An Experimental Geochemical Characterization Analysis of Archaeological Iron from the Contact Period of Newfoundland and Labrador

By

©Tyrone Hamilton

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Department of Archaeology
Faculty of Arts
Memorial University of Newfoundland
St. John's, Newfoundland & Labrador

i. Abstract

This research uses scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX) and inductively coupled plasma-mass spectrometry (ICP-MS) on cross-sections of iron artifacts sectioned from along shafts to determine the elemental constituents of a collection of Inuit and European artifacts from along the coast of Labrador. Hand-wrought iron nails from early historic period (16^{th} – 18^{th} centuries CE) Inuit sites in Labrador were originally manufactured by and acquired from early whalers and fishers of various European nationalities. The purpose of this research was to assess if the elements in different samples are sufficiently homogeneous to be viable for a provenience analysis to discern which Inuit nails were originally derived from which European groups; the Basque, English or French. The consistent relationships between the geochemical signatures of iron nails found in Inuit sites and historic nails derived from specific European groups could provide insights into the prevalence, activity and the nature of indigenous interactions of different European nationalities in the region over time. The results show that the methods applied to evaluate the geochemistry of the nails was not sufficient to detect meaningful patterns because the nails did not demonstrate the necessary degree of chemical uniformity among different samples in the same artifacts.

Keywords: Inuit, Labrador, Iron, Provenance, European Contact, Material Culture, Exchange Networks, Basque, Geochemistry, Scanning Electron Microscopy (SEM)

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Chapter 1: Introduction

1.1 Research Objectives

The objective of this thesis is to assess the applicability of two geochemical characterization techniques in the analysis of archaeological iron from the contact period of Newfoundland and Labrador. The analysis of wrought iron nails, widely used as an iron source by the early Inuit, from 16th to 18th century "early historic" or "contact" period archaeological sites in Newfoundland and Labrador are tested via inductively coupled plasma-mass spectrometry (ICP-MS) and scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX). Heavily corroded hand-wrought iron nails and spikes are among the most abundant artifacts found in historic sites, and they are the earliest and most prolific European artifact found on Labrador Inuit sites. The main goal of this thesis is to test the viability of the analytical methodologies described in chapter four to successfully achieve a proovenance study that analyzes the elemental compositions of the interior of corroded historic iron objects. It also tests whether or not historic iron nails can demonstrate the sufficient consistency among the geochemical signatures of different artifacts that would make them viable for provenience analysis. The presence of geochemical homogeneity among a collection of artifacts is often indicative of a common source or *provenance* of the object's raw material.

The data resulting from such an analysis could provide a greater understanding of the extent of material exchange between the Labrador Inuit with several different groups of Europeans and the distribution of these materials along the Labrador coast. Because of their high prevalence and low museum display value, historic iron fasteners like nails and spikes are ideal objects for destructive archaeomineralogical analysis. Archaeomineralogy is the science of characterization, provenience and dating of archaeological minerals via geochemical analytical methods (Rapp 2009:1). If successful, the geochemical signatures of artifacts have the potential to provide insights into the material exchange patterns of the early contact period of Labrador.

The project described in this thesis used the analytical techniques of ICP-MS and SEM-EDX on a collection of iron nails derived from Inuit, Basque, French and English sites from the early historic period of Newfoundland and Labrador. Although the analytical results of the artifacts' geochemical characterization cannot provide a viable provenience of the iron nails' origins due to the large degree of chemical heterogeneity, this analysis did provide insights into the chemical unconformity present in a single iron nail that is the major contributing factor for this outcome. The first method used, ICP-MS, did not produce viable results due to errors in the methodological procedure. The elemental spectra procured in this research were, instead, obtained with detailed line-scans across the polished diameter of sectioned iron nail interiors with a scanning electron microscope (SEM-EDX). In this instance, the use of invasive analytical geochemistry on iron nails and spikes does not provide data that shows the degree of homogeneity and variation necessary to satisfy the *provenance postulate*. In order to satisfy this postulate, the data would have had to show two or more consistent and homogenous groups of artifacts whose elemental compositions diverged drastically in relation to each other. No pattern was detected.

The effectiveness of the geochemical techniques used in this characterization analysis is also explored with regard to how precise and accurate a picture of the overall geochemical contents of the iron artifacts that they provide. This thesis will emphasize issues that arise when conducting geochemical characterization research on highly heterogeneous material such as early historic iron and will include an assessment of the viability of the methodologies as well as the nature of the material and how it was manufactured, used, preserved and prepared for analysis. Future research may greatly benefit from expanding the range of types, stages of manufacture and geographic span of artifacts or the use of combined quantities and qualitative techniques like XRF or SEM-EDX combined with mineral liberation analysis (MLA).

1.2 Study Area

The coast of Labrador encompasses a vast seaboard of subarctic tundra and boreal forest along the Labrador Sea from the Torngat Mountains National Park Reserve and the Hudson Strait in the north to the Strait of Belle Isle in the south (Fig. 1.1). The major indigenous groups that currently inhabit Labrador are the *Nunatsiavut* Inuit, the *NunatuKavut* Inuit-Métis and the *Innu* First Nation. The early contact period of Newfoundland and Labrador constitutes a span of time between the initial indigenous post-Norse contacts with Europeans around the dawn of the 16th century to the establishment of Moravian missionary settlements in the late 18th century. The early contact period of Labrador, and Inuit-European interaction, remains under-researched despite it being one of the most drawn-out European-Aboriginal contacts in the New World (Brewster 2005:3; Stopp 2002:97). Ethnography was not extensively pursued in the region before 1900 CE (Auger 1991:5).

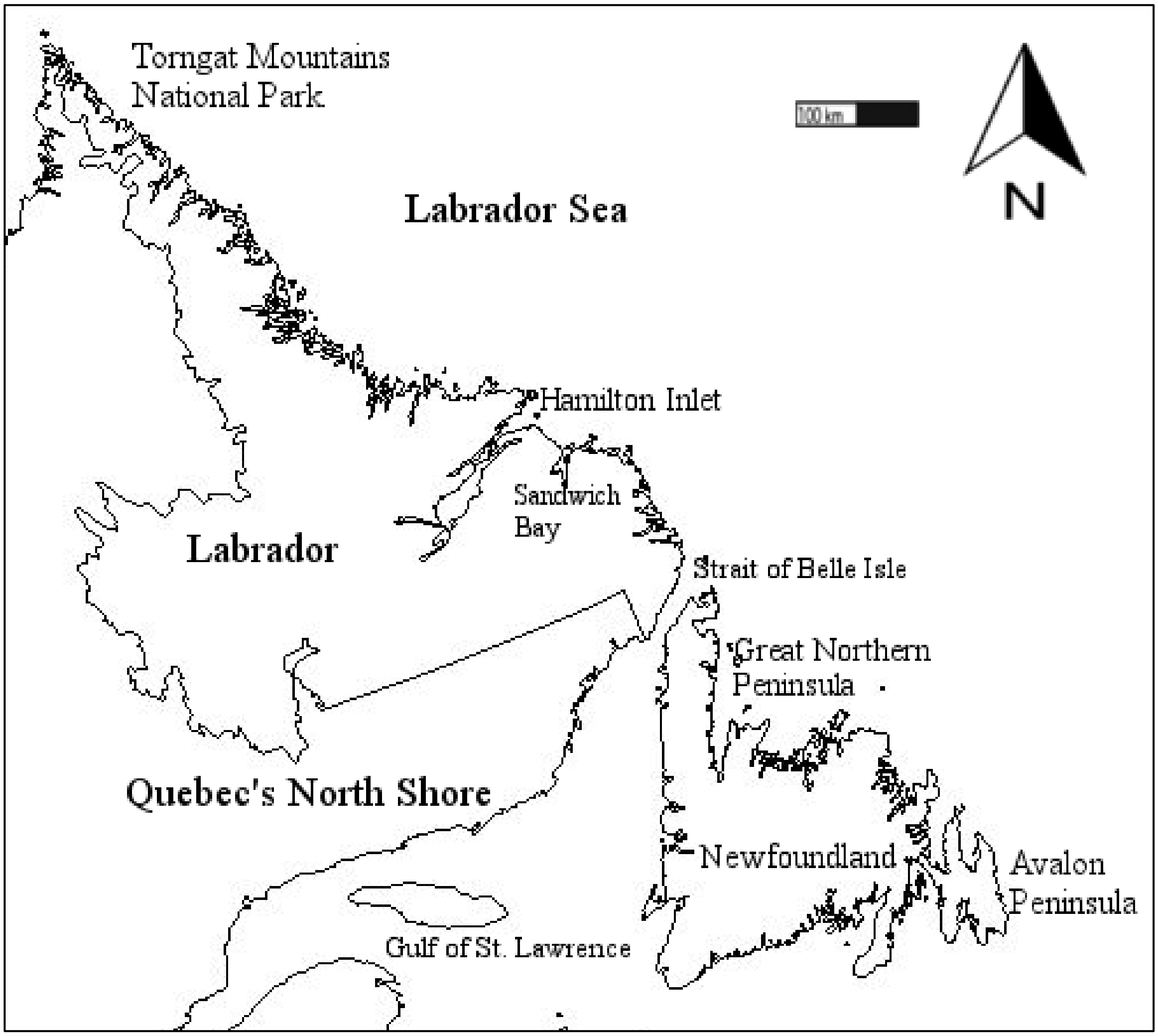

Fig. 1.1 Map of the study area.

This period of time was fraught with erratic and often volatile relations between the Inuit and various European groups eager to exploit the natural resources of the New World. Labrador Inuit archaeological sites along the coast have produced a great quantity of iron procured from European sources and tracing this iron to specific groups of Europeans can help us determine where the Inuit were targeting specific European populations for trading and raiding. The material exchange between European pioneers and the indigenous groups of Labrador was inconsistent and predominantly undocumented. Several different European groups where making contact with the Inuit during this period including the Basque, French, English, Dutch and Portuguese. These different groups had varying degrees of prominence and were more active at various times in the chronology of Labrador. The Basque were perhaps the most prolific European nationality active in Labrador in the early contact period. They were the first major European presence in Labrador over a prolonged period of time in the form of seasonal terrestrial shore stations and many of the iron nails from Inuit sites along the coast of Labrador in the early contact period are presumably of Basque origin.

1.3 Methodological Approach

One objective of this thesis is to examine whether it is possible to identify the source(s) of iron nails from early Labrador Inuit sites back to their original European source through geochemical and X-Ray techniques. The ideal strategy for accomplishing such a project involves a comprehensive analytical methodology that can detect the major elemental constituents of the uncontaminated, non-oxidized mineralogy of an iron nails' interior. These approaches are called solution-based inductively coupled plasma-mass spectrometry (ICP-MS) and scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDX). Both methods are destructive and irreversibly alter the artifacts being analyzed. They both also analyze a small fraction of the object under the assumption that a sample will provide results that will be representative of the overall geochemical constituents of the entire artifact. This analysis is therefore dependent on the artifacts' geochemical homogeneity in order to produce meaningful results and support arguments about the variation of the sample assemblage.

One of the objectives of this thesis is to test the effectiveness of ICP-MS and SEM-EDX in accomplishing this goal. The ICP-MS method proved flawed as the contaminated exterior of the artifacts factored into the analytical results. Establishing consistent geochemical signatures that are distinct to two or more different European groups and then attempting to correlate various artifacts derived from Inuit sites to these signatures is the objective of this thesis' methodology. If geochemical relationships can be established this research has the potential to refine our understanding of the nature of Inuit-European economic and cultural exchange in early historic Newfoundland and Labrador. A particular emphasis was placed to establishing a detectable Basque signature because of the well-documented prolonged Basque presence in southern Labrador in the contact period and the long-purported fondness that the Inuit held for Basque iron.

This research assesses the geochemical homogeneity of the iron artifacts at several different scales of resolution. Sample preparation methods allowed the SEM-EDX data to be broken down to the level of nail section or segments of a composite scan, while the ICP-MS samples can only be assessed at the level of artifacts in relation to sites. By approaching the data from these different scales of resolution, this research examines the degree of geochemical homogeneity contained in historic wrought iron nails and the degree of chemical relationships within the various levels of resolution that can be used to answer different research questions. Whichever of these scales show the most clustering in their data is theoretically the scale that would be the most geochemically homogeneous.

By contrast, heterogeneous data cannot provide meaningful evidence for the geochemical variation and consistency of the data set. Therefore, provenience analysis can only be successful if geochemical homogeneity is evident at the artifact scale. Since this research does not include the analysis of raw material samples, the main priority of this study is to assess the consistency among artifacts as opposed to trace artifacts back to their raw material source. Examples of homogenous materials that are commonly the subject of archaeological provenance include microcrystalline and cryptocrystalline igneous and metamorphic rocks like obsidian, basalt, soapstone and marble as well as cryptocrystalline sedimentary material like chert, chalcedony and clay (Rapp and Hill 2006). Other materials like amber and pigments as well as metals like gold, silver, copper, tin and lead are also sources of provenance data (Rapp and Hill 2006).

1.4 Thesis Overview

This thesis is organized into six major chapters that describe the purpose, procedure and results of this research. Chapter two details the cultural prehistory and protohistory of the coast of Labrador as well as the early modern metallurgical manufacturing process that created the artifacts being analyzed. The historic use of these objects as well as methodological issues in analytical techniques that arise when analyzing archaeological iron is explored in chapter three. The analytical preparation and procedure is outlined in chapter four with regards to both ICP-MS and SEM-EDX. Chapter five presents the analytical results of both methods through the use of biplot graphs and Backscattered Electron Images (BSE). The historic, methodological and geochemical implications and explanatory potential of the results are discussed in chapter six as well as concluding remarks and suggestions for further research.

Chapter 2: Historical Background

2.1 The Labrador Inuit

The Inuit people of the Labrador coast are considered to be the descendants of the Thule people who initially thrived in Alaska around 1000 CE (Rankin 2009:3). They are distinguished in the archaeological record by a bone-based material culture used to support their predominantly sea-mammal-based diet (Maxwell 1985:295; Rankin 2009:3-5). Around one millennium ago the Bering Strait was inhabited with an array of distinct hunter-gatherer cultures with the Thule concentrated on the northwestern coast of Alaska (Friesen and Arnold 2008:534; McGhee 2009:161). The demographic pressure in the area was likely one of the many contributing circumstances that motivated the Thule to begin radiating eastward after 1000 CE (Friesen and Arnold 2008:534; Jordan 1978:175). Conclusively determining the dates of this population movement has always been a provocative issue. Numerous problems involving radiocarbon dating and the viability of using absolute dating methods in the arctic make this date more of a contentious estimate. The obvious lack of tree-ring data in the arctic, the marine reservoir effect and the habit of arctic-adapted peoples to scavenge and recycle material culture, mostly driftwood, acquired from archaeological contexts dated to earlier time periods all contribute to the anomalous results in radiocarbon analysis in the arctic (Friesen and Arnold 2008:528; Nelson and Møhl 2003). As a result of these dating issues some recent research suggests a later date for this migration began closer to 1200 CE (Friesen and Arnold 2008:528; McGhee 2009).

Regardless of the date, the Thule began a rapid and large-scale migration across the Canadian arctic in the first centuries of the second millennium CE (Fig. 2.1). While

1000 CE is a common estimate for the launch of this migration, it is more likely that several waves of movement were undertaken over several centuries (Friesen and Arnold 2008:527, 536; Maxwell 1985:251-253). This migration was exacerbated by the *Medieval Warm Period* making marine resources available in the eastern arctic that were vital to the Thule livelihood (Friesen and Arnold 2008:535; Jordan 1978:176; Maxwell 1985:251; Woolett 2007:71). Apart from bowhead whaling, rumors of ample meteoritic and Norse iron in Greenland, perhaps traded via the Dorset, would have been added motivation for the migration (Friesen and Arnold 2008:535; Gulløv 2008:17; McGhee 2009:161-162). The pace of their easterly journey was brisk as evident by the presence of the Thule culture in southern Baffin Island and northern Greenland soon after 1200 CE (Dugmore et al. 2007:18; McGhee 2009; Rankin 2009:19).

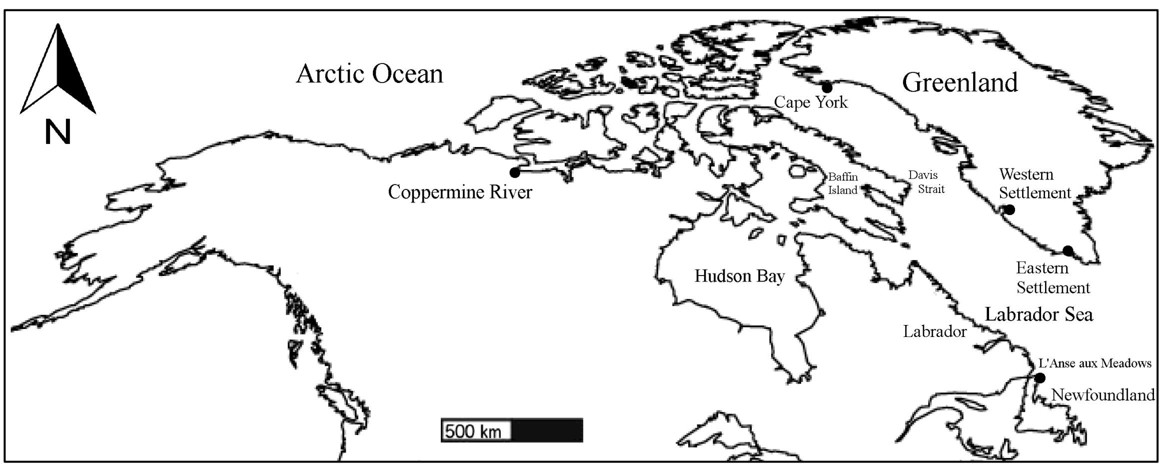

Fig. 2.1 Map of the Canadian Arctic.

Early estimates of the *Classic Thule* migration reaching the northern Labrador Peninsula from Baffin Island range from soon after 1350 to the 1400s CE (Butler 2011:59; Jordan 1978:175; Kaplan 1985:48; Woolett 2007:71). A prominent supply of European iron materials happened to become available on the south coast of Labrador soon after in progressively greater quantities. At the same time the Norse presence in Greenland was discontinuing, providing an alluring incentive for the Thule to make this move from the eastern arctic to Labrador (Fitzhugh 1985:32; Rankin 2009:19-26). This move caused the migrants to fracture into various regionally distinct *Modified Thule* groups that soon developed into the *Historic Inuit* culture (Rankin 2009:19; Woolett 2007:71).

The early material culture of the Historic Inuit of Labrador illustrates the lifestyle of a traditional hunter-gatherer culture. An impressive array of tools ideally adapted to the barren arctic landscape were used by the Inuit that included toggling harpoons, boats made of bone and hide called *umiaks*, as well as lithics predominantly fashioned from slate (Butler 2011:77; Cadigan 2009:20). Points, blades, ulus, knives, scrapers, arrowheads and harpoon heads were also shaped out of iron that the Inuit were acquiring well before they reached Labrador through Norse and meteoritic sources (Jordan 1978:176; Stopp 2002:97). The Inuit dependence on iron increased with time. Its accessibility on the southern coast of Labrador and north shore of Quebec combined with its obvious value as a prestige material provided opportunities for social stratification and aggrandizement that had important implications for the development of Inuit society in Labrador (Jordan 1978:184).

By the 15th century the Inuit began their initial colonization of the north coast of Labrador. The beginning of the 16th century marked a major progression southward along the coast of the peninsula incited by even greater amounts of Thule migrants from Baffin Island on the north coast and a desire for European iron and wood in the south (Butler 2011:61; Fitzhugh et al. 2011:122; Rankin 2009:26; Stopp 2002:72). The central and south coasts of Labrador were settled at the Thule's characteristically fast pace, likely displacing the *Late Dorset* culture and Amerindian peoples in the process (Brewster 2005:4; Cadigan 2009:20, 36; Fitzhugh 1985:26; Jordan 1978:176; Kaplan 1985:45; Rankin 2009:26). The Inuit may even have been openly hostile toward the Amerindians, whom they saw as competitors for access to European resources, compelling them to move away from the coast in favour of a mostly caribou-based terrestrial subsistence while the Inuit tended to settle in coastal sea mammal hunting areas (Brewster 2005:4; Cadigan 2009:37).

Hamilton Inlet, which marks the border between central and south Labrador was already permanently occupied by the Inuit before the end of the 16th century, but the acquisition of European materials had started earlier (Jordan 1978:181; Rankin 2009:20). This southward migration was highly motivated by the desire for European iron (Cadigan 2009:38; Stopp 2002:97). Hamilton Inlet was previously considered to be the frontier of year-round Inuit occupation, from which they dispatched annual scavenging parties south for iron (Jordan 1978:176). However, it seems more likely that southern Labrador was also the location of permanent settlements (Brewster 2005:5; Rankin 2009:28; Stopp 2002:97-98).

By the mid-1500s the Inuit were active on the south coast of Labrador, raiding and scavenging the seasonal Basque and French fishing and whaling stations during the winter months (Butler 2011:61-62; Rankin 2009:19-20; Rankin 2015). The year 1588 CE saw the first recorded instance of the Inuit crossing the Strait of Belle Isle to Newfoundland with the intention of entering into hostilities with local fisherman (Stopp 2002:76). Heavy resistance from Mi'kmaq groups and further expansion of Europeans in

Labrador likely prevented the Inuit from expanding their permanent colonies further south into the Gulf of St. Lawrence, making Labrador and the north shore of Quebec the most southern extent of the Inuit world (Brewster 2005:2; Taylor 1979:49, 55).

The 17th century marked the beginning of gradual cultural change among the Labrador Inuit, perhaps in response to the proliferation of European iron resources (Woolett 2007:69). Large communal houses became more prevalent, progressively supplanting the smaller style of dwellings in the 18th century (Jordan and Kaplan 1980:42; Rankin 2015; Schledermann 1976:32). The development of increased social stratification and aggrandizers with abundant material wealth may have propagated these more amalgamated dwellings. As with the onset of the *Little Ice Age* of the 15th to 19th centuries the consolidation of resources may have become even more significant (Friesen and Arnold 2008:534; Jordan 1978:184; Mann 2002:504; Schledermann 1976:35; Woolett 2007:71). However, communal houses may have resulted from the need to incorporate single women into households as many men were killed while attacking and raiding French fishing stations (Kaplan 1985). Regardless, these changes were greatly exacerbated by the introduction of the European iron in southern Labrador that continued until the establishment of the Moravian missions of northern Labrador in the late 18th century, which profoundly altered the economy of the Inuit (Cadigan 2009:68).

The indigenous peoples of the Canadian arctic were familiar with metals well before they started interacting with Europeans. The Coppermine River on the shore of the Coronation Gulf of the Northwest Territories provided the Inuit of that area with an ample source of copper ore. The strength and durability of copper made it superior to slate but inferior to those chemical properties in iron (Morrison 1987). The geographic exclusivity of this copper source in the western arctic also made iron all the more popular to the Inuit of the eastern arctic. The peoples of the western arctic may have also acquired metals from Asia exchanged via the Bering Strait (McGhee 2009:161; Pringle 1997:766-767).

The Inuit desired the durability and strength properties of iron material which they would adapt and cold-hammer into traditional forms such as harpoon heads (Rankin 2009:11). A potential source of naturally occurring iron ore in the eastern arctic would have been the telluric iron of Disko Island in Greenland. Naturally occurring solid iron is extremely rare and Disko Island is one of the few locations in the world where it can be found (Smith 1966:16). These basaltic inclusions were formed in a lava flow and range in size from small pebbles to large boulders (Buchwald 2005:35; Craddock 1995:101; Smith 1966:16). The Inuit would extract small pieces of these inclusions from the basalt and cold-hammer them into small flat implements (Craddock 1995:103). Telluric iron varies widely in its carbon and nickel content (Craddock 1995:102).

The Inuit also derived a great deal of their iron material from meteorites discovered in Cape York, Greenland (Rapp 2009:168). This area, known as *Perlernerit* to the local Inuit, was the home of three large meteorites referred to as the *tent*, the *dog* and the *woman* by the native Greenlanders (Craddock 1995:103; Henderson 2000:211). Collectively the three meteoritic fragments account for approximately 34.5 metric tons of iron and nickel that broke off from a 200-ton meteor that impacted northwestern Greenland sometime in the last 10,000 years (Buchwald 2005:20; Huntington 2002:53; Pringle 1997:767). The high nickel content in the meteoritic iron makes in very workable and visually distinct with its *Widmanstätten* geometric pattern (Rapp 2009:168).

Harpoons, knives and scrapers were fashioned from pieces derived from these meteorites through cold-hammering as the Inuit did not possess the expertise or sufficient fuel necessary for smelting metal. Cape York was the center of a large trade network that may have reached as far as 2,200 km away (Huntington 2002:56; Pringle 1997:766-767). European settlements, however, would eventually provide the most abundant and reliable source of iron and other metals in the eastern arctic. The Inuit would have acquired metal from a variety of different European groups during this time. Among those, the Norse settlements in southwestern Greenland were likely prominent sources before they eventually disbanded in the 15th century, which coincides chronologically with the Inuit entering Labrador and encountering new sources of European iron (Rankin 2009:24).

About a dozen seasonal Basque and French whaling stations on the coast of southern Labrador were developed in the early 16th century, providing a consistent and abundant source of metal for the indigenous peoples of the region. The Inuit dispatched scavenging expeditions to these whaling stations to procure iron objects, some of which needed to be acquired by destroying the infrastructure (Wolfe 2013:22). The Inuit then cold-hammered the scavenged iron into various tools (Cadigan 2009:36; Logan and Tuck 1990:67; Stopp 2002:97; Wolfe 2013:112). The Inuit appear to have favoured a strategy of complete avoidance of the shore stations until the Basque traveled back to Europe in the autumn because direct interaction almost always escalated to the point of bloodshed (Barkham 1984:518; Stopp 2002:82).

Inuit living in the vicinity of the Basque and French whaling stations would also exchange iron with Inuit living a greater distance from the south in a linear trade distribution up the coast (Fitzhugh 1985:32; Jordan 1978:184; Rankin 2009:25; Rankin 2015). During the late 18th century these Labrador Inuit trade networks broke apart with the establishment of Moravian missions in northern Labrador as part of the English policies of geographically isolating the Inuit to prevent them from attacking and distracting fishing crews (Auger 1991:10-11). The English government's position towards the Inuit during this period was to drive them north toward Moravian settlements where conversion and trade with the Inuit was encouraged (Auger 1991:10-11).

The early contact period in Labrador was a time when iron was increasingly abundant to the Inuit from an expanding array of different European groups like the Portuguese, Basque, Dutch, French and English. Iron was economically significant to the Labrador Inuit throughout their history and its presence in the archaeological record is indicative of the prevalence of European activity in the region over time. The prospect of establishing the presence of relationships between Inuit material culture and specific European groups based on the compositional correlations of iron artifacts could therefore provide meaningful insights of the early history of Labrador. Well before the early contact period, however, the first exposure that the Inuit of the eastern arctic had with European resources came from the Norse five centuries prior to the dawn of whaling and fishing activity in Labrador.

2.2 The Norse

The Norsemen from Scandinavia were the first European presence in the arctic and the entirety of the Americas in the 10th century CE. An Icelandic historian named Ari Þorgilsson the Learned wrote a document describing their activities in the New World several centuries later called *Íslendingabók* (Dugmore et al. 2007:14). It details the exploits of the Icelandic chieftain Eirík Þorvaldsson the Red and his initial colonization of *Grœnland* in 986 CE (Lynnerup 1996:122). The Nordic expansion west across the

Atlantic had started several years earlier with the settlement of the Faroe Islands and Iceland by Norwegian migrants in the early 9th century (Dugmore et al. 2007:13). The politically stifling atmosphere of Iceland made it difficult for aspiring aggrandizers to found a chiefdom of their own.

