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Desalination of Archaeological Composite Objects Consisting of Wrought Iron and Wood/Bone.

With Focus on the Iron Component.

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Cover image:Iron fragment in distilled water with lid of paraffin oil after 6 weeks.

Photograper: Sigríður Þorgeirsdóttir.

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ABSTRACT

The difficulty of choosing a suitable treatment for freshly excavated composite objects from archaeological sites is a well known problem within the field of conservation. The array of different materials, their shape and condition, degree of deterioration and their individual demand on correct treatment along with their effect on each other through time is a complicated combination of factors.

In order to preserve iron, it is important to eliminate as much Cl^- as possible after excavation. This is usually done through treatments in alkaline solutions. However, the high pH of these solutions (pH 12 – 13) is extremely destructive to the organic components in composite objects containing wood and bone.

The Western Australian Museum has been using mineral oil as a seal during the desalination process of iron in alkaline solutions. They report that it is possible to prevent the absorption of carbon dioxide as to avoid the pH dropping in the solution for up to 6 weeks.

The aim of this thesis is to investigate the suitability of the use of mineral oil as a sealing agent to exclude the ingress of oxygen during the desalination process on composite iron/organic objects. The focus of this project lies on the effectiveness of the treatment of the iron component in composite objects.

The results of my work indicate that distilled water with mineral oil as a sealing agent to exclude oxygen for the treatment of composite objects does not work sufficiently. This conclusion is based on the amount of sediment and the iron in the solutions that showed a high level of corrosion on the objects.

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Foreword

I would like to thank everyone who supported me during this thesis; especially my supervisor Jacob Thomas for making this experiment happen, and all the great comments and support. Also, I would like to thank my external supervisor Vivian Smits for bringing up this topic for a thesis, and all the great comments and support. Specially thanks to SVK for access to artefacts, information and equipment. I would like to thank my friend Hildur Gestsdóttir for reading over the thesis and giving me great comment as an archaeologist; and finally, but not least, I would like to thank my fiancé Ólafur Hrafn Nielsen for endless encouragement, support, and patience in preparing this thesis.

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1. INTRODUCTION

1.1 Background

Soon after I started my studies in archaeology and conservation, I started to wonder what would be the best way of treating composite objects, for instance tools, cutlery and other artefacts consisting of wrought iron and any organic material like bone or wood. The difficulty of choosing a suitable treatment for freshly excavated composite objects from archaeological sites is a well known problem within the field of conservation. The array of different materials, their shape and condition, degree of deterioration and their individual demands on correct treatment along with their effect on each other is a complicated combination of different factors.

The most suitable conservation treatment for composite objects is usually defined by the more unstable material or the most valuable one, i.e. the most uncommon, defining or relevant to the archaeological investigation (Cronyn 1990, pp. 93-94). The truth is that treating composite objects usually is rather complicated.

During my internship at Studio Västsvensk Konservering (SVK), in the autumn of 2014 my supervisor, Vivian Smits, brought up the option of using oil in order to exclude oxygen during the process of desalinating iron. The idea came from a comment made by Ian MacLeod of the Western Australian Museum at the Interim Meeting of the ICOM-CC Metal Working Group in September 2013. He stated there that oil was an adequate substitute for many of the methods currently used for excluding oxygen during the desalination of iron. The Western Australian Museum has been using mineral oil as a seal on large tanks and tubs during the desalination process on iron in alkaline solution. They report that it is possible to prevent the absorption of carbon dioxide so as to avoid pH dropping in the solution for up to 6 weeks (Informant 3). Unsuitable pH levels can have a destructive effect on different materials and the access of oxygen is an equally destructive component during the desalination process.

The question of whether this method would be suitable for composite objects was raised. Would it be possible to use a different medium suitable for organic materials, like water, instead of an alkaline solution, and use oil in order to exclude oxygen during the process? Mineral oil would be a cheap alternative to many other methods, and it appeared a method easy to execute!

1.2 Problem Statement

One of the major problems with the conservation of composite objects is that the requirements for an appropriate treatment for the different materials can be mutually exclusive. The effect of a treatment suitable for a certain kind of material can be highly destructive on another component in a composite object.

The problem with treating archaeological objects made of iron and wood[1] that cannot be separated from one another is most obvious in the area where wood and iron are in intimate contact with one another. Since wet wood is slightly acidic (pH range from 4-6), it will cause the iron to corrode when the wood is wet (Baker 1974, p. 1-3).

