Mass-conservation of Archaeological Iron Artefacts

A Case Study at the National Museum of Iceland



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Image on cover shows blisters and akaganéite found on a horseshoe from Reyðarfell, W-Iceland (no. 1960-72:14). The image is taken in a stereomicroscope with x25 magnification. Photographer: Ívar Brynjólfsson, photographer at the National Museum of Iceland.

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ABSTRACT

The aim of this thesis is to investigate the possible benefits and consequences of a mass-conservation system at the National Museum of Iceland through a literature review. There have been periods where few or no conservators specialized in archaeological conservation have been working at the museum. This has left the museum with a large amount of both un-conserved artefacts and artefacts in need of re-conservation. This applies to most material categories but this thesis will only look into the condition of iron and its possible massconservation and how the methods would apply in reality.

The condition of iron artefacts from one site was evaluated and the information logged into a database. This was then used to gather information regarding the condition of the artefacts in the National Museum of Iceland's collection. No actual conservation was done, as this is a theoretical thesis.

General facts on iron and corrosion products are discussed. Iron conservation methods currently in common use in Northern Europe are reviewed in order to get an overview of which methods are applicable in mass-conservation. The goal of this thesis is to find a method to increase the productivity at the conservation department at the National Museum of Iceland within the limitations of low funding and a lack of conservators in the country.

After looking into various treatments of archaeological iron it is recommended in this thesis that only parts of the conservation process in the National Museum of Iceland will be adapted to a mass-conservation setup. That way the process can be sped up and the condition of the artefacts can be evaluated thoroughly.

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Foreword

I would like to thank everybody that supported me during these last few months; especially my mentor Charlotte Gjelstrup Björdal for support and guidance and the National Museum of Iceland for access to artefacts, information and equipment. First and foremost I would like to thank my family and my fiancé Páll Kolka, without whom I could not have written this thesis.

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1 Introduction

1.1 Background

In the summer of 2011 I took part in a project at the National Museum of Iceland that focused on the re-organisation of the museum's archaeological storage. A part of the project was the conservation of iron artefacts from one large-scale excavation conducted in 1980's.¹ Some of the artefacts excavated had been conserved previously, but our work focused on those artefacts that were un-conserved. Only scalpels, ethanol and tannic acid were used to clean and conserve the artefacts, which was very time consuming and, in my mind, inefficient for such a large-scale project. Using these methods creates a backlog of artefacts, which is further exasperated by the fact that there is only one archaeological conservator working at the museum. I feel that this situation could be improved upon.

In the fall of 2011, I spent ten weeks of my practical internship period in Visby at The Swedish National Heritage Board (*sw.* Riksantikvarieämbetet) with a team of conservators that are working on a mass-conservation project. My experience in Visby showed me how large amounts of artefacts could be treated much more efficiently by using the methods of mass-conservation than what was being done at the National Museum of Iceland.

Mass-conservation is well suited for the situation in Iceland, where large amounts of artefacts are in need of conservation and very few archaeological conservators are available. Finding more time efficient and cost effective methods could be something that could move the conservation in the National Museum forward.

1.2 Problem Statement

The National Museum of Iceland is the only party in Iceland that has the facilities for largescale archaeological conservation.² All archaeological finds in Iceland are required to be handed over to The Archaeological Heritage Agency of Iceland (*icel*. Fornleifavernd *ríkisins*), who then hands them over to the National Museum of Iceland (*Þjóðminjalög*, 18 §). All artefacts are required to be handed in within one year of the end of the excavation and they have to be conserved prior to being turned in (*Reglur um veitingu leyfa til fornleifarannsókna*, 4 §). As the National Museum is only one of two parties doing archaeological conservation in Iceland, they carry out a very large part of the conservation work. Some archaeologists have employed conservators from abroad, but these are very few and this has only occurred in the last ten years or so.

There have been periods where few or no conservators specialized in archaeological conservation have been working at the National Museum. As a result, the museum is left with a large amount of both un-conserved artefacts and artefacts in need of re-conservation. This applies to most material categories but due the limitation of this thesis we will only look into the problems concerning archaeological iron.

The methods currently in use at the National Museum are not very efficient in regards to time management or the preservation of artefacts. For example, neither micro-grinding wheels nor air-abrasion are used for cleaning iron artefacts, only scalpels and ethanol. Iron

¹ This was the excavation of the farm mound of Stóra-Borg in the south of Iceland and was dated from 15th to the 19th century. The site was excavated during the years 1978-1990.

² The only other party doing archaeological conservation in Iceland is a privately run company, Fornleifafræðistofan ehf. Fornleifafræðistofan specializes in archaeological research and commission work within that field. In addition it has a small conservation facility and one conservator working there part-time. Fornleifafræðistofan started doing conservation in 2009 (Einarsson 2012).

artefacts are neither desalinated nor kept in dry storage and the storage facilities is not climate controlled.

1.3 Objectives and Goal

The objective of this thesis is to investigate the possible benefits and consequences of the implementation of a mass-conservation system at the National Museum of Iceland through a literature review. Methods, costs, time efficiency are all matters that must be discussed when planning a new conservation strategy. The methods currently in common use in Northern Europe will be reviewed and looked at with regards to their application for mass-conservation. The condition of artefacts from one excavation was to get an idea of the condition of archaeological iron artefacts found in storage at the National Museum of Iceland.

The goal of the thesis is to increase the productivity at the conservation department at the National Museum of Iceland within the limitations of low funding and a lack of conservators in the country. This will be done by making a treatment plan for the conservation process.

The following questions are among those that will be addressed in this thesis:

- What can we expect to find when looking at older artefacts that have been in storage for decades with various degrees and qualities of conservation?
- What types of damage do they suffer from?
- Does the conservation and storage at the National Museum need to be improved and if so, how?
- What methods of iron conservation are best suited for mass-conservation?

1.4 Methodology

As stated above artefacts from one excavation were selected to gain a brief insight into the condition of the museums collection of archaeological iron. The excavation selected was Reyðarfell in Hálsasveit in Borgarfjörður in the west of Iceland. It was selected due to the manageable number of iron artefacts excavated there. Most other large-scale excavations in Iceland have iron artefacts numbering in the hundreds, at least. While a larger selection of artefacts would give a more precise insight into the condition found in the museum's storage, it would be impossible within the limits of this thesis. It was also decided to use material from one excavation rather than gather individual artefacts from various excavations, as it would be impossible to cover the archaeological context of so many sites in this thesis. This would also create so many variables that comparison between the artefacts would be difficult.

A complete finds list of the iron artefacts was done for the purposes this thesis in FileMaker Pro. Excel was used for statistics and diagrams. The condition of the iron artefacts from Reyðarfell was estimated visually and each artefact was given a grade between 1 and 5, 1 representing artefacts in a stable condition and 5 very unstable artefacts. Visual diagnosis of corrosion products was also performed through a stereomicroscope. Conservation methods were identified on those artefacts that had been treated and it was also noted if the artefact had not been treated. The condition of each object was documented by photography. Some artefacts were photographed in a more detail to document corrosion and typical, good or detrimental conservation.

All the images and illustrations in the thesis were done by the author, except for stereomicroscopic images, which were taken by the photographer at National Museum of Iceland, Ívar Brynjólfsson.

1.5 Limitations

Since it is impossible to look at and estimate the condition of every single iron artefact at the museum within the frame of this thesis, iron artefacts from one excavation were selected to get an idea of the overall condition of the museums collection of archaeological iron.

No actual conservation was carried out in this study, but visual examination was done with a stereomicroscope to describe the corrosion layers and possible corrosion products. Only visual analysis was performed to identify corrosion products and previous conservation methods.

The artefacts from Reyðarfell were used as reference to get an idea of the condition of the artefacts in storage, but do not represent the entire collection of archaeological artefacts in the National Museum of Iceland.

1.6 Previous Research

No archaeological artefact analysis has ever been done on the artefacts from Reyðarfell. A bachelor's thesis in archaeology from the University of Iceland written by Rúnar Leifsson discusses the Reyðarfell excavation itself (Leifsson 2004). Leifsson's thesis is an attempt to reinterpret the information gathered at the site, because very little had been published on the excavation, as stated previously.

The manuscript for the final report of the excavation does exist and proved very useful in this thesis, but there was no research done on the artefacts themselves by the excavation team.

Very little has been written on mass-conservation, but there are some articles regarding the conservation of large quantities of iron, such as the article "An approach to handling large quantities of archaeological iron" by Logan published in 1984 and "The conservation of iron objects in archaeological preservation – Application and further development of alkaline sulphite method for conservation of large quantities of iron finds" by Schmutzler and Ebinger-Rist, published in 2008.

Because of how little has been written on mass-conservation, articles on iron conservation in general are used here for specific iron conservation methods and on storage methods. Classic articles were sited, such as "Post excavation changes in iron antiquities" by Turgoose, published in 1982 and "Washing Methods for Chloride Removal from Marine Iron Artefacts" by North and Pearson, published in 1998. Articles on iron conservation general were used extensively, such as "Overview of archaeological iron: the corrosion problem, key factors affecting treatment, and gaps in current knowledge" by Selwyn, published in 2004.

1.7 The Structure of the Thesis

The thesis is divided into seven chapters and one appendix. Chapter 1 introduces the research and its background. Chapter 2 concerns the iron artefacts from Reyðarfell in W-Iceland and the case study carried out in this thesis. The artefacts from Reyðarfell will be discussed, as will their condition, current storage and previous conservation. Chapter 3 deals with the corrosion of iron in the ground and post-excavation, as well as the visual identification of corrosion products.

Chapter 4 looks briefly into the history of iron conservation and chapter 5 covers current methods commonly used for conservation of archaeological iron and their application in mass-conservation. Chapter 6 looks especially into mass-conservation and its possible application on the Reyðarfell material. Chapter 6 discusses the conclusions reached in this thesis and summarizes the thesis. Chapter 7 is the list of references. Appendix I is a print-out from the database created for this thesis of the Reyðarfell artefacts.

2 Iron Material from Reyðarfell in Hálsasveit, W-Iceland

2.1 History of the Material

Reyðarfell is a medieval farm in Hálsasveit in Borgarfjörður in the west of Iceland. It sits on the slopes of mount Húsafell and lies within the boundaries of the farm Húsafell. The excavation at Reyðarfell was conducted by the National Museum of Iceland and headed by Porkell Grímsson. The excavation began in 1960 and continued with reprieves throughout the decade until 1969 (Grímsson 1976, p. 566).

2.1.1 The Location of Reyðarfell

The excavation was one of the first rescue excavations in Iceland. The farmer at Húsafell had intended to extend his hayfields and in the process he disrupted the ruins at the site, which had been declared a National Heritage site in 1931 (*Friðlýsingaskrá*³ 1990, p. 11; Grímsson 1976, p. 565).

The results from the Reyðarfell excavation are quite difficult to interpret as very little has been written about the site, and only one article published in 1976. Grímsson wrote a manuscript for the final report and a progress report from 1960 exists, but neither has been published. These are both stored in the National museum's archives. The manuscript is not dated, however the youngest articles cited are published in 1989 (Grímsson n.d., p. 20). Grímsson was employed at the National museum of Iceland until 1992 (Hallgrímsdóttir 2010), so it is reasonable to assume that the manuscript was written around 1990.

The placement of the farm excavated does not coincide with descriptions of Reyðarfell in older sources. In *Jarðabók Árna Magnússonar og Páls Vídalíns*, a land registry from the early 18th century, the farm is said to be higher on the mountain Húsafell than younger sources claim (*Jarðabók* 1943, p. 255; Jónsson 1893, p. 77-78).

2.1.2 Dating of the Site

The excavation was done on the youngest phase of houses at Reyðarfell and it is not known from an archaeological standpoint, how long Reyðarfell was inhabited, as the older phases of the site were only partially excavated (Grímsson 1976, p. 567). Leifsson claims that it is impossible to date the farm due to lack of excavation data and the poor methodology used on site (Leifsson 2004, p. 70).

Grímsson claims that the farm was abandoned at the end of the medieval period and that written documents from 1504 confirm this. Grímsson notes that Reyðarfell is mentioned in The Book of Settlement⁴ (icel. *Landnámabók*) and based on that hints that it was inhabited since the settlement period of Iceland (ca. 871 ± 2 to 930).

