude oil at archaeological sites and within collections. The following section on Resource Management will consider additional measures potentially required for artifact and collections management (Chin 2011:2-3).

Any additional pretreatment techniques and laboratory procedures for dealing with contaminated collections will entail increased personnel effort and costs. The time requirements and costs in the field and lab are likely to increase proportionately with increased quantities of oil at archaeological sites and within collections, but also in relation to the goals and methods of a research design. An oil spill would more adversely affect large-scale excavations for site mitigation than remote sensing, soil core sampling or site surveys. Section 7.3 will further address the interrelated issues of research design and cost estimates for field research at oiled sites. The final two hypotheses address the potential presence of oil in archaeological deposits at the two control sites, where SCAT teams did not observe oil during the MC252 response.

H8: The presence of oil not associated with MC252 can be detected within archaeological deposits at sites where SCAT teams did not observe oil during the MC252 oil spill response.

As previously stated under H3, 10 of 12 samples (83%) from six sites that tested positive for oil were not definitely matched to MC252. The oil in seven samples (58%) was either inconclusive for MC252 or too weathered to determine the source. Two samples from the two control sites (16SMY17 and 16SB153), where SCAT teams did not observe oil during the MC252 oil spill response, tested negative for oil. Though the chemical analysis of soil samples focused on oiled sites, the absorbed residue analysis detected oil and dispersant in a potsherd from TU 1 (Level 6) at control site 16SB153. The absorbed residues in a pottery sherd from TU 2 (Level 1) at control site 16SMY17 also produced evidence of dispersant. Although there is some support for the hypothesis, the absorbed residue analysis did not undertake chemical fingerprinting for the determination of source. The oil detected at Site 16SB153 and in 10 samples from six other sites may have originated from MC252 or other sources, including smaller oil spills. Dispersant in the absorbed residue of a pottery sherd from control site 16SMY17 offers circumstantial evidence of the extensive use of dispersants during the MC252 cleanup response but cannot be definitively associated with that oil spill.

H9: Evidence of oil not associated with MC252 will be more prevalent in secondary, redeposited contexts at coastal archaeological sites where SCAT teams did not observe oil during the MC252 oil spill response.

There is insufficient evidence to evaluate the final hypothesis due to the small sample size from the control sites. It is worth restating, however, that oil was detected in the absorbed residue of a potsherd from an intact archaeological context in TU 1 (Level 6) at control site 16SB153. A soil sample from the same test unit (TU 1, Level 1) tested negative for oil. The only other chemically-tested soil sample from a control site (16SMY17) came from a redeposited context, but it was negative for oil.

To summarize the evidence in support or partial support of the preceding hypotheses: oil occurs on the surface and in intact archaeological deposits at sites where SCAT teams observed oiled shorelines during the MC252 oil spill response. Oil is also present at control sites where SCAT teams did not record the presence of oil. Some of the oil can be traced to the 2010 MC252 oil spill, although it is mostly too weathered to identify the source. The oil at these archaeological sites occurs mostly in redeposited midden, in shoreline accumulations of wave washed cultural materials. Oil is also present in intact archaeological contexts and in permeable artifacts, such as pottery sherds. There is a correlation between the presence of oil in archaeological contexts at sites oiled by the MC252 spill and contamination of the archaeological record with hydrocarbons. Appropriate pretreatment measures can mitigate the adverse effects of oil and other contaminants in radiocarbon dating and the application of some archaeometric techniques, such as NAA and LA-ICP-MS. Chemical analysis by GC/MS can determine the presence of oil before radiocarbon dating and other analyses. Moderate to severe hydrocarbon contamination impedes absorbed pottery residue analysis, with the greatest loss of information associated with combined oil and dispersant contamination. Pretreatment measures may not be available to mitigate the adverse effects of oil and dispersant in absorbed residue analysis.

## 7.3 Resource Management

The effects of an oil spill on coastal archaeological sites are now evident based on the results of this research. Crude oil and the dispersants used in an oil spill response can be chemically detected on the surface and in subsurface cultural deposits at sites dating from as early as the Tchula period in the Mississippi River delta. Oil and dispersants enter the archaeological record and interact with artifacts and ecofacts as site formation processes, predominantly in redeposited accumulations of wave washed cultural materials and midden, but also enter intact archaeological deposits. In some instances, the effects of contaminants in the archaeological record appear to be negligible for site preservation and archaeometry, as suitable pretreatment measures can effectively mitigate the adverse effects. The introduction of crude oil and dispersants in other instances can result in a loss of information contained in the archaeological record. Coastal erosion, subsidence, and environmental processes influence the conditions under which an oil spill may affect archaeological sites in the delta.

What should be considered now is how these findings might inform appropriate management strategies for archaeological sites affected by an oil spill. As previously described, the second of two major goals of this study is to provide the SHPO and OCD Division of Archaeology with information relevant to CRM planning and regulatory compliance. Archaeologists have known about and tried to manage the potential effects of oil and gas development on sites in Louisiana's coastal wetlands for decades (Neuman 1977a:31; Smith et al. 1983:97–100, 118). In retrospect, it is possible to conclude that more might have been done to preserve sites or mitigate the loss of information contained in the archaeological record (Jones 2014). This study has shown how an oil spill can adversely affect archaeological sites. The following assessment begins with an overview of resource management involving archaeological sites, followed by artifact and collections management, cost estimates and recommendations.

## 7.3.1 Cultural Resource Management Planning

Effective CRM planning in relation to the potential effects of an oil spill ultimately depends on up-to-date knowledge of site conditions, including archaeological integrity. A majority of the recorded prehistoric sites along the coast in the Mississippi River delta are made up of redeposited cultural materials with little or no archaeological integrity, or in conditions where integrity is difficult to assess. HDR, Inc. estimated that 80 percent of the sites recorded during the MC252 cleanup response consisted of redeposited artifact scatters (Cloy and Ostahowski 2015: 7-18). Ostahowski (2015) projects high rates of erosion and subsidence for coastal sites in the delta, with an average loss of one recorded site per year in Plaquemines Parish (Cloy and Ostahowski 2015: 7-18). As seen at the sites assessed for this study, redeposited cultural materials tend to occur in shoreline accumulations of shell hash. Some of these sites, such as Comfort Island (16SB174), Southern Comfort (16SB178), and Scow Island Scatter (16SB182) appear to consist entirely of redeposited cultural materials and reworked shell midden, from presumably destroyed sites offshore. Other sites, such as Chenier St. Denis (16JE2) and Site 16SB153, contain submerged and deeply buried, intact deposits, as well as redeposited cultural materials. These and the other sites assessed by this study are consequently in different stages of deterioration and formation, undergoing processes of erosion, submergence and subsidence.

Avoiding or mitigating the adverse effects of an oil spill on cultural resources in such dynamic, variable environmental conditions requires effective CRM planning. Resource management benefits from informed decision making regarding the most effective uses of finite funds and labor, especially when overseeing endangered resources under time constraints. The management of cultural resources in the U.S. is structured by Federal legislation and regulations pertaining to historic properties and protection of the environment, including Section 106 of the NHPA (54 U.S.C. 306108) and NEPA (King 2013; Lipe 2009). The SHPO and OCD, Division of Archaeology, are responsible for administering the NHPA

within the State of Louisiana (R.S. 41, Ch 13, § 1601, et seq.), while BOEM is charged with regulatory compliance involving historic properties in managing offshore energy development.

Under Section 106 of the NHPA and implementing regulations, Federal agencies are required to consider the effects of an undertaking on historic properties. Because an oil spill is not an "undertaking," the potential impacts of oil on archaeological sites do not fall under the purview of the Section 106 process. The Natural Resources Damage Assessment (NRDA) process addresses the effects of oil, but does not include cultural resources. However, the potential affects of the cleanup response and related activities on archaeological sites are undertakings that fall under the Section 106 process. This includes the use of dispersants during cleanup, the mechanical removal of oil from sites, potential impacts from associated cleanup staging areas, and, as argued after the *Exxon Valdez* spill, site looting and vandalism brought about by increased pedestrian traffic during shoreline cleanup. This loophole in the current Section 106 process effectively means that the oil from spills is not regarded as having potentially adverse affects on cultural resources, even though the cleanup response would be. This is a significant challenge to CRM planning for the SHPO and Federal agencies concerning the effects of an oil spill on archaeological sites (Chip McGimsey, personal correspondence August 10, 2018).

Beyond the Section 106 process, long-term CRM planning should consider historical significance for sites potentially affected by an oil spill, whether directly or indirectly. Determinations of historical significance are made in accordance with implementing regulations of the NHPA concerning the protection of historic properties (36CFR800) and criteria for the listing of properties on the National Register of Historic Places (ACHP 2004; 36CFR60). Historic properties encompass a wide range of cultural resources, including "districts, sites, buildings, structures, and objects that possess integrity of location, design, setting, materials, workmanship, feeling, and association." The National Register criteria for evaluation are applied to evaluate properties based on "quality of significance in American history, architecture, archeology, engineering, and culture" (36CFR60; NPS 1997). Through the regulatory implementation of the NHPA, the standard for historical significance in the U.S. has come to be equated with eligibility for listing on the National Register. Of the following four criteria, the potential eligibility of archaeological sites, especially sites with prehistoric components, is most often determined based on Criterion D (Little et al. 2000:28–29), referring to properties:

- (a) that are associated with events that have made a significant contribution to the broad patterns of our history; or
- (b) that are associated with the lives of persons significant in our past; or
- (c) that embody the distinctive characteristics of a type, period, or method of construction, or that represent the work of a master, or that possess high artistic values, or that represent a significant and distinguishable entity whose components may lack individual distinction; or
- (d) that have yielded, or may be likely to yield, information important in prehistory or history (36CFR60.4).

To be regarded as eligible for listing on the NRHP and therefore historically significant under the NHPA, an archaeological site must meet one or more of the above criteria, which are not mutually exclusive (Little et al. 2000:28–29). Under Criterion D, an archaeological site must produce, or have a clear potential to produce "information important in prehistory or history." The material manifestations and archaeological integrity of a site largely determine its information potential in relation to research design, methods and techniques. Though the manifestation or cultural obtrusiveness of an archaeological site can range from relatively ephemeral to an increasingly obtrusive, multicomponent site, this is not the same as archaeological visibility in the Mississippi River delta. The material culture of an ephemeral artifact

scatter on an eroded shoreline may have greater visibility than a subsided and deeply buried multicomponent site with monumental architecture. Furthermore, redeposited cultural materials and midden in the delta may be physically obtrusive and visible on a shoreline, but the material manifestations may be lacking *in situ* deposits, stratigraphic associations and primary contexts.

The likelihood that a site will yield information is therefore related to its integrity, which can be generally defined as "the ability of a property to convey its significance" (NPS 1997). For properties with standing structures or architecture, this involves "integrity of location, design, setting, materials, workmanship, feeling, and association" (36CFR60.4). Integrity under Criterion D can be more difficult to establish, but is generally regarded as reliant on "the data requirements of the applicable research design" (NPS 1997:23). For archaeological sites in particular, "it is important that the significant data contained in the property remain sufficiently intact to yield the expected important information, if the appropriate study techniques are employed" (NPS 1997:23). If an archaeological site is not "sufficiently intact" to provide information of pertinence to a research design, under the applicable methods and techniques, then it is unlikely to yield "information important in prehistory or history (36CFR60.4). So, though integrity is not a criterion for evaluating the eligibility of properties for listing on the NRHP, a lack of archaeological integrity would likely preclude it from consideration for National Register eligibility under Criterion D. An archaeological site deemed eligible for listing on the NRHP based on its likelihood of producing important information "must have the necessary kinds and configuration of data sets and integrity to address important research questions" (Little et al. 2000:29).

Although the NHPA Section 106 process as currently implemented does not regard the oil from a spill as an adverse affect on historic properties, effective CRM planning for an oil spill will depend in part on determinations of eligibility for listing on the NRHP, as well as archaeological integrity. If an oil spill uniformly impacts a shoreline where two or more sites are located, the effects are likely to be managed differently depending on archaeological integrity and the potential of those sites to yield information. The effects of an oil spill on a site eligible for listing, or listed on the NRHP may necessitate mitigation by recovering information that would otherwise be lost by contamination or ground disturbance from shoreline cleanup. In contrast, the effects of the same oil spill on a site determined to be ineligible may not result in mitigation. If ineligible under Criterion D, such sites are not regarded as being likely to yield important information. HDR, Inc., consequently, recommended that redeposited coastal sites lacking archaeological integrity be ineligible for listing on the NRHP (Cloy and Ostahowski 2015: 7-18–7-21).

This does not mean that redeposited sites in the Mississippi River delta retain no potential for yielding information, but only that the information potential is reduced, altered, or unrecognized. Other forms of site disturbance, such as plowing or cultivation of agricultural fields are recognized as detrimental to archaeological integrity but do not necessarily exclude a site from eligibility under Criterion D (Ford et al. 1972; Hardesty and Little 2009:60–61; Neumann et al. 2010:37–38, 138–139). Sites with different forms and amounts of disturbance may retain cultural information related to provenience, function, composition, and past lifeways, despite the displacement of artifacts and lack of intact cultural features or stratified deposits (Dunnell and Simek 1995; O'Brien and Lewarch 1981; Riordan 1988; Roper 1976). Archaeological integrity is ultimately related to site formation processes and context (Schiffer 1987).

Site disturbance and archaeological integrity play major roles in evaluating eligibility for listing on the NRHP, but both are relative to the kinds and sources of information potentially contained within a site. The information sought from an archaeological site depends, to a large degree, on the research design, including the available methods, analytical techniques, and technologies. There has been little consideration of the information potential of sites that are mostly or entirely shoreline accumulations of redeposited cultural materials, even though the processes of coastal site destruction are ubiquitous and well known (Gagliano 1984; McIntire 1958; Neuman 1977a:31). Two examples from the present study point out the need for additional scrutiny. In the analysis of radiocarbon samples from the Comfort Island (16SB174) and Scow Island Scatter (16SB182) sites, four out of five radiocarbon dates were consistent

with late Coles Creek components as indicated by diagnostic ceramics, even though the samples were from redeposited contexts. Lack of stratified deposits or intact cultural features should not be equated with a lack of culture historical information. Furthermore, absorbed pottery residue analysis detected plant and animal lipids in sherds uncontaminated by hydrocarbons or dispersants from both sites. Surface collections of scattered artifacts may yield previously unanticipated sources of information at redeposited coastal sites. There may be additional reasons to not routinely or entirely exclude such archaeological resources from CRM planning in the event of an oil spill.

Though the effects of an oil spill may be immediately apparent in the widespread shoreline deposits of artifact scatters in the Mississippi River delta, the effects may be less discernible on the deeply buried cultural deposits of subsided sites. The Acorn Mounds site (16SB185), which contains deeply buried but presumably intact deposits on a subsided landform, is likely to yield important information on the Coles Creek period in the delta. Accordingly, HDR, Inc. recommended it to be eligible for listing on the NRHP (Cloy and Ostahowski 2015: 7-19). Based on the depth of the cultural deposits and present site conditions, however, it will be comparatively more difficult and costly for the Acorn Mounds site to yield that information. Deeply buried and subsided sites are not immune to the effects of an oil spill, as hydrocarbon may enter the archaeological record through degradation and percolation (Duffy et al. 1977; Price 1980) or inadvertently during excavation. The presence of oil and dispersant in the absorbed residue of a potsherd from deeply buried deposits (TU 1, Level 6) at Site 16SB153 is a case in point. The infiltration and penetration of hydrocarbons may be accelerated at wet sites in marine environments, especially when combined with dispersants in permeable soils (Amro et al. 2011; Zuijdgeest and Huettel 2012).

The oil from a spill and the dispersants used during the cleanup response may contaminate redeposited and intact archaeological contexts through the tide or wave action along shorelines. In time, oil and dispersants may enter archaeological deposits through the water table or be transported inland by storm surge (Amro et al. 2011). This appears to have been the case at the Chenier St. Denis (16JE2) and Acorn Mounds (16SB185) sites. Shoreline remediation and oil removal methods involving ground disturbance may in some instances prove even more destructive to archaeological resources than initial accumulations of oil. The site monitoring and survey methods developed after the *Exxon Valdez* oil spill (Reger et al. 2000) and adopted by SCAT teams in the Mississippi River delta during the MC252 oil spill response (Cloy and Ostahowski 2015) will continue to serve the interests and CRM responsibilities of the Louisiana SHPO and OCD, Division of Archaeology, in responding to oil spills. Native American consultations and partnerships, including tribal monitoring of cleanup activities, will continue to be essential to site stewardship and conservation.

In managing the potential effects of an oil spill on coastal archaeological sites, conservation planning should be pragmatic and strive to recognize a multiplicity of complementary and sometimes competing heritage values. Though archaeologists and cultural resource managers may focus on the value of scientific research and the production of historically important information, sites are also places with overlapping artistic, economic, environmental, religious, social, and symbolic values (Lipe 2009; Mason and Avrami 2002). Scattered artifacts in a redeposited shoreline midden may be perceived as having little potential for yielding important historical information, but still have important historical associations (Criterion A). The potential effect of an oil spill on an archaeological site that is also a traditional cultural property (TCP) may be more difficult for a resource manager to determine, but is nonetheless included in management planning (Ferguson 2003; King 2003; Moreono and Lee 2015; Parker and King 1998). Consultations and partnerships will be essential to this process.

In CRM planning it will be advantageous and more efficient to prepare in advance and organize potentially effected coastal sites according to a wide range of heritage values and research design strategies. Demas (2002:28) outlines a useful three-tiered planning process involving key constituencies and culturally affiliated groups in site management. The process begins with collecting information on the

expectations of stakeholders, as well as sites, and proceeds to an assessment of current site conditions (including threats), heritage values, and management constraints. The decision-making response involves establishing policies, objectives, and strategies in advance, as part of an established site management plan (Demas 2002:30). Establishing research strategies and setting priorities in advance are essential, since responding to the effects of an oil spill is unlikely to be the only critical issue in managing coastal archaeological sites (Anderson et al. 2017). The final section of the Site Assessment Summary presents recommendations about research strategies and priorities in response to an oil spill.

#### 7.3.2 Artifact and Collections Management

The purpose of this section is to briefly consider artifact and collections management in response to an oil spill. Artifact management is one of the major tasks for accomplishing the goals and objectives in the Project Management Plan. The following is based on a review of the available literature on the treatment and management of artifact collections affected by an oil spill. Because the Louisiana OCD will curate materials from the present study, the PI also conferred with the Louisiana OCD Division of Archaeology Collections Manager about issues and concerns involving the curation of collections contaminated with crude oil. Among the most pertinent issues are whether oiled artifacts and ecofacts will discharge gaseous and potentially harmful emissions and, if so, how this might affect the handling, packaging, and long-term curation of collections. This presentation is not a comprehensive study of materials conservation or techniques for the treatment and curation of artifacts, because there is extensive literature on the subject (Agnew and Bridgland 2006; Brown et al. 1977; Cronyn 1990; Hamilton 1998; Pearson 1987; Rogers 2004; Sease 1994; Smith 2003).

As mentioned in the review of the MC252 response, archaeologists and conservators with the NCPTT provided initial guidance to resource managers and agency officials on materials conservation and the protection of historic structures from crude oil (Chin 2010, 2013). The NCPTT team studied the effects of crude oil on the brick masonry of historic Fort Livingston (16JE49) on Grand Terre Island and tested methods for remediation (Chin and Church 2010). The EPA has approved different products in the National Oil and Hazardous Substances Pollution Contingency Plan for oil spill remediation. These include various bioremediation agents, dispersants, surface washing and collecting agents, and oil spill control agents (EPA 2018; Vora 2011:77). Surface washing agents include surfactant-based cleaners and solvent-based cleaners. Among the NCPTT findings was that some cleaning agents and techniques might cause further damage to materials in the effort to remove crude oil. The NCPTT recommended preventative measures, such as the use of booms, for the avoidance of additional oiling (Chin and Church 2010:6–7).

Building on the NCPTT field research at Fort Livingston, Vora (2011) examined different remedial treatments for the oiling of masonry materials. Although the brick-and-tabby architecture of Fort Livingston differs from the cultural materials at prehistoric sites, there are comparable issues and challenges. These include difficulties in accessing remote site locations in the Mississippi River delta and a lack of fresh water and electricity (Vora 2011). Vora conducted controlled experiments with surface washing agents to find the most effective treatment for removing weathered and unweathered crude oil from historic brick. She tested the following surface washing agents for rate of oil dissolution (Vora 2011:39, 41–42):

- BioSolve (Biosolve Co)
- Clean Green Planet Wash (US Ag, LLC)
- Cytosol Biosolvent (Cytoculture International, Inc.)
- De-Solv-It Clean-Away All Purpose Cleaner (APC) Super Concentrate (Orange-Sol)
- De-Solv-It Industrial Formula (Orange-Sol)
- E-Safe (Plutus Environmental Technologies, Inc.)

- GoldCrew (Environmental Chemical Solutions, Inc.)
- Petro-Clean (Alabaster Co)
- SC-1000 (Gemtek Products)

Vora (2011:59, 64, 78–79) identified Cytosol Biosolvent as the most effective cleansing agent for weathered and unweathered crude oil on brick. Although the results of the controlled experiments are straightforward, field applications must deal with unpredictable environmental conditions.

The NCPTT field and lab studies of surface washing agents for cleaning historic masonry are relevant for treatments of certain classes of artifacts, such as pottery and stone, but may be inappropriate treatments for fragile and porous materials, such as oiled bone (Church 2011; Langdon 2011). As shown in the present study, crude oil and dispersant can be absorbed into pottery sherds and other porous materials. Whether transported by waves, tidal action, or infiltration through the water table, crude oil can enter the archaeological record as tar balls, mousse, or sheen and interact with cultural materials (Chin 2011:1). The historic brick at Fort Livingston is similar to terracotta and some coarse earthenwares in terms of relatively lower firing temperatures, softness, and porosity. Some of the effects of crude oil on brick are likely to be similar for pottery.

The NCPTT produced a series of studies on the treatment and conservation of artifacts contaminated with crude oil (Chin 2011, 2013; Church 2011). These were among the most intensive conservation studies after the MC252 oil spill. The principal issues are the long-term effects of oil in the conservation of artifacts, as well as health and safety issues for conservators. Crude oil is comprised of carbon, hydrogen, sulfur, nitrogen, oxygen, and trace metals, constituting volatile and toxic compounds that include alkanes, polycyclic aromatic hydrocarbons, and hazardous air pollutants. These compounds present known health hazards and remain toxic for extended periods of time. Handling contaminated artifact collections requires appropriate safety equipment, such as respirators and gloves (Chin 2011:2–3).

Besides obvious discoloration and possibly covering decorated surfaces, crude oil can promote the formation of mold and salts by trapping moisture within the ceramic. Along with acidic and corrosive compounds, these can accelerate artifact deterioration and decay. The NCPTT recommends that organic materials and low-fired ceramics such as prehistoric pottery be kept moist after collection. Non-abrasive cleaners, such as Teflon or wooden scrapers and solvents, should be used on a trial basis (Chin 2011:1–4, 7–8). A solution of Dawn detergent and water might be used to clean heavily oiled artifacts that are less porous, such as some historic ceramics and lithic artifacts, but should involve thorough rinsing before artifacts are allowed to dry.

The removal of crude oil from some classes of artifacts and ecofacts can obviously cause further damage than the initial oiling, particularly in dealing with highly porous and fragile materials. The use of a Teflon scraper, rather than an abrasive brush, is recommended for the removal of excess oil from wood, bone, and shell (Church 2011). Solvents, such as acetone, may be appropriate for some wooden objects, but may damage bone and shell. Avoiding acidic solvents is important, particularly in cleaning bone and shell. The NCPTT recommendations on the cleaning of artifacts are tempered by the statement that "little to no scientific research has been done on the removal of crude oil from archeological materials" (Chin 2011:5).

One of the contributions of the present study is in recognizing that information might be damaged or lost with the hydrocarbon contamination of artifacts, such as absorbed pottery residues. The standard operating procedure for this study was to not clean artifacts, so as to assess the effects of crude oil and contaminants. The use of solvents for cleaning may cause further damages and information loss, such as isotope or DNA studies of bone. The cleaning of contaminated artifacts is nonetheless advisable for long-term conservation, to inhibit decomposition and prevent the emission of potentially harmful gaseous fumes. Besides the potential health hazards, the volatile compounds in crude oil can interact with plastics

and storage materials. A small number of oil-contaminated artifacts from the MC252 oil spill response are curated with the Louisiana OCD. These were allowed to off-gas for one to two years before being wrapped in foil and placed in plastic bags for curation (Ashley Fedoroff, personal communication 2 July 2018). Pottery sherds collected as special samples during the present study were wrapped in foil, placed in cloth bags, and kept under refrigeration.

Because it may be preferable to not clean some materials, or impossible to completely remove crude oil from other materials, the NCPTT recommends permanently segregating oiled artifacts from other collections. This may require the separation of collections based on material, such as oiled bone, soil samples or other materials in which the oil cannot be removed (Chin 2011:8). It may involve special storage conditions, such as the use of refrigeration during the present study, at least until collections can be properly cleaned. The potential costs of such measures will be taken up in the following section. The costs and potential hazards of curating collections contaminated with crude oil, particularly bulk items such as shell or materials, such as bone, that might not be adequately cleaned without being destroyed, should be counterbalanced with the potential for the collection to still yield important information. The effects of an oil spill on archaeological sites might ultimately include not collecting certain classes of oiled materials.

#### 7.3.3 Cost Estimates

As described in the preceding section and in regard to the seventh hypothesis, the effects of an oil spill on archaeological sites include increased time requirements and costs for data collection, analyses, conservation and curation. What remains to be considered is the additional amount of time and expense. Since crude oil is hazardous to human health and remains toxic for an extended period of time, fieldwork at oiled sites and laboratory research with contaminated collections requires appropriate safety protocols and equipment, such as respirators and gloves (Chin 2011:2–3). Any additional effort or expenditure that might be required due to the presence of crude oil at a site, or within a collection represents an increase in the overall research cost estimate, and is therefore one of the potential effects of an oil spill on archaeological sites. The NHPA and National Register criteria of eligibility provide an underlying rationale for the linkage between research cost estimates and the effects of an oil spill, because a prehistoric archaeological site that is determined to be eligible is likely to be evaluated based on the prospective information contained in the archaeological deposits. Any increased research expenditure in time or funding due to an oil spill represents indirect adverse effects on a historic property.

The costs of field and lab research are likely to increase in proportion to the amount of oil at archaeological sites and within collections, but also in relation to the goals and methods of research. The additional costs may be negligible for an archaeological survey of a redeposited shell midden that produces few contaminated artifacts from a lightly oiled shoreline. A more intensive investigation involving large-scale excavations of intact archaeological deposits at a heavily oiled site will involve relatively greater expenditures of resources to accomplish field and lab research. For the purposes of this site assessment summary, estimates of the costs of archaeological research are based on a hypothetical Phase III excavation at a heavily oiled coastal site. The regulatory requirement for such data recovery excavations to mitigate the adverse effects of an undertaking on a historic property listed or eligible for listing on the National Register is well established in the Louisiana OCD NHPA Section 106 Field Standards (LDA 2017). The cost estimate for the proposed investigation is based on three months of fieldwork for a ULL crew of five archaeological technicians and a project director. Projected direct costs represent minimum estimates for accomplishing field and laboratory research on a coastal site in the Mississippi River delta.

Minimal estimates of additional costs are based on the time, effort and expenditures required for field and lab research, as well as the conservation and curation of collections. Cost estimates for data collection and analyses are based on the present study and price lists provided by consultants. The increased costs of

fieldwork at a heavily oiled coastal site will, at a minimum, involve additional expenditures for safety equipment and protective clothing for working in a hazardous environment. Assuming archaeological technicians are provided the same personal protective equipment and special training required by the Occupational Safety and Health Administration (OSHA) for oil spill cleanup workers in Hazardous Waste Operations and Emergency Response, this will include oil resistant gloves, boots, disposable coveralls, safety glasses and respirators (OSHA 2018). The additional cost for personal protective equipment is estimated at \$4,040 or an additional 2% based on a budget that includes \$200,000 total direct costs. Additional expenditure of time and effort for excavating at an oiled site and working with contaminated collections is difficult to determine, but, based on the present study, it is conservatively estimated at 10% of projected personnel costs and an overall increase of 7.5% for total direct costs. In addition to crew training for working with hazardous substances, a designated safety officer should be on site during data recovery.

The additional analytical costs include the chemical detection and characterization of oil for 10 soil samples, at \$650 per sample. The need for this GC/MS analysis is based on the findings of this study that crude oil contamination effects absorbed pottery residue analysis and has the potential to affect other analytical techniques. The cost estimate of \$6,500 represents an additional 3.3% of the total direct cost that would not otherwise be needed for data recovery excavations at a coastal site. The cost estimate of \$11,900 for radiocarbon dating by AMS is based on 20 samples at \$595 per sample. Experimental data produced by this study indicate that pretreatment by solvent extraction is recommended for samples contaminated by crude oil. Solvent extraction is included for each sample, at an additional cost of \$185 per sample. This represents a 31% increase in the projected cost of radiocarbon dating services and an additional 1.9% of the total direct cost. The additional cost of pretreating all samples by solvent extraction might be reduced if GC/MS analyses of some associated matrices are negative for oil, but this scenario assumes uniform contamination with crude oil.

Absorbed pottery residue analysis is included for 20 samples, with an increase of 13.5% for working with samples contaminated with oil and/or dispersant. This is a minimal cost estimate for additional time and assumes the analysis is possible. Contamination with oil and dispersant can impede absorbed residue analysis and cause a loss of information. Cost estimates are more problematic for artifact conservation and collections management, as there has been little research on archaeological materials contaminated with crude oil (Chin 2011). For this exercise, the additional cost is based on a conservative estimate of three months of time and effort for a conservator to decontaminate and prepare collections for long term curation. This might be increased or decreased according to the size of the collections.

The estimated increase in the total direct cost for a Phase III investigation of a coastal site affected by an oil spill is \$41,863, or approximately 21% of the initially projected \$200,000 (Table 21). This represents minimal cost estimates in most categories and does not account for indeterminate variables such as changing environmental conditions, irregular amounts or distributions of crude oil at a site, or unforeseen consequences of working with hazardous substances. Other research costs, such as archaeobotanical or zooarchaeological analyses, might be added to the total direct cost. The projected Phase III investigation is relatively small for site mitigation by data collection and might be expanded accordingly, with cost estimates scaled upward to reflect increased expenditures for field and lab research. Although the preceding exercise is largely conjectural, it represents a conservative estimate and best-case scenario for the indirect effects of an oil spill on archaeological research.

Table 21. Cost estimate for a Phase III investigation of a coastal site affected by an oil spill

	Cost Estimate	Additional Cost Estimate	Total Cost
Personnel (including fringe benefits), plus 10% time and effort	149,220	14,922	164,142
Travel (lodging and transportation for three months)	25,800		25,800
Equipment and supplies, plus personal protective equipment (oil resistant gloves, boots, disposable coveralls, safety glasses and respirators)	8,800	4,040	12,840
Chemical characterization of oil by GC/MS (10 samples at \$650 per sample)		6,500	6,500
AMS dating (20 samples at \$595 per sample), plus pretreatment by solvent extraction (\$185 per sample)	11,900	3,700	15,600
Absorbed pottery residue analysis (20 samples at \$74 per sample)	1,480	200	1,680
Curation, plus artifact conservation and collections management	2,800	12,501	15,301
Total estimated direct cost	200,000	41,863	241,863

#### 7.3.4 Recommendations

In assessing the effects of the MC252 oil spill on prehistoric archaeological sites, the objectives of this study were to provide the Louisiana SHPO and OCD Division of Archaeology with information relevant to CRM planning. As described in the preceding sections, the proximate impacts of an oil spill on the archaeological record can include the contamination of artifacts, ecofacts, and cultural deposits, adversely affecting analyses and producing hazardous conditions for fieldworkers and laboratory technicians working with collections. The cleanup response after an oil spill can cause indirect impacts associated with site remediation and environmental restoration. Though the long-term impacts on site formation processes are not yet fully understood, the adverse effects include a potential loss of information for some archaeometric analyses, increased research costs, and subsequent challenges for materials conservation and curation.

