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Provenance studies of iron from Iceland

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In the late nineteen nineties research was done in order to try to determine the provenance of some iron objects from archaeological sites in Iceland, dated to the 9th - 11th centuries. Included in the studies were 112 remains: objects, lightly wrought blooms, ore and slag remains. The method mainly consisted of using the EPMA/WDA to analyse the chemical composition of smelting slag and slag inclusions in the objects and blooms.

Keywords: Iceland - objects - provenance - iron - slag

Introduction

Numerous archaeological sites with vast amounts of slag from iron production have been discovered in Iceland. Thus, these remains indicate extensive iron production in the country at some stage. As a result of research done in the 1920's and a systematic field survey in the 1970's and 1980's over 100 such smelting sites are now known (Nielsen, N, 1926; Friðriksson, Þ, catalogue over smelting sites; Friðriksson, Þ Á & Hermanns-Auðardóttir, M, 1992). The sites cluster in six main areas: 1. Þjórsárdalur; 2. Borgarfjörður/Mýrarsýsla; 3. Snæfellsnes; 4. Dalasýsla; 5. Fnjóskadalur, 6. Fljótsdalshérað (fig. 1).

None of the smelting sites has been thoroughly excavated, but charcoal samples of birch (*Betula pubescens*) collected from some of the sites and radiocarbon dated, give as early dating as the 9th century for the oldest sites, while the most recent ones are from the 13th century (S-Þjms. 27.01. 1989; Hermanns-Auðardóttir, M, 1998).

The great amount of slag remains might thus suggest that at least some of the objects dated to the 9th -13th centuries might have been produced from the bog-iron ore which was available in most areas of the country. As early as in the first half of the 20th century some scholars introduced their theories stating that Icelanders were self-sufficient with iron production during the first centuries of the settlement (Aðils, J, 1906; Jóhannesson, Þ,1943; Þórðarson, M, 1943). Þórðarson argued that at least some of the iron objects dating from the Viking Age and Medieval times were made of Icelandic bog-iron (Þórðarson, M, 1943, 263, 267, 268, 271). The scholars based their theories mainly on the literary evidence which indicates that iron smelting was practiced in Iceland from the time of settlement and was widespread until the 15th century AD, and even practiced in some areas until the 19th century (e.g. Landnámabók, 1843,72,117-118; Dipl. Isl. III, 1896,711; Dipl. Isl. IV, 1897, 689; Ólafsson, E and Pálsson, B, I, 1981, 29; Rafnar, J and Jónsson, Þ M (eds), 1936, 58).

I. Research into iron smelting

In the 1920's, the Danish geologist Niels Nielsen became the first scientist to research into iron smelting in Iceland. Although his interpretation of the furnaces he excavated at Belgsá in Fnjóskadalur (fig. 1) does not hold any more, his registration of the iron smelting sites has been useful for later research. Nielsen registered 46 bog-iron smelting sites, most of which he visited and a few of which he partly excavated. The remains from his research, mainly smelting slag and charcoal, are now stored at the National Museum in Reykjavik. The research was published in *Aarbøger for Nordisk Oldkyndighed og historie* in 1926 (Nielsen, N 1926).

According to Nielsen, the furnace at Belgsá was a rectangular "bowl furnace" lined with stones on the inside and insulated with clay. The sides were circa 40 x 50 cm in diameter and it was 45 cm deep. However, based on the following facts one might conclude that the furnace was a shaft-furnace with a slag-outlet. The ropy surface of the slag found at the site indicates that the slag has run from the furnace (fig. 2). Theories have been put forward that the structure of the phases of the slag from bowl- and shaft-furnaces differs. The structure in the shaft-furnace-slag forming a kind of skeletal structure (fig. 4) while the one from the bowl furnace has a globular structure. That theory, however, is not indisputable as it is known that the structures are different according to when during the smelting process the slag is formed. Finally one must keep in mind that the typical furnace in Europe during this period was the shaft furnace with or without a slag-outlet. One might thus conclude that the remains at Belgsá were the base of a shaft furnace with a slag-outlet, and not a bowl furnace, if a furnace at all