There are two letters in *Diplomatarium Islandicum* $(DI)^5$ regarding Reyðarfell. A bill of sale for Reyðarfell from 1442, were it is noted that the church in Reyðarfell had been abolished. The farm's estimated worth in 1442 was 16 hundreds.⁶ A cartulary of the church

³ Register of National Heritage Sites in Iceland.

⁴ The Book of Settlement was written in the 13th century and has long been used in academia as a factual record of the settlement of Iceland. This uncritical use of the book has been called into question in the last decades by younger generations of scholars, especially by archaeologists.

⁵ *Diplomatarium Islandicum* is a collection of Icelandic letters, cartulary and various documents from the earliest documents found until 1590. The majority of the documents are dated later than 1250.

⁶ One farmland-hundred (*icel.* jarðhundrað) is an Icelandic value unit and was the same as 120 *aurar* of silver and later 120 ells (*icel.* alin) of woollen cloth. 120 ells of woollen cloth were the same as the value of one cow. This was changed in the 17th century and 120 ells of woollen cloth became the value of two cows. The hundred-unit was first used in the 11th century in Iceland and was in use until the 19th century. An average sized farm in Iceland was around 20 hundreds and a small farm around 6-10 hundreds (JGP 2011; Laxnes 1995, p. 213).

of Húsafell, from 1504, states that the farm is worth 24 hundreds and that the bishop has declared the farm a property of the church at Húsafell (*DI* 1897, p. 632; *DI* 1903, p. 737). Grímsson interprets this to mean that the farm had been abandoned in 1504. The fact that the farm's value had increased by eight hundreds from 1442 to 1504 does not support Grímsson's theory that 1504 cartulary proves that the farm was abandoned in 1504. It is however clear that Reyðarfell was abandoned by 1709 when Árni Magnússon and Páll Vídalín surveyed the area for their land registry and had been for quite some time (*Jarðabók* 1943, p. 255).

2.2 The Artefacts From Reyðarfell

All in all, Grímsson lists 201 finds in his manuscript, including samples. 255 finds are registered at the National Museum from Reyðarfell (also including samples). Some of the finds that were on Grímsson's list in his manuscript cannot be found at the National Museum and quite a few that were at the museum are not mentioned in Grímsson's find list.

There are in total 84 iron artefacts from Reyðarfell at the National Museum. In Grímsson's list there were 61, but many of those mentioned there could not be found at the museum. 48 of the artefacts are mentioned both in Grímsson's manuscript and in *Sarpur*, the National Museum's database.

For the purpose of this thesis, a database was designed using FileMaker Pro. The database is a complete list of the iron artefacts from Reyðarfell, with photographs, the artefacts condition and grade, if the artefact has been conserved, what type of coating it has and what type of corrosion product was found on it when examined in a stereomicroscope. A printout of the database can be seen in Appendix I.

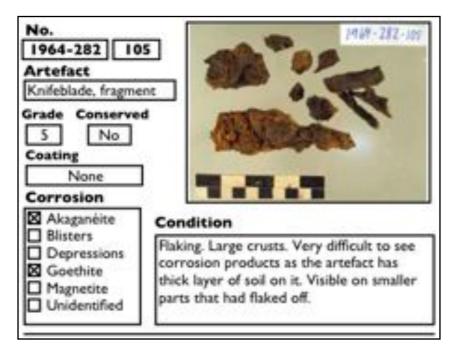


Figure 1. An example of how a record of an artefact looks like in the database.

2.2.1 Previous Conservation

The vast majority of the artefacts (90%) from Reyðarfell have been conserved. These artefacts are generally in better condition than the ones that had not been conserved, but no definite conclusions can be made due to the small sample size. Most of the artefacts that had been treated had been coated with wax, and some had been coated with some sort of varnish (12% of conserved artefacts).

There are no records of previous treatments available for artefacts from Reyðarfell, and Halldóra Ásgeirsdóttir, a conservator who worked at the National Museum from 1983 to 2010 said that judging from the appearance of the artefacts (thick layers of wax and almost no cleaning) and their labelling that they were most likely conserved prior to 1983. She said that Gísli Gestsson, an antiquarian at the National Museum from 1951 to 1977, had treated many artefacts with paraffin wax (Ásgeirsdóttir 2012).

Gestsson had studied chemical engineering in Copenhagen, but had not finished his degree (Björnsson 1984, p. 7). It is reasonable to assume that he had some knowledge of chemistry to aid him in the conserving of the artefacts. In his documents, stored at National Museum's archives, there is a report titled 'Forvarzla – Konservering', from 1971 on the conservation done at the National Museum of Iceland. Also in his documents were reprints from the seminar 'Arkæologi og konservering' held in Bergen in 1975, which he had attended. In his rapport from 1971 Gestsson states that by that time most of the iron artefacts in the museum had been conserved by washing them in tap water or distilled water and subsequently boiled in paraffin wax. He notes that this method is generally considered insufficient for conservation, as the water does not remove all of the chlorides from the National Museum of Iceland, as the storage's RH was below 40% (Gestsson 1971, p. 2). Paraffin wax gained much popularity in the 1960's after the conservator Harold Plenderleith advocated its use and claimed that it did not leave a sticky surface and was easy to remove (Jaeger 2008, p. 218).

It is also possible that Gestsson treated the varnished artefacts as well as those coated with wax. A 1962 article by Gestsson describes the treatment of an iron spear, and is more informative regarding the other methods used at the museum than Gestsson's rapport from 1971. The methods used on the spear were described as follows:

...the spear was mostly whole when it was found, but later became somewhat damaged, and the blade was considerably cracked when it came to the National Museum, but the socket was fortunately whole... A closer examination revealed that it [the socket] had been decorated with silver, but was otherwise covered by 1 cm thick layer of rust on the outside. The rust was extremely hard, and had to be removed from the metal by small chisels, drills, files, and lastly an iron saw, as softening the rust was unsuccessful with those chemicals that did not corrode the underlying metal. It was not possible to clean the blade as there was very little iron in it, as can be seen on X-rays... Afterwards the spear was washed thoroughly in distilled water and then polished again and gaps in the blade were filled with a putty of cellulose varnish and talcum, coloured with coal dust. Lastly, the blade was coated with zapon lacquer (Gestsson 1962, p. 72-73).⁷

This suggests that the non-wax coatings that are on 12% of the artefacts from Reyðarfell could possibly be Zapon lacquer or a cellulose varnish, as mentioned by Gestsston. Zapon lacquer was a commercial cellulose nitrate lacquer, and was one of the first synthetic resins to be prepared on an industrial scale for conservation (Gilberg 1987, p. 112).

Friedrich Rathgen pioneered the application of Zapon lacquer in the conservation of artefacts and published a paper on it in 1904. However, it was later realized that Zapon and

⁷ Eins og fyrr segir, var spjótið að mestu heilt, þegar það fannst, en síðar varð það fyrir nokkru hnjaski, og var fjöðrin talsvert sprungin, er það kom á Þjóðminjasafnið, en falurinn var sem betur fer heill... Við nána athugun mátti sjá, að hann var silfurbúinn, en annars var allt að 1 sm þykkt ryðhrúður utan á honum. Ryðið var ákaflega hart, og varð að losa það frá málminum með smámeitlum, borum, þjölum, og ekki sízt með járnsög, þar eð ekki tókst að mýkja ryðið með neinum þeim efnum, sem ekki tærðu málminn, sem undir var. Ekki voru tiltök að hreinsa fjöðrina, enda var mjög lítið járn eftir í henni eins og sést á röntgenmynd af henni... Síðan var spjótið þvegið vandlega í eimuðu vatni og þá fágað á ný og fyllt upp í bresti í fjöðrinni með kítti úr selluloselakki og talkúm lituðu með koladufti. Seinast var lakkað yfir falinn með zaponlakki (Gestsson 1962, p. 72-73) (Translation by author).

other cellulose nitrate preparations were far too flammable to be used in conservation and Rathgen published a rapport on those findings in 1913 (Gilberg 1987, p. 112-113). Despite its well-documented disadvantages, its use in coating various types of artefacts and archive materials has continued. One of the reasons for its continued use is the fact that it coats metals with a very nearly invisible, thin and firmly adhering film that leaves the artefact's appearance unchanged (Gruber and Ha 2005, p. 239).

In 1969 the National Museum of Iceland received a grant from UNESCO to fund temporary conservation work at the museum. Two conservators from the British Museum came in 1969 and 1970. Each worked for one month each year, conserving various artefacts, but in 1969 mainly metals were treated (Magnússon 1969, p. 161; 1970, p. 130; 1971, p. 139). The grant from UNESCO is mentioned in the museums yearly rapport from 1968 and the necessity of a qualified conservator working full time at the museum is emphasised. It is also mentioned that the museum staff performed 'simpler and rougher' treatments of artefacts (Magnússon 1969, p. 161). It is possible that these conservators conserved the artefacts from Revðarfell that have been treated, perhaps those treated with a thin coating of varnish, but there were no records found in the museum's archives on what artefacts were conserved or what methods were used. The British Museum was contacted for information about the methods used there in 1969 and 1970. Marylin Hockey, conservator at the British Museum replied and said that standard treatments at that time for archaeological iron included mechanical and manual cleaning, electrolytic reduction, hot washing and alkaline sulphite. The specific method chosen would depend on the corrosion and condition of each artefact in question. Protective coatings might have included microcrystalline wax by immersion in molten wax or possibly polyvinyl acetate lacquer. Graphite was often added to the coating (Hockey, 2012).

2.2.2 Condition

For the purposes of this thesis, the condition of the iron artefacts from Reyðarfell was estimated, and each artefact was given a grade between 1 and 5. 1 represented those artefacts in a stable condition and in little or no need of conservation and 5 representing those artefacts that are fragile, unstable and in urgent need of conservation. All of the artefacts from Reyðarfell are made of wrought iron. Points from chapter 2.3 Identifying Corrosion were used when estimating the condition of the artefacts. A stereomicroscope was used to identify akaganéite and blisters caused by weeping.

90% of the iron artefacts had been treated in some way, either with a wax coating or with some sort of varnish. Of the artefacts that had been treated 88% were treated with wax and 12% with varnish. This means that 79% of all of the 84 iron artefacts from Reyðarfell were treated with wax, and were most likely washed prior to coating. In all but two cases⁸ dirt and corrosion products hand not been cleaned off before they were coated. The two artefacts that had been cleaned were given the grade 1 as the seemed very stable and showed no sign of renewed corrosion. They had however been quite aggressively cleaned. 37% of the iron artefacts were graded 4-5, meaning that all of these artefacts are in danger of disintegrating and are very fragile. 40% of the artefacts were graded 1-2. The fact that less than half of the artefacts are in a stable condition is not optimal.

Out of the nine artefacts that were not conserved were seven artefacts graded 4-5, i.e. unstable and very unstable. All of those artefacts that were graded 1-3 had been conserved, except for two artefacts. This means that the steps taken to conserve the artefacts appear to have been in part successful in preserving the artefacts, but despite that fact are 37% of the artefacts are in great danger of disintegrating.

⁸ Those artefacts were number 1960-72-36, a lock and 1961-131-69, a key. These were in fact, perhaps a little to aggressively cleaned.

Grade	Condition	Example of grade
1	In good and stable condition and in little or no need of conservation.	
2	Stable, but has not been cleaned.	
3	Signs of deteriorating condition. Conservation needed, but not urgent.	Cm
4	In need of conservation. Some flaking.	
5	Very fragile. In urgent need of conservation. Very sensitive to handling.	

Table 1. Description of the grade system used when estimating the condition of the artefacts and photographs of examples.

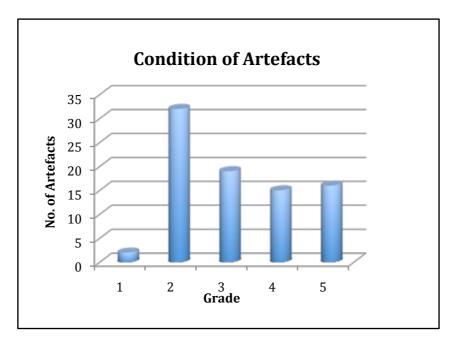


Figure 2. A diagram showing the distribution of the condition of the artefacts.