Recommendations involving archaeological research at sites affected by an oil spill have included pretreatment of radiocarbon samples by solvent extraction. Chemical testing for crude oil by GC/MS is also recommended for archaeological contexts to be sampled for different analytical techniques, as the presence of oil may not always be readily discerned by sensory observation. Chemical testing should include the detection of other contaminants, such as dispersants used in an oil spill response, as these may also affect subsequent analyses. The presence of contaminants, such as crude oil, might then be taken into account when preparing cost estimates for research or planning for collections management and curation. Appropriate safety measures should always be taken in the field and lab when working under conditions where crude oil or other hazardous materials are present.

Looking forward, CRM planning and decision making in regard to oil spills should be an integral part of comprehensive archaeological and historic preservation plans (Doucet and Hobson-Morris 2017:57; Girard et al. 2018:66; Smith et al. 1983). This is especially important for coastal regions such as the north-central GOM, where the oil and gas industry's development of the nation's energy infrastructure entails regulatory compliance with historic preservation and cultural resource legislation. The protection of historic properties and cultural resources is included in the Environmental Planning and Historic Preservation program of the Federal Emergency Management Agency (FEMA 2018). State, tribal, and local emergency management plans, including preparations for oil spill response, should likewise integrate historic preservation and CRM planning (Montgomery 2008). In light of the findings of the present study, recommendations for CRM planning for an oil spill are offered here as a three-tiered process, along the lines presented by Demas (2002:28).

- 1. Plan: Integrate oil spill readiness and response into Louisiana's Comprehensive Archaeological Plan.
- 2. Program: Support a proactive program of regional archaeological survey, monitoring and testing of coastal archaeological sites.
- 3. Prioritize: Identify research strategies and priorities for the mitigation of archaeological sites affected by an oil spill.

CRM planning begins with collecting information on the expectations of various stakeholders, as well as data on the cultural resources and sites, and proceeds to consultations and assessment of current site conditions, including threats to the *in situ* preservation of sites and management constraints. It will be important to incorporate a wide range of heritage values and research design strategies at the planning stage. Consultations with culturally affiliated groups and key constituencies should be geared toward the forming partnerships for heritage stewardship, a collaborative enterprise to be fostered during all stages of the CRM planning process. The management and treatment of human remains and cultural patrimony at coastal sites affected by an oil spill should involve culturally affiliated groups, such as the Chitimacha Tribe of Louisiana, as well as compliance with the Louisiana Unmarked Human Burial Sites Preservation Act (R.S. 8:671-681).

Multiple heritage values and research goals may be complementary or conflicting, so it will be advantageous to have planned in advance. A site determined unlikely to produce information of historical importance due to a lack of archaeological integrity may be eligible for listing on the NRHP through historical associations. The identification and monitoring of traditional cultural properties will require continuing consultations. The potential information contained in one site comprised of redeposited cultural materials and shoreline midden may be limited. However, research designed to examine regional or extraregional interactions and population movements may benefit from the information obtained from the study of many such sites.

Louisiana's Comprehensive Archaeological Plan recognizes the adverse effects of oil and gas development on coastal archaeological sites, and the potential effects of an oil spill (Girard et al. 2018). CRM planning for oil spill readiness and response should be based on the widest possible appraisal of heritage values and resources. In this respect, oil spills are only one of many critical issues in managing cultural resources on the Gulf Coast. Archaeological sites in the Mississippi River delta are endangered by coastal erosion, subsidence, relative sea-level rise, storm surge, wetlands restoration, and a wide range of anthropogenic landscape alterations (Anderson et al. 2017). Integrated CRM planning for the effects of an oil spill should commence and be carried out in this context, as part of Louisiana's Comprehensive Archaeological Plan (Girard et al. 2018; Smith et al. 1983).

Oil and gas development and the effects of an oil spill present challenges and opportunities in managing archaeological sites, but a comprehensive plan should also identify programmatic solutions and possible mitigation measures (Girard et al. 2018). Integrated CRM planning should proceed to programming for the collection of additional data and informed decision making in an emergency oil spill response, building on the first stage of the process. The Louisiana OCD, Division of Archaeology and SHPO should actively pursue proactive programs of regional archaeological survey, monitoring and testing of coastal archaeological sites. Though such recommendations are easy to make, they are more difficult to support with adequate funding. The consequences of inaction are already apparent. Archaeological survey as part of the MC252 oil spill response revealed a high rate of site loss due to coastal erosion and subsidence, resulting in redeposited and submerged sites, and the discovery of two previously unrecorded mound sites and 48 other newly recorded sites (Cloy and Ostahowski 2015). Basic information is still lacking from many coastal sites on cultural affiliation, stratigraphy, integrity, and boundaries, including sites recently

recorded (16SB185) and sites known about for decades (16SMY17). Oil and dispersant have been detected in archaeological deposits at these and other sites.

A comprehensive program of regional survey and site monitoring is needed to examine the long-term effects from the MC252 oil spill of 2010 in relation to changing environmental conditions. The "business as usual" Section 106-based CRM approach of identifying cultural resources and determining potential effects to historic properties within an APE for an undertaking is ill equipped to deal with an emergency response to an oil spill, hurricane or other catastrophic event. The Louisiana OCD, Division of Archaeology lacks the resources for such large-scale emergency CRM response (Girard et al. 2018). In the tradition of earlier investigations (McIntire 1958; Neuman 1977a), the Louisiana OCD, Division of Archaeology might collaborate with the Louisiana Archaeological Survey and Antiquities Commission and State universities to establish and support programs of archaeological research focused on regional survey, monitoring, and testing of endangered coastal sites.

The third stage in the CRM planning process depends on the solid foundation of the first two tiers. Demas (2002:30) addressed site management planning; a regional or State-based approach to CRM planning will be comparable. The decision-making response will involve establishing policies, objectives and strategies in advance, as part of an integrated CRM plan. Identifying and prioritizing research strategies will not be without difficulties or controversy, especially if it means not mitigating the loss of one particular site or the information it might yield. Prioritization should stem from open dialogue to identify collective interests, if not consensus. For example, a decision to not collect, conserve, or study human remains eroding from burials in a shell midden affected by an oil spill might allow research partners to dedicate more resources and time needed for collecting data from deeply buried cultural deposits on a subsided landform. Prioritization might identify alternative mitigation strategies in which the combined effects of an oil spill, coastal erosion, and subsidence on one site lead to the investigation or preservation of other sites.

CRM planning to mitigate the effects of an oil spill on coastal archaeological sites should also prioritize research strategies and data collection, such as information on subsistence patterns and foodways during the Tchula, Marksville and Coles Creek periods, or evidence for extraregional Mississippian interactions with local Plaquemine communities. Building on earlier investigations, research strategies might identify and prioritize different types of sites and site conditions to collect information on long-term histororical ecology and geomorphology in the rapidly changing Mississippi River delta (McIntire 1958; Mehta and Chamberlain 2018). Sites with evidence for intact shell midden and earthen mounds, including deeply buried and submerged sites, will undoubtedly elicit different management decisions and research strategies than redeposited artifact scatters lacking evidence of archaeological integrity. Strategies of site triage might involve preemptive data collection at some sites and inaction at other sites, where data can no longer feasibly be collected or preserved. Integrated CRM planning and programming for the effects of an oil spill will ultimately require establishing priorities and making difficult decisions.

# 8. Summary and Conclusion

In the late spring and summer of 2010 an unprecedented environmental disaster unfolded off of Louisiana's Gulf Coast. Beginning with a catastrophic explosion on the *Deepwater Horizon* drilling rig, the MC252 Macondo wellhead released approximately 4.9 million barrels of crude oil and created the largest marine oil spill in history (Joye 2015; McNutt et al. 2012; Nixon et al. 2016). The MC252 oil spill impacted hundreds of miles of shoreline and wetlands in the Mississippi River delta, the largest river delta in North America (BOEM 2014; Michel et al. 2013). The application of dispersants at the wellhead and by aerial spraying was also unprecedented, as was the scope of the cleanup response (Kujawinski et al. 2011; Seidel et al. 2016; USCG 2011).

The consequences of this environmental disaster are still being investigated eight years later. Scientists are only now beginning to understand the long-term impacts on marine and wetlands ecology (Baker et al. 2017; Beyer et al. 2016; Hester et al. 2016; Joye et al. 2014, 2016; Romero et al. 2015; Schwacke et al. 2014; Valentine et al. 2014; Yang et al. 2016). Cultural resources management was incorporated into the oil spill response from the outset, informed by previous experience following the 1989 *Exxon Valdez* oil spill in Alaska (Cloy and Ostahowski 2015; Chin and Church 2010; HDR 2011; Reger et al. 2000). Yet an oil spill of such magnitude had never occurred in the deltaic and marsh environments of the north-central GOM. The full effects of the MC252 oil spill on underwater and terrestrial cultural resources are only now being systematically described (Hamdan et al. 2018; Salerno et al. 2018).

Questions raised in the aftermath of the MC252 oil spill about its immediate and long-term effects on the archaeological record. Yet there was a critical lack of up to date information on the condition of the affected sites. This was remedied by a comprehensive program of systematic survey and site monitoring along the north-central Gulf Coast, spanning thousands of miles of shoreline from Texas to Florida (Cloy and Ostahowski 2015; HDR 2011). In Louisiana alone, archaeologists with HDR, Inc. revisited 163 previously recorded sites and recorded 50 new sites. A majority of these sites consist of pottery sherds and other cultural materials scattered along shorelines, representing redeposited archaeological components dating from as early as the Tchula period (800 BCE–1 CE). Two sites with earthen monuments—Acorn Mounds (16SB185) and Live Oak Bayou Mounds (16SB186)—were among the newly-recorded sites that HDR. recommended as eligible for listing on the NRHP (Cloy and Ostahowski 2015:1-6, 5-22, 5-31, 6-1, 6-2, 7-1, 7-5, 7-18). Sinking into the marsh on subsided landforms, these mound centers had, unbelievably, escaped the attention of archaeologists for more than a century.

Archaeologists and geologists began to slowly uncover evidence for shifting human habitations in the Mississippi River delta only during the last century (Gagliano 1984; Kniffen 1936; McIntire 1971; Phillips et al 1951). A succession of deltaic lobes dating from the last few millennia of the Holocene Epoch have been home to diverse groups of people living along the river, bayous and coast. Fisher-hunter-gatherers who left potsherds, bone, and shell from as early as 2,800 years ago are known today as Tchefuncte culture of the Tchula period. They were succeeded by cultures that archaeologists named Marksville (1–400 CE), Troyville (400–700 CE), Coles Creek (700–1200 CE) and Plaquemine (1200–1700 CE). Non-local ideas and natives identified as Mississippian arrived in the delta a few centuries before ships from Europe appeared on the horizon. Throughout the millennia, the Mississippi River spread out over its banks, flooded the low-lying wetlands and shifted course, forming new distributaries, building natural levees and lakes. In many places, deltaic progression has erased or obscured the material evidence of human adaptations. Artifacts from successive cultures lie scattered in redeposited shell midden along the coast. At some sites, the archaeological record is still intact and contains an unparalleled cultural and ecological history of human resiliency on the north-central Gulf Coast.

Also during the past century, as archaeologists assembled the culture history of the Mississippi River delta and Gulf Coast, the oil and gas industry developed the nation's energy infrastructure offshore. Anthropogenic changes in Louisiana's coastal wetlands from canals and pipeline construction are the

most recent chapter in a culture history that extends back for millennia, of which archaeology can reveal sustainable human interactions in a shifting landscape. The extraction of oil and gas on the Gulf Coast and OCS has affected cultural resources, impacts that archaeologists have long recognized and the State of Louisiana has managed through legislation and regulations (Neuman 1977a; Smith et al. 1983:97–100, 118). Archaeological sites in the delta have always been impacted by coastal erosion and subsidence, but the accelerated loss of Louisiana's wetlands has been increasingly anthropogenic and severe (Davis 2010).

The effects of the MC252 oil spill on archaeological sites along Louisiana's Gulf Coast must ultimately be understood in relation to long-term cultural and geological processes that have shaped the delta. Archaeological survey and monitoring during the MC252 oil spill response revealed that the ongoing loss of sites to coastal erosion and subsidence has continued apace during the past 50 years (Neuman 1977a; Ostahowski 2015, 2016). The number of submerged, subsided, and redeposited coastal sites outnumbers the recorded terrestrial sites with undisturbed archaeological deposits. Regardless of condition, crude oil from the MC252 spill washed ashore at coastal sites, with virtually unknown effects on artifacts, ecofacts, and cultural deposits. There was little available information on the extent or potential consequences of contamination by hydrocarbons and dispersants used in the response. It was unknown whether crude oil would infiltrate archaeological deposits and contaminate artifacts, or what the consequences of such contamination might entail.

The research design for the present study incorporated many of the questions raised in the wake of the MC252 oil spill. Among the stated goals and objectives were to examine the effects of an oil spill on Native American sites, including the proximate impacts on artifacts, ecofacts, and cultural features that make up the archaeological record, as well as the application of analytical techniques. This study also aimed to assess the potential for long-term and indirect impacts on site formation processes, field and laboratory research, conservation, and curation. In doing so, this study would provide the Louisiana SHPO and OCD Division of Archaeology with information relevant for CRM planning and regulatory compliance. Any lessons learned from investigating archaeological sites affected by the MC252 oil spill should inform CRM decision making and future responses to an oil spill along the Gulf Coast. In evaluating the cultural and environmental impacts, the CRM implications of this research are also relevant to BOEM's mission of managing offshore oil and gas development in the north-central GOM. In pursuit of these goals and objectives, BOEM initiated and supported this study through a cooperative agreement with ULL (Award Number M14AC00022), in collaboration with the Louisiana State Archaeologist and Division of Archaeology.

# 8.1 Summary of the Methods and Results

Over a period of thirteen months, beginning in September of 2014, archaeologists from ULL conducted fieldwork at eight sites on Louisiana's Gulf Coast. SCAT teams and archaeologists had observed oil on the shorelines at six of these sites during the MC252 oil spill response. The selection of two other control sites was based on a lack of reported oiling. Four of the assessed sites are located on eroded remnants of marsh islands in eastern St. Bernard Parish, west of Chandeleur Sound (16SB178, 16SB174, 16SB182, and 16SB185). The first three of these sites (16SB174, 16SB178, and 16SB182) appear to consist entirely of redeposited cultural materials and shell midden from now submerged and eroded sites offshore. Previous investigations and fieldwork for the present study indicate the Comfort Island (16SB174) and Southern Comfort (16SB178) sites are partly made up of redeposited artifacts and midden from Marksville and Tchefuncte components (Cloy and Ostahowski 2015:6-758, 6-767). The Acorn Mounds site (16SB185) stands out, with deeply buried, intact archaeological deposits and three earthen mounds on a subsided marsh island south of Drum Bay. Though surface collections of diagnostic pottery along the shoreline to the east indicate late Marksville and later Troyville components (Cloy and Ostahowski 2015:6-804), the present study produced evidence for the commencement of mound construction during

the first century of the Coles Creek period. The boundaries of this subsided ceremonial site have not yet been determined by systematic subsurface testing.

The fifth site is in Jefferson Parish (16JE2), on Bayou St. Denis in the Barataria Basin. It also contains intact archaeological deposits and has two shell and earth mounds. The Cheniere St. Denis site (16JE2) stands out as one of only two intensively investigated sites included in this study. Previous investigations identified intact Troyville and Coles Creek components, with shell midden and mounds dating from the last century of the Baytown period and first three centuries of the Coles Creek period (ca. 670 to 970 CE). The Cheniere St. Denis site has been recommended eligible for listing on the NRHP (Coughlin et al. 2004; Gagliano et al. 1979). In contrast, the sixth site (16LF293) consists of scattered artifacts in redeposited shoreline midden. The Redfish Slough site (16LF293) in Lafourche Parish lies on the southwestern shoreline of Philo Brice Island in Timbalier Bay. The pottery assemblage from the site indicates Coles Creek, Plaquemine, and Mississippian components (Cloy and Ostahowski 2015:6-374). Cultural deposits may remain intact in deeply-buried contexts or submerged offshore.

The two control sites lie on the northeastern and western peripheries of the study area, in St. Bernard Parish on the south shore of Lake Borgne (16SB153) and in St. Mary Parish on the shore of East Cote Blanche Bay (16SMY17). Site 16SB153 on Lake Borgne is the only other site in this study that has been intensively examined by archaeologists (Weinstein et al. 2012). Intact cultural deposits dating from the Baytown (400–700 CE), Coles Creek (700–1200 CE), and Mississippi (1200–1700 CE) periods are deeply buried along the shoreline, with intact shell midden extending offshore beneath the lakebed. The site includes a historic component possibly associated with the Filipino fishing village of St. Malo. Along with Acorn Mounds and Cheniere St. Denis, 16SB153 is one of the few assessed sites with cultural deposits known to retain archaeological integrity and previously recommended as eligible for inclusion on the NRHP (Weinstein et al. 2012:30–31, 187–188).

Bayou Sale (16SMY17) is the other site included in this study as a control, because it lies approximately 275 miles (443 km) northwest of the MC252 well and SCAT teams did not observe oil along the shoreline during the cleanup response. Although archaeologists have known about this site and revisited it for more than 60 years, little is actually known other than the presence of an extensive shell midden along the shoreline of East Cote Blanche Bay. The midden contains scattered human remains and wave-washed Baytown Plain, *var. unspecified* potsherds from the erosion and redeposition of burials and cultural deposits. A Coles Creek affiliation is suggested by a single Coles Creek Incised, *var. Stoner* sherd. Three AMS dates on turtle bone from TU 2 (Level 4) indicate a late Baytown-early Coles Creek component (2-sigma calibrated calendar ages ranging between 620 and 770 CE). The site boundaries, archaeological integrity, and NRHP eligibility have yet to be determined, with the possibility of intact midden being deeply buried or submerged offshore.

Special sampling procedures and field methods were developed to assess the effects of the MC252 oil spill on these sites and to avoid the inadvertent contamination of cultural deposits during excavation and collection. The ULL field crew excavated a minimum of one square meter at each site, recorded stratigraphic profiles and the proveniences of samples. One hundred and seventy one special samples were collected from all eight sites (Appendix A). These included artifacts, ecofacts, soil samples, column samples, unit cores and soil cores. The investigators took extraordinary measures to avoid the accidental introduction of oil from the ground surface and other sources outside of the cultural deposits and excavated contexts. Special samples were sealed in aluminum foil, clean metal containers and cloth bags, recorded by provenience, placed in air tight, waterproof containers and transported to the ULL lab for processing and analysis.

Although assessing the effects of an oil spill on intact archaeological deposits was a research priority, most of the cultural deposits examined during the fieldwork consist of shoreline accumulations of redeposited artifacts and midden. This is not uncommon in the Mississippi River delta, where coastal

erosion and relative sea-level rise are redrawing Louisiana's coastline (Cloy and Ostahowski 2015:7-17; Wendland 2016). The ULL field crew collected samples from intact archaeological deposits at three sites (16JE2, 16SB153, and 16SB185). Although a majority of the samples from all eight sites were collected from redeposited, secondary contexts, this study has presented evidence that sites lacking archaeological integrity can still yield important information.

In addition to the culture historical and geomorphic associations of diagnostic artifacts, displaced cultural materials from sites otherwise lacking intact cultural features and stratified deposits can still provide useful information on site chronology, subsistence, and the composition and source of artifacts. Despite redeposited contexts, four out of five radiocarbon samples from the Comfort Island and Scow Island Scatter sites produced dates consistent with the late Coles Creek components indicated by diagnostic ceramic types. Absorbed pottery residue analysis can provide information on the edible plants and animals of past foodways, if potsherds are not contaminated with oil and dispersant. Elemental analyses of pottery sherds from sites throughout the region might address extraregional population movements and exchange. As the research potential of redeposited artifacts along shorelines has not been fully realized and these represent a majority of sites in the Mississippi River delta, the effects of an oil spill should not be prematurely dismissed as inconsequential to NRHP eligible properties.

## 8.2 Summary of the Site Assessment

In accomplishing the major tasks of this study, oil source analysis and impact assessment have generated new information and increased knowledge regarding the effects of an oil spill on archaeological sites. The chemical characterization and fingerprinting of oil by GC/MS was especially important given the goals of this study and the subjective nature of visual, olfactory and tactile inspection. Twelve of 28 samples (43%) from the eight assessed sites produced evidence of petroleum hydrocarbon analytes associated with crude oil. The positive samples were collected from the six sites where oil was observed during the oil spill response (Appendix C; Meyer et al. 2017). The LSU DES laboratory analyzed biomarkers for oil source fingerprinting and identified two samples from the Acorn Mounds site (16SB185) as a match for MC252 oil. Three samples from three other sites (16SB174, 16SB178, and 16LF293) were possible matches for MC252. The oil was too weathered in seven samples (58%) to determine the source, or was inconclusive for MC252. The majority of the samples that tested positive for oil were excavated from secondary contexts (n=7; 58%). Two samples (17%) in which oil was detected were collected from the surface, including a sample from the shoreline east of Acorn Mounds (16SB185) that matched MC252 oil (Appendix C; Meyer et al. 2017).

Three of the samples in which the LSU DES lab detected oil came from intact archaeological deposits at the Acorn Mounds (16SB185) and Cheniere St. Denis (16JE2) sites. A core sample (CT 1, CS 1; SBH027) from 0 to 30 cm below surface on the northwest slope of Mound B at the Acorn Mounds site tested positive for oil. Because of the sampling procedures and field methods, the oil is unlikely to have been introduced during the investigation. The oil was too weathered for chemical fingerprinting, so its source cannot be conclusively attributed to MC252. Two samples with oil that matched MC252 came from the shoreline and a shovel test approximately 140 meters to the east. The oil detected in the core sample could have been transported to Mound B by storm surge, or it may represent another oil spill or anthropogenic source (Asl et al. 2016). The two samples from the Cheniere St. Denis site that contained oil (JEB021 and JEB026) consisted of soil matrix and residue from the surfaces of *Rangia* shells collected from a test unit (TU 3, Levels 2 and 3) in the western slope of the northernmost mound. Conceivably introduced by the rising water table during excavation, the oil was too weathered for chemical fingerprinting and an inconclusive match for MC252. Regardless, intact archaeological contexts at Cheniere St. Denis and Acorn Mounds contained crude oil.

One aspect of assessing site impacts involved investigating the effects of an oil spill on radiocarbon dating. Techniques for dealing with contaminants in radiocarbon samples are well established, so this study focused on pretreatment techniques for AMS analysis of samples contaminated with crude oil. Samples were selected from archaeological contexts in which GC/MS analysis had detected crude oil. The samples were subdivided and processed by standard pretreatment or solvent extraction in order to examine the potential effects of hydrocarbon contamination on the results of radiocarbon dating. AMS yielded results for a total of 17 subsamples from six of the eight assessed sites (Section 6.6). The radiocarbon dates for samples from intact archaeological deposits at Cheniere St. Denis, Acorn Mounds, and Site 16SB153 are consistent with Coles Creek affiliations for the first two sites and a Mississippian component at Site 16SB153. The initial results showed no significant differences in radiocarbon assays associated with different pretreatment methods. However, the presence of hydrocarbons in the samples was uncertain because crude oil was detected only in associated strata or surrounding matrices and not in the samples submitted for radiocarbon analysis.

To control for the presence of oil in the pretreatment and radiocarbon dating of samples, two experiments were conducted in which samples were subdivided and half were intentionally contaminated with crude oil from the shoreline of the Southern Comfort site (16SB178-26). In the first experiment, oil was smudged onto two subsamples of bone fragments from Site 16SB153 (16SB153-33). One contaminated subsample was processed by solvent extraction (Beta- 421664) and the other received standard pretreatment (Beta-421663). Two uncontaminated subsamples were also processed by solvent extraction (Beta-421662) and standard pretreatment (Beta-421661). The resulting radiocarbon dates by AMS coincide with a deeply buried Mississippian context in TU 1 at Site 16SB153, regardless of pretreatment technique. Both solvent extraction and standard pretreatment mitigated the effects of surficial or short-term crude oil contamination.

In the second experiment, two subsamples of turtle bone (16SMY17-21) from the Bayou Sale (16SMY17) control site (TU 2, Level 4) were soaked in a mixture of crude oil and seawater for one week. One contaminated subsample was pretreated by solvent extraction (Beta-421668) and the other received standard pretreatment (Beta-421667). Two uncontaminated subsamples were also processed by solvent extraction and standard pretreatment (Beta-421666 and Beta-421665). The AMS results for three subsamples coincided with a late Baytown-early Coles Creek component, with 2-sigma calibrated calendar ages ranging between 620 and 770 CE. The contaminated subsample that received standard pretreatment produced a 2-sigma calibrated calendar age that was 750 to 840 years earlier. The error can be attributed to the failure of standard pretreatment to correct for the presence of crude oil in the sample. Though severe contamination or prolonged exposure to crude oil in seawater can affect the results of AMS dating, pretreatment by solvent extraction mitigates the adverse impacts. Pretreatment by solvent extraction is consequently recommended for radiocarbon samples from coastal sites where oil is present or suspected to be present. The experimental data also indicate crude oil was not introduced into archaeological contexts at the assessed sites in sufficient amounts to cause erroneous AMS results with standard pretreatment.

Trace element analysis of pottery sherds by NAA and LA-ICP-MS does not appear to be affected by the presence of hydrocarbons, at least for the samples analyzed for this study. The MURR Archaeometry Lab analyzed four grog-tempered pottery sherds from oiled contexts at the Comfort Island and Southern Comfort sites, and one sherd from the control site (16SB153) on Lake Borgne. The results of NAA and LA-ICP-MS indicated no increased concentrations of elements known to be present in crude oil, although one sherd (SBC035) from the Southern Comfort site had elevated levels of Arsenic (Appendix D). Elemental analysis was not adversely affected for pottery sherds collected from oiled archaeological contexts. This should also be the case for trace element studies of lithics. Standard pretreatment, such as washing and use of a burring tool to remove oiled surfaces, appears to mitigate the presence of oil. The presence of oil should not adversely affect related provenance studies of ceramics or lithics. Because oil

was chemically detected in associated contexts but not for individual samples, experimental studies might combine GC/MS and NAA or LA-ICP-MS to examine hydrocarbon absorption and its potential effects in different archaeological materials.

In contrast, oil and dispersant can adversely affect the analysis of absorbed pottery residues by GC/MS. Eleanora Reber analyzed 17 pottery sherd samples from seven sites and was able to identify biomarkers for crude oil, as well as the dispersants used in the MC252 oil spill cleanup (Appendix E). Nine of the 17 samples (53%) contained some amount of oil and/or dispersant. Both oil and dispersant were present in the absorbed residues in two sherds, some amount of oil without dispersant was detected in three sherds, and dispersant without oil was present in four sherds. Six of the nine sherds (67%) contaminated with oil and/or dispersant were associated with strata or near soil matrices that independently tested positive for oil. Soil samples from four of these contexts were chemically fingerprinted as possible matches for MC252 oil. There is a correlation between oiled archaeological contexts and the contamination of pottery sherds. Only three sherds (33%) with biomarkers for oil and/or dispersant were collected from contexts that tested negative for oil.

Seven of the nine contaminated sherds came from three oiled sites (16JE2, 16SB178 and 16LF293). Reber surprisingly detected biomarkers for oil and/or dispersant in one sample from each of the control sites. These sherds were from excavated contexts in which overlying or underlying soil matrices had tested negative for oil. Both oil and dispersant were detected in a shell-tempered sherd from an intact, deeply buried context at Site 16SB153 on Lake Borgne (16SB153-15). A single sherd from the Bayou Sale site contained dispersant (16SMY17-18). Although other contaminants were detected, the prevalence of dispersant in the tested samples and in sherds from the control sites may reflect the extensive use of dispersants during the MC252 oil spill response.

Reber calculated relative percentage of contamination per gram of sherd and concluded that moderate to severe contamination (at least 6%) impedes the interpretation of absorbed pottery residues, with the greatest difficulties and information loss associated with combined oil and dispersant contamination (Appendix E). There is no clear association between the percentage of contamination and the depth of archaeological deposits. This may be due to redeposited contexts or small sample size. Nevertheless, the results show that contamination of potsherds with oil and dispersant can impede or cause a loss of information in the interpretation of absorbed animal and plant residues.

#### 8.3 Conclusion

In conclusion, this study has answered many of the questions initially posed in the aftermath of the MC252 oil spill. Oil is present in the archaeological record at sites affected by the MC252 oil spill, including soil matrices and permeable artifacts such as ceramic sherds. There is a correlation between oiled archaeological contexts and the presence of oil and dispersant in absorbed pottery residues. Hydrocarbons from crude oil are also absorbed into bone in combination with seawater. Because this study was principally focused on field research at coastal sites affected by an oil spill, additional experimental studies are needed to more fully address the permeability and contamination of different archaeological materials in relation to crude oil and dispersant.

Assessing the effects of an oil spill on intact, stratified archaeological deposits has proven to be one of the more challenging tasks because a majority of the coastal sites included in this study consist mostly of scattered cultural materials redeposited along shorelines. There was no observed correlation between the depth of intact or redeposited cultural deposits and the presence of oil. Oil was detected in samples from shorelines and from various depths in excavation units at the assessed sites, including intact cultural deposits at Acorn Mounds (16SB185), Cheniere St. Denis (16JE2), and control site 16SB153 on Lake Borgne. The oil at Site 16SB153 was detected with dispersant in absorbed pottery residue from a deeply stratified deposit (TU 1, 125-135 cm). Only two samples of oil, from a shovel test and surface of the

shoreline east of Acorn Mounds (16SB185), were chemically fingerprinted as a match with MC252. The oil at archaeological sites could be from other sources, although some samples were possible matches for MC252 and others, too weathered for chemically fingerprinting, may have degraded in archaeological contexts.

Chemical analysis by GC/MS can, nonetheless, determine the presence of oil in archaeological contexts prior to radiocarbon dating and the application of archaeometric techniques. The LSU DES lab detected oil in samples from all six sites where SCAT teams had observed oil. Pretreatment of radiocarbon samples by solvent extraction is recommended for archaeological contexts affected by an oil spill, especially wet sites, to mitigate for possible hydrocarbon contamination. Standard pretreatment may otherwise result in erroneous results. Absorbed pottery residue analysis can be adversely affected by hydrocarbon contamination, especially in combination with dispersants used in an oil spill cleanup response. Chemical testing for oil should be routine at coastal sites affected by an oil spill prior to absorbed residue analysis. Experimental studies might also assess the potential effects on other archaeometric techniques, such as XRF, TL, isotope analysis, and DNA analysis.

Though radiocarbon samples should be pretreated by solvent extraction to mitigate the adverse effects of hydrocarbon contamination, standard pretreatment techniques may be adequate for elemental analysis of pottery and lithic artifacts. Pretreatment may not be available to mitigate the adverse effects of oil and dispersant in absorbed pottery residue analysis, unless techniques can be established to remove contaminants without destroying the biomarkers of absorbed plant and animal residues. Pretreatments are less likely to be successful when oil and dispersant, or the pretreatment techniques, accelerate material degradation or impair the interpretation of results. Because crude oil presents known health hazards that require safety protocols and precautions (Chin 2011:2–3), field and laboratory research on oiled sites will involve increased cost and time expenditures. A cost estimate analysis for this study conservatively suggests an increase of approximately 21% in the total direct cost for a Phase III investigation of a coastal site affected by an oil spill.

Among the surprising findings of this study was the presence of oil and dispersant at sites where SCAT teams did not record oil on shorelines during the MC252 cleanup response. Absorbed residue analysis detected oil and dispersant in a potsherd from a deeply buried, intact deposit (TU 1, Level 6) at control site 16SB153 on Lake Borgne. Another potsherd that contained dispersant was excavated from a redeposited context (TU 2, Level 1) at the Bayou Sale site (16SMY17), approximately 275 miles (443 km) from the source of the MC252 spill. Because the oil in the sherd from Site 16SB153 was not chemically fingerprinted for a match with MC252, this offers only circumstantial evidence for the extent of the oil spill and extensive use of dispersants during the MC252 cleanup response.

The oil detected in a majority (83%) of samples at the assessed sites was either a possible match for MC252, too weathered for identification, or inconclusive for MC252. Although it may be from other, unidentified spills, the oil has entered archaeological deposits and along with dispersants used in the cleanup response, is now part of the formation processes that encourage or inhibit the *in situ* preservation of information contained within a site. This highlights the need for integrated CRM planning to include the effects of an oil spill on coastal sites, along with shoreline erosion, subsidence and relative sea level rise. The information and recommendations provided by this study should prove useful in this regard.