Therefore, after surveying the coast of Greenland for several years, Eirík the Red departed Iceland with several dozen ships carrying hundreds of settlers (Cadigan 2009:27; Dugmore et al. 2007:16-17; Lynnerup 1996:126). Two major areas of Greenland's southwest coast were colonized. They are now known as the Eastern and Western Settlements. This region was largely void of native groups including the Thule, whose migration would not reach Greenland for two centuries (Lynnerup 1996:122). From these colonies the Norse explored further west, making it to *Helluland* in Baffin Island and soon after encountering Labrador, which they refer to as *Markland* in historic documents (Fitzhugh 1985:28; Sutherland 2009:281). There is insufficient proof that *Markland* was ever colonized; instead Eirík's son Leif the Lucky likely bypassed Labrador on his way to *Vínland* to found the brief and largely unsustainable colony called *Leifsbuðir* at L'Anse aux Meadows, Newfoundland (Lynnerup 1996:133; Rankin 2009:15; Sutherland 2009:280).

In Greenland, the Norse maintained their Scandanavian cultural customs and economic practices. The fjord-riddled coastal landscape of southwestern Greenland is almost identical to the environments in Northern Europe to which the Norse were well-adapted (Cadigan 2009:27; Lynnerup 1996:122). Subsistence was largely derived from the *landnam* farm system that managed groups of domesticated animals called *sætters* (Lynnerup 1996:131). They supplemented this by dispatching expeditions to an area of northern Greenland called *Norðrsetur* to hunt seal, walrus and caribou (Appelt and Gulløv 2009:304; Cadigan 2009:27; Gulløv 2008:19-20). Ivory and meteoritic iron were also important motivations for these excursions (Gulløv 2008:19-20).

The 13th century witnessed the Thule movement into Greenland and displacing the Dorset culture (Dugmore et al. 2007:18). Thule Inuit were acquiring European materials throughout the Norse occupation as demonstrated by sites at Disko Bay on the northwest coast of Greenland where quantities of Norse metal and other artifacts were found. It was a possible place of trade between the Norse and Thule peoples (Dugmore et al. 2007:19; Fitzhugh 1985:30; Gulløv 2008:21-22). Norse iron has also been found as far away as Ellesmere Island and Baffin Island (Gulløv 2008:20; Maxwell 1985:308; Sutherland 2009:280). Despite there being clear evidence of material exchange between the two cultures, the Norse left Greenland having had very little lasting effect or economic interdependence with Inuit culture in the eastern arctic (Cadigan 2009:26; Fitzhugh 1985:36; Gulløv 2008:19-22).

Several reasons contributed to the decline and eventual abandonment of the Norse presence in Greenland. The onset of the *Little Ice Age* in the 14th century made the Greenlandic Norse livelihood increasingly difficult (Dugmore et al. 2007:12; Lynnerup 1996:133). The Norse settlers were predominantly farmers, not Viking warriors, so violent encounters with the New World's *skrælingar*, such as the Inuit, often had devastating results for them (Dugmore et al. 2007:19; Fitzhugh1985:30; Mitchell 2013:320). Iceland was heavily affected by the plague in the early 15th century, creating fresh prospects for dissatisfied Greenlanders (Dugmore et al. 2007:13; Lynnerup 1996:133). The accumulation of all these variables resulted in the western settlement being abandoned in the mid-14th century and a similar fate occurring to the eastern settlement in the early 15th century, with the last historic record of the Norse setting foot in Labrador in 1347 CE (Cadigan 2009:28; Dugmore et al. 2007:13; Fitzhugh 1985:28; Lynnerup 1996:122; Sutherland 2009:282).

2.3 The Portuguese

The Kingdom of Portugal expressed a very early interest in the resources of Labrador that was primarily motivated by competition with England. The Portuguese government perceived the English financial backing of Italian explorer Giovanni Caboto's voyage to Newfoundland as a threat to their newly acquired sovereign domains in the New World promised by Pope Alexander VI in the 1494 Treaty of Tordesillas (Cadigan 2009:31; Major 2001:44). In response, King Emmanuel I of Portugal dispatched a series of expeditions of his own with the purpose of surveying and enforcing his land claims. The Portuguese state's investments in exploring and exploiting the fishery of the coast of Labrador provided a relatively short period of dominance over the English in Atlantic Canada in the earth 16th century even though the Portuguese were still subordinate in the region to their counterparts from Spain and France (Cadigan 2009:32).

Portuguese explorers made numerous excursions to the coast of Labrador in the early 16th century and frequently attempted to interact with its native inhabitants. Gaspar Corte-Real was one of those pioneers commissioned by the Portuguese government to chart areas of Atlantic Canada with the added incentive of potential stewardship over those regions (Cadigan 2009:31; Trudel 1981:140). Despite the disappearance of Gaspar Corte-Real in his 1501 voyage and the subsequent loss of his brother Miguel Corte-Real in a failed rescue mission the following year, substantial areas of the New World were successfully surveyed, including the coast of Labrador (Cadigan 2009:31; Major 2001:45; Trudel 1981:140-141). These excursions included extensive contact and capture of the native peoples of Atlantic Canada described by the Portuguese as "rather human", relatively hospitable and having previously been in possession of European objects as of the beginning of the 16th century (Major 2001:43; Trudel 1981:140-141). Other Portuguese explorers active in the early 16th century, such as João Alvares Fagundes and Labrador's namesake João Fernandes the *Lavrador,* also made efforts to survey and claim land in and around Labrador (Cadigan 2009:31; Gosling 2006:54; Major 2001:45). Despite its initial enthusiasm, Portugal's interests in Labrador proved to be fleeting by the late 16th century as Lisbon neglected the fisheries in the North Atlantic in favour of more worthwhile economic prospects abroad in South America, Asia and Africa (Cadigan 2009:31-32).

2.4 The Basque

Perhaps the most active European presence, and presumably the most likely nationality to provide iron to the Inuit of the early contact period, were the Basque. The Basque people, or *Euskaldunak,* are members of a distinct European ethnic group that exists on the Franco-Spanish frontier where the western extent of the Pyrenees mountain range meets the Bay of Biscay (Loewen and Delmas 2012:214). The Basque have a distinct language called *Eskuara* and call their nation *Euskalerria* while the English-speaking world refers to it as Basque Country (Proulx 2007a:25; Ross 1985:1). Their territory is traditionally comprised of seven provinces that were conquered by larger Iberian and Gallic kingdoms in the 13th century (Loewen and Delmas 2012:214). Of these seven provinces, three are seaside with their major ports of Bilbao, San Sebastián, Bayonne and Saint-Jean-de-Luz having established their economic prosperity through the Castilian wool trade since the 11th century (Barkham 1984:517; Loewen and Delmas 2012:217). By the 16th century the Basque became renowned throughout Europe for their shipbuilding and metallurgical industries: two characteristics that made them capable whalers and fishers throughout the late medieval period (Logan and Tuck 1990:66; Proulx 2007a:30). Their ports had their separate provincial territories that contributed to their economic prosperity and the lucrative whaling *galeón* shipbuilding and outfitting industry (Loewen and Delmas 2012:220-221).

Basque provinces had dissimilar economies in the 16th century that had a direct impact on their differing levels of participation in the transatlantic whaling business. The most eastern and the only French-Basque maritime province is called Lapurdi, and its major ports are Bayonne and Saint-Jean-de-Luz. It lacked the shipbuilding and mineral extraction prowess of the other two provinces but compensated for it though agricultural output. A great deal of the provisions the Basque galleons brought with them on their whaling excursions were from Lapurdi (Loewen and Delmas 2012:219). Because of their less developed iron and shipbuilding trades, the Lapurdi province built smaller galleons and was less prominent in the Gulf of St. Lawrence than their Spanish-Basque counterparts in the 16th century (Barkham 1984:518). By contrast, the most western of the major maritime ports was Bilbao, capital of the Bizkaia province, which enjoyed a more profitable economy based on exporting wool and iron. Bizkaia lacked the infrastructure to support a large shipping industry in the 16th century (Loewen and Delmas 2012:219).

The San Sebastián port in the central maritime Basque province of Gipuzkoa was inarguably the most dominant power among these three factions in the early modern period. Gipuzkoa had the infrastructure, the shipbuilding industry, and the commercial footing to support a substantial international trade network and large whaling enterprise (Loewen and Delmas 2012:219). One of San Sebastián's adjacent ports called Pasaia was the preferred harbour for the Basque for a variety of reasons. Over half of the whaling fleet would dock there to offload their cargo and outfit their voyages to the New World (Barkham 1984:519; Loewen and Delmas 2012:219). San Sebastián was also the headquarters of the bureaucratic body that regulated the distribution and armament of the Spanish-Basque whaling fleet (Loewen and Delmas 2012:223).

Iron production and shipbuilding were highly lucrative industries in the early modern period and contributed the most to the economic prosperity and whaling dominance of the Basque Country in the 16th century (Douglas and Bilbao 1975:67; Light 1992:249). The Basque dominated the European iron market in the late medieval and early modern periods with as much as ninety percent of England's iron imports coming from Basque Country (Childs 1981:46). Basque Country's advantageous location along the Pyrenees Mountains gave it a substantial mineral wealth of several types of metal deposits, predominantly iron, that they have been extracting since Roman times (Monna et al. 2004:197-199). Along with its ore, the Basque Country's ironsmiths also exported high-quality industrial tools, nails and early modern weaponry (Barkham 1984:517). Iron production escalated in the late 16th century to support the golden age of the whaling industry and to keep up with the demand for Basque metallurgy in the European markets (Monna et al. 2004:210).

The Basque shipbuilding industry intensified in the late 15th century when Queen Isabella I of Castile started to subsidize the construction of *galeóns* in her newly consolidated Iberian kingdom (Douglas and Bilbao 1975:68). In the years following Christopher Columbus' "discovery" of the Americas these shipbuilding subsidies served the dual purposes of ensuring the exploitation of the New World's resources and expanding the Spanish armada's naval forces (Loewen and Delmas 2012:223). These galleons were well equipped for warfare if required as they weighed several hundred tons, were crewed by as many as 130 men and were often armed (Barkham 1984:517; Cadigan 2009:32; Proulx 2007b:50). This shipbuilding superiority gave the Basque a substantial advantage over other European groups when it came to whaling in the New World and gave them a quasi-monopoly on fishing and whaling in the Gulf of St. Lawrence in the 16th century (Waddell 1986:137). Basque vessels were also the most abundant group represented in Europe's trade routes with the West Indies (Barkham 1984:519).

Their aptitude in these shipbuilding and ironworking permitted the Basque to dominate the extremely profitable early modern whale oil market (Logan and Tuck 1990:66; Proulx 2007b:77). With the 12th century witnessing the beginning of their whaling industry in the Bay of Biscay the Basque were revered across Europe as some of the most capable and skilled whalers in the world by the 16th century (Barkham 1984:515; Douglas and Bilbao 1975:53). Whale oil was used in a variety of objects like lanterns, soap and medicines (Loewen and Delmas 2012:217). Its most profitable purpose was as an industrial lubricant for the textile trade with most of the Basque's whale oil being exported to the textile industries of Europe and other Spanish territories (Loewen and Delmas 2012:17). Financial backing for these annual whaling ventures usually came from the wealthy proprietors of shipyards and iron mines as well as an early version of an insurance industry (Barkham 1984:517; Proulx 2007b:50).

The watershed moment for the Basque whale oil industry was the late 15th century discovery of what the Basque called *Terranova*, which had an almost immediate and extremely beneficial effect on the Basque economy (Douglas and Bilbao 1975; Ross 1985:1). The Basque were by no means alone in their presence in the New World with France, England and Portugal reaching the Gulf of St. Lawrence and the Strait of Belle Isle either before or at the same time as the Basque in the early 16th century (Barkham 1984:515; Cadigan 2009:33; Waddell 1986:137). All of these European political entities performed varying degrees of cod fishing and whaling operations along with the Basque in the Gulf of St. Lawrence (Fitzhugh et al. 2011:103). By the 1530s the Basque were quickly overtaking their European competitors to dominate the whaling industry in *Terranova* (Barkham 1984:515; Rastogi et al. 2004:1648).

The remainder of the 16th century saw the Basque benefit economically from their labours in the Gulf of St. Lawrence, which the Basque knew as the Grand Bay (Fitzhugh et al. 2011:100). Dozens of seasonal shore stations were visited annually by the Basque with thirteen of them located along the coast of southern Labrador (Loewen and Delmas 2012:228). This period of prosperity was briefly interrupted after the outbreak of a Franco-Spanish war between 1553 to 1559 that spilled over into Labrador as French and Spanish Basque factions attacked and raided each other's galleons and shore stations for whale oil as several documents detailing lawsuits from this period claim (Barkham 1984:518; Loewen and Delmas 2012:219). Once peace broke out in 1559, the consequences of this conflict included the proliferation of armaments on whaling galleons from the 1560s to the 1580s and the domination of the Grand Bay by the Spanish-Basque (Barkham 1984:519). The next several decades saw the pinnacle of Basque activity in the

Grand Bay and southern Labrador with as many as thirty galleons being dispatched to *Terranova* annually (Doroszenko 2009:510; Fitzhugh et al. 2011:100).

The majority of whaling activity in southern Labrador was concentrated at a site they called *Buitres* (Loewen and Delmas 2012:223). Today this site is more commonly called Red Bay after the cliffs comprised of red granite common in the region (Barkham 1984:516). Pack ice was a common hazard in the Strait of Belle Isle between December and June so the Basque would leave Europe for Red Bay in June and early July (Logan and Tuck 1990:66; Ross 1985:1). They would return from Labrador in the autumn with as many as 9,000 barrels of whale oil with each ship being able to store 1,000 barrels in its hold (Barkham 1984:516; Fitzhugh et al. 2011:100). While in Labrador the Basque would supplement their rations with the abundant fauna provided by the sub-arctic coastal tundra around Red Bay, which included fish, caribou and fowl (Barkham 1984:517: Logan and Tuck 1990:66).

While living aboard their *galeóns* the Basque would conduct most of the industrial activities necessary to process cetacean remains into oil on land (Fitzhugh et al. 2011:124). These seasonally inhabited terrestrial shore stations served many purposes related to the butchering and rendering of whale blubber and commonly appeared as open-sided wooden structures called *cabañas* that were roofed with curved tiles made out of red clay (Fitzhugh et al. 2011:100; Proulx 2007b:66). Once a team aboard a smaller vessel called a *chalupa* had successfully hunted a North Atlantic right whale (*Eubalaena glacialis*) or Greenland bowhead whale (*Balaena mysticetus*), the Basque would tow the carcass back to the shore station and proceed to *flense* it or remove the skin and blubber (Cadigan 2009:32-33; Logan and Tuck 1990:66; Proulx 2007b:68-71). The flensed blubber was then taken to an area called the *tryworks* where masonry ovens called *hornos* boiled the blubber in large copper rendering cauldrons (Loewen and Delmas 2012:232; Logan and Tuck 1990:68; Ross 1985:1; Proulx 2007b:72-73). A separate structure was reserved for coopering *barricas* to store the finished product of this rendering process (Loewen and Delmas 2012:232). As many as ninety barrels of oil could be culled from a single animal (Proulx 2007b:73).

Historic documents concerning Red Bay exist from 1536 and mostly relate to legal and financial matters while neglecting the procedural aspects of a whaling station (Barkham 1984:516; Logan and Tuck 1990:66). James Tuck of Memorial University of Newfoundland and Robert Grenier of Parks Canada conducted extensive terrestrial and maritime excavations of the Red Bay site in the 1970s and 1980s in order to account for this deficiency in historic records (Doroszenko 2009:510). Tuck excavated several areas of the terrestrial shore station while Grenier concentrated most of his efforts on exhuming several *chalupas* and the Basque whaling galleon *San Juan* that sank in the autumn of 1565 with an ample cargo aboard before it could return to Pasaia (Barkham 1984:516; Ross 1985:1; Waddell 1986:137). Tens of thousands of artifacts were gained from these excavations including tiles, glass, ceramics and whale remains (Barkham 1984:516; Fitzgerald et al. 1993:46; Loewen and Delmas 2012:228; Rastogi et al. 2004:1648). The moist environment of Labrador was ideal for preserving organic materials like wood, clothing, and faunal remains (Fitzgerald et al. 1993:46; Loewen and Delmas 2012:228; Logan and Tuck 1990:66). Iron artifacts like nails, spikes, harpoons, lances, casks, coins, ladles, weaponry, barrel hoops and whaling equipment are also abundant in the Red Bay collection (Fitzgerald et al. 1993:46; Loewen and Delmas 2012:228; Proulx 2007b:52; Ross 1985:8).

The Basque relationship with the indigenous peoples of Labrador was complicated. According to historic sources there is every reason to believe that the Basque enjoyed predominantly amiable interactions with the Innu First Nations people (Cadigan 2009:37; Logan and Tuck 1990:71). Opinions regarding the Inuit were not as favourable (Auger 1991:15). As one Basque historian wrote in 1625, the Basque encountered people "called *Eskimaos*, who are inhuman, because they suddenly attack our men with their bows and arrows (with which they are very dexterous) and kill and eat them" (Barkham 1984:518). This biased interpretation of early modern Basque-Inuit relations illustrates a general tendency for the Inuit to be hostile toward Europeans when they were not refraining from contact all together (Fitzhugh et al. 2011:102). Their desire for European metals was also one of the major reasons why the Basque preferred to not leave caches of expensive equipment like the copper rendering cauldrons in Labrador and instead brought them back to Basque Country each year (Logan and Tuck 1990:66-67).

Several environmental, political and economic factors contributed to the eventual decline of the Basque whaling industry. King Philip II of Spain heavily taxed the Basque whaling industry to help finance a naval invasion of Queen Elizabeth I's protestant England in 1588 (Cadigan 2009:33; Proulx 2007a:36). This financial burden followed the 1572 bankruptcy of an insurance company in Burgos, Castile, which resulted in a lack of capital necessary to sponsor whaling expeditions, and a 1579 English embargo on Spanish oils, which was caused by an increased anti-Catholic fervor (Loewen and Delmas 2012:224). Since the Spanish-Basque fleet was in the Spanish naval reserves many whaling galleons were recruited into the Great Armada that was devastated in the failed invasion of England in 1588 (Fitzhugh et al. 2011).

After the annihilation of much of the Spanish-Basque fleet, the Dutch, French and English transatlantic fishing, whaling and fur-trading industries proliferated in the Gulf of St. Lawrence and Labrador (Barkham 1984:518; Cadigan 2009:33; Fitzgerald et al. 1993; Fitzhugh et al. 2011:123; Loewen and Delmas 2012:240). By then overhunting of the right and bowhead whales had devastated their North Atlantic populations (Rastogi et al. 2004:1647). The climate was also becoming significantly cooler by the 17th century during a period called the Little Ice Age resulting in more hostile whaling conditions (Proulx 2007a:36; Woolett 2007:72, 81). As a result the Basque and their European rivals began favouring Scandinavian waters for their whaling and fishing grounds (Fitzgerald et al. 1993:46; Loewen and Delmas 2012:217; Proulx 2007a:35). The Spanish government also began reallocating much of their remaining Basque fleet from *Terranova* to their more lucrative trade routes in the West Indies (Fitzhugh et al. 2011:102; Loewen and Delmas 2012:224). The Basque would never again be as prevalent or dominant in Atlantic Canada as they were in the 16th century, but the Basque may be the most prevalent European source of iron represented in early contact period Inuit Sites.

2.5 The Dutch

The Netherlands began conducting whaling, fishing and trading activities off the coast of Atlantic Canada in the early 17th century. The *Northern Company* was the primary Dutch proprietor of intermittent commercial ventures in the vicinity of Davis Strait after the expedition of the Dutch cartographer Jorus Carolus mapped the coast of Labrador and adjacent shores in 1616 (Kaplan 1985:55; Stopp 2002:75-76). Inadequate historical records prevent all but a rudimentary understanding of the exact nature and extent of Dutch enterprises in the North Atlantic in the early contact period (Auger 1991:8). These economic endeavors likely did not ever advance to the construction of permanent or temporary terrestrial settlements in Atlantic Canada or Greenland (Kaplan 1985:55).

Whenever the opportunity arose the Dutch would supplement their fishing and whaling activity with trading commodities with the native population. The Dutch would acquire ivory, baleen, and animal hides in exchange for European goods desired by the Inuit along all of the shores encompassing Davis Strait (Auger 1991:8; Kaplan 1985:55). These transactions were not a routine occurrence and Dutch-Inuit relations were often fraught with the same antagonism that commonly accompanied Inuit interactions with other Europeans (Cardigan 2009:38). While Dutch officials promoted the economic opportunities to be had in the Davis Strait they often cautioned mariners against the well-known hostile nature of the native population (Kaplan 1985:55). Jorus Carolus himself had uncomplimentary things to say about the Inuit in 1634:

> The natives of this land on both sides of the Strait are altogether heathens and wild cannibals. One should not believe their friendly looks. All that they want to trade they tie to the oar with which they want to paddle their canoes. They do not trust anybody and therefore they can also not be trusted (Carolus cited in Auger 1991:8).

On those brief occasions when Dutch-Inuit relations were mutually beneficial they never advanced beyond catering to the material desires of both parties which presumably may have included the exchange of iron nails (Kaplan 1985:55).

2.6 The French

French-Inuit relations along the coast of Labrador date back to the early expeditions in search of the Northwest Passage when it was common for Europeans to acquire baleen, ivory, oil and animal skins from the Inuit on their long journeys (Brewster 2005:4; Rankin 2013b:314; Stopp 2002:75). This practice continued as several maritime French regions like Normandy, Brittany and Gascony started to exploit the fisheries off of Labrador or *Terre-Neufsve* in the 16th century (Major 2001:51; Rankin 2013b:310). In 1713 the French presence in Labrador increased considerably after the Treaty of Utrecht prevented them from profiting from Newfoundland fisheries (Kaplan 1985:58; Rankin 2013b:314). The French presence on the north shore of Quebec and the Strait of Belle Isle rose exponentially as a result of an increased quantity of land concessions given to the French fishery by the government of France (Trudel 1981:316). The French constructed early permanent settlements in Labrador, like Chateau Bay, and the demand for fishing berths was so great that French authorities had to mitigate competition between fishermen by assigning specific harbours to crews (Kaplan 1985:58).

The French generally had a similar attitude towards the Inuit as every other European nationality. A complicated mixture of violence occasionally interrupted with instances of trade dominated interaction between them (Mitchell 2013:321). The French actively sought viable economic relationships with the Inuit who would engage in peaceful commerce, as but many Inuit employed antagonistic tactics to acquire highly sought after European goods including iron that may be represented in the archaeological record (Kaplan 1985:58; Martijn 2009:79; Mitchell 2013:321). Inuit raiding parties and the destruction of shore stations were so common that the French authorities often had to intervene with aggressive force, provoking further retaliation by the Inuit (Kaplan 1985:58; Major 2001:73; Martijn 2009:79; Mitchell 2013:321). Despite this largely unstable rapport the early 18th century witnessed some examples of amicable relations due to the efforts of settlers like Jean-Louis Fournel (Auger 1991:10; Kaplan 1985:58).

2.7 The English

The English have been active in the Atlantic Canada since the early 16th century and they eventually supplanted the French as the major European influence in Labrador by 1763 when the Treaty of Paris was ratified (Kaplan 1985:62; Mitchell 2013:321; Rankin 2013b:316). The English did not fare any better in their dealings with the Inuit of Labrador than any other European group that previously interacted with them. Indeed there was a spike in both the frequency and intensity of violence between the Inuit and Europeans in the years immediately following the treaty (Mitchell 2013:321-324). The Inuit tactics for acquiring European goods without having to trade were getting more sophisticated and aggressive, and the English correspondingly escalated its naval presence along the coast (Mitchell 2013:321-324). Both the Inuit and the English perceived each other with suspicion and contempt (Mitchell 2013:324).

In order to combat the deteriorating situation in Labrador the newly appointed Governor of Newfoundland, Sir Hugh Palliser, enacted reforms to pacify the Inuit and alienate the French for the purpose encouraging the English fishing industry (Martijn 2009:81). Governor Palliser drafted the Peace and Friendship Treaty that was ratified by him and Inuit emissaries in Chateau Bay in 1765 (Mitchell 2013:323). This truce emphasized a trade agreement with the Inuit and cessation of violence on the part of the English in exchange for the Inuit cutting trade links with the French and limiting their territory to northern Labrador (Auger 1991:10; Kaplan 1985:64; Mitchell 2013:323-324). Palliser also put a prohibition on any permanent settlement of Labrador by any Europeans in order to negate French interests off of its shores, with the exception of the English Fort York in Chateau Bay (Auger 1991:10; Cadigan 2009:66; Kaplan 1985:64; Rankin 2013b:310). Due to the loss of revenue resulting from this Inuit-European segregation, the treaty was not effectively executed and aggressive confrontations did not cease (Mitchell 2013:324-325; Rankin 2013b:316).

Ultimately it was the efforts of the Moravian missionaries sponsored in the 1770s by Palliser and his successor Baron Molyneux Shuldham that effectively ended the cycle of violence (Auger 1991:10; Mitchell 2013:324). The Moravians derived from a Czech protestant group that called itself the *United Brethren* and they had plenty of experience in converting and trading with the Inuit of Greenland. The Government of Newfoundland endorsed their efforts to help nullify the Inuit threat to the English fishery (Auger 1991:11; Rankin 2013b:316). When Nain was founded by the Moravians in 1771 it became the first European colony in northern Labrador and drastically changed the dynamics of European-Inuit relations by marginalizing traditional Inuit trade routes, encouraging settlement at the trading stations and religious conversion (Cadigan 2009:68; Kaplan 1985:64; Woolett 2007:72). The establishment of other British settlements such as the 1775 trading post of George Cartwright in Sandwich Bay also improved relations, but also brought Labrador and the Inuit under increasing centralization and helped incorporate the Inuit into a European lifestyle and material culture (Mitchell 2013:325; Rankin 2013b:316). Many of the iron nails present in historic Inuit sites could conceivably be from these Moravian trading posts.

2.8 Conclusion

The Inuit of early contact period Labrador were exposed to interactions with an array of different European nationalities with varying degrees according to their activity in Atlantic Canada. This provided the Inuit with plenty of opportunities to raid, scavenge or trade for European commodities like iron from any one of several Old World sources. Because of the iron's large range of possible origins, the ability to distinguish which nails were derived from which nationality through analytical chemistry could greatly improve our understanding of what European groups were more prolific in Labrador and the nature of their interactions with the Inuit. This analysis assumes that the geochemical signatures of the iron artifacts derived from these different European groups are sufficiently different enough to distinguish regional sources of raw ore or manufacturing centers. However, as the next chapter will explain, there are several issues that arise when attempting to perform a geochemical and X-ray provenance analysis of metallurgy.