For archaeological iron, on the other hand, it is important to remove as much Cl^- as possible to stop the corrosion process. An alkaline solution, pH 12, gives the best results for desalinating iron (Costain 2000, pp. 13-18). However, alkaline treatment, e.g. with sodium hydroxide solution, is extremely aggressive to wood and will damage it. For archaeological wood, water provides the optimal environment for desalination, but without any inhibitors, water initiates aggressive corrosion of iron (MacLeod et al. 1989, p. 245-46).

Yet it remains, that archaeological objects containing elements of iron need to be desalinated in order to be stabilised. The choice of treatment for composite objects consisting of iron and organic material, therefore, cannot follow conventional desalination procedures, since the process will have a destructive effect on any organic components. Suitable treatment for composite objects of iron and wood or bone should:

- maintain a suitable pH for the organic materials – as well as suitable pH for iron;
- exclude oxygen; and,
- remove Cl^- from iron

Identifying such a treatment is a goal for many archaeological conservators.

1.3 Objectives

The objective of this thesis is to investigate the suitability of the use of mineral oil as a sealing agent to exclude the ingress of oxygen into the solution during the desalination process for composite objects. The focus of this project is on the effectiveness of the desalination treatment of the iron components in composite objects.

1.4 Aims

Is desalination with distilled water and mineral oil as a sealing agent in order to exclude oxygen a viable alternative for the treatment of composite objects? As a means to answer this question, I conducted experiments in order to determine the efficacy of the treatment by measuring:

- The total amount (mg/l) of removed chloride ions within a given time;
- The amount of oxygen present during treatment; and,
- Amount of corrosion on the iron in the solution

I compared five different solutions in these experiments:

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[1] From now called composite objects.

- 0.1 M NaOH pH 13 solution
- 1% (w/w) Na_2HPO_4 pH 6.5 buffer solution
- distilled water
- distilled water with mineral oil as an oxygen barrier film
- distilled water degassed with N_2

According to literature is the NaOH solution by far the most effective treatment. Therefore, it serves as a positive control for the effectiveness desalination of iron. The Na_2HPO_4 buffer solution served as a positive control for desalination of iron-organic composite objects. Distilled water served as a negative control for desalination of iron objects. To these three solutions, were compared degassed distilled water and distilled water with a mineral oil oxygen barrier film.

1.5 Limitations

I cannot provide any statistical support on authentic composite objects since these objects are rare and, therefore, very valuable in a scientific and historical point of view. Instead, I rely on a discussion based on what has been done in terms of research on the subject.

The lack of research, absence of documentation on similar experiments carried out and, most important, the limited amount of time available for the experiments affect the outcome of this study.

Due to time limitations, the efficacy of the desalination of 6 weeks treatment is only measured for the soluble chlorides. Desalination of iron (down to an accepted level of <5ppm [Cl^-]) (Selwyn 2004a, p. 39) can take years, depending on the chloride levels contained within the iron.

Because of time limitations for both the desalination treatment and test analysis following evaluation of the effects of different materials, no wood was put into the treatment solutions. Furthermore, it is known that wood does not respond well to long-term alkaline treatments in comparison with neutral aqueous treatments. In light of these limitations, this experiment focused only on the iron component in the evaluation of the potential treatments for composite objects.

1.6 Methods and Materials

This thesis is based primarily on a study of available literature in order to demonstrate the structure of iron, wood, and bone as well as the chemical composition and degradation of archaeological iron, wood, and bone. The literature study sheds light on the limited amount studies on treatment methods that have been carried out and published regarding the conservation of archaeological composite objects. It demonstrates the problem of treating composite objects.

Minor laboratory experiments with distilled water and mineral oil as a seal have been carried out, as well as some more common methods for desalination of iron objects. The amount of oxygen in the solutions has been measured in order to compare the effects of different kinds of solutions in the process of desalination, as well as to evaluate the ease of handling the different methods.

Furthermore, a small experiment in order to compare the effectiveness of synthetic oils and vegetable oils as sealants to exclude oxygen, has been carried out.

1.7 Theoretical approach and ethical issues

In the § 6 evaluation in Code of Ethics from *AIC (American Institute of Conservation)* from 1994 it is stated that:

> "The conservation professional must strive to select methods and materials that, to the best of current knowledge, do not adversely affect cultural property or its future examination, scientific investigation, treatment, or function." (AIC)

These guidelines establish a frame work for the choice of treatment for the preservation of cultural heritage.