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# Appendix A: Samples Collected from Eight Sites

\*Special samples indicated by an asterisk; all others are field samples.

# A1. Samples from Bayou Sale (16SMY17)

No.	Provenience	Sample Type	Description
1	Surface	Surface	pottery
2	ST 2, 0-20 cm	ST	0.25 inch dry screen
3	ST 3, 0-42 cm	ST	0.25 inch wet screen
4	ST 5, 0-30 cm	ST	0.25 inch wet screen
5	ST 5, 30-50 cm	ST	0.25 inch wet screen
6	ST 6, 0-20 cm	ST	0.25 inch dry & wet screen
7	ST 6, 20-45 cm	ST	0.25 inch wet screen
8	ST 4, 0-20 cm	ST	0.25 inch wet screen
9	ST 4, 20-40 cm	ST	0.25 inch wet screen
10*	ST 4 at 10 cm	Unit Core	North wall
11*	ST 4, 0-10 cm	Column	North wall
12*	ST 4, at 20 cm	Unit Core	North wall
13*	ST 6, 0-10 cm	Column	NW wall
14*	ST 6, 20-30 cm	Column	NW wall
15*	ST 6, 30-40 cm	Column	NW wall
16*	ST 5 at 20 cm	Unit Core	North wall
17	TU 1, L 1, 0-10 cm	TU	0.25 inch dry screen
18	TU 2, L 1, 0-10 cm	TU	0.25 inch wet screen
19	TU 2, L 2, 10-20 cm	TU	0.25 inch wet screen
20	TU 2, L 3, 20-30 cm	TU	0.25 inch wet screen
21	TU 2, L 4, 30-35 cm	TU	0.25 inch wet screen
22*	TU 2, L 1, 0-10 cm	Column	East wall
23*	TU 2, L 2, 10-20 cm	Column	East wall
24*	TU 2, L 3, 20-30 cm	Column	East wall
25*	TU 2, L 4, 30-38 cm	Column	East wall
26*	TU 2 at 22 cm	Unit Core	North wall
27*	TU 2, L 1, 0-10 cm	Soil	South wall
28*	TU 2, L 2,10-20 cm	Soil	South wall
29*	TU 2, L 3, 20-30 cm	Soil	South wall

# A2. Samples from Cheniere St. Denis (16JE2)

No.	Provenience	Sample Type	Description
1	Surface, Areas A and B	Surface	Pottery
2	TU 1, L 1, 0-10 cm	TU	0.25 inch dry mesh
3*	TU 1, 0-8 cm	Specimen	Pottery
4*	TU 1, L 1, 5-10 cm	Specimen	Fauna, fish bone
5	TU 1, L 2, 10-20 cm	TU	0.25 inch wet screen
6*	TU 1, 10-19 cm, south half	Soil	Soil
7*	TU 1, 15-19 cm	Specimen	Fauna, fish bone
8*	TU 1, 20 cm	Specimen	Pottery
9	TU 2, L 1, 0-15 cm	TU	0.25 inch dry screen
10	TU 2, L 2, 15-20 cm	TU	0.25 inch dry screen
11	TU 2, L 3, 20-30 cm	TU	0.25 inch dry screen
12*	TU 2, 5-9 cm	Soil	Soil
13*	TU 2, 8 cm	Specimen	Pottery
14*	TU 2, 25 cm	Specimen	Pottery
15*	TU 2, 23-26 cm	Specimen	Fauna, bone
16*	TU 2, 26 cm	Specimen	Shell
17	TU 2, L 4, 30-40 cm	TU	0.25 inch dry screen
18*	TU 2, 35 cm	Specimen	Pottery
19	TU 3, L 1, 0-15 cm	TU	0.25 inch dry screen
20	TU 3, L 2, 15-25 cm	TU	0.25 inch wet screen
21*	TU 3, 17-25 cm	Specimen	Shell
22*	TU 3, 17-25 cm	Specimen	Pottery
23*	TU 3, 23-25 cm	Specimen	Shell
24	TU 3, L 3, 25-35 cm	TU	0.25 inch wet screen
25*	TU 3, 30-35 cm	Specimen	Pottery
26*	TU 3, 35 cm	Specimen	Shell
27	TU 3, L 4, 35-45 cm	TU	0.25 inch wet screen
28*	TU 3, 38 cm	Specimen	Lithic
29*	TU 3, 39 cm	Specimen	Ceramic
30	TU 2, L 5, 40-50 cm	TU	0.25 inch dry screen
31*	TU 3, 0-10 cm	Column	North Wall
32*	TU 3, 10-20 cm	Column	North Wall
33*	TU 3, 20-30 cm	Column	North Wall

No.	Provenience	Sample Type	Description
34	TU 4, L 1, 0-10 cm	TU	0.25 inch wet screen
35*	TU 4, 2-8 cm	Soil	Soil
36	TU 4, L 2 10-20 cm	TU	0.25 inch wet screen
37	TU 1, L 3, 20-30 cm	TU	0.25 inch wet screen
38*	TU 1, L 3, 22-30 cm	Soil	Soil
39	TU 2, L 6, 50-60 cm	TU	0.25 inch dry screen
40*	TU 2, 60 cm	Specimen	Pottery
41	TU 2, L 7, 60-70 cm	TU	0.25 inch dry screen
42*	TU 2, L 6, 55-60 cm	Specimen	Fauna, bone
43*	TU 2, L 7, 65-70 cm	Specimen	Pottery
44*	TU 2, L 7, 68-70 cm	Specimen	Lithic
45	TU 2, 70-85 cm	Auger	0.25 inch dry screen
46	TU 2, 85-95 cm	Auger	0.25 inch dry screen
47*	TU 2, 85-95 cm	Auger, specimen	Shell
48*	TU 2, L 1, 4-10 cm	Unit Core	North Wall
49*	TU 2, L 2, 10-20 cm	Column	North Wall
50*	TU 2, L 3, 21-27 cm	Unit Core	North Wall
51*	TU 2, L 3, 28-34 cm	Unit Core	North Wall
52*	TU 2, L 4, 34-40 cm	Unit Core	North Wall
53*	TU 2, 40-50 cm	Column	North Wall
54*	TU 2, 50-60 cm	Column	North Wall
55*	TU 2, 55-65 cm	Column	East Wall
56*	TU 1, 0-10 cm	Column	North Wall
57*	TU 1, 10-20 cm	Column	North Wall
58*	TU 1, 20-28 cm	Column	North Wall
59*	TU 4, 11-18cm	Specimen	Pottery

# A3. Samples from Southern Comfort (16SB178)

No.	Provenience	Sample Type	Description
1	General Surface	Surface	Pottery
2	ST 1, L 1, 0-30 cm	ST	0.25 inch dry screened
3	ST 1, L 2, 30-50 cm	ST	0.25 inch dry screened
4	Auger 1, 0-26 cm	Auger	0.25 inch dry screened
5	ST 2, L 1, 0-11 cm	ST	0.25 inch dry screened

No.	Provenience	Sample Type	Description
6	ST 3, L 1, 0-20 cm	ST	0.25 inch dry screened
7	ST 3, L 2, 20-40 cm	ST	0.25 inch wet screened
8	ST 3, L 3, 40-62 cm	ST	0.25 inch wet screened
9	ST 4, L 1, 0-20 cm	ST	0.25 inch dry screened
10	ST 4, L 2, 20-50 cm	ST	0.25 inch dry screened
11	ST 5, L 1, 0-20 cm	ST	0.25 inch dry screened
12	ST 5, L 2, 20-40 cm	ST	0.25 inch dry screened
13	ST 6, L 1, 0-10 cm	ST	0.25 inch dry screened
14	ST 6, L 2, 0-30 cm	ST	0.25 inch wet screened
15	ST 7, L 1, 0-50 cm	ST	0.25 inch wet screened
16	TU 1, L 1, 0-10 cm	TU	0.25 inch dry screened
17	TU 1, L 2, 10-20 cm	TU	0.25 inch dry screened
18	TU 1, L 3, 20-30 cm	TU	0.25 inch dry screened
19	TU 1, L 4, 30-40 cm	TU	0.25 inch dry screened
20	TU 2, L 1, 0-10 cm	TU	0.25 inch dry screened
21	TU 2, L 2, 10-20 cm	TU	0.25 inch dry screened
22	TU 2, L 3, 20-30 cm	TU	0.25 inch dry screened
23*	TU 1, L 1, 7-10 cm	Soil	North Half
24*	TU 1, L 2, 16-20 cm	Soil	South Half
25*	TU 1, L 3, 35-40 cm	Soil	South Half
26*	Surface, near TU 2	Soil	Soil
27*	TU 2, L 1, 4-10 cm	Soil	Soil
28*	Surface, near TU 1	Specimen	Pottery
29*	Surface, near TU 2	Specimen	Pottery
30*	Surface, near TU 2	Soil	Soil
31*	TU 2, L 1, 0-10 cm	Column	North Wall
32*	TU 2, L 2, 10-20 cm	Column	North Wall
33*	TU 2, L 3, 20-30 cm	Column	North Wall
34*	TU 2, L 4, 30-40 cm	Column	North Wall
35*	TU 2, L 1, 5-10 cm	Specimen	Pottery
36*	Surface, near TU 2	Specimen	Pottery
37*	TU 2, surface of L 1	Specimen	Pottery
38*	TU 1, L 1, 2-10 cm	Unit Core	West Wall
39*	TU 1, L 2, 10-20 cm	Column	West Wall

No.	Provenience	Sample Type	Description
40*	TU 1, L 3, 20-30 cm	Column	West Wall
41*	TU 1, L 4, 30-40 cm	Column	West Wall

# A4. Samples from Comfort Island (16SB174)

No.	Provenience	Sample Type	Description
1	General Surface	Surface	Pottery
2	TU 1, L 1, 0-10 cm	TU	0.25 inch dry screened
3	TU 1, L 2, 10-20 cm	TU	0.25 inch wet screened
4*	TU 1, 0-10 cm	Column	East Wall
5*	TU 1, 10-20 cm	Column	East Wall
6*	TU 1, 20-30 cm	Column	East Wall
7*	TU 1, 20-30 cm	Soil	South Wall
8*	TU 1, 10-20 cm	Soil	West Wall
9	TU 1, L 3, 20-30 cm	TU	0.25 inch dry screened
10	TU 2, L 1, 0-10 cm	TU	0.25 inch dry screened
11	TU 2, L 2, 10-20 cm	TU	0.25 inch dry screened
12*	TU 2, 15-20 cm	Soil	Soil
13	TU 2, L 3, 20-30 cm	TU	0.25 inch dry screened
14*	TU 2, 21-26 cm	Soil	North Half
15*	TU 2, 20-27 cm	Unit Core	North Wall
16*	TU 2, 10-20 cm	Column	North Wall
17*	TU 2, 0-10 cm	Column	North Wall
18*	TU 2, 30-40 cm	Column	West Wall
19*	TU 2, 20-30 cm	Column	East Wall

# A5. Samples from Site 16SB153

No.	Provenience	Sample Type	Description
1	Near Jahnckes Ditch	Surface	Pottery
2	Shell midden SW of TU	Surface	Pottery
3	TU 1, L 1, 75-85 cm	TU	0.25 inch wet screened
4	TU 1, L 2, 85-95 cm	TU	0.25 inch wet screened
5	TU 1, 90-110 cm, SW quad	TU	0.25 inch wet screened

No.	Provenience	Sample Type	Description
6	TU 1, 90-110 cm, SW quad	TU	0.25 inch wet screened
7	TU 1, 110-128 cm, SW quad	TU	0.25 inch wet screened
8	TU 1, L 3, 95-105 cm	TU	0.25 inch wet screened
9*	TU 1, L 3, 95-100 cm, NE quad	Soil	0.25 dry & 1/16 in. wet screen
10*	TU 1, L 4, 107-112 cm, NE quad	Soil	0.25 dry & 0.0625 in. wet screen
11	TU 1, L 4, 105-115 cm	TU	0.25 inch wet screened
12	TU 1, 128-138 cm, SW quad	TU	0.25 inch wet screened
13	TU 1, L 5, 5 115-125 cm	TU	0.25 inch wet screened
14	TU 1, 138-158 cm, SW quad	TU	0.25 inch wet screened
15	TU 1, L 6, 125-135 cm	TU	0.25 inch wet screened
16*	TU 1, 125-132 cm, NE quad	Soil	0.25 dry & 0.0625 in. wet screen
17	TU 1, L 7, 135-145 cm	TU	0.25 inch wet screened
18*	TU 1, L 7, 135-140 cm, NE quad	Soil	0.25 dry & 0.0625 in. wet screen
19	TU 1, 158-178 cm, SW quad	TU	0.25 inch wet screened
20	TU 1, Wall profiles	TU	0.25 inch wet screened
21*	TU 1, West Wall, 120-130 cm	Soil	0.25 dry & 0.0625 in. wet screen
22*	TU 1, 0-5 cm	Column	North Wall
23*	TU 1, 0-10 cm	Column	North Wall
24*	TU 1, 10-20 cm	Column	North Wall
25*	TU 1, 20-30 cm	Column	North Wall
26*	TU 1, 50-60 cm	Column	North Wall
27*	TU 1, 60-70 cm	Column	North Wall
28*	TU 1, 82-90 cm	Column	North Wall
29*	TU 1, 90-100 cm	Column	North Wall
30*	TU 1, 100-110 cm	Column	North Wall
31*	TU 1, 113-123 cm	Column	North Wall
32*	TU 1, 123-130 cm	Column	North Wall
33*	TU 1, 134-139 cm	Unit Core	West Wall

# A6. Samples from Scow Island Scatter (16SB182)

No.	Provenience	Sample Type	Description
1	Area A	Surface	Pottery
2	ST 2, L 1, 15-20 cm	ST	0.25 inch dry screened

No.	Provenience	Sample Type	Description
3	n/a	not collected	n/a
4	ST 5, L 2, 20-30 cm	ST	0.25 inch dry screened
5	ST 4 L 2, 20-40 cm	ST	0.25 inch dry screened
6	TU 1, L 1, 0-10 cm	TU	0.25 inch dry screened
7*	TU 1, 5-10 cm	Specimen	pottery
8	TU 1, L 2, 10-20 cm	TU	0.25 inch dry screened
9*	TU 1, 10-17 cm	Specimen	pottery
10	TU 1, L 3, 20-30 cm	TU	0.25 inch dry screened
11	TU 1, L 3, 20-28 cm	TU	soil sample
12	ST 6, L 1, 0-20cm	ST	0.25 inch dry screened
13	ST 6, L 2, 20-90 cm	ST	0.25 inch dry screened
14	TU 2, L 1, 0-10 cm	TU	0.25 inch dry screened
15	TU 2, L 2, 10-20 cm	TU	0.25 inch dry screened
16*	TU 2, L 2, 19 cm, East Half	Specimen	charcoal
17*	TU 2, L 2, 19 cm, East Half	Specimen	pottery
18*	TU 2, L 2, 19-34 cm, East Half	Unit Core	Floor
19	TU 2, L 3, 20-30 cm	TU	0.25 inch dry screened
20*	TU 2, L 1, 0-10 cm	Column	North Wall
21*	TU 2, L 2, 10-20 cm	Column	North Wall
22*	TU 2, L 3, 20-30 cm	Column	West Wall
23*	TU 1, L 1, 0-10 cm	Column	West Wall
24*	TU 1, L 2, 10-20 cm	Column	West Wall
25*	TU 1, L 3, 20-30 cm	Column	West Wall
26*	TU 1, L 2, 14-20 cm	Unit Core	North Wall
27	Surface, Area B	Surface	Pottery
28	Surface, Area C	Surface	Pottery
29	ST 5, L 2, 35-40 cm	ST	0.25 inch dry screened

# A7. Samples from Redfish Slough (16LF293)

No.	Provenience	Sample Type	Description
1a	Area A	Surface	Pottery
1b	Area B	Surface	Pottery
2	ST 1, L 1, 0-20 cm	ST	0.25 inch dry screen
3	ST 1, L 2, 20-40 cm	ST	0.25 inch dry screen

No.	Provenience	Sample Type	Description
4	ST 1, L 3, 40-60 cm	ST	0.25 inch dry and wet screen
5	ST 3, L 1, 0-20 cm	ST	0.25 inch dry screen
6	ST 3, L 3, 40-60 cm	ST	0.25 inch wet screen
7	ST 4, L 1, 0-20 cm	ST	0.25 inch dry screen
8	ST 5, L 1, 0-20 cm	ST	0.25 inch dry screen
9	ST 5, L 2, 20-40 cm	ST	0.25 inch dry screen
10	ST 4, L 2, 20-40 cm	ST	0.25 inch dry screen
11	ST 5, L 3, 40-60 cm	ST	0.25 inch dry screen
12	ST 6, L 1, 0-20 cm	ST	0.25 inch dry screen
13	ST 6, L 2, 20-40 cm	ST	0.25 inch wet screen
14	ST 7, L 1, 0-22 cm	ST	0.25 inch wet screen
15	ST 7, L 3, 40-60 cm	ST	0.25 inch wet screen
16	TU 1, L 1, 0-10 cm	TU	0.25 inch dry screen
17	TU 1, L 2, 10-20 cm	TU	0.25 inch dry screen
18*	TU 1, 10 cm	Specimen	Pottery
19*	TU 1, 10-14 cm	Specimen	Pottery
20*	TU 1, 16 cm	Specimen	Fauna, bone
21	TU 1, L 3, 20-30 cm	TU	0.25 inch dry screen
22	TU 1, L 4, 30-40 cm	TU	0.25 inch dry screen
23*	TU 1, 25-30 cm	Specimen	Pottery
24*	TU 1, 0-10 cm	Column	North Wall
25*	TU 1, 10-20 cm	Column	North Wall
26*	TU 1, 20-30 cm	Column	East Wall
27*	TU 1, 30-40 cm	Column	East Wall
28	ST 8, L 1, 0-20 cm	ST	0.25 inch dry screen
29	ST 8, L 2, 20-40 cm	ST	0.25 inch dry screen
30	ST 8, L 3, 40-50 cm	ST	0.25 inch dry screen
31	ST 9, L 1, 0-20 cm	ST	0.25 inch dry screen
32	ST 9, L 2, 20-40 cm	ST	0.25 inch dry and wet screen
33	TU 2, L 1, 0-10 cm	TU	0.25 inch dry screen
34	TU 2, L 2, 10-20 cm	TU	0.25 inch dry screen
35	TU 2, L 3, 20-30 cm	TU	0.25 inch dry screen
36	TU 2, L 4, 30-40 cm	TU	0.25 inch dry screen
37	TU 2, L 5, 40-50 cm	TU	0.25 inch dry screen

No.	Provenience	Sample Type	Description
38*	TU 2, 36 cm	Specimen	Fauna, bone
39*	TU 1, 10-20 cm	Column	North Wall
40*	TU 2, 22-30 cm	Soil	Soil
41*	TU 2, 30-40 cm	Soil	Soil
42*	TU 2, 45-50 cm	Soil	Soil
43*	TU 2, 17-23 cm	Unit Core	West Wall
44*	TU 2, 0-10 cm	Column	North Wall
45*	TU 2, 10-20 cm	Column	South Wall
46*	TU 2, 20-30 cm	Column	South Wall
47*	TU 2, 30-40 cm	Column	South Wall
48*	TU 2, 40-50 cm	Column	South Wall
50*	TU 1, L 4, 35-40 cm	Specimen	Pottery

# A8. Samples from Acorn Mounds (16SB185)

No.	Provenience	Sample Type	Description
1	Area "A" near shoreline	Surface	Artifacts
2*	Area "A" near shoreline	Surface	Soil and tar balls
3*	Area "A" near shoreline	Surface	Soil and tar
4*	ST 2, L1, 20 cm	Soil	Soil and tar ball
5	TU 1, L 1, 82-92 cm	TU	0.25 inch dry screened
6	TU 1, 97-130 cm	TU	0.25 inch dry screened
7	TU 1, L 2, 95-105 cm	TU	0.25 inch dry screened
8*	TU 1, L3 105-115 cm	Soil	SW
9*	TU 1, L6 135-145 cm	Soil	NW
10*	TU 1, L4, 115-125 cm	Soil	SW
11*	TU 1, 0-10 cm	Column	West wall
12*	TU 1, 30-40 cm	Column	West wall
13*	TU 1, 65-75 cm	Column	West wall
14*	TU 1, 75-85 cm	Column	East wall
15*	TU 1, 50-56 cm	Unit core	South wall
16*	TU 1, 87-93 cm	Unit core	West wall
17*	AT 2, L1, 0-10 cm	AT	Soil
18*	AT 2, L3, 45-55 cm	AT	Soil
19*	AT 2, L4, 55-65 cm	AT	Soil

No.	Provenience	Sample Type	Description
20*	AT 2, L5, 70-78 cm	AT	Soil
21*	AT 2, L6, 80-95 cm	AT	shell and soil
22	TU 2, L 1, 0-10 cm	TU	0.25 inch dry screened
23	TU 2, L 2, 10-20 cm	TU	0.25 inch dry screened
24*	TU 2, L 2, 16-20 cm	Soil	Soil
25*	TU 2, L 3, 20-25 cm	Soil	Soil
26	AT 4, 0-175 cm	AT	0.25 inch dry screened
27*	CT 1, CS 1, 0-30 cm	Core	N flank of Md B
28*	CT 2, CS 1, 0-28 cm	Core	N flank of Md B
29*	CT 2, CS 2, 75-105 cm	Core	N flank of Md B
30*	CT 2, CS 3, 125-152 cm	Core	N flank of Md B
31*	CT 2, CS 4, 152-180 cm	Core	N flank of Md B
32*	CT 2, CS 5, 210-239 cm	Core	N flank of Md B
33*	CT 3, CS 1, 0-30 cm	Core	Md B summit
34*	CT 4, CS 1, 180-216 cm	Core	Md B summit; bottom of AT 4
35*	CT 5, CS 1, 0-33 cm	Core	N of Md B
36*	CT 5, CS 2, 114-141 cm	Core	N of Md B
37*	CT 5, CS 3, 195-226 cm	Core	N of Md B
38*	CT 5, CS 4, 225-340 cm	Core	N of Md B
39*	CT 6, CS 1, 0-30 cm	Core	N of Md B, S of channel
40*	CT 7, CS 1, 0-30 cm	Core	W of Keelboat Pass
41*	CT 7, CS 2, 93-123 cm	Core	W of Keelboat Pass
42*	CT 7, CS 3, 161-192 cm	Core	W of Keelboat Pass
43*	CT 7, CS 4, 280-310 cm	Core	W of Keelboat Pass

# Appendix B: Samples Submitted for Analysis

Site	No.	ANID	Provenience	Sample	Consultant	Technique	Result
16JE2	21	JEB021	TU3 L2, 17-25 cm, specimen sample	Rangia and soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16JE2	22	JEB022	TU3 L2 at 17-25cm, specimen sample	Baytown Plain sherd	Residue Lab	GC/MS	Small amount of oil; Difficult to interpret; meat and plant lipids
16JE2	24	JEB024	TU3 L2 at 25-35 cm, field sample	Bone (unid mammal)	Beta Analytic	AMS	No statistically significant difference between pretreatment.
16JE2	24	JEBA024	TU3 L2 at 25-35 cm, field sample	Bone (unid mammal)	Beta Analytic	AMS	Used to supplement JEB024
16JE2	25	JEB025	TU3 L3, 30-35cm, specimen sample	Baytown Plain sherd	Residue Lab	GC/MS	Dispersant; not easily interpretable; small amount of mixed plant and animal based lipid:
16JE2	26	JEB026	TU3 W half, L3, at 35cm, specimen sample	Rangia and soil	DES, LSU	GC/MS	Oil detected; inconclusive for MC252 oil
16JE2	29	JEB029	TU3 L4 at 39cm, east half	Baytown Plain sherd	Residue Lab	GC/MS	No contaminants, insufficient residue for interpretation
16JE2	33	JEB033	TU3 N wall, 20-30 cm, column sample	soil	DES, LSU	GC/MS	Negative
16JE2	35	JEB035	TU4 L1, 2-8cm	soil	DES, LSU	GC/MS	Negative
16JE2	43	JEB043	TU2 L 7 at 65-70cm, specimen sample	Grog tempered sherd	Residue Lab	GC/MS	Hand cream or surfactant; difficult to interpret; primarily plan lipids with a small amount of fish/shellfish
16JE2	53	JEB053	TU2 N wall, 40-50cm column sample	soil	DES, LSU	GC/MS	Negative
16JE2	55	JEB055	TU2 E wall, L 6-7, 55- 65 cm, column sample	soil	DES, LSU	GC/MS	Negative
16JE2	59	JEB059	TU4 L 2 at 11-18cm, specimen sample	Baytown Plain sherd	Residue Lab	GC/MS	No contaminants, insufficient residue for interpretation
16LF293	18	LFG018	TU1 at 10cm , specimen sample	Baytown Plain sherd	Residue Lab	GC/MS	Oil contamination; Primarily terrestrial plant, with small amount of animal lipids
16LF293	20	LFG020	TU1 at 16 cm, specimen sample	Bone ( <i>Ondatra</i> <i>Zibethicus</i> , femur)	Beta Analytic	AMS	Failed collagen extraction
16LF293	24	LFG024	TU1 L1, N wall, 0-10 cm column sample	Soil	DES, LSU	GC/MS	Negative
16LF293	34	LFG034	TU2 L2 10-20cm, field sample	Baytown Plain sherd	Residue Lab	GC/MS	Dispersant contamination; Primarily terrestrial plants, with small amount of animal lipids
16LF293	38	LFG038	TU2 at 36 cm, specimen sample	Bone (Mississippi ensis, jugal)		AMS	Failed collagen extraction
16LF293	40	LFGA040	TU2 L3, E half, 22-30 cm	Soil	DES, LSU	GC/MS	Negative
16LF293	45	LFG045	TU2 L2, S wall, 10-20 cm column sample	Soil	DES, LSU	GC/MS	Positive; possible match for MC252 oil

Site	No.	ANID	Provenience	Sample	Consultant	Technique	Result
16LF293	47	LFG047	TU2 L 4 at 30-40cm S wall, column sample	Grog tempered sherd	Residue Lab	GC/MS	Dispersant contamination; Difficult to interpret, probably a mixture of animal and plants, possibly including marine algal lipids
16LF293	48	LFG048	TU2 L5, S wall, 40-50 cm column sample		DES, LSU	GC/MS	Negative
16SB153	15	SBE015	TU1 L6 at 125-135cm, field sample	Shell tempered sherd	Residue Lab	GC/MS	Oil and dispersant contamination; mixture of animal and terrestria plants
16SB153	17	SBE017	TU1 L7, 135-145 cm, field sample	1 grog and shell- tempered sherd	MURR	ICP-MS	No effect
16SB153	23	SBE023	TU1 N wall, 0-10 cm column sample	Soil	DES, LSU	GC/MS	Negative
16SB153	33	SBE033	TU1 at 134-139 cm W wall, core sample	Bone (unid fragments)	Beta Analytic	AMS	No statistically significant difference between pretreatment
16SB153	33	SBEA033	TU1 at 134-139 cm W wall, core sample	Bone (unid fragments)	Beta Analytic	AMS	No statistically significant difference between pretreatment
16SB174	6	SBD006	TU1 L3, E wall, 20- 30cm column sample	Soil	DES, LSU	GC/MS	Negative
16SB174	10	SBD010	TU2 L1, 0-10cm, specimen sample	Grog tempered, burnished	Residue Lab	GC/MS	No contaminants; Mixture of plant and meat
16SB174	11	SBDA011	TU2 L2, 10-20cm, field sample	1 grog- tempered sherd	MURR	ICP-MS	No effect
16SB174	11	SBDB011	TU2 L2, 10-20 cm, field sample	1 grog- tempered sherd	MURR	ICP-MS	No effect
16SB174	12	SBD012	TU2 L2 15-20cm, sediment sample	1 grog- tempered sherd	MURR	ICP-MS	No effect
16SB174	12	SBDA012	TU2 L2 at 15-20cm, sediment sample	Wood charcoal	Beta Analytic	AMS	No statistical significan difference between pretreatment
16SB174	15	SBDA015	TU2 L3 N wall, 20-27 cm core sample	Soil	DES, LSU	GC/MS	Positive; possible match for MC252 oil
16SB174	16	SBD016	TU2 L2, N wall, 10-20 cm column sample	Soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16SB178	33	SBC033	TU2 L3, N wall, 20-30 cm column sample	Soil	DES, LSU	GC/MS	Negative
16SB178	26	SBC026	Collected near TU2	Soil	DES, LSU	GC/MS	Positive; possible match for MC252 oil
16SB178	31	SBC031	TU2 L1, N wall, 0-10 cm column sample	Soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16SB178	35	SBC035	TU2 L1, 5-10 cm, specimen sample	1 grog- tempered sherd	MURR	NAA	Elevated levels of Arsenic
16SB178	32	SBC032	TU2 L2, N wall, 10-20 cm column sample	soil	DES, LSU	GC/MS	Negative
16SB178	28	SBC028	Near TU1, specimen sample	Grog tempered sherd	Residue Lab	GC/MS	No contaminants; difficult to interpret; algal lipids, with plants lipids
16SB178	37	SBC037	TU2 L1, surface, specimen sample	Grog tempered sherd	Residue Lab	GC/MS	Oil and dispersant; Mixture of lipids from animals and terrestrial plants

Site	No.	ANID	Provenience	Sample	Consultant	Technique	Result
16SB178	37	SBCA037	TU2 L1, surface, specimen sample	Baytown Plain	Residue Lab	GC/MS	No contaminants; Shellfish, plant lipids and perhaps meat
16SB178	29	SBC029	Near TU2, specimen sample	Baytown Plain	Residue Lab	GC/MS	Not interpretable due to oil and dispersant contamination
16SB182	7	SBF007	TU1 L1 at 5-10 cm, specimen sample	Grog tempered sherd	Residue Lab	GC/MS	No contaminants; Difficult to interpret; primarily plant/fish, with animal lipids
16SB182	10	SBF010	TU1 L3 at 20-30cm, field sample	Bone (unid fragment)	Beta Analytic	AMS	Produced historic or possibly modern dates
16SB182	16	SBF016	TU2 L2 at 19cm, E half, specimen sample	Wood charcoal	Beta Analytic	AMS	No statistically significant difference between pretreatment
16SB182	17	SBF017	TU2 L2 at 19 cm, E half, specimen sample	Baytown Plain sherd	Residue Lab	GC/MS	No contaminants; Not interpretable
16SB182	18	SBF018	TU2 E half, 19-34 cm, core sample	soil	DES, LSU	GC/MS	Negative
16SB182	21	SBF021	TU2 L2 N wall, 10-20 cm, column sample	soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16SB182	23	SBF023	TU1 L1, W wall, 0-10 column sample	soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16SB185	2	SBH002	Surface collected from Area A	Tar ball	DES, LSU	GC/MS	Positive match for MC252 oil
16SB185	4	SBH004	ST2 L1, N wall, 20 cm	Tar ball	DES, LSU	GC/MS	Positive match for MC252 oil
16SB185	11	SBH011	TU 1 W wall, 0-10 cm column sample	soil	DES, LSU	GC/MS	Negative
16SB185	27	SBH027	CT 1, 0-30 cm, core sample 1	soil	DES, LSU	GC/MS	Oil detected; too weathered for fingerprinting
16SB185	34	SBHA034	Mound B, CT4 at 210- 216cm, core sample	Wood charcoal	Beta Analytic	AMS	No statistically significant difference between pretreatment
16SB185	34	SBH034	Mound B, CT4 at 195- 199cm, core sample	Bone (unid fragments)	Beta Analytic	AMS	Failed collagen extraction
16SB185	35	SBH035	CT5, 0-33 cm, core sample 1	soil	DES, LSU	GC/MS	Negative
16SB185	39	SBH039	CT6, 0-30 cm, core sample 1	soil	DES, LSU	GC/MS	Negative
16SMY17	18	SYA018	TU2 L1 at 0-10cm, field sample	Baytown Plain sherd	Residue Lab	GC/MS	Dispersant, DEET and fragrance contaminated; mixture of meat and terrestrial plants
16SMY17	21	SYA021	TU2 L4 at 30-35 cm, field sample	Bone, unid. turtle	Beta Analytic	AMS	No statistically significant difference between pretreatment
16SMY17	21	SYAA021	TU2 L4 at 30-35 cm, field sample	Bone, unid. turtle	Beta Analytic	AMS	A statistically significar difference between pretreatment
16SMY17	21	SYAB021	TU2 L4 at 30-35 cm, field sample	Bone, unid. turtle	Beta Analytic	AMS	Completed, supplemented SYA021
16SMY17	21	SYAC021	TU2 L4 at 30-35 cm, field sample	Bone, unid. turtle	Beta Analytic	AMS	Completed, supplemented SYAA021
16SMY17	25	SYA025	TU2 30-38 cm E wall, column sample	soil	DES, LSU	GC/MS	Negative

# Appendix C. Chemical Characterization and Oil Source Fingerprinting

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#### **C.1** Introduction

The following appendix details the extraction, instrumental, and data processing methodologies used by LSU-RCAT to determine concentrations of oil analytes of interest (Table 1) and for qualitative and quantitative oil source fingerprinting of samples collected for the University of Louisiana–Lafayette (ULL)-Bureau of Ocean Energy Management (BOEM) project. Table 1 includes compounds that are typically petrogenic (i.e., oil derived) in origin and include parent aromatic hydrocarbons and their alkyl homologs, saturate compounds from n-C<sub>10</sub> to n-C<sub>35</sub>, isoprenoids pristane and phytane, and four groups of oil "biomarkers". The oil biomarkers are routinely used for oil source fingerprinting and include the triterpanes (including hopanes), diasteranes and regular steranes,  $14\beta(H)$ -steranes, and the triaromatic steroids.