Chapter 3: Archaeometallurgical Background

3.1 Iron Mineralogy

Iron is one of the most abundant metals found in nature, but it is seldom found in a pure form as it easily merges with many other varieties of elements to usually form dark-coloured veins of carbon and sulfur-rich minerals such as hematite, magnetite, limonite, siderite and goethite (Buchwald 2005:63; Henderson 2000:211, 214; Rapp 2009:166). The melting temperature of pure iron is approximately 1558°C, but this number varies depending on the nature of the impurities in the iron (Buchwald 2005:63; Craddock 1995:235). The metamorphosis of the crystal structure or *allotropy* is a major property of iron chemistry that affects the chemical nature of the product of the manufacturing process. Ferrite (α-Fe) is the cold, strong and stable form of iron while austenite (γ-Fe) is the malleable, non-magnetic, easily forged and easily chemically bonded state of iron created at ~912°C (Buchwald 2005:63-66). Several elements are routinely found in raw iron minerals such as oxygen, silicon, sulfur, carbon, manganese, phosphorus, calcium, aluminum and titanium with manganese and manganese oxides being particularly prevalent (Buchwald 2005:63; Frurip et al. 1983:11; Rapp 2009:166-167). Historically iron was frequently combined with carbon to form steel but that alloy was not heavily exploited for use in nails until the 19th century (Wells 1998:80-81).

The raw mineral form of iron has the greatest geochemical legacy on the finished artifact and high manganese content is ubiquitous with iron ore sources in Western Europe including those in the Pyrenees (Leroy et al. 2012:1080; Paynter 2006:272). An artifact's geochemistry could also be influenced by the selection of more desirable raw materials over others in a process called *beneficiation*, further affecting the chemical constituents of the slag mixture (Agricola 1950:270; Henderson 2000:220; Paynter 2006:272). Several *cupellation* or treatment steps preceded the smelting process including granulating, roasting, sorting and rinsing that also influence the geochemical contents of the finished product (Agricola 1950:267; Ferrer-Eres et al. 2010:298).

Two types of iron resulted from early modern metallurgy, called cast iron and wrought iron, with each having distinctive physical characteristics and applications. Cast or *pig* iron is a highly impure and fragile material produced in a blast furnace that is not viable for nail manufacture (Frurip et al. 1983:9-10; Starley 1999:1128). Instead, the more pure and ductile wrought iron needed to be fashioned from cast iron ingots in a bloomery. Wrought iron, in contrast to cast iron, is a mostly pure iron matrix with slag inclusions scattered throughout the material in the form of long strands down the length of the shaft that contribute to the structural and chemical durability of the nail (Frurip et al. 1983:12-13; Wells 1998:79). Time, temperature, craftsmanship and the origin of the raw ore are all factors that alter the chemical constituents of these iron-rich slag; inclusions that can suggest the geographic and temporal context of their manufacture due to the quantity and quality of different slags specific to regional and chronological conditions (Frurip et al. 1983:13; Starley 1999:1128; Wells 1998:80-81).

3.2 Historic Iron Nail Use

Europeans benefitted from the use of iron since at least the 5th century BCE and have used hand-wrought iron nails since the medieval period, ensuring that they are one of the most prominent types of objects found in historic archaeological sites (Bodey 1983:7; Hume 1970:252; Rapp 2009:166). In the Middle Ages, the hand-wrought iron nail manufacturing industry produced artifacts that were widely variable and unstandardized in size (Hume 1970:252; Nelson 1968). In the post-medieval period nails began to be labeled by their physical characteristics at the same time as the iron industry began to proliferate in demand and output in the European economy (Nelson 1968; Starley 1999:1127). One of the major factors contributing to this iron boom in the European market beginning in the 16th century was the trade in wrought iron nails that remained a lucrative business for several centuries (Bodey 1983:11; Hume 1970:525; Nelson 1968). The shipbuilding industry was a major consumer of these wrought iron nails with more than 8000 flat-headed nails being discovered in the hull of the Basque whaling galleon *San Juan* shipwreck in Red Bay (Light 1992:249-255). The construction of trywork *cabañas* in Basque shore stations would also require numerous iron nails (Proulx 2007b:66). Wrought nails had become out of fashion by the 19th century as cut nails became the nail industry standard (Hume 1970:253).

3.3 Historic Iron Nail Production

Early modern iron nails would have been made out of the earliest form of processed iron called wrought iron that was easily worked and durable (Mathias 1998:40). Historically, there have been two different techniques for processing wrought iron after ore is extracted. The oldest (and most primitive method) was the *direct* method that involved the unrefined creation of wrought iron by melting granular ore in a bloomery furnace (Aronson et al. 2013:110; Buchwald and Wivel 1998:73). This included granulating and roasting the ore to get rid of sulfur and water, melting the ore in a charcoal or wood furnace and then working the iron into the desired shape (Agricola 1950:273; Buckwald 2001:9-12). This process left the finished product highly impure and prone to enormous quantities of slag inclusions (Starley 1999:1127). The direct method would have been the only approach available to the Norse at the time of their initial colonization of Greenland and their North Atlantic explorations (Buchwald 2001:8; Buchwald and Wivel 1998:73). The direct method was introduced to Iberia by the Islamic caliphate in the early Middle Ages in the form of the *Catalan* method, and continued to be a widespread practice in the Pyrenees region along with the indirect method well into the 19th century (Smith 1966:21; Tomàs 1999:228).

By contrast, the *indirect* process of wrought iron creation involved granulating and roasting the ore before being placed in a blast furnace to reduce the melt into the highly fragile cast or *pig* iron that is then converted into wrought iron in *finery* and *chafery* hearths in a forge to remove excess carbon and silicon (Frurip et al. 1983:6-11; Henderson 2000:220; Rapp 2009:167; Starley 1999:1127; Smith 1966:23; Wells 1998:79-80). The benefit of the intermediary blast furnace step is the two to five percent level of carbon in the cast iron that lowers the iron's melting temperature and removes much of the separated liquid slag in the blast furnace stage (Buchwald 2005:68; Frurip et al. 1983:6-11; Henderson 2000:232; Starley 1999:1127). The iron was then worked with a hammer, and heated or *annealed*, so that most of the slag was relegated into inclusions in the form of long miniscule strands down the length of the object surrounded by relatively pure iron, giving wrought iron a grain that steel lacks (Frurip et al. 1983:11; Henderson 2000:232; Light 2000:330; Wells 1998:80). The indirect method was first used in Europe in the 12th century and by the 14th century the use of the blast furnace was widespread in European metallurgy (Rapp 2009:167-168; Starley 1999:1127). The 18th century saw the inclusion of charcoal into the iron smelting process (Frurip et al. 1983:7; Rapp 2009:167).

Several factors during the smelting process can influence the elemental quantities, distribution and crystal structure of wrought iron to an extent that it may only be possible to trace a finished artifact to a production center (Charlton et al. 2012:2291; Coustures et al. 2003:609). Skills, experience and specific procedural decisions made by the ironsmith have enormous implications for element concentrations like ore selectivity, direct or indirect methods used and cultural traditions (Blakelock et al. 2009:1745; Rehren and Pernicka 2008:235). The type of furnaces used in the forge can influence temperature and the walls of the furnace can greatly affect the slag concentrations (Blakelock et al. 2009:1745; Gimeno-Adelantado et al. 2003:908; Starley 1999:1128). Provenience studies on wrought iron forged using the indirect method cannot be traced to an ore using trace elements (Desaulty et al. 2009:2461). Some provenience studies of wrought iron may only be limited to individual smelts due to the different temperatures, ores, ashes, clays, sands, species of plants used for charcoals and furnace linings used that can be distinct to a single smelting event (Chirikure and Bandama 2014:300; Paynter 2006:272; Schwab et al. 2006:442).

Basque metallurgy in the 16th century was not standardized, resulting in a great deal of inconsistency in nail making techniques (Light 1992:254). While the Basque were very active in the iron nail making industry in the early modern period, other European groups may have also contributed to the manufacture of artifacts acquired by the Labrador Inuit. The blacksmiths of early modern Basque Country and Europe would have created iron nails with characteristics that are unique to the person who manufactured them (Light 1992:254-257; Wells 1998:80-81). Despite this variability there were some generally widespread aspects to the process of making iron nails. The major similarity would be the preparation of nail-blanks from the wrought material.

This labour intensive process involved beating the iron into a bar before it was hammered into a rod or flattening the iron into a plate that needed to be chiseled or sheared along the grain by hand into smaller rods (Bodey 1983:7; Light 1992:253-254; Wells 1998:80). The rods resulting from any one of these processes were then forged and hammered by hand into the desired dimensions of the nail or spike shaft (Wells 1998:81). After the 16th century the slitting mill became more prevalent in Europe as a means of efficiently dividing wrought iron bars into rods by machine that can be more conveniently worked into nails and spikes (Light 1992:253-257; Smith 1966:23; Wells 1998:79-80).

An assortment of techniques were used for ascribing the head to the nail dependent on both the purpose and manufacturer of the nail following the forging stage (Hume 1970:252; Light 1992:254). The end of the nail would usually be heated into the malleable austenite form of iron and *upset* or flattened before being placed in a vice or header where the head was formed by the blacksmith (Light 1992:254-255; Nelson 1968). The flattened end might be further *mushroomed* or contorted and the shaft of the nail may have needed to be manipulated in order to fit into the header (Light 1992:255-256). At this stage the other end of the nail is also forged and worked into a point (Bodey 1983:8).

3.4 Characterization Research of Archaeological Metals

Geochemical provenience and characterization is done under the assumption that elements leave a signature in the slag that remain relatively consistent throughout the smelting process and inconsistent among different sources and forging procedures. Other than the obvious iron, major elements that are commonly found in and used to geochemically characterize ferrous artifacts include Al, As, Cd, Ca, C, Cr, Co, Cu, Pb, Mg, Mn, Ni, O, P, K, Si, Na, S, Ti, V, Zn and Zr (Aronson et al. 2013:117-118; Ashkenazi et al. 2013:248; Balassone et al. 2009:53; Buchwald and Wivel 1998:74; Cvikel et al. 2013:208; Devos et al. 2000:875; Gimeno-Adelantado et al. 2003:904; Piatak and Seal II 2012:641). Co, Cu, and Ni are typically bonded to the iron metal part of the artifact, while much of the Ca, Mn, P and Na in an artifact are found in the inclusions (Starley 1999:1128). Large quantities of phosphorus make iron artifacts brittle so it began to be phased out of the smelting process in the 17th century (Aronson et al. 2013:111; Rapp 2009:167). Sulfur tended to be avoided while much of the silicon was extruded when an iron piece was hammered and worked (Starley 1999:1128). Basque Country is known to produce ores that are very abundant in manganese and the quantity of this element in artifacts' geochemistry is highly influenced by the smelting process (Desaulty et al. 2009:2459; Starley 1999:1128).

There are different techniques for assessing geochemical correlations between iron ore, slag inclusions and metal. Isotopes from lead, strontium and osmium have been examined in geochemical research in archaeology for the purposes of chemical characterization and raw material provenance (Balassone et al. 2009; Brauns et al. 2013; Degryse et al. 2007; Schwab et al. 2006). Iron geochemistry characterization has also targeted major and trace element concentrations in either the iron metal or the slag inclusions of an artifact to assess the original sources of material culture and the sophistication of historic trade networks (Leroy et al. 2012:1081). Some research uses *non-reduced compounds* (NRC), compounds that have not been disassembled into individual elements, as opposed to breaking down the data as a method for assessing the geochemical consistency amongst samples (Blakelock et al. 2009:1756). These studies work under the assumption that these elements and isotopes vary in abundance according to the geographical, historical and technological contexts in which they were manufactured. It has also been speculated that manufacturing techniques are sufficiently comparable enough to produce objects of regionally consistent composition so artifacts can be traced to broadly defined iron-making centers where similar ores and manufacturing techniques were employed (Coustures et al. 2003:609).

Smelting had implications for the geochemical constituents of the finished product. Much of the smelting process is supposed to remove impurities in the iron, but some element signatures persist in the form of slag inclusions (Blakelock et al. 2009:1745; Buchwald and Wivel 1998:75). Carbon, manganese, phosphorus and silicon inclusions oxidize at different rates during the forging process (Starley 1999:1128). The ferrite and austenite forms of iron have different bonding properties with other elements and oxides (Buchwald 2005:66). Lead isotope levels are not substantially affected during the early forging process, but are often too low in iron artifacts to provide detectable ratios, while strontium is highly abundant in ore and the addition of substances like clay, charcoal and lime during the forging process will considerably increase strontium levels (Balassone et al. 2009:46; Brauns et al. 2013:842). Like Al, Ba, Ca, Fe, K, Mg, Mn, Na and Ti, strontium is a *lithophile* element, which means it oxidizes easily and is more likely to be found in slag inclusions (Allaby 2008; Brauns et al. 2013:842). In contrast, lead is a *chalcophile* like As, Cu, Sb and Zn meaning that it has a strong attraction to sulfur and will be found in both the inclusions and the surrounding metal (Allaby 2008; Brauns et al. 2013:842).

Non-reduced compounds are elemental conglomerates that were never divided in the smelting process (Dillmann and L'Héritier 2007:1813). Aluminum oxide, calcium oxide, magnesium oxide, manganese oxide, potassium oxide, titanium dioxide have all been targeted for analysis as well as silicon dioxide, which is typically the most abundant NRC in ore (Charlton et al. 2012:2281-2281; Chirikure and Bandama 2014:300). Non-reduced compounds can be an indicator of furnace pollutants as well as charcoal and ore compositions (Desaulty et al. 2009:2461). Dillmann and L'Héritier (2007), for example, used SEM-EDX to target NRC in slag inclusions for medieval French metallurgy. They detected a large degree of variability within the NRC contents of slag inclusions for 161 artifacts (Dillmann and L'Héritier 2007:1815). Heterogeneous results were encountered in all but a few of the samples by targeting NRCs in slag inclusion with SEM-EDX (Dillmann and L'Héritier 2007:1822). This inconsistency is caused by various stages like the smelt, refining procedure and forge in the manufacturing process, which has the potential to make slag inclusions within the same objects prohibitively variable in provenance analysis (Schwab et al. 2006:439).

A visual analysis on an assemblage of iron nails is very limited in the amount of information that it can provide. The size and shape of the artifact is not indicative of how it was made and it is nearly impossible to determine whether the direct or indirect method was used in the manufacture of an iron nail by visual examination (Wells 1998:79). Visual analysis of iron nails from Labrador Inuit sites does not produce a variation in nail types up the coast (Wolfe 2013:109). Nails found in Inuit sites were often modified from their original shape to perform a traditional function (Wolfe 2013:112). In some instances iron nails can be used to date sites into the very broad temporal categories of hand-wrought forged nails that were made from at least the medieval period until the 19^{th} century, machine-cut nails in the 19^{th} and 20^{th} centuries, and the contemporary wire nails (Nelson 1968). Generally, however, visual analysis is an extremely imprecise and unviable means of geographic provenance for historic iron nails.

Several methods have been used to detect the geochemical constituents of objects, or analytes, in metal artifacts that have often been used in combination with each other. These techniques can include both laser ablation and solution-based inductively coupled plasma-mass spectrometry (Brauns et al. 2013; Coustures et al. 2003; Desaulty et al. 2008; Desaulty et al. 2009; Devos et al. 2000; Marco 2012; Schwab et al. 2006). These studies often specifically target trace elements in slag inclusions for their provenance analysis of iron artifacts and ores. For instance, Desaulty et al. (2008) used solution-based ICP-MS to correlate a collection of ore, slag and cast irons to one of several French iron making centers. They used acid digestion on powdered samples with a "mixture composed of 2 ml of HNO_3 (65% Normatom), 1 ml $HClO_4$ (68% Normatom) and 1 ml HF (47% Normatom)" (Desaulty et al. 2008:1256). The results of the analysis were able to use trace elements such as Ce, Co, Cs, Ba, Eu, Hf, La, Rb, Sc, Sm, Th, U and Yb to source samples to very broad iron-producing regions.

An example of laser ablation ICP-MS being successfully implemented as a means of provenance for archaeological iron comes from Coustures et al. (2003). This study used a Nd:YAG, 266 nm laser to target the trace element concentrations in slag inclusions to source iron blooms and artifacts to iron ores from the Roman era of France (Coustures et al. 2003:602). They were able to establish consistent geochemical signatures from several iron making regions and trace element ratios were sufficient to correlate blooms to these sites. Devos et al. (2000) performed a similar preliminary analysis on polished, epoxy resin embedded cross-sections of medieval Swiss irons. Trace elements proved to be a reliable indicator of sources in this instance as well.

Multiple geochemical characterization studies have been done on historic wrought iron nails and other archaeological metals using scanning electron microscopy in conjunction with energy dispersive X-ray spectrometry (Aronson et al. 2013; Ashkenazi et al. 2013; Balassone et al. 2009; Blakelock et al. 2009; Brauns et al. 2013; Campos and Solórzano 2004; Charlton et al. 2012; Coustures et al. 2003; Cvikel et al. 2013; Faifar et al. 2013; Ferrer-Eres et al. 2008; Ferrer-Eres et al. 2010; Gelegdorj et al. 2007; Gimeno-Adelantado et al. 2003; Leroy et al. 2012; Lv et al. 2011; Maldonado and Rehren 2009; Mapelli et al. 2007; Marco 2012; Martinón-Torres et al. 2007; Martinón-Torres et al. 2012; McCowan et al. 2011; Mödlinger et al. 2013; Murillo-Barroso et al. 2010; Neff et al. 2004; Neff et al. 2005, Neff et al. 2006; Park et al. 2010; Paynter 2006; Piatak and Seal II 2012; Reguer et al. 2007; Remazeilles et al. 2010; Renzi et al. 2009). SEM has been applied in archaeology since the late 1960s, but this method was not widely used for metallurgical analysis until relevant technological advancements were made in the last three decades (Frahm 2014:6489-6491).

The sample preparation process typically involves extracting samples in transverse cross-sections along the shaft of the nails with a diamond saw using an oil-based lubricant, embedding them in an epoxy resin and polishing them with silicon carbide abrasive paper to an 1 μm finish (Desaulty et al. 2009:2446; Leroy et al. 2012:1084; Neff et al. 2005:119; Remazeilles et al. 2010:389; Rimmer and Wang 2010:80). Sonication steps usually accompany these cutting and polishing stages using ethanol and ultrapure water in ultrasonic baths (Desaulty et al. 2009:2446). Then samples may be carbon coated to make them more conductive to the EDX beam (Rimmer and Wang 2010:80). In contrast to ICP-MS, which target trace elements, SEM-EDX analyses usually target major elements like Al, Ca, Fe, K, Mg, Mn, O, P, Si, Ti, Na (Desaulty et al. 2009:2447).

SEM-EDX has previously been implemented to perform characterization and provenance on archaeological iron and copper from Spain with promising results (Ferrer-Eres et al. 2010; Gimeno-Adelantado et al. 2003; Renzi et al. 2009). Gimeno-Adelantado et al. (2003) conducted a SEM-EDX analysis on a collection of iron ores, slags and artifacts, among them iron nails, from Iberia in the first millennium BCE. Samples were prepared by embedding cross-sections in acrylic resin pucks, polishing with silicon carbide abrasive paper, exposed to an ethanol-nitric acid solution called *nital* and washed in deionized water with the absence of a carbon or metal coating step because of the inherent conductive properties of the samples (Gimeno-Adelantado et al. 2003:901). The study managed to identify chemical correlations throughout the entire range of the samples taken from various stages along the iron manufacturing process (Gimeno-Adelantado et al. 2003:910). The samples in this study were all taken from the same geographic vicinity and proved that the slag inclusions remained more or less consistent throughout the manufacturing process and traceable to the original ore (Gimeno-Adelantado et al. 2003:910).

ICP-MS is often used in conjunction with SEM-EDX to more thoroughly analyze an assemblage of artifacts. Leroy et al. (2012), for example, used SEM-EDX to analyze major elements and used ICP-MS to target trace elements in a collection of medieval wrought iron, including nails, from the Ariège region of the French Pyrenees. These artifacts were compared to the geochemical signatures of four different medieval iron mines in the same region. The ore samples were crushed into powdered pellets and analyzed for major elements by SEM-EDX before undergoing acid digestion and ICP-MS for bulk trace element concentrations. The artifact samples were cross-sectioned, polished using SiC abrasive paper and analyzed for major elements using SEM-EDX and trace elements using LA-ICP-MS (Leroy et al. 2012:1083-1085). The analysis of the artifacts specifically targeted slag inclusions. The results of this study successfully traced most of the artifacts analyzed to one of the four possible medieval ore extraction centers in the region.

Other methods used to analyze archaeological metal include X-ray florescence spectrometry (Aronson et al. 2013; Cvikel et al. 2013; Maldonado and Rehren 2009; Martinón-Torres et al. 2012; Piatak and Seal II 2012). One of the methods used by Martinón-Torres et al. (2012) was portable X-ray florescence spectrometry because of its non-destructive analytical capabilities. They used it and PIXE on a collection of valuable artifacts made out of gold and gold alloys from pre- and post-Columbian Cuba. The analysis involved scanning each artifact with a handheld Alpha 8000 LZX portable XRF three times and factoring the average reading into the results to acquire a more accurate geochemical signature (Martinón-Torres et al. 2012:441-442). The results showed a good degree of consistency between *p*XRF and PIXE results (Martinón-Torres et al. 2012:442).

Several analyses have made use of neutron activation analysis (Desaulty et al. 2008; Desaulty et al. 2009; Michelaki et al. 2013; Selwyn and Argyropoulos 2006). Michelaki et al. (2013) recently used instrumental NAA on aboriginal copper artifacts from17th century Ontario. The University of Toronto's SLOWPOKE reactor facility was used to analyze 425 copper and brass samples of European and aboriginal origin where they were irradiated for 3 minutes and assayed for 5 minutes each (Michelaki et al. 2013:1252). The objective was to geochemically characterize copper and brass fragments found at a Wendat village site to assess how many artifacts they originally came from. Their findings traced these fragments to the geochemical signatures of as many as three-dozen artifacts (Michelaki et al. 2013:1258).

An example of a more infrequently used technique is thermal ionization mass spectrometry (Degryse et al. 2007; Renzi et al. 2009). TIMS is ideal for detecting isotope ratios as in the case of Degryse et al. (2007) where lead and strontium levels were targeted on ferrous artifacts and ores from ancient Anatolia. This method also requires the destructive acid digestion of the uncorrupted cores of artifacts (Degryse et al. 2007:78). This study found strontium isotopes to be far more reliable as indicators of provenance than lead when it comes to tracing iron artifacts to ores (Degryse et al. 2007:84).

Another method that is less common in geochemical characterization of archaeological metal is proton or particle-induced X-ray emissions (Bourgarit and Thomas 2012; Martinón-Torres et al. 2012; Mödlinger et al. 2013). PIXE was performed on a collection of 161 late medieval French copper artifacts based on known manufacturing methods by Bourgarit and Thomas (2012). The procedure involved having to first scrape corrosion layers off of the surface of the artifacts before the cleaned areas were targeted by a PIXE μ-beam (Bourgarit and Thomas 2012:3053-3054). The results produced at least eight consistent geochemical signatures (Bourgarit and Thomas 2012: 3069).

Light optical microscopy is often used to supplement these methods with high-resolution visual data (Faifar et al. 2013; Mapelli et al. 2007; Martinón-Torres et al. 2007; Martinón-Torres et al. 2012; Murillo-Barroso et al. 2010; Neff et al. 2006). Both plane and cross polarized light (PPL and XPL) optical microscopy was used by Murillo-Barroso et al. (2010:1763), for concentrating their SEM-EDX on areas of interest on samples of Asiatic metals from the first millennium BCE. Mapelli et al. (2007:1052) and Faifer et al. (2013:235) also used optical microscopy before SEM-EDX analyses to better understand the microstructures of medieval swords from the Mediterranean region. All of these studies benefited from the use of optical microscopy to refine their search to select regions of interest on the SEM-EDX samples.

Many of these geochemical characterization analyses were for the purpose of determining the natural source of the mineral called *provenience* or researching the artifact's historic lifespan and use called *provenance* (Weiner 2010:36-37). If discernable and consistent geochemical distinctions are identified between artifacts sources then it is possible to trace an artifact's *provenience* and gain a better understanding of the artifact's *provenance* (Charlton et al. 2012:2282; Coustures et al. 2003:611). In iron artifacts the slag inclusions are the preferred target of geochemical analysis because the slag tends to maintain much of the distinct element concentrations that were present in the ore or the smelting furnace where it was manufactured (Gimeno-Adelantado et al. 2003:895; Paynter 2006:272; Rehren and Pernicka 2008:235; Starley 1999:1128). It can often be difficult to locate the precise mine or forge in which an iron artifact was originated so sourcing an object may only be able to trace it back to a broad geographical territory or time period (Blakelock et al. 2009:1756; Coustures et al. 2003:609; Paynter 2006:273).

Iron made from the direct process is considered better suited to provenience analysis and intermediate iron products like ingots are more ideal for tracing back to an ore source than a finished metallurgical object (Charlton et al. 2012:2281).

3.5 Problems in Archaeomineralogical Analysis of Iron Nails

3.5.1 Corrosion and Contamination

Several issues arise in the analysis of the nature of metal artifacts at the microscopic or archaeomineralogical level. Among the most significant of these research problems are the exclusion of iron oxide and other corrosive components of the artifact from the geochemical results. Iron atoms bond easily with water and oxygen, causing the creation of iron oxide or rust which destabilizes the structural integrity of an iron artifact's surface (Buchwald 2005:69). Chloride ions are also heavily corrosive to iron and hydroxychlorides can form on the exterior oxide layer with prolonged exposure to humidity (Buchwald 2005:69; Reguer et al. 2007:66-67; Selwyn and Argyropoulous 2006:3). The presence of chlorine in the interior of archaeological iron may be indicative of contamination and leaching from the surrounding soil environment (Buchwald and Wivel 1998:94). Archaeological preservation with chemicals like sodium hydroxide may also affect geochemical characterization as treatment of iron nails can manipulate results (Rimmer and Wang 2010:84).

Iron oxide is the most common corrosion material on the surface of iron artifacts, and must be considered in geochemical characterization studies of archaeological metals. Hematite, goethite, maghemite, magnetite and wüstite are common types of oxides found on an iron object's outer layers and calcium carbonate typically forms on iron buried in marine environments (Ashkenazi et al. 2013:241; Balassone et al. 2009:58; Selwyn

2004:295). Iron submerged in a marine context undergoes rapid electrochemical processes as oxygen and chlorine form iron oxychloride in fractures (Argo 1981:42; Neff et al. 2004:533; Selwyn 2004:294). For example, iron nails from the Basque whaling station of Red Bay were exposed frequently to seawater contributing to a quantity of akaganeite on the artifacts (Argo 1981:42-43). The interiors of the iron nails likely remain relatively unaffected by this chemical transformation and the exclusion of the rust and chemical compounds on the exterior of the artifact will presumably not influence the results. Sodium hydroxide is often introduced to the surface of the artifact after excavation in order to preserve it, which has chemical ramifications as the oxide can become more porous as a result (Selwyn 2004:301). For this reason, as demonstrated through this study, it is necessary to only access the interior, un-altered portion of an iron artifact when performing geochemical analysis.

Changing the chemistry of the artifact during the analytical sample preparation process can change the geochemical results of a characterization analysis. Metals always undergo a chemical change when exposed to solvents like hydrochloric acid or nitric acid (Mascetta 2003:241). Solution-based analysis in which the iron is broken down or diluted in a chemical acid may inevitably create different chemical compounds that could suppress the analytes of interest. Specifically when ferrous metal is exposed to nitric acid (HNO_3) or hydrochloric acid (HCl) to break down the samples before an ICP-MS analysis it could result in the creation of chemical byproducts that could suppress analyte signals (Pollard et al. 2007:304). Overheating is an issue when preparing samples of solid iron nails with a non-cooling saw that operates without lubricant, or a plasma cutter (Marco 2012:14). Oxidation is an issue during the incremental washing and sonication steps on samples. Therefore ethanol is used in addition to ultrapure water to slow the creation of rust and decrease the chances of iron oxide from entering the results (Desaulty et al. 2009:2446).