Corrosion products were examined visually using a stereomicroscope. Akaganéite was identified by its typical strands growing out of the surface (see chapter 3.3) as a corrosion product on 50% of the artefacts and blisters on 14%. It was impossible to investigate the corrosion products on 30% of the artefacts because of thick layers of wax. Other corrosion products were not identified in the examination, but it was noted when magnetite or goethite crystals were clearly visible. Unknown corrosion products were noted, marked under 'Other' in the database and described briefly as well. These are however only visual identifications and have not been confirmed by analytical equipment such as scanning electron microscope, X-ray diffraction.

Seeing as akaganéite is one of the greatest dangers facing archaeological iron postexcavation it is quite serious that 50% of the Reyðarfell artefacts apparently suffer from it. They need to be put in a desiccated storage as soon as possible. As stated before, akaganéite only forms under conditions of fairly high concentrations of chloride ions (Ståhl et al. 2003, p. 2564), so its presence therefore suggests that the artefacts are contaminated with chloride and need to be desalinated.

2.2.3 Current Storage

The archaeological storage facility in the National Museum does not have the equipment to control the climate carefully. The aim is to keep the temperature at an even level, but the building is originally built for a factory, not for museum storage, so it is hard to control the temperature when there are storms or drastically varying temperatures, as is often the case during winter in Iceland.

The artefacts are packed in clear, hard plastic boxes of polystyrene, supported by acidfree silk tissue paper. These are then packed into acid-free cardboard boxes. Ethafoam supports are made for fragile artefacts or larger items in need of support, such as swords and spears. The shelves are fixed shelves of aluminium.

Some artefacts are kept in polyethylene bags, but research has shown that these are not optimal for the storage of iron. Nails stored only in polyethylene bags have been shown to increase in weight by 7,4% after three years, due to the formation of corrosion products (Mathias et al. 2004, p. 36).



Figures 3 and 4. Aluminium shelves stacked with acid free cardboard boxes at the National Museum of Iceland. In between the cardboard boxes are PE boxes desiccated with silica gel for the storage of unstable artefacts. Figure 4 shows a close up of one those. Humidity strip and the numbers of the artefacts in the box can also be seen.

Fragile artefacts in need of conservation or re-conservation are kept in polyethylene (PE) boxes with silica gel for desiccation. The silica gel is kept in perforated polyethylene bags that are labelled with the date that they were placed in the box. Humidity strips are placed in the box to monitor the humidity within. This procedure is recent in the storage and has not been done with all of the collection, but the possibility of poor maintenance of the silica gel is high due to the limited availability of staff in the museum.

3 Corrosion of Iron

Iron is a chemical element with the symbol Fe, derived form the Latin word *ferrum*. Iron is a common element in nature and was extracted from bog iron or soil high in iron oxide for the production of iron artefacts. Iron is a hard, plastic and ductile metal that can be processed and formed in annealed condition. Pure iron has a high melting temperature, about 1535°C and a density of 7.9 g/cm³ (Fjæstad 1999, p. 85).

All prehistoric iron artefacts found in Europe are made of wrought iron. Wrought iron contains less than 0,5% carbon and has different quantities of slag, phosphorus and sulphur (Fjæstad 1999, p. 85). The prefix 'wrought' is an old English word, meaning 'worked', as in iron worked in a smithy (Sörenson 2003, p. 1230). Steel is achieved by increasing the iron's carbon content to about 1%. Iron containing 2% or more carbon is called cast iron and is quite brittle due to its high carbon content. Therefore it cannot be formed like wrought iron or steel, but is cast into moulds (Fjæstad 1999, p. 86).

3.1 Iron Corrosion in the Ground

Iron is a relatively unstable metal and corrodes easily, which involves a transformation of the material and metals by oxidation. Oxidation means that the metal atoms give off one or more of the electrons to a substance in the environment, a so-called oxidizing agent. Oxygen is the most common oxidizing agent of archaeological metals (Borg et al. 1995, p. 60).

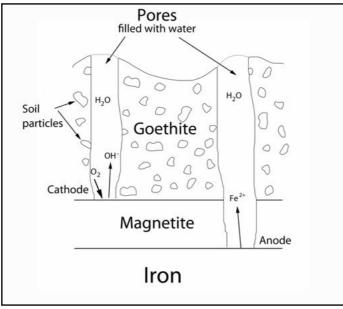


Figure 5. A schematic illustration showing the iron corrosion process and the stratigraphy of the corrosion layers of an iron artefact.

Corrosion is an electrochemical process and like all electrochemical processes requires an anode, a cathode and an electrolyte. When iron corrodes in the ground the surface is the anode and at the beginning of the corrosion process another part of the metal surface is the cathode. As layers of corrosion products build up the cathode is more likely to be an area of magnetite, Fe₃O₄, which is an electrically conducting corrosion product. The electrolyte in this process is the soil water, which contains chloride from dissolved salts (Knight 1997, p. 36). Chloride ions in the soil then accumulate at anodic sites in archaeological iron that is buried in a moist aerated context where they exist as iron chloride solutions satisfying the

charge balance of the Fe^{2+} ions produced by the corrosion process (Watkinson and Lewis 2004, p. 241). These chlorides will then later act as corrosion accelerators post-excavation (Rimmer et al. 2012, p. 29).

Pure iron is covered with a thin, oxide film, which forms by exposure to air. When iron is buried and covered by soil it is exposed to an aqueous electrolyte. The thin oxide film does not protect it from these electrolytes, so the iron corrodes. Iron oxidizes at anodic sites to Fe²⁺ ions that dissolve in the following process: Fe (s) \rightarrow Fe²⁺ + 2e⁻. Fe²⁺ ions can oxidize further to Fe³⁺ ions: Fe²⁺ \rightarrow Fe³⁺+ le⁻. As corrosion is electrochemical, a counterbalancing reduction reaction at must occur at the cathodic sites to consume the electrons generated in equation (Selwyn et al. 1999, p. 217). The most common reduction reactions are oxygen reduction and hydrogen evolution. Hydrogen evolution however only occurs at pH 4 or below so it is not as common as oxygen reduction.

Table 2. Oxygen reduction and iron oxidation (Selwyn et al.1999, p. 217-218).

Oxygen Reduction	Iron Oxidation
$O_2 + 2H_2O + 4e \rightarrow 4OH$ -	$Fe + \frac{1}{2}O_2 + H_2O \rightarrow Fe^{2+} + 2OH^-$
or	or
$O_2 + 4H^+ + 4e - \rightarrow 2H_2O$	$Fe^{+\frac{1}{2}O_2+2H+} \rightarrow Fe^{2+}+H_2O$

After the initial corrosion process and OH- ions have formed solid Iron(II) hydroxide, $Fe(OH)_2$ the corrosion rate decreases and passivation can occur. With time, when $Fe(OH)_2$ has oxidized to iron(III) hydroxide, $Fe(OH)_3$ can then transform to goethite. This process passivates the iron further as magnetite and goethite are thermodynamically stable. Magnetite forms under lower oxygen levels and goethite at higher oxygen levels. The corrosion process decreases gradually as the iron is covered with insoluble iron corrosion products cemented with soil particles, dirt and sand (Selwyn et al. 1999, p. 218).

3.2 Iron Corrosion Post-Excavation

When an artefact is excavated it is covered with a layer of corrosion products. The corrosion products are typically goethite, but sometimes a siderite, a FeCO₃ matrix in which magnetite, Fe₃O₄ or strips of maghemite (γ -Fe₂O₃) are embedded. Underneath this is another layer of iron corrosion products in a lower oxidation state, usually magnetite. This layer lies on top of the remaining metal (Selwyn 2004, p. 295; Watkinson 2010 p. 3311).

Post-excavation corrosion caused by chlorides is one of the most frequent and serious problems regarding archaeological iron finds (Schmutzler and Ebinger-Rist 2008, p. 248). The damage caused by these processes is irreversible and can result in the complete loss of the artefacts. Examples of the problems are e.g. cracking and the expansion of the corrosion layers, which causes flaking (Réguer et al. 2007, p. 2727).

The artefact's layer of corrosion is covered in pores or tunnels that contain an acidic solution of ferrous chloride, FeCl₂•4H₂O. When the artefact is excavated and brought into the atmosphere it is exposed to a new environment, usually with a lower relative humidity (RH) and higher oxygen content than in its burial context. The artefact begins to dry out and the pores in the corrosion layer are slowly aerated (Knight 1997, p. 36). When ferrous chloride is exposed to ambient conditions the reaction is as follows:

 $4Fe^{2+} + O_2 + 6H_2O \rightarrow 4FeOOH + 8H^+$ (Turgoose 1982, p. 98).

As the iron dries, the acidic solution and other salts crystallize and expand, cracking open the corrosion layers and increasing the access of oxygen to the remaining metal. Rapid drying of freshly excavated iron can also result in the formation of yellow crystals of Iron(II) chloride, FeCl₂ (Selwyn 2004, p. 295-296). This reaction to changes in the environment causes physical and chemical damage to the artefact. Chemical damage is caused by the formation of hydrochloric acid, HCl and physical damage is caused by the formation of new iron oxyhydroxides corrosion layers, which increases stress and causes cracks. One visual symptom of corrosion problems on excavated iron is the formation of either weeping or sweating iron (yellow droplets) or blistering (dry, hollow red spherical shells). Weeping is caused by the hygroscopic nature of iron chloride salts. When the relative humidity is high, the salts, e.g. Iron(II) chloride, absorb water, dissolve, and form wet droplets of orange coloured liquid. Iron oxyhydroxides precipitate around the outside of the droplets and form the framework for the blisters (Selwyn 2004, p. 296).

If the artefact dries rapidly in a RH lower than 18%, ferrous chloride will crystallise in the pores. But if the RH is higher than 18% the ferrous chloride will remain soluble and will slowly oxidise to form akaganéite if it is in contact with the iron core. It is the formation of akaganéite that causes artefacts to crack and flake when stored in ambient conditions. The other problem with akaganéite is that it acts as a reservoir of chloride ions, which can stimulate renewed corrosion of the iron core when they are released as the akaganéite decomposes (Knight 1997, p. 37).

Because akaganéite only forms under conditions of fairly high concentrations of chloride ions its presence on archaeological iron suggests that the artefact is contaminated with chloride (Ståhl et al. 2003, p. 2564). Akaganéite is believed to form on archaeological iron only when it is exposed to air after excavation (Mathias et al. 2004, p. 34).

3.3 Identifying Iron Corrosion products

According to Canadian Conservation Institute's (CCI) notes on *Caring for Collections*, stable archaeological artefacts "have compact and adherent corroded surfaces that vary in colour from blue-black to red-brown" (Logan 2007 a), p. 1-2). Unstable iron artefacts are those that suffer from active corrosion that can rapidly disintegrate the artefact. The corrosion occurs at the interface between the remaining metal core and the outer corrosion layer. The pressure of the corrosion between these layers causes cracking, flaking, and the detachment of the outer corrosion layers. Active iron corrosion can be indicated by fragments lying around the artefact and depressions on the metal surface with orange spots in the centres (Logan 2007 a), p. 1; Logan 2007 c), p. 1-2).

Close examination of an artefact can reveal active iron corrosion, usually either in the form of akaganéite, weeping or sweating, i.e. droplets on the artefact. These are only liquid at 55% RH or higher and at lower levels of RH they form blisters (<50% RH). When viewed in a microscope these blisters look like broken bubbles that are fragile, shiny and empty (Logan 2007 a), p. 1; Logan 2007 c), p. 1-2).

Akaganéite is considered as one of the greatest risks facing archaeological iron (Mathias et al. 2004, p. 34). It forms at the interface of the metal and the corrosion and appears as long, thin, orange crystals that are fuzzy or velvety in appearance with the naked eye (see figure 7). With a stereomicroscope they contain elongated particles that appear to be growing out of the surface. These are very fragile and brake off easily, they will also bend over when they grow far out of the surface (Selwyn and Logan 1993, p. 804; Selwyn et al. 1999, p. 229).

Corrosion products can be identified in numerous ways, e.g. by scanning electron microscope (SEM), X-ray diffraction (XRD), X-ray florescence (XRF) and Raman spectroscopy or Fourier transform infrared (FTIR) spectroscopy. However, due to the

limitations of this thesis and to the expense of such analysis, visual examination and estimation of the artefacts condition was used here. This is a well-known method and often used when estimating results of conservation methods (e.g. Costain, 2000). The following images were shot using a Dino-Lite handheld microscope and the stereomicroscope pictures were taken in a Wild Heerbrugg M3C stereomicroscope using a Canon EOS 5D Mark II camera.