Table C1. Targeted Petroleum Hydrocarbon Analytes

Anthracene	Fluoranthene	C-1 Phenanthrenes/Anthracenes
Benz[a]anthracene	Fluorene	C-2 Phenanthrenes/Anthracenes
Benzo[a]pyrene	C-1 Fluorenes	C-3 Phenanthrenes/Anthracenes
Benzo[b]fluorene	C-2 Fluorenes	C-4 Phenanthrenes/Anthracenes
Benzo[e]pyrene	C-3 Fluorenes	Pyrene
Benzo[g,h,i]perylene	Indeno[1,2,3-cd]pyrene	C-1 Fluoranthenes/Pyrenes
Benzo[k]fluorene	Naphthalene	C-2 Fluoranthenes/Pyrenes
Chrysene	C-1 Naphthalenes	C-3 Fluoranthenes/Pyrenes
C-1 Chrysenes	C-2 Naphthalenes	C-4 Fluoranthenes/Pyrenes
C-2 Chrysenes	C-3 Naphthalenes	Saturate Hydrocarbons:
C-3 Chrysenes	C-4 Naphthalenes	nC <sub>10</sub> -nC <sub>35</sub>
C-4 Chrysenes	Naphthobenzothiophene (NBT)	Oil Biomarkers:
Dibenz[a,h]anthracene	C-1 NBTs	Triterpanes (m/z 191)
Dibenzothiophene (DBT)	C-2 NBTs	Diasteranes & Regular Steranes (m/z 217)
C-1 DBTs	C-3 NBTs	14β(H) Steranes ( <i>m/z</i> 218)
C-2 DBTs	Perylene	Triaromatic Steroids (m/z 231)
C-3 DBTs	Phenanthrene	

### C.2 Methodology

#### C.2.1 Solvents

Only pesticide/reagent grade solvents (>99.9%) are used in all analytical standard preparations, sample analyses and dish washing procedures. All standards described below are part of LSU-RCAT's routine quality assurance/quality control (QA/QC) procedures.

### **C.2.2 Analytical Standards**

A commercially-prepared oil analysis standard (Absolute Standards, Hamden, CT) was used to prepare a five-point calibration curve. The oil analysis standard contained normal alkanes (n-C<sub>10</sub> through n-C<sub>35</sub>) and parent polycyclic aromatic hydrocarbons. A continuing calibration standard (one point of this initial five-point calibration curve) was analyzed in each batch of samples, or each 12-hour period during which analyses were performed. The acceptance criterion for the continuing calibration standard is  $\pm 20\%$  of the average relative response factor calculated from the initial five-point curve. If the acceptance criterion was not met, all analyses were stopped until the instrument was performing at optimum conditions.

The surrogate standards were 5-alpha androstane (alkanes) and phenanthrene- $d_{10}$  (aromatics). The surrogate standards were purchased from Accustandard, Inc. (New Haven, CT) and stored individually until they were mixed to a final concentration of 200  $\mu$ g mL<sup>-1</sup>. Surrogate standards were added to samples prior to extraction and were used to evaluate the extraction efficiency. Extraction efficiency for each sample was evaluated based on the percent recovery of the surrogate standard. The acceptable percent recovery range was 70–120%.

The internal standards were naphthalene- $d_8$ , acenaphthene- $d_{10}$ , chrysene- $d_{10}$ , and perylene- $d_{12}$ . The internal standards were bought (Accustandard, Inc., New Haven, CT) and stored individually until they were mixed to make the internal standard solution with a final concentration of 1000  $\mu$ g mL<sup>-1</sup>. Internal standard (10 ng  $\mu$ L<sup>-1</sup>) was added to each sample extract just before analysis, and was used for quantitating compounds listed in Table 1.

The source oil used for these analyses and oil source fingerprinting was Mississippi Canyon 252 (MC252), collected by BP through a riser vent pipe from the damaged wellhead of the *Deepwater Horizon* drilling rig on May 20, 2010. MC252 oil standards are prepared by extracting 1 gram of pure oil in 40 mL of hexane (or equivalent ratio of 1 g:40 mL, e.g. 0.50 g:20 mL). The MC252 source oil extract was analyzed in each sample batch as an additional QA/QC sample (a laboratory control sample).

#### **C.2.3 Sample Extraction Procedures**

Sediment and/or soil samples were homogenized by vigorous stirring then sub-sampled for analysis. The amount of sample extracted was dependent on the amount available and ranged from approximately 10 to 50 g. The sample was weighed to the nearest 0.01 g into a pre-cleaned 500-mL beaker. The sample was spiked with 1 mL of surrogate standard, and then pre-cleaned, granular, anhydrous sodium sulfate was added and mixed into the sample until a "dry" sand-like matrix was created. The samples were extracted using a modified EPA SW-846 Method 3550B, Ultrasonic Extraction. At the completion of the extraction procedure the extraction solvent was concentrated to 1 to 2 mL by a combination of rotary evaporation and nitrogen blowdown. Final extracts were transferred with a clean graduated, gas-tight syringe into a 2 mL autosampler vial. Internal standard was added, the vial was capped, and the sample was then ready for analysis.

Sample results were calculated based on dry weight by determining the moisture of each individual sample. A portion of the sediment and/or soil sample was prepared for drying in an oven overnight. To determine the dry weight of a sample, 5 to 10 g of sample was weighed in a pre-weighed aluminum weigh boat. The weigh boat with the sample was placed in a 105°C oven overnight. The sample was then

removed and allowed to cool in a desiccator before determining the final, oven-dried weight of the sample. Percent moisture was then calculated.

In some instances, samples were analyzed as "fingerprint only". In this case, samples were extracted in the same manner as described above, the only difference for "fingerprint only" samples is that no internal standard was added to the extract before analysis. As a result, concentrations of target analytes cannot be reported. Analyzing samples as fingerprint only does not exclude them from oil source fingerprinting because these techniques rely on other quantitative data for analysis.

### C.2.4 Instrumental Analysis

#### C.2.4.1 GC Operation

Chemical characterization of all samples was carried out using gas chromatography/mass spectrometry (GC/MS) operated in selected ion monitoring (SIM). The GC/MS methodology has been developed specifically for detection and quantifying compounds that are commonly associated with contamination from oil spills. Chemical characterization of samples was performed using an Agilent 7890 GC equipped with an Agilent 5975 inert XL MSD or an Agilent 6890 GC equipped with an Agilent 5973 MSD. Both instrument systems were fitted a 5% diphenyl/95% dimethyl polysiloxane high-resolution capillary column (Zebron-5MSi, 30 m x 0.25 mm x 0.25  $\mu$ m). Instrumental acquisition was identical for both instruments and QA/QC assured that data was comparable between both systems.

The carrier gas was ultrahigh purity helium (AirGas, Radnor, PA) at a constant flow rate of 1 mL min<sup>-1</sup>. An Agilent 7683B or Agilent 7693 autosampler was used for making splitless injections. The injector port was set at 280°C and was fitted with an Agilent deactivated borosilicate liner. The oven temperature program was as follows: the initial temperature was set to 60°C and was held for 3 minutes; the temperature was then increased to 280°C at a rate of 5°C min<sup>-1</sup> and held for 3 minutes. The oven was then heated from 280°C to 300°C at a rate of 1.5°C per minute and held at 300°C for two minutes.

#### C.2.4.2 MS Operation

The MS is operated in the selected ion monitoring (SIM) to maximize the detection of several trace target constituents unique to crude oil. The instrument is operated such that the selected ions for each acquisition window are scanned at a rate greater than 1.5 scans/sec with a dwell time of 60 milli-seconds. The temperature of the MSD interface to MS was set at 300°C. The mass spectrometer had an ion source temperature of 230°C, quadrupole temperature of 150°C, and ionization energy of 70 eV. At the start of each analysis period, or every twelve hours, the MS was tuned to PFTBA, an internal instrument standard. Laboratory reference standards such as a source oil and a continuing calibration standard were also analyzed with each sample batch as part of the QA/QC procedures.

#### C.2.5 Data Analysis

#### C.2.5.1 Quantitative Analysis

GC/MS data was processed by Chemstation<sup>TM</sup> Software using a customized data analysis method developed by LSU-RCAT. The customized data processing method creates a custom report that contains the raw integration data that was exported to a spreadsheet for quantitative analysis. Integration results for each data file were carefully reviewed and reintegrated as required. In addition to the raw integration data, a macro printout is also generated and contains the extracted ion chromatography data, or oil fingerprints, to be qualitatively compared to the source oil.

Quantitative analysis of target compounds was performed using average response factors calculated from the five point oil analysis calibration curve. Alkylated PAH homologs (C-1 through C-4) were quantified using the response factors generated from the unsubstituted parent PAH (C-0) compounds as alkylated homolog standards were not commercially available for all targeted compounds of interest. Normal alkane concentrations were reported as  $\mu g/g$  (i.e., parts per million), and aromatic concentrations were

reported as ng/g (i.e., parts per billion). All individual analyte concentrations were surrogate corrected and reported as a function of dry weight. The final results of the quantitative analysis are reported at three significant figures.

### C.2.5.2 Oil Source Fingerprinting

Oil source fingerprinting is an environmental forensics technique that was originally adapted from the field of petroleum geochemistry. Oil source fingerprinting uses analytical chemistry to compare samples that contain recognizable oil profiles to a suspected source. Compounds known as oil biomarkers are targeted in all oil source fingerprinting techniques because the relative distribution of these compounds are unique for different types and blends of petroleum products and source oils, and, as a result, provide unique chemical fingerprinting information that can distinguish one oil from another, including oils with similar geographic origins (Daling et al., 2002; Hansen et al., 2007; Peters et al., 2005; Stout et al., 2002; Wang and Fingas, 1995; Wang and Fingas, 2003; Wang et al., 2006).

All ULL-BOEM samples underwent three oil source fingerprinting techniques: qualitative comparison, diagnostic biomarker ratio analysis, and chemometrics. The goal of all three techniques was to determine whether any oil detected in a sample was *Deepwater Horizon* oil (MC252 oil).

#### C.2.5.3 Qualitative Comparison

GC/MS oil biomarker profiles for each sample analyzed were visually compared to the same *Deepwater Horizon* oil (MC252 oil) profiles. This visual, or qualitative, comparison was initially performed as outlined by ASTM 5739-00 (ASTM, 2000). All qualitative comparisons considered the possibility of MC252 oil biomarker weathering that likely occurs in a predictable pattern as outlined in Meyer et al. (2017).

#### C.2.5.4 Diagnostic Biomarker Ratio Analysis

Diagnostic biomarker ratio analysis of the ULL-BOEM samples was accomplished by identifying and integrating peak heights of specific triterpanes, steranes, and triaromatic steroids (Tables 2 and 3) in the GC/MS data. This method of quantitative oil source fingerprinting was adapted from the Center for European Norms (CEN) methodology (Hansen et al., 2007; CEN, 2012). Peak heights were then used to calculate diagnostic ratios for each sample and were compared to the same ratios that are specific to MC252 source oil. All the MC252 source oil diagnostic ratios chosen had a percent relative standard deviation (%RSD) less than 5%, a quality criterion set forth in the CEN method. The sample ratios were then statistically compared to the average (n=15) MC252 source oil ratios using the critical difference (CD) method. The CD between a sample ratio and the MC252 ratio cannot be more than 14%, which represents the repeatability limit at a 95% confidence interval ( $\mathbf{r}_{95\%}$ ). Repeatability, in terms of diagnostic biomarker ratio analysis, is used to compare individual diagnostic biomarker ratios with the assumption that oil in the unknown sample is the same as the source oil in question. Once the CD was calculated for each ratio, each sample was given a final oil source fingerprinting score by dividing the number of "matching" ratios by 23 and multiplying by 100 to yield a percentage. Oil source fingerprinting categories were determined by the final ratio score and are given below:

- 87-100 = Match between the sample and MC252 oil
- 79-86 = Probable match between the sample and MC252 oil
- <79% = Inconclusive between the sample and MC252 oil

These categories were established by analyzing both fresh and weathered MC252 crude oil that provided information on how weathering affected the diagnostic biomarker ratio results specifically for MC252 oil.

#### C.2.5.5 Chemometric Analysis

Chemometrics is an exploratory data analysis technique that recognizes patterns using multivariate pattern recognition algorithms and classifies samples into related groupings, or clusters (Peters et al., 2005; Peters et al., 2007; Peters et al., 2008; Lorenson et al., 2011; Peters et al., 2013). The two most common chemometric analyses are hierarchical cluster analysis (HCA) and principal component analysis (PCA).

The extracted ion chromatograms (EIC) of the triterpanes (m/z 191), the diasteranes and regular steranes (m/z 217), the 14 $\beta$ (H)-steranes (m/z 218), and the triaromatic steroids (m/z 231) of each sample and MC252 source oil analyzed within the same sample batch were converted into peak intensity data points recorded approximately every second within a limited time window for each respective biomarker ion group. For example, the conversion of the GC/MS data of the diasteranes and regular steranes (m/z 217) from 42.5-52 minutes into peak intensity data resulted in 638 data points that were used as variables in the chemometric analysis. The peak intensity data were then transferred into the chemometric software package Pirouette® (Infometrix, Bothell, WA) for subsequent HCA and PCA analysis. The HCA and PCA analyses graphically display samples in clusters based on a similarity matrix and accentuates the relationship among the samples. Samples that cluster with MC252 oil in the HCA and PCA analyses are considered to be "genetically" similar to each other. The HCA and PCA analyses are also useful for displaying samples that are outliers (dissimilar) to all other samples.

### C.3 Results and Discussion

A total of 28 samples were analyzed, with three out of the 28 analyzed as fingerprint only (i.e., quantitative concentrations of target analytes were not calculated). Results of the GC/MS analysis and oil source fingerprinting of all 28 samples are given in Table 4. Of the 28 samples analyzed, two were a match to *Deepwater Horizon* oil (MC252 oil) based on all three oil source fingerprinting techniques (i.e., qualitative comparison, diagnostic biomarker ratio analysis, and chemometrics). Three of the samples were considered to be a possible match to MC252 oil. Oil biomarkers are typically considered to be resistant to environmental weathering processes (i.e., evaporation, photo-oxidation, microbial degradation); however, analysis of Louisiana coastal marsh surface sediments collected from 2010 to 2017 have indicated that environmental weathering can profoundly affect these compounds. This is evident in six samples in Table 4 where oil was detected, but the oil residue was too weathered to make any oil source fingerprinting assertions.

The samples that were a match to MC252 oil had target alkane concentrations between 76 and 100 parts per million, and PAH concentrations between 20 and 26 parts per billion. The higher alkane concentrations, compared to all the other samples, indicate that weathering had occurred but not to an extent that adversely effected the oil source fingerprinting outcome. It should be noted that the oil source fingerprinting results presented in Table 4 can be interpreted as individual results (i.e., based on the qualitative technique alone, 5 samples would be a match to MC252 oil). Using the results of all three techniques results in a more conservative approach to oil source fingerprinting; however, if all three techniques indicate a match or possible match to *Deepwater Horizon* oil the confidence level in this result is higher than using just one technique.

Table C2. List of Common Oil Biomarker Compounds Used for Oil Source Fingerprinting

Abbreviation	Name	m/z	Abbreviation	Name	m/z
TC28R	C28 Tricyclic triterpene-22R	191	C27dBaS	13β(H),17α(H)-Diacholestane-20S (Diasterane)	217
TC28S	C28 Tricyclic triterpene-22S	191	C27dBaR	13β(H),17α(H)-Diacholestane-20R (Diasterane)	217
TC29R	C29 Tricyclic triterpene-22R	191	C27aaS	5α(H),14α(H),17α(H)-Cholestane- 20S	217
TC29S	C29 Tricyclic triterpene-22S	191	C29DBaS	13β(H),17α(H)-Ethyldiacholestane- 20S	217
C27Ts	C27 18α(H)-22,29,30- Trisnorhopane	191	C27aaR	5α(H),14α(H),17α(H)-Cholestane- 20R	217
C27Tm	C27 17α(H)-22,29,30- Trisnorhopane	191	C29DBaR	13β(H),17α(H)-Ethyldiacholestane- 20R	217
C28aB	C28 17α(H),21β(H)-28,30- Bisnorhopane	191	C28aaaS	24-methyl-5α(H),14α(H),17α(H)- Cholestane-20S	217
C25nor	C29 17α(H),21β(H)-25- Norhopane	191	C28BBS	24-methyl-5β(H),14β(H),17β(H)- Cholestane-20S	217
C29aB	C29 17α(H),21β(H)-30- Norhopane	191	C28BBR	24-methyl-5α(H),14β(H),17β(H)- Cholestane-20R	217
C29Ts	C29 18α(H)-30- Norneohopane	191	C28aaaR	24-methyl-5α(H),14α(H),17α(H)- Cholestane-20R	217
C30d	C30 15α-methyl-17α(H)-27- Norhopane (diahopane)	191	C29aaaS	24-ethyl-5α(H),14α(H),17α(H),24- Cholestane-20S	217
C29Ba	C29 17β(H),21α(H)- Norhopane (normoretane)	191	C29BBR	24-ethyl-5α(H),14β(H),17β(H)- Cholestane-20R	217
C30 O	C30 18α(H)- and 18β(H)- Oleanane	191	C29BBS	24-ethyl-5α(H),14β(H),17β(H)- Cholestane-20S	217
C30aB	C30 17α(H),21β(H)-Hopane	191	C29aaaR	24-ethyl-5α(H),14α(H),17α(H)- Cholestane-20R	217
C30Ba	C30 17β(H),21α(H)-Hopane (moretane)	191	C27BBR	5α(H),14β(H),17β(H)-Cholestane- 20R	218
C31aBS	C31 17α(H),21β(H)- Homohopane-22S	191	C27BBS	5α(H),14β(H),17β(H)-Cholestane- 20S	218
C31aBR	C31 17α(H),21β(H)- Homohopane-22R	191	C28BBR	24-methyl-5α(H),14β(H),17β(H)- Cholestane-20R	218
C30G	C30 Gammacerane	191	C28BBS	24-methyl-5α(H),14β(H),17β(H)- Cholestane-20S	218
C32aBS	C32 17α(H),21β(H)- Bishomohopane-22S	191	C29BBR	24-ethyl-5α(H),14β(H),17β(H)- Cholestane-20R	218
C32aBR	C32 17α(H),21β(H)- Bishomohopane-22R	191	C29bBS	24-ethyl-5α(H),14β(H),17β(H)- Cholestane-20S	218
C33aBS	C33 17α(H),21β(H)- Trihomohopane-22S	191	C20TA	C20 Triaromatic steroid (Pregnane)	231
C33aBR	C33 17α(H),21β(H)- Trihomohopane-22R	191	C21TA	C21 Triaromatic steroid (Homopregnane)	231
C34aBS	C34 17α(H),21β(H)- Tetrahomohopane-22S	191	SC26TA	C26 20S-Triaromatic Steroid (Cholestane)	231
C34aBR	C34 17α(H),21β(H)- Tetrahomohopane-22R	191	RC26TA+SC2 7TA	C26 20R- + C27 20S-Triaromatic steroids	231

Abbreviation	Name	m/z	Abbreviation	Name	m/z
C35aBS	C35 17α(H),21β(H)- Pentahomohopane-22S	191	SC28TA	C28 Triaromatic steroid-20S (Ethylcholestane)	231
C35aBR	C35 17α(H),21β(H)- Pentahomohopane-22R	191	RC27TA	C27 Triaromatic steroid-20R (Methylcholestane)	231
			RC28TA	C28 Triaromatic steroid-20R (Ethylcholestane)	231

Table C3. List of MC252 Diagnostic Ratios used for Oil Source Fingerprinting

Class	Diagnostic Ratio*
	C27Ts/C27Tm
	C29aB/C29Ts
Tri- and Pentacyclic Triterpanes (Hopanes)	C29aB/C30aB
(m/z 191)	C31aB(S+R)/C32aB(S+R) + C33aB(S+R)
	C32aB(S+R)/C31aB(S+R) + C33aB(S+R)
	C33aB(S+R)/C31aB(S+R) + C32aB(S+R)
	C27DBaS/C27DBaR
	C29DBaS/C29DBaR
Diasteranes and Regular 14α(H)-steranes	C28aaaR/C29aaaR
(m/z 217)	C29aaaS/C29aaaR
	C29BBR/C29BBS
	C29aaaS/C29aaa(R+S)
	C27BBR/C27BBS
	C28BBR/C28BBS
14β(H)-Steranes	C29BBR/C29BBS
( <i>m/z</i> 218)	C27BB(R+S)/[C28BB(R+S)+C29BB(R+S)]
	C28BB(R+S)/[C27BB(R+S)+C29BB(R+S)]
	C29BB(R+S)/[C27BB(R+S)+C28BB(R+S)]
	C20TA/C21 TA
Triaromatic Steroids	SC26TA/SC28TA
(m/z 231)	RC27TA/RC28TA
Inter-Ion Biomarker Ratios	C27BB(R+S)/C30aB
( <i>m/z</i> 218/191)	C29BB(R+S)/C30aB

<sup>\*</sup> Compound names for abbreviations are provided in Table 2.

Table C4. GC/MS and Oil Source Fingerprinting Results

		e et	g) et	0			
LSU ID	Field ID	Total Target Alkanes (ug/g)	Total Target PAHs (ng/g)	Qualitative Comparison to MC252	Diagnostic Biomarker Ratio Analysis Score	Chemometrics	Comments
2014322-01	SYA025	0.28	28.6				No oil detected
2014322-02	JEB033	0.24	11.5				No oil detected
2014322-03	JEB035	0.03	0.61				No oil detected
2014322-04	JEB053	0.28	0.21				No oil detected
2015049-01	SBC031	0.65	43.5			Inconclusive	Oil detected; too weathered
2015049-02	SBC032	1.57	45.7				No oil detected
2015049-03	SBD006	2.11	2.76		-		No oil detected
2015049-04	SBD016	0.29	34.5			Inconclusive	Oil detected; too weathered
2015191-01	SBC026 (Soil)	5.28	18,700	Match	91	Possible Match	Possible Match
2015191-02	JEB021 (Shells)	Fingerprint Only	Fingerprint Only			Inconclusive	Oil detected; too weathered
2015230-01	SBF023	0.76	1.27				Oil detected; too weathered
2015230-02	SBF021	0.64	1.98				Oil detected; too weathered
2015230-03	LFG024	3.64	47.1				No Oil
2015230-04	LFG045	Fingerprint Only	Fingerprint Only	Match	30	Possible Match	Possible Match
2015230-05	SBH002	102	26,000	Match	83	Match	MATCH
2015230-06	SBH004	76.5	19,700	Match	83	Match	MATCH
2015230-07	SBH011	1.36	17.0				No Oil
2015230-08	SBE023	0.56	27.8				No Oil
2016022-01	LFGA040	5.54	116				No Oil
2016022-02	LFG048	0.72	26.8				No Oil
2016022-03	JEB026	Fingerprint Only	Fingerprint Only	Inconclusive	35	Inconclusive	Inconclusive
2016022-04	SBC033	0.74	14.4				No Oil
2016022-05	SBDA015	0.25	38.4	Match	26	Possible Match	Possible Match
2016022-06	SBH027	0.49	8.38				Oil detected; too weathered
2016022-07	SBH035	1.01	26.2				No Oil
2016022-08	SBH039	0.25	7.50				No Oil
2016022-09	JEB055	0.29	9.44				No Oil
2016022-10	SBF018	0.14	10.6				No Oil

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## **Appendix D:** Analysis of Trace Elements in Pottery Samples

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### **Acknowledgments**

We wish to acknowledge Mr. Brett Johnson for preparing the samples for NAA, Dr. William Gilstrap for performing LA-ICP-MS on the sherds, and the University of Missouri Research Reactor (MURR) for supporting the Archaeometry Laboratory. The Archaeometry Laboratory at MURR is supported in part by National Science Foundation (NSF) grant #1415403. We also acknowledge an earlier NSF Major Research Instrumentation grant #0922374, which made it possible to purchase the laser and quadrupole ICP-MS used in this study.

#### D.1 Abstract

This report describes the analysis of pottery samples from coastal Louisiana contaminated by crude oil from the 2010 *Deepwater Horizon* accident and subsequent oil spill. Five samples of pottery were submitted to the Archaeometry Lab at MURR for analysis. The samples were analyzed by neutron activation analysis (NAA) and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS). The NAA results indicate that the only element possibly compromised by contact with crude oil was arsenic. None of the other 32 elements by NAA were effected. The LA-ICP-MS results are inconclusive. The potential impact on provenance studies is negligible. Heating the sample will remove the odor and most of the unbound arsenic.

## **D.2 Materials and Sampling Methods**

The pottery samples were delivered as sherds. Four of the sherds (SBDA011, SBDB011, SBD012, SBC035) came from coastal sites contaminated by the *Deepwater Horizon* oil spill. The fifth sherd (SBE017) was used as a control. Upon their arrival, a distinct odor of oil was noticeably emanating from all four of the oil spill samples.

The five pottery samples were prepared for NAA according to standard operating procedures at MURR (Glascock 1992; Glascock and Neff 2003) by removing a 1 cm<sup>2</sup> portion from each sherd. A small dremel tool was used to burr away the exposed surface areas and to obtain the inner portion of the sherd (or paste). The paste samples were then ground to powder, dried in an oven overnight, and weighed into vials for NAA. Two analytical samples were prepared from each powdered sample for short and long irradiations. The short irradiation sample used 150 mg of powder weighed into a density poly vial. The long irradiation sample used 200 mg of powder weighed into a high-purity quartz vial. Standard reference materials made from SRM-1633b flyash, SRM-278 obsidian rock, SRM-699 basalt rock, and Ohio Red Clay were similarly prepared. Samples for LA-ICP-MS were prepared by mounting a small portion from each sherd onto a glass slide along with a sample of Ohio Red Clay, SRM-612 glass, and other standard reference materials.

#### D.2.1 NAA Analysis at MURR

The NAA samples were irradiated under conditions described previously by Glascock (1992) and Glascock and Neff (2003). The short irradiation samples and standards were irradiated in a neutron flux of 8x1013 n cm-2 s-1 for five seconds, allowed to decay for 25 minutes, and counted for 12 minutes each on a high-purity germanium detector. This measurement produces data for the following short-lived elements: Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V. The long irradiation samples and standards were rolled

up into a bundle with aluminum foil and submitted for a 24-hour irradiation in a neutron flux of 5x1013 n cm-2 s-1. The irradiation was followed by two separate counts. The first count was performed after seven days of decay using 30 minute counting time to measure the following medium-lived elements: As, La, Lu, Nd, Sm, U, and Yb.

Three weeks later the samples were counted a second time for 2.5 hours each to measure the long-lived elements: Ce, Co, Cr, Cs, Eu, Fe, Hf, Ni, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, and Zr. The NAA results are listed in Table 1 in parts per million for each element.

Table D1. Concentrations in parts per million for the elements measured in sherds contaminated by oil from the *Deepwater Horizon* spill

Sample SBE017-ref is a control sample.

- r											
ANID	Na	Al	K	Ca	Sc	Ti	٧	Cr	Mn	Fe	Со
SBDA011	5915	75269	12550	3907	10.8	4267	101.4	71.7	183	35694	5.9
SBDB011	10216	85149	20125	8533	13.6	4584	138.9	82.7	881	48019	14.1
SBD012	12232	83786	19796	8785	13.8	4171	128.3	81.7	1603	45611	14.8
SBC035	12685	93403	22690	7965	13.9	4517	123.2	91.4	995	36765	41
SBE017-ref	9694	70127	22489	6696	9.7	3210	96.8	62.7	1184	34154	14.3

ANID	Ni	Zn	As	Rb	Sr	Zr	Sb	Cs	Ва	La	Се
SBDA011	0	59.4	27.5	55.8	124.2	195.6	0.63	3.68	446	33.3	64.8
SBDB011	44.5	104.1	27.5	90.9	201.1	183.4	0.88	4.85	621	41.4	81.2
SBD012	31	111.4	26.6	102.5	274.8	156.4	0.91	5.4	768	40.4	80.1
SBC035	52.4	115.2	33.6	78.8	157.8	131.5	1.18	4.06	698	39.9	82.7
SBE017-ref	0	88.4	12.8	90	208.7	151.8	0.85	4.83	755	34.2	68.4

ANID	Nd	Sm	Eu	Tb	Dy	Yb	Lu	Hf	Та	Th	U
SBDA011	31.1	5.63	1.04	0.74	4.62	2.83	0.39	8.42	1.16	11.3	3.48
SBDB011	39	7.3	1.44	0.83	5.36	3.08	0.44	6	1.12	13	4.02
SBD012	35.9	7.2	1.42	0.92	5.43	2.84	0.49	5.47	1.21	12.5	4.31
SBC035	36.8	7.46	1.52	1.03	6.35	3.58	0.54	5.55	1.25	13.6	3.82
SBE017-ref	31.1	6.08	1.24	0.79	4.65	2.57	0.41	6	0.92	10.2	3.05

#### D.2.2 LA-ICP-MS Analysis at MURR

The sherds were mounted on the glass slide such that scans of the profile from the exterior to interior of each sherd could be performed. Due to the porosity and granularity of the sherds, continuous profiles were not possible. As a result, five laser spots were identified on each sherd using three different depths from the exterior to interior. Depth #1 was near the exterior surface, depth #2 was near the core, and depth #3 was near the interior surface.

The Photon Machines laser ablation system operated with a 40 micron diameter laser beam was used to ablate the samples. An argon sweep gas transported the ablation particulate from the laser to a Nexion 300X quadurople ICP-MS where the elemental data were collected. The data from the five spots were averaged to produce three data points for each of the measured elements on each sherd. The elements

analyzed were: Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Rb, La, Ce, Nd, Sm, Eu, Tb, Dy, Yb, Lu, Hf, Ta, Pb, Th, and U.

### D.3 Results and Interpretation

To compare the contaminated sherds to the control-reference sherd, ratios of concentrations for contaminated to reference sherd were calculated. A line plot showing the four resulting ratios is presented in Figure D1 (a, b, c). A value of one or nearby should indicate little or no additional amount of the element to the contaminated sherd relative to the control. Our expectation was that elements commonly known to be present in crude oil might cause increased concentrations for those elements in the sherds. According to Hitchon and Filby (1983), several trace elements in crude oils have been measured in the parts per million range. They observed the following maximum amounts in Alberta crude oils: V (176 ppm); Mn (3.8 ppm), Fe (140 ppm), Co (2 ppm), Ni (74 ppm), and As (2 ppm).

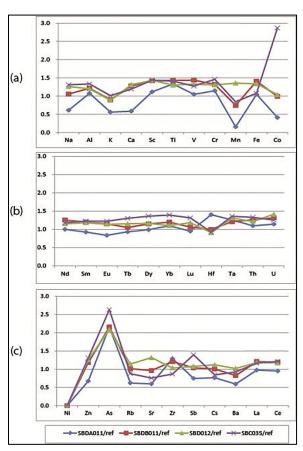


Figure D1. Ratios of elements in contaminated samples to the control reference sample.

Note that the element nickel was below detection in several of the samples.