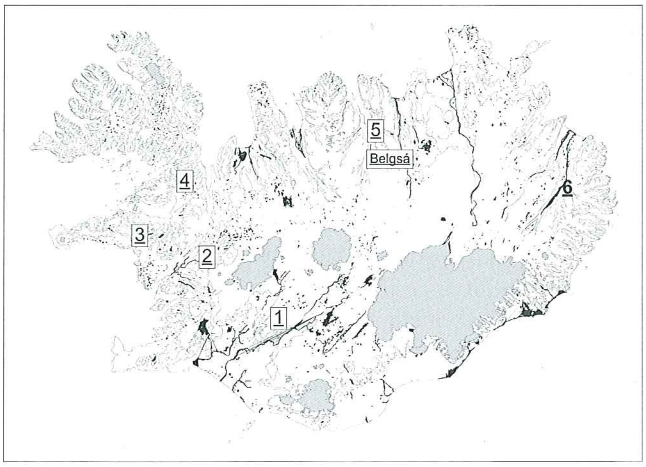

Figure 1. The main iron smelting areas in Iceland: 1. Þjórsárdalur; 2. Borgarfjörður/Mýrarsýsla; 3. Snæfellsnes; 4. Dalasýsla; 5. Fnjóskadalur; 6. Fljótsdalshérað.

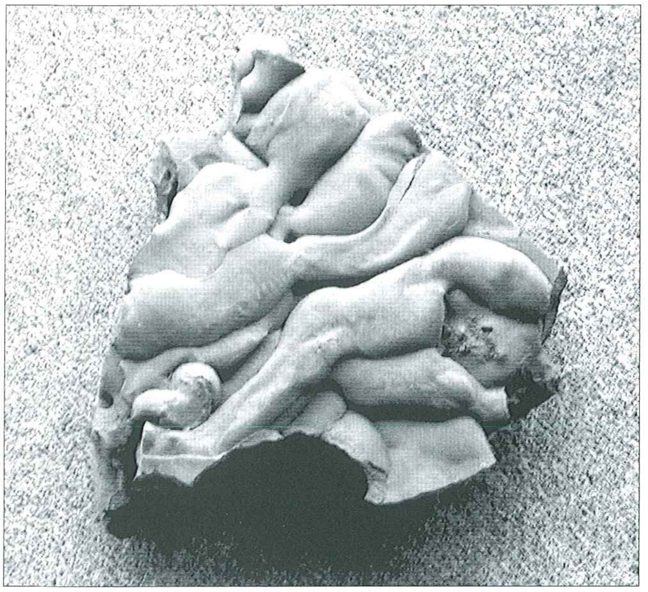

Figure 2. Iron smelting slag from Iceland. Note the ropy surface.

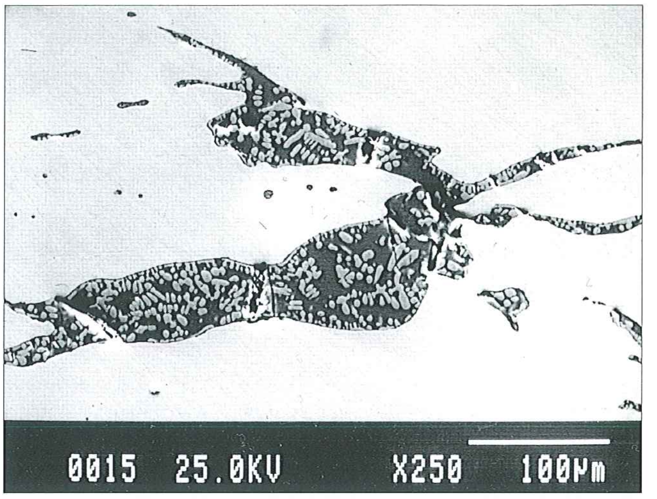

Figure 3. The dark strings are slag inclusions in the sword: Þjms. 4781.

