INVESTIGATIONS OF MEDIEVAL SLAG FROM GOTLAND, SWEDEN

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AUTHORIZATION TO SUBMIT THESIS

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Abstract

This thesis examines metallurgy in the late Viking Age and early medieval period of Gotland, Sweden. This is done through the chemical analysis of slag samples, waste material from metal working operations, from three different sites from the island. The three sites studied include two farmstead sites, Little Hultungs and Långume, and a trading port, Fröjel. Chemical analysis includes Inductively Coupled Plasma Mass Spectrometry and Instrumental Neutron Activation Analysis for determining elemental composition of slag, and Thermal Ionization Mass Spectrometry to determine lead isotope ratios. The lead isotope data was inconclusive for determining sources for copper. The elemental composition of slag samples was compared among the three sites and it was determined that two of the sites, Fröjel and Långume, were smelting and working iron, with more successful smelting occurring at Fröjel, and that one of the sites, Little Hultungs, was casting bronze.

Acknowledgements

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Chapter 1: Introduction

Gotland, Sweden is the largest island in the Baltic Sea. It is also a place with a very rich cultural history. Gotland is known to historians and archaeologists today for its prominence during the Viking period, around 750 to 1050 CE. This recognition comes from the fact that vast amounts of Viking age silver hoards have been found on the island. More than half of all the silver hoards found in Sweden have been found on this island, which is very small compared to the rest of Sweden. A great deal of research has been directed toward the phenomenon of silver hoards, revealing that Gotland was an ideal trade location. Its central geographical location in the Baltic Sea made it an ideal stopping point for nearly all Baltic trade. This situation continued into the Middle Ages with Gotland's major city, Visby, becoming a member of the Hanseatic League, a powerful trade organization in the Baltic and surrounding regions.

Most of the archaeological research on Gotland has, therefore, focused upon investigating these silver hoards in particular and Viking and medieval period trade in general. Overall, there has been a great deal of treasure hunting on the island as well as an understandable emphasis on the exciting high-value hoards that have been recovered all across the island. However, more recent work by archaeologists has focused upon daily life on the island through several excavations of Viking and medieval period farmsteads. While trade was important for the island, Gotland was mostly settled and occupied by single families. As such, the farmsteads that dot the island today and those that archaeologists have been exploring are more representative of the realities of daily life on the island than the silver hoards that have attracted so much attention. The research trend to focus on local daily life is being led by Dr. Dan Carlsson from the

University of Gotland. Carlsson, who leads the Viking Discovery Program on Gotland, has excavated several farmsteads from 2007 to 2010. During the summer of 2009, I traveled to Gotland with my wife to participate in the Viking Discovery Program. That summer we excavated part of a farmstead known as Little Hultungs. Little Hultungs is a farmstead located in Northern Gotland dating from 1150 to 1400 CE, a site that will be discussed in greater detail later in this thesis.

Continuing the trend of examining daily life on Gotland, this thesis is focused upon understanding the metallurgical practices taking place on Gotland during the late Viking and medieval periods, from about 900 to 1400 CE. My study of Gotland’s metallurgical processes is based on the analysis of slag samples from three archaeological sites. Slag comes in two varieties. The first form is as a waste product produced from extracting metal from ores, known as smelting slag. The second is from spillage while casting metal into moulds, which is known as crucible slag. This study is primarily based upon smelting slag.

Although mundane sounding, the investigation of historical metallurgy is potentially very important to understanding past human lifeways. It is important for the simple reason that the invention of metallurgy was an extremely important technological development in human history. Metallurgy first developed around 5000 BCE in Bulgaria long before the time period I am working in. The history of metallurgy is characterized by many technological changes over time as well as considerable variation in implementation from region to region (Bork et al. 2005:1, Anthony 2007:162). Metallurgy has many uses but a key one for humankind is the fact that it has greatly expanded the tool-making resources available to humans. Metal tools often have many advantages over wood and stone tools, such as durability and malleability. During the Viking and Medieval periods, metal provided many necessary objects for their way of life, such as nails, knives, horseshoes, and other everyday objects (Bork et al. 2005:2-3). Manufacturing these metal objects became an integral part of life during these periods.

As metal objects became parts of everyday life they also become objects (and processes) that are of interest to archaeologists. Topics examined in this thesis are metallurgical trade and metallurgical techniques. Slag samples from three different sites on Gotland have been compared to examine similarities and differences in technologies as well as to explore what metallurgy may reveal about trade practices. The sites used in this study are Little Hultungs and Långume, which are farmstead sites, and Fröjel, which was a trading center. The slag samples were analyzed to determine the elemental composition of the slag and to attempt to source copper slag. Detailed discussion of these techniques used can be found in chapter four.

This thesis incorporates techniques from chemistry to answer anthropological questions about human behavior in the past. The project also demonstrates some of what can be done with international cooperation. A great deal of archaeometallurgical studies across the world have been assessed, including some parts of Scandinavia, but Gotland has had only preliminary work done in this field of research. The research will provide a starting point for future archaeometallurgy research in Gotland, and expands the work being done specifically in Scandinavia. Development of metallurgy marked a significant transition in human technology and has seen many changes throughout history. The time period this project examined (800-1400 CE) was one that was reliant upon metal technology for many objects; including metal tipped plows, nails and rivets for building houses, metal decorative jewelry, weapons, and other tools.

The intellectual contributions of this project are varied. First, this research expands our understanding of everyday Viking age and medieval life. This type of work, using multidisciplinary approaches to understand a process such as metallurgy, has not yet been done in Gotland. Archaeometallurgy has seen only limited application in Scandinavia, and while this project is limited in scope, it provides a basis for future research by other scholars. My work focuses upon three specific sites on Gotland, but it provides data that others will be able to be used for comparison and as the basis for future study, both in Gotland and the region. This research will provide examples of what types of analysis are possible using different analytical tools to understand metallurgy in Gotland over 1000 years ago. This thesis also provides insights into difficulties presented by metallurgical analysis and offers suggestions for future avenues of archaeometalurgical scholarship.

Chapter 2: History of Gotland

The island of Gotland is the largest island in the Baltic Sea, around 3,140 square kilometers. Gotland is located about 90 kilometers from mainland Sweden, 150 kilometers from Latvia, and 220 kilometers from Poland (Figure 2.1). Gotland is primarily made up of limestone. Evidence suggests that the island was first inhabited about 8,000 to 7,000 years ago (Spencer 1974:39, Saers n.d.).

Figure 2.1: Map of the Baltic

The *Guta Saga* offers a traditional history of Gotland based on oral tradition and written in the thirteenth century (Peel 1999). The text states that the island was first settled by a man named Tjelvar (Spencer 1974:39, Peel 1999:3). Tjelvar is said to have come to the island that sank by day. When he brought fire to the island it stopped sinking. There is some geological evidence to support this myth as the water levels fluctuated around the island of Gotland since the end of the last Ice Age until about 8,000 years ago (Spencer 1974:19-21). The *Guta Saga* then tells of Tjelvar's three grandsons and how the island was divided among the three, with one given the northern part, one given the middle part, and the third given the southern part; this three part division is reflected in the modern political organization of the island.

Archaeological evidence shows that the early Gotlanders were seal hunters and strung together seal teeth into necklaces (Spencer 1974:39-40). During the early period of settlement, around 5000 BCE, marine life was likely the primary resource utilized, but there is also some evidence of utilizing terrestrial fauna such as pigs (Saers n.d.). There is also archaeological evidence that even in Gotland's early history it was a center of trade. An excavation at Västerbjärs in eastern Gotland during the 1930s showed that early people traded for beautiful stone axeheads from across the Baltic, primarily from southern Scandinavia (Spencer 1974:40, Saers n.d.).

Trade continued to be an important feature for Gotland as it entered the Bronze Age (1500 to 500 BCE). Gotlanders served as middlemen between Denmark, Southern Sweden and Indo-Germanic peoples to the south and east (Spencer 1974:40). The primary trade goods during the Bronze Age in Gotland were Baltic amber and bronze.

Bronze Age Gotland is famous for the large ship-graves constructed on the island. Ship graves were a specific form of monumental burial. These graves could extend for more than 40 meters and could be up to seven meters wide with stones arranged in the shape of a ship with "high prows, raking lines and solid sterns" (Spencer 1974:41). One of the most famous ship graves on the island has been named Tjelvar's Grave, although this would certainly not be the grave of the first person on the island as

Figure 2.2: Photograph of Ship Setting Grave

people lived on the island for roughly 3,000 years before this grave was constructed. Figure 2.2 shows an example of a ship grave. The largest ship-graves contain multiple individual cremations, possibly serving as family burial places (Carlsson Personal Communication, Sprague 2005:14-17).

During the Bronze Age, farming became more prominent in Swedish society. On the mainland there is evidence of the use of domesticated horses and other farm animals (Sprague 2005:9). Evidence of horse domestication can be found on the island (Saers n.d.). This indicates that domesticated animals were being introduced onto Gotland during this time period (1500-500 BCE). To date there are no traces of permanent buildings from the Bronze Age.

The *Guta Saga* also discusses an overpopulation of the island that forced a mass emigration, an event that may have occurred during the Bronze Age or the early Iron Age (Peel 1999:xxv-xxvii). This episode is described in detail beginning with casting lots to determine who had to leave the island. The *Guta Saga* details that the people first took refuge at a fortress known as Torsburgen, the largest hill-fort found in modern Sweden (Peel 1999:3). Torsburgen is thought to have been large enough to shelter the entire population of Gotland within its defensive fortifications (Spencer 1974:49, Peel 1999:xxvii-xxviii). This fort was situated on a high point on Gotland where natural defensive cliffs served to provide about half of the total outer barrier. The story goes on to state that the people were forced to leave Torsburgen and made their way to Farö, a small island just north of Gotland, and from Farö to the island of Dagö off of the coast of Estonia (Peel 1999:3-5). The travelers could not support themselves in either Farö or Dagö, so from there they moved south into the Byzantine Empire. According to the *Guta Saga* the settlers were able to trick the Emperor into agreeing to let them settle there permanently by getting him to agree to let them stay for “the waxing and waning” (Peel 1999:5). The emperor thought that this statement meant that they would be able to stay for a month, one waxing and waning of the moon, but the Gotlanders pointed out that the moon continues to wax and wane forever.

The Iron Age, beginning about 500 BCE and continuing until the Viking Age about 800 CE, saw not only a shift in new materials but also a dramatic shift of climate. Northern Europe became dramatically cooler around the beginning of this period. There is evidence that iron smelting was happening on Gotland as early as 200 BCE, earlier than mainland Sweden (Spencer 1974:44). The evidence of early smelting may suggest that, due to their trade networks, Gotland received new ideas before mainland Sweden.

The culture on Gotland started to see a dramatic shift during this time. The Iron Age saw the first evidence of permanent buildings on Gotland (Spencer 1974:47). Evidence of these structures includes post holes and stone foundations. The structures are typically rectangular in shape with rounded corners (Spencer 1974:47). Most often these structures were located in small numbers near cultivated fields, indicating a family farmstead, but sometimes larger clusters were identified. These larger clusters may indicate seasonal fishing villages or trading centers, as they are found along the coast.

One example of a larger cluster is at Vallhagar near Fröjel in western Gotland where 24 houses were found (Spencer 1974:47). There is evidence of many houses being subject to violent destruction, such as by fire from 450-500 CE (Spencer 1974:47).

Direct evidence of farming is apparent in the Little Hultungs excavations where archaeologists have identified the remains of ard lines (Figure 2.3). Ard lines appear as alternating dark and light areas of soil where the soil was pressed down with an ard, a tool similar to a plow, but that was used just to break the soil in a single line for planting. Carlsson theorizes that most of the farmsteads on Gotland were first established during this period and were then consistently inhabited into the historical period (Carlsson 2009).

Figure 2.3: Photograph of Ard Lines

Another trend during the later part of the Iron Age, beginning around 400 CE, was the construction of picture stones (Spencer 1974:46, Öhrman 2001:1). Picture stones are similar to rune stones found in other parts of Scandinavia but are unique to Gotland. Picture stones are large stones with carved surfaces depicting a variety of scenes often erected as memorials. “Early picture-stones have simple but elegant geometrical patterns, and mystic symbols such as the ‘Sun-Wheel’ or ‘Wheel of Life’” (Spencer 1974:46) while later stones often depict scenes from typical Norse mythology or normal life (Carlsson 2009, Spencer 1974:46-47). Rune stones found in other parts of

Scandinavia typically have simple patterns and many are just decorated with runes. Text in the form of runes on picture stones is usually some sort of dedication. Carlsson believes that most are memorials erected to honor people who have died while away from the island (Carlsson 2009). Picture stones, while first appearing in the late Iron Age, remained prominent throughout the Viking Age (750-1100 CE) and started to disappear during the early Medieval Age (Öhrman 2001:1). Figure 2.4 shows a famous example of a picture stone that can be found at the Bunge Museum in northern Gotland.

Figure 2.4: Photograph of Picture Stone

The Viking Age, around 750 CE to 1100 CE, is perhaps the most famous period of Scandinavian history. During this age, Gotland continued its tradition as a prime trading location (Montelius 1969:148; Spencer 1974:51, 61-63). Gotland served as a point where eastern goods from Byzantium and the Middle East met with western goods from Scandinavia and Britain (Spencer 1974:51, 61-63; Clarke 1985). Glass, silk and spices from the East were traded for honey, wax and furs from the West. Around 800 CE Gotland agreed to come under Swedish rule in return for protection and exemption from tolls (Spencer 1974:50, Peel 1999:7). The *Guta Saga* relates the tale that Gotland had been under assault by various kings, and many Gotlanders tried to negotiate some sort of peace accord (Peel 1999:7). Eventually Avair

Strabain of Alva parish "entered into a binding treaty with the king of the Swedes. Sixty marks of silver in respect of each year is the Gotlanders' tax, divided so the king of Sweden should have forty marks of silver out of the sixty, and the jarl twenty marks of silver" (Peel 1999:7). A jarl is a nobleman of high rank. This protection helped ensure the continuation of their trade networks and also exempted the Gotlanders from any tolls, or other charges in Sweden, enabling them to expand their operations in Sweden.

Gotlandic trading contacts ranged far and wide. Figure 2.5 shows a map of various Viking trade routes. The routes in green, to the east of Sweden, are the typical routes of Swedish Vikings, such as the Gotlanders.

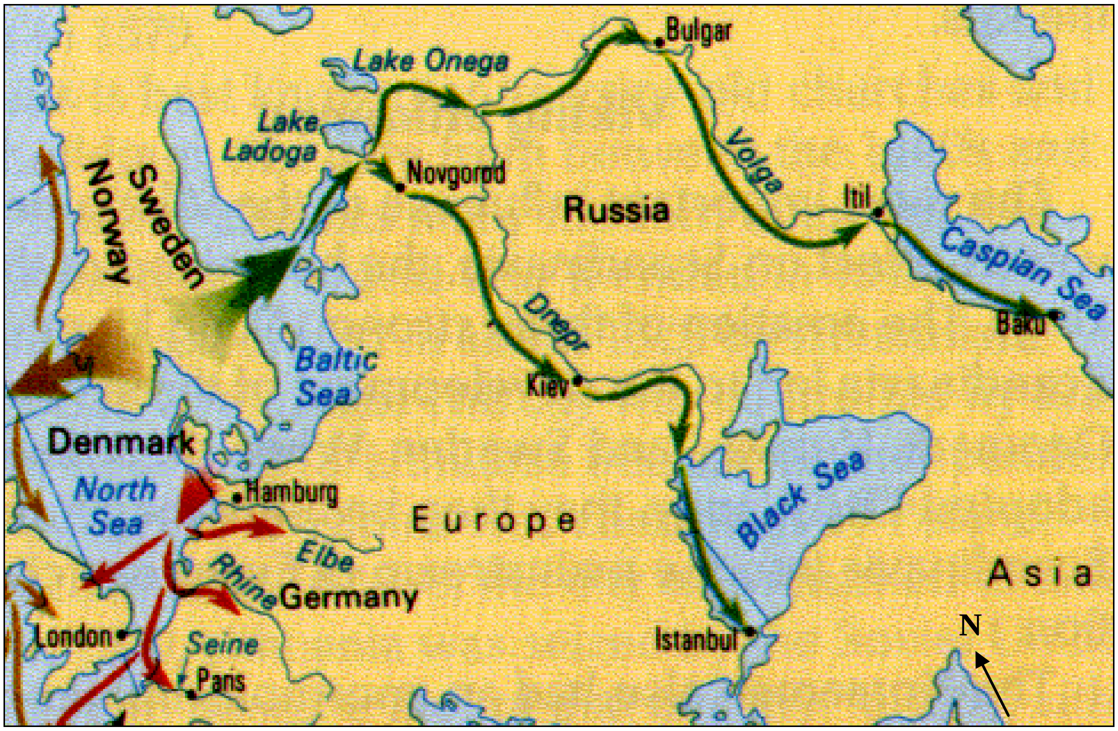

Figure 2.5: Map of Viking Trade Routes

Viking traders were able to penetrate very far east and south through their ability to move their boats across land from river to river, enabling them to navigate to the Black and Caspian Seas (Clements 2005:99-105). Moving the boats was accomplished by pulling them completely out of the water and carrying them or rolling them on logs; this method was possible due to the boats' very shallow keels. These travels facilitated trade contacts with Constantinople, the capital of the Byzantine Empire, and Itil, a Muslim trade city located where the Volga river runs into the Caspian Sea (Clements 2005: 104, 106).

As a result of extensive trading Gotland saw an influx of Arabic silver during this time period. More than half of all silver finds from Sweden are Gotlandic silver finds including over 140,000 silver coins; these coins generally date to around 900 AD and can be identified as Islamic, German or English (Sprague 2005:34). Silver coins are only one example of the numerous goods that were obtained in trade. "Persian glass, Chinese silk, narrow-necked bronze bottles from east of the Caspian, exotic purses from India, spices and wines, all these found their way to Novgorod, Gotland, … in exchange for slaves, weapons, honey and wax, and an odorous plenitude of furs" (Jones 1984:253). The Vikings were especially able to help meet the Arab demand for slaves, as the Christians were prohibited from selling slaves to "heathen" customers (Spencer 1974:51).