The natural abundances of elements Mn and Fe in the sherd are sufficiently high such that contamination is unlikely to be observable. For this reason, we anticipated that the elements V and Ni might be the best indicators of contamination and producing a negative effect on trace-element analysis for provenance research. However, neither of the two elements (V or Ni) produced a high ratio between contaminated and control. On the other hand, the calculations for As produced ratios between 2 and 3 times higher in all four of the contaminated samples than in the control sample. One sample out of four has a high ratio for cobalt. The increase in arsenic concentrations is consistent and is our only evidence suggesting a possible influence on provenance determination if this element included. To demonstrate this effect, we compared

the sherds from this study to five sherds from the Lafourche Parish analyzed previously by NAA in our laboratory.

RQ-Mode Principal components analysis (Neff 1994) was performed on the sherds from this study along with five sherds from the Lafourche Parish previously analyzed by the Archaeometry Lab. Figure D2 (a, b) shows biplots of principal components #1 compared to #2 and #2 compared to #3, respectively. In both plots, the element vectors indicate that the main differentiating element is arsenic. Due to the limited number of samples analyzed and many other possible factors, are unable to say unconditionally that arsenic in the four *Deepwater Horizon* sherds came from crude oil. Arsenic is a volatile element that can be removed by heating.

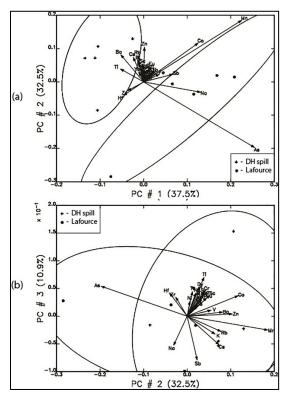


Figure D2. RQ-mode biplot of sherds from the *Deepwater Horizon* oil spill with sherds from Lafourche Parish, LA.

The data show that arsenic is the element exhibiting the greatest overall difference between sherd groups.

Unfortunately, the data from LA-ICP-MS were very erratic as we show in Figure D3 (a, b, c, d, e, f). The best explanation we have for this is that the pottery is very porous and granular. Thus, the presence or absence of small mineral inclusions may produce higher and lower amounts of the trace elements. The data are not conclusive evidence for greater contamination by trace elements on near the surface than at the other depths within the sherd.

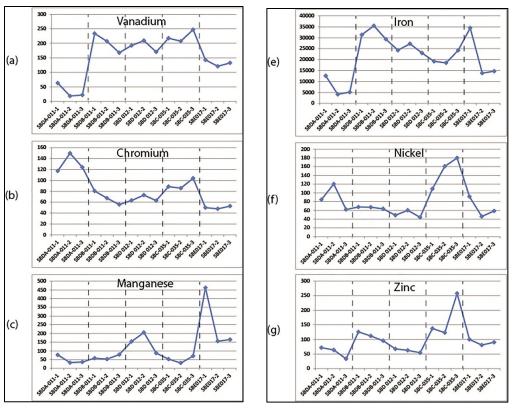


Figure D3. Line plots of LA-ICP-MS at three depths (1, 2, 3) within each sherd.

#### **D.4 Conclusions**

Analysis of four sherds from the *Deepwater Horizon* oil spill by NAA and LA-ICP-MS suggests that the effects of trace-element contamination on the sherds by crude oil are not significant enough to cause difficulty with provenance research by methods that measure trace elements. Although arsenic is observed at concentrations on the order of two or three higher than a control sherd and a set of five reference sherds from Lafourche Parish, this element is rarely used for most provenance research studies. If one is concerned about the presence of arsenic in sherds, we recommend heating the sherds for two hours at 400 degrees Celsius; this will drive off most of the arsenic and have no significant effect on the presence of other trace elements.

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## **Appendix E:** Absorbed Pottery Residue Analysis

Analysis of 17 Sherds from the Louisiana Gulf Coast: The Effects of the *Deepwater Horizon* Oil Spill and Subsequent Clean-up on Absorbed Pottery Residue Analysis

Eleanora A. Reber, UNCW Archaeological Residue Laboratory, University of North Carolina at Wilmington. UNCW Anthropological Papers 33; Papers of the UNCW Residue Lab 24.

Seventeen pottery sherds were submitted for absorbed pottery residue analysis from a variety of sites along the Louisiana Gulf Coast. Contamination was quantified by separately quantifying total lipid/g of sherd, and total biomarkers of contamination/g of sherd. These results could be used to determine a very approximate percentage of contamination. It is important to note that this number only includes biomarkers of contamination, and therefore underestimates both the degree of contamination present and the difficulty of interpreting residues in contaminated sherds. Of the seventeen sherds tested, one was severely contaminated by the oil spill and was uninterpretable (100% contaminated). Nine appeared to be uncontaminated by either oil or dispersants (0%). One was moderately severely contaminated (at least 6%), two were moderately contaminated (at least 2%), one was moderately to lightly contaminated (at least 1%) and three were lightly contaminated (less than 1%). Valid archaeometric interpretations could be made for all of the residues that were moderately and less contaminated. Future absorbed pottery residue studies on potentially oil-spill impacted sherds would benefit from analyzing soil samples from each site in parallel with the pottery residues, to control for contaminants in the soil.

#### **E.1 Introduction**

Seventeen pottery sherds were submitted for absorbed pottery residue analysis from the Louisiana Gulf Coast. At least some of the sherds had probably been exposed to pollution from the *Deepwater Horizon* oil spill and the dispersants used as part of the clean-up attempts. The purpose of the study was to determine the presence or absence of contamination by oil hydrocarbons and by dispersants. When present, the contaminants were quantified and evaluated to determine the effect of the contamination on the interpretation of pottery residues. If such contamination could be ameliorated during analysis, we also attempted to determine the analytical costs of any additional steps to the analysis, as explained below.

Absorbed residue analysis involves the extraction, identification, and interpretation of compounds that are absorbed within the ceramic matrix of a potsherd. These compounds are usually the result of the slow absorption of chemical components of resources processed in a pottery vessel over its use-lifetime, but may come from a variety of sources, including depositional contamination. Over the use-lifetime of a pottery vessel, a newly fired pottery vessel begins to absorb lipids from various cultural uses that take place over time, such as cooking, processing of non-food items, etc. All the different chemical components from many separate uses slowly accumulate within the ceramic matrix, some undergoing microbial degradation, pyrolytic reactions, and oxidation while still in use. Upon burial, these components may continue to undergo changes, including being washed out of the pottery vessel by groundwater, hydrolysis, and further oxidation and microbial breakdown. Compounds in the soil may also wash into the vessel, including modern forms of pollution or contamination, such as plasticizers, fertilizers, pesticides, oil, and dispersants (Reber 2012). Further, following excavation, postdepositional contamination may take place, including sunscreen and bugspray from excavators, as well as plasticizers from plastic bags. In order to be preserved within the matrix of the pottery, components must be hydrophilic enough to dissolve in cooking liquid, but hydrophobic enough that they do not wash out of the pot during archaeological deposition. Lipids chemically fit this description most closely, and lipids therefore make up the large majority of chemical components in absorbed pottery residues, deriving from both archaeological, depositional, and postdepositional sources.

The final absorbed residue extracted from the pot, therefore, represents layers of residue formation, transformation, subtraction, and addition. The interpretation of such final absorbed residues is notoriously complex, although the actual chemistry involved in extracting the residues and identifying the components is fairly simple.

## E.2 Methods

The samples were collected in the field in aluminum foil, with special protocols to minimize inadvertent postdepositional contamination during excavation or storage. Minimal archaeological information was given to the residue lab prior to and during analysis. As a result, the study was at least singly blind—the residue lab staff did not know which sherd came from which site, or which sherds were more likely to be contaminated than others.

Once sherds arrived at the UNCW Residue lab, they were assigned a UNCW Lab number, sketched, and the temper and surface treatment of the sherd was described. It is important to note that we are not familiar with Louisiana pottery types, and did not attempt to assign a specific ware title. Further, due to the difficulties in distinguishing between grit- and grog-tempered pottery in hand-section, sherds were generally described as grit- or grog-tempered, rather than grog-tempered.

Absorbed residues were extracted using the methodology published by Richard P. Evershed, et al. (1990). Sherds were cleaned with a solvent-washed model drill to remove surface impurities, crushed in a solvent-washed mortar and pestle, an internal standard of  $20\mu L$  *n*-tetratriacontane was added, and the sherd was extracted with approximately 10 mL of 2:1 v/v chloroform/methanol per 2 g of powdered sherd. Each sample vial was then ultrasonicated for 20 min x 2, with a 10 min cooling period. The samples were centrifuged at 2000 rpm for 20 min, the supernatant was pipetted into solvent-washed vials, and samples were then filtered through ashed, solvent-washed glass filter paper (1.5  $\mu$ l mesh) to remove the remaining fine particles from the residue-impregnated solvent.

The clean solvent-residue mixture was evaporated under  $N_2$  gas and mild heat to dryness. An aliquot of this residue was derivatized with approximately 200  $\mu$ l N,O-bis(trimethylsilyl)fluoroacetamide (BSTFA) +1% trimethylchlorosilane (TMCS) and analyzed in a Fisons 8065 gas chromatograph interfaced to a Trio 1000 mass spectrometer, using a DB-1HT 15 m x .32 mm column with .1 $\mu$ l film thickness and with a column head pressure of 7.5 psi. The temperature was held at 50° for 2 min, then ramped at 10°/min until 350°, followed by a 10 min hold at that temperature. Total runtime was 42 min. Before analysis each day, the GC/MS was tuned with DFTPP to EPA standards to ensure consistent and precise mass spectrometry. This portion of the analysis is called the total lipid extract (TLE) since it contains all the components in the residue without saponification.

Residue samples were also separated into neutral and fatty acid (FA) fractions for better quantification and analysis of the various compounds in the residue. Approximately 60% of the total residue extracted from sherds was transferred to solvent-washed culture tubes, then saponified with 2 mL NaOH/methanol and heated at 75° for 1 h. The saponified residues were then extracted with 3 x 2 mL hexane, which was blown down. This extraction became the neutral fraction, and contained compounds such as alkanes, long-chain alcohols, sterols, and terpenoids. This fraction was stored under N<sub>2</sub> gas and refrigeration until analyzed using the same instrument and temperature program as the TLE.

The remainder of the residue, containing primarily free fatty acids, was acidified to pH 3-4 with 2 M HCl, and extracted with 3 x 2mL hexane into cleaned and solvent-rinsed culture tubes. This solution was evaporated, stored under N<sub>2</sub> and refrigerated until analyzed. Approximately half of the fatty acid fraction was derivatized to trimethylsilyl esters with BSTFA and analyzed using the same instrument and column as the TLE, but with a temperature program ramping from 50-150° C at 15°C min<sup>-1</sup>, followed by 150-250° C at 3° C min<sup>-1</sup>, and a 10 min hold at 250° C.

Semi-quantification was done for the absolute amount of residue present in each sample by adding the amount of all identified lipids from the TLE fraction, and calculating the amount based on the amount of the internal standard known to be present in the sample. This quantification was an interesting, but not conclusive measure of viable residue present in a potsherd. Because amounts of heavy components such as triacylglycerols and waxes are less accurately quantified on a GC/MS, samples with more of these heavy components had lower residue quantities measured. Since these are also the best-preserved residues, it led to a situation where better-preserved residues looked less abundant than badly degraded ones. Richard Evershed has suggested that  $5 \mu g/g$  is the lower limit for correct interpretation of an archaeological sample (2008). In this study, lipid amounts ranged from 2.8-132.0  $\mu g$  lipid/g sherd.

This information was also used to roughly quantify the degree of contamination in residues. The amount of known biomarkers for contaminants in each TLE were also calculated and quantified in  $\mu g$  contaminant/g of sherd. The quantity of contaminant biomarkers was divided by the quantity of total compounds in the residue to determine a percentage of contamination per sherd. It is important to note that when a sherd, for example, is described as 'at least 1% contaminated,' the quantification refers *only* to biomarkers of contamination. Both oil and dispersants are complex chemical mixtures that include only a small percentage of biomarkers in their total make, as described below. The quantification therefore certainly underestimates the total degree of contamination present in residues. The quantification is best thought of as a broad guideline to the degree of contamination, and not a definite measure. Table 1 shows the assigned lab numbers, original sample designations, sherd descriptions, lipid quantification, approximate contamination percentage, and a basic interpretation of the contents of each sherd.

Solvent blanks were run in parallel with the archaeological samples, and used to control for laboratory contamination. Blanks were generally clean for this project, suggesting that there was no significant laboratory contamination.

# E.3 How to Identify Oil and Dispersant Contamination

Identifying oil and dispersant contamination is quite complicated, as mentioned above, because neither crude oil nor the dispersants used in an oil spill are single, unique compounds. Instead, both are complex mixtures of compounds, all of which can degrade in a variety of different ways in the environment and absorb within a ceramic residue.

For example, the original chemical composition of oil tends to vary slightly based on the location from which it was extracted (Polichtchouk and Yashchenko 2006). Further, the compounds in the oil may be degraded by bacterial action, oxidation, hydrolysis, and cross-reaction with other chemicals in the environment...including, naturally, dispersants (Aeppli, et al. 2012; Seidel, et al. 2016). The result of the interaction between crude oil components and environmentally-caused breakdown of these components is often a large, undifferentiated and unidentifiable group of compounds, known technically as the 'Unresolved Complex Mixture' or 'hump,' because the mixture appears as a large hump on the gas chromatograph. That said, all crude oil is known to generally include alkanes, steranes, terpanes, including the hopane terpenoids (hopanoids), and polyaromatic hydrocarbons. Of these compound types, hopanes and polyaromatic hydrocarbons (PAHs) are often used as biomarkers for the presence of oil contamination (Romero, et al. 2015). PAHs should generally be used cautiously as a biomarker, as they are also produced through the burning of biomass, and are also used as a biomarker for the presence of soot and extensive burning (Guerin 1999; Masclet, et al. 1995). Therefore, the presence of PAHs indicates either one of these two situations. Oil may therefore be detected fairly conclusively either by the presence of the unresolved complex mixture or hopane terpenoids, and PAHs may be used to support the presence of oil contamination.

# **E.4 Dispersants**

The dispersants used primarily in the *Deepwater Horizon* oil spill were Corexit 9500 and Corexit 9527. As indicated above, each of these dispersants was composed of a complicated mixture of compounds, including 1,2-propanediol; a 1:1 sodium salt of 1,4-bis (2-ethylhexyl) ester 2-sulfo-Butanedioic acid (dioctyl sodium sulfosuccinate) [often known as DOSS]; poly (oxy-1,2-ethanediyl) derivatives of Sorbitan mono-(9Z)-9-octadecenoate [this compound is generally known as TWEEN 80 or Polysorbate 80]; poly(oxy-1,2-ethanediyl) derivatives of Sorbitan tri-9-octadecenoate [this compound is generally known as polysorbate 85]; 1-(2-butoxy-1-methylethoxy)-2-propanol; and light petroleum distillates, which is a broad and undefined class of light hydrocarbons.

Of these compounds, DOSS is generally used as a biomarker for the presence of Corexit. It is not generally found in nature and it was the proprietary compound in both Corexit 9500 and 9527 that was viewed as distinguishing it from other products (Nalco 2008a, 2008b). The compound does break down in the environment into smaller, still distinctive products (Seidel, et al. 2016), as shown in Figure 1. Although we looked for DOSS as well as its degradation products in this study, we did not find the compounds in any residues. It therefore seems likely that DOSS and its breakdown products do not absorb within pottery. This would probably be due to the anionic sodium structure, but there has been little study on the mechanisms by which compounds are absorbed within ceramics to produce residues.

Figure E1. Molecular structure of DOSS and its breakdown products.

(Seidel, et al. 2016:Figure 5)

Distinguishing many of the non-DOSS components of Corexit was quite difficult, because they often contained molecular components that are common in the environment. For example, polysorbate 80 and 85 both consist of a sorbitan sugar molecule connected to one or more oleic acid (C<sub>18:1</sub>) fatty acid [polysorbate 80] or connected to a tri-olein triacylglycerol [polysorbate 85]. The structures of polysorbates 80 and 85 are shown in Figure 2. The linkage between the sorbitan and the acyl lipids is easily broken, and the compound falls apart into a mixture of oleic acid, tri-olein, and sorbitan sugars, all of which can break down further in the environment, and are relatively common. A recent study of breakdown products of Sorbitan 80 demonstrated that the compound degraded into a wide variety of compounds of varying complexity, including polyoxyethylene esters of fatty acids (POE esters) (Kishore, et al. 2011).

Figure E2. Sorbitan-based ingredients of Corexit.

On the top is Polysorbate 80, while on the bottom is Polysorbate 85. Both consist of a sorbitan sugar, the pentacyclic molecule, linked to oleic acid through one or more ethoxy, or oxy-1,2-ethanediyl groups.

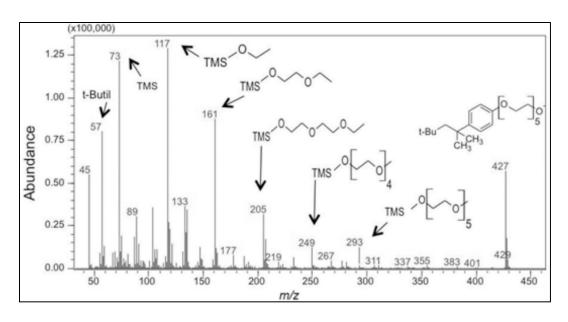
In the event, the most identifiable biomarker for oil dispersants using a GC/MS on pottery residues turned out to be POE fatty acid esters, and other long-chain POE compounds, broadly described as "polyethoxylates" in this paper. These compounds resulted from the breakdown of the polysorbate compounds and the cross-linking of the polyethoxyl functional groups to each other, and to environmental

fatty acids, probably caused by an oxidation reaction in the presence of ultraviolet light and oxygen, as illustrated in Figure 3 (Kishore, et al. 2011).

Figure E3. Proposed mechanism for the production of POE esters of environmental fatty acids from polysorbate 80.

(Kishore, et al. 2011:Scheme 4)

These compounds were easily detected in the mass spectrum by peaks at 161, representing the TMS derivative of two linked ethoxyl groups, as shown in Figure 4. These compounds were used as a biomarker for dispersants in this study. Although these compounds are not usually used as a biomarker for Corexit, it seems acceptable to use them as such in this study. First, polyethoxylates are not common in nature, and are generally produced by humans during plastic, dispersant, or surfactant synthesis. Second, these polyethoxylates were abundant and ubiquitous in several of the residues. And third, given the known presence of oil and Corexit dispersants in the area, and the known fact that POE esters of fatty acids are produced by the breakdown of polysorbates in the presence of oxygen and ultraviolet light, it seems most parsimonious to assume that these compounds derived from Corexit, rather than a completely unknown source.



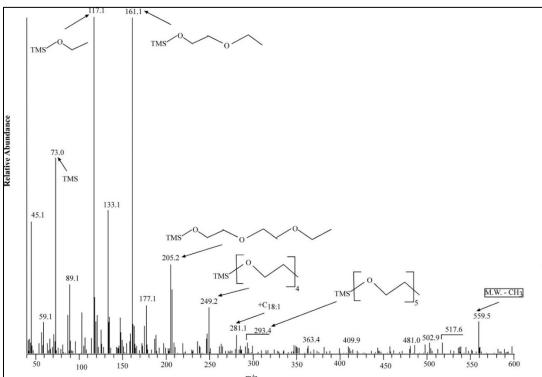


Figure E4. Comparison of a known polyethoyl compound, octylphenol polyoxyethylene (OPEO30) (Soares, et al. 2015:Figure 3) (top), with one of the polyethoxylates detected in this study (bottom), and identified as the POE ester of C<sub>18:1</sub>.

Note the similar peaks at 117, 161, 205, 249, and 293, representing portions of the polyoxyethylene chain. The molecular weight peak differs because the compounds linked to the polyethoxyethylene chain are different between the OPEO and the polyoxyethylene esters of fatty acids found in absorbed residues in this study; the molecular weight of the POE ester of  $C_{18:1}$  is 574.

# E.5 How to Interpret a lipid residue in the absence of serious contamination

When interpreting a largely uncontaminated lipid residue, several different classes of compounds are examined. The fatty acid relative abundances, particularly in terms of chain length and saturation, are examined to determine the general overall composition of the residue, as described above. Saturation is the number of double bonds present in a carbon chain. Fatty acids are generally written in the form C<sub>carbon</sub> chain length: # of double bonds. Fatty acids most commonly occur linked to a glycerol backbone in the form of triacylglycerols, which are the most abundant constituents of fats and oils in nature. Free fatty acids, although present in normal lipids, occur in only small amounts and tend to dissolve in water more easily than the glycerol forms (R.P. Evershed 1993; Richard P. Evershed, et al. 1992) and many others.

In most cases, fatty acids with more unsaturated fatty acids, particularly  $C_{16:1}$  and  $C_{18:0}$ , and more  $C_{16:0}$  than  $C_{18:0}$ , tend to originate in either vegetables or fish. Fatty acids with less unsaturated fatty acids and more  $C_{18:0}$  than  $C_{16:0}$  tend to be comprised primarily of meat lipids. Odd chain fatty acids often originate in bacterial or fungal lipids. Also, fatty acids with shorter chain lengths tend to wash out of absorbed residues earlier, while more unsaturated fatty acids are more prone to hydrolysis or oxidation. Due these and other issues described at length in other publications (Richard P. Evershed 2008; Reber and Evershed 2004), this preliminary interpretation of fatty acid composition must be paired with the interpretation of other compound types. In most cases, a residue containing highly unsaturated fatty acids can only be interpreted as 'primarily plant/fish' in origin, due to the difficulty of distinguishing between unsaturated fatty acids originating in plants and fish. In this project, this is a particular handicap.

Due to the tendency of unsaturated fatty acids to undergo hydrolysis or oxidation, it is unusual for an archaeological residue to be very strongly unsaturated. This can be defined such that an unsaturated fatty acid makes up more than 50% of the total fatty acid fraction of a residue. If a residue is that strongly unsaturated, it suggests either that the residue was comprised almost completely of plant or marine resources, or that the residue was contaminated. Modern oils and lotions are often very highly unsaturated. If a residue with a strongly unsaturated fatty acid fraction is present in a residue also containing biomarkers of modern contamination, such as DEET, vitamin E, or sunscreen compounds, then it must be classified as so contaminated that interpretation is either very difficult or impossible. If those biomarkers of modern contamination are not present, however, it is possible that the archaeological residue was comprised almost entirely of plant, fish, or shellfish resources. One of the suggested indicators for a primarily fish/shellfish residue is a high degree of unsaturation, a  $C_{18:0}/C_{16:0}$  ratio lower than .48, and the presence of cholesterol (Isaksson and Hallgren 2012). Other, less common indicators for fish and shellfish are the presence of isoprenoid fatty acids, (Baeten, et al. 2013; Corr, et al. 2008), and the presence of ω-(o-alkylphenyl) alkanoic acids, pyrolytically formed from isoprenoid fatty acids (Hansel, et al. 2004).

Sterols are one of the compound types most likely to produce general category biomarkers. Cholesterol is a biomarker for the presence of meat resources, while there is a series of plant biomarkers, including sitosterol, campesterol, and stigmasterol that indicate the presence of plant resources. The presence of cholesterol or plant sterols can help support a fatty acid composition interpretation, as well as definitively determining whether plant and meat resources were present in the lipid residue. Unfortunately, sterols are not as common as fatty acids, and are not always present. When they are present, however, they provide valuable and clear information concerning vessel contents. In this study, every sample contained sterols, some rather obscure.

Terpenoids are another compound type particularly useful in interpreting residues. They are plant biomarkers; pentacyclic triterpenoids are commonly found in non-pine plant resins and surface waxes (Glastrup 1989; Harborne and Tomas-Barberan 1991; Langenheim 2003). Diterpenoids, particularly those with the pimarane and abietane carbon skeletons, are often biomarkers for pine resin. Labdane

diterpenoids occur both in pine resins and in resin from other plants, and thus can be used as a category biomarker for plant resin, but not for any particular class of plants.

Alkanols are long-chain alcohols—carbon chain lengths of 12-34 are often found in lipid residues. Alkanols often originate in wax esters, linked to alkanes. As such, alkanols give valuable information concerning the presence of waxes in the lipid residue. Waxes occur in all resource types, but even-chain alkanols are particularly prevalent in higher plant waxes (Kolattukudy 1976). In this report, alkanols will be notated by the form OL<sub>chain length</sub>. By carefully examining references on plant waxes, sometimes a plant resource or a range of resources may be identified partially through alkanol composition. For example, very long-chain alkanols, such as OL<sub>32</sub> are rare in most plants but relatively common in panicoid grasses (Bianchi, et al. 1984; Reber, et al. 2004). Panicoid grasses are a large subfamily of about 2000 grasses, including maize and many other grasses from around the world. The presence of this compound indicates that a panicoid grass or grasses may be present in the residue. Additionally, most (but not all) plant waxes consist of a small number of alkanols esterified with a range of alkanes, or of a range of alkanols with a gradual increase in abundance of chain length to the most abundant alkanol, followed by a gradual decrease in chain length abundances (Kolattukudy 1976). Residues containing a wide range of alkanols, particularly those of very different chain length and not fitting either of these patterns, probably indicate that more than one plant resource is present.

Alkanes are unsaturated carbon chains, usually originally found linked to alkanols in waxes, or to sterols. Alkanes are described in this paper in the form  $AL_{carbon\ chain\ length}$ . Like alkanols, they occur in all resource classes. Higher plant alkanes usually have odd carbon chains; highly branched alkanes often indicate microbial or fungal breakdown of the original wax ester. Furthermore, the alkane  $AL_{29}$  can be used as a biomarker for higher plant epicuticular wax (Evershed 2008: 898). They can also be used to determine whether more than one resource source is present in a lipid similarly to the way alkanols are used.

It is important to remember that all residue interpretation must be done with some knowledge of the local biome of the site being investigated, or at least with the knowledge that more knowledge of the local biome is needed. For example, coniferous resins can be easily identified in a residue through the presence of abietane and pimarane diterpenoids, which are well-established biomarkers for this type of resin. Determining the source of such a resin, however, requires knowledge of what coniferous trees would be found near the site and likely to be utilized by the ancient inhabitants of the site. From a residue standpoint, a coniferous resin from Connecticut and one from Mississippi look identical, but the interpretation of the source and use of the resin would be different in the two places, based on environmental and cultural considerations. This is why collaboration between residue analysts, site archaeologists, and paleoethnobotanists is so crucial to a successful residue analysis.

## E.6 Results

# E.6.1 Severely contaminated—Uninterpretable due to oil and dispersant contamination

One sherd in the study, SBC029 (RL 333) was so badly contaminated by apparently degraded crude oil that it could not be interpreted at all. Almost the entire TLE consisted of the unresolved complex mixture, as shown in Figure 5. There were some compounds in the residue that could be separated from the background, including a series of polyethoxylates almost certainly resulting from the oxidation of Corexit dispersants, as described above. Hopane terpenoids were also present in the TLE of the residue, although PAHs were not. The combination of the unresolved complex mixture, hopane, and dispersants make it clear that this sherd was impacted by the oil spill and cleanup. Because of the large amounts of unresolved complex mixture, all compounds in the sherd must be assumed to derive from the contamination to some degree or another. Even if this is not completely the case, it is impossible to determine which compounds derived from contamination, and which from prior archaeological use.

Quantification was difficult, due to the hump on the baseline. That said, when the amount of known contaminants, including the Unresolved Complex Mixture was divided by the total lipids in the sherd, the residue was functionally 100% contaminated. This sherd contained the most residue of any sample in the study, as shown in Table 1. All this residue, however, appears to have derived from crude oil and dispersant.

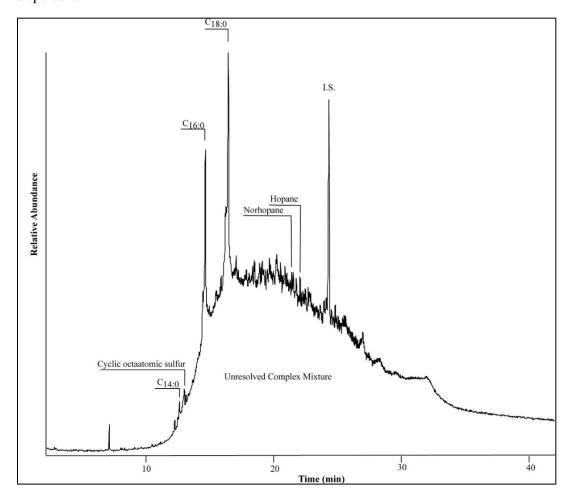


Figure E5. Gas chromatogram of the total lipid extract from SBC029 (RL 333), showing the large amounts of unresolved complex mixture resulting from serious crude oil contamination.

Other important compounds are also labelled, including two hopane terpenoids.

Table E1. Sherd provenience, lipid quantification, % contaminated, initial description, laboratory description, and residue description and interpretation for all samples in the project.