(Rosenqvist, A M, 1983, 133; Buchwald, V F and Voss, O, 1992, 31; Martens, I, 1988, 76, 141-142; Nielsen, N, 1926, 147; Serning, I, 1973).

Until the Middle Ages, iron in Scandinavia was produced in a single operation called the direct process. During this process the iron forms a solid mass of sponge iron (bloom) which is then forged into workable iron. One of the characteristics of the iron produced by the direct smelting process was a large number of slag inclusions in the bloomery iron and the lightly wrought bloom. Some minute slag inclusions are also usually left in the objects. The slag inclusions in the objects do not only originate in the blooms but can be formed during primary and secondary smithing.

Some slag was thus formed during the blacksmithing process, mainly from sand used during welding.

II. Provenance studies

Provenance of iron has never been a straight forward subject and a few unsuccessful attempts have been made to determine the origin of iron objects. The pioneers in this field chiefly used bulk analysis, like mass-spectrometric analysis and atomic absorption spectrometry analysis to compare the chemical composition of the metal and slag in objects and currency bars to that of iron ore. Mostly without satisfying results (e.g. Christie H , Alfsen B and Rosenqvist A M 1979; Hansson, T and Modin S 1973; Thålin, L 1973; Tomtlund, J E, 1973).

It became evident that the chemical composition of the ore was often similar from various localities within the country and that people were also mixing ores from different areas as raw material for the iron smelting. It was thus impossible for the researchers to link the objects to a certain area. Besides, by using the bulk method, corrosion products and other pollution in the sample distort the measurements. More promising methods, based on spot analysis, were developed in the 1980's and 1990's, some producing good results and others not (Fells, S, 1983; Photos, E 1987; Buchwald, W F, 1994; Jouttijärvi, A, 1992). Particularly, the methods developed by Buchwald and Jouttijärvi appeared to show good results. By using the scanning electron microscope with energy dispersive analysis (SEM/EDAX) comparing the chemical composition of smelting slag to those of the slag inclusions in blooms and currency bars. As a result they managed to show difference in the chemical composition of Danish, Swedish and Norwegian smelting slag as well as slag from other areas in Europe. They also managed to detect distinct characteristics in the chemical composition of the slag inclusions in the objects and blooms that have allowed them to place the provenance of the finds within certain geological areas (e.g. Buchwald, V F, 1994, 38; Jouttijärvi, A, 1992).

III. The EPMA/WDA analysis

In my research I decided to use a method little different to that of the Danes, using the electron probe microanalyses with a wavelength dispersive analyser (EPMA/WDA). The EPMA/WDA compared each of the elements to individual standards. Thus it measured each element per se and was better equipped to measure trace elements. I started by using the SEM/EDAX in order to decide which elements to measure. Involved in the study were 112 remains: objects, lightly wrought blooms, ore and slag remains from 50 sites around Iceland.

The comparative material for analysis was smelting slag from Norway, Sweden and Denmark and publications of similar analysis from the British Isles, mainland Europe and Scandinavia.

There were three main questions I wanted to answer through this research. Firstly, whether spot analysis, by using EPMA/WDA, showed any difference in the chemical composition of the smelting slag from the four Nordic countries: Denmark, Iceland, Norway and Sweden. Secondly, whether it was possible to distinguish between the chemical composition of smelting slag from different areas in Iceland and thirdly, whether, as was hoped, the smelting slag and slag inclusions in the objects had such distinctive elemental characteristics that it would be possible to determine whether the objects were of Icelandic or foreign origin.