During the expansion of trade networks to the south and east, Gotlanders and Swedes established lasting communities along their trade routes. Evidence from cemeteries in these communities suggests that the mainland Swedes were a strong military presence, while Gotlanders were primarily civilians (Jones 1984:243). Many Gotlanders even brought their wives to these settlements. The evidence of a civilian lifestyle suggests that the Gotlanders were less interested in piracy or war, but instead were interested in more peaceful trade (Jones 1984:243). The two most important communities were established in Novgorod and Kiev. These became destination points for trade and for stopping along the long route to Constantinople or Itil (Clements 2005:102-103). These places also became permanent settlements of Gotlanders and

Swedes whom the locals referred to as the Rus or Rootsi (Clements 2005:98-99). Gotland was prominent in this arrangement as it provided an ideal place for ships from these trading points to stop on their way across the Baltic to other parts of Scandinavia.

The Viking Age is certainly most famous for the raids that came out of the various northlands. Gotland almost certainly participated in some raiding, but likely emphasized trade first. The fame of Viking raiding often comes from sources of those who were raided, mainly Christian monks. While raiding certainly occurred all across Europe and into the near East, many of the stories may have been exaggerated by authors. The Vikings often settled in lands that they had started out raiding. This increased trade contacts throughout the Viking world. Some evidence of raiding that may be associated with the Gotlanders includes the Viking attacks on Constantinople (Clements 2005:104-115).

While there was considerable movement of people through raids and trade journeys in the Viking era, Gotland continued to be dominated by single family farmsteads with few larger settlements (Spencer 1974:59, 62; Siltberg 1998:68-69). Two larger settlements during the Viking Age were Visby, currently the capital of Gotland, and Ridanäs which was located near modern Fröjel, a trading port south of Visby (Figure 2.6) (Spencer 1974:12-13, Carlsson 2004). There were other smaller towns and ports on Gotland, but most of the island was dominated by

Figure 2.6: Map of Gotland

farming (Siltberg 1998, Carlsson 2004). Socially, Gotland was divided among a dominant farmer class, a landless working class, and small groups of tenants (Stiltberg 1998:71). The members of the farming class were the effective leaders of the region. They were politically active, involved in administration of justice, guarded harbors, and performed other political tasks through their Gutensten (a council of representatives from all across the island) (Stiltberg 1998:68). In comparison, the other two groups were largely unrepresented in the island's administration. The dominant land owning farming class was also engaged in the trade activity that was done on the island.

Everyday life in Viking Age Gotland shares some similarities with everyday life of mainland Sweden during this era. While many Vikings were primarily traders, they also raised cattle and farmed between voyages (Sprague 2005:41). However, Gotland and the mainland had some significant differences in the distribution of wealth during this time period. "[Mainland Swedish] Society consisted of an upper class of rich and free farmers who owned ships and large areas of land, a middle class of regular farmers who owned smaller pieces of land and cattle, and slaves who owned nothing and worked long hours for others in return for room and board" (Sprague 2005:43). Slavery in Sweden during the Viking era was usually more like indentured servitude, but in some cases it was the result of people captured while raiding who were truly treated as slaves and not given the opportunity of eventual freedom. All male members of both the upper and middle class farmers were able to speak during political councils and participated in electing leaders, including the Swedish king in Uppsala (Sprague 2005:25-26,41).

Rune stones and archaeological evidence suggests that women were considered to have similar status to men during the Viking age (Sprague 2005:43). While men and women had different duties, these could be overlapped, and were equally valued. Women were in charge of a family's finances and household, as they were the ones that carried the keys to the home and strongbox. There have also been indications that women were engaged in trade as can be seen with finding merchant's scales in female graves (Sprague 2005:44). Women often had to take on extra responsibilities when men were off on voyages of trade or warfare. Archaeological evidence in the form of grave goods suggests that with age women tended to gain status, while men tended to lose status (Sprague 2005:44). This may be due to that idea that a man's status was grounded in physical strength, while a woman's status was grounded in experience and wisdom.

The transition into the Middle Ages in Gotland can be marked around 1050 CE. The single most influential change between the Viking Age and the medieval period is the adoption of Christianity. On Gotland this can be seen by the construction of churches and a transition toward using Christian iconography on picture stones. Like many major cultural changes this occurred gradually across the island. The *Guta Saga* discusses that some Gotlandic merchants decided to become baptized and brought priests back to the island (Peel 1999:9). Not all of the Gotlanders welcomed these new ideas. The *Guta Saga* provides the account of the first church built on Gotland:

> *Botair of Akebäck was the name of the one who first built a church, in that place which is now called Kulstäde. The islanders were not prepared to tolerate that, but burned it… Then later on, there was a sacrifice at Vi. There Botair built a second church. The people of the island also wanted to burn this particular church. Then Botair went up on top of the church himself and said, 'If you want to burn it, you will have to burn me along with this church.' He was himself influential, he had as his wife a daughter of the most powerful man, called Likkair Snielli … Likkair … supported Botair … As a result, that church was allowed to stand unburnt.* (Peel 1999:9).

The *Guta Saga* goes on to relate that after a few churches were built in various parts of the island and the Gotlanders saw Christian ways, Christianity was adopted by the Gotlanders of their own free will without duress (Peel 1999:11). After Christianity became accepted many churches were built across the island (Figure 2.7). Of the 94 churches in use on Gotland in 1974, 93 were finished before the end of the fourteenth century (Spencer 1974:123). There were other churches built during the Middle Ages that are not still in use, including 15 churches in the city of Visby.

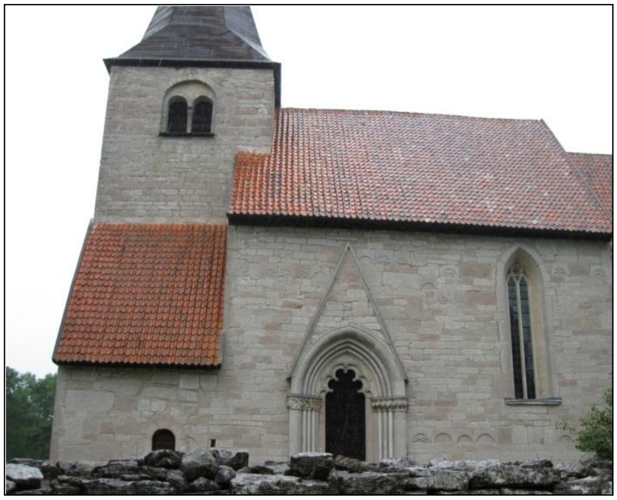

Figure 2.7: Photograph of Bro Church, Gotland

The transition into the medieval period was more than just religious. There was also a marked change in the structure of society. There was an increased centralization of power, including a hierarchal class structure and regulated trade. With Christianity came a more rigid division of classes that included hereditary nobility that further separated the rich farmers from the poorer farmers on mainland Sweden (Sprague 2005:65-66). Gotland still maintained their traditional social classes and relative independence during the early part of the medieval era (Spencer 1974:64).

Although trade prospered during the early part of this period, there was growing competition from Germany. In 1161, the Gotlanders were forced into a trade agreement with Henry the Lion, Duke of Saxony, and the city of Lübeck (Spencer 1974:64). The rise of cities, such as Visby, also brought the rise of a merchant class, usually at the expense of the free farmers (Sprague 2005:66). Visby became an influential international city during this time period. During the early medieval period Visby was comparable with London and Paris as one of the great cities of Europe in terms of wealth and importance (Spencer 1974:61).

While Visby was flourishing, the rest of the island was also doing very well. Several medieval farmhouses still stand on the island indicate a well-to-do farming class (Spencer 1974:62). With the trade agreement in place between Visby and Lübeck, there were increasing numbers of foreign merchants working in Visby. The foreign merchants operated with efficient organizations in contrast to the farmer-traders of Gotland (Spencer 1964:65). Visby began to monopolize trade on the island during this period, leading to the abandonment of other trading centers on the island such as Ridanäs (Spencer 1974:65). This created a great deal of tension between Visby and the other inhabitants of the island. In 1288 CE, Visby built an outer wall around the city enabling it to charge a toll on anyone entering the city with trade goods from the countryside, sparking a civil war (Spencer 1974:65). The rural residents were defeated, but civil war brought Gotland back to the attention of the Swedish King Magnus Ladulås (Spencer 1974:65). The King was reminded of his rights on Gotland and began asserting them through raising taxes. This marked the beginning of Gotland’s decline in international affairs (Spencer 1974:65). While Visby remained prosperous, it became more of a secondary trade center rather than the center of Northern Europe it had once been (Spencer 1974:65). The countryside however saw the start of a more dramatic decline in wealth as increased taxes and reduced trade had a noticeable impact on the island.

Nearly one hundred years later, the final decline for Gotland struck. In 1361, the Danish King, Vlademar Atterdag, invaded the island (Spencer 1974:66). This was part of a larger struggle between Denmark and Sweden. During the conquest of the island the farmers raised two forces that engaged the Danes. Both of these engagements led to horrific defeats for the farmers, resulting in the loss of about half of the male population outside Visby (Spencer 1974:66-67, Carlsson 2009). The city of Visby surrendered to the Danes without fighting them at all (Spencer 1974:67).

Site Histories

Figure 2.8: Map of Gotland, showing excavation sties

Data was used from three excavated sites for this thesis (Figure 2.8). The habitation period of all three sites overlap with time ranges from the Viking age into the late medieval period. Two of the sites (Little Hultungs, and Långume) were single family farmsteads, while the third site (Fröjel) was a trading port. These sites were chosen to examine possible differences in manufacturing techniques and materials between different types of sites, as well as availability to materials from these sites.

Little Hultungs

Little Hultungs is located on the northern end of Gotland, in Bunge parish. The site was excavated in the summer of 2009. The excavation was led by Dr. Dan Carlsson of Gotland University. This site was located by examining a map from the late seventeenth century that had an old name in the area, which translates into Little

Hultungs or Hultungs Down Under. There was no obvious physical trace of this place on the contemporary landscape. Even when that map was first made; the label appears to have been given just from tradition. Initial archaeological testing at the site included phosphate mapping and metal detecting to determine site boundaries as well as where to excavate. Phosphate mapping indicated human settlement in the area, and metal detecting turned up a number of non-ferrous artifacts (ferrous objects were ignored during the survey, due to the abundance of possible modern objects), including some Viking age objects. Figure 2.9 shows the phosphate mapping done at Little Hultungs. The large boxes with color bands show phosphate concentrations, with dark red indicating the highest phosphate concentrations.

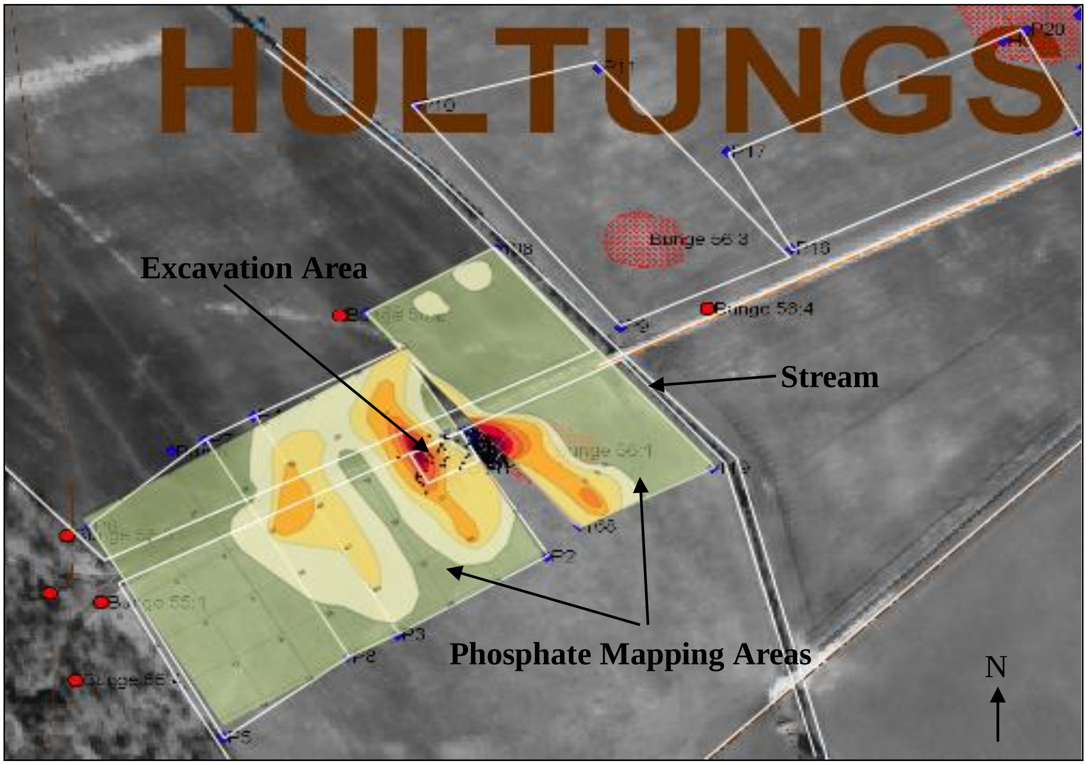

Figure 2.9 Phosphate Map from Little Hultungs

The site at Little Hultungs is situated in fields that have been primarily used for grazing sheep, but have been plowed for crops on a few occasions. The presence of a plow zone in the area does mean that objects may have been moved about within the area.

The habitation at the site was dated through two methods. The first was based upon the six silver coins that were found at the site. The identifiable coins were minted on Gotland, something that started in the year 1150 CE. The second method was based upon the ceramic types and styles recovered during excavations. These were matched to existing time lines to give an approximate date of occupation from around 1100 CE to sometime earlier than 1400 CE.

The first major feature found at the site was a house foundation somewhat broken up by modern plowing. The most intact portion of the foundation was where the fireplace had been located. The foundation being disturbed led to a few questions about its structure. Medieval houses on Gotland were two room structures consisting of a smaller entry way and a larger living space. In general in medieval houses on Gotland, and specifically at Little Hultungs, the fireplace is found inside the larger living space.

Figure 2.10 is a diagram of the excavation at Little Hultungs. The proposed house outlines are marked in red lines. Various features from the site are also shown, including ard traces, modern plow marks, and the fireplace. The outline of the house was determined based upon the part of the foundation that was still intact as well as the identification of a post hole in the south eastern corner of the structure. With only a limited amount of the foundation identified, other options were considered to more accurately determine the location of the interior wall. It was determined that the interior wall was on the east side of the hearth feature, placing the entrance on the eastern end of the structure. This determination was made due to artifact distributions at the site.

Distributions of bone fragments and ceramics show much higher concentrations in the western portion of the house.

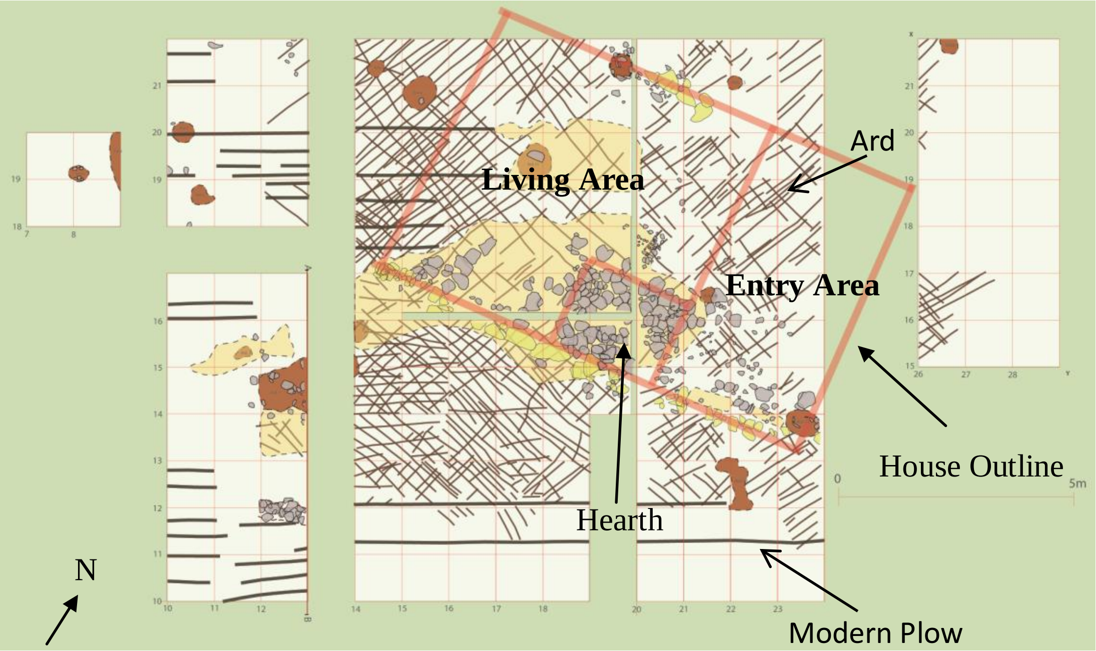

Figure 2.10: Excavation Map of Little Hultungs

Figure 2.11 is a photograph of a different medieval house foundation excavated at Fjäle on Gotland by Dr. Carlsson. This photograph is included as an example of a typical medieval house in Gotland.

Figure 2.11: Photograph from Fjäle of a Medieval House

The second major feature identified at Little Hultungs was traces of an Iron Age ard. The ard traces were located mostly under the house foundation and extended south of the house. These types of traces have been found at sites throughout Scandinavia. The traces were likely preserved under the house due to the protection it offered from modern plowing. The ard lines indicate that the area was used as a cultivated field sometime from 100 to 500 CE.

Fröjel

Fröjel is located on the west coast of Gotland, south of Visby. It is also the former location of the Viking trade settlement of Ridanäs. During the Viking Age, Ridanäs was one of the largest and most important ports on Gotland (Carlsson 2004:6). Excavations revealed that the port was in use around the late 6th century and was abandoned around 1180 CE, a period when the site was a port, trading center, and manufacturing center (Carlsson 2004:6). The site was likely abandoned due to a drop in the sea level making the strait too shallow for ships, and from increased competition from Visby (Carlsson 2004:16). There was very little development or exploitation of the area following its abandonment, resulting in excellent the preservation of the Viking era settlement.

The discovery of Fröjel by archaeologists is attributable to a combination of events. First, the presence of a still-standing parish church as well as the remains of a defensive tower was an immediate indicator of long-term occupation of the area. Second, local residents had reported numerous isolated finds in the region over many years. Third, its physical location was fairly well protected from the winds and storm swells, making it a logical place for people to have settled over a thousand years ago. (Carlsson 2004:7). These factors led to archaeologists testing the area by phosphate mapping. The results indicated a large area of very high phosphate content near the modern shore, suggesting sustained human activities (Carlsson 2004:7).