3.5.2 Geochemical Heterogeneity

The heterogeneity of a sample is also a concern as wrought iron artifacts may not produce viable results to demonstrate geochemical correlations or inconsistencies within a data set due to ores, forging procedures and other contaminants (Starley 1999:1127-1128). Raw iron ore sources can be heterogeneous within the same mineral vein even though larger geographic sources can show some geochemical consistency (Coustures et al. 2003:611; Paynter 2006:272). This phenomenon was encountered when the study conducted by Coustures et al. (2003) used ICP-MS to assess the trace element concentration data from two major Roman iron making sites; one in the Montagne Noire mountain range of southern France and one in the Loiret region of northern France. They detected a large degree of chemical heterogeneity within the same iron production area but an even larger amount of heterogeneity between the two regions (Coustures et al. 2003:611). Their findings show that the relative homogeneity within a region when compared to the other region can be sufficient to provenance artifacts to one of these two broad iron production centers.

Artifacts manufactured at the same forge can also demonstrate a large degree of geochemical variability as a result of differing smelting techniques, diverse slag compositions, or using ores from more than one source (Buchwald and Wivel 1998:86; Chirikure and Bandama 2014:300; Paynter 2006:272, 288). As a result, studies have detected significant geochemical variability within different parts of a single wrought iron nail (Frurip et al. 1983:21). Buchwald and Wivel (1998) emphasize this point in their SEM-EDX provenance study of iron objects from over three millennia of Scandinavian history. They encountered high and low-carbon and phosphorus zones within the same iron nails, a phenomenon that they attributed to hot forging manipulating the crystal structure (Buchwald and Wivel 1998:74). Different components of the same wrought iron object can demonstrate localized chemical homogeneity while demonstrating chemical heterogeneity within the same object overall, particularly in the slag inclusions (Paynter 2006:288).

Tracing wrought iron artifacts back to the original natural context of the raw ore is extremely problematic and considered exceedingly challenging or impossible by many (Devos et al. 2000:879). This is mostly attributed to the radical manipulation in elemental concentrations that an ore undergoes during the smelting process as a result of temperature changes, fluxes, fuel types, furnace linings, technical expertise and selectivity in the choice of ore used (Blakelock et al. 2009:1745; Devos et al. 2000:879; Rehren and Pernicka 2008:234). Provenience of iron to an ore source may only be possible with a partially completed implement earlier in the smelting process or if materials demonstrate substantial quantities or lack of major and trace elements in slag inclusions where elements from the ore tend to congregate (Charlton et al. 2012:2281; Coustures et al. 2003:599; Devos et al. 2000:879). The smelting process influences the suitability of elements to provide viable provenance data because of chemical additives and the tendency for lithophile and chalcophile elements to congregate in different parts of the artifact's microstructure (Desaulty et al. 2008:1253; Schwab et al. 2006:448).

Absolute provenience of an iron artifact to a raw ore source may only be possible in certain instances when trace or major element ratios are favourably consistent between the artifacts and the original source material (Charlton et al. 2012:2284; Desaulty et al. 2009:2446). A very limited array of major and trace elements in slag inclusions may only be viable for the purposes of provenience since inclusions are believed to house most of the original ore (Coustures et al. 2003:609, 611; Schwab et al. 2006:442). It may only be possible for a geochemical characterization analysis of wrought iron to determine how an object was manufactured as opposed to tracing it to where it came from (Blakelock et al. 2009:1746). In order to determine the absolute provenience of iron artifacts to their raw material source it is necessary to be able to correlate the geochemical signature of a collection of artifacts to a specific raw material location that differs considerably from the geochemical signature of other raw material samples. This principle is the main emphasis of the provenance postulate and without this observable pattern of artifacts' elemental signatures being consistent to specific raw material sources as opposed to others an absolute provenance cannot be determined via geochemical means (Glascock and Neff 2003:1521).

3.6 Conclusion

Several variables related to the nature of the artifacts being examined and the particular features of different techniques factor into the development of an appropriate methodology for a research design in the archaeological sciences. Depending on the types of artifacts being analyzed, samples vary in the quantity of corrosion, contamination and geochemical homogeneity they possess and different analytical approaches are more effective and advantageous based on the specific circumstances of the samples. Many techniques in analytical chemistry have a destructive component to them, which can present limitations in terms of the sample size and quality available with archaeological artifacts. Expenses and time restrictions on research can make some options too cost prohibitive and time consuming to be feasible. Some methods are more widely accessible than others and vicinity to the suitable research facilities and expertise is also a major concern when deciding upon the most viable analytical alternatives.

All of the different groups of Europeans that were active to varying degrees along the coast of Labrador during the early modern period provided the Inuit with at least six major European groups from which to acquire anthropogenic iron. The diversity of potential sources is further complicated by the numerous historic manufacturing hand-wrought ironworking techniques that were often highly customized at the level of the individual blacksmith. This chapter explained the possibility that these various nationalities and iron manufacturing centers in Europe produced different geochemical signatures. The remainder of the thesis assesses the viability that these signatures can be used to consistently trace Inuit artifacts to these broad European iron-manufacturing centers. The succeeding chapter describes the analysis conducted on a collection of 32 iron nails taken from nine Inuit, Basque, and French and English sites across the Labrador coast and Newfoundland (Fig. 4.2).

Chapter 4: Methodology

4.1 Inductively Coupled Plasma-Mass Spectrometry

Inductively coupled plasma-mass spectrometry (ICP-MS) is an analytical method for detecting major and trace elements, as well as isotopes in archaeological metals (Henderson 2000:10; Malainey 2011:370-371). ICP-MS is sensitive and able to analyze most elements on the periodic table concurrently (Desaulty et al. 2008:1262; Malainey 2011:371; Pollard et al. 2007:195). Several ICP-MS analyses have been conducted on archaeological iron (Balassone et al. 2009; Brauns et al. 2013; Coustures et al. 2003; Desaulty et al. 2008; Desaulty et al. 2009; Devos et al. 2000; Marco 2012; Schwab et al. 2006). Solution-based analysis and laser ablation are the two major types of ICP-MS. In a similar study, Desaulty et al. (2008) used solution-based ICP-MS to successfully perform a trace element provenance analysis of archaeological iron from medieval France.

Solution analysis involves immersing the sample in nitric acid in order to liquefy the metal and break down the compounds in the material with hydrochloric acid and sulfuric acid. This can create suppressive compounds in the form of molecules created as a product of chemical additives bonding with atoms from the sample material and manipulating the results (Malainey 2011:371; Pollard et al. 2007:304). The sample solution is then transported from the nebulizer to a plasma torch where the sample is ionized by argon plasma before the resulting positively charged *cations* are sorted by a *quadrupole* and detected by a mass spectrometer (Pollard et al. 2007:196). The resulting element concentrations are then converted to parts-per-million (ppm) and presented in a software spreadsheet that is used to create scatterplot matrices to detect clusters and patterns in the data by contrasting the quantity of various elements.

4.1.1 Sample Selection for ICP-MS

Eight Inuit and European contact period sites were sampled from locations along the coasts of Newfoundland and Labrador. A total of 32 iron artifacts were involved in the ICP-MS analysis (see Table 4.1 and Appendix 1). Half of these samples were from two sites on Huntingdon Island in Sandwich Bay. The site on the west coast of the island is called Indian Harbour or Huntingdon Island 5 (FkBg-03). This site is an Inuit settlement located on Indian Island, a promontory of land connected to the rest of Huntingdon at low tide. The site was occupied as early as the 17th century and includes at least five houses and six tent rings (Kelvin 2011:108).

Table 4.1 List of sites and their approximate dates of occupation.

Site	**Borden Number**	**Cultural Affiliation**	**Dates of Occupation (CE)**
Huntingdon Island 5	FkBg-03	Inuit	17th - 18th Century
Snack Cove 3	FkBe-03	Inuit	16th - 17th Century
Pigeon Cove	FlBf-06	Inuit	Early 18th Century
Great Caribou Island 1	FbAv-13	Inuit	Late 18th - Early 19th Century
North Island 1	FeAx-03	Inuit	Late 16th - Mid-18th Century
Nachvak	IgCx-03	Inuit	16th - 18th Century
Saddle Island	EkBc-01	Basque	16th - 17th Century
Ferryland	CgAf-02	English	17th Century
Dos de Cheval	EfAx-09	French	16th - 18th Century

The largest structure at this site is House 4, an 18th century occupation where 936 artifacts of European origin were excavated; of those 393 were iron implements (Rankin 2012:128). House 3 also dates to the 18th century with Houses 1 and 2 being of the 17th century (Rankin 2012:127-129). Three or four iron nails were examined from each of these houses and one from Tent Ring 4 (also from the 18th century) to examine if geochemical signatures among the artifacts associated with different structures could be temporally correlated to different sources (Fig. 4.1).

Fig. 4.1 Photograph of an iron nail from Huntingdon Island 5 (FkBg-03); catalogue number: 1344; SEM sample number: TH-22.

The site on the east coast of Huntingdon Island is called Snack Cove 3 (FkBe-03). Snack Cove dates to the early 17th century and is home to a minimum of three Inuit sod houses (Brewster 2004:8). A large number of European iron nails have been found in this site's dwellings and most of these artifacts were modified by the Inuit (Brewster 2005:84). Like its counterpart on the opposite side of the island, Snack Cove may be representative of the southerly migration of Labrador Inuit motivated by European sources of iron to the south (Brewster 2005:120). Two iron nails were analyzed from House 3 of this site.

A smaller island called Newfoundland Island is located immediately north of Huntingdon Island in Sandwich Bay and the Pigeon Cove (FlBf-06) site is situated on its west coast (Fig. 4.2). Two iron nails were acquired from this site for analysis that, like House 3 and 4 of Huntingdon Island 5, likely dates back to the early 18th century (Rankin 2013a:128). It only contains one structure in the form of a winter house that contains European and Inuit artifacts numbering 4220 objects (Rankin 2013a:128). This structure's isolation and material wealth suggest that it may have been home to an individual of great social importance (Rankin 2013a:128).

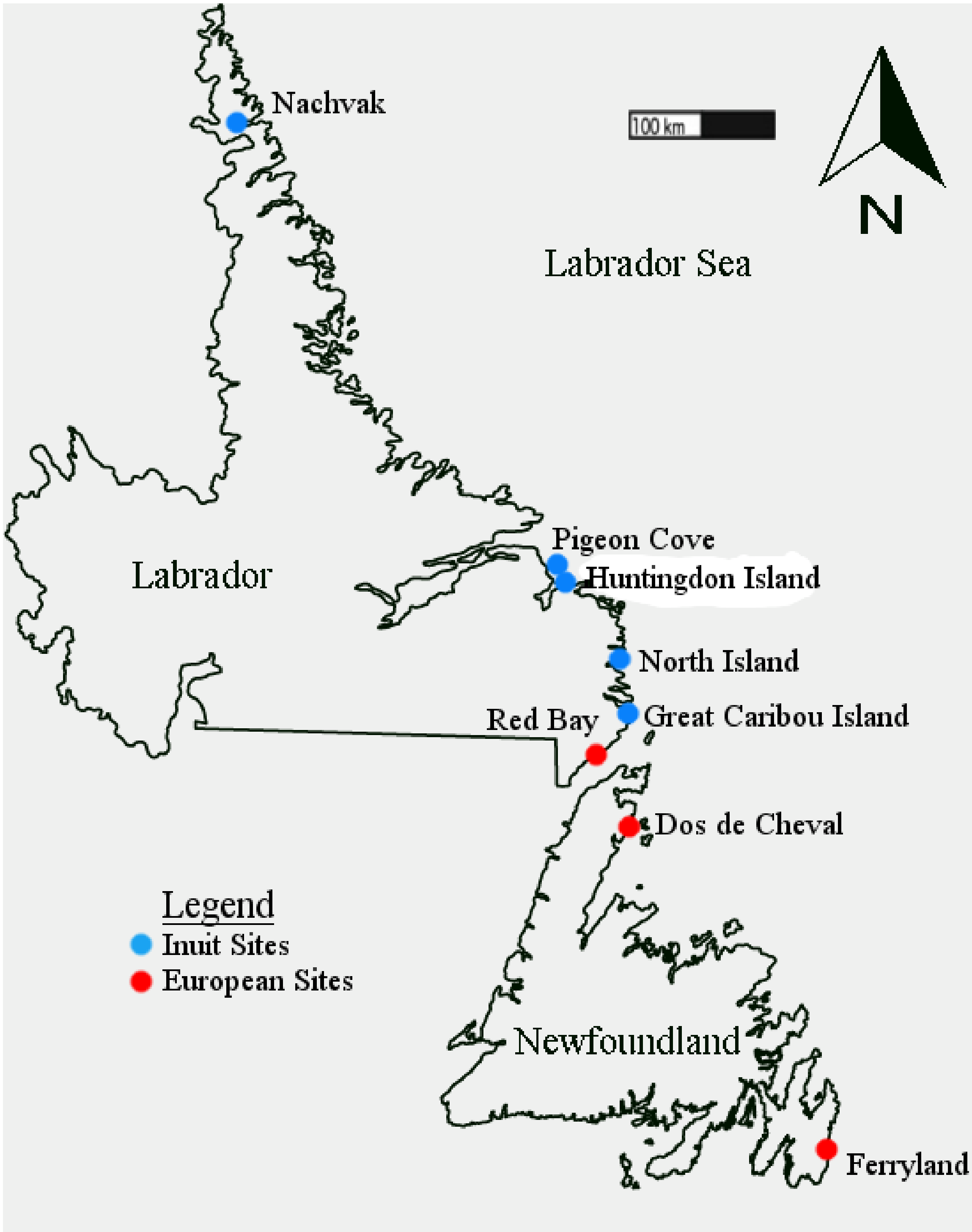

Fig. 4.2 Map of Inuit and European sites in Newfoundland and Labrador.

In between Sandwich Bay and the Strait of Belle Isle are two other sites important to this study: Great Caribou Island 1 and North Island 1. The Great Caribou Island 1 (FbAv-13) site is located in St. Lewis Inlet near Green Cove on the west coast of the island and it dates from the late 18th to the early 19th century, though it has been occupied periodically since the Palaeoeskimo period (Stopp and Jalbert 2010:157). The site contains two historic Inuit sod houses on an elevated cobble stone beach (Stopp and Jalbert 2010:157). Two iron nails were analyzed from each of the two houses at this site. The site called North Island 1 (FeAx-03) is positioned north of Great Caribou Island in St. Michael's Bay next to Schooner Cove and dates from the late 16th century to the mid-18th century (Stopp 2012:166). Like Great Caribou Island 1, the site is made up of two Inuit sod houses that are located on a stone terrace overlooking a protected cove (Stopp and Jalbert 2010:158). Because of its earlier chronology in relation to Great Caribou Island 1, two iron samples from this site were involved in the analysis.

The most geographically northern site involved in this analysis is the Inuit village of Nachvak (IgCx-03) situated in Nachvak Fjord in the Torngat Mountains National Park Reserve of northern Labrador. The village is late Thule in origin and was occupied by the historic Inuit from the 16th century to the 18th century (Higdon 2008:1). The site is comprised of 16 dwellings in the form of sod houses in anthropogenic depressions (Whitridge 2004:16). Two iron artifacts where examined from this village.

Six artifacts came from European sites including four from the seasonal Basque whaling station concentrated on the northern coast of Saddle Island (EkBc-01) at the mouth of Red Bay in the Strait of Belle Isle. As mentioned in chapter 2, this terrestrial whale blubber-rendering settlement was predominantly occupied by the Basque in the

16th and early 17th centuries (Barkham 1984). This particular area of the shore station has a large density of infrastructure with over a dozen structures like ovens and cooperages as well as several middens and a graveyard with the remains of over a hundred Basque whalers (Loewen and Delmas 2012:226-227). The nails from Red Bay were sampled from the tryworks on Saddle Island to avoid inadvertently sampling nails from other European populations who have since exploited Red Bay.

The historic English fishing settlement of Ferryland (CgAf-02) offered a possible alternative European source of iron for the Labrador Inuit. Located on the east coast of the Avalon Peninsula of Newfoundland, this brief permanent settlement was founded in 1621 and devastated by the French in 1696 during which it was a major headquarters for the cod-fishery and English interests in the New World (Guiry et al. 2012:2013). Hundreds of European artifacts, including iron, have been excavated from this site (Gaulton et al. 2010:64). Nails were obtained from the Ferryland site to examine the presence of a significant geochemical difference in iron signatures between Basque and English sites. All of the artifacts involved in this study were derived from sites dating to the contact period of Labrador and Newfoundland and nails discovered in the older soil strata of the sites were targeted (see Table 4.2). Most of occupations from the Inuit sites involved in this study are suspected of having been inhabited briefly.

Table 4.2 Table of individual ICP-MS and SEM-EDX artifact lab numbers as well as their associated sites, features and catalogue numbers.

SEM Lab Number	ICP-MS Lab Number	Site	Associated Feature	Borden Number	Catalogue Number
TH-01	FlBf06-2292	Pigeon Cove	House 1	FlBf-06	2292
TH-02	FkBg03-2708	Huntingdon Island 5	House 4	FkBg-03	2708
TH-03	FkBg03-123	Huntingdon Island 5	House 1	FkBg-03	123
TH-04	FlBf06-1778	Pigeon Cove	House 1	FlBf-06	1778
TH-05	FkBg03-2991	Huntingdon Island 5	House 4	FkBg-03	2991
TH-06	FeAx03-240	North Island 1	-	FeAx-03	240
TH-07	FkBg03-2948	Huntingdon Island 5	House 4	FkBg-03	2948
TH-08	FkBg03-297	Huntingdon Island 5	House 3	FkBg-03	297
TH-09	FkBg03-4307	Huntingdon Island 5	Tent Ring 4	FkBg-03	4307
TH-10	FkBg03-147	Huntingdon Island 5	House 1	FkBg-03	147
TH-11	FkBg03-184	Huntingdon Island 5	House 1	FkBg-03	184
TH-12	Not ICP-MS sample	Snack Cove 3	House 3	FkBe-03	1228
TH-13	Not ICP-MS sample	Great Caribou Island 1	House B	FbAv-13	-
TH-14	Not ICP-MS sample	Great Caribou Island 1	House A	FbAv-13	-
TH-15	FbAv13-HA.1-W21	Great Caribou Island 1	House A	FbAv-13	-
TH-16	FkBg03-118	Huntingdon Island 5	House 1	FkBg-03	118
TH-17	EkBc01-23190a-r	Saddle Island	Tryworks	EkBc-01	23190
TH-18	EkBc01-23152a-j	Saddle Island	Tryworks	EkBc-01	23152
TH-19	Not ICP-MS sample	Dos de Cheval	-	EfAx-09	11141
TH-20	Not ICP-MS sample	Snack Cove 3	House 3	FkBe-03	1288
TH-21	FkBg03-1344	Huntingdon Island 5	House 2	FkBg-03	1344
TH-22	FkBg03-318	Huntingdon Island 5	House 2	FkBg-03	318
TH-23	FkBg03-615	Huntingdon Island 5	House 2	FkBg-03	615

TH-24	FkBg03-373	Huntingdon Island 5	House 3	FkBg-03	373
TH-25	FkBg03-1248	Huntingdon Island 5	House 3	FkBg-03	1248
TH-26	Not ICP-MS sample	Dos de Cheval	-	EfAx-09	11144
TH-27	FbAv13-HB.1-20-30	Great Caribou Island 1	House B	FbAv-13	-
TH-28	Not ICP-MS sample	North Island 1	-	FeAx-03	222
TH-29	Not ICP-MS sample	Nachvak	House 2	IgCx-03	219
TH-30	Not ICP-MS sample	Nachvak	House 2	IgCx-03	241
TH-31	Not ICP-MS sample	Saddle Island	Tryworks	EkBc-01	29508
TH-32	EkBc01-25985c	Saddle Island	Tryworks	EkBc-01	25985
Not SEM sample	FkBe03-1229	Snack Cove 3	House 3	FkBe-03	1229
Not SEM sample	FkBe03-1244	Snack Cove 3	House 3	FkBe-03	1244
Not SEM sample	FeAx03-207	North Island 1	-	FeAx-03	207
Not SEM sample	FbAv13-HA.2-W24	Great Caribou Island 1	House A	FbAv-13	-
Not SEM sample	FbAv13-HB.2-30-40	Great Caribou Island 1	House B	FbAv-13	-
Not SEM sample	EkBc01-25619c	Saddle Island	Tryworks	EkBc-01	25619
Not SEM sample	IgCx03-782	Nachvak	House 2	IgCx-03	782
Not SEM sample	IgCx03-4048	Nachvak	House 6	IgCx-03	4048
Not SEM sample	FldB-794.1	Ferryland	Event 794	CgAf-02	-
Not SEM sample	FldB-794.2	Ferryland	Event 794	CgAf-02	-

In order to assess whether preservation had a significant impact on the geochemical results, two of the artifacts tested from House 1 on Huntington Island were not exposed to sodium hydroxide while two of them were. All 28 of the other artifacts have been treated in an aqueous sodium hydroxide (NaOH) solution to protect the artifacts from corrosive ions and chloride that contribute to the decomposition of the artifacts exterior surface (Selwyn and Argyropoulos 2005:81). The purpose of this experiment was to evaluate the possible impact these preservative chemicals may have on the interior chemistry of the artifacts. Of course, this experiment assumes that because those artifacts were from the same structure that they would share the same geochemical signature.

4.1.2 Sample Preparation for ICP-MS

Approximately 100 mg of iron was extracted from each artifact by stripping the exterior of the artifact off with pliers and placing it in a 15 ml Teflon container. Acid digestion began with the addition of 2 ml of concentrated 6N hydrochloric acid and heating for twenty-four hours on a hotplate at 60°C. The samples were washed with 8N nitric acid and dried on the hotplate at 70°C. Once they were dried, 2 ml of 8N nitric acid was added to each container and the samples were thoroughly liquefied on a hotplate at 70°C. Samples that were not completely dissolved were exposed to a 30% hydrogen peroxide solution at 60°C for twenty-four hours. The samples were reduced in 60 g of nano-pure water, particles were filtered out through NO1 Whatman filter paper. The solution was sealed in a snap-capped 120 ml tube. The solution was further diluting into 10 g samples in 11 ml test tubes with sample solution and 0.2N nitric acid at a ratio of 1:49.

4.1.3 Analytical Procedure for ICP-MS

The ICP-MS analysis was conducted at the Earth Resources Research and Analysis Facility (TERRA) in the Department of Earth Sciences at Memorial University of Newfoundland. A PerkinElmer ELAN DRC II inductively coupled plasma-mass spectrometer was used to analyze the elemental composition of the iron artifacts. Thirty-nine elements were targeted by the mass spectrometer including Al, Sb, As, Ba, Be, Bi, B, Br, Cd, Ca, Ce, Cs, Cl, Cr, Co, Cu, Fe, I, La, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, Rd, Se, Si, Ag, Sr, S, Sn, Ti, Tl, U, V and Zn. The instrument was calibrated and operated by TERRA's ICP-MS research laboratory coordinator. TERRA's solution-based ICP-MS laboratory uses the analytical procedures described by Jenner et al. (1990:136), which included external calibration, internal standardization and standard addition strategies.

External calibration involves analyzing two solution standards containing known elemental concentrations to those solutions containing samples to correct for signal drift (Longerich et al. 1990:108). This lab also uses internal standardization with the inclusion of Mo, Nb, Ta and W to the sample in order to determine solution instability (Jenner et al. 1990:136). Standard addition creates spikes of known quantities of the elements being analyzed and compares them with the stable results (Longerich et al. 1990:108). Data was compiled in an Excel spreadsheet while further quantitative analysis was conducted on the JMP 11 statistical software program. The values are represented in parts-per-million (ppm).

4.2 Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry

Scanning electron microscopy used in combination with energy dispersive X-ray spectrometry (SEM-EDX or SEM-EDS) can provide an analysis of multiple elements on a geochemically heterogeneous surface (Henderson 2000:18; Pollard et al. 2007:111). The SEM-EDX is particularly beneficial when it comes to analyzing spatial differences, relative quantitative simplicity, better detection limits and compositional variability than methods like X-ray florescence spectrometry and the inclusion of visual data (Henderson 2000:19; Pollard et al. 2007:109, 113).

The scanning electron microscope works by bombarding the surface of the sample on a plane with an electron beam emitted by an electron gun (Henderson 2000:18; Pollard et al. 2007:109). The SEM uses a combination of low-energy, contour sensitive *secondary electrons* that are responsible for imaging and high-energy, nucleus penetrating *backscatter electrons* that provide the geochemical data (Henderson 2000:18; Pollard et al. 2007:109). The end product of the SEM-EDS analysis is a Backscattered Electron (BSE) Image that provides both visual qualitative and quantitative data across the diameter of the sample (Henderson 2000:19; Pollard et al. 2007:11). Before the SEM stage, however, samples preparation requires a series of steps that typically involves first cutting the artifact with a diamond saw, embedding the iron sample in a plastic epoxy, polishing the surface to a 1 μm finish, sonicating it, washing it in ethanol and then carbon-coating to make the surface of the sample more conductive for the EDX (Ashkenazi et al. 2013:242; Blakelock et al. 2009:1747; Desaulty et al. 2009:2447; Dillmann and L'Héritier 2007:1812; Henderson 2000:19; Pollard et al. 2007:111; Rimmer and Wang 2010:80).

4.2.1 Sample Selection for SEM-EDX

Twenty-two of the same nails that were used in the ICP-MS analysis were also used in the SEM-EDX analysis, but ten nails needed to be replaced due to their irregular shape or extreme corrosion that made them unviable for SEM-EDX (see Table 4.3 and Appendix 2). One artifact from North Island, one from Saddle Island and two from Great Caribou Island needed to be replaced as well as all of the artifacts from Snack Cove and Nachvak. The artifacts from Ferryland were too corroded and irregular to be sectioned by the precision sectioning saw and therefore not analyzed via SEM (see Appendix 4). Like the ICP-MS analysis, the SEM-EDX analysis involved the same two artifacts from House 1 on Huntingdon Island (FkBg-03) that were not exposed to chemical preservatives and two artifacts from the same site that were treated to sodium hydroxide treatment to assess the possible impact of preservation on the results.

Table 4.3 Number of individual SEM-EDX samples taken from each artifact.