As a result of the analysis in the SEM/EDAX, the decision was made to measure the following elements in the EPMA/WDA: Mg, Fe, Al, Ni, Si, Ti, As, S, Ca, Co, Mn, P, K, Cr, Ba and Sr. In some cases Cu, Cl and Na were included.

On average 6 -7 spots were examined in each sample; in some cases fewer spots were measured as the slag inclusions were to small or badly preserved, while in other cases a larger number of spots were measured. This was done in order to find out whether there was a difference between the readings of the various slag inclusions within the sample. The amount, size and preservation of the slag inclusions vary in the objects from Iceland. Although many of them are well preserved, too many are friable and others are to small to be used for analysis. It was this author´s intention to analyse the concentration in each of the phases in the slag. It was often hard to do so as the phases were too small for the beam. As a result there was the danger that one would measure the chemical concentration of the other phases in the slag inclusion, or penetrate into the surrounding metal.

The slag inclusions in the objects from Iceland were of three types: two-phased glass and iron-oxide, (usually wüstite) or iron silicate (usually fayalite), three-phased (glass, wüstite and fayalite) and single-phased (mainly glass or fayalite). The multi-phased slag are usually thought to originate in the bloomery process (Tylecote, R F, 1987, 316).

It was thus the multi-phased slag that were chemically analysed in this study and compared to the analyses of the smelting slag. An attempt was made to detect all the main phases of the slag inclusions and smelting slag: the most common phases as shown in fig. 4 were the iron silicate/fayalite (Fay), glass matrix (G) and the iron oxide/wüstite (Feox). Tests were also made for metals (M) to establish the elements they might contain.

The EPMA/WDA readings did show some difference between smelting slag from the different countries on the basis of the propositions of the magnesium (Mg), aluminium (Al), calcium (Ca) and phosphorus (P). These elements are mainly preserved in the glass phase, apart from the Mg which concentrates in the fayalite and other Fe-richer phases such as the pyroxene groups and hercynite.

As examples of the analysis and its result, I will take the Al and P readings of the smelting slag and the same elements in the swords and the nails/rivets.

The Swedish samples have one high reading of Al, 25.1 wt%, as did one of the Icelandic samples (23.2 wt%). Apart from that, the Norwegian samples had the highest readings of Al and this could be detected both in the glass and hercynite phases (fig. 5). High readings were also detected in the samples from Sweden (7.7-9.8 wt%) and Iceland (many readings between 7-8 wt%). The readings in the glass phase showed lower concentrations of Al than in the iron silicate phases thus indicating hercynite. The differences in the readings between these three countries are not so great as to make it possible to discriminate between them. On the other hand, the analysis of Al may suggest that the Danish slag have lower Al contents than the smelting slag from the other countries,. The concentration in the Danish samples analysed reached a maximum of 3.5 wt% while all the other were over that limit. The results of the readings of the samples analysed in this study indicated that it may be possible to use the Al to distinguish Danish slag from slag from other countries (fig. 5).

In the smelting slag the P concentrates mainly in the glass phase (fig. 6). The Danish and Icelandic samples show a few high readings but in general it is hard to distinguish between the Icelandic, Danish and Swedish slag on the basis of P content, since many of the readings cluster near to each other. The Norwegian slag, on the other hand, have far lower concentration of P in the slag than the other countries, not

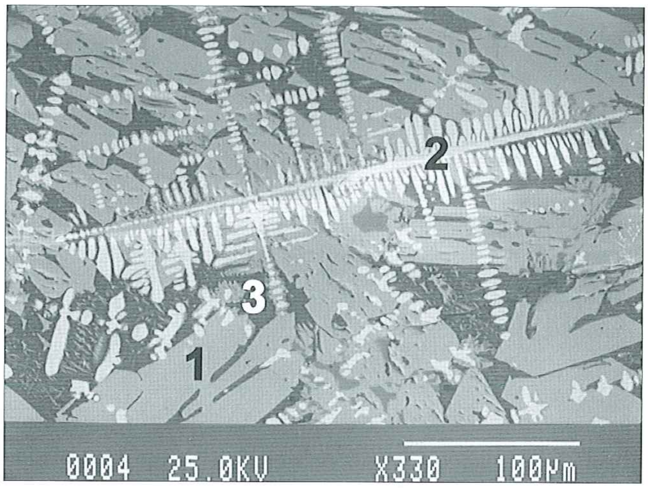

Figure 4. 1. iron silicate/fayalite (Fay), 2. iron oxide/wüstite (Feox), 3. glass matrix (G).