The site at Fröjel occupies around 10ha, with several cemeteries and traces of many buildings. There is a regular pattern to the settlement with rows of houses identified by postholes and occasionally the remains of wood fences (Carlsson 2004:7). By 2004, 1,200 square meters of the site had been excavated in a series of trenches covering most of the harbor area and 35,000 objects had been collected (Carlsson 2004:9). In addition to the artifacts recovered there was clear evidence of manufacturing including large quantities of animal bones, burnt clay, slag, flint and charcoal (Carlsson 2004:9).

The large number and varied qualities of the artifacts recovered here shed light on the various activities that occurred here. Evidence of trade can be seen through walrus ivory from the north Atlantic, semi-precious stones from the Arabian peninsula and the area around the Black Sea, imported raw glass material from Italy and coins from the Caliphate, Germany, England, and Denmark (Carlsson 2004:9-10).

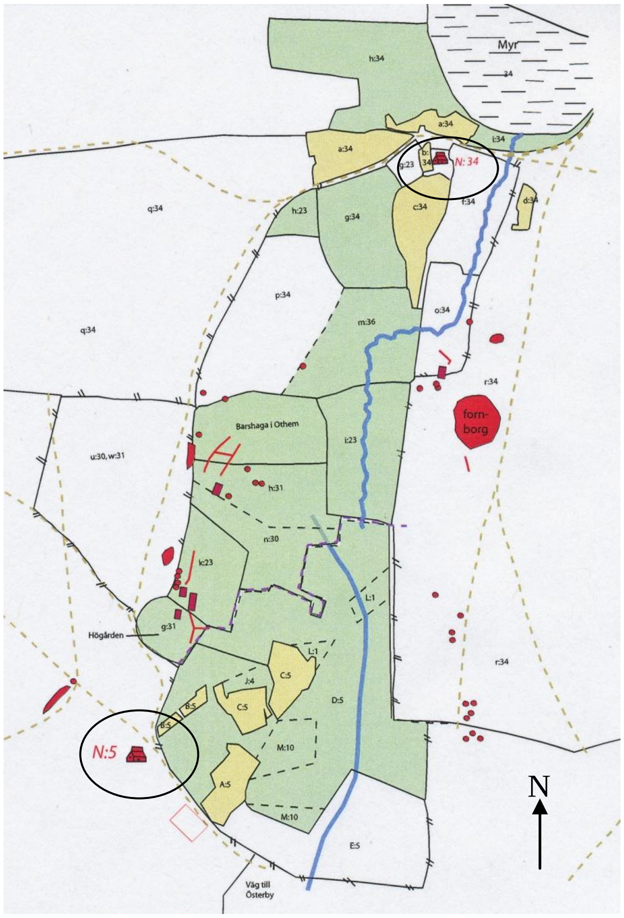

Figure 2.12: Map of Långume

Långume

Långume is located in the northern part of Othem parish in northeastern Gotland. This farmstead is a rare example of one that has never been divided into parcels or reconfigured. The area was first documented in 1696 (Carlsson 2007:2). Figure 2.12 shows the farmstead (marked N:5) of Långume circa 1696, along with the farmstead of Gisslause (marked N:34) in the southern end of Larbro parish, also shown are three iron age farmsteads marked in red and an iron age fort (marked forn-borg). Some bone, charcoal, a slate whetstone, and an unfinished balance weight were uncovered at Långume in 1949. These finds indicated the possibility of a dwelling from the Viking period (Carlsson 2007:5).

In the spring of 2007, phosphate mapping was done on the fields north of the modern farmstead, focusing on dark areas of soil and indications from the 1696 map. As expected, high phosphate values were identified within and near the areas of dark soil (Carlsson 2007:5). Metal detecting was then done with the goal of examining the hypothesis that a Viking age dwelling was located here. Finds included parts of bronze pots (probably from the later middle ages), a coin from the 1600's, and a tool ring for a belt (probably from the later Viking period) (Carlsson 2007:6-7). The findings suggest that the dwelling area in the fields north of the modern Långume farmstead dates from the late Viking period to the early Middle Ages (Carlsson 2007:7).

Chapter 3: Smelting and Metallurgy

Introduction to Metallurgy

Metals are very abundant in nature as more than three fourths of all elements are metals. Most of these are not found in a “pure” state and are combined with other elements and do not have metallic qualities that we are familiar with. Metal containing minerals, including iron oxide and copper ores, were first utilized as pigments. The primary metals that occur naturally in a relatively pure state include: gold, silver, copper, and iron (from meteorites). Platinum is also naturally occurring, but was not recognized as a unique metal until the sixteenth century. Silver, iron, and mercury are fairly rare, thus the first metals widely used by humans were gold and copper. Both gold and copper were found in the form of nuggets that could be worked into other shapes and objects. Gold is quite soft and easy to shape, while copper required more effort and possibly heating to make it more pliable. Gold is too soft to form useful tools and was therefore used primarily for ornamental purposes. Copper, while much softer than iron or steel, is strong enough to make functional tools, but was generally used for decorative objects. Small copper objects, such as beads, pins, and awls, are seen as early as 9000-7000 BCE in Anatolia (Tylecote 1976:1).

Smelting ores to extract metals and create alloys dramatically increased the uses of metals. Ores are minerals with a high metal content that do not exhibit metal traits and tend to be brittle. While there are a variety of contemporary methods for extracting metals from ores, the earliest common method of metal extraction was smelting. This is done by using heat and some sort of chemical reducing agent (often charcoal during the early development of smelting) along with the ore (Tylecote 1976:5). The process results in the metal and a waste product known as slag, made up of the mostly non-metallic parts of the ore. Humans have been smelting metal since about 5000 BCE, and it remains a common method of metal extraction (Anthony 2007:163).

Adding different materials into metals one can make stronger and harder metals. These are called alloys. Alloys are formed by including a second metal or element in small amounts with the primary metal. The first alloys used were bronzes. The earliest bronzes were created by 3700-3500 BCE. These were arsenical bronzes which were created by including arsenic with copper during the smelting process (Anthony 2007:124-125). Smelted coppers usually contain around one percent arsenic, but arsenic ores occur naturally with copper ores. Including an increased amount of arsenic produced a metal that is lighter in color, harder when cool and less viscous when molten, making it much easier to cast (Anthony 2007:124). Eventually it was discovered that using tin in similar amounts instead of arsenic produced an even better bronze that was harder and more easily worked. Tin ores, however, do not occur near copper ores and until reliable sources of tin were discovered tin bronzes remained less common. Other alloys discovered and used later include: brass made from copper with zinc added, and steel made from iron with carbon added. Alloys tend to have much higher tensile strength and shear strength.

Brief History of Metallurgy

Metals and metal working are very important in contemporary society, and they have been increasingly significant to humans over the past several thousand years. The development of metallurgy has had a dramatic impact on human technology. Metallurgy is the branch of science concerned with the properties of metals. Metal working is the process of utilizing metals for creating various objects. This process has greatly expanded the tool making resources available to people. Metal tools often have advantages over wood and stone tools, such as durability and malleability.

Some of the earliest uses of metals included iron oxide for pigments and copper minerals for coloring ceramics. Other early uses of metals began with the discovery of native copper and meteoric iron. These metals occur naturally in a nearly pure state that can be worked without the need for smelting (Tylecote 1976:1-4). Early metal work was done generally using relatively pure metal nuggets and pounding them into the desired shape. To make the metal easier to shape it may be heated or annealed, which makes it more malleable. Naturally occurring metals often have crystalline impurities making such metals brittle (Tylecote 1976:1-2). Most early metal objects are decorative such as beads, but some tools were also produced including awls and knives. An example of a knife from North Greenland has small flakes of meteoric iron set into walrus ivory (Tylecote 1976:2).

Humans began smelting copper around 5200-5000 BCE in Bulgaria and expanded into the Pontic-Caspian steppe societies around 4600 BCE (Anthony 2007:162). Pontic-Caspian steppe societies had developed pottery kilns that reached temperatures ranging from 800 to 1,000 degrees Celsius. The kilns were already low-oxygen, reducing environments used to produce black ceramics, but lent themselves to smelting copper from azurite and malachite, two different copper ores (Anthony 2007:163). Around 4800-4600 BCE Balkan smiths learned to make molds that would withstand the heat of molten copper enabling them to cast copper to create tools (Anthony 2007:163). This requires heating the copper to 1,083 degrees Celsius to liquefy the metal. Copper smelting techniques eventually reached Scandinavia around 1500 BCE (Lossin 2005:2).

The first improvement to copper smelting technology was the inclusion of arsenic and tin with the copper (Tylecote 1976:14). Arsenic frequently occurs with copper deposits and this innovation was likely discovered by accident. To retain the arsenic during smelting, oxidation must be avoided. Oxidation is the chemical process where atoms lose electrons. As tin became more available, the arsenic no longer needed to be preserved in the copper for added strength. Bronzes, copper alloyed with tin, were regularly used in Mesopotamia around 3000 BCE to 2800 BCE, and spread rapidly, reaching central Europe by 1800 BCE to 1500 BCE (Tylecote 1976:19-24). Tin bronze was more popular than arsenical bronze due to dangers of smelting arsenical bronze. Smelting arsenical bronze produces highly toxic fumes and people were able to identify metal working as a dangerous profession, as metal workers consistently had poor health.

The use of bronze also dramatically impacted the way metal tools were made. Copper had to be heated and hammered into objects, because furnaces were not hot enough to melt copper for casting, but bronze has a much lower melting point, ranging from 850 to 1,000 degrees Celsius depending on the amount of tin, and could be cast much more easily than copper. Casting objects became much more common because it was much easier to do with bronze. Molds for casting objects have been found made of stone, clay, bronze and copper from this period (Tylecote 1976:32). A great variety of objects were made by casting, including: small articles of jewelry, axes, knives, arrowheads, and a multitude of wood working tools.

The next advance in metallurgical processes was the use of iron. Iron was smelted from ores much like copper. Evidence suggests that iron was also first smelted in the Anatolian-Iranian region around 1500-1000 BCE (Tylecote 1976:40). The practice spread rather rapidly reaching Greece around 900 BCE, Spain by 800 BCE and Northern Europe, including Scandinavia and Britain by 500 BCE (Tylecote 1976:40).

Early iron smelting was done by a process called bloomery smelting. The iron ore along with the reducing agent, charcoal, would be heated to 1,150 degrees Celsius (Tylecote 1976:41-42). This produced a “bloom” which was a solid piece of iron with bits of slag and unburnt pieces of charcoal. The bloom could then be hammered to remove the slag and charcoal impurities. The temperature needed to melt pure iron, 1,540 degrees Celsius, was not easily achievable until the 19th century (Tylecote 1976:41). However, casting iron was possible through the use of cast iron. Cast iron is an alloy of iron and carbon that is produced by using a large fuel to ore ratio with an efficient bellows; the incorporated carbon comes from the combustion of wood or coal. Cast iron has a melting point of about 1,200 degrees Celsius and can then be cast with molds much like bronze (Tylecote 1976:65). This was rarely done in the West, but was common in China.

Other metals were also being used in new ways during the early Iron Age. Silver and gold were being used for coinage, especially in Greece (Tylecote 1976:48). Bronze materials were still in use as a fairly strong metal that was resistant to oxidation and for decorative purposes. Copper was mixed with electrum, an alloy of silver and gold, to bring out the natural gold color. Lead was also being used for sealing cramps, which are bars that held stone together, for joining masonry and as cladding in ships’ bottoms

(Tylecote 1976:48-49). Silver was generally extracted from lead deposits, so a great deal of lead was also produced, but prior to Roman times that lead had only limited uses.

During the Roman period, 500 BCE to 500 CE, the biggest change in metallurgy was the scale of production. Slag waste could easily be measured in tons rather than kilograms at major production sites. Brass was also introduced during this period, around 50 BCE (Tylecote 1976:58). Brass is an alloy of zinc and copper. This metal was primarily used in coinage until about 160 CE, when it was replaced by gunmetal. Gunmetal is an alloy of copper, tin and zinc (Tylecote 1976:58). This metal was named for its much later popular use in making guns, before iron could be cast regularly. Gunmetal and bronze remained the primary nonferrous metals in use for objects during the Roman period from about 50 BCE to 400 CE, though lead still had a great deal of prominence for the multitude of plumbing projects in which the Romans engaged. These plumbing projects, of course, had long term health effects. Lead remained primarily a by-product of silver production, but was much more widely used.

During the middle ages there were few innovations in metallurgy. One of them was the recognition of the value of steel. Steel was sometimes produced during iron smelting as it is a high carbon alloy of iron (Tylecote 1976:68). It has increased tensile strength and holds a sharp edge with greater durability than iron. Steel was highly valued and fairly rare, as it was produced from smelting only particular iron ores. The iron most commonly produced at this time was high in phosphorus, which made it unsuitable for steel production (Tylecote 1976:68). This made steel a valuable trade item and it was imported from areas that had iron ores more suitable to producing the steels. Because steel was a valued commodity, it was often welded onto wrought iron (Tylecote

1976:68). This means a wrought iron knife might have steel welded onto the cutting surface to improve its function.

The metallurgy technologies initially developed during the Roman period remained fairly static until the introduction of water power for metallurgy during the early 15th century. In 1408 the Bishop of Durham established the first water powered bloomery in Britain (Tylecote 1976:65). Evidence suggests that water power was employed to run the bellows. The development of water power, contact with the East where cast iron was commonly used, and the need for cast iron to create efficient guns all contributed to the development of the blast furnace. The blast furnace reduces the melting point of iron by carburizing it in a strongly reducing environment and introduces a flux to enable slag to separate from of the iron (Tylecote 1976:65).

Bloomery smelting was the method used throughout the Viking Age and the Medieval period (Senn et al. 2010:132-133; Blakelock et al. 2009:1745-1746; Paynter 2006:271). During this process the iron remains solid while the slag becomes molten, leaving a spongy metal bloom (Senn et al. 2010:132-133). The bloom produced is mostly iron but with slag and charcoal impurities. Smelting the ore produces the majority of the slag, but the next step of the processing also produces some slag. The second step is to hammer the bloom into a billet or bar; this hammering removes adhering slag and charcoal and helps remove additional impurities from the iron (Blakelock et al. 2009:1746).

Another possible source of slag from this period is from metals being melted down for reuse or from casting. Slag from this process is known as crucible slag, while slag from smelting is called smelting slag. Slag is in-between metal objects and stone objects in terms of preservation. Some of the metallic content of the slag can oxidize and corrode, but most of the material preserves very well, due to being mostly silicate in nature.

Iron production in the Viking Age, in Scandinavia, was primarily done by farmers, not specialist blacksmiths (Christiansen 2006:203). This helped farmers supplement their income and be more self-sufficient. Most of Scandinavia is marginal arable land. While no detailed accounts exist about iron working during the Medieval period, iron working continued to be a household industry into the nineteenth century in parts of Scandinavia (Christiansen 2006:203).

The process of this household iron working is quite well established. The first step was to locate bogs or shallow lakes where small lumps of iron ore could be obtained. This is known as bog ore and is a renewable resource. The ore was identified based upon color, size, context and taste. It was then dried and stored. Next the site for smelting is prepared, including building furnaces and creating charcoal. Then the ore was smelted in the furnaces and a bloom was obtained. The bloom was then processed as described above (hammered into a billet or bar). This could then be sold or processed into other objects (Christiansen 2006:203-204).

There are two likely sources of slag from Fröjel, Långume, and Little Hultungs. The first type of slag expected would be smelting slag, especially from iron through the bloomery smelting process. The second type of slag that is probable is crucible slag from melting finished products and using the metal to cast new objects. This second process would be expected with bronze, gold, or silver rather than iron. Melting iron requires very high temperatures that would have been nearly impossible to achieve at these sites during their periods of occupation.

Analytical Techniques for Studying Metals

There are many different analytical techniques that can be used to study metal artifacts and slag. For metals containing lead, a principal method is to examine lead isotope ratios to determine the geographic origin of a lead containing object. Different ore sources have distinctly different lead isotope ratios, enabling provenanceing of lead containing metal objects (Brill 1970:144). The difference in isotope ratios is due to the difference in the geological time period of the lead deposits. Lead isotopes can be measured by Inductively Coupled Plasma Mass Spectrometry, usually referred to as ICP-MS. Another popular method for determining lead isotope ratios is Thermal Ionization Mass Spectrometry (TIMS). In archaeology this technique is used with lead, silver, copper, and bronze objects and slag. This method is reliable due to the fact that lead isotope ratios are insensitive to the chemical history of the object, in other words, chemical changes do not affect the lead isotope ratios (Brill 1970:145, Meliksetian et al. 2007:7-8). The only way that the ratios will not reflect the true ore origin is if lead from different sources is mixed.

Examining sources of lead-containing metal objects can be useful to understanding possible trade connections. Analysis of smelting slag can show where people were getting raw materials rather than trading for finished goods. This can be especially revealing in areas that do not have native resources of the metal in question.

Unfortunately this cannot be used with ferrous objects or slag, as lead does not occur naturally in iron ore deposits.

Chemical analysis of copper artifacts can also be used to identify objects that are made from smelted copper rather than native copper. Smelted copper objects have substantial traces of arsenic and nickel. These trace elements can be identified using several analytical chemical techniques, including ICP-MS, X-ray Fluorescence (XRF) Spectrometry, Atomic Absorption Spectrometry and others. ICP-MS is commonly used for copper sourcing and slag analysis (Senn et al. 2010:135). Trace element analysis is useful to examine very early copper artifacts, as it enables one to determine if native copper sources are being used or if copper ore is being smelted. These techniques can also be applied to other aspects of metallurgy. They help identify possible alloys and impurities. Examining the elemental composition of metals can help create a better understanding of how and why these objects were produced. Elemental composition can show intentional alloyed metals, can indicate provenance, or reveal technical processes due to the identification of impurities. For example slag from efficiently smelted iron will have much less iron remaining than slag from more poorly smelted iron.