Artifact lab number	**Site**	**Number of Samples (3 mm slices)**
TH-01	Pigeon Cove (House 1)	2
TH-02	Huntingdon Island 5 (House 4)	2
TH-03	Huntingdon Island 5 (House 1)	2
TH-04	Pigeon Cove (House 1)	2
TH-05	Huntingdon Island 5 (House 4)	2
TH-06	North Island 1	2
TH-07	Huntingdon Island 5 (House 4)	3
TH-08	Huntingdon Island 5 (House 3)	3
TH-09	Huntingdon Island 5 (Tent Ring 4)	3
TH-10	Huntingdon Island 5 (House 1)	3
TH-11	Huntingdon Island 5 (House 1)	2
TH-12	Snack Cove 3 (House 3)	2
TH-13	Great Caribou Island 1 (House B)	2
TH-14	Great Caribou Island 1 (House A)	2
TH-15	Great Caribou Island 1 (House A)	2
TH-16	Huntingdon Island 5 (House 1)	2
TH-17	Saddle Island (Tryworks)	2
TH-18	Saddle Island (Tryworks)	2
TH-19	Dos de Cheval	2
TH-20	Snack Cove 3 (House 3)	2
TH-21	Huntingdon Island 5 (House 2)	3
TH-22	Huntingdon Island 5 (House 2)	3
TH-23	Huntingdon Island 5 (House 2)	2
TH-24	Huntingdon Island 5 (House 3)	2
TH-25	Huntingdon Island 5 (House 3)	3
TH-26	Dos de Cheval	2
TH-27	Great Caribou Island 1 (House B)	3
TH-28	North Island 1	3
TH-29	Nachvak (House 2)	2
TH-30	Nachvak (House 2)	2
TH-31	Saddle Island (Tryworks)	2
TH-32	Saddle Island (Tryworks)	2
	Total:	73

Two additional nails were sampled from the historic French site of Dos de Cheval located in Cape Rouge Harbour on the northeast coast of Newfoundland's Great Northern Peninsula. The site was called *Champs Paya* by the French and was used by French fisherman mostly from Brittany from the 16th century until the 18th century (Guiry et al. 2012:2013). This fishing station has produced ceramics, iron and other archaeological artifacts of European origin (Noël 2010:186). Its vicinity to Labrador and early involvement in the transatlantic cod-fishery makes this site a likely source of European iron for the Inuit in the early contact period. These nails were taken from Dos de Cheval to see if there is a significant difference in the geochemical signatures of iron artifacts between Basque and French sites and if any artifacts from Inuit sites can be traced to this signature.

4.2.2 Sample Preparation for SEM-EDX

To avoid the friable outer oxide layer of the nails from disintegrating while cutting, LePage epoxy steel was applied to sections of the nails' that were to be cross-sectioned along the shafts to consolidate the outer layer of the artifact (Fig. 4.3). After the epoxy cured, the nails were cut with a Buehler-IsoMet 1000 precision saw using a lubricant solution of diluted Buehler IsoCut fluid and de-ionized water at a ratio of 1:9. Slices 3 mm long were cut from two to three sections along the shaft of the artifact (see Appendix 3). The number of sections taken for a particular artifact depended on the length of the nail. Nails longer than 10 cm provided more than two sections. The cut sections were rinsed in a 95% ETOH solution to avoid further oxidation, left to dry, then embedded in 30 mm round molds using a Buehler EpoThin epoxy resin and hardener at a ratio of 5:1.95. Approximately 1.5 mm of the puck was cut to remove excess epoxy. The embedded cut nail samples were sonicated in a 95% ETOH solution for one minute instead of water to avoid corrosion and the creation of iron oxide.

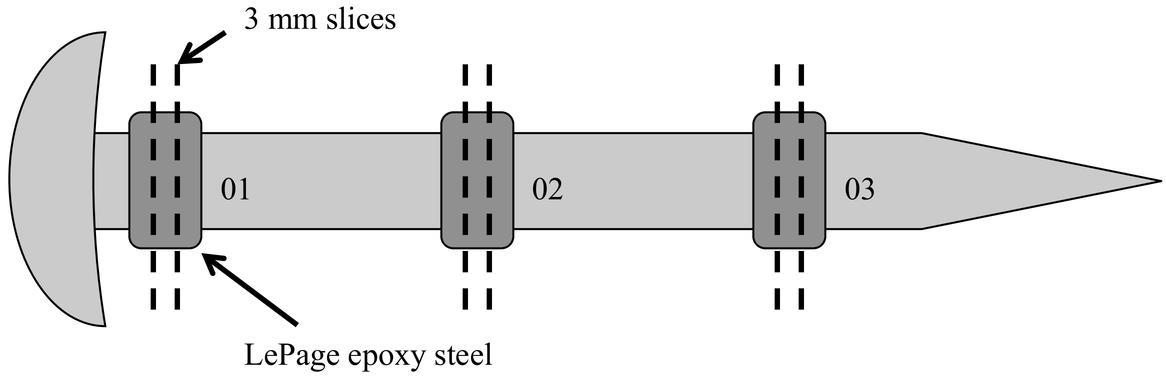

Fig. 4.3 Diagram showing methodology for cross-sectioning an iron artifact. Darker grey areas are locations where LePage epoxy steel was applied to secure the outer iron oxide layer. Hatched lines depict points where the precision sectioning saw cut the artifact in 3 mm sections.

Polishing the epoxy pucks was first attempted using a Buehler MetaServ 250 grinder-polisher in the Applied Archaeological Sciences Lab in the Department of Archaeology at Memorial University of Newfoundland. The samples were held by hand on the polishing wheel and moved in a figure-eight motion using 600 grit, 800 grit, 9 µm, 6 µm, 3 µm and 1 µm diamond abrasive SiC disks at 400 rpm. Samples were sonication in a 95% ETOH solution for one minute between polishing steps. This method failed to produce a sufficiently polished surface on the samples necessary for SEM analysis, which is a polished surface at 1 µm (Fig. 4.4). The sample preparation of iron surfaces for this methodology required the use of automatic instruments to prevent striations and sufficiently create the surface quality necessary for scanning electron microscopy.

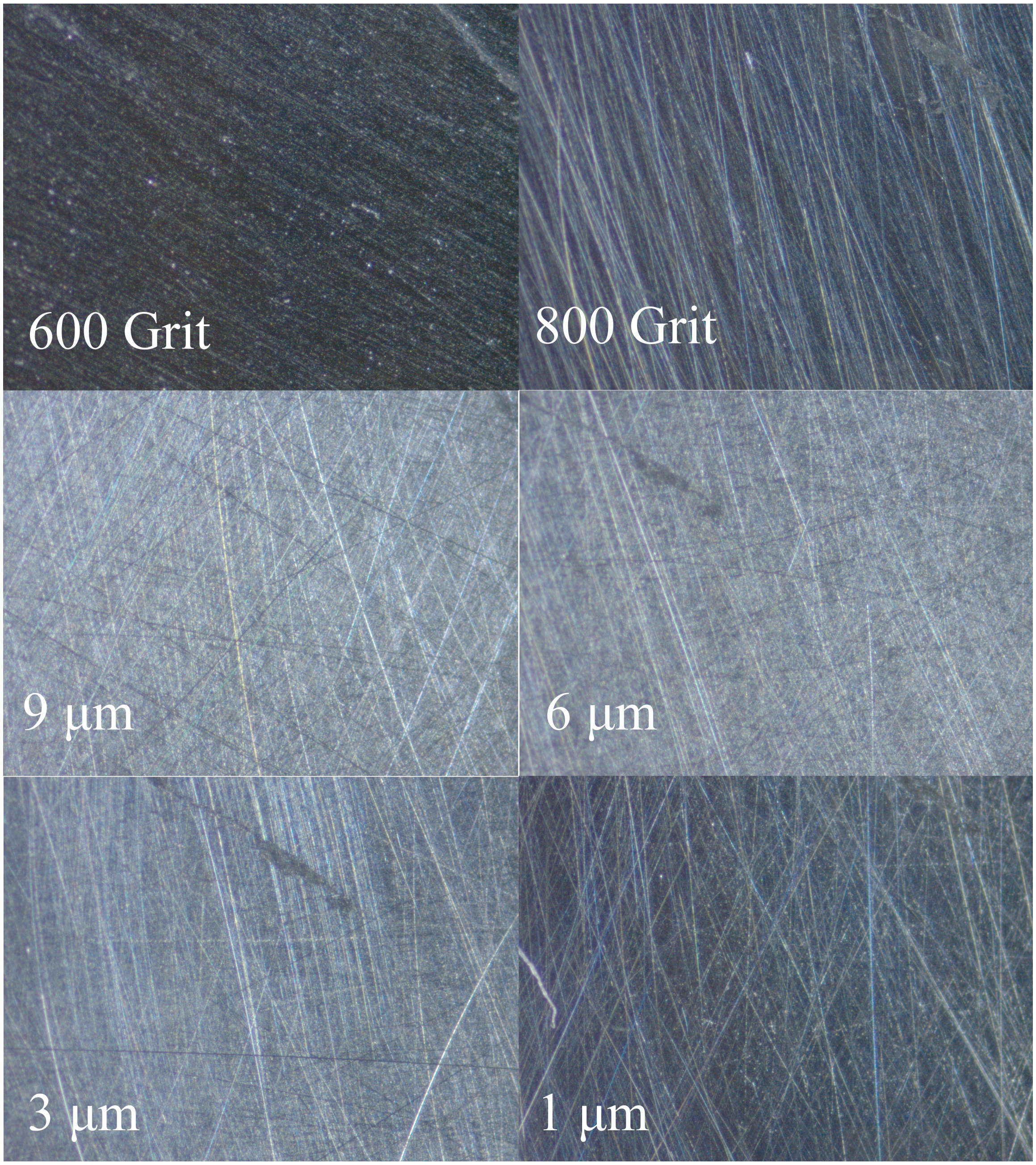

Fig. 4.4 Images of the surface of a sample from artifact number 241 (TH-30-01) from Nachvak (IgCx-03), House 2 under a magnification of 50x. These images were taken after various stages of polishing using abrasive paper with progressively smaller levels of coarseness by hand at 400 rpm with a Buehler MetaServ 250 grinder-polisher. These images illustrate the difficulty of attempting to create a polish of 1 micron in the absence of even and consistent pressure applied for several minutes ranging from approximately 20 to 40 minutes.

To obtain a surface that is observably polished to 1 μm at a magnification of 50x for SEM, the use of an automated system is required. At the Micro Analysis Facility (MAF-IIC) at Memorial University of Newfoundland, a Struers Tegramin-30 grinder-polisher and a Struers TegraPol-31 automated grinder-polisher were used. The embedded nail samples (Fig. 4.5) were placed in specimen holders and six samples were automatically polished simultaneously with an even application of force in a circular motion. The samples were polished for thirty minutes on a 6 μm Largo plate, forty minutes on a 6 μm DUR plate, forty minutes on a 3 μm DUR plate and twenty minutes on a 1 μm DUR plate. The grinder-polisher was lubricated with a Struers DP-Lubricant Green solution and all of the polishing stages had intermittent sonication steps in a 95% ETOH solution for five minutes. All of the samples were then carbon-coated before SEM analysis to make the sample conductive.

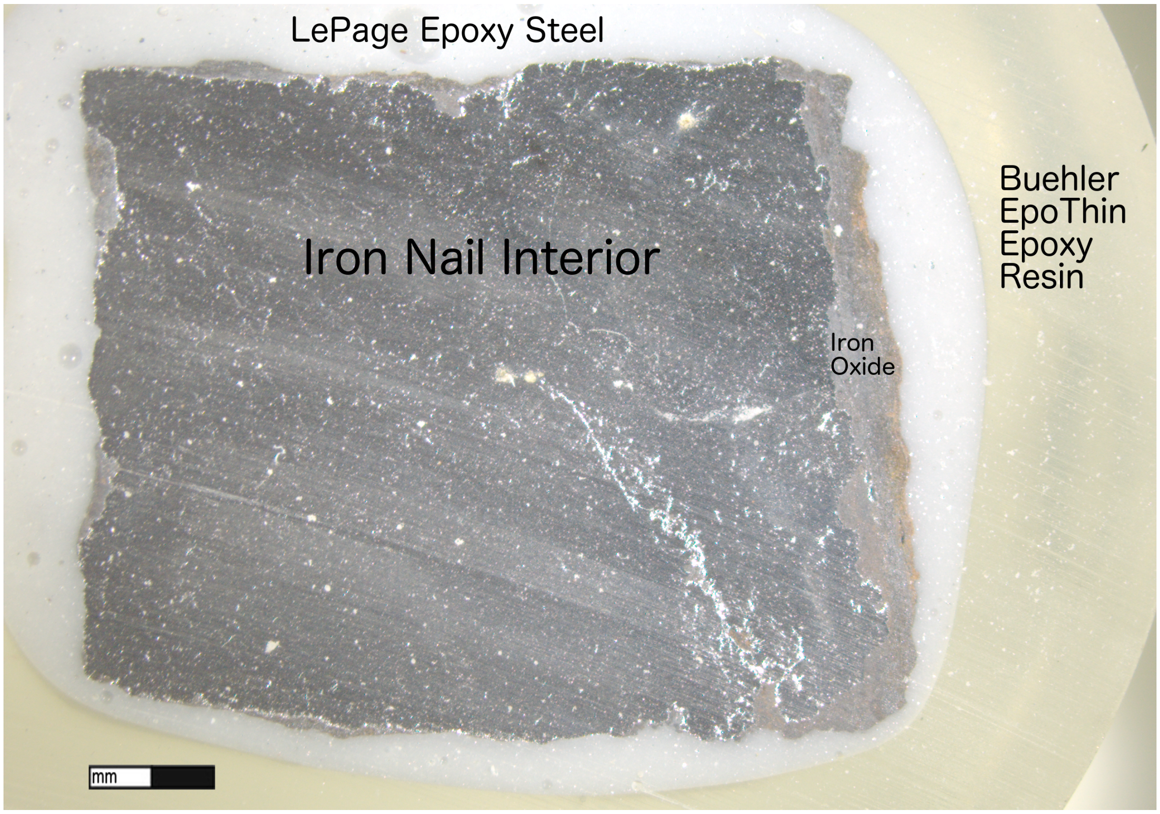

Fig. 4.5 Image of the cross-sectioned SEM-EDX sample from House 4 of the Indian Harbour, Huntingdon Island 5, FkBg-03 (TH-07-01) pre-grinder-polisher at a magnification of 50x.

4.2.3 Analytical Procedure for SEM-EDX

The SEM-EDX analysis was also performed in the Micro Analysis Facility (MAF-IIC) at Memorial University of Newfoundland. The type of instrument used was a FEI MLA 650F FEG scanning electron microscope that includes dual Bruker 5th generation XFlash silicon drift X-ray detectors (SDDs). Several scans were taken horizontally from left to right on each sample to composite a line-scan. Each scan is exactly 3000 μm across and the number of scans taken from a sample depended on the width of the individual thin-section (Fig. 4.6). A total of 341 scans were taken from the 73 prepared samples. Seventeen elements were analyzed including Al, Ca, Cr, Cl, Co, Cu, Fe, Mg, Mn, Ni, P, K, Si, S, Sn, Ti and Zn. The resulting data was compiled in a spreadsheet in Excel while further quantitative analysis was conducted on the JMP 11 statistical software program.

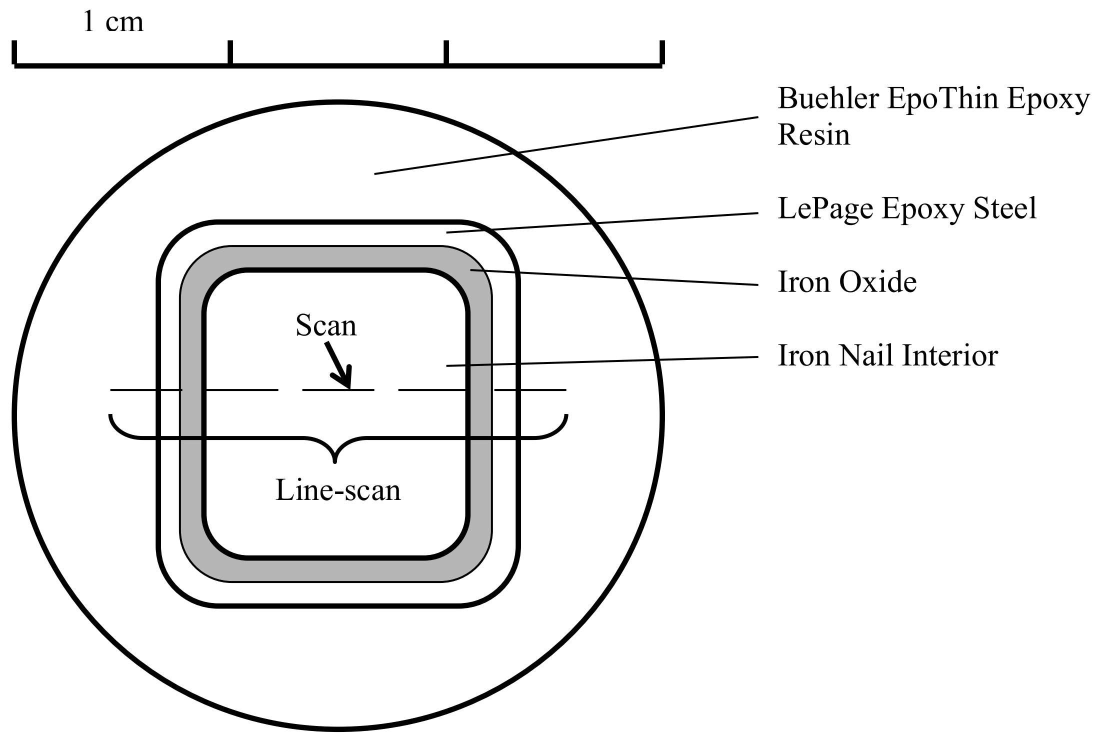

Fig. 4.6 Diagram of a line-scan for SEM-EDX on a nail cross-section sample. The 3 mm slices vary in size, shape and oxidation. This figure illustrates how a line-scan represents between three to seven stitched scans across the nail section.

The SEM-EDX data came in the form of 341 Excel spreadsheets of individual scans. These scans are 3mm long and each spreadsheet provided 100 chemical concentrations per element from different points along the scan. The accompanying *Backscattered Electron* (BSE) image illustrated the presence of epoxy as well as layers and veins of iron oxide that needed to be removed from the data to avoid contamination in the final results (Fig. 4.7). This was done by going through each individual Excel spreadsheet and omitting data where iron oxide was present in the accompanying BSE image. After iron oxide was purged from the individual scan data, these Excel spreadsheets were then conglomerated into spreadsheets that represent the spectra of an entire line-scan of the nails' uncontaminated interior (Table 4.4). In order to further condense the sample data, the statistical mean ($\bar{x}$) concentration was taken from each element in each data set of a sample and compiled in a table that was analyzed in JMP 11.

Table 4.4 SEM-EDX scales of analysis.

Scale of Analysis	**Definition**	**Number**
Site	Locations where the artifacts were found	8 (13 structures)
Artifact	Wrought iron nail or spike	32
Sample (Line-scan)	Thin-section of an artifact embedded in an epoxy puck	73
Scan	Individual scans that combine to form a single horizontal line-scan of an epoxy puck	341

Because of the qualitative nature of the data the type of statistics used to graph the results of this analysis were biplots contrasting two mean elemental concentrations for the purpose of visually detecting a correlation among sites, samples or artifacts. Significant overlap of elemental concentrations among these groups of samples would represent similar geochemical signatures and a high probability that they share the same provenance. More quantitative tests like principal component analyses and cluster analyses would be unnecessary as the objective of this study is to identify qualitative alignments and disparities among geochemical signatures as opposed to judging quantitative nuance that would be of little use in displaying the existence of desired patterns.

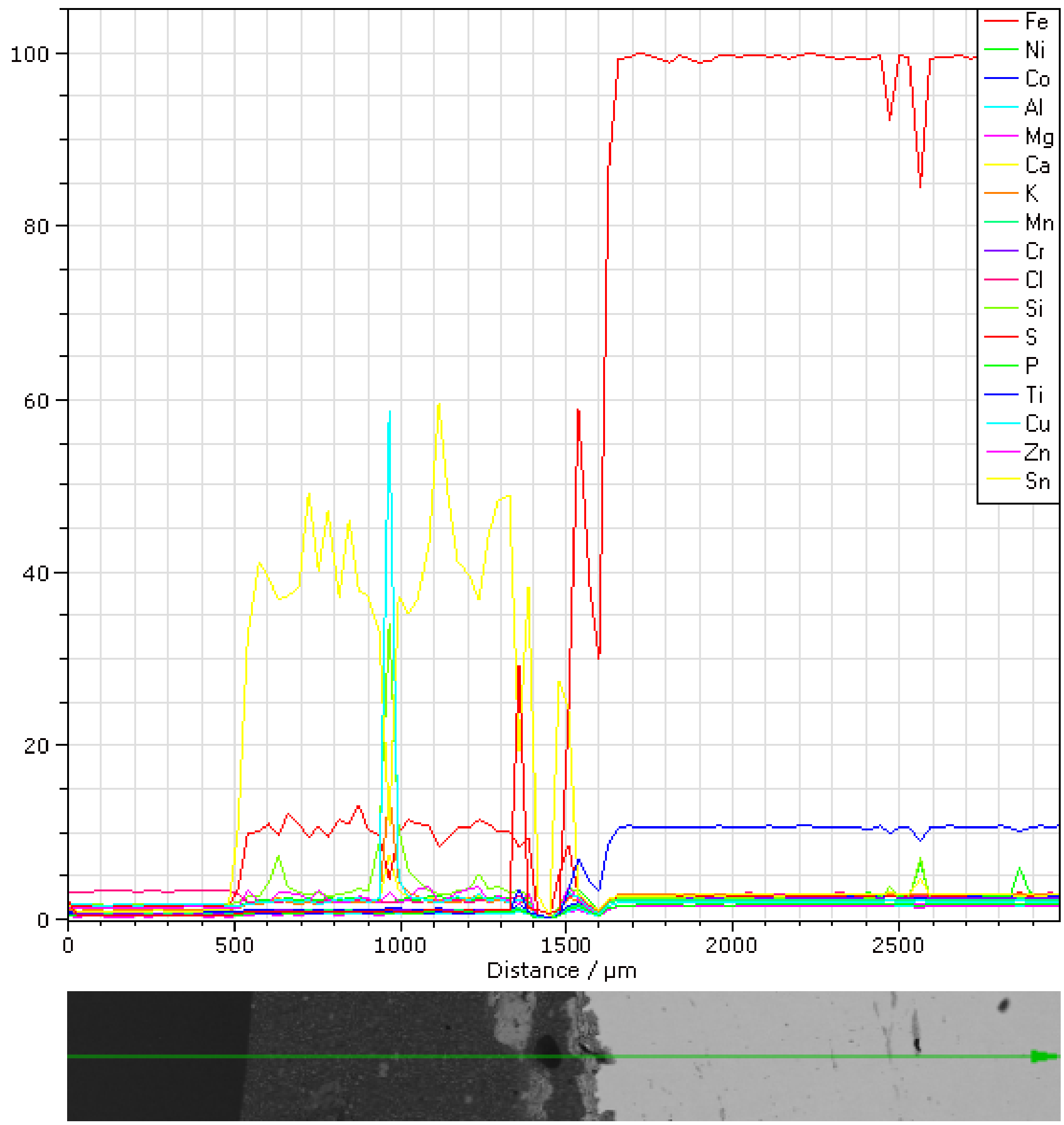

Fig. 4.7 Backscattered Electron (BSE) Image for scan 1 of a sample from Huntingdon Island 5 (FkBg-03), House 4 (TH-02-01). The y-axis represents percentage of elemental concentrations. The black area to the left represents the epoxy resin puck while the dark grey material observable from 500 to 1500 µm is the LePage epoxy steel. A thin layer of oxide is visible from 1500 to 1600 µm while the light grey area to the right represents the interior of the iron nail. A small impurity of unknown origin is present in the spike between 2500 and 2600 µm.

Chapter 5: Results

5.1 Inductively Coupled Plasma-Mass Spectrometry

The data obtained from the analysis of 32 samples analyzed via ICP-MS were organized in MS Excel before being transferred to JMP 11 for statistical analysis. A scatterplot matrix analysis involving all 39 elements was performed to assess the presence of patterns in the data and which combinations of elements, if any, demonstrate an observable clustering of data-points. Because of the inclusion of iron oxide in the results, the scatterplot matrix did not show patterns in the data of any meaningful kind (Fig. 5.1). Iron artifacts from the same site showed no geochemical consistency and as a result no observations about correlations or contrasts in the artifacts' provenience could be made. Artifacts excavated from the same sites would not necessarily have the same European origin. However, the fact that there were no clusters among artifacts, regardless of site affiliation, shows that the sample preparation, methodology and analytical capacity of the ICP-MS was unable to detect consistent geochemical signatures. ICP-MS was also unable to detect consistencies within the oxidized material (outer rusted surface of a nail) as well.

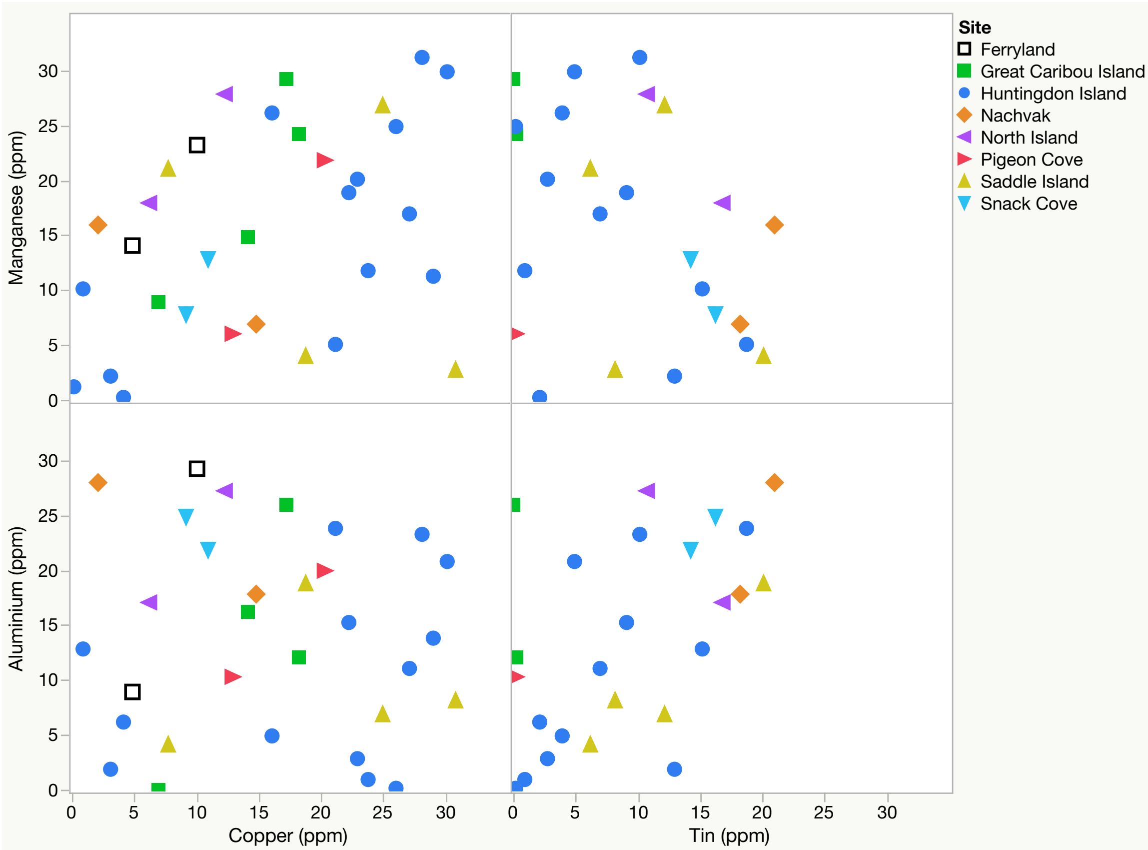

Fig. 5.1 Biplots of the elemental concentrations of Al, Mn, Cu and Sn for each of the thirty-two iron artifacts 'tips' sampled for ICP-MS.

5.2 Scanning Electron Microscopy-Energy Dispersive X-Ray Spectrometry

5.2.1 Individual Nails

Figure 5.2 displays the geochemical consistency among individual scans from the same line-scans from an artifact (TH-21) from House 2 of the Inuit site called Indian Harbour, Huntingdon Island 5 (FkBg-03) in central Labrador. It illustrates the difference between the geochemical signatures of different sections of the same artifact that can occur as demonstrated by the geochemical consistency among individual scans from the same line-scan in relation to the scans from other line-scans from the same artifact. The extent of geochemical grouping among different line-scans in relation to each other shows a significant amount of geochemical homogeneity within the same section of an artifact while the artifact demonstrates a substantial degree of geochemical heterogeneity overall.