exceeding 0.3 wt% in the glass phase. Phosphorus content is thus useful for excluding Norwegian origin of the slag.

Two double-edged swords are represented among the objects: Þjms 1962: 124, which was determined as type M and thus dated to the 10th century, and Þjms 4781 which was not categorized by type as the hilt was missing (Eldjárn, K, 1956, 150; Grímsson, Þ, 1966, 78-86).

Þjms 1962:124 is an example of a high degree of skill in metal smithing showing good competence in welding. It is made of three main components, a centre of low carbon ferrite and the two cutting edges made of three-layered structure of steel of varying carbon content.

Þjms 4781 was not as sophisticated a product as Þjms 1962:124, It is also double edged with a fuller along the whole length. The tip was broken off. The sword is made of iron with a ferrite microstructure. After etching with Nital one could see dark phases along the edges of the sword, most likely of carbon diffusion. It might thus have been case-hardened. There are great amounts of two-phased slag inclusions of glass and iron oxide in the sword. Some of the inclusions are quite large, others smaller and elongated. The structure of the slag inclusions was similar to the slag in the blooms. The amount of entrapped slag in objects has often been used as a measure of the skill (or lack of it) of the early iron producers in expelling slag during consolidation of the bloom (Scott, B G, 1990, 16). Judging from the amount of slag in the sample this sword seems to have been made from a badly worked bloom.

As can be seen in figs. 5 and 6, none of the readings of P and Al from the objects fall exactly within the range of the slag. The same accounted for the other elements measured.

The result of the comparison was that it was not possible to pinpoint the origin of the objects by using the EPMA/WDA method. Due to wide range of readings of each of the elements in the slag from each of the countries, and due to the fact that the readings from the objects did not fall distinctively within the reading in the slag, the method is not promising for determining the provenance of iron objects. The results of the attempt to find the provenience of the objects were as follows:

By comparing the measurements of P and Al in the slag and swords, both the swords, Þjms 4781 and Þjms 1962:124, could possibly be made from Icelandic bog iron as some of the readings in the slag inclusions were similar