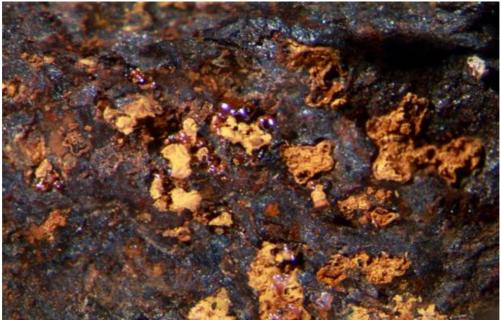


Figure 6. Fragile, empty and shiny blisters most likely resulting from weeping, viewed in a stereomicroscope. The blisters can be seen just above the middle of the image. The magnification is x25 (Logan 2007 a), p. 1) (Artefact no. 1960-72:14). Photographer: Ívar Brynjólfsson.



Figure 7. An example of a fuzzy corrosion layer, similar to the description of the appearance of akaganéite. The scale is around 10 mm (Artefact no. 1966-175:165).



Figure 8. Akaganéite viewed in a microscope. The magnification is about x25. The strands that are characteristic of the akaganéite corrosion product appear to grow out of the artefacts surface (Selwyn et al. 1999, p. 229) (Artefact no. 1960-72:9). Photographer: Ívar Brynjólfsson.



Figure 9. A close up of an artefact with active corrosion. Active iron corrosion can be identified by fragments surrounding the artefacts, depressions on the metal surface with orange spots in the centres of these depressions (Logan 2007 a), p. 1). The scale on the picture is just under 10 mm (Artefact no. 1960-72:14).

4 History of Iron Conservation

Although modern conservation was not developed until the late 19th century, the earliest written evidence for the conservation of antiquities comes from Pliny the Elder in the first century AD and the foundations of conservation were established during the Renaissance. Later, excavations at sites like Pompeii and Herculaneum in the 18th century led to techniques to preserve artefacts, rather than to restore them, to be developed. At the end of the 18th and in the early 19th century, scientists became increasingly more interested in problems concerning archaeological materials. The contributions of scientists such as Friedrich Rathgen in Berlin and Gustaf Rosenberg in Copenhagen formed the discipline of modern archaeological conservation of electrochemical reduction for the treatment of metal artefacts and established the guidelines for its proper use in conservation Another pioneer in early conservation was the Danish chemist Axel Krefting, who described the use of electrochemical reduction for cleaning iron artefacts in 1892. (Gilberg 1987, p. 110).

The greatest advances in the conservation of archaeological artefacts were achieved at the end of the 19th century. It was in 1882 the chemist Edward Krause, who worked in the Royla Museums in Berlin who first recognized the importance of salts in the corrosion process of iron and suggested desalination in hot and cold distilled water to eliminate them (Jakobsen 1988, p. 51-52). Friedrich Rathgen became the first director of the chemical laboratory of the Royal Museums in Berlin the when it was founded in 1888. His laboratory was the first museum research laboratory in the world (Gilberg 1987, p. 106). Rathgen published the first book devoted to the conservation of antiquities in 1898 and many of the methods recommended by him were in use in museums until the 1980's. Those methods were, among others, the mechanical removal of corrosion, heat treatment, electrochemical reduction and steeping in warm water followed by impregnation with paraffin wax or varnish (Knight 1997, p. 36). In 1855 impregnation is mentioned as the standard treatment of unstable iron artefacts in the Royal Museum of Nordic Antiquities, the predecessor to the National Museum of Denmark. Rathgen mentions in 1898 some of the materials used for impregnation, e.g. immersion in oils, waxes, lacquers and rubber coatings, but these were considered obsolete by the 1890's (Jakobsen 1988, p. 55-56).

Rosenberg revised in 1917 the electrolytic methods that had previously been used on iron. In Rosenberg's method the iron was heated up to 800°C for 15 minutes and up to two hours. After heating, and while still hot the artefacts were plunged into saturated sodium or potassium carbonate solution. After drying the artefacts were covered in wax. This method was used, with some modifications, at the Danish National Museum until 1977 (Jakobsen 1984, p. 84.22.8).

Until the 1970s, there were few treatments options for iron. Among those were boiling in purified water, reduction using electrolysis, or soaking in sodium carbonate. These methods were widely found to be unsuccessful and artefacts frequently re-corroded within a few years. Methods that have been introduced in recent decades in conservation include hydrogen reduction, chemical reduction, and gas plasma reduction. These are very aggressive treatments, some of which rely on heat, and can destroy the fragile corrosion surface and the metallurgical evidence found on the artefacts surface (Keene 1994, p. 250).

Washing methods have been a part of conservation treatments for iron for over a hundred years. For most of this time the goal has been to wash out chlorides. The work of Turgoose (1982) and Gilberg and Seeley (1982) has done much to explain the corrosion mechanisms sustained by archaeological iron after excavation and the role of akaganéite in the process (Keene 1994, p. 250). The first chemical study of akaganéite was done in 1960, were it was established that the compound always contains chloride (Reguer et al. 2009, p. 2796).

5 Current Methods in Iron Conservation

5.1 Mechanical Cleaning

Mechanical cleaning of corroded iron is a relatively common practice in iron conservation, and can be performed with different tools such as a scalpel, dental tools and micro air abrasion depending on how thick and hard the corrosion layer is and on the robustness of the artefact. The shape of the artefact can be found underneath the outer corrosion layer, in the denser layer of magnetite. Controlled mechanical cleaning is used to expose this layer and thereby the artefact's shape (Selwyn and Argyropoulos 2005, p. 85).

The goal of mechanical cleaning is not to remove all of the corrosion layers but to reveal the artefacts original surface. Mechanical cleaning offers a much greater control of how much of the corrosion layer is removed than treatments such as electrolysis or plasma reduction, as the conservator can estimate where the original surface is and can judge whether mineralized organic inclusions in the corrosion layer should be left behind. This process cannot be undone, so great care should be taken when cleaning the artefact. In fact, Cronyn compares this stage to archaeology, because material is permanently removed and documented to reveal structures below (Cronyn 1990, p. 63).

Despite the fact that mechanical cleaning has many advantages, it only results in a minimal stabilizing effect on the iron, so it can only be used in combination with chloride removal treatment. Another drawback is the fact that in removing the corrosion layer, sources of metallurgic information can be lost.

The most common tools in dry mechanical cleaning are scalpels, pincers, soft and stiff brushes (animal or synthetic hair), fibreglass brushes and dental picks. This type of cleaning is often done under a magnifying glass or a microscope. Electrical tools, such as microgrinding wheels and micro air abrasion are often used in cleaning artefacts, but do not offer as much control as the purely manual techniques (scalpels and brushes). In micro air abrasion aluminium oxide, Al₂O₃ or micro glass beads are most often used as the abrasion medium. Artefacts are often X-rayed prior to mechanical cleaning for better information on the artefacts condition underneath the corrosion layer and for clues about the original surface (Watkinson 2010, p. 3310).

Original surface has been defined as the limit between the materials that comprise the artefact and the surrounding soil when the artefact was buried, before the corrosion process began. When the artefact corrodes, its surface changes but it is still possible to find the limit of the original surface (called limitos) within the corrosion layer. Corrosion layers located under the limitos are recognizable because they contain slag inclusions. Those corrosion layers located above the limitos are recognized by the presence of soil minerals (Neff et al. 2004, p. 740).

5.2 Immersion Treatments

The aim of immersion treatments, as with all desalination treatments, is to remove as much of the chloride ions as possible, as chloride is the major corrosion accelerator in archaeological iron (Watkinson 1996, p. 208). Chloride removal from artefacts has become one of the biggest challenges in conservation for the last hundred years. The emphasis of chloride removal has increased even more since Turgoose demonstrated that they could have effect at an RH as low as 20% post-excavation (Turgoose 1982, p. 97). Many methods have been used to try to remove chloride from iron artefacts, including electrolysis, hot washing, and plasma treatment. The method most commonly used today is chemical desalination using alkaline solutions (Rimmer and Wang 2010, p. 79).

Studies have shown that chlorinated corrosion phases of iron artefacts can form in the presence of very low chloride levels in the surrounding environment (Reguer et al. 2009, p. 2795). This causes increased degradation of the artefacts that need highly controlled environments to be stable. However, if enough Cl⁻ ions are removed, the artefacts should be able to resist corrosion when stored or displayed in a controlled museum environment without special storage conditions, according to Selwyn. Iron artefacts from archaeological contexts have a much higher rate of survival if treated with some kind of desalination treatment than those artefacts that are left untreated (Selwyn 2004, p. 298).

Immersion treatments of archaeological iron involve placing the artefact in an aqueous solution for the Cl⁻ ions to diffuse out. Usually the solution used has a pH close to neutral or alkaline. Research has shown that the washing of iron artefacts in desalinated water is not an effective stabilizing treatment because the Cl⁻ ions are trapped in the lattice of akaganéite and in micro-cracks (Scott and Seely 1987, p. 73; Selwyn and Logan 1993, s. 806). Even though the method preserves information about the metallurgy and other information that could be found in the corrosion layers, the stabilizing effect is so minimal that the negatives outweigh the positives (Scott and Seeley 1987, s. 73). Archaeological iron that is partially mineralized and cracked, cannot withstand treatment in strongly reducing solutions, like alkaline sulphite and such material can disintegrate during treatment. There is, as yet, no ideal treatment for highly mineralized iron (Scott and Seely 1987, p. 73). Fully mineralized artefacts do however not need chloride extraction treatment, as they are unlikely to corrode further because no metal is left in the artefact (Watkinson 1983, p. 89).

There are two key factors that influence the ability of dissolved Cl⁻ ions to diffuse out of archaeological iron, whether the iron metal is continuing to corrode and if the corrosion layer is porous. The Cl⁻ ions will diffuse into the solution if the corrosion can be stopped, and increased porosity will make the diffusion easier (Selwyn 2004, p. 298). Porosity can be increased by placing artefacts in alkaline solutions, as many inorganic and organic materials found in corrosion layers are more soluble in alkaline solutions than neutral ones. Greasy dirt, fatty compounds, oils, cellulose and protein are broken down into water-soluble compounds in alkaline solutions and quartz becomes more soluble in pH above 9 (Selwyn 2004, p. 300).

Determining the amount of chloride left within the artefact after treatment is difficult. It requires the digestion of artefacts after the treatment is completed, destroying the artefact in the process. As a result there is a limited amount of information available with regard to the amount of chlorides left after desalination treatment. The small amount of data available suggests that some desalination treatments are considerably better than others and extract a consistent amount of chloride. Chloride extracted into the solution. The problem with this method is that it does not guarantee that the artefact is chloride free (Watkinson 2010, p. 3319). Watkinson claims that to guarantee that iron treated with desalination methods does not corrode post-treatment it should be stored in a controlled environment to the same standard as is used for untreated iron. This makes storage costs the same as for both categories (Watkinson 2010, p. 3318-3319), which negates one of the greater advantages of desalination treatment, i.e. that it does not need special storage condition as claimed by Selwyn (2004, p. 298).

5.3 Sodium Hydroxide

As stated above, desalination treatments with water alone are not considered an effective stabilization treatment for archaeological iron. One of the most common treatments for archaeological iron is an aqueous sodium hydroxide solution. The concentrations most often used are 0.1M to 0.5M, with the pH of 13–14. Sodium hydroxide, NaOH is fairly cheap and readily available and has been shown to be highly effective in removing Cl⁻ ions from

archaeological iron (North and Pearson 1978, p. 183; Selwyn 2004, p. 299). This is why it is one of the most widely used stabilization treatments for archaeological iron.

One of the reasons sodium hydroxide is such a successful method is its high pH. High pH passivates the iron and reduces the corrosion rate, as an adherent layer of insoluble corrosion products forms on the iron. When iron corrodes in an alkaline solution, Fe^{2+} ions precipitate as $Fe(OH)_2$ which is then easily oxidized and hydrolyzed to an insoluble film of $Fe(OH)_3$. If this corrosion film is formed in direct contact with the iron surface, it can prevent the transfer of Fe^{2+} ions from the iron metal to the solution, preventing further corrosion (Selwyn 2004, p. 298).