#	Provenience	μg Lipid/g sherd	% contam- inated	Initial Description	Laboratory description	Interpretation	Reason
RL 333	SBC029	132.0	100%	Baytown Plain	grit- or grog-tempered, fabric impressed or eroded cord- impressed surface	Uninterpretable due to oil and dispersant contamination.	Large hump on the baseline, prob. unresolved complex mixture, hopanoids, abundant polyethoxylates. Compounds with large peaks at 191 and 217, probably related to sorbitan.
RL 348	LFG047	10.3	6.4	grog- tempered	Shell-tempered, plain or eroded surface	Difficult to interpret, probably contained a mixture of marine algal lipids and terrestrial plant lipids.	Cholesterol, cholestanone, brassicasterol, and campesterol in neutral fraction, $AL_{29}$ present in N, as were alkanols. Fatty acids difficult to interpret due to probable dispersants.
RL 345	SYA018	8.0	2.5	Baytown Plain, grog- tempered	Thin, red sherd, grit or grog-tempered, plain surface	Dispersant contamination, DEET and fragrance contamination, some microbial presence, but terrestrial plants and perhaps meat-based resources.	Fatty acids hard to interpret, but branching and odd-chain more abundant than usual, alkanols from 11-28, most abundant is OL <sub>12</sub> , AL <sub>18-36</sub> , AL <sub>22</sub> most abundant, AL <sub>29</sub> present, labdane diterpenoids, trace of DHA in TLE, polyethoxylates, oxalic acid in N, hydroxybutyric acid in N, isolongifolene? in N, cholesterol in N
RL 347	SBC037	6.0	2.2	grog- tempered, plain surface	grit- or shell- tempered, plain (?) surface	Oil and dispersant contaminated, otherwise a mixture of lipids from animal sources and terrestrial plants.	Fatty acids primarily meat? Hard to tell, alkanols OL <sub>22-32</sub> , OL <sub>30</sub> most abundant, alkanes 28-36, AL <sub>29</sub> present, massive quantities of C <sub>17</sub> br in TLE, polyethoxylates, PAHs in N, phytol in N, cholesterol, stigmasterol, avenasterol in N
RL 343	SBE015	12.3	1.7	Shell- tempered black surface	Shell-tempered, plain/corrugated surface	Lightly to moderately contaminated with oil and dispersant, also plasticizers, otherwise a mixture of bacterial lipids, and lipids from animal sources and terrestrial plants.	Polyethoxylate, propylene glycol, and PAH present, wide range of fatty acids, particularly on light side, a lot of branching, otherwise unsaturated but hard to tell, alkanols very abundant, wide range from 10-32, most abundant are OL <sub>12</sub> and <sub>16</sub> with poss additional focus at OL <sub>20</sub> , alkanes 18-36, AL <sub>22</sub> most abundant, plasticizer contamination, labdane dieterpenoids present, bacterial breakdown product of BPA?? DEET in N, many short-chain unknowns in N, cholesterol in N, hydroxybenzoic acid in FA
RL 349	JEB025	2.8	0.5	Baytown plain	grit- or grog-tempered, plain surface	Slightly contaminated by dispersants, otherwise probably contains a small amount of mixed plant and animal-based lipids.	Polyethoxylates present, Fatty acids primarily meat? Hard to tell, wide range of alkanols OL <sub>12-32</sub> , OL <sub>28</sub> most abundant, AL <sub>25-35</sub> , foci at AL <sub>29</sub> and <sub>31</sub> ? Oxalic acid in N, hydroxybutyric acid in N, cholesterol, stigmasterol, 5a stigmastanol in N

#	Provenience	μg Lipid/g sherd	% contam- inated	Initial Description	Laboratory description	Interpretation	Reason
RL 338	LFG034	14.7	0.3	Baytown Plain	Shell-tempered, plain surface, possible visible residues on interior.	Slightly contaminated by dispersants, otherwise contains primarily plant resins from an unusual terrestrial plant, with some animal-based lipids present.	Fatty acids primarily plant/fish?, alkanol foci at OL <sub>14, 18, and 30,</sub> AL <sub>20-23</sub> , AL <sub>23</sub> most abundant, □-amyrin and □-amyrin, taraxasterol in TLE and N, polyethoxylate in TLE, cholesterol, germanicol, lanostatrienol, lupeol in N
RL 337	LFG018	43	0.2	Baytown Plain	Shell-tempered necksherd, plain (?) surface	Slightly contaminated by oil and dispersants, otherwise contains primarily plant resins from an unusual terrestrial plant, with some animal-based lipids present.	Propylene glycol, wide range of fatty acids, primarily plant/fish, only alkanol OL <sub>32</sub> , no alkanes, trace of phytanic acid in TLE, ursadienone, lupenone, lupeol, □□amyrin, □□amyrin, taraxasterol, germanicol, triterps in TLE, cholesterol, lanostatrienol in N, long series of unknown terpenoids in fatty acids.
RL 334	JEB043	9.6	0	Grog- tempered, plain surface	Shell-tempered, plain surface, possible visible residues on interior.	Seems most likely to be contaminated by modern hand cream, surfactant, or lubricant. If not, then contains primarily plant lipids with a small amount of fish.	Highly unsaturated fatty acids, primarily plant/fish/shellfish or contamination, two foci for alkanols, OL <sub>18</sub> and <sub>30</sub> , wide range of alkanols AL <sub>20-36</sub> , wide range of alkanoes including focus at AL <sub>29</sub> , lots of TAG 16:1, 16:1, 16:1, cholesterol, campesterol, stigmasterol in N
RL 346	SBCA037	11.7	0	Baytown Plain	grit- or grog-tempered, plain (?) surface	Some contamination from biodegradable plastics, also contained shellfish, plant lipids, and perhaps meat.	Fatty acids unsaturated but hard to tell, alkanols from OL <sub>12·34</sub> , most abundant is OL <sub>32</sub> , OL <sub>22 and 24</sub> also foci, AL <sub>24·36</sub> , no obvious focus, AL <sub>29</sub> present, oxalic acid in N, ethoxyamine in N, hydroxyvalerate and hydroxybutyrinfrom biodegradable plastics? Phytol in N, occelasterol in N, cholesterol, campesterol, stigmasterol, sitostanol in N
RL 340	SBD010	6.4	0	Grog- tempered, burnished surface	Shell-tempered, burnished surface	Mixture of plant and meat- based components, including plant resins.	Fatty acids indeterminate, very wide range of alkanols, from OL <sub>12-32</sub> , no obvious foci, but a lot of different ones, AL <sub>18-32</sub> present, no odd/even pattern, AL <sub>22</sub> most abundant, Labdane diterpenoids present, DHA in N, cholesterol and sitosterol in N, Fatty acids dominated by cyclic octaatomic sulfur
RL 336	JEB022	4.2	0	Baytown plain	Limestone tempered, plain (?) surface, sherd moist during sampling	Some PAHs in neutral fraction, but not TLE, some plasticizer contamination. Strong bacterial contribution, meat and plant lipids present.	Very branchy Fatty acids, a lot of C <sub>19:1</sub> , lots of branched C <sub>17</sub> , lots of alkanols, with foci apparently at OL <sub>18, 24, and 30,</sub> alkanes present at AL <sub>27, 29, 32, 36</sub> only, Oxalic acid present, 9-methyl phenanthrene present, dimethyl benzocinnoline present, plasticizer contamination, long sugar close to corexit? Lots of oxalic acid in N, cholesterol, cholestanone, sitostanol in N

#	Provenience	μg Lipid/g sherd	% contam- inated	Initial Description	Laboratory description	Interpretation	Reason
RL 335	SBF007	3.9	0	grog- tempered, plain?	Shell-tempered, plain (?) surface	Primarily plant/fish, with plant lipids, animal lipids, and some unknown compounds in the neutral fraction.	Fatty acids primarily plant/fish, wide range of alkanols, with apparent foci at OL <sub>22</sub> and 30, alkanes present at 27 and 29 (and 36) only, cholesterol, cholestanone, campesterol, campestanol, stigmasterol, 5a stigmastanol in N, bunch of heavy unknowns in N
RL 341	SBF017	3.6	0	Baytown Plain	Grit- or shell- tempered, cordmarked (?) surface	Uninterpretable	Fatty acids more meat-based but no cholesterol in N, no alkanols, no alkanes, huge amount of oxalic acid in N, malonic acid in Fatty acid fraction, may be polymer cross-link?
RL 342	SBC028	3.2	0	Grog- tempered	Rusty colored, grog or clay tempered, eroded surface. Sherd soft and squishy	Algal lipids, with plant lipids also present, including one from an unusual source containing taraxasterol.	Fatty acids difficult to interpret, alkanols abundant $OL_{15\cdot34}$ , $OL_{32}$ most abundant, alkanes 25-36, no obvious focus $AL_{29}$ present, sitosterol, campesterol, stigmasterol present in TLE, phytol in N, cholesterol, brassicasterol, campesterol, stigmasterol, taraxasterol in N
RL 332	JEB029	2.9	0	Baytown plain	grit- or grog-tempered, plain surface, sherd moist during sampling	Insufficient residue to interpret	Fatty acids primarily meat? Hard to tell. No alkanols, only alkane is $AL_{36}$ , no sterols in N
RL 339	JEB059	2.2	0	Baytown Plain	Grog-tempered, plain surface, blackened interior	Insufficient residue to interpret	Fatty acids more meat-based, no alkanols, only alkane AL <sub>15</sub> .

# E.6.2 Moderately severely contaminated—More than 5% contaminated

LFG047 (RL 348) was determined to contain at least 6% contaminants. This was due to an abundant series of polyethoxylates, indicating the presence of dispersants. The residue did not contain any biomarkers for oil or hydrocarbon contamination. There was abundant cyclic octaatomic sulfur in the residue, as shown in Tables 2 and 3; however, this sulfur-based molecule may have derived from natural soil chemistry, or possibly the breakdown of the sulfur-containing DOSS.

Interpreting this residue aside from the contamination was quite difficult. Because  $C_{18:1}$  (oleic acid) is present in Corexit complexed either by itself with sorbitan, or in triacyl form, all  $C_{18:1}$  in this residue must be assumed to derive at least in part from contamination. The residue contained  $C_{18:1}$ , although not in unusually large quantities. This makes interpretation of the fatty acid relative abundances, described above, unusually difficult and inconclusive. As a result, interpretation of these residues must derive almost completely from the neutral compounds. These neutral compounds included several sterols: cholesterol and cholestanone, campesterol, and brassicasterol. Generally, cholesterol and cholestanone are used as biomarkers for meat, campesterol is used as a biomarker for terrestrial plants and brassicasterol is a biomarker for marine algal lipids, although it is also found in some terrestrial plants (Volkman 1986). That said, all of these lipids, except cholestanone, are found commonly in marine algae (Volkman 1986); and cholestanone is a well-known breakdown produce of cholesterol. As a result, the sterol biomarkers either indicate a mixture of terrestrial plant and meat resources, or a large amount of marine algal lipids.

The alkanol and alkane evidence was somewhat inconclusive, and suggested a mixture of algal and plant-based lipids. The most abundant alkanols in the neutral fraction were OL16 and 18, both which are abundant in algae (Kolattukudy 1976:380), and OL 28-32, all of which are more typical of terrestrial vascular plants.  $OL_{32}$ , in particular, is known to be abundant in panicoic grasses, although not limited to this plant class (Bianchi, et al. 1984; Reber, et al. 2004). The alkanes were limited, in the range  $AL_{27-36}$ , with AL 32 and 36 likely representing contaminants or algal alkanes.  $AL_{29}$ , however, was present, which is often used as a biomarker for terrestrial plants, as mentioned above (Richard P. Evershed 2008).

This moderately-to-severely contaminated sherd, therefore, probably contained a mixture of algal lipids, probably from environmental contamination during deposition, and lipids from terrestrial plants, which may have derived either from anthropogenic usage prior to burial, or be also due to depositional contamination. The interpretation of the contents of the vessel were dramatically impacted by the presence of contamination—some interpretation is possible, but it requires in-depth analysis of the neutral components, which appear to be highly mixed.

# Table E2. Percentage of Total Lipid Extraction (TLE) fraction for each compound in each residue in the project.

Compounds are organized by variety, and then by chain length; fatty acids, alkanols, and alkanes are labelled as described in the 'How to Interpret a Lipid Residue in the Absence of Serious Contamination' section of the paper. Unknowns are labeled by elution time, and then by important fragments and tentative interpretation of compound class. DAG stands for diacylglycerol, TAG for triacylglycerol, and MAG for monoacylglycerol. POE esters are described as 'Polyethoxy' and the retention time given. This is because there are multiple similar identifications of, for example, POE ester of C18:1, as various esters contain varying numbers of ethoxyl groups attached to the fatty acid. Because the amounts are reported by percentage, the results are comparable across residues containing different amounts of residues; however, absolute amounts of components are not given.

Compound	RL	RL3	RL3	RL	RL												
	332	333	334	335	336	337	338	339	340	341	342	343	345	46	47	348	349
C <sub>12:0</sub>	-	-	-	-	-	-	-	-	1	-	-	1	-	-	-	-	-
C <sub>14:0</sub>	-	2	-	-	-	-	-	-	-	1	1	2	1	1	2	1	1
C <sub>15:0</sub>	-	-	-	-	1	-	-	-	-	-	-	1	-	-	-	-	-
C <sub>16:1</sub>	-	-	2	-	4	-	-	-	-	1	-	3	-	-	-	-	-
C <sub>16:0</sub>	39	28	8	9	26	3	3	19	13	15	37	12	10	30	21	18	35
C <sub>17:1</sub>	-	-	-	-	1	-	-	-	-	-	-	1	-	-	-	-	-
C <sub>17:0</sub>	-	-	-	-	1	-	-	-	-	-	-	1	-	-	-	-	1
C <sub>18:1</sub>	1	-	1	-	3	-	-	-	-	1	3	3	1	1	-	-	2
C <sub>18:0</sub>	53	33	16	10	33	6	2	37	6	24	45	16	13	59	43	20	55
C <sub>19:1</sub>	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>19:0</sub>	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>20:0</sub>	-	-	-	-	1	-	-	-	-	-	-	-	1	1	-	-	-
C <sub>22:0</sub>	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>24:0</sub>	-	-	1	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>26:0</sub>	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>28:0</sub>	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>30:0</sub>	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>15</sub> br	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>16</sub> br	-	-	-	1	1	-	-	-	-	-	1	-	-	-	-	-	1
C <sub>17</sub> br	-	-	-	-	7	-	-	-	-	-	-	1	1	-	21	-	-
C <sub>18</sub> br	-	7	-	-	1	-	-	-	-	-	-	-	-	-	1	-	1
DAG 14, 16:1	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
DAG 16, 16	-	-	-	-	1	1	-	-	-	-	-	-	-	-	-	-	-
TAG 16:1, 16:1, 16:1	-	-	51	-	-	-	-	-	-	-	-	-	-	-	-	-	-
TAG 16:1, 16:1, 16	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-
TAG 16:1, 16:1, 18:1	-	-	12	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Cyclic octaatomic sulfur	2	3	-	75	-	10	65	41	-	49	-	8	-	-	3	51	-
Propylene glycol	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-	-
DEET	-	-	-	-	-	-	-	-	-	-	-	6	1	-	-	-	-
Diiso-butyrin	-	-	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-
Hydroxy 1,3 dimethylbutyrin	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
2-methyl 1-(1,1 dimethylethyl) 2-methyl,	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
1,3 propanediyle ester propanoic acid																	
1,1'-Dodecylidenebis(4-methylbenzene)	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
Plasticizer 11.73	-	-	-	-	1	-	-	-	-	-	-	1	-	-	-	-	-
Phthalate 12.20	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
Phthalate 13.08	-	-	-	-	-	-	-	-	-	-	-	2	3	-	-	-	-
Phthalate 18.11	-	-	-	-	-	-	1	-	-	1	1	1	1	-	-	-	1
Diphthalate 19.66	-	-	-	-	-	-	-	-	-	-	1	1	-	-	-	-	-
Polyethoxy M.W. 515	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Polyethoxy 25.39	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Polyethoxy 25.65		2	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
Polyethoxy 26.64	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Polyethoxy 26.99	-	6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Polyethoxy 27.96	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Polyethoxy 28.30	-	4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Polyethoxylic acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Isolongifolene?	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-
7-isopropyl octahydrophenanthrene	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-
Ursadienone	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
Lupeol	-	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-
Labdane diterpenoid 12.67	-	-	-	-	-	-	-	-	2	-	-	-	3	-	-	-	-
Labdane diterpenoid 13.37	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
Labdadienol	-	-	-	-	-	-	-	-	8	-	-	3	-	-	-	-	-
□-Amyrin	-	-	<b>†</b> -	-	-	3	1	-	-	-	-	-	-	-	-	-	-
□-Amyrin	-	-	-	-	-	10	2	-	-	-	-	-	-	-	-	-	-
Cholesterol	-	-	-	1	-	-	1	-	1	-	3	1	1	-	-	-	-
Stigmasterol	-	-	-	-	-	-	-	_	-	-	1	-	<u> </u>	-	-	-	_
Campesterol	-	-	<b>†</b> -	-	-	-	-	-	-	1	1	-	-	-	-	-	-
Sitosterol	-	2	-	3	-	-	-	-	1	2	2	_	-	-	-	-	_
5□-stigmastanol	-	-	-	-	1	-	-	-	-	-	-	_	-	-	-	-	_
Lanostatrienol (?)	-	-	-	-	-	4	1	-	-	-	-	_	-	-	-	-	_
Taraxasterol	_	-	-	-	-	10	1	_	_	-	-	_	_	-	-	-	_
Germanicol	-	-	-	-	-	1	-	_	-	-	-	_	-	-	-	-	_
Lupenone?	_	-	<b>-</b>	-	-	4	-	_	_	-	-	_	_	-	-	-	_
Hopane	_	3	-	-	-	-	-	_	_	-	-	_	_	-	-	-	_
OL <sub>12</sub>	_	-	-	-	-	-	-	_	8	-	-	1	4	-	-	-	_
OL <sub>13</sub>	_	-	-	-	-	-	-	_	3	-	-	1	2	-	-	-	_
OL <sub>14</sub>	-	-	t <u>-</u>	-	l -	l -	-	_	3	-	-	1	1	-	-	-	_
OL <sub>16</sub>	-	-	-	-	-	-	-	-	2	1	-	2	1	-	<b> </b> -	-	-
OL <sub>18</sub>	-	-	-	-	-	-	-	_	-	<u> </u>	_	1	1	-	-	_	-
OL <sub>20</sub>	-	-	-	-	-	-	-	_	-	_	_	-	1	-	-	_	-
OL <sub>22</sub>	_	-	-	-	-	-	_	_	1	_	_	_	<u> </u>	-	-	_	_
OL <sub>28</sub>	_	-	-	1	-	-	-	_	<u> </u>	_	1	_	_	-	-	_	_
OL <sub>30</sub>	-	-	-	1	-	-	-	-	-	-	-	_	-	-	-	-	-
OL <sub>32</sub>	-	-	-	-	-	-	-	-	-	-	1	_	-	-	-	-	-
AL <sub>17:1</sub>	-	-	+-	-	-	-	-	_	3	-	-	1	4	-	-	-	-
/ N=1/(1		1	1	<u> </u>	1	1					1	_ '					1

Compound	RL	RL3	RL3	RL	RL												
A.I.	332	333	334	335	336	337	338	339	340	341	342	343	345	46 -	47	348	349
AL <sub>17</sub>	-	-	-	-	-	-	-	-	6	-	-	2	5	-	-	-	-
AL <sub>18</sub>	-	-	-	-	-	-	-	-	4	-	-	1	1	-	-	-	-
AL <sub>19</sub>	-	1	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-
AL <sub>20</sub>	-	-	-	-	-	-	-	-	2	-	-	-	2	-	-	-	-
AL <sub>21</sub>	-	-	-	-	-	-	-	-	5	-	-	1	2	-	-	-	-
$AL_{22}$	-	-	-	-	-	-	-	-	-	-	-	2	2	-	-	-	-
AL <sub>23</sub>	-	-	-	-	-	-	1	-	3	-	-	1	2	-	-	-	-
AL <sub>24:1</sub>	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
$AL_{24}$	-	4	-	-	-	-	1	-	3	-	-	-	1	-	-	-	-
AL <sub>25</sub>	-	-	-	-	-	-	1	-	3	-	-	-	1	-	-	-	-
AL <sub>26</sub>	-	-	-	-	-	-	-	-	2	-	-	-	1	-	-	-	-
AL <sub>27</sub>	-	-	-	-	-	-	-	-	1	-	-	-	1	-	-	-	-
AL <sub>30</sub>	-	-	1	-	-	-	-	-	-	-	1	-	-	-	-	-	-
AL <sub>31</sub>	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
AL <sub>32</sub>	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
AL <sub>35</sub>	-	2	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-
AL <sub>36</sub>	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
AL <sub>17</sub> br	-	-	-	-	-	-	-	-	1	-	-	-	2	-	-	-	-
AL <sub>18</sub> br	•	1	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-
AL <sub>23</sub> br	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
Benzealdehyde	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 7.15 69, 143	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
? 81, 137	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-
? 145, 160, 230, 107	-	-	-	-	-	-	-	-	3	-	-	-	2	-	-	-	-
? 10.29 73, 75, 103, 117, 187	-	-	-	-	-	-	-	-	-	-	-	1	2	-	-	-	-
? 11.95 143, 185, 157, 200, 270	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
? 13.93 195	-	-	-	-	-	-	-	-	-	-	-	1	2	-	-	-	-
? 14.79 257 wax or br alkane	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
? 15.18 163, 245, 201, 189, 286, 271, 69	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
? 187, 199, 269, 284	-	-	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-
? 17.61 147, 339, 73	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 20.31 very branchy	-	-	-	-	-	-	-	-	-	-	-	1	2	-	-	-	-
? 20.84 406, 391, 255, 253, 269,	-	-	-	-	-	1	1	-	-	-	-	-	-	-	-	-	-
295,123																	
? 21.34 triterpenoid M.W. 408	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 21.88 420, 269, 405, 202, 267	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 22.13 420, 267, 405, 171, 387	ı	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 23.08 triterp. 218, 496, 255, 391, 295, 73, 133	-	-	-	-	-	9	1	-	-	-	-	-	-	-	-	-	-
? 23.87 triterp. 218, 205, 283, 512, 426, 467	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 24.28 130, 133, 117, 57, 299, 342	-	-	-	-	-	-	-	-	-	-	-	-	-	-	_	-	1

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
? 24.30 135, 273, 232, 410, 423, 438,	-	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-
189, 95						7											
? 24.34 421, 436, 231, 286, 135	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
?24.35 203, 217, 147 sugar/glucoside	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 24.36 135, 73, 273, 232, 175, 383, 512, 469	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 24.70 273, 135, 383, 73, 512, 469, 497	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 25.32 191 217 sugar/glucoside	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 26.41 218, 103, 130, 57, 85	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
triterpenoid																	
? 255, 295, 391, 407, 57, 69, 133	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-
? 29.22 triterpenoid 218, 131, 261	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
? 29.31 long sugar, close to Corexit	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-
? 29.42 218, 131, 408 triterp.	-	-	-	-	-	1	6	-	-	-	-	-	-	-	-	1	-
273, 135, 232, 189, 367, 410, 423	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
? 273, 135, 232, 423, 407, 379	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
? 31.19 255, 295, 391, 407, 253, 207	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 31.35 218 triterp.	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 31.54 218, 408	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-
? 32.57 273, 232, 423, 407	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-

# E.6.3 Moderately contaminated—at least 2% contamination

Two sherds were described as moderately contaminated, with at least 2% of the total lipid extract comprised of biomarkers for oil or dispersant contamination: SYA018 (RL 345) and SBD037 (RL 347). SYA018 contained a series of polyethoxylates, probably indicating dispersants. There were no biomarkers for oil contamination in this residue. SBD037, however, contained both a series of polyethoxylates and also PAHs, probably indicating oil contamination.

SYA018 also contained also abundant short-chained, branched compounds after fractionation, including hydroxybutyric acid and oxalic acid. These compounds are not present in any of the dispersants known to have been used following the MC252 oil spill. Because of the composition of Corexit, however, the 1,2 propanediol and 1-(2-butoxy-1-methylethoxy)-2-propanol may well have broken down into a variety of short-chain, branched components, or might have derived from the 'light petroleum distillate' portion of the dispersant. In addition, a small amount of DEET and apparent fragrance contamination was present in the TLE form this sherd, probably deriving from bug spray in the field.

Because of the presence of contamination from both dispersants and bug spray, interpretation of this residue was quite complicated, and interpretation of fatty acids was again difficult, due to the probable  $C_{18:1}$  contamination from Corexit. The fatty acids did have one distinctive trait, however (shown in Table 4) with slightly higher than usual abundances of branched and odd-chain fatty acids, which are generally believed to be produced by bacteria (Kaneda 1991). There does seem to be some bacterial contribution to the residue, though whether this contribution derives from the action of bacteria during the ancient use-lifetime of the pottery, or during deposition is impossible to tell. The abundant short-chain alkanols and alkanes (shown in Table 3), may suggest bacterial and/or algal lipids as well.

The residue also contained cholesterol, usually a biomarker for animal lipids, as described above, as well as several labdane diterpenoids, which are biomarkers for plant resins. Sadly, this type of diterpenoid is found in a wide range of plant resins (Mills and White 1977). The presence of AL<sub>29</sub> likewise suggests a terrestrial plant.

SBD037, as mentioned above, contained contamination from dispersants, and probably from oil as well. The fatty acids were again difficult to interpret. The TLE contained large amounts of branched fatty acids, shown in Table 2, probably again indicating a bacterial contribution to the lipids. The sterols included cholesterol, stigmasterol, and avenasterol; cholesterol is a biomarker for meat, while both stigmasterol and avenasterol are biomarkers for plant oil. It is an unusual sterol distribution, in that normally a residue containing both stigmasterol and avenasterol would also contain sitosterol, which is probably the most abundant plant sterol in nature; there are a few plants, however, with avenasterol and stigmasterol more abundant than sitosterol; interestingly, one of these is quinoa oil (Fanali, et al. 2015). Both alkanols and alkanes are present in the residue, at long chain lengths consistent primarily with terrestrial plant waxes. At the present time, therefore, the best interpretation for this residue seems to be that the residue within the sherd contains a mixture of lipids deriving from plant and animal resources.

Table E3. Percentage of neutral fraction for each compound in each residue in the project

Compounds are organized by variety, and then by chain length. Unknowns are labeled by elution time and significant peak as in Table 2.

I I I I I I I I I I I I I I I I I I I	l rain	1		1	1.0.19	1		0 0 .0		1		<u> </u>	1	l pour			
Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
Cholesterol	-	-	2	1	1	1	1	-	3	-	15	1	2	4	6	10	4
Brassicasterol	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-	1	-
Campesterol	-	-	5	5	-	-	-	-	-	-	5	-	-	1	-	1	-
Stigmasterol	-	-	1	1	-	-	-	-	-	-	3	-	-	1	3	-	3
Germanicol	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
Taraxasterol	-	-	-	-	-	18	14	-	-	-	3	-	-	-	-	-	-
Sitosterol	-	-	6	7	5	-	-	-	1	-	14	-	1	3	7	2	5
Avenasterol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	7	-	-
Occelasterol	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-
Cholestanone	-	-	-	2	2	-	-	-	-	-	-	-	-	-	-	1	-
Lanostatrienol	-	-	-	-	-	10	3	-	-	-	-	-	-	-	-	-	-
Campestanol	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-	-
Sitostanol	-	-	-	-	5	-	-	-	-	-	-	-	-	2	-	-	-
5□-Stigmastanol	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-	-	3
Oxalic acid	-	-	-	-	10	-	-	-	1	64	-	-	8	3	-	-	4
Benzoic acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-
Salicyluric acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
Glycerol																	
1,2,3,5-tetramethyl-4-(3- methylbutyl)benzene	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
Ethoxyamine?	-	-	-	-	-	-	-	-	-	-	-	-	-	13	-	-	-
Hydroxybutyric acid	-	-	-	-	-	-	-	-	-	-	-	-	1	5	-	-	2
4-hydroxy hydroxyvalerate	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-
3,8 dimethyl benzocinnoline	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-
Ethoxy chain 10.13	-	-	-		-	-	-			-	-	-	1	-		-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
Dehydroabietic acid	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-
Methyl deabietate	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-
□□Amyrin	-	-	-	-	-	13	3	-	-	-	-	-	-	-	-	-	-
□□Amyrin	-	-	-	-	-	5	13	-	-	-	-	-	-	-	-	-	-
Triterp. 23.41	-	-	-	-	-	17	-	-	-	-	-	-	-	-	-	-	-
Lupeol	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-
Labdane? 12.46	-	-	-	-	-	-	-	-	-	-	-	-	4	-	-	-	-
Labdane? 13.29	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-
Labdane? 13.49	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-
Labdane? 13.84	-	-	-	-	-	-	-	-	-	-	-	3	-	-	-	-	-
Labdane? 14.32	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
CAS 57397-02-1	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
Fluoranthene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	4	-	-
Anthracene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-
Benzofluoranthene	-	-	-	-	-	-	-	-	-	-	-	-	-	-	6	-	-
PAH related to anthracene 19.39	-	-	-	-	-	-	-	-	-	-	-	-	-	-	4	-	-
Cyclic octaatomic sulfur	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	67	-
Isolongifolene ?	-	-	-	-	-	-	-	-	1	-	-	-	1	-	-	-	-
DEET	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
OL <sub>8</sub>	-	-	-	-	-	-	-	-	-	-	-	1	1	2	-	-	-
OL <sub>12</sub>	-	-	-	-	-	-	-	-	2	-	-	2	5	1	-	-	1
OL <sub>13</sub>	-	-	-	-	-	-	-	-	1	-	-	1	2	-	-	-	-
OL <sub>14</sub>	-	-	-	-	-	-	1	_	1	_	-	1	2	-	-	-	-
OL <sub>15</sub>	-	-	-	-	-	-	-	_	_	_	1	-	-	-	-	-	-
OL <sub>16</sub>	-	-	1	-	-	-	1	-	2	-	3	2	2	1	-	1	1
OL <sub>18</sub>	-	-	3	2	1	-	2	-	2	-	2	1	1	1	-	1	1
OL <sub>19</sub>	-	-	1	-	-	-	-	-	-	-	-	-	1	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
OL <sub>20:1</sub>	-	-	-	-	-	-	-	-	-	-	-	1	1	-	-	-	-
OL <sub>20</sub>	-	-	1	-	1	-	-	-	1	-	-	1	-	1	-	-	2
OL <sub>22</sub>	-	-	2	3	3	-	-	-	1	-	4	-	-	4	3	-	3
OL <sub>24</sub>	-	-	3	1	3	-	-	-	1	-	3	-	-	3	3	-	3
OL <sub>25</sub>	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
OL <sub>26</sub>	-	-	2	3	2	-	-	-	1	-	2	-	-	2	3	-	3
OL <sub>28</sub>	-	-	4	3	2	-	-	-	1	-	-	-	1	2	3	1	6
OL <sub>29</sub>	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-
OL <sub>30</sub>	-	-	5	5	4	-	1	-	1	-	1	-	-	2	5	1	3
OL <sub>32</sub>	-	-	3	2	3	-	1	-	1	-	6	-	-	5	4	1	3
OL <sub>34</sub>	-	-	1	-	-	-	-	-	-	-	1	-	-	1	-	-	-
OL <sub>36</sub>																	
Di-OL <sub>6</sub>	-	-	1	-	-	-	-	-	-	-	-	3	-	-	-	-	-
Phytol	-	-	-	-	-	-	-	-	-	-	5	-	-	1	4	1	-
OL <sub>16</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
OL <sub>18</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
OL <sub>20</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
OL <sub>22</sub> br	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
AL <sub>15</sub>	-	-	-	-	-	-	-	100	-	-	-	-	-	-	-	-	-
AL <sub>17:1</sub>	-	-	-	-	-	-	-	-	3	-	-	1	2	-	-	-	-
AL <sub>17</sub>	-	-	-	-	-	-	-	-	5	-	-	1	5	-	-	-	-
AL <sub>18</sub>	-	-	-	-	-	-	-	-	3	-	-	1	2	-	-	-	-
AL <sub>19:1</sub>	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
AL <sub>19</sub>	-	-	-	-	-	-	-	-	3	-	-	1	3	-	-	-	-
AL <sub>20</sub>	-	-	1	-	-	-	1	-	4	-	-	-	3	-	-	-	-
AL <sub>21</sub>	-	-	1	-	-	-	1	-	6	-	-	1	3	-	-	-	-
AL <sub>22</sub>	-	-	1	-	-	-	4	-	6	-	-	1	3	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
AL <sub>23</sub>	-	-	2	-	-	-	4	-	6	-	-	1	2	-	-	-	-
AL <sub>24</sub>	-	-	1	-	-	-	-	-	4	-	-	1	-	-	-	-	-
AL <sub>25</sub>	-	-	3	-	-	-	-	-	4	-	2	-	1	1	-	-	1
AL <sub>26</sub>	-	-	2	-	-	-	-	-	2	-	-	-	1	1	-	-	1
AL <sub>27</sub>	-	-	3	4	-	-	-	-	1	-	1	-	1	1	-	1	2
AL <sub>28</sub>	-	-	2	-	-	-	-	-	1	-	1	-	-	1	2	-	4
AL <sub>29</sub>	-	-	18	1	2	-	-	-	1	-	3	-	1	1	2	1	4
AL <sub>30</sub>	-	-	2	-	-	-	-	-	1	-	-	-	1	1	2	-	1
AL <sub>31</sub>	-	-	6	-	-	-	-	-	1	-	3	-	1	1	4	-	5
AL <sub>32</sub>	-	-	2	-	4	-	-	-	1	-	4	-	1	1	4	2	4
AL <sub>33</sub>	-	-	3	-	-	-	-	-	-	-	1	-	1	-	-	1	2
AL <sub>35</sub>	-	100	-	-	1	-	-	-	-	-	-	-	-	-	-	-	2
AL <sub>36</sub>	100	0	3	2	7	-	1	-	1	-	5	-	1	2	6	7	7
AL <sub>38</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2
AL <sub>17</sub> br	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
AL <sub>18</sub> br	-	-	-	-	-	-	-	-	1	-	-	1	1	-	-	-	-
AL <sub>21</sub> br	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
AL <sub>22</sub> br	-	-	-	-	-	-	3	-	1	-	-	-	-	-	-	-	-
AL <sub>24</sub> br	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-
AL <sub>27</sub> br	-	-	-	2	-	-	-	-	1	-	3	-	-	-	3	-	-
AL <sub>28</sub> br	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-
AL <sub>29</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
AL <sub>32</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
AL <sub>34</sub> br	-	-	1	-	13	-	-	-	-	-	-	-	-	1	-	-	-
AL <sub>36</sub> br	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
? 3.10 238, 221	-	-	-	-	-	-	-	-	-	-	-	1	1	-	-	-	-
? 3.55 73, 130, 188, 74	-	-	-	-	-	-	-	-	-	-	-	-	-	_	-	-	5