One of the most popular methods for examining elemental compositions of metal artifacts and slag is X-Ray Fluorescence (XRF) spectrometry. In XRF the atoms of the sample are irradiated with high energy x-ray photons (Shackley 2009:1). This removes electrons from the atoms, converting them into ions. To become more stable electrons from the outer orbitals redistribute to lower energy orbitals, thereby releasing energy (Shackley 2009:1). This released energy is measured and each element (e.g. carbon, copper etc.) produces a characteristic spectrum, so that the elemental composition of the sample can be determined. Differing intensities of the peak readings indicate the proportion of constituent elements (Shackley 2009:2). XRF analysis is commonly used to study slag compositions, because it is a cost-effective method of analysis (Humphris et al. 2009:362; Senn et al. 2010:135). Ores from different sources typically have different elemental compositions. It is however necessary to analyze a number of samples because slag, even from the same smelt are heterogeneous, meaning their composition is different from one another (Paynter 2006:272).

Neutron Activation Analysis is another chemical method, sometimes used to determine impurities in silver coins. This method is preferable due to its nondestructive nature. Neutron Activation analysis is done by bombarding the sample with neutrons to create artificial radioisotopes that can then be measured (Gordus 1970:169). Neutron Activation Analysis has been used primarily to determine how much various silver coins were debased in specific times and places, and to examine why people were debasing coins in certain times and places (Gordus 1970:169). Debasing coins is the process of mixing less valuable metals into the coins to reduce the production costs.

Metallography differs from chemical analysis, but is useful for examining different aspects of metal artifacts. Rather than examining the specific chemical makeup of an object, metallography examines the physical properties of the object, usually at a microscopic level. This approach is used to examine alloy content, impurity patterns, and inclusion analysis (Northover 1985:58). Metals, particularly smelted metal alloys, can have slag inclusions, similar to the way diamonds often have dark black carbon inclusions. Metallography starts by acid etching a surface that is then examined under a microscope. The type of microscope used is determined by the specific needs of the analysis. Metallographic processes examine the microstructure to understand how an object was created, such as being cast or wrought. Other aspects that can be examined include: inclusion types and morphology, hardness, measure of carburization in ferrous metals, grain size and more. These aspects all help one understand finished objects. Metallography can explain specific manufacturing techniques, such as forging, welding, or casting, and can also explain possible function through examination of the physical properties (Northover 1985:58-59).

For this research TIMS was used to determine lead isotope ratios in slag from bronze smelting to determine the source of the copper used. While much is known about general trade connections on Gotland, little research has been done regarding metals trade. Gotland has very limited metal resources and no copper resources, being mostly a limestone outcrop in the Baltic.

The second goal of this research is to examine smelting techniques. Using ICP-MS to determine the elemental composition of slag to examine inclusions and impurities enables this examination of smelting techniques. This approach is being applied to both ferrous and cupreous slag. As a prominent trade location the island may have been introduced to new ideas more rapidly than places with less contact. Examining techniques such as metal working in areas with a great deal of contact may help provide insight on how ideas spread.

Chapter 4: Methods Used for Slag Analysis

Introduction to Methods

The slag analysis has been conducted using two techniques. The first technique for the analysis of slag is Inductively Coupled Plasma Mass Spectrometry (ICP-MS). In doing ICP, samples are ionized in a plasma and mass analyzed in a mass spectrometer (PerkinElmer Instruments 2001). ICP mass spectrometers are capable of resolving the isotope distributions of each element in the sample. This produces very accurate results, revealing even very minute differences in trace elements, as noted above, including isotope compositions of many elements that vary with source (PerkinElmer Instruments 2001). This analysis was employed to identify elemental make up of slag samples.

The second technique used was Thermal Ionization Mass Spectrometry (TIMS) to determine lead isotope ratios in cupreous, or copper containing, slag samples. This process is able to very accurately determine isotope ratios in a sample. The sample is ionized thermally by passing electric current through a ribbon in a vacuum (Mueller and Vervoot n.d.). The ions are then focused into a beam that passes through a magnetic field that separates the beam into different parts based upon electric charge and mass (Mueller and Vervoot n.d.). Isotopes are examples of the same element with different amounts of neutrons, which makes them have measurably different mass. This process separates different isotopes from one another; the amounts of each can then be measured and the isotope ratios determined.

Origin of Samples and Site Descriptions

Slag samples were obtained from excavations at three different sites on Gotland: Little Hultungs, Långume, and Fröjel. These samples were initially characterized by their type according to physical appearance. There were two different type classifications: ferrous slag, identified by reddish areas on the slag or mostly reddish samples, and cupreous slag, identified either by a greenish color or by various grey or brown colors.

Site 1: Little Hultungs

Little Hultungs was excavated in the summer of 2009 and is located on the northern end of Gotland (Figure 2.8). The site was determined to be a single family farmstead dating from c.a. 1150 to 1400 CE. Excavations uncovered the family farmhouse and some of the surrounding area. See Figures 2.9 and 2.10 for the excavation area and the surrounding locations. Due to modern plowing slag was found scattered around the site including within the boundaries of the house. Two-thirds of the slag found was in the eastern half of the excavated area. There is a small stream to the east-north-east of the house. The concentration of slag in the eastern half of the area and the location of the stream indicate the likely location of a workshop or work area in-between the house and the stream. The area excavated only extended about three meters from the expected edge of the house toward the stream. There were no clear indications of any particular dumping place for the slag, but that is likely due to the disturbance of modern plowing and the limited area of excavation. Slag found at this site was classified as cupreous slag.

Site 2: Långume

Långume is another single family farmstead site located in the northeastern part of the island, dating from c.a. 900 to 1200 CE (Figure 2.8). Slag found at Långume was classified as ferrous slag. Much like the site at Little Hultungs there were no clear deposits of slag in a single area indicating a possible dump.

Site 3: Fröjel

Fröjel is different from the other sites in that it was a prominent trading community during Viking times (c.a. 800 to 1150 CE) and into the early medieval period (c.a. 1150 to 1250) (Figure 2.8). During the Viking period it was a very prominent port known as Ridanäs. In the summer of 2000 a metal workshop was uncovered in Fröjel (Gustafsson and Soderberg 2005:14). This included finding two furnaces which had been filled in with moulds, hearth lining, crucibles, and slag. The first furnace was radio-carbon dated to 970-1160 CE and the second furnace dated to 1110-1230 CE (Gustafsson and Soderberg 2005:14). Hearth lining material was identified as originating from a cupellation hearth that was used for refining silver. Further evidence supports the production of silver jewelry in this workshop, including moulds for armbands, and a small collection of silver shavings (Gustafsson and Soderberg 2005:16). Armbands of the type produced from the moulds found in Fröjel were very common in the late Viking period and were made from both silver and bronze. Evidence of iron work was also present, inferred from the presence of tube-shaped clay packages which are thought to have been used in carburization of steel for tools and weapons (Gustafsson and Soderberg 2005:17). Samples of both ferrous and cupreous slag were classified from Fröjel.

A selection of slag samples from each of the three sites were sent to the University of Idaho by Dr. Dan Carlsson. There were a total of 199 samples, with a total mass of 1276.0 grams. The majority of the samples were from Fröjel, with 165 samples and a mass of 528.1 grams. There were 24 samples sent from Långume with a mass of 425.0 grams, and there were 10 samples sent from Little Hultungs with a mass of 322.9 grams.

Samples sent from Fröjel were divided into two primary sets, the first set, containing 145 of the samples were identified as probably ferrous, while 20 of the samples were identified as possibly cupreous. Six samples from the cupreous set were selected to be tested, with one of those samples selected for lead isotopic analysis. The sample selected for lead isotopic analysis had a distinct green spot on the sample. The other samples were selected based upon color and selected from the different layers and trenches represented.

Six samples were selected for testing from Långume. The samples were selected to represent a variety of different physical characteristics present, including color and texture.

The ten samples were sent from Little Hultungs. The six samples selected for testing were based upon color and texture to be representative of the range of possibilities.

Table 4.1 shows how many samples from each site were tested and whether they were identified as likely to be cupreous or ferrous.

Table 4.1 Slag Sample Classification

Slag Type	Fröjel	Långume	Hultungs
Cupreous	6	0	6
Ferrous	6	6	0

Methods of Chemical Analysis

All but two samples in this study were subjected to Inductively Coupled Plasma Mass Spectrometry (ICP-MS) in order to examine element composition of both ferrous and cupreous slag samples. The two samples that were not tested by ICP-MS were due to a miscommunication with the laboratory. The two samples not tested by ICP-MS are the two samples used in the lead isotopic analysis described below.

Lead (Pb) isotopic analysis by Thermal Ionization Mass Spectrometry (TIMS) was used on two cupreous samples to determine lead isotope ratios in slag from bronze smelting with the ultimate goal of determining the source of the copper used. Only two samples were selected due to budget constraints. One sample came from Fröjel while the other sample came from Hultungs. There were no samples identified as cupreous from Långume, so no samples were taken from there for this analysis.

Ferrous Slag Analysis

Analyses were done by Activation Laboratories of Ontario Canada. The ferrous slag samples were analyzed using the Activation Laboratories Code 4 Litho method.

This method includes Fusion Inductively Coupled Plasma (FUS-ICP) to examine the mineral properties of the sample and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to determine trace element composition.

> “Fused sample is diluted and analyzed by Perkin Elmer Sciex ELAN 6000, 6100 or 9000 ICP/MS. Three blanks and five controls (three before sample group and two after) are analyzed per group of samples. Duplicates are fused and analyzed every 15 samples. Instrument is recalibrated every 40 samples” (Activation Laboratories 2011 4Litho).

Cupreous Slag Analysis

Cupreous slag was analyzed using the Activation Laboratories’ MIT package analysis. The MIT package is a two step analysis. The first part is an analysis by Instrumental Neutron Activation Analysis (INAA). The second part is done with Inductively Coupled Plasma (ICP). The INAA portion of the process is detailed below.

> “Samples are encapsulated in polyethylene irradiation vials and irradiated for 5 minutes at a thermal neutron flux of $7X10^{12}$ n.cm^{-1}sec at the McMaster Nuclear Reactor [at McMaster University, Hamilton, Ontario]. Samples are allowed to decay and are measured on a high purity Germanium detector with resolution of 1.7 KeV for the 1332 KeV photopeak. Calibration is achieved using an internal calibration reference by NiCr flux wires with NIST [the National Institute of Standards and Testing] metal standards used as controls” (Hoffman 2011).

The ICP portion is described below:

> “Samples are digested in Teflon test tubes with nitric, perchloric, hydrofluoric and hydrochloric acids for 18 hours at temperatures to 260 degrees C. They are diluted and analyzed with a Varian/Agilent 735 ICP. NIST standards are used as controls” (Hoffman 2011).

Lead Isotope Ratio Analysis

Lead isotope ratios were determined using the Activation Laboratories’ Thermal Ionization Mass Spectrometry (TIMS) analysis. This analysis is described below:

> “Pb were separated using the ion-exchange technique with Bio-Rad 1x8. Pb isotope compositions were analyzed on VG Sector 54 multicollector mass spectrometer. The measured Pb isotope ratios were corrected for mass fractionation calculated from replicate measurements of Pb isotope composition in NBS SRM – 981 standards” (Kapusta 2011).

Samples were tested based upon the suggestions of Activations Laboratory after I had explained the nature of the samples and the goals of my research. The cupreous samples were tested differently based upon the upper detection limits of copper from the methods used for the ferrous samples.

Chapter 5: Results of Slag Analysis

Slag samples from the various sites had been assigned categories of ferrous or cupreous based upon visual identification (seen in Table 4.1). Looking at the data from the elemental analysis of the samples this appears to be inaccurate. The samples identified as cupreous from Fröjel are much more indicative of being ferrous slag samples. Rather than organize the data with misidentified categories the data will be organized by site. In all of the samples metals and oxides are presented as percentage of the sample. There are four oxides and six metals that are of primary interest to the study of the slag samples.

The first oxide listed is SiO_2. This is a measure of how much silicon is in the sample and is an indicator of how mineral-like the sample is compared to how metal-like it would be. Silicon is the most abundant element in Earth's crust and is present in nearly all ores. The more refined the iron sample becomes, the lower the SiO_2 percentage in the iron sample and the higher the SiO_2 percentage in the discarded slag samples. Slag samples are expected to be high in SiO_2 and low in the element refined from the sample (iron for ferrous slag for example).

The second oxide is Al_2O_3. Aluminum is found in most ores and many minerals. For the purpose of either copper or iron smelting aluminum is an undesirable impurity. There should be a higher percentage of Al_2O_3 left in the slag than there was present in the original ore as nearly all of it should be left behind. Refined metal samples should be very low in Al_2O_3.

The third oxide is Fe_2O_3. This is a measure of how much iron is in the sample. As one of the two desirable metals for the purpose of this study it should be expected to be fairly low in the slag samples. Iron smelting slag should contain a noticeable, but very low percentage of iron. The process of smelting the ore should extract most of the iron, but especially with earlier smelting techniques such as bloomery smelting, there should still be some iron present in the slag. Copper smelting slag should expect to see moderately high amounts of iron, as iron is not being extracted from copper smelting.

The forth oxide is CaO. Calcium is present to some degree in most ores and many minerals. Certain types of ores may be more likely to have higher amounts of CaO than other ores. Much like SiO_2, the more CaO left in the sample the less likely it is a refined sample of a metal. CaO can also be added to the smelting process as a flux to help the slag run off from the metal. This is usually done during blast furnace smelting and is likely not an explanation for any CaO found in the samples studied from these three sites.

The first metal on the list is Ni, or nickel. Nickel is commonly found in meteoric iron samples and therefore is expected in only trace amounts in these samples. A noticeable percentage of nickel would likely indicate the presence of meteoric iron. As meteoric iron was primarily used before iron smelting became popular it is not expected in any samples.

The second metal is Cu, or copper. Copper is one of the two desirable metals in this study (the other being iron). There are two possible explanations for slag with moderate to high amounts of copper. The first would be copper smelting slag which would have fairly low, but noticeable amounts copper much like iron slag with Fe_2O_3. The second possibility would be crucible slag. Crucible slag is refined metal that has been lost during either re-melting the metal or from casting the metal. High amounts of copper would be expected from crucible slags, possibly with fairly large amounts of arsenic, tin, and lead, as examples of bronzes.

The third metal is Zn, or zinc. Only trace amounts of zinc are expected to be present in any of the samples. More than trace amounts of zinc along with copper would be indicative of a brass alloy or of a gunmetal alloy if tin was also present. Neither of these alloys are expected in any of the samples.

The fourth metal is As, or arsenic. Arsenic is also only really expected in trace amounts in any of the samples. It is expected to be a bit higher in cupreous samples than in ferric ones, as arsenic is commonly occurs within copper ores. More than trace amounts of arsenic found with copper could indicate an arsenical bronze, but this is not expected as tin bronzes had replaced arsenical bronzes by the Viking Age.

The fifth metal is Sn, or tin. Tin is added to copper to produce bronze. Bronze is stronger than pure copper. Tin might be found in samples that are crucible slags from bronze casting or re-melting. This would be found along with copper. Tin should only be present in smelting slags of either iron or copper in trace amounts as it does not occur at the same places as iron or copper.

The sixth and final metal is Pb, or lead. Lead is sometimes added to copper or bronze to make the material less viscous and easier to melt (Gale and Stos-Gale 1982:13). This makes it easier to cast, but also makes the metal softer. While adding lead to bronze may make it softer, this was generally not a problem during the Viking Age and the early medieval period as bronze was used primarily for decorative and ornamental purposes.

Results of Samples from Fröjel

Table 5.1 shows the results of the element analysis for all of the samples from Fröjel that were tested except sample 13. Sample 13 was one of the two samples selected for the lead isotope examination by TIMS, but by mistake was not analyzed for elemental composition. This table shows iron content as Fe_2O_3, this is the most common form of oxidized iron, commonly referred to as rust.

Table 5.1 shows the experimental percentage of each of the oxides and metals of interest for the slag samples. The samples from Fröjel show varying amounts of SiO_2. Nine of the eleven samples have a range of 64.56 percent to 73.98 percent SiO_2. This is expected in smelting slag. The goal is to remove the impurities from the iron and the waste produced is the slag. Smelting slags are generally expected to be high in SiO_2. These same samples have a range of 3.19 percent to 14.21 percent Fe_2O_3. The amount of iron in these samples is expected in smelting slags. Early smelting was inexact and did not extract all of the desired metal from the ore, leaving some metal present in the slag. Samples 2, 3, 4, 5, 15, 16, 17, 18, and 19 all have numbers that support that these are ferrous iron smelting slag samples. Samples 15, 16, 17, 18, and 19 were originally identified visually as cupreous slag samples, but given the content of the samples these all appear to be ferrous samples.

Table 5.1 Compositional Analysis of Fröjel Samples

Sample Number	1	2	3	4	5	6	15	16	17	18	19	Total	Mean	Standard Deviation
SiO_2	6.68	70.53	73.51	70.57	71.46	29.45	64.56	67.56	73.98	66.36	66.25	661.34	60.12	21.62
AL_2O_3	0.72	11.12	8.84	6.28	11.00	4.68	7.45	11.11	8.83	8.58	10.15	88.76	8.07	3.19
Fe_2O_3	95.02	5.59	5.29	14.21	5.79	39.05	8.56	7.74	4.54	3.19	4.40	193.38	17.58	27.63
CaO	1.61	3.76	4.5	4.08	3.66	20.26	12.45	5.58	2.32	3.16	8.27	69.65	6.33	5.54
Ni	0.008	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.006	0.007	0.007	0.028	0.003	0.004
Cu	0.013	0.005	0.008	0.004	0.005	0.006	0.404	0.266	>1.000	>1.000	2.540	5.251	0.477	0.786
Zn	<0.003	0.007	0.008	<0.003	0.008	0.003	<0.003	0.008	>1.000	0.012	0.009	1.055	0.096	0.300
As	0.0046	<0.0005	<0.0005	<0.0005	<0.0005	0.0054	0.0045	0.0025	0.0026	0.0336	0.0056	0.0588	0.0053	0.0096
Sn	0.0038	0.0091	0.1400	0.0004	0.0004	0.0002	0.0075	0.0580	0.1520	0.0125	0.0200	0.4039	0.0367	0.5652
Pb	<0.0005	0.0011	0.0021	0.0006	0.0026	0.0007	0.0171	0.0504	0.5110	0.4070	0.4880	1.4806	0.1346	0.2164

Sample 1 and Sample 6 are much lower in SiO_2, and much higher in Fe_2O_3. Sample 1 is especially high in Fe_2O_3, with 95.02 percent, and 6.68 percent SiO_2 reported. Sample 1 is also unusual in that the percentages reported add to 104, suggesting some margin of error, perhaps due to the very large amount of Fe_2O_3 present. Sample 12 from Långume is very similar with 94.64 percent Fe_2O_3 and 6.79 percent SiO_2 and with 104.9 total percent reported. The high percentage of Fe_2O_3 in Sample 1 argues against it being a slag sample, but there at least two other possible explanations. The first explanation of Sample 1 is that it may be a misidentified, highly corroded iron object. The second explanation is that it could be a piece knocked off of an iron bloom. Iron blooms are spongy with impurities and slag inclusions. The bloom is hammered to remove impure portions and Sample 1 could have been removed from a bloom, despite having high iron content.