Such a passivating layer of insoluble corrosion products will also contribute to a more efficient desalination, as active corrosion would greatly limit the diffusion of chloride ions by those attracted by the Fe^{2+} ions produced during the corrosion process. Generally, the corrosion rate is almost negligible if the pH of the solution is over 12. The limitation of this model is that very high concentrations of chloride ions will be able to prevent the formation of a passivating layer (Selwyn 2004, p. 298).

There are further drawbacks to the method. Turgoose points out that the passivation process can fail if the corrosion layer is very thick, as this will prevent OH- ions to be present in a high enough concentration simultaneously on the artefact's whole surface (Turgoose 1985, p. 15). Treatment efficiency will therefore be dependent on the chloride levels in the artefact and to some extent on the thickness of the corrosion layer. This issue can be solved by mechanical cleaning prior to desalination treatment, but that is not always an option as it can remove information about the artefact found in the corrosion layer, such as organic or mineralized organic matter. As no perfect stabilization treatment has been found for archaeological iron, sodium hydroxide treatment is generally considered an effective treatment that improves the stability of iron artefacts (Costain 2000, p. 18).

When it comes to the potential metallurgic information that can be obtained from the artefact it can be argued that sodium hydroxide treatment is a less invasive treatment than both plasma treatment and electrolysis (see chapters 5.4 and 5.5), as the treatment does not change the metallographic structure or nor is it likely to remove the original surface. The treatment's effects on possible organic or mineralized organic matter found in the corrosion layer is more uncertain, as there has been relatively little research done on this aspect. In general it is assumed that strong alkaline solutions are not good for organic materials (Knight 1997, p. 38). The increase in porosity that alkaline solutions, such as sodium hydroxide causes, can be unfortunate in this context, as this often leads to increased brittleness and cracks in the corrosion layers (Selwyn 2004, p. 301).

It has been pointed out that sodium hydroxide is unlikely to break down akaganéite, as research has shown that akaganéite needs to be heated up to 200°C to transform or release chloride (Stål et al. 2003, p. 2574). Watkinson however claims that solid ferrous chloride and surface adsorbed chloride on akaganéite dissolve in sodium hydroxide. This makes the akaganéite less hygroscopic and prevents post-treatment corrosion of the iron. Akaganéite can transform and release chlorides into the wash solution in solutions with high pH. As the ionic size of chloride trapped in tunnels within akaganéite is larger than the entrances of the tunnel, the chloride remains trapped. It therefore presents no corrosion threat post-treatment, unless conversion of akaganéite to goethite releases this chloride. The hydrolysis of akaganéite to goethite can occur post-treatment but very little research has been done on this topic (Watkinson 2010, p. 3318-3319).

5.3.1 Alkaline Sulphite

In the alkaline sulphite reduction treatment, sodium hydroxide, NaOH and sodium sulphite, Na₂SO₃ are mixed in an aqueous solution. The treatment requires sealed containers in order

to reduce the oxygen. The original method was first described by North and Pearson in 1975 and used an aqueous solution of 0.5M sodium hydroxide and 0.5M sodium sulphite, heated to 70°C (North and Pearson 1975). Later modifications to the treatment have included lower concentrations with 0.1M sodium hydroxide and 0.05M sodium sulphite and room temperature treatment (Rimmer and Watkinson 2010, p. 17).

Alkaline sulphite treatment is based on the same principals as desalination with sodium hydroxide. It is however also expected to improve the latter treatment, as the addition of sodium sulphite deoxygenates the solution. Fe^{2+} ions in the artefact react with OH- ions in the solution to produce insoluble Iron(II) hydroxide that later oxidizes to iron oxides (Rimmer and Watkinson 2010, p. 17).

It has long been considered necessary for artefacts to be washed after alkaline sulphite treatment to remove chemical residues from treatment that could increase the risk of corrosion post-treatment (Schmutzler and Ebinger-Rist 2008, p. 251). Rimmer and Watkinson have however concluded that this procedure is unnecessary. It increases treatment time, causes iron to corrode in the oxygenated aqueous wash solutions and has an unknown efficiency. Barium hydroxide rinsing has been used for this purpose, in an attempt to immobilize SO₃^{2-/}SO₄²⁻ ions as BaSO₄. This has been shown to produce a white residue and it is unknown how effective the rinsing is (Keene 1994, p. 263; Rimmer and Watkinson 2010, p. 16). Rimmer and Watkinson believe that rinsing is not necessary after the treatment as their research showed that residual sulphates did not cause increased iron corrosion below 75% RH (Rimmer and Watkinson 2010, p. 17).

Various researchers have concluded that alkaline sulphite treatment is a highly effective treatment for the stabilization of archaeological iron, to an even greater extent than sodium hydroxide treatment (Selwyn and Logan 1993, p. 805; Wang 2007, p. 132). The main reason for the success of the alkaline sulphite treatment is thought to be the precipitation of fresh iron compounds, such as ferric hydroxide. It has been pointed out that even though the method has advantages over other desalination treatments, it also has significant disadvantages as the reduction of Fe(III) to Fe(II,III) compounds can only occur when there is an excess of Fe₂₊ ions present, such as in artefacts from marine archaeological environment and artefacts that have been recently excavated (Selwyn 2004, p. 301; Schmutzler and Ebinger-Rist 2008, p. 251). The artefact must have been kept wet at all times and have a thick corrosion layer. When iron that meets these standards is treated with alkaline sulphite, some of the iron oxyhydroxides within the artefact may be converted to magnetite, thereby opening up the corrosion layer's pore structure and allowing faster diffusion of the Cl⁻ ions into the treatment solution (Selwyn 2004, p. 301). On artefacts from a non-marine context or dry artefacts this precipitation of fresh iron compounds does not occur. This then acts as a diffusion barrier for chloride ions (Schmutzler and Ebinger-Rist 2008, p. 251).

Alkaline sulphite is a more aggressive treatment than sodium hydroxide, as it can damage original surfaces (Keene 1994, p. 258). Highly mineralized artefacts will become soft in the alkaline sulphite solution, and fragile, heavily mineralized artefacts can disintegrate (Keene 1994, p. 262). This can have a detrimental effect on all the mineralized organic matter preserved in the corrosion layers. All artefacts should be X-rayed prior to alkaline sulphite treatment in order to sort out highly mineralized artefacts and exclude them from treatment, as they are too fragile to withstand it (Argyropoulos et al. 1997, p. 154). They are also unlikely to corrode further post-excavation (Watkinson 1983, p. 89). This applies to sodium hydroxide treatment as well. Heavily mineralized artefacts should only be in alkaline solutions long enough to remove as much of the chlorides as possible without damage to the shape of the artefact (Argyropoulos et al. 1997, p. 154).

Schmutzler and Ebinger-Rist main argument against the alkaline sulphite treatment is the fact that the setup of an alkaline sulphite desalination facility is very time consuming and

the equipment and operating system are expensive (Schmutzler and Ebinger-Rist 2008, p. 252).

It can be concluded that the even though researchers have shown that alkaline sulphite treatment is a very effective treatment for some types of iron artefacts, it is not ideal for material that has been in dry storage for a long period of time, such as the Reyðarfell material and most of the iron artefacts in the National Museum of Iceland.

5.4 Electrolytic Cleaning

Electrolytic cleaning is nearly the reverse of the process that occurs when electrochemical corrosion takes place. It involves an electric current where the voltage is supplied by an external source, such as a battery or a similar effect can be achieved by mixing chemicals. When no electrical source is used the process is known as electrochemical cleaning (Cronyn 1990, p. 174-175).

In electrolytic cleaning the artefact functions as a cathode and is connected to the negative terminal of a low-voltage direct current source. An inert metal (e.g. stainless steel) functions as an anode and is connected to the positive terminal. Both the anode and the cathode (the artefact) are immersed in an electrolyte, such as NaOH (Cronyn 1990, p. 174-175). An imposed current protects the artefact cathodically and frees chloride from its counter-ion role. This allows the chlorides to diffuse out of the artefact and they are drawn to the inert anode. A reduction of iron oxide to magnetite can occur which increases the porosity of the corrosion layer, and is believed to facilitate chloride diffusion (Watkinson 2010, p. 3321). The porosity increases because the molar volume (per mole of iron) of magnetite is smaller than the molar volumes of the iron oxyhydroxides: goethite, akaganéite, and lepidocrocite. The smaller volume occupied by magnetite means a greater pore space in the corrosion layer (Selwyn 2004, 301).

Electrolysis can be used to either to entirely remove corrosion products from an artefact or to aid the diffusion of soluble corrosion, while retaining corrosion layers on the metal (Watkinson 2010, p. 3321). The complete removal of corrosion products is no longer widely done in archaeological conservation. However, early electrochemical methods that were used in conservation were developed in the 19th century and were very radical. These removed all the corrosion products, leaving only the clean metal surface. Later the importance of the corrosion products was recognized as source of information of e.g. the artefacts original surface or decorations. These extreme electrolysis treatments were abandoned in the 1970's and 80's (Cano et al. 2010, p. 385).

Whether the presence of an electric field has any major effect on the rate at which the chloride ions diffuse out has been called into question. Studies have shown that the presence of the electric field has an insignificant effect on the rate of the removal of chloride ion. This is claimed to be due to the fact that the negative component of the current is carried mainly by the OH- ions rather than Cl⁻ ions (Selwyn 2004, p. 299).

Electrolytic treatments, like most chloride removal treatments need to run for long periods of time. They are more often used for larger artefacts, as electrolytic treatments need little input from the conservator after the initial process, the artefact remains cathodically protected and the treatment is not labour intensive in the same way as, e.g. sodium hydroxide treatment (Watkinson 2010, p. 3321).

Quantitative measurement has shown that electrolysis in NaOH solution is no better at extracting chloride than aqueous washing in NaOH solution, but there are no quantitative measurements for the post-treatment stability of artefacts treated with electrolysis (Watkinson 2010, p. 3322). Conservators have often discarded electrolytic techniques as they are considered as too complicated, as some basic knowledge on electrochemical parameters and processes is required. Another factor that deters conservators from using the technique is that

the facilities required for electrolysis are not as easy to access or to set up as in e.g. alkaline washing (Degrigny 2010, p. 353; 360).

5.5 Plasma Reduction

Gas plasma reduction is a heat assisted reduction process. In its present form the method has been used to aid chloride removal from iron by reducing iron oxide to magnetite in order to produce a more porous corrosion layer facilitating the removal of chloride in subsequent aqueous alkaline treatments (Watkinson 2010, p. 3324). Plasma reduction is similar to electrolysis as it aims to stabilize iron artefacts though a reduction process. While the reduction in the electrolysis takes place in an aqueous electrolyte, hydrogen gas acts as the electrolyte in plasma reduction (Sjøgren and Buchwald 1991, p. 161). The artefact becomes negatively charged in the plasma and this causes positive ions to bombard the artefact' s surface. As the ions neutralise in the atomic hydrogen they act as a strong reducing agent and the corrosion products can be chemically reduced to a lower oxidation state (Schmidt-Ott 2004, p. 235-236). Akaganéite, goethite and maghemite are reduced to magnetite during treatment, which increases the porosity of the corrosion layer and chloride ions can be removed completely (Sjøgren and Buchwald 1991, p. 166).

When gas plasma reduction was first applied to archaeological iron, temperatures up to 400°C were used. To remove chloride ions completely from an artefact treatment temperature needs to 300–400 °C for eight days with low-pressure hydrogen plasma. Such high temperatures can have a detrimental effect on the metallographic information in the metal core. After treatment at such high temperatures it will no longer be possible to make interpretations about grain characteristics, carbon content or welding technology on the artefact (Tylecote and Black 1980, p. 95). Mineralized organic matter may be reduced during treatment, making it very brittle and at worst impossible to interpret (Schmidt-Ott and Boissonnas 2002, p. 84). Another aspect that has raised questions is the method's impact on the location of the original surface, as the corrosion products reduce to magnetite, making the original surface definition difficult to distinguish, and a lack of surface contours can be expected on X-ray images (Tylecote and Black 1991, p. 92).