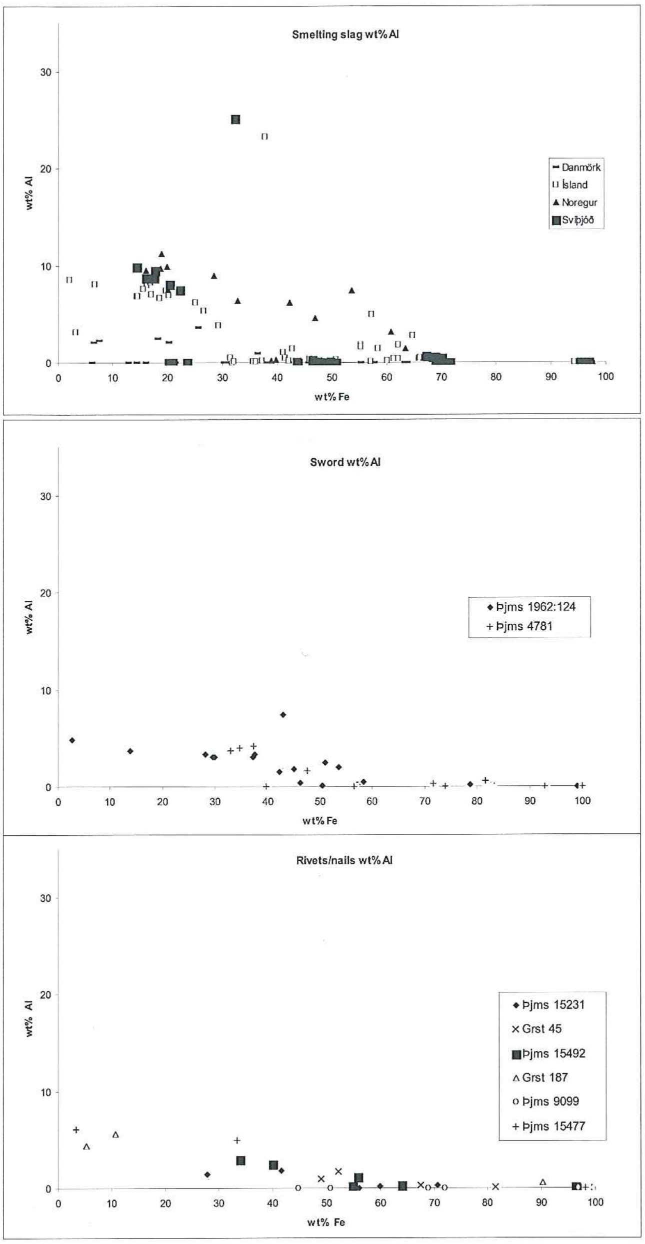

Figure 5. Wt% Al in smelting slag, sword and rivets/nails.

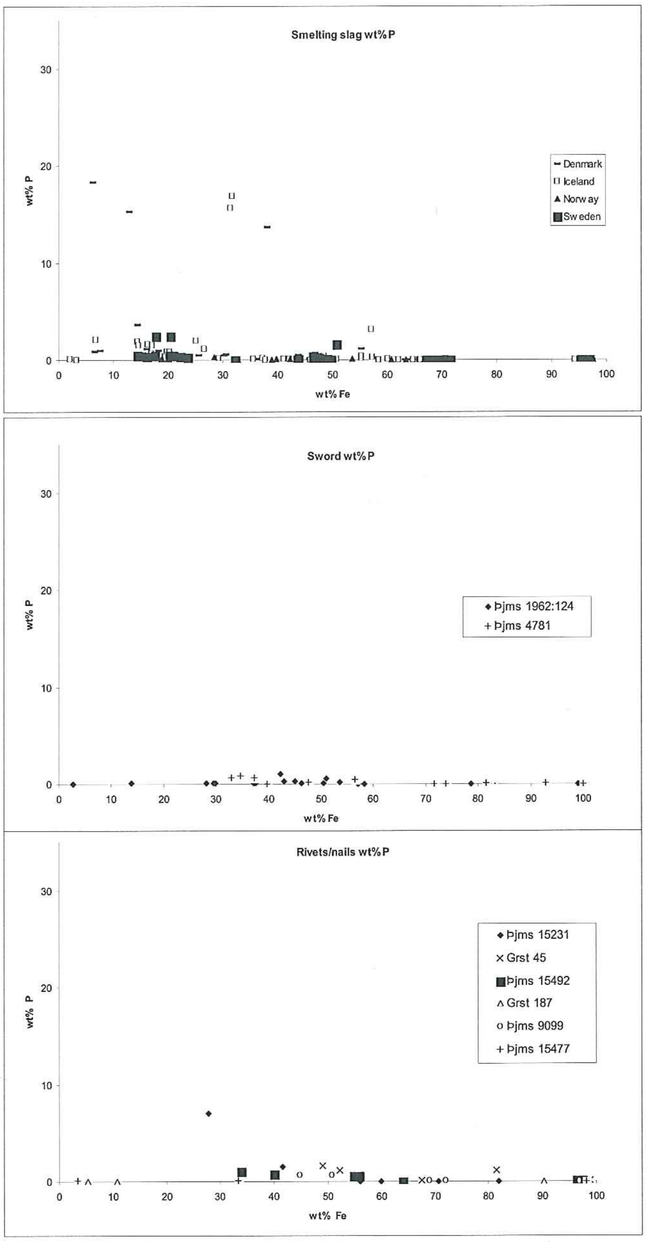

Figure 6. Wt% P in smelting slag, sword and rivets/nails.