Sample 6 is much lower in Fe_2O_3 than Sample 1, but still more than doubles any of the other samples from Fröjel. This sample has 39.05 percent Fe_2O_3, and 29.45 percent SiO_2. Sample 6 is the highest in CaO of the samples from Fröjel, with 20.26 percent reported. These numbers could indicate a copper smelting slag, but it has only a trace amount of copper, indicating that this is not the case. It would be nearly impossible to refine copper ore to leave only trace amounts of copper behind.

All of the samples from Fröjel are ferric in nature and at least nine of the tested samples appear to be iron smelting samples. There are some possible explanations for the other two samples and this will be explored further in Chapter 6.

Results of Samples from Långume

Table 5.2 shows the results of the oxide and metal content for all six samples tested from Långume.

Table 5.2 Compositional Analysis of Långume Samples

Sample Number	7	8	9	10	11	12	Total	Mean	Standard Deviation
SiO_2	15.05	30.02	53.62	22.47	44.8	6.79	173.20	28.87	17.75
AL_2O_3	2.57	3.95	3.93	2.05	8.17	0.68	21.35	3.56	2.57
Fe_2O_3	75.88	53.17	26.29	66.47	4.22	94.64	320.67	53.45	33.26
CaO	6.08	10.57	12.07	7.50	7.89	2.82	46.93	7.82	3.28
Ni	<0.002	<0.002	<0.002	<0.002	0.004	<0.002	0.004	0.001	0.002
Cu	0.006	0.004	0.005	0.005	0.004	0.005	0.029	0.005	0.001
Zn	0.004	<0.003	<0.003	0.004	0.009	0.007	0.024	0.004	0.004
As	<0.0005	<0.0005	<0.0005	0.0005	0.0007	<0.0005	0.0012	0.0002	0.0003
Sn	0.0931	0.0012	0.0005	0.0016	0.0005	0.0005	0.0974	0.0162	0.0377
Pb	0.0024	0.0008	<0.0005	0.0007	0.0039	0.0006	0.0084	0.0014	0.0015

The samples from Långume show considerable variability. Generally the samples have higher Fe_2O_3 and lower SiO_2 numbers than the samples from Fröjel. Again there are only trace amounts of copper, suggesting that these are not cupreous samples. The samples seem to have higher amounts of Fe_2O_3 than would be expected in most smelting slag samples. The only samples that fit into expected slag ranges are sample 11, which fits very well, and sample 9, which fits only marginally. Much like sample 6 from Fröjel, many of these samples could indicate copper production except for the only trace amount of copper found. This suggests that the samples from this site are either very poorly refined, or possibly unrefined.

In general, there is also a much higher amount of CaO compared to SiO_2 in the Långume samples than in the Fröjel samples. This could suggest a different origin for the iron ore. On Gotland the only source of iron ore is bog iron. Bog iron composition will be highly variable from source to source.

Sample 12, is very similar to Sample 1 from Fröjel. It has a very high concentration of iron content at 94.64 percent Fe_2O_3. This again suggests the possibility of it being a misidentified refined ferric object of some sort, or a piece hammered off of an iron bloom.

Results of Samples from Little Hultungs

Table 5.3 shows the compositional results for the four of the samples from Little Hultungs, excluding Sample 22. Such small amount of data was provided for Sample 22, that it appears the results for that sample are unreliable. Sample 22 was also the smallest of the samples from Little Hultungs submitted for analyses, and it may have been insufficient for the analysis.

Table 5.3 Compositional Analysis of Little Hultungs Samples

Sample Number	20	21	23	24	Total	Mean	Standard Deviation
SiO_2	0.39	4.57	5.34	7.05	17.35	4.34	2.83
AL_2O_3	0.08	0.24	0.33	0.43	1.08	0.27	0.15
Fe_2O_3	0.29	2.05	2.20	2.29	6.83	1.71	0.95
CaO	0.26	0.36	0.64	0.92	2.18	0.55	0.30
Ni	0.024	0.020	0.023	0.022	0.089	0.022	0.002
Cu	68.5	61.6	59.1	51.2	240.4	60.1	7.1
Zn	0.008	0.010	0.004	0.022	0.044	0.011	0.008
As	0.138	0.124	0.188	0.180	0.630	0.158	0.031
Sn	15.6	12.2	14.3	12.9	55.0	13.8	1.5
Pb	8.36	5.32	0.87	8.39	22.94	5.74	3.55

The samples from Little Hultungs are very different than the samples from Fröjel and Långume. Both the SiO_2 and Fe_2O_3 levels are quite low in comparison. The low

$Si0_2$ suggests these are more metal than rock, while the low Fe_2O_3 levels could indicate iron slag, the low values of SiO_2 and CaO suggest that these are not smelting slag. All four samples have very significant amounts of Cu and Sn (copper and tin). The ratio of copper to tin in the samples is within the expected ranges of a refined tin bronze. Trace amounts of zinc and arsenic rule out these samples as being brass, gunmetal, or arsenical bronze. The composition of these samples suggests that these are bronze crucible slag rather than any sort of smelting slag.

There is a significant amount of lead found in three of the four samples. Lead was commonly added to bronze to make the material less viscous at high temperature and make the metal easier to cast. This supports that evidence that these samples are crucible slag, waste run off from either melting the metal or casting the metal.

Silver and Gold Content from samples

None of the samples tested had more than trace amounts of silver (Ag) or gold (Au). Silver for all samples ranged from less than 0.00005 to 0.00828 percent, and gold for all samples ranged from less than 0.00001 to 0.00081 percent. Gold analyses were only performed for cupreous slag samples from Fröjel and Little Hultungs, while silver was analyzed in all samples. Results indicate that none of the samples have added silver or gold, or are attempts at silver smelting.

Isotope Ratio Results

Lead isotope ratios were determined for two samples. Sample number 13 from Fröjel and sample number 14 from Little Hultungs. This purpose for collecting this data was to attempt to provenance copper sources for each site. Table 5.4 shows the lead isotope ratios reported by Activation Laboratories which are referenced to ^{204}Pb. This is not referencing common to the archaeological literature which instead uses ^{206}Pb as the reference for ^{207}Pb and ^{208}Pb. Table 5.5 shows the values for samples 13 and 14 converted to ^{206}Pb reference that is most commonly used in archaeological lead isotope ratio studies for provenancing (Stos-Gale and Gale 2009).

Table 5.4: Lead Isotope Ratios Reported by Activation Laboratories

Sample	Site	^{206}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{208}Pb/^{204}Pb
13	Fröjel	18.497	15.721	38.718
14	Hultungs	17.591	15.633	37.630

Table 5.5: Lead Isotope Ratios for Provenancing

Sample	Site	^{208}Pb/^{206}Pb	^{207}Pb/^{206}Pb	^{206}Pb/^{204}Pb
13	Fröjel	2.093	0.850	18.497
14	Hultungs	2.139	0.889	17.591

Most of the data collected for provenancing copper has been reported for archaeological sites centered around the Middle East and the Aegean (Höppner et al. 2005, Klein et al. 2009, Stos-Gale and Gale 2009, Stos-Gale and Gale 2010). Some work has been done in central Europe including Germany and Austria (Frotzscher et al. 2007, Stos-Gale and Gale 2009), but extensive literature searching has uncovered no published data from Northern Europe including Scandinavia. The Oxford Archaeological Lead Isotope Database (OXALID) maintains a data base of lead isotope data by country and region shown in Table 5.6 (Stos-Gale and Gale 2009).

Table 5.6 Analyses of Lead Isotope Data on OXALID (Stos-Gale and Gale 2009: 209)

Region or Country	Number of Analyses
Greece	858
Cyprus	430
India and Iran	166
Turkey	367
Spain	101
Italy	332
Bulgaria	147
British Isles	464
Ireland	11
Germany, Austria, Bohemia	464
Red Sea Region	74

No other element analyses were performed on these two samples due to miscommunication with Activation Laboratories. Both samples 13 and 14 were originally thought to be cupreous slag samples. Judging by the element content data gathered from the other samples from Fröjel and Little Hultungs, there is significant doubt that sample 13 is cupreous, while sample 14 is likely a finished bronze. Sample 13 is likely a ferrous slag since all of the other samples from Fröjel that were analyzed were ferrous. Sample 14, however, is likely a refined bronzed based upon the results of the other samples from Little Hultungs.

Sample 13 from Fröjel has lead isotope values that are consistent with many of the values from the literature reported in Table 5.7. The closest value on Table 5.7, to sample 13, is an average for samples from Massif Central in southeast France. This is difficult to interpret for a few reasons. First, there is considerable doubt as to whether sample 13 is cupreous or not. Second, having no data for Scandinavian copper sources and only one sample tested from Fröjel also complicates interpretation. Lead isotope ratios from geographically separate areas can sometimes be similar. In the absence of ratios from sites in Scandinavia, it is difficult to assume its origin. It would be best to compare the numbers first to Scandinavian sites and then expand the search if there were no matches, as closer locations would be more likely to be the source for materials.

The sample from Little Hultungs is unique in that it has values that exceed the values found in the literature, including locations in Austria, France, Spain, and Turkey as seen in Table 5.7. The numbers for $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$ are higher than any values found in the literature, while the number for $^{206}Pb/^{204}Pb$ is lower than any of the literature values. The closest values to sample 14 are also from the Massif Central area of southeast France.

The isotope numbers for sample 14 may surpass other values due to mixing multiple sources of lead into the finished product. The samples from Hultungs are all indicative of finished bronzes and there is reason to believe that bronze objects were being recycled. Three of the four bronze slag samples from Hultungs also exhibit very high levels of lead (as discussed above), indicating that lead was being added to the bronze to aid casting.

Table 5.7 Average Lead Isotope Ratios

Location	$^{208}Pb/^{206}Pb$	$^{207}Pb/^{206}Pb$	$^{206}Pb/^{204}Pb$
South Spain	2.0775	0.84292	18.57854
Inn Valley (Austria)	2.0369	0.82525	19.043
Southern Turkey	2.06086	0.82871	19.001
Massif Central (South East France)	2.08946	0.84752	18.514
Fröjel (Gotland)	2.093	0.850	18.497
Little Hultungs (Gotland)	2.139	0.889	17.591

Chapter 6: Conclusions

Conclusions of Elemental Analysis

All of the slag samples tested were ferrous, except the four samples from Little Hultungs. The ferrous slag samples seem mostly to be smelting slag, while the cupreous samples are all crucible slag. Smelting slag is the direct waste from extracting metal from the ore, while crucible slags are waste globs that form from overfilling moulds and other drippings during the casting of already smelted metals. This makes sense as there are no native copper deposits on Gotland, while there are some limited sources of iron. It seems reasonable to believe that copper and bronze were imported to Gotland already refined rather than in the form of ore, while iron was being smelted from the local resources that were available. Smelting the metal before trade means moving less material as the waste (slag) has already been removed. The imported copper and bronze may have taken the forms of trade ingots or finished goods.

None of the samples showed any evidence of meteoric iron, brass, gunmetal, or arsenical bronze. This is expected. Meteoric iron is generally rare, and was used primarily before smelting technology was developed. Arsenical bronze was also only really used before the development of tin bronze and would be very unlikely to be found at these sites. Brass and gunmetal were generally not in use in Scandinavia during this time period.

Fröjel Discussion

Nine of the eleven samples analyzed for content from Fröjel show good evidence of smelting slag. Those samples, 2-5 and 15-19, have a mean of 6.59 percent Fe_2O_3 with a standard deviation of 3.30. This low Fe_2O_3 level in the samples shows that good yields of iron were produced from the smelting. These samples are also all high in SiO_2, with a mean of 69.47 percent and a standard deviation of 3.34. This is also expected in smelting slag, as this is undesirable waste material.

Sample 6, has significantly more Fe_2O_3 present with 39.05 percent. This suggests a few possibilities. First, this sample may be from a different ore source than the other samples, an idea supported by the high CaO percentage in this sample. If it is ore from a different source, the smelting process used at Fröjel may not have been as successful as with other sources. Second, this sample might represent a poor quality unrefined ore sample rather than a smelting slag sample. Third, it may just be an example of slag from a less successful smelt than were the other samples.

Sample 1 is significantly different from the other samples found at Fröjel. With 95.02 percent Fe_2O_3, this does not represent normal smelting slag or a piece of iron ore. The iron content is too high for either of those possibilities. This suggests that this sample is either a rather purely refined piece knocked off a bloom, or a highly corroded iron object that was misidentified as slag. The second explanation is quite likely as small pieces of highly corroded iron objects can be very hard to identify as such.

There is very clear evidence of successful iron smelting at Fröjel. This is expected as Fröjel was one of the major trading ports on Gotland during the late Viking and early medieval periods. There may be some evidence of ore from multiple sources being used in Fröjel with the differences in Sample 6. More examination of samples may reveal a trend. The most likely source of iron ore for Fröjel would be local bog iron from Gotland. Bog iron is usually fairly low in CaO impurities, but considering Gotland is mostly limestone, there may be more CaO in some Gotlandic bog ores. It is also possible, but not likely that Fröjel was getting ore from the mainland. Transporting refined iron, even in the form of billets or bars rather than finished tools would be preferable to transporting ore. With smelting occurring in Fröjel it is likely that iron working was occurring as well.

Långume Discussion

The samples from Långume are quite variable. This makes these more difficult to interpret as a group. If all of the samples are iron smelting slag samples then the techniques used at Långume not as well practiced as the techniques used at Fröjel. Most of the samples are so high in iron that it seems unlikely that they are smelting slags. Four of the six samples had more than 50 percent Fe_2O_3; a very high amount if iron was already extracted from these samples. A more likely explanation of these samples may be that they are unprocessed ore samples. The other two samples have 26.29 and 4.22 percent Fe_2O_3. These could also be unprocessed ore, though of lower quality than the other samples, or these could be smelting slags. If Sample 9, having more than 26 percent Fe_2O_3, is a smelting slag, then this process may have been less successful at Långume than at Fröjel.

If these samples are examples of unprocessed ore, a very distinct possibility, then it may be that the family that lived at Långume gathered iron ore to supplement their income without actually processing iron themselves. Another possibility is that most of these samples had been gathered, but not yet processed. Two of the samples suggest the possibility of processing at Långume, and tradition supports farmers doing small iron working production on the side in Scandinavia.

Little Hultungs Discussion

The four samples from Little Hultungs were the only samples that tested as cupreous. Each one of these samples are examples of highly refined bronzes, due to the amount of copper and tin in each sample. They are clearly examples of crucible slag; as this is the only type of slag that consists of refined metals. This suggests that bronze objects were being cast at this site. Three of the four samples also contain significant amounts of lead. Lead was often added to bronze to make the material more fluid at lower temperatures. This makes the bronze much easier to cast.

There is also other evidence at Little Hultungs that suggests that finished bronze objects may have been modified and recycled there. A bronze penannular brooch, a typical male late Viking Age style (ca 900-1050 CE), was found at the site, seen in Figure 6.1. These brooches are typically round, as seen in Figure 6.2 (next page). This brooch fragment (Figure 6.1), found at Little Hultungs, shows signs of being flattened and possibly broken up for scrap. The brooch style predates the likely period of occupation and was used as a source for bronze.

Figure 6.1: Pennannular Brooch Fragment

Figure 6.2: Pennanular Brooch (reconstruction)

Little Hultungs is unique as it represents a farmstead that is a second farm that was divided from the main branch of the Hultungs farmstead. This may indicate that the family living at Little Hultungs did not have enough resources to procure their own full farmstead. Recycling old bronze objects may have been a source of income for this family to supplement their farming. Another possibility is that many of the farmers on the island were also traveling traders and they may have been casting new bronze objects to use in trade. Bronze was a substance that was used primarily as a decorative material during the early medieval period (ca. 1050-1300 CE) as iron was common and a stronger material for tools and other goods. A final possibility is that the farmers at Little Hultungs were recycling bronze for their own private use.

The lack of evidence of iron working at Little Hultungs is a bit surprising given the many uses of iron during this time period and the evidence at both the other sites. Some possible explanations are that since Little Hultungs was a splinter off of the primary Hultungs farmstead, all of the needed iron work may have been done at the primary Hultungs farmstead for both families. The second possibility is that due to the location of Little Hultungs there may have been no convenient sources of iron ore to use in iron work.

Lead Isotope Discussion

The lead isotope analysis results are unclear for multiple reasons. The first problem is that elemental analysis was not done on the samples due to miscommunication. This makes it uncertain whether the samples were cupreous or not. It may be reasonable to assume that the sample from Fröjel was actually ferrous, as all the other Fröjel samples have been shown to be, and similarly that the sample from Little Hultungs was cupreous. This however, cannot be said with certainty without having an element analysis from each sample.

The fact that there were only two samples tested makes it difficult to draw any conclusions from the data, as there is very little. Ideally ten to twenty samples, already identified as cupreous samples, from each site should have been tested. For this project, the cost was prohibitive, each sample tested at activation laboratories cost $450, and the total budget for this project was $3,000.

Another problem is that there is no lead isotope source data for any sources in Scandinavia. To properly source the material it makes sense to start with possible sources closest to Gotland and then move outward. For this type of project to be carried out in the future, copper ores from various mines in Scandinavia need to be tested to compare results from the samples. This would be a costly endeavor as the general consensus is that twenty samples must be tested from each mining site, though this number may be too low for certain statistical analyses (Baxter et al. 2000).

The next problem with this analysis is the evidence at Little Hultungs for recycling bronze objects. Recycling bronze objects can point to mixing bronze from multiple sources. This will provide unreliable lead isotope data, which are hybrids of ratios from multiple sources.

The elemental analysis of the slags from Little Hultungs also shows significant amounts of lead in three of the four samples. Lead is sometimes added to bronze to improve casting. Added lead makes the bronze more fluid when casting. This property makes it flow into moulds more easily and more easily captures the shapes from the moulds. The addition of lead for this purpose will also skew the lead isotope data.