The plasma reduction method has been developed in recent years and adapted so that metallurgic information can be saved. Previously a mixture of hydrogen and argon was used as an electrolyte in plasma reduction, with the argon being used to stabilize the plasma and to enhance the effect of the surface interaction. Tests have shown that excluding the argon and using pure hydrogen plasma instead at low pressure, reduces the iron artefact's temperature to about 80°C during treatment. The addition of argon causes the artefact to become warmer during reduction, so this observation improved the method in regards to lowering the temperature and decreasing the risk of a change of the metallographic information contained in the artefact. The complete removal of chlorides is unfortunately not possible with plasma reduction treatment and subsequent desalination in alkaline sulphite is necessary (Schmidt-Ott 2004, p. 236).

5.6 Protective Coatings and Consolidants

The aim of coatings in conservation is mostly to protect the artefact from atmospheric humidity and corrosive salts that can be deposited on the artefact during handling. Coatings can prevent fresh corrosion of metal and most treatments of archaeological iron, such as immersion treatment and plasma-reduction, are followed by some type of coating or consolidation (Keene 1984, p. 104).

The materials that are most often used on archaeological iron presently are microcrystalline wax, Paraloid B-72 and Paraloid B44. Previously polyvinyl acetate,

cellulose nitrate, beeswax and paraffin wax were the most common coatings and consolidants but they have now fallen out of favour (Keene 1984, p. 104; Moffett 1996, p. 4; Wang 2007, p. 131; Jaeger 2008; p. 218). Charcoal was often mixed in with the beeswax or paraffin wax to create a more uniform surface (Wang 2007, p. 131). Anti-corrosion agents are also used, often in conjunction with coatings.

5.6.1 Microcrystalline Wax

Waxes have previously been used on artefacts that had not been desalinated to prevent corrosion by excluding atmospheric water vapour (Keene 1984, p. 104). This method has a long history in the conservation of iron and paraffin wax has been used for more than a hundred years as a protective coating on archaeological iron (Horie 2010, p. 128). It has now mostly been replaced by microcrystalline wax.

Microcrystalline wax is made from fractions that are derived from the distillation of petroleum. They come in various formulas that have different melting points, ranging between 52°C and 57°C (Mills and White 1999, p. 53). Microcrystalline wax is composed of mainly irregular molecules that are highly branched and are larger than paraffin waxes. Microcrystalline waxes are less brittle than paraffin waxes and a mixture of the two is stronger than either separately (Horie 2010, p. 125). Microcrystalline wax is not soluble in polar solvents and it is a fairly un-reactive substance (Johnson 1984, p. 107).

Wax coating is usually done by immersing the artefact in molten wax and is often done in vacuum, as it minimizes the chance of air pockets forming and reduces their size if they do form. Using vacuum also ensures the maximum penetration of the wax. Microcrystalline wax can also be applied locally using a hot-air blower or heated spatula (Jaeger 2008, p. 218).

Waxes are no longer considered ideal in conservation as they photooxidize, causing cross-linking, embrittlement, and discoloration in the wax. Microcrystalline wax is more susceptible than paraffin wax in this regard (Horie 2010, p. 126). Wax coatings can also attract dust and lint (Jaeger 2008, p. 218) and obscure details found on the artefacts surface (Moffett 1996, p. 6). They are also incompatible with most adhesives (Horie 2010, p. 259). Additionally, studies have shown that wax coatings to be ineffective at stabilizing archaeological iron (Johnson 1984, p. 107; Keene 1984, p. 105).

Even though coatings are a still a necessary part of the treatment of iron artefacts, they do have their drawbacks. If applied to porous corrosion layers, they can become very hard to remove, should the artefact start to actively re-corrode. This applies especially to waxes, as they are particularly difficult to remove from heavily corroded iron surfaces. Therefore CCI does not normally recommend the use of waxes on rusted iron (Logan 2007 c), p. 3). The National Heritage Board in Sweden compared the performance of microcrystalline wax to 14 corrosion inhibitors and the results showed that it was one of the three coatings that performed the worst in the corrosion test (Norlander et al. 2007, p. 11; 54).

5.6.2 Paraloid B-44 and B-72

As was stated above, protective coatings are still necessary, as artefacts cannot always be stored under ideal conditions. More reversible protective coatings than microcrystalline wax e.g. Paraloid B-44 or B-72, are being used today, with B-72 being the more common of the two.

Paraloid B-72 is a thermoplastic acrylic resin (a co-polymer of methyl acrylate and ethyl methacrylate) and is usually applied on the artefacts after desalination. It is soluble in a wide range of organic solvents, but acetone and toluene are most often used. Paraloid B-72 possesses a high tolerance for ethanol, and up to 40% ethanol can be added to the solution of Paraloid B-72 and acetone or toluene. This improves the control over the drying time of the solution (Hamilton 1999, p. 9).

Schmitt-Ott concluded that Paraloid B-44 was better suited than B-72 for artefacts treated with plasma reduction, as it is successful as a protective coating for mineralized organic remains during desalination treatments with alkaline sulphite solutions. Paraloid B-72 has a lower glass transition temperature⁹ (Tg) of 40°C than B-44 that has a Tg of 60°C. Paraloid B-72 does therefore not provide the necessary protection in baths heated to a temperature of 50°C. Paraloid B-44 does however have a glossy appearance but Schmitt-Ott states that matt surface appearance can be achieved by using toluene as a solvent (Schmitt-Ott 2002, p. 84). Studies have shown that Paraloid B-44 retains at a more stable pH over time than Paraloid B-72, which has a tendency to become more acidic and oxidize (Down et al. 1996, p. 27). Even so, it does so very slowly and has become the standard of stability (Horie 2010, p. 159). Recent testing comparing Paraloid B-72 to other coatings on various metals showed that there is no need to replace Paraloid B-72 as a coating for iron artefacts, because of its well-proven reversibility and how familiar conservators are with its effects and uses (Wolfram et al. 2010, p. 172).

5.7 Anti-corrosion Agents

Anti-corrosion agents act by serving as a moisture barrier, oxygen barrier or by containg substances that inhibit electrochemical reactions, i.e. they prevent anodic or cathodic processes (Norlander et al. 2007, p. 8). Anodic inhibitors slow the anodic reaction, iron corrosion, and cathodic inhibitors slow the cathodic reaction, oxygen reduction. For the inhibitor to work it has to reach the area where the anodic or cathodic reactions occur. Therefore, anodic inhibitors need to reach the metal surface and cathodic inhibitors need to reach the electronic conductors making contact with the corroding iron, e.g. magnetite, a conducting corrosion product (Selwyn 2004, p. 300).

Tannic acid is an anodic iron corrosion inhibitor and has been used at the National Museum of Iceland. Tannic acid is an organic acid found in most plants. The tannic acid reacts with the iron, forming a somewhat porous blue-black film of ferric tannate. Tannic acid will inhibit the artefacts most vulnerable areas from reacting with water vapour for a limited amount of time (Logan 2007 b), p. 1). Tannic acid is applied onto the artefact by immersion in tannin solutions or with the more common method of painting the artefact with it (Watkinson 2010, 3334).

The ferric tannate film created by tannic acid does not provide the artefact with a vapour barrier. It will delay corrosion by forming a protective film over iron, but this effect will not last indefinitely. The ferric tannate film can last longer if the artefact is stored in a stable RH of around 50%. Wearing gloves during handling the artefact and protecting the surface from dust and abrasion also prolongs the films life. Ferric tannate can rub off onto other materials it is in contact with. Artefacts treated with tannic acid should therefore not be in contact with artefacts that stain easily, such as paper, textiles, leather, bone, and wood. Wax coating after tannic acid treatment can reduce this problem and the wax will also provide a vapour barrier and will in addition consolidate the corrosion layers on the metal (Logan 2007 b), p. 3).

The National Heritage Board in Sweden conducted a project with the aim of finding a suitable corrosion inhibitor for archaeological and ethnological iron. The results showed that for the treatment to be effective on archaeological iron it needs to be desalinated before being treated with corrosion inhibitors. Inhibitors that remained sticky for a long time after application fared best during the tests. Dinitrol paste was the most successful of the 14

⁹ Glass transition temperature is the transition of amorphous materials from a hard and brittle state into a softer, flaccid state. Softer coatings attract dust particles and do not provide the protection intended with the treatment (Horie 2010, p. 24-26).

inhibitors tested.¹⁰ Corrosion inhibitors that dried quickly and had a hard, dry surface fared the worst (Norlander et al. 2007, p. 53-53).

The problem with Dinotrol paste is that sticky corrosion inhibitors attract dust particles, which in turn attract moisture. Dinotrol paste dries very slowly and could spread to packaging and other objects through handling. Both of the anti-corrosion agents discussed here have the disadvantage of being able to rub off onto other materials, which could cause problems in regards to storage. Tannic acid treated artefacts will still need controlled storage, and both Dinitrol paste and tannic acid are most commonly applied by brush individually so they are not practical in regards to mass-conservation.

5.8 Preventive conservation

Preventive conservation means choosing suitable storage facilities and ensuring that the climate is controlled and suitable for artefacts storage. Climate control means in particular that the relative humidity can be controlled so that it suits a particular material. For metals, this means that the climate should be kept as dry as possible, RH should not exceed 50% and fluctuations in both temperature and RH should be kept at a minimum (Norlander 2007, p. 19).

Ambient humidity can supply moisture to solvate ions within corroded metals and create electrolytes. It is well known that hygroscopic salts such as sodium chloride can lower the RH threshold for corrosion to occur in artefacts. Surface adsorbed chloride found on hygroscopic akaganéite becomes mobile within atmospheric moisture and can corrode iron that it is in contact with at 15% RH. Studies have shown that for chloride infested iron stored at 20°C, no corrosion occurs at 12% RH, akaganéite causes slow corrosion of iron at 15–20% RH and above 21% RH both akaganéite and iron(II) chloride tetrahydride, FeCl2•4H2O contribute to corrosion (Watkinson 2010, p. 3314-3315).

Controlling humidity is therefore very important in preventive conservation. Using either active dehumidification mechanical plants or passive desiccation using desiccants like silica gel in storage boxes or display areas, is recommended. Controlling corrosion by desiccation has significant advantages, as they remain easily accessible, especially if an entire storeroom is desiccated as opposed to enclosed individual boxes. Using a mechanical desiccation plant has the disadvantage of being demanding with regard to both finance and energy, as opposed to small-scale control of climate by silica gel in individual boxes. These require more attention of museum staff though as the gradual hydration of the silica gel will eventually raise the RH within the box to values that could cause corrosion. This corrosion continues to increase as relative humidity in the box rises, until it reaches equilibrium with the external environment. When this happens the artefacts lie in a continually aggressive environment, just like they would in an uncontrolled storage (Watkinson 2010, p. 3315). Using desiccated boxes requires diligence in monitoring the RH of the boxes from the conservator and storage personnel.

Regularly inspecting artefacts for renewed corrosion is therefore a vital part of their care. Because many corrosion reactions occur quickly, the CCI recommends that metal artefacts should be inspected monthly. Deterioration can then be noted at an early stage and preventive measures taken before damage becomes severe (Logan 2007 a), p. 3). This is hardly a realistic goal in a museum storage where tens of thousands of artefacts are stored, such as in the National Museum of Iceland's stores.

¹⁰ 15 coatings were tested in all. However, one those coatings was microcrystalline wax, which does not have corrosion inhibitive qualities (Norlander et al. 2007, p. 11; 54).

Although preventive conservation is an indispensible part of artefact maintenance it does not replace active treatment, which appears to have a much greater effect than does controlled storage at low relative humidity (Keene 1994, p. 259).

Preventive conservation extends further than to just climate control. Careless handling of corroded artefacts can cause considerable damage. Some archaeological metal artefacts can be fully mineralized and are therefore very fragile. If such an artefact is stored in a too small box, so that a part hangs out or if it is lifted at one end, there is a risk that the artefact can break under its own weight. Using gloves is also an important part of preventive conservation, as sweat contains chlorides and caustic substances such as ammonia (Fjæstad 1999, p. 72).

6 Application of Treatment in Mass-conservation

6.1 Methods in Mass-conservation

Some archaeological sites, mostly ones from historic periods, can yield thousands of artefacts. A large amount of these finds are often smaller iron artefacts, such as nails, rivets and decorations. Another large material category is leather and excavations at medieval sites in particular are well known to result in large amounts of leather artefacts. In order to minimize costs and increase time efficiency so-called 'bulk treatments' have been developed to treat large material categories. These methods are also called 'batch treatments' or 'batch processing' in the literature, but will be mostly be referred to as 'mass-conservation' in this thesis.