to those in the Icelandic smelting slag. But as seen in figs. 5 and 6 they could just as well originate in the other Nordic countries.

The same accounts for the nails and rivets, as shown in figs. 5 and 6. By comparing the Al, nail Þjms. 9099, which is thought to come from a horse harness, could come from any of the countries. It is hard to say from which of the countries Grst 45, which comes from a dwelling house, might originate. But, by comparing the Al in the nails/rivets to that of the smelting slag, the other five, Þjms. 15231, thought to come from a boat, Þjms. 15492, which probably comes from a horse harness, Grst 187, which comes from the same house as Grst 45, and Þjms. 15477, which probably comes from a coffin, one could say that these nails might be made of Icelandic bog-iron ore as they had readings that lie near to the Icelandic ones (Eldjárn, K, 1956, 28-31, 45-46, 98-101, 117-121; Einarsson, B F, 1995, 197).

When comparing the P measurements, Grst 45 could be of Swedish origin and 15231 and 15492 of Icelandic origin, while Grst 187, 9099 and 15477 could originate in all of the countries.

IV. Conclusion

Although the elements Mg, Al, Ca and P show some variation in the smelting slags from the different countries, the readings from the different countries tend to overlap, as can be seen in the cases of Al and P in figs. 5 and 6. The same accounts for the slag from the different localities in Iceland. When it comes to comparing the objects to the slag, the plots from the objects do not lie exactly in the same ranges as the elements in the slag, but close to them. And although the readings from some of the elements in the objects are similar to the readings of slag from one of the countries, the readings of other elements in the objects are more similar to those of other countries. It is thus not possible to determine with any certainty that the objects originate in one particular country. Although some trends can be seen in the plots, there are so many questions that cause the results of the analysis to be unclear.

There is the question as to whether the smelting slag remains from countries other than those included in this study might have given similar results, i.e. indicated that the objects might be from those countries.

Another problem is that one does not know exactly which methods were used during the smelting and smithing process and thus which elements, and what amount of them, might come from the charcoal used during the different processes, or from bone ash, sand, slag, or possibly from the furnace lining.

One of the aims of this research was to distinguish between the slag from the different countries. Due to the geological differences between Iceland and the other countries, one could expect that the elements from ore, fuel, flux and possibly furnace lining might vary between the countries and that this would show in the slag by giving a different chemical composition to the slag from the other countries.

Another point worth mentioning is that the elements are effected during the analysis in the EPMA/WDA as pointed out by Fells (1983, 101, 132).

Yet another problem is the beam. There is the danger that the beam does not hit the phases in the slag inclusions that are to be measured. Thus, there is the possibility that one was measuring a phase other than the intended one, or the edge of the slag inclusion and the area around it, or even that one was penetrating too deep and analysing material beneath the slag inclusions, into the metal.

When reviewing the analysis method for provenance tested in this paper, the EPMA/WDA, one can say that the method was useful in giving some indication of differences between the chemical compositions of the smelting slag samples from the different countries. It does not show concrete characteristics in most cases as there is too much overlap between the readings for the slag from the different countries. It does not show any difference between different areas in Iceland. When it comes to comparing the chemical composition of the slag to those of the objects, the task becomes even more difficult. The chemical analysis of the objects does not give grounds for stating that any of the objects are without doubt from any one country. The study leaves the present author thus with more questions in mind than in the starting phases of the research. There are questionable points at every stage of the process.

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