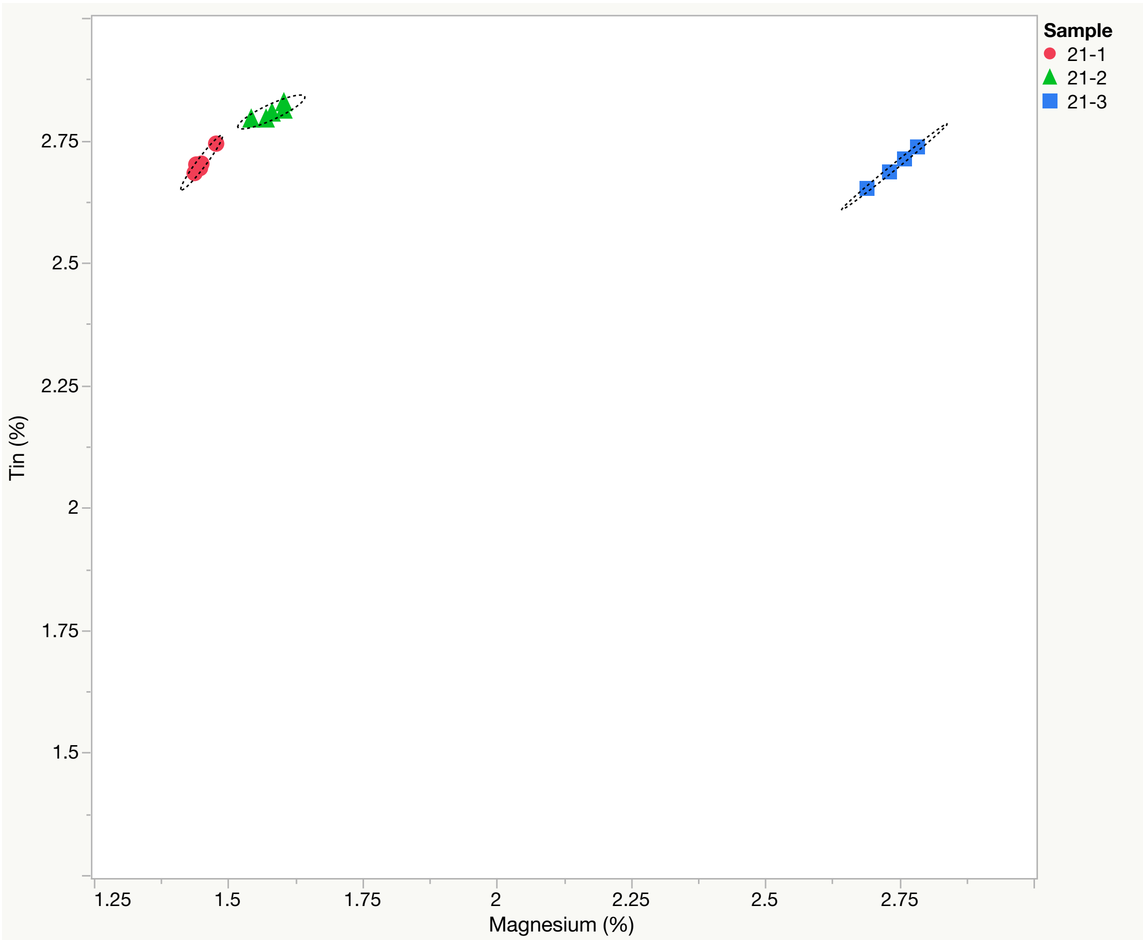

Fig. 5.2 Biplots of mean ($\bar{x}$) elemental concentrations of Sn and Mg of individual scans for one European-derived Inuit nail (TH-21) from House 2 in Indian Harbour on Huntingdon Island. Density ellipses surround scans from the same sample or line-scan with 95% confidence.

Figures 5.3, 5.4 and 5.5 show the spectra of elemental concentrations for several elements along the path of the SEM composite line-scans for the three samples from an artifact (TH-21) from Huntingdon Island 5, House 2 while figures 5.7, 5.8 and 5.9 illustrates the same for the three sections from an artifact (TH-10) from Huntingdon Island 5, House 1. These chemical spectra display how inconsistencies in concentrations of various elements are dispersed along the path of the SEM-EDX beam from left to right. Compared to the figures that illustrate the spectra of the other two samples from the same artifacts, the courses of these elements diverge considerably. This inconsistent path, along with the substantial quantity of data, is why the statistical mean ($\bar{x}$) concentration of elements in the uncontaminated interior of the nails needs to by calculated for each scan or, in the case of Fig. 5.15, each line-scan.

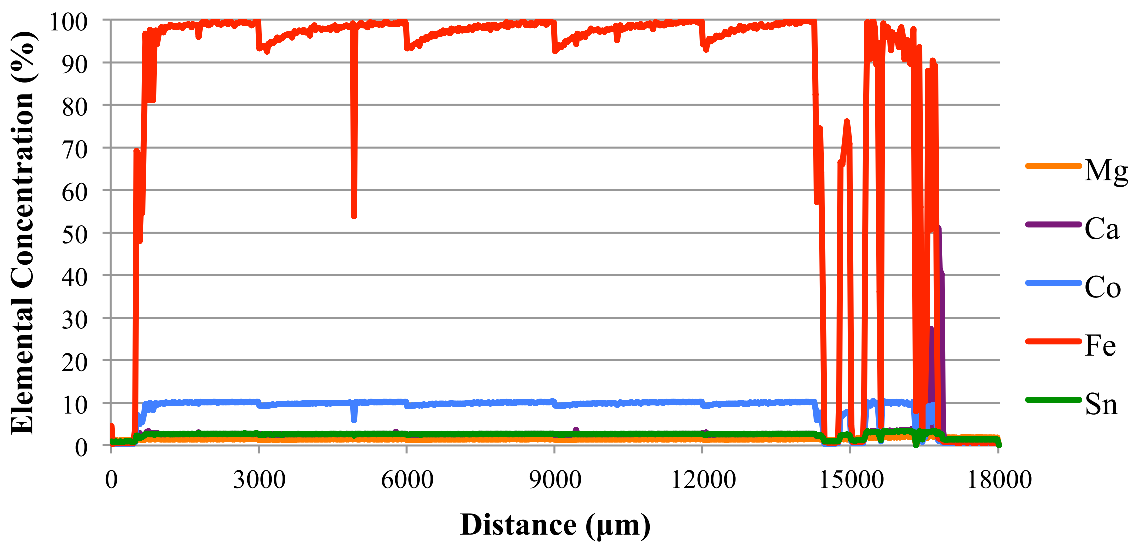

Fig. 5.3 Elemental spectrum of the composite line-scan for sample TH-21-01 from an European-derived Inuit nail from House 2 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

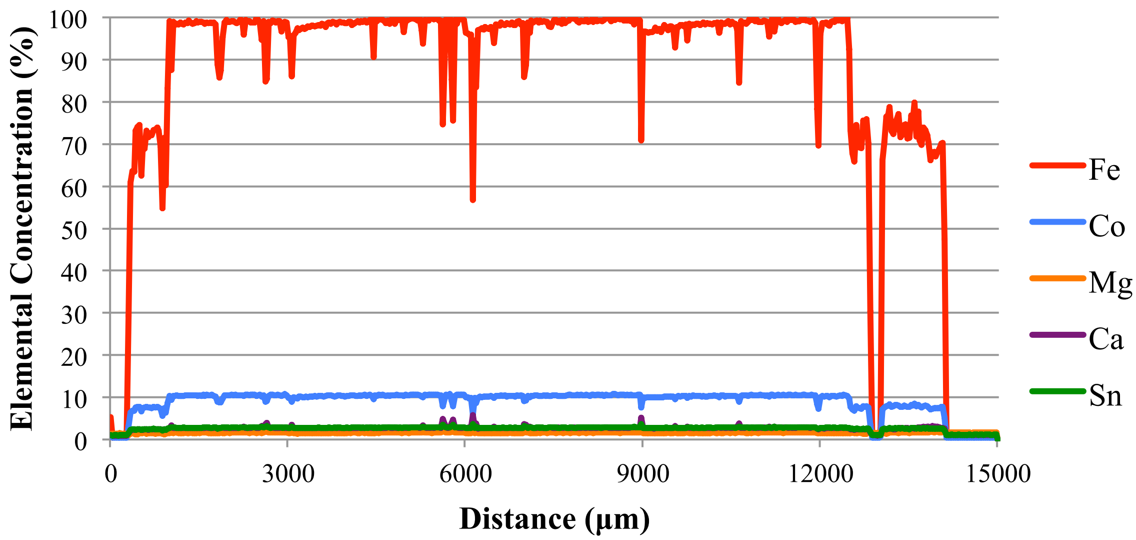

Fig. 5.4 Elemental spectrum of the composite line-scan for sample TH-21-02 from an European-derived Inuit nail from House 2 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

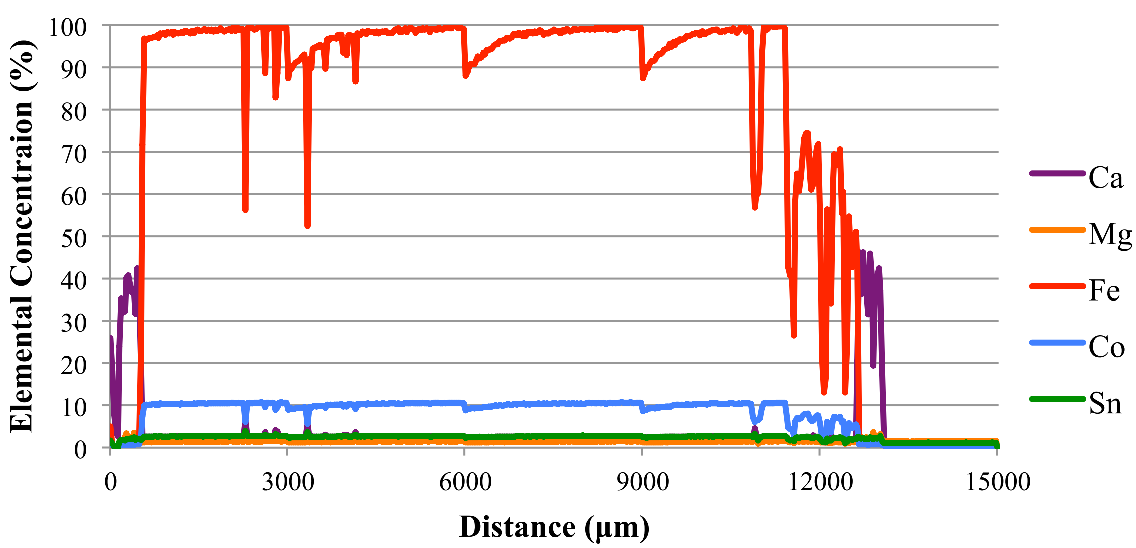

Fig. 5.5 Elemental spectrum of the composite line-scan for sample TH-21-03 from an European-derived Inuit nail from House 2 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

Figure 5.6 shows the individual scans from an artifact (TH-10) from House 1 of Huntingdon Island 5. This iron nail demonstrates the same pattern as TH-21 with individual scans clustering more tightly together in groups associated with line-scans. These clusters vary in levels of chemical uniformity, which means that some geochemical signatures are more ambiguous than others. These figures make it clearly observable that individual scans tend to cluster more closely together in relation to individual samples, thus making apparent the problem of chemical heterogeneity within this type of artifact. Different components of the shaft of nails TH-10 and TH-21 from Houses 1 and 2 on Huntingdon Island 5 are geochemically diverse to the extent that this suggests that the artifacts are too internally inconsistent to be able to provide the conclusive chemical signature representative of the entire object necessary to offer viable provenance data.

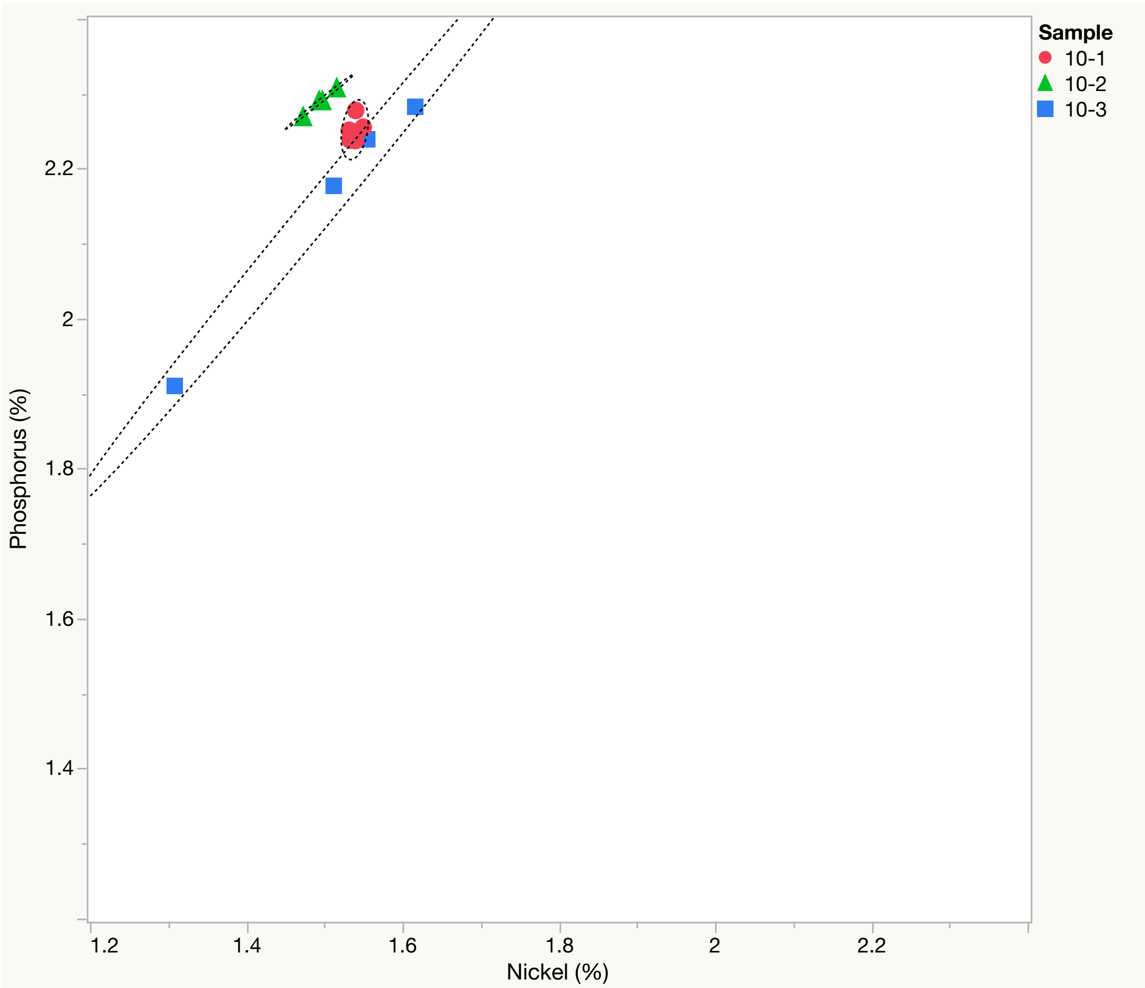

Fig. 5.6 Biplots of the mean ($\bar{x}$) elemental concentrations of P and Ni of individual scans for one European-derived Inuit nail (TH-10) from House 1 in Indian Harbour on Huntingdon Island. Density ellipses surround scans from the same sample or line-scan with 95% confidence.

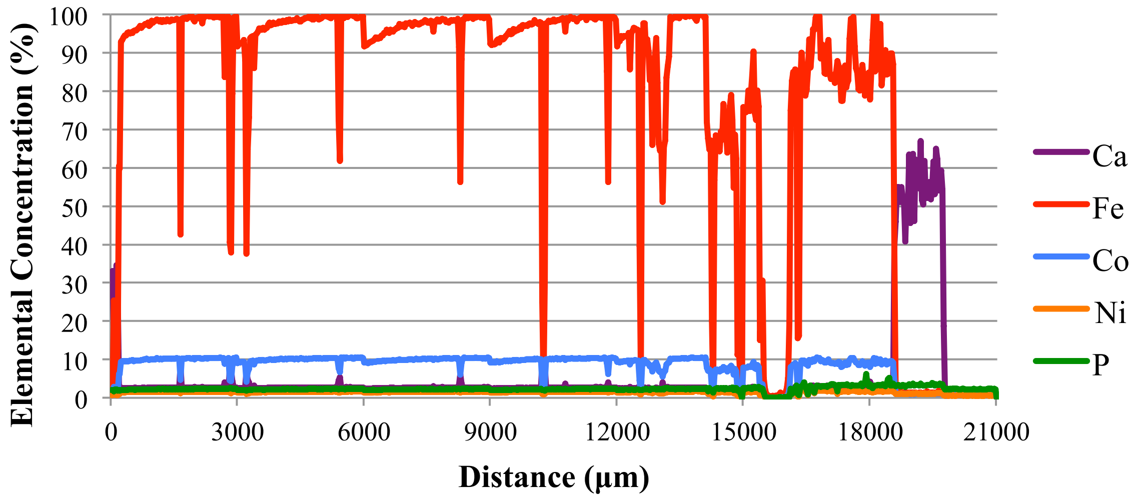

Fig. 5.7 Elemental spectrum of the composite line-scan for sample TH-10-01 from an European-derived Inuit nail from House 1 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

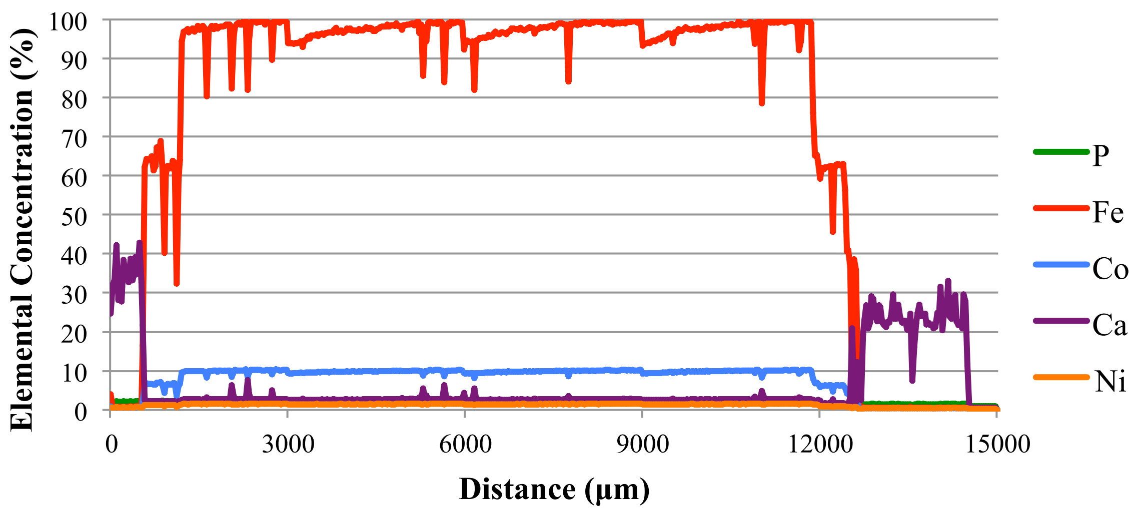

Fig. 5.8 Elemental spectrum of the composite line-scan for sample TH-10-02 from an European-derived Inuit nail from House 1 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

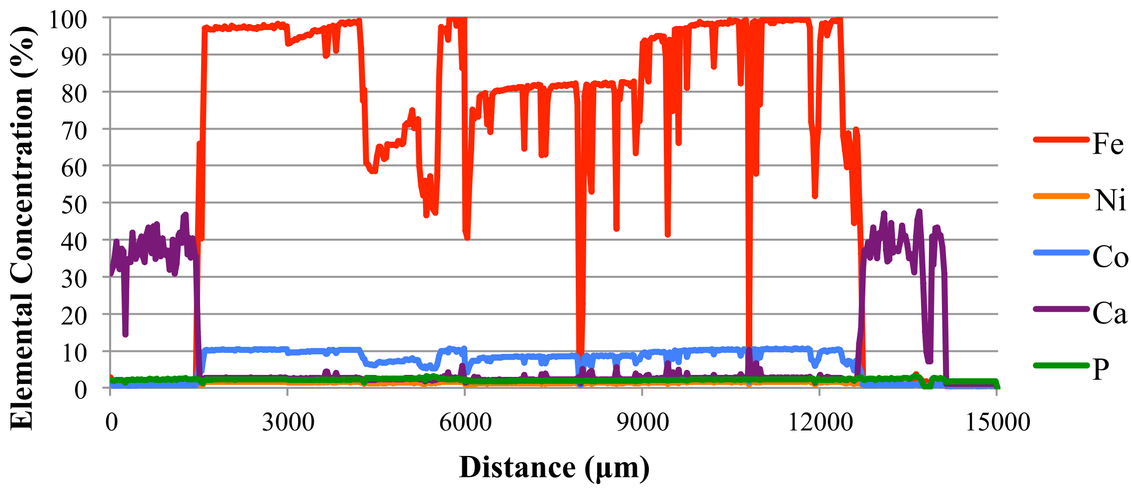

Fig. 5.9 Elemental spectrum of the composite line-scan for sample TH-10-03 from an European-derived Inuit nail from House 1 in Indian Harbour on Huntingdon Island. Each individual scan in a line-scan is 3 mm long. The sharp fluctuations in elemental concentrations represent iron oxide and other anomalies that were omitted from the final dataset prior to analysis.

Placing the individual scan data from both TH-10 and TH-21 in the same biplot illustrates how clusters representing different line-scans may have more in common chemically with the line-scans of other artifacts than with line-scans from the same artifact (Fig. 5.10). These groupings further suggest that these artifacts are not chemically homogeneous enough to produce a signature that is representative of the entire nail. If a single iron nail can demonstrate a geochemical signature that varies considerably from one end to the other then the consistency of the natural source of the iron could also vary if subjected to a provenance analysis. Since these two artifacts demonstrate that one line-scan can diverge more from other line-scans from the same artifact than line-scans from another artifact collected from another site, this data is unviable for a provenance analysis as distinct geochemical signatures may be regionally specific to different sections of the same artifact.

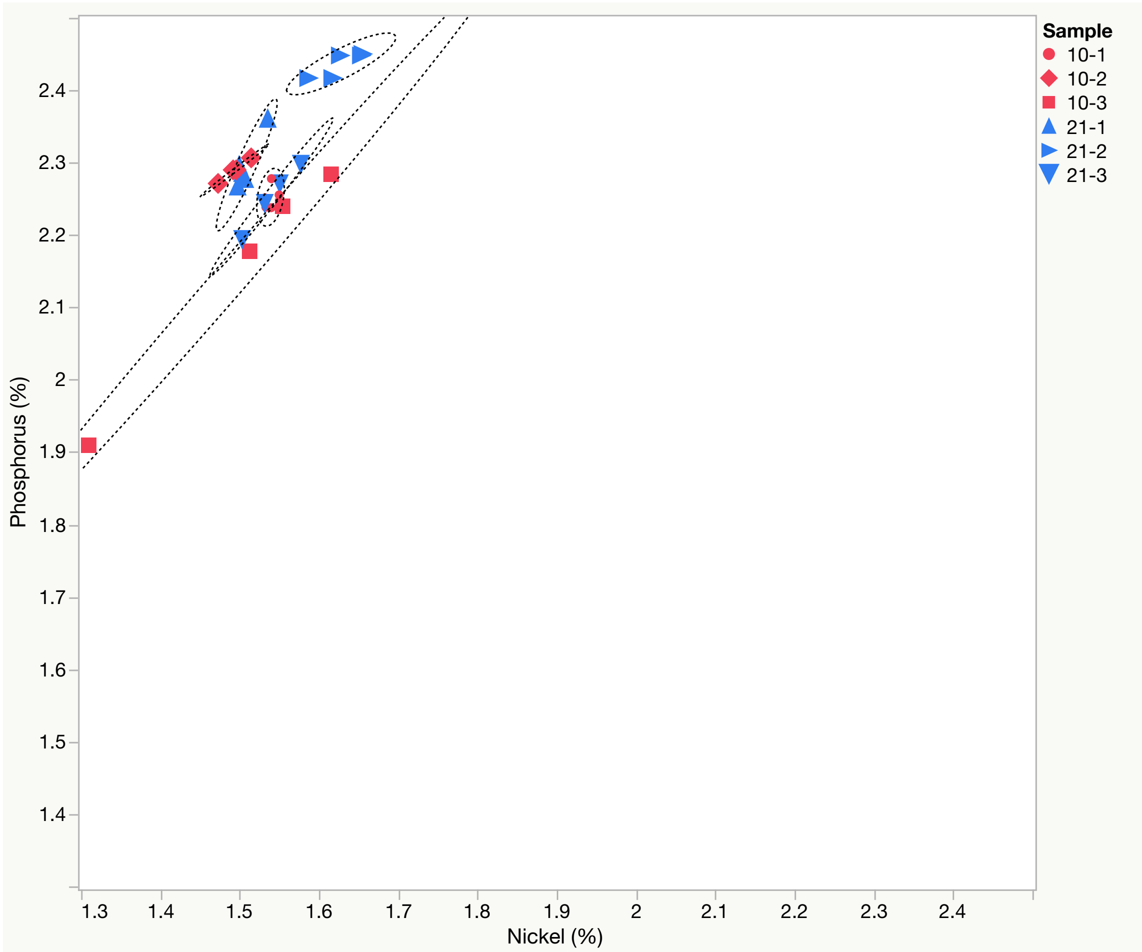

Fig. 5.10 Biplots of mean ($\bar{x}$) elemental concentrations of P and Ni of individual scans for two European-derived Inuit nails (TH-10 and TH-21) from Houses 1 and 2 in Indian Harbour on Huntingdon Island. Density ellipses surround scans from the same sample or line-scan with 95% confidence.

5.2.2 Inuit Site Samples

Figure 5.11 shows the geochemical signatures of individual scans derived from House 1 of the Inuit site from Indian Harbour, Huntingdon Island 5 (FkBg-03) off the coast of central Labrador. The combination of elemental concentrations that demonstrated the most defined clustering amongst scans in the scatterplot matrix for this group of four artifacts was P and Cu. These groupings illustrate how individual scans from the same artifact cluster together in relation to other artifacts from the same site. While the individual scans from multiple artifacts do demonstrate the presence of a geochemical signature, it is generally broader than those clusters encountered in the data of the previous section.