CCI has been influential in developing methods for dealing with large quantities of finds after they began working with archaeologists in Newfoundland and Labrador where sites producing large quantities of well preserved finds were being excavated, starting in 1978 (Mathias 1993, p. 313).

Treating artefacts individually results in better control and closer monitoring during treatment and is the ideal. However, this is not always possible, and if mass-conservation is necessary, the artefacts must be sorted by their degree of mineralization, so that similar types of artefacts from the same site can be treated together for the best results. Artefacts that are treated in the same container should never be in direct contact with each other and should be separated by plastic dividers or packed in some sort of netting or gauze. This is also important should they fall apart or parts of them come loose in treatment. That way no parts of the artefact will be lost and the artefact can possibly be reassembled after treatment. Each artefact must be clearly labelled using material resistant to the chemicals it will be subjected to during treatment. The same applies to materials used to fasten the labels (Argyropoulos et al. 1997, p. 154). Argyropoulos et al. recommend using Teflon strips fastened by polyethylene coated wire twist ties (1997, p. 154).

Immersion treatments are a quite common method used in mass-conservation, as they are easily adapted to larger quantities of artefacts. Both sodium hydroxide treatment and alkaline sulphite treatment are common mass-conservation treatments and studies by Mathias et al. support that sodium hydroxide is a suitable tool for use in mass-conservation (Mathias et al. 2004, p. 32).

Studio Västsvensk Konservering (SVK) in Gothenburg where I was an intern in the fall of 2011 and gave me some insight into the methods they use. SVK does not actively use mass-conservation per se, but they do perform some stages of the treatment in bulk, such as desalination, dehydration and wax coating. For desalination they use sodium hydroxide for treating terrestrial iron and for most marine artefacts, but in some cases they have used alkaline sulphite for marine artefacts. The artefacts treated with sodium hydroxide are placed in clear hard plastic containers (ca. 30x20x5 cm) that can be divided up and the site and number of each artefact is written on the container for identification. The shape of each artefact is drawn on the lid of the container for further identification. Every artefact is Xrayed before treatment.

In Visby the Swedish National Heritage Board has a mass-conservation project, Projekt Arkeologisk Konservering (PAK), with over 300.000 artefacts that must be treated (*Riksantikvarieämbetet*, 2010). Not all of the artefacts are iron artefacts but a large proportion is. Like with SVK I got some insight into the methods used by the PAK team. They use deionised water deoxygenated by nitrogen gas (N_2) for desalination in a large container that is hooked up to a supply of N_2 . The artefacts are placed on steel trays and packed in tubes of

netting, separated by knots, strings or plastic clips. Each tube belongs to one site and is labelled by site. Smaller and more fragile artefacts are packed in fine meshed mosquito netting, to reduce the risk of parts falling out. Each artefact is photocopied¹¹ prior to treatment for identification after treatment as the artefacts are only labelled by site and also in case the label falls off during treatment.

Both in SVK and in the PAK project artefacts are mechanically cleaned prior to desalination, but mass-conservation is not possible in those cases, as each artefact must be cleaned individually. Most artefacts can be cleaned using micro-grinding wheels and micro air abrasion, which significantly speeds up the cleaning process, as opposed to cleaning the artefact with a scalpel.

After desalination the artefacts are dehydrated in ethanol and coated in microcrystalline wax in batches in both SVK and PAK. Wax coating is done by immersion in hot wax under vacuum. SVK coats their artefacts with Dinitrol inhibitor prior to waxing by brushing it on, which must be done on each artefact individually. Waxing is practical for use in mass-conservation as it can be done by immersion in batches. That way the artefacts can be coated in batches and the process sped up. Excess wax can be removed by using a hot air gun after immersion. Some other coatings can also be done by immersion in vacuum, such as Paraloid B-44 or B-72 (Wolfram 2010, p. 170-171). These types of coatings are however most often applied by brushing them on the artefact.

Due to the vast amount of artefacts involved in the PAK project they have chosen a very drastic mass-conservation route. That is, they have adapted most of the steps involved in the treatment process to mass-conservation. They have, e.g. created a registration method that speeds up the documentation process. They use a file created in Excel where the basic information about the artefact is put, i.e. material, site etc. and use a checklist in the same file to register the artefacts condition. They only X-ray a few selected artefacts and have a digital X-ray, which speeds up the process significantly. Only artefacts that need specific treatment are photographed and when there are artefacts that need specific treatment, the team's methods are adapted to that artefact and it is given the time it needs. Even though the methods are highly adapted to mass-conservation, there are still parts of treatment that are done on each artefact individually, such as mechanical cleaning.

6.2 Suggested Treatment of the Reyðarfell Artefacts

6.2.1 Condition

In the table below a plan of the re-conservation of the Reyðarfell artefacts can be seen. 90% of the artefacts from Reyðarfell, and according to Gestsson's rapport from 1971, a large portion of the artefacts at the National Museum, have been coated with paraffin wax. This in fact, appears to have stabilized many of the artefacts as can be seen when they are compared to the un-conserved artefacts form Reyðarfell. 78% of the un-conserved artefacts from Reyðarfell were graded 4-5, meaning that they are considered unstable. 9 artefacts had not been conserved and 7 of those were judged to be unstable. The artefacts graded 1-3 and are mostly in stable condition but have not been cleaned of dirt and corrosion products. Because of this, they are not in a condition to be displayed. So, treatment could be an option for these artefacts for aesthetic reasons and for further desalination. But seeing as the artefacts are quite stable, it would not be advised to disrupt their condition.

Artefacts graded 4-5 are however in great danger of completely disintegrating within the span of some years. Akaganéite was identified on 50% of the Reyðarfell iron artefacts,

¹¹ Artefacts are usually not photographed by the PAK team but are placed in a photocopier, protected by a supporting frame of Ethafoam.

which indicates high chloride content, so there is a need to desalinate these artefacts. Desalination is a very good method to use in mass-conservation and has been used for 30 years in treating large quantities of finds (Logan 1984).



Figures 10 and 11. Most of the artefacts treated with varnish are in a poor condition. An example can be seen on the left in figure 11 (artefact no. 1961-131:80). Many of the artefacts treated with wax are also in a very unstable condition (artefact no. 1960-72:2).

In this context it is therefore very urgent to estimate the condition of all the older iron artefacts in archaeological storage at the National Museum. This step is already in progress, and is being done parallel to the reorganisation work that is being done in the storage, as mentioned in chapter 1.1. The artefacts in need of conservation or re-conservation are put in boxes desiccated with silica gel and marked 'In need of conservation' or 'In need of urgent conservation'. These artefacts are unfortunately not going to the conservation department at this point, as there is only one conservator whose job it is to handle all conservation of exhibit artefacts, new acquisitions, artefacts returned from loan, artefacts going on loan and as well as conserving or re-conserve artefacts in storage.

Grade	Condition	Treatment
1	In good and stable condition and in little or no need of conservation.	No treatment needed.
2	Stable, but has not been cleaned.	Removal of wax or coating with micro air abrasion and removal of corrosion and soil particles with air abrasion and micro-grinding wheels. Immersion treatment with sodium hydroxide. Coat with Paraloid B-72. Not urgent.
3	Signs of deteriorating condition. Conservation needed, but not urgent.	Removal of wax or coating with micro air abrasion and removal of corrosion and soil particles with air abrasion and micro-grinding wheels. Immersion treatment with sodium hydroxide. Coat with Paraloid B-72. Not urgent. Store in dry storage until conservation is possible.
4	In need of conservation	Removal of wax or coating with micro air abrasion and removal of corrosion and soil particles with air abrasion and micro-grinding wheels. Immersion treatment with sodium hydroxide, 0,1 M. Coat with Paraloid B-72. Quite urgent. Store in dry storage until conservation is possible.
5	Very fragile. In urgent need of conservation.	Removal of wax or coating with micro air abrasion and removal of corrosion and soil particles with air abrasion and micro-grinding wheels. Immersion treatment with sodium hydroxide. Coat with Paraloid B-72. Very urgent. Store in dry storage until conservation is possible.

Table 3. Plan of action for conservation, depending on estimated condition of artefact.

6.2.2 Treatment

There are only around 150.000 artefacts¹² in the archaeological storage in the National Museum, and although the Reyðarfell artefacts can never represent all the stored artefacts, they can give an idea of their condition. The artefacts from Reyðarfell have been conserved using the same methods as most of the artefacts in the museum excavated prior to 1971, and most likely prior to 1977, which is the year Gísli Gestsson retired. This is quite a large portion of the artefacts from Reyðarfell need some sort of re-conservation, due to high chloride content. Using Reyðarfell as a yardstick, would mean that around 75.000 artefacts in the National Museum could need some amount of re-conservation.

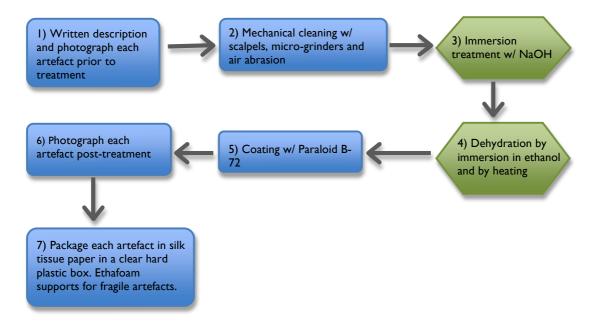


Figure 12. A flowchart showing the seven steps involved in the mass-conservation process recommended in this thesis. The steps shown in rectangles are individual treatment and the two hexagonal ones on the right are steps done by mass-treatment.

The condition of the Reyðarfell iron artefacts is quite varied, as figure 7 on page 22 shows. This would require separate treatment for the various stages of deterioration of the artefacts. Even if this does result in increased time for sorting and evaluating the condition of the artefacts, it is still more effective than the individual treatment of every artefact and mass-conservation is very difficult to apply to all stages of the conservation process. Stages such as documentation can be modified, and before-and-after pictures can be reduced to apply only to the occasional artefact needing special attention. But if mechanical cleaning with scalpels, micro-grinding wheels and micro air-abrasion is to be used, like in this case, it can still only be performed by individual treatment. It is therefore recommended that parts of the treatment in the National Museum of Iceland, such as desalination, will be changed to a mass-conservation setup. That way the process can be sped up and the condition of the artefacts can be evaluated thoroughly.

¹² This number is only a rough estimation as there is no definite information on how many artefacts are in the National Museum's collection. This is due to its database's (Sarpur 2.0) inability to show a complete number of registrations in it. It is also not able to show how many artefacts are behind each registration. This problem is to be resolved in the near future when a new version of the database will be fully functioning. At this point (May 2012) the process of moving information from Sarpur 2.0 to Sarpur 3.0 is ongoing, and is expected to be finished by early June 2012 (Böðvarsson 2012).

All in all are seven stages involved in the mass-conservation process chosen in this thesis. The stages of the conservation process can be seen in a flow chart in figure 12. Step 1 involves the documentation of the artefacts condition with both written descriptions and with photographs prior to any treatment. Adapting the documentation process is not recommended here, because while appropriate in large-scale projects like in Visby, it is not necessary where the artefacts are fewer. Thorough documentation is a very important part of the conservation process and in article 10 in the European Confederation of Conservator-restorers' Organisations's (E.C.C.O.) Guidelines, Code of Ethics states: "The conservation-restoration treatment of cultural heritage should be documented in written and pictorial records of the diagnostic examination, any conservation / restoration intervention and other relevant information. The report should also include the names of all those who have carried out the work" (E.C.C.O., 2002).

In this thesis it is recommended cleaning will be done by mechanical methods and step 2 involves this stage. Should artefacts previously coated with paraffin wax need to be reconserved, the paraffin wax would be removed from the artefacts prior to desalination, otherwise the wax could, in theory, hinder the chloride diffusion out of the corrosion layers. Tests have however shown that waxed artefacts actually yield more chloride than non-waxed artefacts (Johnson 1984). There are nonetheless other reasons for removing wax, such as the fact that it attracts dust, obscures surface details and does not provide an impervious barrier for water and oxygen (Moffett 1996, p. 4; Jaeger 2008, p. 218).