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
? 4.06 dihydroxy carbonate?	-	-	-	-	-	-	-	-	-	-	-	-	-	13	-	-	-
? 4.33 73, 119	-	-	-	-	-	-	-	-	-	-	-	-	-	5	-	-	-
? 4.46 73, 102, 242, 271	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 4.75 73, 117, 282	-	-	-	-	-	-	-	-	-	-	-	32	4	4	-	-	-
? 4.92 281, 147, 73	-	-	-	-	-	-	-	-	-	-	-	-	4	-	-	-	-
? 5.05 280, 73	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
? 5.10 266, 73	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-	-
? 5.21 ethoxy	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-
? 5.53 147, 117, 157, 73, 218	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
? 5.65 280, 238, 73	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 5.92 280, 73, 124	-	-	-	-	-	-	-	-	-	-	-	11	1	-	-	-	-
? 6.06 73, 193, 174, 170	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 6.36 147, 73, 191	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-
? 6.37 294, 73, 238, 124	-	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-	-
? 6.75 73, 238, 294, 124, 189	-	-	-	-	-	-	-	-	-	-	-	9	-	-	-	-	-
? 6.95 294, 73	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 7.03 73, 119, 57, 147, 207	-	-	-	-	-	-	-	-	-	-	-	1	2	-	-	-	-
? 7.75 187, 73	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
? 8.19 188, 73, 146	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 8.39 156	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 8.45 163, 123, 191	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	-
? 10.02 81, 137	-	-	-	-	-	-	-	-	1	-	-	-	1	-	-	-	-
? 10.14 145, 160, 230, 107	-	-	-	-	-	-	-	-	1	-	-	-	1	-	-	-	-
? 12.71 211, 245, 105, 69	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
? 13.02 123	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
? 14.22 293	-	-	-	1	2	-	-	-	-	-	-	-	-	-	-	-	-
? 14.43 73, 147, 221, 313, 341, 355 sugar?	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
? 14.96 143, 257	-	-	-	-	-	-	-	-	13	-	-	-	-	-	-	-	-
? 18.35 73, 116, 356	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 19.80 73, 116, 384	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 20.13 v. branchy	-	-	-	-	-	-	-	-	-	-	-	1	3	-	-	-	-
? 20.81 406, 255, 253, 391	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 20.74 255, 406, 123, 391	-	-	-	-	-	2	6	-	-	-	-	-	-	-	-	-	-
? 20.92 218, 203, 189, 408	-	-	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-
? 21.18 218, 189, 203, 408, 393 triterp.	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 21.24 218, 189, 203, 133, 119 triterp.	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 21.76 202, 203, 269, 420, 405	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 22.01 421, 406, 387, 267, 255	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-
? 420, 405, 439, 267	-	-	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-
? 22.06 205, 147	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 22.32 69, 255, 425, 421	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 22.56 135, 407, 422, 273, 232	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 22.64 424, 255, 133, 232, 271, 295	-	-	-	-	-	5	5	-	-	-	-	-	-	-	-	-	-
? 22.70 73, 255, 496, 424, 391 sterol or terp.	-	-	-	-	-	3	11	-	-	-	-	-	-	-	-	-	-
? 22.58 br diol	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
? 218, 189, 496 triterp.	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-
? 189, 103, 219, M.W. 496 triterp.	-	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-
? 119	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-
? 23.35 189, 203, 73 triterp.	-	-	-	-	11	-	-	-	-	-	-	-	-	-	-	-	-
? 57, 483	-	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-
? 43, 95, 135, 189, 370, 353, 410	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-
? 191	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-
? 23.72	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 57, 135, 231, 272, 383, 469, 512	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-
? 23.82 189, 109, 369, 498 terp.	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 24.17 189, 135, 232, 370, 353, 586 terp.	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 24.23 130, 299, 342, 356	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5
? 24.25 189, 205, 130, 133, 73 terp.	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 204.31 189, 191, 390, 509	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 24.35 130, 133, 299, 342, 356 FA ester?	-	-	-	7	-	-	-	-	-	-	-	-	-	-	-	-	-
? 24.36 □-hydroxy FA	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-	-
? 24.43 73, 135, 175, 232, 273, 383, 512	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 24.56 73, 135, 175, 232, 273, 383, 512	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-
? 191, 217, 95, 367, 369	-	-	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-
? 24.94 73, 147, 497, 353, 407	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL 340	RL 341	RL 342	RL 343	RL 345	RL3 46	RL3 47	RL 348	RL 349
? 25.45 145, 130, 83	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 25.69 69, 83, 131, 97, 133, 145, 265, 354, 221, 311	-	-	-	12	-	-	-	-	-	-	-	-	-	-	-	-	-
? 25.85 191, 217, 95, 307	-	-	-	-	-	-	-	-	-	-	-	-	-	1	3	-	4
? 26.00 217, 191, 133, 130, 57, 69, 81, 307, 327	-	-	-	3	3	-	-	-	-	-	4	-	-	-	-	-	-
? 26.11 like AL, with 145	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	2
? 26.20 130, 133, 117, 57, 71	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-	-	-
? 26.27 69, 207, 217, 145	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
? 26.30 218, 103 terp.	-	-	-	-	-	-	-	-	-	36	-	-	-	-	9	-	-
? 26.45 218, 71, 130, 135, 85, 103	-	-	-	7	-	-	-	-	-	-	-	-	-	-	-	-	-
? 26.54 171, 73, 131	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 26.63 217, 205, 307, 149	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 26.68 130, 143, 566, 328, 382	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-
? 26.81 145	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-	-
? 27.03 191, 229, 352	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2	-	-
? 27.06 130, 71, 85, 57	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-	-
? 27.17 69, 133, 191, 229, 351	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-	-
? 29.33 191, 73, 117, 95, 271, 451	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-	-	-

# E.6.4 Moderately to lightly contaminated—1–2% biomarker contamination

One residue, from SBE015 (RL 343), was moderately to lightly contaminated, with about 1.7% of the TLE consisting of biomarkers for contaminants. These contaminants were quite minor, consisting of one ethoxylate, propylene glycol, and one PAH, probably with an alkane chain attached. Even though only two dispersant-based compounds were found in this residue, it still seems likely that at least some of the  $C_{18:1}$  in the fatty acid fraction of this residue derived from dispersant contamination. There was also a small amount of DEET and fragrance contamination, probably deriving from bug spray in the field. The fatty acid composition of this residue overall was unusually unsaturated, with 10% of the fatty acid fraction consisting of  $C_{16:1}$ . That said,  $C_{16:1}$  and unsaturated fatty acids in general are often constituents in creams, lotions, and bug spray. There were many branched fatty acids in the TLE, although these components were not unusually abundant in the fatty acid fraction, which suggests that there may have been a bacterial contribution to the residue. There were also some phthalate plasticizers, although it is unclear where they came from, given the procedures for collecting the sherds directly into aluminum foil in the field.

In terms of neutral compounds, cholesterol was present, alkanols were present for a wide range of carbon chain lengths, with the shorter lengths predominating— $OL_{12}$  is the most abundant alkanol in the residue, which is unusual and may suggest a bacterial or algal contribution to the lipid (Kolattukudy 1976). Alkanes are also predominately shorter chain. That said, there are long-chain alkanols, up to  $OL_{32}$ , and  $AL_{29}$  is also present, suggesting that there were terrestrial plant lipids present within the sherd. The best interpretation of the residue in this sample is that it contained contamination from oil, dispersant, bug spray, and probably plasticizers, but that it also contained lipids from both bacterial or algal sources as well as terrestrial plants and probably meat.

# E.6.5 Lightly contaminated sherds—Less than 1% biomarker contamination

Three sherds were lightly contaminated, meaning that biomarkers for oil and dispersant contamination made up less than 1% of the total lipid extract. JEB025 (RL 349) contained the most contaminant of these three sherds, with two polyethoxylates comprising about 0.5% of the TLE. The residue itself was not very abundant, only  $28 \,\mu\text{g/g}$ . The residue did contain a wide variety of compounds, however, that made some interpretation possible. Aside from the issues with  $C_{18:1}$ , the fatty acid fraction of this residue was indeterminate. The neutral fraction contained cholesterol, stigmasterol, and sitosterol, as well as  $5\alpha$ -stigmastanol, a well-known breakdown product of sitosterol, suggesting the presence of both plant-based and animal-based lipids. The alkanols and alkanes were also fairly typical for terrestrial plants, focusing on long chains. The best interpretation of this residue, therefore, is that it contained a very small amount of a mixture of plant and animal-based lipids.

LFG034 (RL338) contained fairly abundant residue, of which 0.3% was a single polyethoxylate, suggesting a small amount of dispersant contamination. This residue was described as oil contaminated in the preliminary report, due to the abundance of highly unusual, heavy compounds that it contained. Further research suggests, however, that many of these compounds are triterpenoids. Although very unusual, these compounds are not biomarker for oil contamination, and the interpretation of the residue in this sherd has changed dramatically since the preliminary report. Ignoring the C<sub>18:1</sub> in the fatty acid fraction from this residue, which may be due to dispersant contamination, the fatty acid fraction still appears to derive primarily from plants or fish. This is indicated by the higher abundance of C<sub>16:0</sub> over C<sub>18:0</sub>, as described above. The most interesting compounds in the residue, however, are the sterols and terpenoids. The sterols include cholesterol and germanicol (present in algal and plant lipids) but most interestingly, 14% of the neutral fraction from this sherd is comprised of taraxasterol, a very unusual plant sterol. There is also a sterol tentatively identified as lanostatrienol in the neutral fraction. Taraxasterol is found in abundance in members of genus *Taraxacum*, which includes dandelions. It is also present in

other new world plants, including *Phytolacca Americana* (pokeweed), and *Helianthus annus* (sunflower) (Sharma and Zafar 2015:Table 1). Similarly, there is a long series of unusual, presently unidentified terpenoids that appear to have fractionated into the neutral fraction (Table 4). Some terpenoids were identified, including  $\alpha$ - and  $\beta$ -amyrin, both of which are found in resins from nonconiferous plants. The alkanols and alkanes are unusually short, and look more reflective of bacterial or algal lipids; there is no AL<sub>29</sub>, despite the abundant plant sterols. This residue appears to consist primarily of plant lipids, but contains very few plant waxes—it looks more like a resin, sap, or possibly an oil. There is a small amount of meat-based lipids contributing to the residue, as shown by the cholesterol. The residue is primarily made up of the unusual plant lipids, however. It is very similar to the similarly slightly contaminated LFG018 (RL 336) residue.

The residue from LFG018 (RL 336) is remarkably similar to that from LFG034. The only contaminant biomarkers present in this residue are an apparent hopene triterpenoid, a biomarker for oil, and propylene glycol, which probably indicates degraded dispersant. Otherwise, the residue is remarkably similar to LFG 034, containing unusually abundant  $C_{16:0}$  compared to  $C_{18:0}$ , large amounts of taraxasterol, lanostatrienol, a small amount of cholesterol, and a wide range of terpenoids, including a wider range of identified terpenoids: lupeol, lupenone, ursadienone,  $\alpha$ -amyrin, and  $\beta$ -amyrin. All these are triterpenoids found in non-coniferous plants, and are not biomarkers for any particular group of non-coniferous plants (Langenheim 2003). There is also a similar, and wider, range of unknown triterpenoids. The alkanol and alkane portions of the residue are even more limited than LFG034, with  $OL_{32}$  the only alkanol present in LFG018, and no alkanes at all. This again suggests that the source of these lipids was not a wax-containing portion of the plant, presumably deriving from almost pure sap or resin. Further research may be able to determine the source of the residues in these two sherds with more precision. Both residues are remarkably unique and interesting. The dispersant and oil contamination, although it did affect the interpretation of the sherds, did not preclude the identification of the many unique compounds in the residues.

Table E4. Percentage of fatty acid fraction for each compound in each residue in the project.

Compounds are organized by variety, and then by chain length. Unknowns are labeled by elution time and significant peaks similarly to Tables 2 and 3.

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL3 40	RL 341	RL3 42	RL3 43	RL3 45	RL3 46	RL3 47	RL3 48	RL 349
C <sub>10:0</sub>	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
C <sub>12:0</sub>	_	-	-	-	-	-	-	-	-	-	-	2	1	-	-	-	-
C <sub>14:1</sub>	_	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
C <sub>14:0</sub>	1	2	-	2	1	1	2	1	-	2	1	4	3	2	1	1	1
C <sub>15:0</sub>	-	-	-	1	1	1	1	1	-	1	-	2	1	-	-	-	-
C <sub>16:1</sub>	_	1	65	17	5	-	2	2	-	-	4	10	5	5	1	-	1
C <sub>16:0</sub>	37	29	12	55	25	48	60	36	4	32	36	27	29	27	31	16	33
C <sub>17:1</sub>	-	-	-	-	2	-	-	-	-	-	1	1	2	-	-	-	-
C <sub>17:0</sub>	1	1	-	-	-	1	1	-	-	-	1	1	2	1	-	-	1
C <sub>18:2</sub>	-	3	-	2	-	1	-	-	-	-	-	-	2	1	-	-	-
C <sub>18:1</sub>	2	5	9	10	5	6	3	3	-	2	4	7	9	5	1	1	2
C <sub>18:0</sub>	54	51	10	4	27	17	20	53	2	49	41	31	34	52	61	22	58
C <sub>19:1</sub>	1	1	-	-	6	-	1	1	-	-	2	-	-	1	1	-	1
C <sub>20:1</sub>	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-	-	
C <sub>20:0</sub>	1	1	-	-	1	1	1	1	-	-	1	-	-	1	1	-	-
C <sub>22:0</sub>	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
C <sub>24:0</sub>	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
C <sub>12</sub> br	-	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-
C <sub>13</sub> br	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
C <sub>14</sub> br	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>15</sub> br	1	1	-	-	6	1	2	-	-	-	2	2	3	1	1	-	1
C <sub>16</sub> br	1	1	-	1	3	1	3	1	-	-	2	-	5	-	-	-	1
C <sub>17:1</sub> br	-	-	-	-	1	-	-	-	-		-	-	-	-	-	-	<u> </u>
C <sub>17</sub> br	2	3	1	3	12	2	2	-	-	1	4	2	3	2	1	1	1

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL3 40	RL 341	RL3 42	RL3 43	RL3 45	RL3 46	RL3 47	RL3 48	RL 349
C <sub>18</sub> br	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-	-
C <sub>19</sub> br	-	-	-	-	1	-	-	-	-	-	-	-					
Malonic acid	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
2-hydroxy propanal	-	-	-	-	-	-	-	-	-	2	-	-	-	-	-	-	-
o-hydroxy benzoic acid	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
3-hydroxy decanoic acid	-	-	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-
Benzonaptho- cinnoline	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
Cyclic octaatomic sulfur	-	-	-	5	-	-	-	-	93	-	-	-	-	-	-	58	-
? 3.45 73, 130, 174, 188	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
? 4.30 73, 282, 163	-	-	-	-	-	-	-	-	-	5	-	-	-	-	-	-	-
? 4.41 179, 135, 105, 281	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
? 4.50 73, 221, 248, 266	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
? 4.60 73, 147, 192, 151	-	-	-	-	-	-	-	-	-	1	-	-	-	-	-	-	-
? 21.85 205, 73, 147, 133, 117	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
? 35.55 218, 135, 271, 409, 424	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 36.29 73, 496, 204, 189, 215, 229, 391, 441, 481	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 38.35 43, 163, 205, 340, 426, 445, 460	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 38.84 189	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-

Compound	RL 332	RL 333	RL 334	RL 335	RL 336	RL 337	RL 338	RL 339	RL3 40	RL 341	RL3 42	RL3 43	RL3 45	RL3 46	RL3 47	RL3 48	RL 349
? 39.07 135, 232, 273, 248, 299, 423, 438, 528, 476	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 39.66 135, 273, 232, 299, 410, 423, 438	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 38.84 135, 271, 286, 231, 421, 436, 495, 510	-	-	-	-	-	1	-	-	-	-	-	-	-	-	-	-	-
? 40.12 189, 73, 371, 354, 444, 411, 485, 500 triterp.	-	-	-	-	-	3	-	-	-	-	-	-	-	-	-	-	-
? 40.89 135, 273, 73, 175, 232, 383, 512, 469, 497	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-	-
? 41.01 273, 135, 384, 73, 232, 175, 512, 497, 469	-	-	-	-	-	-	2	-	-	-	-	-	-	-	-	-	-
? 41.39 273, 135, 73, 383, 175, 512, 469, 497	-	-	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-

#### E.6.6 Uncontaminated sherds

The remaining nine sherds in the study showed no evidence for contamination by oil or dispersant. One of these sherds, JEB 043 (RL 334), ironically, was almost certainly contaminated by some type of modern lubricant or surfactant, such as hand cream, although it is unclear how this might have happened, given the special collection techniques used in this study. More than half of the TLE was comprised of a triacylglycerol made up of three  $C_{16:1}$  fatty acids, also known as glyceryl tripalmitoleate. Naturally produced lipids seldom contain this triacylglycerol. It is, however, commonly used in hand creams and other synthesized lubricants and surfactants. The residue is therefore probably functionally uninterpretable. If the glyceryl tripalmitoleate was not due to contamination, then the residue is comprised almost completely of plant lipids with a small amount of animal-based lipid. That said, contamination looks most likely.

SBCA037 (RL 346) contained the most residue of the uncontaminated sherds. Its most interesting component was the sterol occelasterol, which is generally found in shellfish lipids (Phillips, et al. 2012). This sterol may well suggest a shellfish contribution in the residue. Other sterols present in the residue include cholesterol, campesterol, stigmasterol and sitostanol, which might derive from shellfish lipids or from plant and meat components. The alkanols and alkanes in the residue, however, contain compounds typical of terrestrial plant waxes and lipids. The residue probably is made up of shellfish lipids, plant lipids, and *perhaps* meat, although it is difficult to tell.

SBD010 (RL 340) seems likely to have contained a mixture of plant and animal-based components, including plant resins. This interpretation is based on the presence of cholesterol and sitosterol, labdane diterpenoids, and the overall profiles of the alkanols and alkanes in the residue.

JEB022 (RL 336) was unusual in that it contained no biomarkers for contaminants in the TLE, but there were small amounts of PAHs in the neutral fraction. This residue, therefore, might have been slightly contaminated, but not in a measurable fashion, as the contamination was measured in this project. There was also some plasticizer contamination in the residue. The fatty acids were unusually branched and odd-chained, as can be seen in Table 4. This probably represents a strong bacterial contribution to the residue. Aside from the contamination and bacterial contribution, the residue appears to contain a mixture of meat and plant-based lipids, with cholesterol, cholestanone, and sitostanol present in the neutral fraction, and alkanols and alkanes typical of terrestrial plant waxes.

SBF077 (RL 335) contained a small amount of residue with a highly unsaturated fatty acid fraction. It appears to be comprised almost completely of plant-based or fish-based lipids, with the sterols, alkanols, and alkanes pointing primarily to plant-based lipids with a small animal-based contribution. There are some unknown compounds in the neutral fraction of this residue that may benefit from further analysis.

SBF017 (RL 341) contained a small amount of residue that was not interpretable. The fatty acids were fairly nondiagnostic, and there were no sterols, alkanols, or alkanes. This may represent a sherd that was not used prior to deposition, or that was used to contain dry components that did not form a lipid residue.

SBC028 (RL 342) contained less absolute lipid than SBF017. This residue, however, contained several diagnostic compounds including brassicasterol, indicating some lipids from marine algae. This may be the source of the cholesterol and some of the other plant sterols; however, taraxasterol is also present in this residue, which is not known to be present in algal lipids, as are alkanols and alkanes typical of terrestrial plant waxes. As a result, this sherd can be interpreted as containing both marine algal lipids and plant lipids. The source of the taraxasterol may or may not be the same as in LFG018 and 034; if not, it would be another of the taraxasterol-containing plants present in North America.

JEB029 and JEB059 (RL 332 and 339) both contained too little residue to interpret. Although there were some fatty acids in these residues, they were very small amounts of these compounds, and there were no sterols, alkanols, or alkanes.

## E.7 Discussion

Oil and dispersant contamination can be detected in absorbed pottery residues, although the process was more complex in pottery residues than expected. The use of polyethoxylates to determine Corexit contamination has apparently never been done before, probably because DOSS is normally an excellent biomarker for Corexit contamination. The lack of DOSS in the pottery residues is probably due to the complexities inherent in absorbing compounds within pottery, as opposed to soil. For the reasons outlined above, however, I am confident that the polyethoxylates found in the absorbed pottery residues reflects Corexit contamination.

Of the seventeen sherds submitted for this study, one (5% of the total) was too contaminated by crude oil to interpret. Seven (41% of the total) were contaminated to varying degrees by either oil or dispersants, but could be interpreted with greater or lesser degrees of difficulty, as described above. In general, it seems likely that interpretations from the 'moderately to lightly' contaminated and 'contaminated' categories could generally provide valid interpretations of archaeological residues. Nine (53% of the total) were not contaminated to the best of our ability to detect oil and dispersants.

To provide some control for the possibility of unusual soil lipids washing into pottery residues, it would be wise to analyze site soil samples in tandem with pottery residue samples. This would allow the comparison of soil lipids from the site area. This would be particularly helpful in the case of sherds containing clear evidence for algal or bacterial lipids—such lipids could derive either from archaeological sources, or by washing in from the soil during deposition. This would further be helpful when sherds show some evidence for unusual contaminants, such as biodegradable plastics. Analyzing a soil sample in tandem with each absorbed pottery residue sample would double the price of the overall analysis. One or two soil samples could be utilized from each site, however, so that if more than two pottery residue samples were taken from a site, the overall cost increase would be less. In any case, the ability to control for the obviously complex soil lipids present in the area would, in my view, make up for the additional expense.

In terms of contamination from oil and dispersants the problem is generally not from the biomarkers for these contaminants, which can fairly easily be identified and discounted. The threat to the archaeometric resource of pottery residues derives from the common compounds found in both oil and dispersants. The sources of these common compounds cannot easily be distinguished during residue analysis, so that compounds within a contaminated residue would derive from a mixture of sources, both modern contaminants and ancient anthropogenic resources. The result would be a badly distorted interpretation of the results.

Somewhat adding to the difficulty, the mixture of compounds in oil and Corexit dispersants are quite different. Corexit contamination contains such a large amount of  $C_{18:1}$  fatty acid that fatty acid interpretations are rendered by difficult in the presence of this contamination. Conversely, crude oil is made up of a complex mixture of hydrocarbons that include alkanes, alkanols, and steranes, making the interpretation of at least part of the neutral fraction very challenging. Luckily, in the case of extreme oil contamination, the Unresolved Complex Mixture makes the nature and extent of the contamination obvious. In less extreme cases, however, contamination with both oil and dispersants simultaneously require diligent and careful interpretation, and ideally a soil sample from the site for comparison.

## E.8 Conclusion

Archaeological and archaeometric resources were clearly threatened by both the MC252 oil spill and the cleanup. In many cases, notably those in which the percentage of contaminant biomarker is 3% or less of the total lipid extract, contaminated residues can still provide useful archaeological information. Parallel analysis of a soil sample with an absorbed pottery residue samples allows the best control for site contamination, and would allow the most accurate archaeological interpretation of a pottery residue sample with a small amount of contamination.

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Appendix F: Carbon-14 Analysis, by Beta Analytic, Inc.



Consistent Accuracy . . . . Delivered On-time

Beta Analytic Inc. 4985 SW 74 Court Miami, Florida 33155 USA Tel: 305 667 5167 Fax: 305 663 0964 Beta@radiocarbon.com www.radiocarbon.com Darden Hood President

Ronald Hatfield Christopher Patrick Deputy Directors

December 1, 2015 Dr. Mark A. Rees

University of Louisiana at Lafayette Department of Sociology/Anthropology Mouton Hall Room 108

Lafayette, LA 70504

RE: Radiocarbon Dating Results For Samples SBE033 standard pretreatment, SBE033 solvent extraction, SBEA033 standard pretreatment, SBEA033 solvent extraction, SYA021 & SYAB021 standard pretreatment, SYA021 & SYAB021 solvent extraction, SYAA021 & SYAC021 standard pretreatment, SYAC021 solvent extraction, SBHA034 standard pretreatment, SBHA034 solvent extraction, SBF016 standard pretreatment, SBF016 solvent extraction, SBF010 solvent extraction, SBDA012 standard pretreatment, SBDA012 standard pretreatment, JEB024 & JEBA024 standard pretreatment, JEB024 & JEBA024 solvent extraction

### Dear Dr. Rees:

Enclosed are the radiocarbon dating results for 17 samples recently sent to us. As usual, the method of analysis is listed on the report with the results and calibration data is provided where applicable. The Conventional Radiocarbon Ages have all been corrected for total fractionation effects and where applicable, calibration was performed using 2013 calibration databases (cited on the graph pages).

You will notice that Beta-421680 (SBF010 solvent extraction) is reported with the units "pMC" rather than BP. "pMC" stands for "percent modern carbon". Results are reported in the pMC format when the analyzed material had more <sup>14</sup>C than did the modern (AD 1950) reference standard. The source of this "extra" <sup>14</sup>C in the atmosphere is thermo-nuclear bomb testing which on-set in the 1950s. Its presence generally indicates the material analyzed was part of a system that was respiring carbon after the on-set of the testing (AD 1950s). On occasion, the two sigma lower limit will extend into the time region before this "bomb-carbon" onset (i.e. less than 100 pMC). In those cases, there is some probability for 18th, 19th, or 20th century antiquity.

Reported results are accredited to ISO/IEC 17025:2005 Testing Accreditation PJLA #59423 standards and all chemistry was performed here in our laboratory and counted in our own accelerators here. Since Beta is not a teaching laboratory, only graduates trained to strict protocols of the ISO/IEC 17025:2005 Testing Accreditation PJLA #59423 program participated in the analyses.

As always Conventional Radiocarbon Ages and sigmas are rounded to the nearest 10 years per the conventions of the 1977 International Radiocarbon Conference. When counting statistics produce sigmas lower than +/- 30 years, a conservative +/- 30 BP is cited for the result. The reported d13C values were measured separately in an IRMS (isotope ratio mass spectrometer). They are NOT the AMS d13C which would include fractionation effects from natural, chemistry and AMS induced sources.

Our invoice will be emailed separately. Please, forward it to the appropriate officer or send a credit card authorization. Thank you. As always, if you have any questions or would like to discuss the results, don't hesitate to contact me.

Sincerely

Darden Hood



## REPORT OF RADIOCARBON DATING ANALYSES

Dr. Mark A. Rees Report Date: 12/1/2015

University of Louisiana at Lafayette

Material Received: 10/19/2015

590 +/- 30 BP

Sample Data	Measured	d13C	Conventional			
	Radiocarbon Age	Radiocarbon Age(*)				
Beta - 421661	310 +/- 30 BP	-13.6 o/oo d15N= +7.3 o/oo	500 +/- 30 BP			
SAMPLE: SBE033 standard pretrea ANALYSIS: AMS-Standard deliver MATERIAL/PRETREATMENT: (0 2 SIGMA CALIBRATION:	ry					
Beta - 421662	250 +/- 30 BP	-13.0 o/oo d15N= +8.3 o/oo	450 +/- 30 BP			
SAMPLE : SBE033 solvent extraction ANALYSIS : AMS-Standard deliver	· <del></del>					
MATERIAL/PRETREATMENT: (0 2 SIGMA CALIBRATION: 0	oone collagen): collagen extract Cal AD 1420 to 1465 (Cal BP 5		ction			
Beta - 421663	300 +/- 30 BP	-12.8 o/oo	500 +/- 30 BP			
		d15N = +7.9  o/oo				
SAMPLE: SBEA033 standard pretr						
ANALYSIS: AMS-Standard deliver MATERIAL/PRETREATMENT: (1		ion: with alkali				

SAMPLE : SBEA033 solvent extraction ANALYSIS : AMS-Standard delivery

2 SIGMA CALIBRATION :

Beta - 421664

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali and solvent extraction

Cal AD 1405 to 1445 (Cal BP 545 to 505)

400 +/- 30 BP

2 SIGMA CALIBRATION : Cal AD 1295 to 1370 (Cal BP 655 to 580) and Cal AD 1380 to 1415 (Cal BP 570 to 535)

Dates are reported as RCYBP (radiocarbon years before present, "present" = AD 1950). By international convention, the modern reference standard was 95% the 14C activity of the National Institute of Standards and Technology (NIST) Oxalic Acid (SRM 4990C) and calculated using the Libby 14C half-life (5568 years). Quoted errors represent 1 relative standard deviation statistics (68% probability) counting errors based on the combined measurements of the sample, background, and modern reference standards. Measured 13C/12C ratios (delta 13C) were calculated relative to the PDB-1 standard.

The Conventional Radiocarbon Age represents the Measured Radiocarbon Age corrected for isotopic fractionation, calculated using the delta 13C. On rare occasion where the Conventional Radiocarbon Age was calculated using an assumed delta 13C, the ratio and the Conventional Radiocarbon Age will be followed by "\*". The Conventional Radiocarbon Age is not calendar calibrated. When available, the Calendar Calibrated result is calculated from the Conventional Radiocarbon Age and is listed as the "Two Sigma Calibrated Result" for each sample.

-13.6 o/oo

d15N = +8.4 o/oo



## REPORT OF RADIOCARBON DATING ANALYSES

Dr. Mark A. Rees Report Date: 12/1/2015

Sample Data Measured d13C Conventional Radiocarbon Age Radiocarbon Age(\*)

Beta - 421665 1260 +/- 30 BP -17.7 o/oo 1380 +/- 30 BP d15N= +8.9 o/oo

SAMPLE: SYA021 & SYAB021 standard pretreatment

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali 2 SIGMA CALIBRATION: Cal AD 620 to 670 (Cal BP 1330 to 1280)

Beta - 421666 1160 +/- 30 BP -17.0 o/oo 1290 +/- 30 BP

d15N = +10.2 o/oo

SAMPLE: SYA021 & SYAB021 solvent extraction

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali and solvent extraction

2 SIGMA CALIBRATION : Cal AD 660 to 770 (Cal BP 1290 to 1180)

Beta - 421667 2030 +/- 30 BP -18.7 o/oo 2130 +/- 30 BP

d15N = +10.1 o/oo

SAMPLE: SYAA021 & SYAC021 standard pretreatment

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali

2 SIGMA CALIBRATION : Cal BC 345 to 320 (Cal BP 2295 to 2270) and Cal BC 205 to 85 (Cal BP 2155 to 2035) and Cal

BC 75 to 55 (Cal BP 2025 to 2005)

Beta - 421668 1230 +/- 30 BP -18.4 o/oo 1340 +/- 30 BP

d15N = +11.1 o/oo

SAMPLE: SYAA021 & SYAC021 solvent extraction

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali and solvent extraction

2 SIGMA CALIBRATION : Cal AD 650 to 690 (Cal BP 1300 to 1260) and Cal AD 750 to 760 (Cal BP 1200 to 1190)

Dates are reported as RCYBP (radiocarbon years before present, "present" = AD 1950). By international convention, the modern reference standard was 95% the 14C activity of the National Institute of Standards and Technology (NIST) Oxalic Acid (SRM 4990C) and calculated using the Libby 14C half-life (5568 years). Quoted errors represent 1 relative standard deviation statistics (68% probability) counting errors based on the combined measurements of the sample, background, and modern reference standards. Measured 13C/12C ratios (delta 13C) were calculated relative to the PDB-1 standard.



## REPORT OF RADIOCARBON DATING ANALYSES

Dr. Mark A. Rees Report Date: 12/1/2015

Sample Data Measured d13C Conventional Radiocarbon Age Radiocarbon Age(\*) Beta - 421673 1040 +/- 30 BP -25.4 o/oo 1030 +/- 30 BP SAMPLE: SBHA034 standard pretreatment ANALYSIS: AMS-Standard delivery MATERIAL/PRETREATMENT: (charred material): acid/alkali/acid Cal AD 975 to 1030 (Cal BP 975 to 920) 2 SIGMA CALIBRATION : 1110 +/- 30 BP Beta - 421674 -24.1 o/oo 1120 +/- 30 BP SAMPLE: SBHA034 solvent extraction ANALYSIS: AMS-Standard delivery MATERIAL/PRETREATMENT: (charred material): acid/alkali/acid/solvent extraction 2 SIGMA CALIBRATION : Cal AD 780 to 785 (Cal BP 1170 to 1165) and Cal AD 880 to 990 (Cal BP 1070 to 960) -25.7 o/oo Beta - 421677 980 +/- 30 BP 970 +/- 30 BP SAMPLE: SBF016 standard pretreatment ANALYSIS: AMS-Standard delivery

 $MATERIAL/PRETREATMENT: (charred\ material):\ acid/alkali/acid$ 

2 SIGMA CALIBRATION : Cal AD 1015 to 1155 (Cal BP 935 to 795)

SAMPLE : SBF016 solvent extraction ANALYSIS : AMS-Standard delivery

MATERIAL/PRETREATMENT: (charred material): acid/alkali/acid/solvent extraction

2 SIGMA CALIBRATION : Cal AD 980 to 1035 (Cal BP 970 to 915)

Dates are reported as RCYBP (radiocarbon years before present, "present" = AD 1950). By international convention, the modern reference standard was 95% the 14C activity of the National Institute of Standards and Technology (NIST) Oxalic Acid (SRM 4990C) and calculated using the Libby 14C half-life (5568 years). Quoted errors represent 1 relative standard deviation statistics (68% probability) counting errors based on the combined measurements of the sample, background, and modern reference standards. Measured 13C/12C ratios (delta 13C) were calculated relative to the PDB-1 standard.