A possible way to avoid problems of mixed sources is to try and find trade ingots to test directly for the lead isotope data. Other finished goods may also be able to provide better information than slag. But before copper sourcing can be done in Gotland, Scandinavian copper sources must be identified and tested to be able to use in comparison.

Complications

The number of slag samples tested for this thesis is rather small. Ideally each slag sample should be tested in multiple places in order to obtain statistically meaningful results, as slag samples can be heterogeneous. Testing different points on the sample may provide different results, and results from multiple points would more accurately reflect the actual comprehensive composition of each sample. Tests should be run with more samples and on many more sites on each sample.

Performing preliminary elemental analysis that is limited to expected major elements, such as copper, iron, and silicon, would benefit initial classification of slag type. Clearly the use of visual identification is not optimum as illustrated by the fact that visual identification of the “cupreous slag samples” from Fröjel actually contained fairly low amounts of copper. Identification of slag type by visual clues seems only effective when very clear color indications are in the samples. For example, reddish slag is nearly always ferrous while greenish slag is nearly always cupreous. Slag without these distinct colorations is much more difficult to identify visually. Identification of whether a slag is from smelting or from casting (a smelting slag versus a crucible slag) is also very difficult from visual analysis.

Comparisons of the Three Sites

There is evidence of ferrous slag at both Fröjel and Långume. However there is some question on whether iron working was done at Långume. Tradition supports the idea of iron working farmers, as do at least two of the slag samples. It is likely that at Långume iron smelting was done only occasionally to produce needed goods or to use as a minor income supplement, with the possibility of selling unrefined ore as well as smelted material. At Fröjel, however, the smelting seems to be much more consistent and more successful. This is likely due to the fact that as a trading port there was likely a specialized smith at Fröjel who worked with iron all year round.

Little Hultungs is the only site with evidence for working bronze, and has no evidence of working iron. This may be due in part to its nature as a splinter off of the main Hultungs farmstead. Perhaps all of the ironworking that needed to be done was accomplished by the main Hultungs farmstead, so at Little Hultungs they instead recycled old bronze objects. This would have likely been done at least in part to supplement income, but could also have been for personal use. Many of the farmers on Gotland also engaged in trade, as there were only a few trading ports on the island. It is believed that many farmers went on occasional trade voyages. This could also explain the bronze working being done at Little Hultungs.

There are some clear conclusions that can be made and some others considered from this data. There is very clear evidence of successful smelting at Fröjel, probably by an experienced smith. There is some evidence of potential iron smelting at Långume, but also the possibility exists that only ore was gathered, with the probable intention of selling or trading to others. At Little Hultungs there is evidence of recycling bronze objects into new objects. There is no current evidence of iron smelting or ore gathering at Little Hultungs. The lack of iron working at Little Hultungs could be due to difficulty of getting iron ore, or possibly from not having a need of it due to the possibility of obtaining iron objects from the primary Hultungs farmstead.

Trade specialization was relatively unknown during the Viking Age in Scandinavia, but begins developing during the early medieval period. This transition may be seen in the fact that there appears to be specialized metal working going on at Fröjel and possibly at Little Hultungs as well, while the tradition of non-specialized work can be seen at Långume. Specialized metal working seems to be clearly indicated at Fröjel. At Little Hultungs, the only site with evidence of bronze working, some specialization may have been happening alongside farming. This would likely be mostly practiced during the winter, when the demands of farming were lessened. At Långume, evidence seems to indicate metalworking as primarily a side occurrence at the farmstead. Smelting seems to have been only marginally successful there. Like at Little Hultungs, most of the metal working would likely have been done during the winter.

Chapter 7: Epilogue

In many theses issues often come up that are unexpected and my thesis was no exception. Since I had taken on a project that was exploring both a place and materials that few archaeologists have investigated I certainly ran into my share of issues. These few pages are a look back at what should have been done differently and hopefully how to avoid some of the problems I faced should someone choose to build off of this project. First, the slag samples should not have been assumed to be from a certain type of material without some analysis. I was quick to accept the preliminary categories of cupreous or ferrous. These categories ultimately proved to be useless for the final analysis, and the data had to be reorganized in order to be interpreted successfully.

Second, I did not spend enough time and effort to determine the availability of local analysis for the samples. I was quickly discouraged after contact with two local labs, and took one of their suggestions in contacting Activation Laboratories in Ontario. Local analysis was available and would have been significantly less expensive. This would have enabled the testing of more samples to provide a larger data set that may have been easier to interpret especially considering the considerable variability in the samples from Långume. Having a more thorough understanding of the options available for analysis before contacting the laboratory would also have been of great help. I was very quick to just accept Activation Laboratory's suggestions on which types of analysis to use on my samples.

X-Ray Fluorescence (XRF) would have been a less costly and non-destructive method to use for elemental analysis of the samples. The TIMS (Thermal Ionization Mass Spectrometry) analysis was a very expensive route to take for lead isotope ratio analysis, ICP-MS would have been a reasonable technique to use for determination of lead isotope ratios.

If I had done a more thorough search of lead isotope ratio literature before submitting my samples for analysis, a great deal of cost could have been avoided. I did not realize that there was no data for sources of copper in Scandinavia until after the samples had been analyzed for lead isotope ratios. This factor, combined with the lack of elemental analysis of the lead isotope ratio samples, made the latter information completely useless. The lack of elemental analysis is also something I have learned about. I thought that the lead isotope analysis would include an elemental analysis of the samples. I was not clear in my communications with Activation Laboratories to make certain that this was preformed. In general the lesson learned is that in some instances an extra level of research is necessary before beginning analysis to make sure there is adequate comparative data and clarity in communication when contracting with labs is crucial.

Research on metal smelting and working on Gotland is still something that can be pursued further with certain considerations. To examine sources of copper for the island, Scandinavian copper sources need to be analyzed by lead isotope ratio analysis to provide a comparison point. Something else that would help ensure the success of this type of analysis would be to perform a preliminary analysis of the cupreous samples to avoid samples with significant amounts of lead and to ensure that the samples analyzed are in fact cupreous.

A different approach to continuing metal working research on Gotland would be to try and examine where sources of bog iron are on the island and compare the composition of these ores to samples found in different places on Gotland. This would be for looking at the smaller picture of ore movement on the island. Also to trying to locate and excavate rural workshops would also add to the understanding of rural metal working. This may reveal smelting furnaces or molds for casting which would provide a great deal of insight into rural metal working on Gotland. Samples collected from slag dumps or hearths may also more clearly represent smelting slag regardless of composition. Examining these types of samples may reveal more about smelting practices on Gotland.

Overall this research was not as successful as I would have liked. There were many problems which arose primarily from an attempt to proceed as quickly as possible instead of taking time to fully understand what I was trying to accomplish. I believe that this rush ultimately delayed the research further. I believe that the conclusions made would have been reached much more quickly with a better plan of research. Problems aside, it was still ultimately gratifying to try, and partially succeed, in exploring a question that has been largely unexplored and to work in a part of the world I am passionate about. I really appreciate everyone that helped me with this project and I believe that I have grown substantially as a researcher through this process.

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Appendix A: List of Figures with Citations

- Figure 2.1
  - Map obtained at Wikimedia Commons: http://commons.wikimedia.org/wiki/File:Baltic_Sea_map.png
  - Created by Norman Einstein, May 25, 2006, modified by author, accessed Feb. 9, 2011
- Figure 2.2
  - Photo by author, taken July 5, 2009, at Gnisvärd
- Figure 2.3
  - Photo by author, taken July 21, 2009, at Little Hultungs Excavation
- Figure 2.4
  - Photo by author, taken July 4, 2009, at the Bunge Museum
- Figure 2.5
  - Map of Viking Trade Routes, from 1000 Petals http://1000petals.wordpress.com/2008/08/
  - Accessed Feb. 9, 2011, some labels added by author
- Figure 2.6
  - Map obtained at Wikimedia Commons: http://commons.wikimedia.org/wiki/File:Sweden_Gotland_location_map_modified.svg
  - author: Erik Frohne, NordNordWest. May 9, 2009, retrieved October 4, 2010 Text added to map by author.
- Figure 2.7
  - Photo by author, taken July 3, 2009, at the Bro Church, Gotland
- Figure 2.8
  - Same map as figure 6, different text by author
- Figure 2.9
  - Phosphate Mapping Map from Little Hultungs,
- Figure 2.10
  - Excavation map of Little Hultungs, showing major features, and possible house outline
- Figure 2.11
  - Photo by Dan Carlsson, from Fjäle of a medieval house dating from 1250-1360
- Figure 2.12
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- Table 4.1
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- Table 5.1
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- Table 5.2
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Table 5.4

Lead Isotope Ratios Reported by Activation Laboratories, see Appendix C, page 86 for report

Table 5.5

Lead Isotope Ratios for Provenancing, calculated by author

Table 5.6

Analysis of Lead Isotope Data from OXALID (Stos-Gale 2009: 209)

Table 5.7

Average Lead Isotope Ratios for Various Locations (Höppner 2005, Klein 2009, Stos-Gale 2010)

Figure 6.1

Photo by author, taken July 30, 2009, at Little Hultungs Excavation

Figure 6.2

Photography of a Reproduced Viking Brooch obtained at: http://www.jorgencraft.com/files/penannular-brooch-z-7s.jpg, accessed October 24, 2011.

Appendix B: Elemental Analysis Report

Quality Analysis ... **Actlabs** *Innovative Technologies*

Date Submitted: 03-Nov-10
Invoice No.: A10-7878
Invoice Date: 03-Mar-11
Your Reference: UNDERSTANDING A WAY OF L

University of Idaho
875 Perimeter Drive
Moscow ID 83844-1110

ATTN: Timothy Mace

CERTIFICATE OF ANALYSIS

24 Rock samples were submitted for analysis.

The following analytical packages were requested:	Code 10A Pb/Nd/Sr-TIMS Pb, Nd, Sr TIMS Code 4LITHO (11+) Major Elements Fusion ICP(WRA)/Trace Elements Fusion ICP/MS(WRA4B2) Code MIT Pkg INAA(INAAGEO)/Total Digestion ICP(TOTAL)
REPORT **A10-7878**	

This report may be reproduced without our consent. If only selected portions of the report are reproduced, permission must be obtained. If no instructions were given at time of sample submittal regarding excess material, it will be discarded within 90 days of this report. Our liability is limited solely to the analytical cost of these analyses. Test results are representative only of material submitted for analysis.

Notes:
We recommend using option 4B1 for accurate levels of the base metals Cu, Pb, Zn, Ni and Ag. Option 4B-INAA for As, Sb, high W >100ppm, Cr >1000ppm and Sn >50ppm by Code 5D. Values for these elements provided by Fusion ICP/MS, are order of magnitude only and are provided for general information. Mineralized samples should have the Quant option selected or request assays for values which exceed the range of option 4B1. Total includes all elements in % oxide to the left of total.

Footnote: Missing data for some samples due to insufficient sample material.

CERTIFIED BY :

Emmanuel Eseme , Ph.D.
Quality Control

ISO/IEC 17025
SCC Accredited
LAB 266
LAB 266
Accrédité CCN

ACTIVATION LABORATORIES LTD.
1336 Sandhill Drive, Ancaster, Ontario Canada L9G 4V5 TELEPHONE +1.905.648.9611 or +1.888.228.5227 FAX +1.905.648.9613
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Activation Laboratories Ltd. Report: A10-7878 rev 2

Analyte Symbol	SiO2	Al2O3	Fe2O3(T)	MnO	MgO	CaO	Na2O	K2O	TiO2	P2O5	LOI	Total	Sc	Be	V	Ba	Sr	Y	Zr	Cr	Co	Ni	Cu	Zn
Unit Symbol	%	%	%	%	%	%	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	0.01	0.01	0.01	0.001	0.01	0.01	0.01	0.01	0.001	0.01		0.01	1	1	5	3	2	2	4	20	1	20	10	30
Analysis Method	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS
1	6.68	0.72	95.02	0.158	0.26	1.61	0.07	0.16	0.030	0.32	-6.17	98.86	1	< 1	7	21	24	< 2	12	< 20	129	80	130	< 30
2	70.53	11.12	5.69	0.084	2.05	3.76	0.95	3.20	0.472	0.45	1.10	99.40	10	2	57	418	144	23	134	150	11	< 20	50	70
3	73.51	8.84	5.29	0.113	1.41	4.50	1.24	3.22	0.349	0.31	0.50	99.29	7	2	56	371	114	24	128	30	9	< 20	80	80
4	70.57	6.28	14.21	0.099	0.65	4.08	0.88	1.98	0.301	0.16	-0.22	98.98	4	< 1	26	339	90	15	234	120	10	< 20	40	< 30
5	71.46	11.00	5.79	0.126	1.42	3.66	0.97	3.21	0.438	0.18	1.47	99.73	10	2	57	424	103	31	186	30	9	< 20	50	80
6	29.45	4.68	39.05	0.215	3.91	20.26	0.45	1.33	0.240	0.50	-0.25	99.83	5	< 1	16	302	228	18	110	< 20	7	< 20	60	30
7	15.50	2.57	75.88	0.152	0.69	6.08	0.20	0.61	0.140	0.42	-2.91	99.34	3	< 1	31	124	49	2	43	30	15	< 20	60	40
8	30.01	3.95	53.17	0.098	1.27	10.57	0.39	1.17	0.177	0.25	-1.33	99.73	3	< 1	20	162	65	7	84	< 20	2	< 20	40	< 30
9	53.62	3.93	26.29	0.128	1.06	12.07	0.73	1.76	0.124	0.38	-0.27	99.82	2	< 1	12	261	115	9	76	< 20	10	< 20	40	< 30
10	22.47	2.05	66.47	0.112	0.77	7.50	0.29	0.82	0.080	0.37	-1.60	99.33	2	< 1	18	114	49	< 2	42	< 20	8	< 20	50	40
11	44.80	8.17	4.22	0.056	1.28	7.89	0.74	2.15	0.407	0.13	28.85	98.70	8	2	70	385	117	20	187	230	10	40	40	90
12	6.79	0.68	94.64	0.100	0.37	2.82	0.09	0.25	0.029	0.19	-5.43	100.5	1	< 1	8	35	17	< 2	17	< 20	24	< 20	50	70
15	64.56	7.45	8.56	0.176	2.02	12.45	1.08	2.92	0.257	0.25	0.37	100.1	5	1	30	391	220	18	137	30	23	< 20	4040	< 30
16	67.99	11.11	7.74	0.074	1.86	5.58	1.26	3.81	0.496	0.16	0.33	100.4	10	2	71	464	148	25	201	40	8	< 20	2660	80
17	73.98	8.83	4.54	0.078	0.91	2.32	0.96	2.83	0.406	0.22	0.70	95.79	8	1	46	418	95	28	236	40	22	60	> 10000	> 10000
18	66.36	8.58	3.19	0.099	1.29	3.16	1.05	2.63	0.383	0.15	0.41	87.30	8	1	57	406	105	24	223	40	8	70	> 10000	120
19	66.25	10.15	4.40	0.083	1.78	8.27	1.01	2.84	0.474	0.19	0.44	95.88	9	2	60	425	152	26	236	40	24	70		90
20	0.39	0.08	0.29	0.005	0.02	0.26	0.02	< 0.01	0.003	0.13	-8.24	< 0.01	7	< 1	69	7	< 2	< 2	4	< 20	71	240		80
21	4.57	0.24	2.05	0.064	0.11	0.36	0.03	0.02	1.803	0.11	10.54	19.89	7	< 1	92	16	6	2	157	30	65	200		100
22																								
23	5.34	0.33	2.20	0.065	0.23	0.64	0.04	0.03	1.418	0.13	-7.36	3.06	8	< 1	87	19	18	2	85	40	42	230		40
24	7.05	0.43	2.29	0.066	0.29	0.92	0.06	0.04	1.694	0.24	-2.52	10.57	7	< 1	95	24	18	2	168	40	66	220		220

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Analyte Symbol	Ga	Ge	As	Rb	Nb	Mo	Ag	In	Sn	Sb	Cs	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb
Unit Symbol	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	1	1	5	2	1	2	0.5	0.2	1	0.5	0.5	0.1	0.1	0.05	0.1	0.1	0.05	0.1	0.1	0.1	0.1	0.1	0.05	0.1
Analysis Method	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS
1	5	< 1	46	3	< 1	10	< 0.5	< 0.2	39	3.4	< 0.5	3.7	6.4	0.82	2.8	0.7	0.13	0.6	< 0.1	0.5	0.1	0.3	< 0.05	0.3
2	14	1	< 5	112	9	11	0.6	< 0.2	91	< 0.5	4.2	45.8	77.3	10.5	35.2	7.0	1.16	5.9	0.8	4.8	1.0	2.8	0.42	2.7
3	11	1	< 5	99	7	2	0.6	0.8	1400	0.7	2.7	33.7	57.9	7.95	26.5	5.3	1.06	4.8	0.7	4.4	0.9	2.8	0.44	2.7
4	6	1	< 5	65	6	13	0.9	< 0.2	4	< 0.5	1.5	27.2	47.3	5.86	19.4	3.8	0.71	3.5	0.5	3.2	0.7	2.1	0.32	2.1
5	13	1	< 5	115	9	< 2	0.6	< 0.2	4	0.8	4.1	46.6	69.9	10.8	37.0	7.4	1.48	6.6	1.0	5.6	1.1	3.4	0.51	3.1
6	5	< 1	54	30	5	4	< 0.5	< 0.2	2	5.5	0.8	29.1	51.8	7.39	24.9	5.0	1.05	4.7	0.7	3.9	0.8	2.3	0.31	1.9
7	7	< 1	< 5	17	3	21	< 0.5	0.5	931	< 0.5	< 0.5	10.3	19.5	2.45	8.5	1.7	0.28	1.4	0.2	1.2	0.3	0.8	0.12	0.7
8	14	2	< 5	35	4	< 2	< 0.5	< 0.2	12	7.5	0.8	15.2	24.5	3.49	11.5	2.4	0.45	2.2	0.3	2.0	0.4	1.2	0.19	1.2
9	6	< 1	< 5	39	3	4	< 0.5	< 0.2	5	< 0.5	0.6	12.6	24.8	3.03	10.4	2.0	0.43	1.8	0.3	1.8	0.4	1.0	0.16	1.0
10	12	< 1	5	20	2	3	< 0.5	< 0.2	16	0.7	< 0.5	7.6	12.4	1.72	5.9	1.2	0.23	1.1	0.2	1.0	0.2	0.6	0.09	0.6
11	13	2	7	104	12	20	0.7	< 0.2	5	1.9	5.5	39.3	79.4	9.34	34.9	6.8	1.17	5.6	0.9	5.0	1.0	2.9	0.46	3.0
12	10	< 1	< 5	4	< 1	10	< 0.5	< 0.2	5	4.1	< 0.5	2.8	4.4	0.59	1.9	0.4	0.07	0.4	< 0.1	0.3	< 0.1	0.2	< 0.05	0.2
15	6	< 1	45	65	6	< 2	1.4	0.8	75	16.0	1.6	29.2	47.2	6.57	21.7	4.1	0.87	3.8	0.6	3.3	0.7	2.0	0.30	1.9
16	13	1	25	110	10	< 2	2.6	0.3	580	10.1	3.9	40.9	68.3	9.20	30.9	6.1	1.21	5.4	0.8	4.8	1.0	3.0	0.47	2.9
17	12	2	26	95	8	< 2	7.8	1.9	1520	161	2.8	34.0	64.0	8.27	30.6	6.2	1.21	5.3	0.9	4.8	1.0	2.9	0.44	2.9
18	11	1	336	84	7	< 2	82.8	0.8	125	609	2.7	30.0	63.9	7.27	26.5	5.4	1.05	4.5	0.8	4.1	0.8	2.4	0.38	2.4
19	11	1	56	98	10	< 2	12.7	8.2	200	589	3.7	41.8	69.8	9.58	32.1	6.5	1.23	5.7	0.8	5.0	1.0	3.0	0.47	3.0
20	< 1	< 1	1380	< 2	< 1	< 2		55.2		1610	< 0.5	1.0	0.6	0.12	0.4	< 0.1	< 0.05	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.05	< 0.1
21	< 1	< 1	1240	< 2	5	< 2	71.0	42.9		1880	< 0.5	5.0	9.8	0.96	3.3	0.6	0.15	0.5	< 0.1	0.4	< 0.1	0.2	< 0.05	0.3
22																								
23	< 1	< 1	1880	< 2	2	< 2	87.4	32.6		1120	< 0.5	12.9	26.0	2.66	9.2	1.4	0.34	0.9	0.1	0.6	0.1	0.3	0.05	0.4
24	< 1	< 1	1800	< 2	4	< 2	82.9	51.5		1890	< 0.5	8.2	16.6	1.73	5.9	1.0	0.25	0.7	0.1	0.6	0.1	0.3	< 0.05	0.4