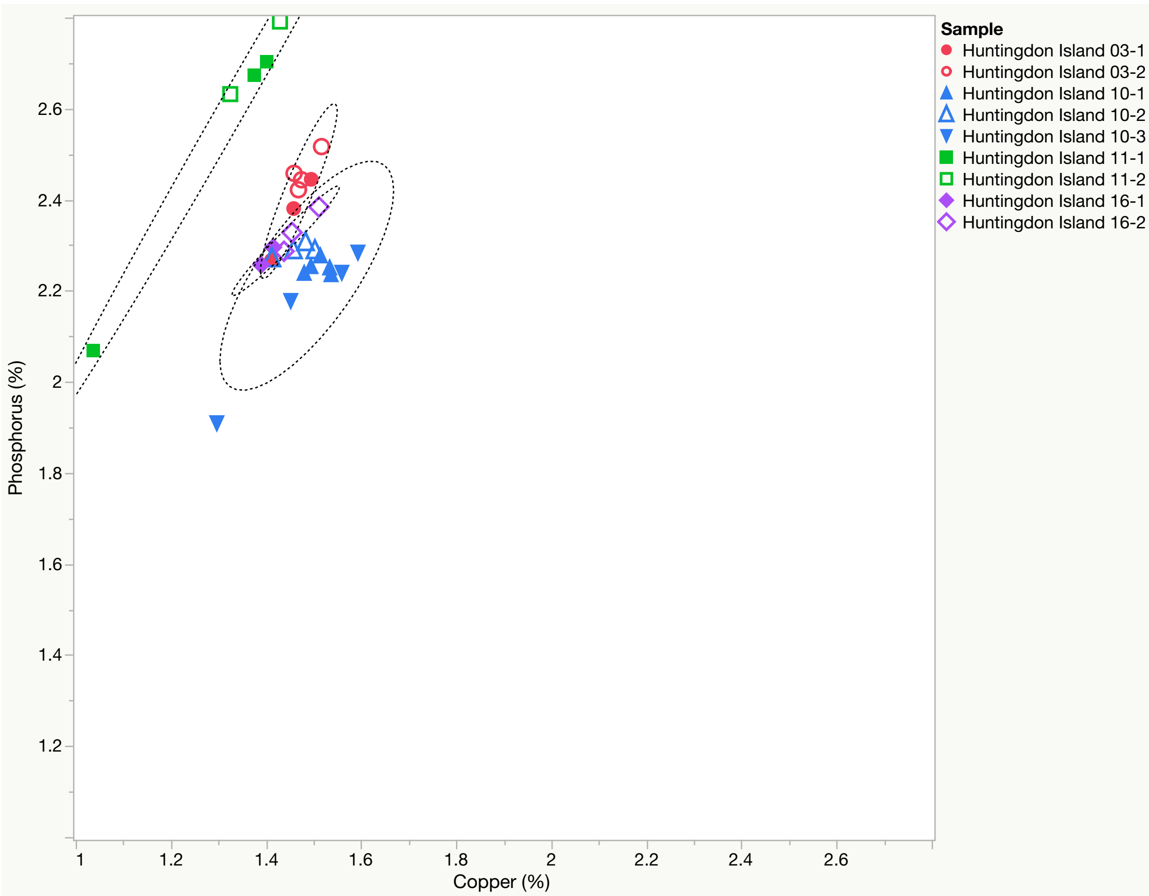

Fig. 5.11 Biplots of the mean ($\bar{x}$) elemental concentrations of P and Cu of individual SEX-EDX line scans for four European-derived Inuit nails (TH-03, TH-10, TH-11, TH-16) from House 1 in Indian Harbour on Huntingdon Island. Density ellipses surround scans from the same artifact with 95% confidence.

Samples TH-03 and TH-10 were not subjected to chemical preservation while samples TH-11 and TH-16 were exposed to sodium hydroxide treatment like all of the other artifacts in the analysis. No observable similarity is apparent between TH-03, TH-10 and most of the other nails from House 1 and Nachvak (Fig. 5.12). This likely implies that either conservation treatment does not affect the interior of the preserved nails' geochemistry or that this method was also unable to detect differences between these samples that may have been caused by chemical treatment.

Figure 5.12 displays the geochemical signatures of the individual scans obtained from House 1 of Huntingdon Island 5 compared to the Individual scan data taken from two artifacts from House 2 of Nachvak (IgCx-03) in northern Labrador. While the extent of grouping among scans varies among different artifacts and there is some overlap, there is an observable degree of geochemical homogeneity among individual scans from the same artifact. The presence of any difference among the geochemical signatures between different sites, if it exists, is more difficult to discern. Some signatures are more nebulous than others, such as TH-10 and TH-11. All of the groupings are more ill-defined than those clusters distinguished among the individual scans of a single artifact, such as TH-21 in Fig. 5.2, which makes differentiating multiple sources problematic.

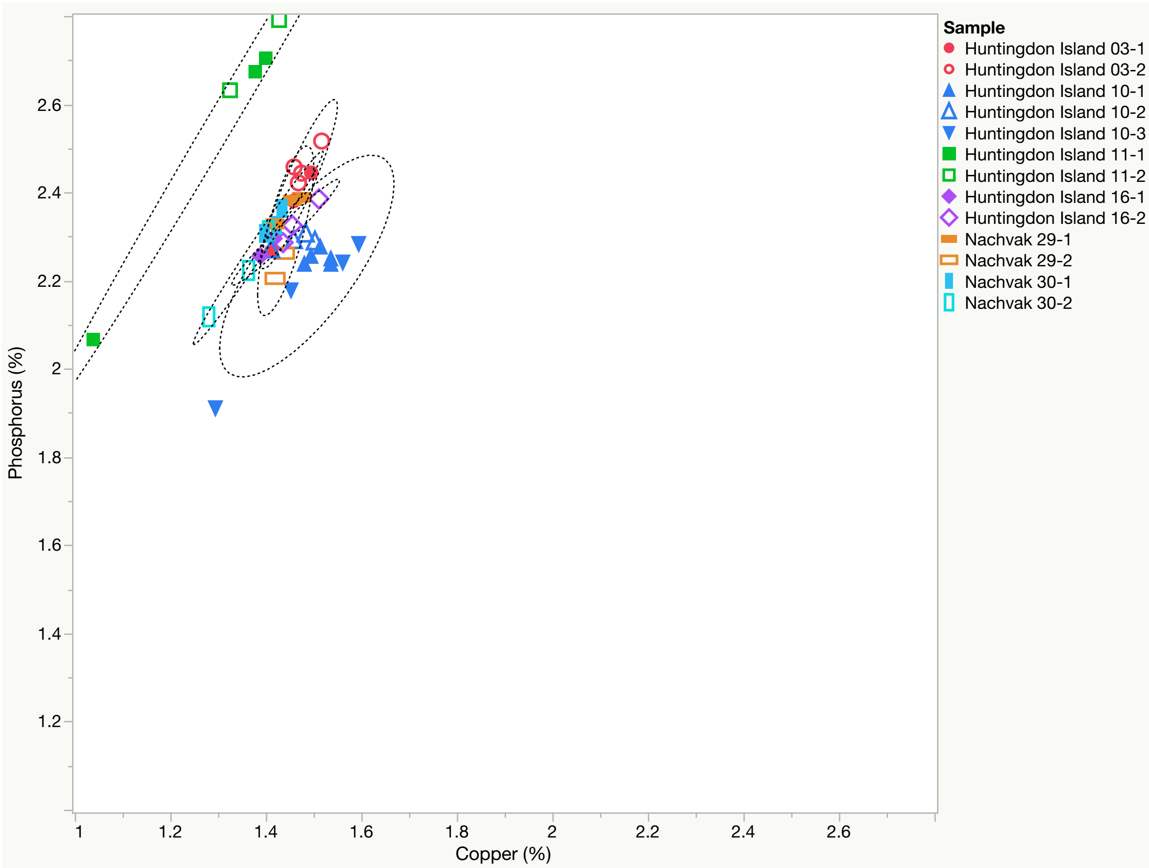

Fig. 5.12 Biplots of the mean ($\bar{x}$) elemental concentrations of P and Cu of individual scans for four European-derived Inuit nails from House 1 in Indian Harbour on Huntingdon Island (TH-03, TH-10, TH-11, TH-16) and two European-derived Inuit nails from Nachvak (TH-29, TH-30). Density ellipses surround scans from the same artifact with 95% confidence.

5.2.3 Basque and French Site Samples

Figure 5.13 shows similar geochemical patterns observed in the previous section with European samples taken from the seasonal Basque whaling station on Saddle Island in Red Bay (EkBc-01) in southern Labrador. Different artifacts illustrate varying degrees of clustering among their individual scans and there is an observable amount of overlap in elemental concentrations with the exception of sample TH-32 from Saddle Island, which exhibits approximately 0.12 % more copper than the other nails from the same site. The chemical consistency is more ambiguous with TH-18 and TH-31 than it is with the groupings of TH-17 and TH-32. As these artifacts were meant to provide a consistent geochemical signature with which to compare and possibly provenance iron nails found at Inuit sites, this observable variability in the contents of Basque nails does not provide a reliable source material.

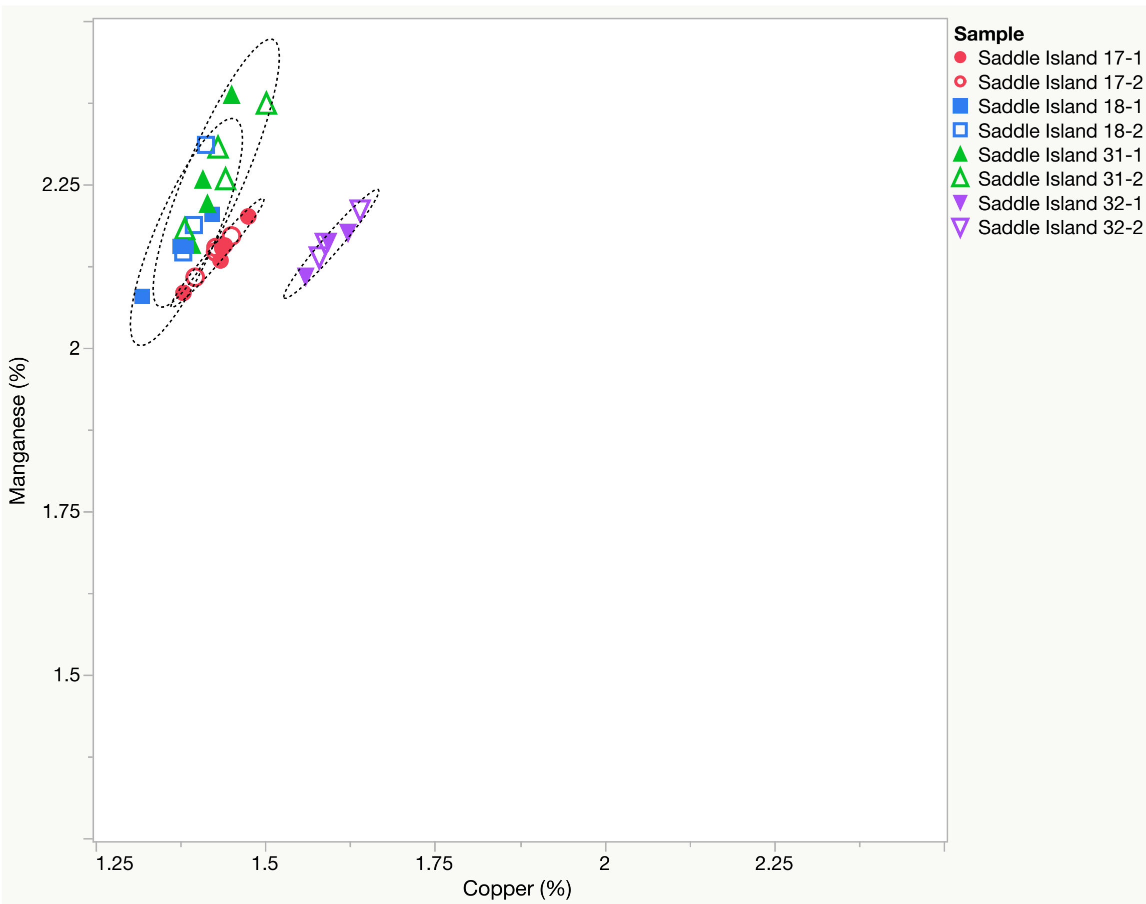

Fig. 5.13 Biplots of the mean ($\bar{x}$) elemental concentrations of Mn and Cu of individual scans for four Basque nails (TH-17, TH-18, TH-31, TH-32) from Saddle Island in Red Bay. Density ellipses surround scans from the same artifact with 95% confidence.

Figure 5.14 illustrates those artifacts from Saddle Island as well as the two iron nails from the French settlement of Dos de Cheval (EfAx-09) on the eastern coast of Newfoundland’s Great Northern Peninsula. In order to perform a provenance analysis that regards these two sites as possible sources, they would need to demonstrate two distinct and consistent geochemical signatures. The individual scans in this figure do not establish such a pattern. Like the individual scans from the Inuit and Basque artifacts, the data from the French nails showed two different degrees of consistency. French and Basque artifacts do not illustrate two distinguishable groupings segregated by the nationalities of the iron nails' archaeological contexts. Without this pattern, establishing more than one distinct source for a provenance analysis is impossible.

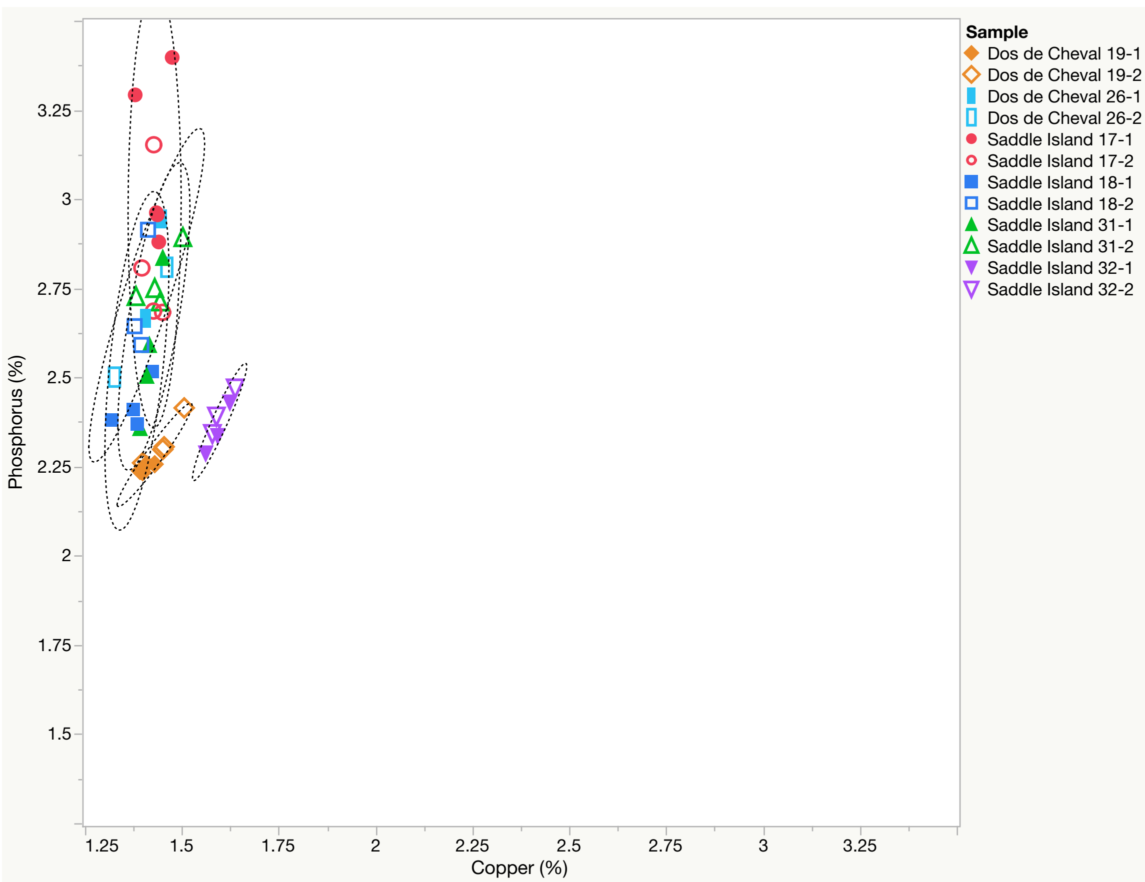

Fig. 5.14 Biplots of the mean ($\bar{x}$) elemental concentrations of P and Cu of individual scans for four Basque nails from Saddle Island in Red Bay (TH-17, TH-18, TH-31, TH-32) and two French nails from Dos de Cheval (TH-19, TH-26). Density ellipses surround scans from the same artifact with 95% confidence.

5.2.4 SEM-EDX Line-Scan Results

The mean elemental quantities of the 73 SEM-EDX line-scans do not illustrate any clear patterns among the artifacts in a scatterplot matrix (Fig. 5.15). In order to detect a greater resolution of geochemical variability within the nails, the data needed to be reduced from the mean elemental concentrations of entire composite line-scans to the mean elemental concentrations of individual scans in the previous sections. The individual scans in Figs. 5.11-5.14 illustrate more concentrated clustering in the biplots with the scans of some artifacts displaying more tightly clustered groupings than the scans of other artifacts from the same or different sites.

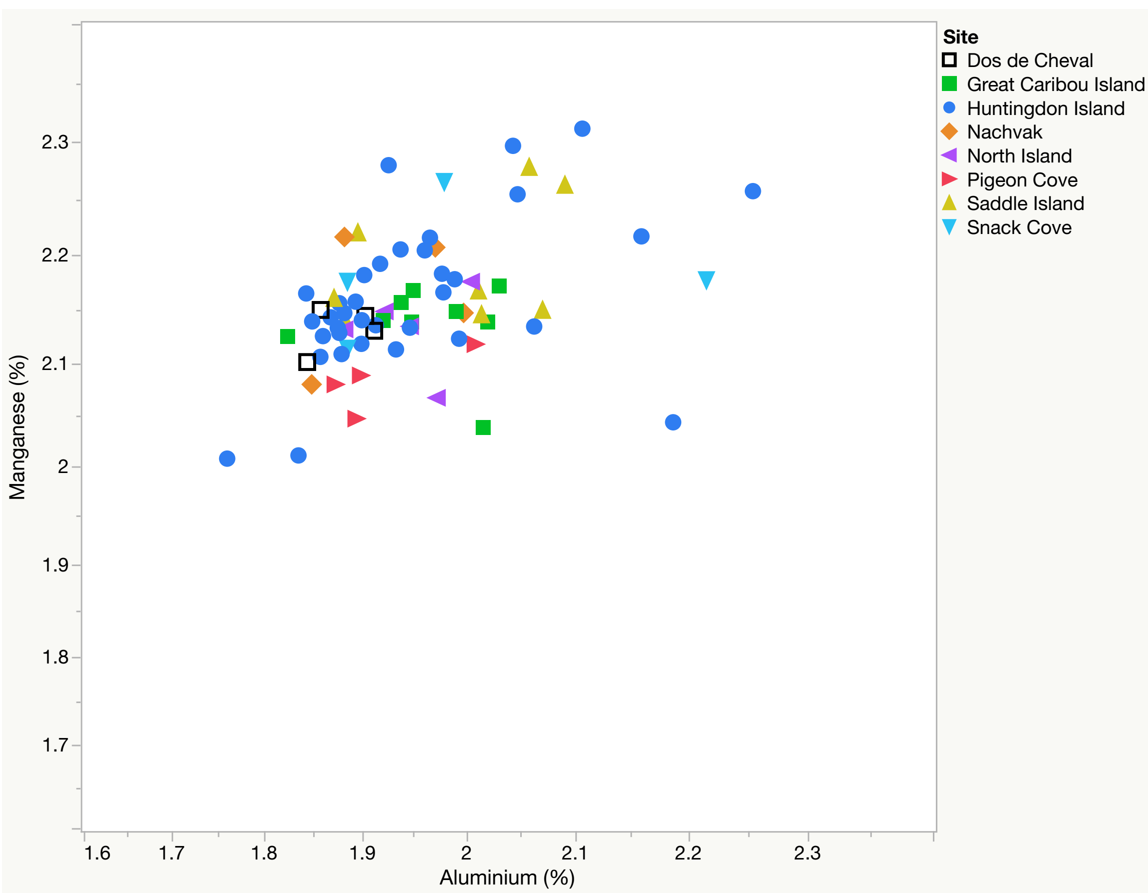

Fig. 5.15 Biplots of the mean ($\bar{x}$) elemental concentrations of Al and Mn for each of the 73 samples or line-scans involved in the SEM-EDX analysis.

Dividing the data tables from composite line-scan into individual scans before calculating the statistical mean of the elemental concentrations produces more geochemical homogeneity and individual scans are far more consistently organized into groups according to line-scan than composite line-scans are organized according to artifacts (Fig. 5.16). The greatest amount of chemical consistency was found in the individual scans from the same line-scan, which demonstrate the significant level ($p = <0.05$) of chemical homogeneity contained amongst the line-scans from the same artifact (Fig 5.2 and 5.6).

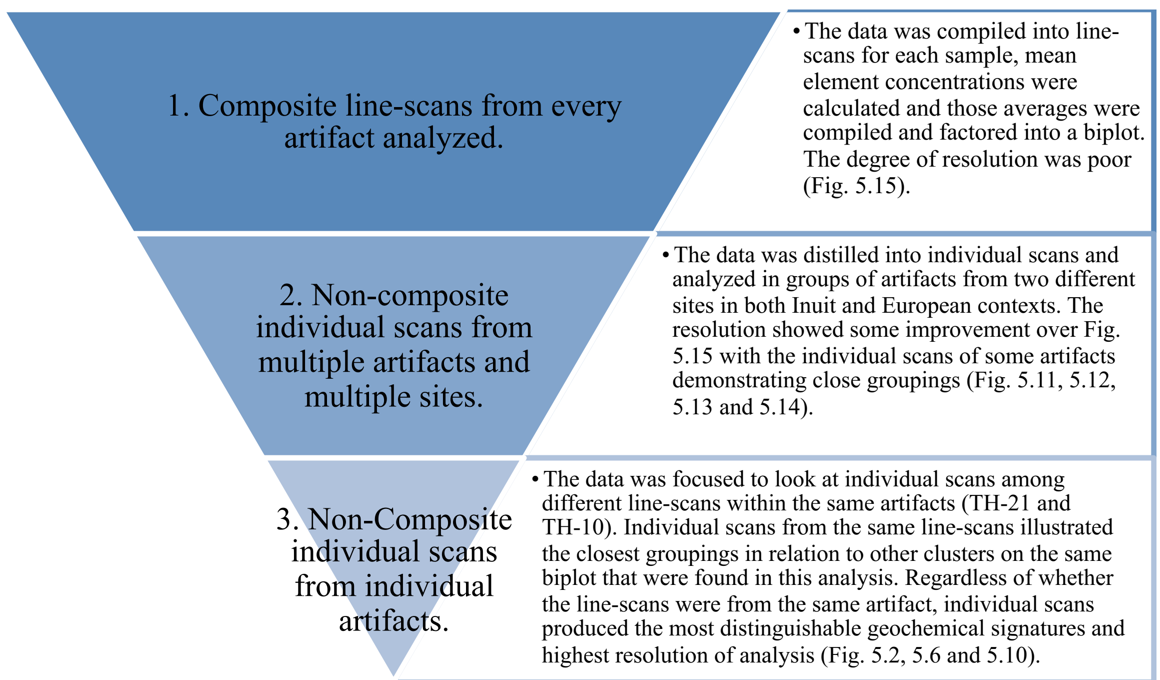

Fig. 5.16 Diagram illustrating the scales of analysis for geochemical signatures of iron nails resulting from this SEM-EDX analysis in the chronological order of the approaches taken to analyzing the data from the poorest resolution at the top to the highest resolution at the bottom.

5.3 Conclusion

The geochemical results described in this section show the varying degrees of success in detecting geochemical homogeneity in historic iron artifacts at different scales of analysis using two alternative techniques. The ICP-MS method was unable to identify consistent geochemical signatures among any artifacts. This is, as previously discussed, most likely the results of insufficient sample preparation and by analyzing the corroded exterior of the nail. In contrast, SEM-EDX technique was sophisticated enough to be able to detect homogeneity at its most consistent within specific sections of individual artifacts based on their mean elemental concentrations. This is likely due not only to the analytical methods that do not involve further corrosion of the sample with the acid washes (like ICP-MS), but also because the un-corroded, original sample was analyzed.

While individual scans from composite line-scans demonstrate the most amount of homogeneity, there is an observable degree of geochemical consistency among samples from the same artifact in relation to other artifacts. This is illustrated most clearly in the case of Fig. 5.2 and Fig 5.6 where the individual scans coming from samples derived from artifacts from the same sites tend to cluster more closely with scans from the same samples and artifacts. However, the level of homogeneity detected within the iron artifacts through SEM-EDX is still not adequate enough to establish chemical correlations between different artifacts. This is made apparent with the example of TH-21 in Fig. 5.2 and in the case of TH-10 in Fig. 5.6, which show a clear geochemical inconsistency among samples from the same artifacts in relation to scans within the same samples.

Chapter 6: Discussion and Conclusion

6.1 Geochemical Implications

The SEM-EDX analytical results described and illustrated in the previous chapter demonstrate a considerable degree of geochemical homogeneity among the three individuals scans within the same composite line-scan. Different sections from the same iron artifact (nail or spike) can show a considerable degree of chemical similarity within the same section but substantial dissimilarity between sections within the same iron nail. Figures 5.2 and 5.6 demonstrate the significant overall geochemical differences that can occur between line-scans comprising different sections within the same artifact in samples from Huntingdon Island 5, House 1 (TH-10) and Huntingdon Island 5, House 2 (TH-21) respectively. The fact that individual scans within each line-scan do show relative homogeneity compared to different line-scans within the same artifact suggests that geochemical homogeneity within an iron artifact is only possible within different areas of an iron object.

The degree of chemical heterogeneity resulting from this analysis consequently makes extrapolating information of archaeological and historic significance from this data exceedingly problematic. While the goal of this analysis was to identify geochemical signatures associated with nails coming from different European areas and determine the prevalence of these signatures in Inuit material culture, the results cannot contribute to new insights into the nature of European activity or Inuit trade in the early contact period of Labrador. It has, however, supplied a basis to develop methodologies to better analyze iron artifacts using visual means and analytical chemistry. It also tested the degree to which conservation techniques can affect geochemical results, which does not appear to be a major problem and more nails can therefore be tested that have gone through a sodium hydroxide treatment.

There are several possible explanations for this apparent heterogeneity contained in the artifacts' chemical composition. One of the most likely causes of this phenomenon is the manufacturing techniques originally employed to make the artifacts, which as discussed in chapter three, were highly varied and unstandardized. The various resulting chemical additives from these smelting inconsistencies could have affected different sections of the artifact more than others. The geochemical heterogeneity may have been established earlier in the natural source of the metal. The original iron ore or the use of different ores in tandem may have created various pockets of elemental impurities that were not evenly distributed throughout the artifacts' composition. A combination of these factors could have contributed to the issue of heterogeneity within the artifacts, making broader chemical correlations among different objects, sites and cultural groups unfeasible based on the data provided by the SEM-EDX technique using only line-scans.

Provenience analysis requires a substantial degree of geochemical homogeneity among different artifacts as well as within the same artifact. Since the different sections of the same artifact have shown a significant quantity of chemical divergence the explanatory potential of the results described in this thesis cannot accommodate the basic principle of chemical provenience. This fundamental concept called the provenance postulate emphasizes how "the raw material source responsible for an artefact can be successfully determined through chemical analysis as long as between-source chemical differences exceed within-source differences" (Glascock and Neff 2003:1521). In the ideal analytical scenario artifacts from the same source should have a detectable and consistent chemical signature they share amongst each other that artifacts from a different source do not possess. Since the data demonstrates that different sections from the same artifact have significant chemical inconsistencies, chemical correlations at the level of artifact assemblages among different sites are impractical. The level of geochemical homogeneity detectable within the artifacts is insufficient to satisfy the parameters of the provenance postulate.