Studies have shown that solvents at elevated temperatures are the most effective way of removing wax coatings (Moffett 1996, p. 5). Other methods, such as boiling water have been tried but found to be unsuccessful (Johnson 1984, p. 107). As heated solvents have proved most successful they were used in an experiment for the mass-treatment of iron artefacts excavated in London and had been treated with microcrystalline wax, without desalination. When it was decided later on to desalinate these artefacts, the conservators were faced with the problem of removing the wax (Johnson 1984, p. 107).

Treating hundreds of artefacts in heated solvents requires specialized equipment and commercial solvent cleaning system was used in treating the London artefacts. A similar method was used with a solvent recycling still on artefacts that were damaged in a 2003 flood in Virginia in the United States of America. This was done as most solvents are toxic and highly flammable, and using them in the amounts needed poses a distinct health and safety hazard (Jaeger 2008, p. 219).

Using these systems is not really an option for the National Museum of Iceland, as that would require buying a solvent cleaning system and the destruction of solvents after treatment would result in further costs for the museum. Jane Hamill, the current conservator at the National Museum, has had to remove wax coatings from artefacts at the museum. Hamill's experience in removing the wax has shown her that micro air abrasion with aluminium oxide at some distance is by far the most effective removal technique (Hamill 2012).

The next stage in the conservation process, step 3, involves immersion treatment with sodium hydroxide. It was chosen here as it has proven to be the most successful method for terrestrial iron and marine artefacts that have been allowed to dry, which applies to all of the artefacts in the National Museum of Iceland. The treatment was also used in the 1990's by the National Museum, so all equipment required for the treatment is available there, which makes it financially the most effective approach.

After the immersion treatment is complete will the artefacts require dehydration, which is step 4 in the process. This will be done both with immersion in ethanol, and by heating in oven at 50°C. These are well known and often used methods for dehydrating archaeological iron (Norlander 2007, p. 39). Step 5 is consolidation by coating and Paraloid B-72 has been

chosen here for availability, effect, reversibility and its proven stability. Microcrystalline wax is not recommended by CCI as it is very difficult to remove from porous surfaces such as corroded iron (Logan 2007 c), p. 3). It can attract dust and lint and disguise surface details, so it is not recommended here. Inhibitors such as tannic acid, which have previously been used by the National Museum of Iceland or Dinitrol, which has been recommended by the Swedish National Heritage Board, could be an option for the National Museum as there is a severe lack of staff to monitor the artefacts condition. The disadvantage of these corrosion inhibitors is that, both can rub off on to other more absorbent material, putting it at risk for discoloration.

Step 6 is connected to step 1, as it is a continuation of the documentation process. It involves photographing the artefacts individually after treatment. Step 7 is the final step and is the repackaging of the artefacts. The current methods used by the National Museum of Iceland will not be changed here and the continued use of clear hard boxes of polystyrene is recommended. Silk tissue paper will be used for support in the boxes and ethafoam supports will be made for fragile artefacts and large artefacts in need of additional support. The boxed artefacts will then be packed into acid-free cardboard boxes.

7 Conclusions

7.1 Conclusions and Discussion

Some archaeological sites can reveal tens of thousands of artefacts. A large amount of these finds are often smaller iron artefacts, such as nails, rivets and decorations. In order to minimize costs and increase time efficiency mass-conservation methods have been developed to treat these large material categories. Immersion treatments are commonly used in mass-conservation, as they are easily adapted to larger quantities of artefacts. Certain stages of the conservation process can however not be adapted to mass-conservation, such as mechanical cleaning. While there are parts of the documentation process that can be cut out in order to adapt to mass-conservation, this should not be done unilaterally but rather be done on case-by-case basis. The artefacts from Reyðarfell were used to get an idea of the condition of artefacts found in the archaeological storage of the National Museum of Iceland. Their condition is quite poor and many of them need desalination, which is very adaptable to mass-conservation. In this thesis, evaluation and suggestions for such action is given.

Although there are many methods available for iron conservation, not all of them are adaptable for mass-conservation and some are too expensive to set-up to be cost-effective, such as electrolysis and plasma reduction. While most of the stages in iron conservation can be adapted to mass-conservation in some way, some cannot, such as mechanical cleaning, which must be done individually. There will also be occasions when artefacts will need specialized treatment and cannot be treated along with others. The ethical standpoints are very important in all conservation and must be considered. The thorough documentation of the conservation process is very important and its importance is emphasized in E.C.C.O.'s guidelines code ethics. From this standpoint it is doubtful that adapting all stages to massconservation would be desirable. While retaining some individual treatment the artefacts can be better sorted and the mass-conservation stages can be better adapted to the artefacts condition.

The adaptation of some stages of treatment process is therefore considered here to be the best outcome for the National Museum of Iceland. There are seven steps involved in the process. These are: photographing before and after treatment: mechanical cleaning with scalpels, micro-grinders and air abrasion, immersion treatment with sodium hydroxide, dehydration with ethanol and finally coating with Paraloid B-72. Each artefact would be packed into a hard clear box of polystyrene with silk tissue paper for support. Ethafoam supports would be made for those artefacts needing additional support. Only two of these seven steps would involve mass-conservation. Those are the immersion treatment and the dehydration process.

The goal by analyzing the Reyðarfell artefacts was to get an idea of the condition of artefacts that are in storage at the National Museum. The results were that the condition is very likely quite varied. The Reyðarfell artefacts were in a diverse range of conditions, even though these artefacts came from a single site and 79% of the artefacts had most likely been conserved with the same treatment (waxed and most likely washed). Artefacts from different sites, which have been treated with other methods, can only add to this variation.

When going through the artefacts from Reyðarfell it was evident that they had been conserved previously, but no records could be found of this treatment. When looking for information about the artefacts in the National Museum's archives, I found a document stating that by 1971 most of the iron artefacts in the museum had been treated by washing them in water and boiling in paraffin wax. This gives us an idea of what type of treatment the artefacts in the storage need, should they need re-treatment. This would apply to all iron

artefacts that came to the museum prior to 1977, as Gestsson, the only person at the museum doing conservation in those years, retired that year. There are some artefacts in storage that have been treated with other methods, such as the artefacts from Reyðarfell that had been coated by varnish. Conservators from the British Museum came to the National Museum in 1969 and 1970 will have been treated some of the artefacts and possibly with different methods, but these artefacts must be considered a minority, judging from Gestsson's statement about paraffin wax in 1971.

If we continue to use the Reyðarfell artefacts as a parameter to get an idea of the condition of the artefacts in storage in the National Museum, we can assume that quite many of them have high chloride content, as 50% of the Reyðarfell artefacts had suffered from akaganéite corrosion, which is an indicator of high chloride content. These artefacts would, if treated, need to have the paraffin wax removed and then be desalinated. At this time the most important step is to estimate the condition of the entire collection and sort out those iron artefacts that are unstable and place them in a desiccated storage. This process has already started, in the re-organisation of the archaeological storage.

The situation in the National Museum of Iceland, where there is little or no documentation about older treatments is not unique and can most likely be found in many other museums around the world. Old sins, old methods that have now been discontinued or revised are a problem for the museum staff of today and will most likely continue to be so. The fact that the National Museum of Iceland is making steps to rectify this situation by instigating a reorganisation of their storages is a very positive development. The new system of sorting out artefacts in need of conservation is especially good, however, what is lacking in this system is integrated collaboration with the museum's conservation team. This is difficult to accomplish as only one archaeological conservator is working at the museum, who has multiple responsibilities on her plate already and adding even more is no solution in the long term. Adding more archaeological conservators to the museum staff is necessary step for a successful reorganisation of the storage. The role of the National Museum is by Icelandic law to collect, catalogue, preserve, conserve and research Icelandic national heritage (*Þjóðminjalög*, 5 §). By financially starving the museum with regards to conservation the purpose of this act not achieved, i.e. artefacts are disintegrating in the storages due to the lack of qualified staff to treat it.

7.2 Summary

Iron, is a chemical element with the symbol Fe and is a relatively unstable metal that corrodes easily. Corrosion involves a transformation of material and metals corrode by oxidation, meaning that the metal atoms give off one or more of the electrons to a substance in the environment, a so-called oxidizing agent, such as oxygen. This process builds up layers of corrosion products; the outer layer of this corrosion crust is a mixture of iron corrosion products cemented with soil particles.

Once excavated the artefact is introduced to a new environment with very different conditions than those in the ground. While the iron is in the ground, chlorides accumulate within and post-excavation corrosion caused by these chlorides is one of the most frequent and serious problems regarding archaeological iron finds. The damage caused by this process is irreversible and can result in the complete loss of the artefacts. The corrosion occurs at the interface between the remaining metal core and the outer corrosion layer. The pressure of the corrosion between these layers causes cracking, flaking, and detachment of the outer corrosion layers. Akaganéite is the most common culprit and is the only corrosion that occurs post-excavation, it is also indicative of high chloride content.

The artefacts that were used as reference material in this thesis were excavated at the medieval farm site of Reyðarfell, in the west of Iceland. The excavation at Reyðarfell was

conducted by the National Museum of Iceland from 1960-1969 and headed by Þorkell Grímsson. There are in total 84 iron artefacts from Reyðarfell at the National Museum. 90% of the iron artefacts had been conserved, mostly with a wax coating or with a varnish. Of the artefacts that had been treated 88% were coated with wax and 12% were coated in varnish. Less than half of the artefacts were judged to be in a stable condition and akaganéite was identified on 50% of the artefacts, making them very susceptible to fluctuations in RH. The archaeological storage facility in the National Museum does not have equipment to control climate. This can result in a serious problem if the RH rises, if many of the artefacts are highly contaminated with chlorides. The museum is in the process of re-organising the archaeological storage and putting unstable metal artefacts in polyethylene boxes desiccated with silica gel.

Today, there are diverse options regarding iron conservation. Conservation has a long history and is a constantly developing field. The earliest written evidence for the conservation of artefacts comes from Pliny the Elder in the first century AD and the foundations of conservation were actually established during the Renaissance. It was, however, the contribution of scientists such as Friedrich Rathgen in Berlin and Gustaf Rosenberg in Copenhagen in the 19th and 20th century that formed the discipline of modern archaeological conservation.

One of the most common methods for cleaning corroded iron today is mechanical cleaning, which can be performed with different tools such as a scalpel, dental tools and with micro air abrasion. Another common treatment is immersion treatment, which aims to remove as much of the chloride ions from the iron artefact as possible, as chloride is the major corrosion accelerator in archaeological iron. Chloride removal from artefacts has become one of the biggest challenges in iron conservation for the last hundred years. The emphasis on this problem has increased further since Turgoose demonstrated that they could have an effect post-excavation at an RH as low as 20%. Many methods have been used to try to remove chloride from iron artefacts, including electrolysis and hot washing. The method most commonly used today is chemical desalination using alkaline solutions, such as sodium hydroxide and alkaline sulphite. Chlorides can also be removed by electrolysis and plasma-reduction, but these methods have not proven as effective and are more expensive.

Coating iron artefacts after desalination treatment is often done with the aim of protecting the artefact from atmospheric humidity and the corrosive salts that can be deposited on the artefact during handling. Coatings can also prevent fresh corrosion of metal and most treatments of archaeological iron, such as immersion treatment and plasma-reduction. Coatings are often used in conjunction with anti-corrosion agents that serve as a moisture barrier, oxygen barrier or contain substances that inhibit or prevent a chemical reaction.

Preventive conservation is a vital part of extending the life span of iron artefacts. It involves ensuring that the climate is controlled and suitable for the artefacts to be stored there. Climate control means controlling the relative humidity so that it suits a particular material. For iron, this means that the climate should be kept as dry as possible, RH should not exceed 50% and fluctuations in both temperature and RH should be kept at a minimum.

One of the goals of the thesis was to see what methods of archaeological iron conservation are best suited for mass-conservation for the National Museum of Iceland. It is recommended here that only parts of the treatment will be changed to a mass-conservation setup. All in all the process would be done in seven steps, only two of which involve mass-conservation. These are desalination by immersion treatment and dehydration. Adapting all seven of the steps would greatly diminish the documentation of the artefact and the conservation process, which is compatible with E.C.C.O.'s guidelines code ethics.

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Appendix I. Database for the Reyðarfell Artefacts