Analyte Symbol	Lu	Hf	Ta	W	Tl	Pb	Bi	Th	U	Au	Ag	As	Ba	Br	Co	Cr	Cs	Hf	Hg	Ir	Mo	Na	Rb	Sb
Unit Symbol	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	ppm	%
Detection Limit	0.04	0.2	0.1	1	0.1	5	0.4	0.1	0.1	0.1	0.001	0.001	50	0.5	1	5	1	1	1	5	1	0.01	15	0.001
Analysis Method	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA
1	0.04	0.4	< 0.1	18	< 0.1	< 5	< 0.4	0.7	0.3															
2	0.42	3.5	0.8	3	0.3	11	< 0.4	13.3	2.3															
3	0.45	3.2	0.5	13	< 0.1	21	< 0.4	7.9	1.8															
4	0.35	5.5	0.5	< 1	< 0.1	6	< 0.4	6.2	1.8															
5	0.52	4.5	0.7	< 1	0.4	26	< 0.4	10.4	2.2															
6	0.31	2.7	0.3	< 1	< 0.1	7	< 0.4	5.3	1.2															
7	0.12	1.2	0.2	< 1	< 0.1	24	< 0.4	2.5	1.0															
8	0.19	2.1	0.3	12	< 0.1	8	< 0.4	3.7	1.1															
9	0.17	1.7	0.2	5	< 0.1	< 5	< 0.4	2.9	1.1															
10	0.10	1.1	0.1	19	< 0.1	7	< 0.4	1.7	0.6															
11	0.49	7.0	1.1	9	< 0.1	39	< 0.4	12.5	3.0															
12	< 0.04	0.4	< 0.1	19	< 0.1	6	< 0.4	0.5	0.2															
15	0.30	3.2	0.4	< 1	< 0.1	171	< 0.4	6.8	1.5	< 0.1	< 0.001	0.006	470	15.6	34	51	4	4	< 1	< 5	< 1	0.85	73	0.002
16	0.46	4.7	0.8	< 1	0.1	504	0.7	9.6	2.1	< 0.1	< 0.001	0.004	590	17.8	14	65	5	7	< 1	< 5	< 1	0.99	144	0.001
17	0.45	5.1	0.7	< 1	0.3	5110	14.0	10.2	2.2	< 0.1	< 0.001	0.003	550	24.3	25	50	5	7	< 1	< 5	< 1	0.82	111	0.010
18	0.37	4.9	0.6	< 1	0.5	4070	51.2	9.2	1.8	0.4	0.008	0.031	140	12.2	11	54	< 1	8	< 1	< 5	< 1	0.88	94	0.036
19	0.47	5.5	0.8	< 1	0.3	4880	3.8	9.9	2.2	< 0.1	0.001	0.007	550	52.2	35	66	4	7	< 1	< 5	< 1	0.85	105	0.053
20	< 0.04	< 0.2	< 0.1	< 1	< 0.1		313	< 0.1	0.2	8.1	0.066	0.352	< 50	292	107	< 5	< 1	< 1	< 1	< 5	< 1	0.06	< 15	0.247
21	0.07	3.2	0.7	< 1	< 0.1		196	1.5	0.5	6.8	0.045	0.254	< 50	113	116	53	< 1	6	< 1	< 5	< 1	0.07	< 15	0.164
22										65.8	0.080	0.144	< 50	157	8	< 5	< 1	< 1	< 1	< 5	< 1	0.07	< 15	0.097
23	0.06	1.3	0.3	< 1	< 0.1	8710	127	1.8	0.5	4.3	0.043	0.313	< 50	356	60	< 5	< 1	1	< 1	< 5	< 1	0.08	< 15	0.187
24	0.09	3.5	0.3	< 1	< 0.1		242	1.8	0.6	7.5	0.058	0.271	< 50	402	90	34	< 1	7	< 1	< 5	< 1	0.10	< 15	0.199

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Analyte Symbol	Sc	Se	Sn	Sr	Ta	Th	U	W	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Mass	Cu	Pb
Unit Symbol	ppm	ppm	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	g	%	%
Detection Limit	0.1	3	0.01	0.05	0.5	0.2	0.5	1	0.5	3	5	0.1	0.2	0.5	0.2	0.05		0.001	0.003
Analysis Method	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	INAA	ICP-OES	ICP-OES
1																			
2																			
3																			
4																			
5																			
6																			
7																			
8																			
9																			
10																			
11																			
12																			
15	6.6	< 3	< 0.01	< 0.05	< 0.5	8.8	< 0.5	2	35.0	66	19	5.1	1.4	< 0.5	2.6	0.12	1.01		
16	11.5	< 3	< 0.01	< 0.05	< 0.5	13.2	2.2	< 1	48.6	90	28	6.9	1.6	< 0.5	3.6	0.42	1.00		
17	9.5	< 3		< 0.05	< 0.5	11.2	3.4	< 1	46.2	84	30	6.9	1.6	< 0.5	3.8	0.43	1.00		
18	8.7	< 3	< 0.01	< 0.05	< 0.5	11.3	3.4	< 1	40.2	76	31	6.1	1.2	< 0.5	3.5	0.46	0.985		
19	10.9	< 3	< 0.01	< 0.05	< 0.5	12.0	< 0.5	< 1	47.9	92	39	7.4	1.6	< 0.5	3.8	0.42	1.01	2.54	
20	0.6	< 3	15.6	< 0.05	< 0.5	< 0.2	< 0.5	< 1	< 0.5	< 3	< 5	< 0.1	< 0.2	< 0.5	< 0.2	< 0.05	1.01	68.5	8.36
21	3.5	< 3	12.1	< 0.05	< 0.5	< 0.2	< 0.5	< 1	9.3	24	< 5	0.7	< 0.2	< 0.5	< 0.2	< 0.05	1.00	61.6	5.32
22	1.4	< 3	< 0.01	< 0.05	< 0.5	< 0.2	< 0.5	< 1	3.1	15	< 5	0.3	< 0.2	< 0.5	< 0.2	< 0.05	1.01		
23	3.2	< 3	14.3	< 0.05	4.7	4.7	< 0.5	< 1	17.8	30	< 5	1.6	< 0.2	< 0.5	< 0.2	< 0.05	1.00	59.1	
24	3.2	< 3	12.9	< 0.05	< 0.5	< 0.2	< 0.5	< 1	12.3	< 3	< 5	1.1	< 0.2	< 0.5	< 0.2	< 0.05	1.01	51.2	8.39

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Quality Control

Analyte Symbol	SiO2	Al2O3	Fe2O3(T)	MnO	MgO	CaO	Na2O	K2O	TiO2	P2O5	Sc	Be	V	Ba	Sr	Y	Zr	Cr	Co	Ni	Cu	Zn	Ga	Ge
Unit Symbol	%	%	%	%	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	0.01	0.01	0.01	0.001	0.01	0.01	0.01	0.01	0.001	0.01	1	1	5	3	2	2	4	20	1	20	10	30	1	1
Analysis Method	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS
WMG-1 Meas																		750	203	2840	6160	190	10	
WMG-1 Cert																		770	200	2700	5900	110	10.3	
NIST 694 Meas	11.55	1.88	0.75	0.011	0.36	43.84	0.86	0.55	0.119	30.21			1684											
NIST 694 Cert	11.2	1.80	0.790	0.0116	0.330	43.6	0.860	0.510	0.110	30.2			1740											
NIST 694 Meas	11.56	1.91	0.75	0.012	0.35	43.68	0.87	0.55	0.119	30.26			1688											
NIST 694 Cert	11.2	1.80	0.790	0.0116	0.330	43.6	0.860	0.510	0.110	30.2			1740											
DNC-1 Meas	47.15	18.38	9.84	0.147	10.15	11.21	1.88	0.23	0.479	0.06	31		154	104	140	15	38	280	58	250	100	90		
DNC-1 Cert	47.15	18.34	9.97	0.150	10.13	11.49	1.890	0.234	0.480	0.070	31		148.0	118	144.0	18.0	38	270.0	57.0	247	100.0	70.0		
DNC-1 Meas	47.01	18.40	9.91	0.148	10.07	11.30	1.90	0.23	0.479	0.06	31		155	104	142	16	36	290	64	300	100	100		
DNC-1 Cert	47.15	18.34	9.97	0.150	10.13	11.49	1.890	0.234	0.480	0.070	31		148.0	118	144.0	18.0	38	270.0	57.0	247	100.0	70.0		
GBW 07113 Meas	71.67	12.70	3.27	0.142	0.14	0.58	2.45	5.41	0.285	0.03	5	4	< 5	492	39	46	413							
GBW 07113 Cert	72.8	13.0	3.21	0.140	0.160	0.590	2.57	5.43	0.300	0.0500	5.00	4.00	5.00	506	43.0	43.0	403							
GBW 07113 Meas	72.70	12.82	3.29	0.143	0.14	0.59	2.50	5.47	0.291	0.03	5	4	< 5	496	39	45	413							
GBW 07113 Cert	72.8	13.0	3.21	0.140	0.160	0.590	2.57	5.43	0.300	0.0500	5.00	4.00	5.00	506	43.0	43.0	403							
KC-1A Meas																								
KC-1A Cert																								
CZN-3 Meas																								
CZN-3 Cert																								
CCU-1C Meas																								
CCU-1C Cert																								
LKSD-3 Meas																		80	31	50	40			
LKSD-3 Cert																		87.0	30.0	47.0	35.0			
LKSD-3 Meas																		90	35	60	40			
LKSD-3 Cert																		87.0	30.0	47.0	35.0			
NIST 1633b Meas	48.93	28.39	11.17	0.020	0.78	2.12	0.26	2.31	1.306	0.52	41		306	698	1041									
NIST 1633b Cert	49.2	28.4	11.1	0.0200	0.800	2.11	0.270	2.35	1.32	0.530	41.0		296	709	1040									
NIST 1633b Meas	48.18	27.82	10.93	0.020	0.76	2.13	0.26	2.28	1.286	0.52	40		303	693	1025									
NIST 1633b Cert	49.2	28.4	11.1	0.0200	0.800	2.11	0.270	2.35	1.32	0.530	41.0		296	709	1040									
TDB-1 Meas																		240		100	340	180		
TDB-1 Cert																		251		92	323	155		
BCS 183/4 Meas																								
BCS 183/4 Cert																								
BCS 207/2 Meas																								
BCS 207/2 Cert																								
NBS 158A Meas																								
NBS 158A Cert																								
CPB-1 Meas																								
CPB-1 Cert																								
PTC-1a Meas																								
PTC-1a Cert																								
W-2a Meas	52.98	15.26	10.81	0.168	6.39	10.85	2.20	0.62	1.099	0.14	36	< 1	276	171	193	20	93	90	43	70		80	17	2
W-2a Cert	52.4	15.4	10.7	0.163	6.37	10.9	2.14	0.626	1.06	0.130	36.0	1.30	262	182	190	24.0	94.0	92.0	43.0	70.0		80.0	17.0	1.00
W-2a Meas																					110			
W-2a Cert																					110			
W-2a Meas																		90	43	60		90	17	2
W-2a Cert																		92.0	43.0	70.0		80.0	17.0	1.00
OREAS 14P Meas																								
OREAS 14P Cert																								
SY-4 Meas	50.31	20.50	6.24	0.108	0.51	7.94	7.01	1.67	0.292	0.14	1	3	< 5	345	1204	119	610							
SY-4 Cert	49.9	20.69	6.21	0.108	0.54	8.05	7.10	1.66	0.287	0.131	1.1	2.6	8.0	340	1191	119	517							
SY-4 Meas	49.70	20.86	6.27	0.107	0.51	7.95	6.98	1.67	0.288	0.13	1	3	< 5	343	1220	112	564							
SY-4 Cert	49.9	20.69	6.21	0.108	0.54	8.05	7.10	1.66	0.287	0.131	1.1	2.6	8.0	340	1191	119	517							
CTA-AC-1 Meas																			< 1		60	30		
CTA-AC-1 Cert																			2.72		54.0	38.0		
CTA-AC-1 Meas																								
CTA-AC-1 Cert																								
CTA-AC-1 Meas																			< 1		60	< 30		

Quality Control

Analyte Symbol	SiO2	Al2O3	Fe2O3(T)	MnO	MgO	CaO	Na2O	K2O	TiO2	P2O5	Sc	Be	V	Ba	Sr	Y	Zr	Cr	Co	Ni	Cu	Zn	Ga	Ge
Unit Symbol	%	%	%	%	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	0.01	0.01	0.01	0.001	0.01	0.01	0.01	0.01	0.001	0.01	1	1	5	3	2	2	4	20	1	20	10	30	1	1
Analysis Method	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS
CTA-AC-1 Cert																			2.72		54.0	38.0		
BIR-1a Meas	48.13	15.58	11.32	0.171	9.55	13.18	1.79	0.02	0.968	0.02	43	< 1	335	7	105	15	18	380	52	160	130	70	15	
BIR-1a Cert	47.96	15.50	11.30	0.175	9.700	13.30	1.82	0.030	0.96	0.021	44	0.58	310	6	110	16	18	370	52	170	125	70	16	
BIR-1a Meas	47.32	15.42	11.14	0.170	9.49	13.19	1.79	0.02	0.969	0.02	44	< 1	335	7	105	13	16	430	61	190	140	80	17	
BIR-1a Cert	47.96	15.50	11.30	0.175	9.700	13.30	1.82	0.030	0.96	0.021	44	0.58	310	6	110	16	18	370	52	170	125	70	16	
NCS DC86312 Meas																								
NCS DC86312 Cert																								
NCS DC86312 Meas																								
NCS DC86312 Cert																								
NCS DC70014 Meas																			26	70	2600	7400	25	
NCS DC70014 Cert																			26.2	70.9	2600.00	7400.00	25.2	
NCS DC70014 Meas																			27	70	2730	7400	24	
NCS DC70014 Cert																			26.2	70.9	2600.00	7400.00	25.2	
NCS DC70009 (GBW07241) Meas																		30	4	< 20	970	110	17	11
NCS DC70009 (GBW07241) Cert																		30	3.7	2.8	960.000	100.000	16.5	11.2
NCS DC70009 (GBW07241) Meas																		30	4	< 20	960	100	16	11
NCS DC70009 (GBW07241) Cert																		30	3.7	2.8	960.000	100.000	16.5	11.2
MP-1b Meas																								
MP-1b Cert																								
OREAS 100a (Fusion) Meas																			17		170			
OREAS 100a (Fusion) Cert																			18.1		169			
OREAS 100a (Fusion) Meas																			17		170			
OREAS 100a (Fusion) Cert																			18.1		169			
OREAS 101a (Fusion) Meas																			48		430			
OREAS 101a (Fusion) Cert																			48.8		434			
OREAS 101a (Fusion) Meas																			50		440			
OREAS 101a (Fusion) Cert																			48.8		434			
OREAS 101b (Fusion) Meas																			44	< 20	420			
OREAS 101b (Fusion) Cert																			47	9	416			
JR-1 Meas																		< 20	1	< 20	< 10	40	17	3
JR-1 Cert																		2.83	0.83	1.67	2.68	30.6	16.1	1.88
JR-1 Meas																								
JR-1 Cert																								
JR-1 Meas																		< 20	1	< 20	< 10	40	17	3
JR-1 Cert																		2.83	0.83	1.67	2.68	30.6	16.1	1.88
SARM 3 Meas																								
SARM 3 Cert																								
OREAS 13b (4-Acid) Meas																								
OREAS 13b (4-Acid) Cert																								
DMMAS 111 Meas																								
DMMAS 111 Cert																								
11 Orig	44.46	8.10	4.20	0.056	1.28	7.84	0.74	2.13	0.404	0.13	8	2	86	380	115	20	182	230	10	40	40	80	13	2
11 Dup	45.13	8.24	4.25	0.056	1.29	7.94	0.75	2.17	0.410	0.13	8	2	54	389	118	21	193	230	10	40	40	90	14	2
Method Blank Method Blank																		< 20	< 1	< 20	< 10	< 30	< 1	< 1