## REPORT OF RADIOCARBON DATING ANALYSES

Dr. Mark A. Rees Report Date: 12/1/2015

Sample Data Measured d13C Conventional Radiocarbon Age Radiocarbon Age(\*)

Beta - 421680 127.8 +/- 0.3 pMC -15.1 o/oo 125.3 +/- 0.3 pMC

d15N = +6.7 o/oo

SAMPLE : SBF010 solvent extraction ANALYSIS : AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali and solvent extraction

COMMENT: The reported result indicates an age of post 0 BP and has been reported as a % of the modern reference standard,

indicating the material was living about the last 60 years or so ("pMC" = percent modern carbon).

Beta - 421681 1150 +/- 30 BP -23.9 o/oo 1170 +/- 30 BP

SAMPLE : SBDA012 standard pretreatment ANALYSIS : AMS-Standard delivery

MATERIAL/PRETREATMENT: (charred material): acid/alkali/acid

2 SIGMA CALIBRATION : Cal AD 770 to 905 (Cal BP 1180 to 1045) and Cal AD 920 to 965 (Cal BP 1030 to 985)

Beta - 421682 1260 +/- 30 BP -26.7 o/oo 1230 +/- 30 BP

SAMPLE : SBDA012 solvent extraction ANALYSIS : AMS-Standard delivery

MATERIAL/PRETREATMENT: (charred material): acid/alkali/acid/solvent extraction

2 SIGMA CALIBRATION : Cal AD 685 to 885 (Cal BP 1265 to 1065)

Beta - 421683 NA NA NA 780 +/- 30 BP

SAMPLE: JEB024 & JEBA024 standard pretreatment

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT : (bone collagen): collagen extraction: with alkali 2 SIGMA CALIBRATION : Cal AD 1215 to 1280 (Cal BP 735 to 670)

COMMENT: The original sample was too small to provide a d13C on the original material. However, a ratio including both natural and laboratory effects was measured during the 14C detection to calculate the true Conventional Radiocarbon Age.

Dates are reported as RCYBP (radiocarbon years before present, "present" = AD 1950). By international convention, the modern reference standard was 95% the 14C activity of the National Institute of Standards and Technology (NIST) Oxalic Acid (SRM 4990C) and calculated using the Libby 14C half-life (5568 years). Quoted errors represent 1 relative standard deviation statistics (68% probability) counting errors based on the combined measurements of the sample, background, and modern reference standards. Measured 13C/12C ratios (delta 13C) were calculated relative to the PDB-1 standard.



## REPORT OF RADIOCARBON DATING ANALYSES

Dr. Mark A. Rees Report Date: 12/1/2015

Sample Data	Measured	d13C	Conventional			
	Radiocarbon Age		Radiocarbon Age(*)			
Beta - 421684	830 +/- 30 BP	-22.5 o/oo d15N= +10.4 o/oo	870 +/- 30 BP			

SAMPLE: JEB024 & JEBA024 solvent extraction

ANALYSIS: AMS-Standard delivery

MATERIAL/PRETREATMENT: (bone collagen): collagen extraction: with alkali and solvent extraction

2 SIGMA CALIBRATION : Cal AD 1050 to 1085 (Cal BP 900 to 865) and Cal AD 1125 to 1140 (Cal BP 825 to 810) and

Cal AD 1150 to 1225 (Cal BP 800 to 725)

Dates are reported as RCYBP (radiocarbon years before present, "present" = AD 1950). By international convention, the modern reference standard was 95% the 14C activity of the National Institute of Standards and Technology (NIST) Oxalic Acid (SRM 4990C) and calculated using the Libby 14C half-life (5568 years). Quoted errors represent 1 relative standard deviation statistics (68% probability) counting errors based on the combined measurements of the sample, background, and modern reference standards. Measured 13C/12C ratios (delta 13C) were calculated relative to the PDB-1 standard.

(Variables: C13/C12 = -13.6 o/oo : lab. mult = 1)

Laboratory number Beta-421661 : SBE033 STANDARD PRETREATMENT

Conventional radiocarbon age 500 ± 30 BP

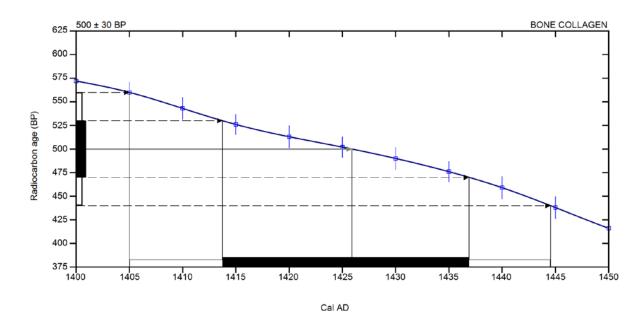
Calibrated Result (95% Probability) Cal AD 1405 to 1445 (Cal BP 545 to 505)

Intercept of radiocarbon age with calibration curve

Cal AD 1425 (Cal BP 525)

Calibrated Result (68% Probability)

Cal AD 1415 to 1435 (Cal BP 535 to 515)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine 13 radiocarbon age calibration curves 0-50,000 years cal BP. Radiocarbon 55(4):1869-1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -13 o/oo : lab. mult = 1)

Laboratory number Beta-421662 : SBE033 SOLVENT EXTRACTION

Conventional radiocarbon age 450 ± 30 BP

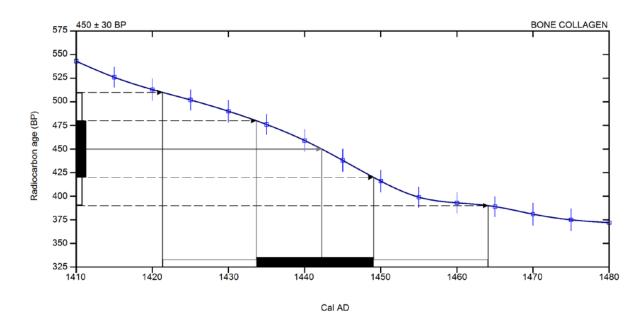
Calibrated Result (95% Probability) Cal AD 1420 to 1465 (Cal BP 530 to 485)

Intercept of radiocarbon age with calibration curve

Cal AD 1440 (Cal BP 510)

Calibrated Result (68% Probability)

Cal AD 1435 to 1450 (Cal BP 515 to 500)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0-50,000 years cal BP. Radiocarbon 55(4):1869-1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -12.8 o/oo : lab. mult = 1)

Laboratory number Beta-421663 : SBEA033 STANDARD PRETREATMENT

Conventional radiocarbon age 500 ± 30 BP

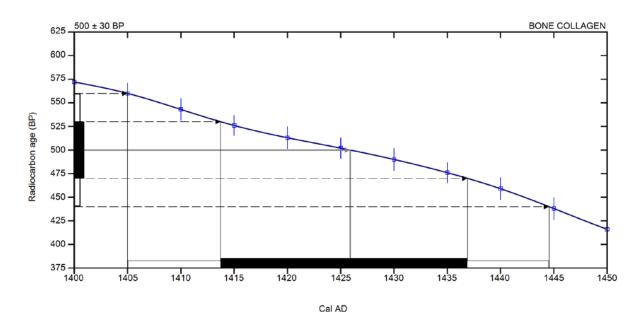
Calibrated Result (95% Probability) Cal AD 1405 to 1445 (Cal BP 545 to 505)

Intercept of radiocarbon age with calibration curve

Cal AD 1425 (Cal BP 525)

Calibrated Result (68% Probability)

Cal AD 1415 to 1435 (Cal BP 535 to 515)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0-50,000 years cal BP. Radiocarbon 55(4):1869-1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -13.6 o/oo : lab. mult = 1)

Laboratory number Beta-421664 : SBEA033 SOLVENT EXTRACTION

Conventional radiocarbon age 590 ± 30 BP

Calibrated Result (95% Probability) Cal AD 1295 to 1370 (Cal BP 655 to 580)
Cal AD 1380 to 1415 (Cal BP 570 to 535)

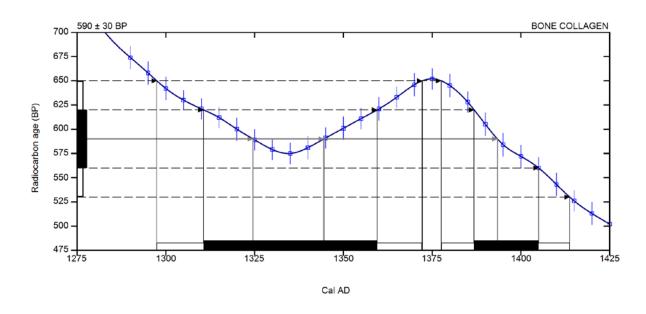
Intercept of radiocarbon age with calibration curve Cal AD 1325 (Cal BP 625)
Cal AD 1345 (Cal BP 605)

Cal AD 1395 (Cal BP 555)

Calibrated Result (68% Probability)

Cal AD 1310 to 1360 (Cal BP 640 to 590)

Cal AD 1385 to 1405 (Cal BP 565 to 545)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0-50,000 years cal BP. Radiocarbon 55(4):1869-1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -17.7 o/oo : lab. mult = 1)

Laboratory number Beta-421665 : SYA021 & SYAB021 STANDARD PRETREATMENT

Conventional radiocarbon age 1380 ± 30 BP

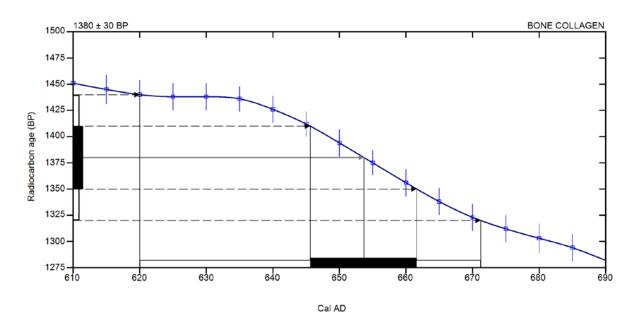
Calibrated Result (95% Probability) Cal AD 620 to 670 (Cal BP 1330 to 1280)

Intercept of radiocarbon age with calibration curve

Cal AD 655 (Cal BP 1295)

Calibrated Result (68% Probability)

Cal AD 645 to 660 (Cal BP 1305 to 1290)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -17 o/oo : lab. mult = 1)

Laboratory number Beta-421666 : SYA021 & SYAB021 SOLVENT EXTRACTION

Conventional radiocarbon age 1290 ± 30 BP

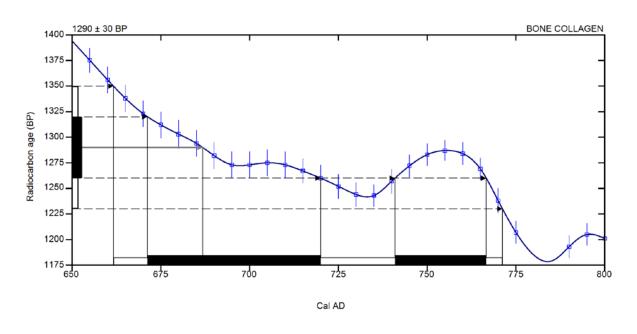
Calibrated Result (95% Probability) Cal AD 660 to 770 (Cal BP 1290 to 1180)

Intercept of radiocarbon age with calibration curve

Cal AD 685 (Cal BP 1265)

Calibrated Result (68% Probability)

Cal AD 670 to 720 (Cal BP 1280 to 1230) Cal AD 740 to 765 (Cal BP 1210 to 1185)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -18.7 o/oo : lab. mult = 1)

Laboratory number Beta-421667 : SYAA021 & SYAC021 STANDARD PRETREATMENT

Conventional radiocarbon age 2130 ± 30 BP

Calibrated Result (95% Probability) Cal BC 345 to 320 (Cal BP 2295 to 2270)

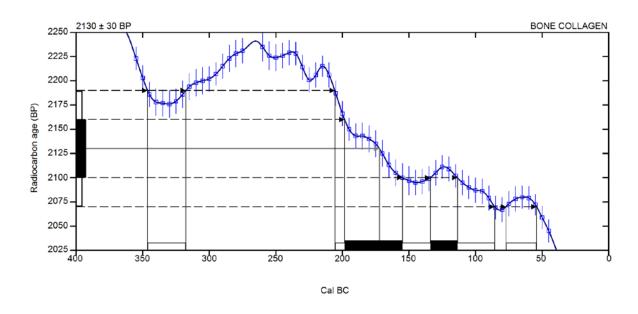
Cal BC 205 to 85 (Cal BP 2155 to 2035) Cal BC 75 to 55 (Cal BP 2025 to 2005)

Intercept of radiocarbon age with calibration curve

Cal BC 170 (Cal BP 2120)

Calibrated Result (68% Probability)

Cal BC 200 to 155 (Cal BP 2150 to 2105) Cal BC 135 to 115 (Cal BP 2085 to 2065)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -18.4 o/oo : lab. mult = 1)

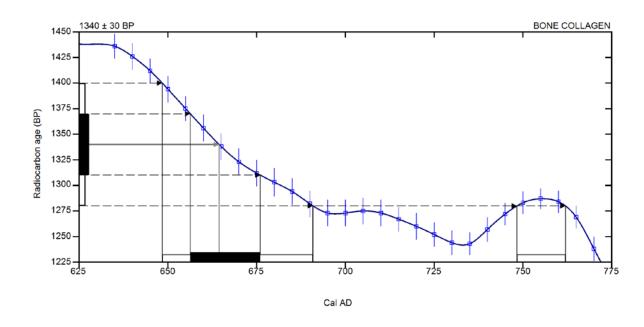
Laboratory number Beta-421668 : SYAA021 & SYAC021 SOLVENT EXTRACTION

Conventional radiocarbon age 1340 ± 30 BP

Calibrated Result (95% Probability) Cal AD 650 to 690 (Cal BP 1300 to 1260)
Cal AD 750 to 760 (Cal BP 1200 to 1190)

Intercept of radiocarbon age with calibration Cal AD 665 (Cal BP 1285)

Calibrated Result (68% Probability) Cal AD 655 to 675 (Cal BP 1295 to 1275)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -25.4 o/oo : lab. mult = 1)

Laboratory number Beta-421673 : SBHA034 STANDARD PRETREATMENT

Conventional radiocarbon age 1030 ± 30 BP

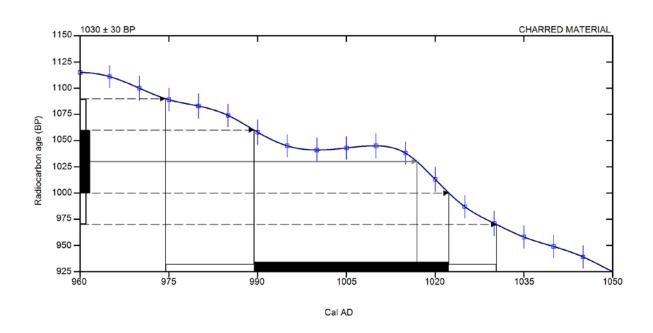
Calibrated Result (95% Probability) Cal AD 975 to 1030 (Cal BP 975 to 920)

Intercept of radiocarbon age with calibration curve

Cal AD 1015 (Cal BP 935)

Calibrated Result (68% Probability)

Cal AD 990 to 1020 (Cal BP 960 to 930)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -24.1 o/oo : lab. mult = 1)

Laboratory number Beta-421674 : SBHA034 SOLVENT EXTRACTION

Conventional radiocarbon age 1120 ± 30 BP

Calibrated Result (95% Probability)

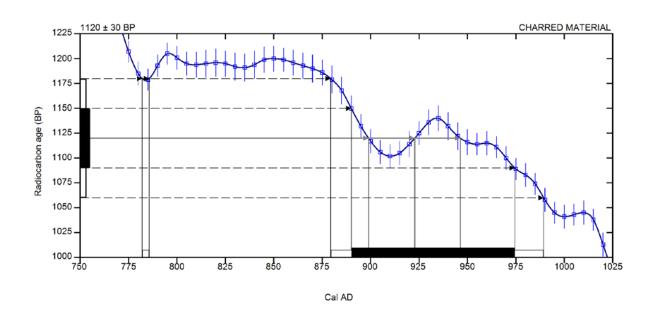
Cal AD 780 to 785 (Cal BP 1170 to 1165)

Cal AD 880 to 990 (Cal BP 1070 to 960)

Intercept of radiocarbon age with calibration curve Cal AD 900 (Cal BP 1050) Cal AD 925 (Cal BP 1025)

Cal AD 945 (Cal BP 1005)

Calibrated Result (68% Probability) Cal AD 890 to 975 (Cal BP 1060 to 975)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -25.7 o/oo : lab. mult = 1)

Laboratory number Beta-421677 : SBF016 STANDARD PRETREATMENT

Conventional radiocarbon age 970 ± 30 BP

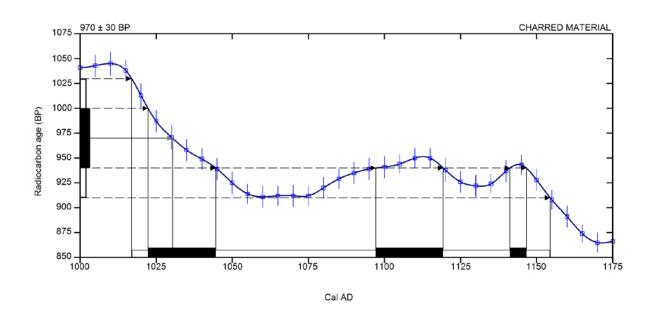
Calibrated Result (95% Probability) Cal AD 1015 to 1155 (Cal BP 935 to 795)

Intercept of radiocarbon age with calibration

Cal AD 1030 (Cal BP 920)

Calibrated Result (68% Probability)

Cal AD 1020 to 1045 (Cal BP 930 to 905) Cal AD 1095 to 1120 (Cal BP 855 to 830) Cal AD 1140 to 1145 (Cal BP 810 to 805)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -26 o/oo : lab. mult = 1)

Laboratory number Beta-421678 : SBF016 SOLVENT EXTRACTION

Conventional radiocarbon age 1020 ± 30 BP

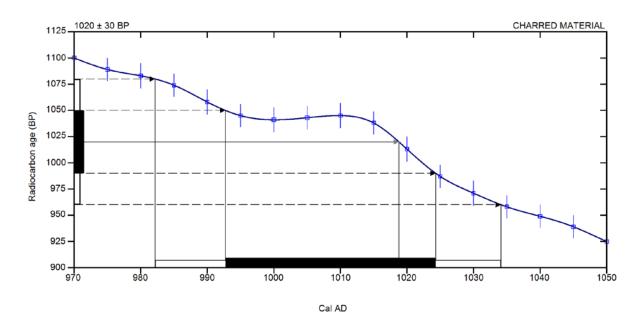
Calibrated Result (95% Probability) Cal AD 980 to 1035 (Cal BP 970 to 915)

Intercept of radiocarbon age with calibration curve

Cal AD 1020 (Cal BP 930)

Calibrated Result (68% Probability)

Cal AD 995 to 1025 (Cal BP 955 to 925)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -23.9 o/oo : lab. mult = 1)

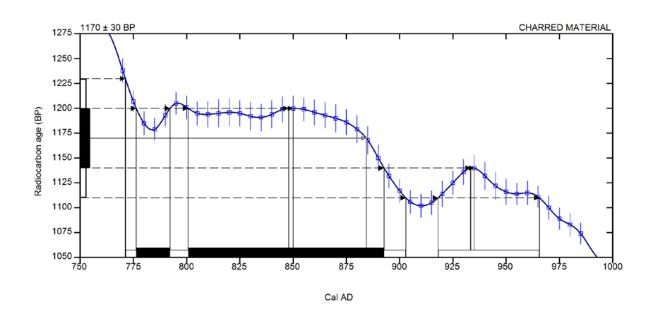
Laboratory number Beta-421681 : SBDA012 STANDARD PRETREATMENT

Conventional radiocarbon age 1170 ± 30 BP

Calibrated Result (95% Probability) Cal AD 770 to 905 (Cal BP 1180 to 1045)
Cal AD 920 to 965 (Cal BP 1030 to 985)

Intercept of radiocarbon age with calibration Cal AD 885 (Cal BP 1065)

Cal AD 775 to 790 (Cal BP 1175 to 1160)
Cal AD 800 to 895 (Cal BP 1150 to 1055)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -26.7 o/oo : lab. mult = 1)

Laboratory number Beta-421682 : SBDA012 SOLVENT EXTRACTION

Conventional radiocarbon age 1230 ± 30 BP

Calibrated Result (95% Probability) Cal AD 685 to 885 (Cal BP 1265 to 1065)

Intercept of radiocarbon age with calibration

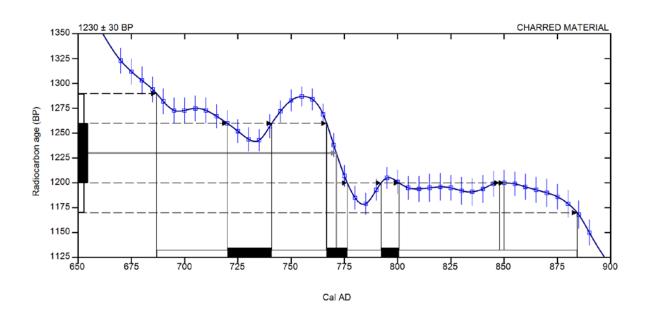
curv

Cal AD 770 (Cal BP 1180)

Calibrated Result (68% Probability) Cal AD 720 to 740 (Cal BP 1230 to 1210)

Cal AD 765 to 775 (Cal BP 1185 to 1175)

Cal AD 790 to 800 (Cal BP 1160 to 1150)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = N/A: lab. mult = 1)

Laboratory number Beta-421683 : JEB024 & JEBA024 STANDARD PRETREATMENT

Conventional radiocarbon age 780 ± 30 BP

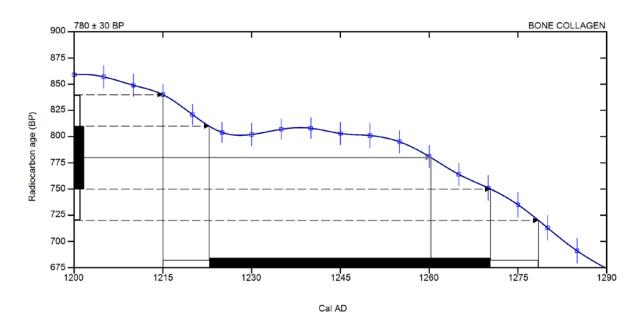
Calibrated Result (95% Probability) Cal AD 1215 to 1280 (Cal BP 735 to 670)

Intercept of radiocarbon age with calibration curve

Cal AD 1260 (Cal BP 690)

Calibrated Result (68% Probability)

Cal AD 1225 to 1270 (Cal BP 725 to 680)



# Database used INTCAL13

#### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**

(Variables: C13/C12 = -22.5 o/oo : lab. mult = 1)

Laboratory number Beta-421684 : JEB024 & JEBA024 SOLVENT EXTRACTION

Conventional radiocarbon age 870 ± 30 BP

Calibrated Result (95% Probability) Cal AD 1050 to 1085 (Cal BP 900 to 865)

Cal AD 1125 to 1140 (Cal BP 825 to 810)

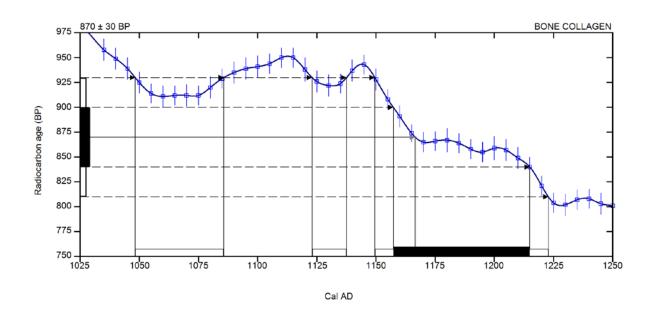
Cal AD 1150 to 1225 (Cal BP 800 to 725)

Intercept of radiocarbon age with calibration

Cal AD 1165 (Cal BP 785)

Calibrated Result (68% Probability)

Cal AD 1155 to 1215 (Cal BP 795 to 735)



# Database used INTCAL13

### References

Mathematics used for calibration scenario

A Simplified Approach to Calibrating C14 Dates, Talma, A. S., Vogel, J. C., 1993, Radiocarbon 35(2):317-322

References to INTCAL13 database

Reimer PJ et al. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. Radiocarbon 55(4):1869–1887., 2013.

### **Beta Analytic Radiocarbon Dating Laboratory**



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Fax: 305-663-0964 info@betalabservices.com www.betalabservices.com Mr. Darden Hood

President

Mr. Ronald Hatfield Mr. Christopher Patrick Deputy Directors

The Radiocarbon Laboratory Accredited to ISO/IEC 17025:2005 Testing Accreditation PJLA #59423

## **Quality Assurance Report**

This report provides the results of reference materials used to validate radiocarbon analyses prior to reporting. Known value reference materials were analyzed quasi-simultaneously with the unknowns. Results are reported as expected values vs measured values. Reported values are calculated relative to NIST SRM -4990B and corrected for isotopic fractionation. Results are reported using the direct analytical measure percent modern carbon (pMC) with one relative standard deviation.

**Report Date:** December 01, 2015 **Submitter:** Dr. Mark A. Rees

#### **QA MEASUREMENTS**

Reference 1 Expected Value: 96.7 +/- 0.5 pMC

Measured Value: 96.8 +/- 0.4 pMC

Agreement: Accepted

Reference 2 Expected Value: 129.4 +/- 0.1 pMC

Measured Value: 129.7 +/- 0.3 pMC

Agreement: Accepted

Reference 3 Expected Value: 2.2 +/- 0.2 pMC

Measured Value: 2.2 +/- 0.1 pMC

Agreement: Accepted

COMMENT: All measurements passed

acceptance tests.

Date: December 01,

2015

Dardew Hood

Validation:

Date:

December 01, 2015

## **Appendix G: Faunal Analysis**

### A. J. Delahoussaye

The analysis of faunal material for this study included samples from each of the eight assessed sites. The faunal analysis focused on the identification of element and taxon. This involved recording the number of identified specimens (NISP) and bone weight by provenience for each taxon, along with evidence of burning, other modifications, and whole or fragmented size. Bone not identifiable by taxon or element due to fragmentary condition was classified as unidentified bird, unidentified fish, unidentified mammal, unidentified reptile, or unidentified specimen. Taxon and element were also recorded for samples of bone submitted for radiocarbon analysis. Table G1 presents the number of identified specimens from the eight assessed sites.

Other than NISP, the faunal analysis is limited to a few observations regarding the presence of certain taxa and condition of the material. Inferences regarding past subsistence patterns are problematic given the small sample size from most sites, the lack of representative samples and redeposited contexts. There was no discernible odor or discoloration of the fauna that might be associated with petroleum or crude oil. The proportions of mammals are generally representative of most samples from prehistoric archaeological sites along the Gulf Coast. The smaller mammals, rather than larger ones like white-tailed deer, make up the largest NISP in the examined faunal material. The muskrat is usually common in faunal samples from the coast, and combined with the category of unidentified other mammal, constitute the largest percentage of mammals (N=688; 90.6%) in the samples from the eight sites. The unidentified other mammal category is mostly made up of fractured long bones, such as femurs. Larger mammals such as deer may not have been as readily available to the inhabitants of these coastal sites, or were obtained less frequently than smaller mammals like muskrat, mink, and raccoon.

The squirrel identified in a sample from Redfish Slough (16LF293) is somewhat unusual for the present-day environment of Philo Brice Island in Timbalier Bay, with vegetation predominantly consisting of *Spartina* marsh grass, black mangrove, and saltwort. This provides some indication of a markedly different forest habitat at some time in the past. Unidentified turtle are well represented in samples from five of the eight sites. This is because of the durability of turtle bone, particularly the carapace and plastron.

Fish are by far the most numerous animals represented in the samples, particularly at Redfish Slough (16LF293) and the Cheniere St. Denis site (16JE2). Gar, bowfin, and catfish dominate the NISP for all vertebrates combined; garfish is the most common. The numbers shown for NISP for gar do not include gar scales. These would otherwise over represent the significance of gar, so the scales are recorded separately. Most of the identified fish are, unsurprisingly, associated with saltwater, including red drum, black drum, sheepshead, stingray, and shark. However, the latter three can also exist in estuarine to fresh water conditions.

Table G1. NISP of Fauna from Eight Sites

		93		74	53	117	82	78	85	
		16LF293	6JE2	16SB174	16SB153	16SMY17	16SB182	16SB178	6SB185	
Taxon	Common Name	161	16.	168	168	168	169	168	169	Total
Rodentia	Rodent	5		14						19
Ondatra zibethicus	Muskrat	255	167	11			1	1		435
Sciurus niger	Fox squirrel	1								1
Mephitis mephitis	Striped skunk	1								1
Lontra canadensis	River otter	1								1
Neovison vison	Mink	1								1
Canis	Canid		1					1		2
Procyon lotor	Raccoon	3	3		1					7
Carnivora	Carnivore	1	1	5	1	1				9
Odocoileus virginianus	White-tailed deer	6	1		2	2		2		13
Unidentified large mammal	(Deer or larger)	1				15	1			17
Unidentified other mammal	(Smaller than deer)	131	11	16	5	84		6		253
Subtotal	dccij	406	184	46	9	102	2	10		759
Anatidae	Duck	1	104	70		102		10		1
Unidentified medium bird	(Robin-sized bird)	23	1							24
Ondertailed mediam bild	(Smaller than	20								27
Unidentified small bird	robin)	2	1							3
Subtotal	,	26	2							28
Alligator mississippiensis	American alligator	10		1	2	5				18
Kinosternidae	Mud turtle	1								1
Unidentified turtle		55	30		20	37		1	14	157
Unidentified snake		3								3
Unidentified other reptile						4				4
Subtotal		69	30	1	22	46		1	14	183
Anura	Unidentified frog					1				1
Lithobates catesbianus	Bullfrog	1	1							2
Subtotal		1	1			1				3
Lepisosteidae	Gar	252	319		3	2		1		577
Atractosteus spatula	Alligator gar		6							6
Amia calva	Bowfin	94	6							100
Ictaluridae	Catfish	64	13	5	5	3				90
Catostomidae	Sucker		1		1					2
Sciaenidae	Drum	9	12	10	2		1	3	1	38
Sciaenops ocellatus	Red drum	3	1	5						9
Pogonias chromis	Black drum		2							2
Archosargus probatocephalus	Sheepshead	1			2			1		4
Carchariniformes cf	Shark	1								1
Dasyatidae	Stingray	1								1
Unidentified fish		342	364	18	24	1		7	1	757
Subtotal		767	724	38	37	6	1	12	2	1587
Total Identified		1269	941	85	68	155	4	23	16	2560
Unidentified		3389	264	823	258	584	5	336	22	5681
TOTAL		4658	1205	908	326	739	9	359	38	8241
Lepisosteidae (scales only)	Gar	2469	3130	681	44	2		11		
Atractosteus spatula (scales only)	Alligator gar		35							



## **Department of the Interior (DOI)**

The Department of the Interior protects and manages the Nation's natural resources and cultural heritage; provides scientific and other information about those resources; and honors the Nation's trust responsibilities or special commitments to American Indians, Alaska Natives, and affiliated island communities.



## **Bureau of Ocean Energy Management (BOEM)**

The mission of the Bureau of Ocean Energy Management is to manage development of U.S. Outer Continental Shelf energy and mineral resources in an environmentally and economically responsible way.

## **BOEM Environmental Studies Program**

The mission of the Environmental Studies Program is to provide the information needed to predict, assess, and manage impacts from offshore energy and marine mineral exploration, development, and production activities on human, marine, and coastal environments. The proposal, selection, research, review, collaboration, production, and dissemination of each of BOEM's Environmental Studies follows the DOI Code of Scientific and Scholarly Conduct, in support of a culture of scientific and professional integrity, as set out in the DOI Departmental Manual (305 DM 3).