Science became a part of conservation in the mid-twentieth century, when focus shifted to objects instead of merely preservation of architectural heritage (Muños Viñas 2005, pp. 69-81). The implementation of science into the field of conservation has had a major impact on the knowledge and understanding of deterioration processes of different groups of materials. A scientific approach is necessary for the understanding of the need for ethical guidelines in conservation.

1.8 The Structure of the thesis

This thesis is divided into six chapters. Chapter 1 introduces the background of my research as well as the experiments. Chapter 2 focuses on the problem of treating composite objects. It also contains an overview on available publications on the subject and how composite objects are treated today at SVK, Gothenburg, Acta, Stockholm, and the National Museum of Denmark. Chapter 3 addresses the degradation process of iron as well as the degradation of wood and bone. Carried out experiments are introduced in Chapter 4, and Chapter 5 discusses the results from the experiment. Chapter 6 contains the conclusion of the thesis.

2 Composite objects – iron and wood/bone

2.1 Objects COMPOSED of composite material

Figure 1: Composite object; iron knife with wooden handle.

Archaeological composite artefacts are defined as objects which are made up of two or more materials. Composite artefacts are commonly made up of a combination of organic and inorganic materials, for example, iron knifes, cutlery, or tools, with handles made of wood, or bone.

The biggest challenge for these types of artefacts is to reduce the corrosion of iron. For iron objects in combination with wood, the corrosion can cause the two materials to fuse. This will result in a disfiguring staining on the wood as well as accelerated degradation of the cellulose (Hawley 1989, p. 224). Terrestrially found archaeological iron is usually contaminated with hygroscopic salts (Cl^-) and other impurities from the burial environment which have accumulated in the object over centuries. Access to oxygen in combination with a high humidity and the presence of chloride ions in the iron, will cause corrosion of the iron (Mattsson 1996, pp. 29). It is known that the iron III corrosion products catalyse the degradation of cellulose, as the corrosion products are inherently acidic as a consequence of the hydrolysis of the ferric ions (MacLeod et al., 1994, p. 199). The action of the ferric ions on the hydrolysis of cellulose often shows in the form of loss of strength of the wood surrounding the corroding iron (MacLeod et al., 1989, p. 245). The corrosion will have a destructive effect on the physical and chemical integrity of the object as a whole. Iron ions contaminate the surrounding materials causing discolouration, deformation, flaking and cracks, and will eventually disintegrate the entire object. This is why it is necessary to remove as much of the chloride ions from the iron as is possible.

The most effective desalination treatments available today are based on the diffusion process with an alkaline solution. Treatment with non-alkaline solutions are far less effective (Costain 2000, p. 19). However, the desalination process is destructive to organic materials due to the high pH in the alkaline solution (Mattsson 1996, p. 27).

Wet organic archaeological materials like wood and bone are normally gently cleaned in water. Chelating agents like DTPA[2] or EDTA[3] are sometimes used to remove contaminations from the soil. After this, the object will preferably be treated with a PEG[4] solution, especially the wood objects, then frozen and often then freeze-dried. Waterlogged bone objects are often dehydrated in e.g. changes of ethanol and not freeze-dried. The PEG treatment however, will cause problems on iron components in a composite object since its low pH (4.5-7) will trigger corrosion of the metal (Selwyn et al., 1993, p. 180).

To slow down corrosion of iron in composite objects, a corrosion inhibitor, for example, Hostacor IT, can be added to the PEG solution. The carboxylate anion of the Hostacor molecule passivates iron spontaneously using dissolved oxygen in the solution to oxidise Fe^{2+} ions to Fe^{3+} ions, raising the local pH and forming a protective layer between the Fe^{3+} ions and the inhibitor. The Hostacor only acts on the surface of the exposed iron (Argyropoulos et al., 2000, pp. 253-254) and it is unclear what happens to the metal parts contained within the wood or bone being treated. What happens to the metal on a molecular level when frozen (i.e. whether or not the process is destructive due to the formation of ice-crystals within the metal attire), is not known as research on the topic is limited.

2.2 Earlier methods for composite objects

Different methods have been used and tested for the conservation of composite objects over the years. None of the tested methods has given satisfactory results. Janet K Hawley, from *The Historic Park Resource Conservation Branch Laboratory of Environment Canada*, carried out a survey among laboratories around the world in the early 1980s on the treatment of waterlogged metal and wood composite objects. The results were published in the conference monograph: *Conservation of Wet Wood and Metal: Proceedings of the ICOM Conservation Groups on Wet Organic Archaeological Materials and Metals, Fremantle 1987* (MacLeod 1989). In one of these papers, Hawley (1989, p. 231) describes how the *Historic Park Resource Conservation Branch Laboratory of Environment Canada* carried out the desalination process on a composite object, a ship wheel constructed of wood, iron and copper alloys. The object was placed in a large tank of refrigerated water (2°C) in order to desalinate the iron and reduce microbial growth. To reduce dissolved oxygen and to minimize corrosion, nitrogen gas was gently bubbled through the solution.