Quality Control

Analyte Symbol	SiO2	Al2O3	Fe2O3(T)	MnO	MgO	CaO	Na2O	K2O	TiO2	P2O5	Sc	Be	V	Ba	Sr	Y	Zr	Cr	Co	Ni	Cu	Zn	Ga	Ge
Unit Symbol	%	%	%	%	%	%	%	%	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Detection Limit	0.01	0.01	0.01	0.001	0.01	0.01	0.01	0.01	0.001	0.01	1	1	5	3	2	2	4	20	1	20	10	30	1	1
Analysis Method	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-ICP	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS
Method Blank Method Blank																		< 20	< 1	< 20	< 10	< 30	< 1	< 1
Method Blank Method Blank																								

Quality Control

Analyte Symbol	As	Rb	Nb	Mo	Ag	Sn	Sb	La	Ce	Pr	Eu	Tb	Dy	Ho	Tm	Yb	Lu	W	Tl	Pb	Au	Ag	As	Mo
Unit Symbol	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	ppm
Detection Limit	5	2	1	2	0.5	1	0.5	0.1	0.1	0.05	0.05	0.1	0.1	0.1	0.05	0.1	0.04	1	0.1	5	0.1	0.001	0.001	1
Analysis Method	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	INAA	INAA	INAA	INAA
CTA-AC-1 Cert								2176	3326		46.7	13.9				11.4	1.08							
BIR-1a Meas	< 5		< 1					0.7	2.0		0.52					1.7				< 5				
BIR-1a Cert	0.44		0.6					0.63	1.9		0.55					1.7				3				
BIR-1a Meas	< 5		< 1					0.8	2.0		0.55					1.7				< 5				
BIR-1a Cert	0.44		0.6					0.63	1.9		0.55					1.7				3				
NCS DC86312 Meas								> 2000	177			34.1	183	35.7	14.4	87.3	12.1							
NCS DC86312 Cert								2360.000	190.000			34.6	183.00	35.70	15.1	87.79	11.96							
NCS DC86312 Meas								> 2000	180			34.3	183	35.7	14.4	87.4	12.0							
NCS DC86312 Cert								2360.000	190.000			34.6	183.00	35.70	15.1	87.79	11.96							
NCS DC70014 Meas				> 100	16.8		180	43.3	84.3	10.00	1.79	1.2	6.4	1.3	0.54	3.4	0.46			> 10000				
NCS DC70014 Cert				270.000	16.7		180.000	45.3	87.0	10.8	1.8	1.1	6.7	1.3	0.57	3.3	0.50			27200.00				
NCS DC70014 Meas				> 100	16.8		180	44.9	86.5	10.3	1.76	1.2	6.8	1.2	0.52	3.3	0.47			> 10000				
NCS DC70014 Cert				270.000	16.7		180.000	45.3	87.0	10.8	1.8	1.1	6.7	1.3	0.57	3.3	0.50			27200.00				
NCS DC70009 (GBW07241) Meas	70	503			1.7	> 1000	3.1	23.8	59.9	7.99		3.3	20.7	4.3	2.33	16.1	2.22	2200						
NCS DC70009 (GBW07241) Cert	69.9	500.00			1.8	1701.000	3.1	23.7	60.3	7.9		3.3	20.7	4.5	2.2	14.9	2.4	2200.00						
NCS DC70009 (GBW07241) Meas	70	502			1.6	1700		23.5	61.4	7.90		3.3	20.6	4.3	2.31	15.9	2.25	2200						
NCS DC70009 (GBW07241) Cert	69.9	500.00			1.8	1701.000		23.7	60.3	7.9		3.3	20.7	4.5	2.2	14.9	2.4	2200.00						
MP-1b Meas																						0.005	2.28	292
MP-1b Cert																						0.0	2.30	285
OREAS 100a (Fusion) Meas				23				260	462	46.6	3.59	3.7	22.8	4.9	2.39	15.3	2.14							
OREAS 100a (Fusion) Cert				24.1				260	463	47.1	3.71	3.80	23.2	4.81	2.31	14.9	2.26							
OREAS 100a (Fusion) Meas				21				258	475	45.4	3.61	3.7	22.6	4.9	2.36	15.3	2.13							
OREAS 100a (Fusion) Cert				24.1				260	463	47.1	3.71	3.80	23.2	4.81	2.31	14.9	2.26							
OREAS 101a (Fusion) Meas				21				798	1360	130	7.92	5.5	31.8	6.6	2.95	18.4	2.50							
OREAS 101a (Fusion) Cert				21.9				816	1396	134	8.06	5.92	33.3	6.46	2.90	17.5	2.66							
OREAS 101a (Fusion) Meas				19				802	1440	128	8.08	5.6	31.8	6.6	2.95	18.5	2.52							
OREAS 101a (Fusion) Cert				21.9				816	1396	134	8.06	5.92	33.3	6.46	2.90	17.5	2.66							
OREAS 101b (Fusion) Meas				20				768	1310	125	7.60	5.4	30.4	6.3	2.90	18.1	2.47							
OREAS 101b (Fusion) Cert				20.9				789	1331	127	7.77	5.37	32.1	6.34	2.66	17.6	2.58							
JR-1 Meas	16	251	14	3	< 0.5	3		20.3	48.4	6.07	0.29	1.1			0.71	4.8	0.69		1.6	19				
JR-1 Cert	16.3	257	15.2	3.25	0.031	2.86		19.7	47.2	5.58	0.30	1.01			0.67	4.55	0.71		1.56	19.3				
JR-1 Meas																								
JR-1 Cert																								
JR-1 Meas	16	250	15	2	< 0.5	3		20.1	49.0	5.98	0.28	1.1			0.71	4.8	0.70		1.6	19				
JR-1 Cert	16.3	257	15.2	3.25	0.031	2.86		19.7	47.2	5.58	0.30	1.01			0.67	4.55	0.71		1.56	19.3				
SARM 3 Meas			973																					
SARM 3 Cert			978																					
OREAS 13b (4-Acid) Meas																								
OREAS 13b (4-Acid) Cert																								
DMMAS 111 Meas	1510																							
DMMAS 111 Cert	1450																							
11 Orig	7	102	12	20	0.6	5	1.8	39.2	79.0	9.28	1.17	0.9	5.0	1.0	0.46	2.9	0.49	7	< 0.1	39				
11 Dup	8	106	13	21	0.7	5	2.0	39.4	79.8	9.39	1.17	0.9	5.1	1.0	0.47	3.0	0.49	10	< 0.1	40				
Method Blank Method Blank	< 5	< 2	< 1	< 2	< 0.5	< 1	< 0.5	< 0.1	< 0.1	< 0.05	< 0.05	< 0.1	< 0.1	< 0.1	< 0.05	< 0.1	< 0.04	< 1	< 0.1	< 5				

Quality Control

Analyte Symbol	As	Rb	Nb	Mo	Ag	Sn	Sb	La	Ce	Pr	Eu	Tb	Dy	Ho	Tm	Yb	Lu	W	Tl	Pb	Au	Ag	As	Mo
Unit Symbol	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	ppm
Detection Limit	5	2	1	2	0.5	1	0.5	0.1	0.1	0.05	0.05	0.1	0.1	0.1	0.05	0.1	0.04	1	0.1	5	0.1	0.001	0.001	1
Analysis Method	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	INAA	INAA	INAA	INAA
Method Blank Method Blank	< 5	< 2	< 1	< 2	< 0.5	< 1	< 0.5	< 0.1	< 0.1	< 0.05	< 0.05	< 0.1	< 0.1	< 0.1	< 0.05	< 0.1	< 0.04	< 1	< 0.1	< 5				
Method Blank Method Blank																								

Quality Control

Analyte Symbol	Sb	Sn	W	In	Cs	Bi	Nd	Sm	Gd	Er	Hf	Ta	Th	U	LOI	Total	Cu	Pb
Unit Symbol	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	%	%
Detection Limit	0.001	0.01	1	0.2	0.5	0.4	0.1	0.1	0.1	0.1	0.2	0.1	0.1	0.1		0.01	0.001	0.003
Analysis Method	INAA	INAA	INAA	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-ICP	FUS-ICP	ICP-OES	ICP-OES
WMG-1 Meas					< 0.5						1.4	0.4		0.8				
WMG-1 Cert					0.480						1.30	0.500		0.650				
NIST 694 Meas																		
NIST 694 Cert																		
NIST 694 Meas																		
NIST 694 Cert																		
DNC-1 Meas							5.3											
DNC-1 Cert							5.20											
DNC-1 Meas							5.2											
DNC-1 Cert							5.20											
GBW 07113 Meas																		
GBW 07113 Cert																		
GBW 07113 Meas																		
GBW 07113 Cert																		
KC-1A Meas																	0.624	2.24
KC-1A Cert																	0.629	2.24
CZN-3 Meas																	0.685	0.107
CZN-3 Cert																	0.685	0.113
CCU-1C Meas																	25.6	
CCU-1C Cert																	25.6	
LKSD-3 Meas					2.4		45.5	8.3				0.7	10.8	4.6				
LKSD-3 Cert					2.30		44.0	8.00				0.700	11.4	4.60				
LKSD-3 Meas					2.3		45.5	8.4				0.7	10.9	4.6				
LKSD-3 Cert					2.30		44.0	8.00				0.700	11.4	4.60				
NIST 1633b Meas																		
NIST 1633b Cert																		
NIST 1633b Meas																		
NIST 1633b Cert																		
TDB-1 Meas							24.0						3.1					
TDB-1 Cert							23						2.7					
BCS 183/4 Meas	0.230	7.28																
BCS 183/4 Cert	0.230	7.27																
BCS 207/2 Meas	0.100	9.88																
BCS 207/2 Cert	0.100	9.74																
NBS 158A Meas		0.99																
NBS 156A Cert		0.96																
CPB-1 Meas																		
CPB-1 Cert																		
PTC-1a Meas																	13.5	0.054
PTC-1a Cert																	13.51	0.05
W-2a Meas					0.9	< 0.4	12.9	3.3		2.2	2.4	0.5	2.4	0.6				
W-2a Cert					0.990	0.0300	13.0	3.30		2.50	2.60	0.500	2.40	0.530				
W-2a Meas																		
W-2a Cert																		
W-2a Meas					1.0	< 0.4	13.3	3.5		2.2	2.6	0.5	2.4	0.5				
W-2a Cert					0.990	0.0300	13.0	3.30		2.50	2.60	0.500	2.40	0.530				
OREAS 14P Meas																	0.950	
OREAS 14P Cert																	0.997	
SY-4 Meas																		
SY-4 Cert																		
SY-4 Meas																		
SY-4 Cert																		
CTA-AC-1 Meas							1150	170	128			2.8	23.9	4.3				
CTA-AC-1 Cert							1067	162	124			2.65	21.8	4.4				
CTA-AC-1 Meas											1.2							
CTA-AC-1 Cert											1.13							
CTA-AC-1 Meas							1110	163	125			2.9	23.8	4.3				

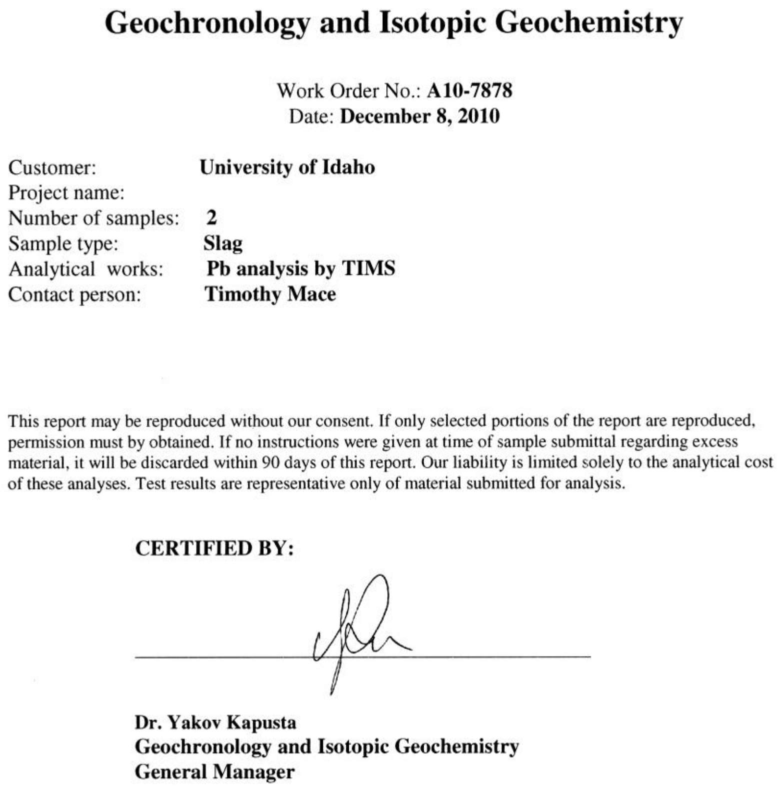
Quality Control

Analyte Symbol	Sb	Sn	W	In	Cs	Bi	Nd	Sm	Gd	Er	Hf	Ta	Th	U	LOI	Total	Cu	Pb
Unit Symbol	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	%	%
Detection Limit	0.001	0.01	1	0.2	0.5	0.4	0.1	0.1	0.1	0.1	0.2	0.1	0.1	0.1		0.01	0.001	0.003
Analysis Method	INAA	INAA	INAA	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-ICP	FUS-ICP	ICP-OES	ICP-OES
CTA-AC-1 Cert							1087	162	124			2.65	21.8	4.4				
BIR-1a Meas							2.7	1.2	2.0		0.5							
BIR-1a Cert							2.5	1.1	2.0		0.60							
BIR-1a Meas							2.6	1.2	1.8		0.6							
BIR-1a Cert							2.5	1.1	2.0		0.60							
NCS DC86312 Meas							1550		225	96.3			25.0					
NCS DC86312 Cert							1600.000		225.0	96.2			23.6					
NCS DC86312 Meas							1580		226	96.3			25.2					
NCS DC86312 Cert							1600.000		225.0	96.2			23.6					
NCS DC70014 Meas						80.3	37.2	7.8	6.9	3.5								
NCS DC70014 Cert						80.3	39.9	8.0	7.4	3.5								
NCS DC70014 Meas						80.3	40.2	7.6	7.2	3.3								
NCS DC70014 Cert						80.3	39.9	8.0	7.4	3.5								
NCS DC70009 (GBW07241) Meas				1.3	43.1		32.0	12.5	14.2	12.9			28.4					
NCS DC70009 (GBW07241) Cert				1.3	41		32.9	12.5	14.8	13.4			28.3					
NCS DC70009 (GBW07241) Meas				1.3	43.7		32.7	12.8	14.3	13.1			28.2					
NCS DC70009 (GBW07241) Cert				1.3	41		32.9	12.5	14.8	13.4			28.3					
MP-1b Meas	0.005	1.62	1110															
MP-1b Cert	0.0	1.610	1100.000															
OREAS 100a (Fusion) Meas							148	24.1	22.3	14.5			50.9	139				
OREAS 100a (Fusion) Cert							152	23.6	23.6	14.9			51.6	135				
OREAS 100a (Fusion) Meas							150	24.2	22.7	14.4			50.8	137				
OREAS 100a (Fusion) Cert							152	23.6	23.6	14.9			51.6	135				
OREAS 101a (Fusion) Meas							389	50.1		18.9			35.5	421				
OREAS 101a (Fusion) Cert							403	48.8		19.5			36.6	422				
OREAS 101a (Fusion) Meas							396	50.8		19.0			35.6	421				
OREAS 101a (Fusion) Cert							403	48.8		19.5			36.6	422				
OREAS 101b (Fusion) Meas							371	47.7		18.5			38.1	414				
OREAS 101b (Fusion) Cert							378	48		18.7			37.1	396				
JR-1 Meas				< 0.2	20.9	0.6	23.7	5.9	5.0		4.6	1.9	26.8	9.2				
JR-1 Cert				0.028	20.8	0.56	23.3	6.03	5.06		4.51	1.86	26.7	8.88				
JR-1 Meas										3.7								
JR-1 Cert										3.61								
JR-1 Meas				< 0.2	20.9	0.6	24.1	6.0	5.1		4.2	1.8	26.5	9.1				
JR-1 Cert				0.028	20.8	0.56	23.3	6.03	5.06		4.51	1.86	26.7	8.88				
SARM 3 Meas																		
SARM 3 Cert																		
OREAS 13b (4-Acid) Meas																	0.233	
OREAS 13b (4-Acid) Cert																	0	
DMMAS 111 Meas																		
DMMAS 111 Cert																		
11 Orig				< 0.2	5.4	< 0.4	34.6	6.7	5.5	2.9	6.8	1.1	12.4	3.0	28.85	98.18		
11 Dup				< 0.2	5.6	< 0.4	35.2	6.8	5.6	3.0	7.3	1.1	12.6	3.1	28.85	99.21		
Method Blank Method Blank				< 0.2	< 0.5	< 0.4	< 0.1	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1	< 0.1				

Quality Control

Analyte Symbol	Sb	Sn	W	In	Cs	Bi	Nd	Sm	Gd	Er	Hf	Ta	Th	U	LOI	Total	Cu	Pb
Unit Symbol	%	%	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	%	%	%	%
Detection Limit	0.001	0.01	1	0.2	0.5	0.4	0.1	0.1	0.1	0.1	0.2	0.1	0.1	0.1		0.01	0.001	0.003
Analysis Method	INAA	INAA	INAA	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-MS	FUS-ICP	FUS-ICP	ICP-OES	ICP-OES
Method Blank Method Blank				< 0.2	< 0.5	< 0.4	< 0.1	< 0.1	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1	< 0.1				
Method Blank Method Blank																	< 0.001	< 0.003

Appendix C: Lead (Pb) Isotope Analysis Report



Geochronology and Isotopic Geochemistry

Work Order No.: **A10-7878**
Date: **December 8, 2010**

Customer:	**University of Idaho**
Project name:	
Number of samples:	**2**
Sample type:	**Slag**
Analytical works:	**Pb analysis by TIMS**
Contact person:	**Timothy Mace**

CERTIFIED BY:

Dr. Yakov Kapusta
Geochronology and Isotopic Geochemistry
General Manager

I. Pb Isotope Analysis

Sample	$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$
13	18.497	15.721	38.718
14	17.591	15.633	37.630

(1) Measured ratios corrected for mass fractionation based on replicate analyses of NBS-981;
(2) precision of ratios is ≤0.1%