6.2 Archaeological Implications

The results of both analytical methods of analysis emphasize the problematic nature of performing a geochemical provenance analysis of iron implements that were manufactured using late medieval and early modern smelting techniques. The internal mineralogy of the iron nail artifacts was too heterogeneous to demonstrate meaningful insights regarding cultural exchange, chronology, trading patterns or the specific regional European affiliation of the raw material in nails acquired from Inuit sites. The main implication provided by the findings of this analysis emphasizes discretion and questioning certain assumptions about iron artifact homogeneity in archaeological provenance research.

Numerous studies such as those listed in chapter three have been conducted under the premise that the artifact mineralogy of iron nails has the potential to demonstrate homogeneity to a degree that pacifies the provenance postulate. The danger in assuming or exaggerating the homogeneity of iron artifacts is an issue that needs to be more extensively addressed in provenance research. Archaeologists needs to be cautious that they are not analyzing artifacts under the fallacy that all artifacts demonstrate the exact same geochemical result regardless of what part of the object it is taken from. A comprehensive analysis of multiple components taken from individual artifacts is first necessary to demonstrate that the artifact is suitably homogenous enough to establish a consistent geochemical signature that represents the entire artifact. Caution must also be exercised when designing a methodology to ensure that the chemistry of the results is not unintentionally altered due to supplementing the samples with chemical additives.

This thesis did prove a direct correlation between the materials analyzed, but not at a scale of analysis that would have proved useful for provenance research. This studies' hypothesis that there exists a geochemical relationship between the numerous artifacts of various cultural, geographic and temporal affiliations involved in this research is therefore null. Neither inductively couple ICP-MS or SEM-EDX could detect significant parallel geochemical signatures beyond different scans in the same line-scan of an individual nail section. Based on the results of this research there is no alternative but to conclude that a material culture relationship between early contact period European and Inuit groups based on the chemical constituents of iron nails cannot be established through these methods alone. This result suggests that either the methodological techniques or statistical tests used in this thesis were not sophisticated enough to detect the presence of a correlation or perhaps such a relationship does not exist, and if it did, it is not possible to detect with the analytical methods available at present.

6.3 Methodological Issues

The methodology of this thesis had to address several procedural issues in order to insure that the integrity of the samples were not compromised and that the results would be as accurate as they could possibly be. The ICP-MS approach presented various challenges where contamination and error were major concerns. The manner in which samples of the iron interior of nail artifacts were extracted was ill-equipped to prevent the inclusion of iron oxide and preservatives on the exterior of the nail during the collection of the samples. The introduction of nitric acid and hydrochloric acid in the process of the sample digestion was also an issue that likely initiated the creation of chemical compounds such as iron nitrate and iron chloride that caused a substantial chemical manipulation of the samples and therefore an alteration of the final results. The ICP-MS component of this study only included a single sample taken from each artifact as opposed to multiple samples in the SEM-EDX analysis. Much of the disorder visible in the ICP-MS data likely arose from inadequate sample preparation and methodological issues from insufficient sample size from each iron nail.

The SEM-EDX analysis was more successful in anticipating issues involving contaminants and chemical manipulation. The sample preparation process took precautions to avoid the irreversible manipulation of the chemical structure and contents by applying lubrication to the saw-sectioning process to obstruct the creation of sparks and sonication in 95% ethanol to prevent oxidation. The polishing procedure of the sample preparation process was the subject of several complications. Polishing the epoxy pucks containing the samples by hand on the Buehler MetaServ 250 grinder-polisher in the *Memorial Applied Archaeological Sciences Lab* proved to be inadequate in eliminating large striations resulting from the sectioning saw. This step is required to analyze the surface of the sample with the SEM-EDX beam without analytical interference resulting from uneven surface contours. The even and consistent pressure applied to the sample pucks in the *Micro Analysis Facility* on the automated Struers grinding and polishing equipment was far more effective in reducing the size and quantity of striations to the desired minimum.

6.4 Suggestions for Further Research

There are several possible procedural adjustments to this research that if implemented could greatly improve the scope of this analysis. This thesis emphasizes the great necessity for the interdisciplinary collaboration required in conducting an optimally productive provenance study. Apart from historical and anthropological insights, analytical chemistry and materials science expertise is also of vital importance when approaching research questions in the archaeological sciences. As archaeologists often have a minimal understanding of these areas, the relevant specialists should be consulted for guidance. For instance, a more prudent research design would first try to establish whether consistent geochemical signatures from known sources could be discerned before proceeding with a more comprehensive analysis or a large collection of samples.

This thesis only made use of one type of iron object in particular and future studies may benefit from assessing the viability of using a more diverse range of artifact types to evaluate whether or not the manufacturing technique specific to iron nails has a large impact on the artifacts' heterogeneity. Subsequent iron provenance research in early contact period Labrador should also consider the inclusion of raw material sources or artifacts with confirmed provenance from broad manufacturing centers in Europe rather than just a comparison of the geochemical signatures of artifacts from different sites. Acquiring samples of raw iron ore and artifacts confirmed to be manufactured in the Basque Country and elsewhere in Europe may help determine the geochemical differences between the pre- and post-manufactured iron material. However, even with raw material from a ‘known source’, the data may not provide the information archaeologists need to pursue the provenience postulate. Simply because ore is from Basque Country does not mean that it was used. Further to this, the impurities introduced into the smelt can also obscure the interpretation of data even when known ore sources are analyzed.

There are several different types of analytical techniques that may or may not be more effective in geochemical provenance research of iron artifacts. Using laboratory X-ray florescence spectrometry (*l*XRF) or portable X-ray florescence spectrometry (*p*XRF) may be a viable option to explore. X-ray florescence spectrometry is similar to SEM-EDX in that it uses beams of X-rays to acquire a spectral reading that represents the various elemental concentrations of a material (Rapp and Hill 2006:237). Another analytical approach could be to pursue a more extensive type of scanning electron microscopy-energy dispersive X-ray spectrometry in the form of elemental area-scans as opposed to a line-scan. The SEM-EDX area-scan provides a two-dimensional elemental false colour image or ‘map’ of a sample’s surface in contrast to the one-dimensional line-scan through mineral liberation analysis (MLA). This approach could vastly improve the level of precision and accuracy via a more thorough assessment of the elemental constituents of the samples. Instrumental Neutron Activation Analysis (INAA) is also a viable option that lacks many issues involved with ICP-MS like matrix interference, though it requires access to a facility with a reactor (Rapp and Hill 2006:236).

The major methodological conclusion to be drawn from this study’s analysis is the necessity of being more selective in what areas of the nails’ microstructure are analyzed and increasingly cautious of their extreme heterogeneity as a result of their manufacture and corrosion history. The SEM-EDX results are consistent with some previous articles' interpretations of the behavior of major elements (Al, Ca, Cu, Fe, K, Mg, Mn, O, P, Pb, Si, S, Ti, Na, Ni, etc.) between ore and finished product that suggest the various manufacturing stages cause them to be too variable within the same source areas and inconsistent within the same artifact to provide reliable provenance data (Leroy et al. 2012:1085; Schwab et al. 2006:439, 442). Instead, targeting trace elements seem to be the most promising option when attempting to determine geochemical correlations between ores and artifacts, as they tend to congregate in slag inclusions and remain consistent throughout the smelting process (Leroy et al. 2012:1085; Schwab et al. 2006:442). SEM-EDX line-scans are unable to precisely detect a sufficiently representative amount of slag inclusions to produce a reliable interpretation of their overall internal chemistry.

SEM-EDX area-scans can provide the spatially comprehensive data necessary to detect and target the inclusions containing trace elements that would provide more precise geochemical information. But trace elements can present analytical issues of their own since the indirect process of wrought iron manufacture may effectively delete much of the trace element signature of the ore in the inclusions during the bloomery procedure (Desaulty et al. 2008:1261; Schwab et al. 2006:448). Giving the analysis more spatial dimension on the sample surface would also allow for more thorough discrimination between what inclusion are included in the data. These inclusion may come from several different mixtures due to iron's high melting temperature and the wrought iron nails' accumulating different slags in their internal structures over a complex multi-phased manufacturing process (Desaulty et al. 2008:1253; Schwab et al. 2006:437).

The geochemical provenance of wrought iron remains a highly problematic endeavor and requires a more rigorous and precise methodology than the techniques applied in this thesis to tackle the major issues of extreme heterogeneity, variability in the manufacturing process, as well as the cultural modifications to the artifacts through time. If these obstacles can be sufficiently overcome then meaningful provenance data may yet be achievable. The purpose of this research project was to provide new insights into the contact period interactions between the Labrador Inuit and European pioneers on the east coast of Canada. Although no such conclusions were drawn in this instance, the performance of a more comprehensive provenance analysis of iron nails would yield valuable contributions to our archaeological understanding of the region's economic, demographic and cultural history.

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Appendices

Appendix 1 Measurements of the iron nails involved in the ICP-MS analysis.

ICP-MS Sample	Weight (g)	Length (mm)	Width at Tip (mm)	Width at Base of Head (mm)	Thickness at Tip (mm)	Thickness at Base of Head (mm)	Diameter of Nail Head (mm)	Width of Head (mm)
FkBg03-147	86.30	147.65	4.19	14.08	3.00	13.65	28.21	7.45
FkBg03-123	70.60	99.74	3.87	22.81	8.59	15.12	32.44	14.82
FkBg03-118	21.30	80.18	4.81	9.84	3.71	9.46	15.62	5.63
FkBg03-184	34.10	90.88	17.57	10.66	10.26	8.68	18.05	6.99
FkBg03-318	11.09	229.97	16.67	5.79	4.38	10.86	-	-
FkBg03-1344	131.30	147.12	7.03	16.02	14.85	4.48	35.17	12.30
FkBg03-615	51.70	82.91	5.95	14.25	3.92	12.95	33.91	7.17
FkBg03-297	114.50	179.13	9.08	12.79	4.32	12.71	30.22	10.09
FkBg03-1248	61.70	145.52	5.63	12.40	3.71	11.12	20.85	6.80
FkBg03-373	65.00	111.36	6.21	13.11	5.66	10.88	29.51	8.36
FkBg03-2948	137.30	194.30	7.45	14.38	6.28	12.32	-	-
FkBg03-2708	26.10	102.66	8.00	5.61	3.03	5.78	23.46	8.12
FkBg03-2991	22.60	77.97	4.62	13.09	6.02	2.70	-	-
FkBg03-4307	92.10	159.37	6.62	10.96	4.87	15.84	-	-
FlBf06-2292	49.00	84.54	13.16	14.29	1.88	11.12	-	-
FlBf06-1778	48.00	107.36	6.66	12.82	4.32	13.26	13.82	7.24
FkBe03-1229	22.60	59.24	-	29.48	-	9.18	-	-
FkBe03-1244	5.90	41.19	-	16.56	-	5.64	-	-
FeAx03-207	-	-	-	-	-	-	-	-
FeAx03-240	17.50	85.55	5.06	9.32	4.75	5.64	17.68	5.35
FbAv13-HA.1	67.60	91.90	6.90	8.40	10.16	12.36	17.84	11.55
FbAv13-HA.2	-	-	-	-	-	-	-	-
FbAv13-HB.1	26.80	94.21	6.94	13.76	4.53	9.55	16.44	7.62
FbAv13-HB.2	-	-	-	-	-	-	-	-
EkBc01-25619c	-	-	-	-	-	-	-	-
EkBc01-23190a-r	49.20	90.20	6.05	11.65	8.28	11.32	18.90	9.53
EkBc01-23152a-j	33.40	82.60	7.26	9.79	5.60	9.74	17.80	8.00
EkBc01-25985c	23.80	66.10	6.77	9.81	4.86	9.93	24.60	7.30
IgCx03-782	-	-	-	-	-	-	-	-
IgCx03-4048	-	-	-	-	-	-	-	-
FldB-794.1	36.50	72.87	24.00	28.06	23.51	13.74	-	-
FldB-794.2	94.00	86.29	19.37	25.40	27.97	15.91	-	-

Appendix 2 Measurements of the iron nails involved in the SEM-EDX analysis.

SEM-EDX Artifact	Weight (g)	Length (mm)	Width at Tip (mm)	Width at Base of Head (mm)	Thickness at Tip (mm)	Thickness at Base of Head (mm)	Diameter of Nail Head (mm)	Width of Head (mm)
TH-01	49.00	84.54	13.16	14.29	1.88	11.12	-	-
TH-02	26.10	102.66	8.00	5.61	3.03	5.78	23.46	8.12
TH-03	70.60	99.74	3.87	22.81	8.59	15.12	32.44	14.82
TH-04	48.00	107.36	6.66	12.82	4.32	13.26	13.82	7.24
TH-05	22.60	77.97	4.62	13.09	6.02	2.70	-	-
TH-06	17.50	85.55	5.06	9.32	4.75	5.64	17.68	5.35
TH-07	137.30	194.30	7.45	14.38	6.28	12.32	-	-
TH-08	114.50	179.13	9.08	12.79	4.32	12.71	30.22	10.09
TH-09	92.10	159.37	6.62	10.96	4.87	15.84	-	-
TH-10	86.30	147.65	4.19	14.08	3.00	13.65	28.21	7.45
TH-11	34.10	90.88	17.57	10.66	10.26	8.68	18.05	6.99
TH-12	24.10	84.24	5.92	8.58	3.40	10.69	17.11	3.41
TH-13	82.70	140.61	10.61	11.00	10.32	12.40	18.80	11.41
TH-14	60.30	100.31	9.44	13.82	10.47	13.67	19.75	8.96
TH-15	67.60	91.90	6.90	8.40	10.16	12.36	17.84	11.55
TH-16	21.30	80.18	4.81	9.84	3.71	9.46	15.62	5.63
TH-17	49.20	90.20	6.05	11.65	8.28	11.32	18.90	9.53
TH-18	33.40	82.60	7.26	9.79	5.60	9.74	17.80	8.00
TH-19	77.00	128.02	2.04	154.49	6.03	17.38	28.35	12.26
TH-20	33.70	77.87	2.86	10.72	5.37	10.43	23.09	6.66
TH-21	131.30	147.12	7.03	16.02	14.85	4.48	35.17	12.30
TH-22	11.09	229.97	16.67	5.79	4.38	10.86	-	-
TH-23	51.70	82.91	5.95	14.25	3.92	12.95	33.91	7.17
TH-24	65.00	111.36	6.21	13.11	5.66	10.88	29.51	8.36
TH-25	61.70	145.52	5.63	12.40	3.71	11.12	20.85	6.80
TH-26	6.00	65.55	4.94	5.46	5.02	5.34	-	-
TH-27	26.80	94.21	6.94	13.76	4.53	9.55	16.44	7.62
TH-28	100.30	152.56	5.43	12.36	6.60	9.32	-	-
TH-29	62.10	141.95	6.84	13.38	6.47	12.62	22.30	11.30
TH-30	49.70	140.26	2.21	11.42	3.96	11.60	18.57	10.56
TH-31	37.90	85.11	8.32	9.76	8.41	9.17	18.53	8.63
TH-32	23.80	66.10	6.77	9.81	4.86	9.93	24.60	7.30

Appendix 3 Measurements of the SEM-EDX cross-sectioned samples.

SEM-EDX Sample	Distance from Head (mm)	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)
TH-01-1	26.82	12.19	14.09	3.00	1.50
TH-01-2	67.57	5.98	10.77	3.00	1.20
TH-02-1	28.52	6.75	8.55	3.00	0.60
TH-02-2	66.94	7.16	9.51	3.00	0.60
TH-03-1	32.83	5.57	8.33	3.00	1.10
TH-03-2	71.93	11.65	11.77	3.00	1.10
TH-04-1	31.86	11.51	12.31	3.00	1.50
TH-04-2	81.15	7.50	10.37	3.00	1.00
TH-05-1	25.01	8.61	10.31	3.00	0.90
TH-05-2	56.60	9.14	9.35	3.00	0.70
TH-06-1	21.93	7.00	8.93	3.00	0.40
TH-06-2	67.85	7.60	8.85	3.00	0.60
TH-07-1	20.81	12.92	15.49	3.00	3.00
TH-07-2	90.20	13.27	13.33	3.00	1.90
TH-07-3	154.23	9.67	11.87	3.00	1.00
TH-08-1	27.16	16.04	17.04	3.00	1.80
TH-08-2	79.72	12.79	15.21	3.00	2.00
TH-08-3	157.69	10.50	10.54	3.00	0.80
TH-09-1	27.10	15.73	15.98	3.00	2.10
TH-09-2	62.33	14.02	15.01	3.00	2.30
TH-09-3	123.24	13.10	13.59	3.00	1.30
TH-10-1	20.98	14.72	16.22	3.00	2.60
TH-10-2	60.49	12.62	13.87	3.00	1.60
TH-10-3	98.01	10.21	12.51	3.00	0.90
TH-11-1	23.63	12.57	14.10	3.00	1.10
TH-11-2	66.27	11.63	13.02	3.00	0.80
TH-12-1	25.56	10.41	10.77	3.00	1.00
TH-12-2	57.64	8.50	8.61	3.00	0.60
TH-13-1	19.59	10.10	14.98	3.00	1.40
TH-13-2	62.42	6.62	7.17	3.00	0.50
TH-14-1	30.15	14.42	16.65	3.00	1.70
TH-14-2	75.00	11.66	14.82	3.00	1.40
TH-15-1	27.25	11.15	13.47	3.00	1.80
TH-15-2	63.74	12.30	14.13	3.00	2.20
TH-16-1	22.77	11.64	11.71	3.00	1.00
TH-16-2	49.91	10.47	11.58	3.00	0.70
TH-17-1	24.85	13.47	13.90	3.00	1.90
TH-17-2	65.70	10.14	10.52	3.00	1.00
TH-18-1	24.05	10.62	11.17	3.00	1.10

TH-18-2	57.69	10.90	11.39	3.00	1.00
TH-19-1	23.49	10.93	11.27	3.00	1.80
TH-19-2	85.58	11.09	11.34	3.00	1.00
TH-20-1	18.50	10.09	11.50	3.00	1.00
TH-20-2	44.46	9.09	10.54	3.00	0.90
TH-21-1	23.14	13.51	14.48	3.00	2.30
TH-21-2	63.98	10.68	12.02	3.00	1.40
TH-21-3	118.01	9.07	9.55	3.00	1.00
TH-22-1	50.27	12.64	12.81	3.00	1.60
TH-22-2	121.31	13.31	15.64	3.00	2.30
TH-22-3	195.37	11.43	13.28	3.00	1.20
TH-23-1	19.95	14.90	16.26	3.00	2.10
TH-23-2	52.02	10.53	12.06	3.00	1.00
TH-24-1	24.12	13.45	16.03	3.00	2.30
TH-24-2	63.07	10.76	11.70	3.00	1.20
TH-25-1	18.82	13.08	13.80	3.00	1.90
TH-25-2	63.23	10.28	12.16	3.00	1.20
TH-25-3	106.05	10.41	11.51	3.00	0.90
TH-26-1	20.78	9.95	10.65	3.00	0.60
TH-26-2	46.99	6.92	7.56	3.00	0.20
TH-27-1	17.93	15.70	15.80	3.00	2.20
TH-27-2	59.46	12.32	13.09	3.00	1.60
TH-27-3	112.62	9.90	11.79	3.00	1.20
TH-28-1	30.78	14.49	15.66	3.00	2.00
TH-28-2	71.10	13.91	14.51	3.00	2.00
TH-28-3	113.69	13.98	14.05	3.00	2.20
TH-29-1	17.02	14.60	15.12	3.00	2.30
TH-29-2	82.64	8.13	9.29	3.00	0.70
TH-30-1	20.54	13.33	16.99	3.00	1.80
TH-30-2	76.05	10.73	11.67	3.00	0.60
TH-31-1	23.69	12.07	13.01	3.00	1.00
TH-31-2	62.09	11.56	12.85	3.00	1.20
TH-32-1	19.54	12.49	18.45	3.00	1.10
TH-32-2	47.12	8.28	10.60	3.00	0.50

Appendix 4 Photographs of Iron Nails.

Huntingdon Island 5 (FkBg-03), House 1, #147 (TH-10)

Huntingdon Island 5 (FkBg-03), House 1, #123 (TH-03)

Huntingdon Island 5 (FkBg-03), House 1, #118 (TH-16)

Huntingdon Island 5 (FkBg-03), House 1, #184 (TH-11)

Huntingdon Island 5 (FkBg-03), House 2, #318 (TH-22)

Huntingdon Island 5 (FkBg-03), House 2, #1344 (TH-21)

Huntingdon Island 5 (FkBg-03), House 2, #615 (TH-23)

Huntingdon Island 5 (FkBg-03), House 3, #297 (TH-08)

Huntingdon Island 5 (FkBg-03), House 3, #1248 (TH-25)

Huntingdon Island 5 (FkBg-03), House 3, #373 (TH-24)

Huntingdon Island 5 (FkBg-03), House 4, #2948 (TH-07)

Huntingdon Island 5 (FkBg-03), House 4, #2708 (TH-02)

Huntingdon Island 5 (FkBg-03), House 4, #2991 (TH-05)

Huntingdon Island 5 (FkBg-03), Tent Ring 4, #4307 (TH-09)

Pigeon Cove (FlBf-06), House 1, #2292 (TH-01)

Pigeon Cove (FlBf-06), House 1, #1778 (TH-04)

North Island 1 (FeAx-03), #240 (TH-06)

Great Caribou Island 1 (FbAv-13), House A.1 (TH-15)

Great Caribou Island 1 (FbAv-13), House B.1 (TH-27)

Saddle Island (EkBc-01), #23190a-r (TH-17)

Saddle Island (EkBc-01), #23152a-j (TH-18)

Saddle Island (EkBc-01), #25985c (TH-32)

Snack Cove 3 (FkBe-03), House 3, #1228 (TH-12)

Snack Cove 3 (FkBe-03), House 3, #1288 (TH-20)

North Island 1 (FeAx-03), #222 (TH-28)

Great Caribou Island 1 (FbAv-13), House A.2 (TH-14)

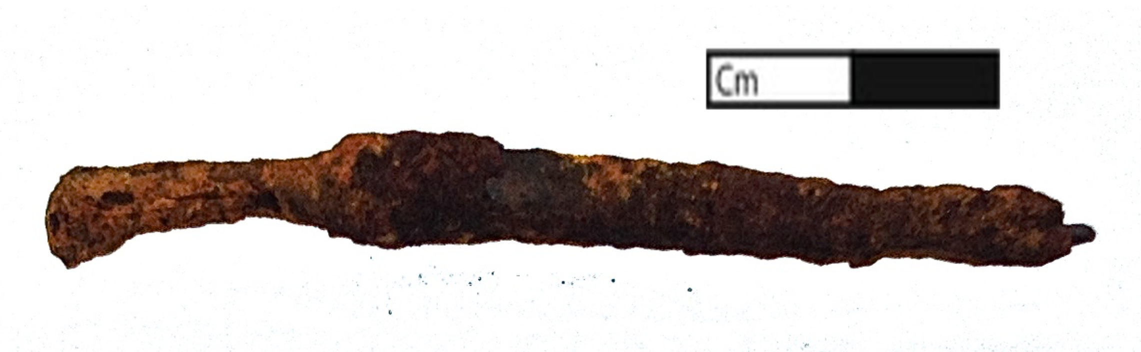
Great Caribou Island 1 (FbAv-13), House B.2 (TH-13)

Saddle Island (EkBc-01), #29508c (TH-31)

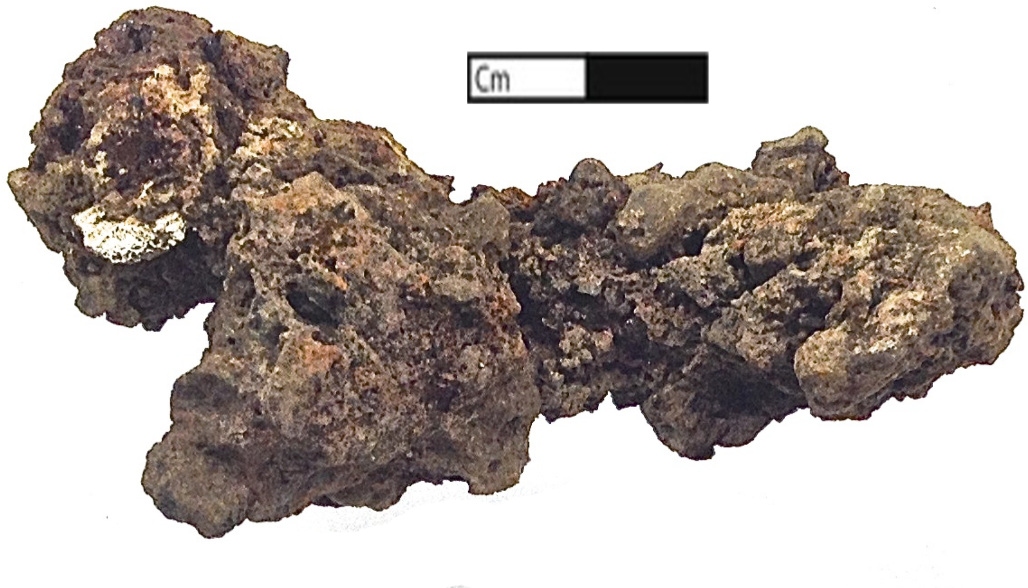
Nachvak (IgCx-03), House 2, #219 (TH-29)

Nachvak (IgCx-03), House 2, #241 (TH-30)

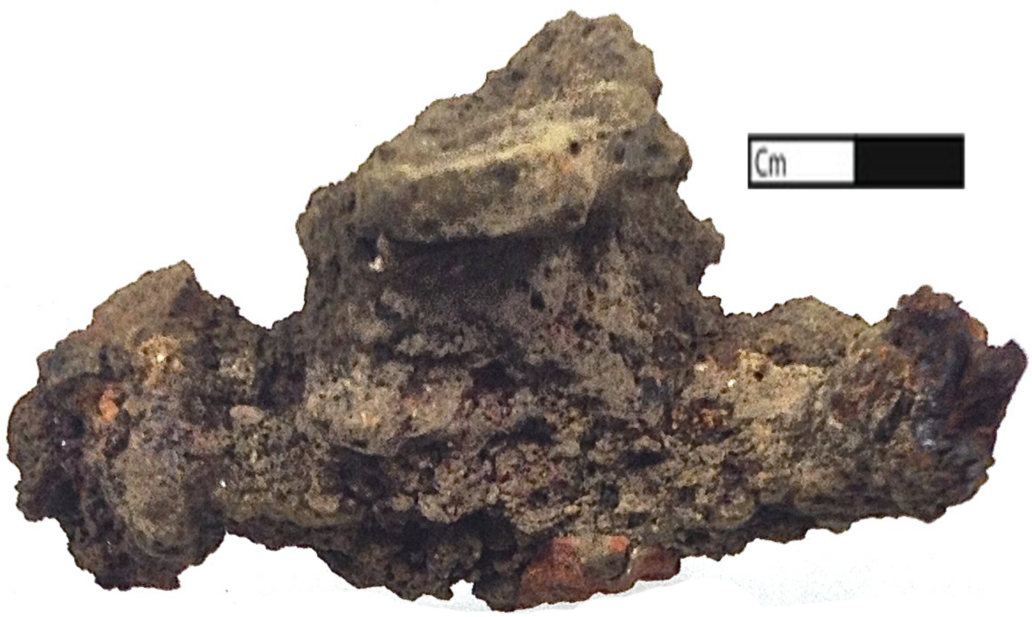
Dos de Cheval (EfAx-09), #11141 (TH-19)



Dos de Cheval (EfAx-09), #11144 (TH-26)



Ferryland (CgAf-02), Event #794



Ferryland (CgAf-02), Event #794