In the same paper other museum and institutes gave answers to the question about treatment of composite objects, but only a few gave answer describing the desalination process.

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[2] Diethylene triamine pentaacetic acid (DTPA)

[3] Ethylenediaminetetraacetic acid (EDTA)

[4] Polyethylene glycol (PEG)

Jim Spriggs at the *York Archaeological Trust, York, U.K*, describes three methods the Trust uses to desalinate objects: in a humidity cabin under a blanket of nitrogen; in a 1% sodium hydroxide solution; and, in water containing a dissolved vapour phase inhibitor, cyclo hexylamine carbonate. Spriggs however does not present any results from those treatments in his answers (Hawley 1989, p. 236).

Howard Murry at the *Mary Rose Trust, Portsmouth, U.K*, describes the treatment of composite objects with the so called mannitol process. The composites were generally stored in 5% Borax ® or benzotriazole, and desalinated in 5% sodium sesquicarbonate or under cascades of water, immersed for a few days in 15% mannitol and then freeze-dried (Hawley 1989, p. 236).

Kristen Jespersen at the Nationalmuseet Konerveringsdelingen for *Jordund, Brede, Denmark*, describes the treatment of composite objects in baths of EDTA, followed by washing in distilled water to remove any remaining salts (Hawley 1989, p. 238).

Anne Daldorff at the Universititetet i Tromsø, Tromsø Museum, *Institutt for Museumcirksomhet Tromsø, Norway*, reported using diaminoethylene as a method of desalinating. However, the results were inconsistent. Attempts were made to desalinate composite objects by boiling them in water for at least three to four months. However, that process proved to be harmful to the wood components. Therefore she recommended the use of 2.5 -5% sodium sesquicarbonate to remove salts, followed by Soxhlet extraction of the carbonate (Hawley 1989, p. 238).

Karl Peters from the *University of Auckland, Anthropology Department Auckland, New Zealand*, described removing the soluble chlorides by washing the objects in running distilled water; whereas, the corrosion products are subsequently removed by mechanical cleaning (Hawley 1989, p. 239).

Ian MacLeod, Fiona M. Fraser and Vicki L. Richards at *the Department of Material Conservation, Western Australian Maritime Museum*, carried out an experiment on the factors affecting the stabilization of corroded iron-wood composites. The purpose of the experiment was to inspect the ability of aqueous PEG solutions to extract iron and chloride ions from iron-impregnated concretions and wood recovered from the sea. These ions were specifically studied because the removal of chlorides is known to stabilise iron and the removal of iron cations will protect the wood from further degradation. The results demonstrated that a PEG 1500 solution in the range of 5-10% (w/v) was optimum for extracting chloride ions from the iron-containing concretion and wood, and that the chloride removal rate was faster in this solution than in distilled water. The PEG solution also removed significant amounts of soluble iron salts. Based on these result MacLeod et al. (1989, pp. 245-250) suggested a two stage treatment for composite iron-wood objects. First stage is to treat the composite to remove iron and chloride ions, and then use a more concentrated PEG solution to consolidate the wood.

Another team led by Ian MacLeod, Paul Mardikian and Vicki L. Richards at the *Department of Material Conservation, Western Australian Maritime Museum*, carried out a desalination experiment on corroded wrought iron from a marine environment. The experiment was performed with six different solutions: distilled water, 2% (w/v) ammonium citrate, 2% (w/v) sodium hydroxide, 5% (w/v) PEG 400, 5% (w/v) PEG 400/2% ammonium citrate and electrolysed in 2% (w/v) ammonium citrate. The results of the experiment demonstrated that 5% (w/v) PEG 400/2% ammonium citrate with neutral pH gave the most effective extraction of chlorides from corroded iron. In addition, the corrosion-promoting chloride ions were extracted from the iron at a reasonable rate, and the PEG stabilised the waterlogged wood. Therefore, this solution is deemed a suitable storage medium for wood and iron composite objects. It is, however, recommended to add a corrosion inhibitor, such as oxalate or tannate to the solutions to minimise the corrosion of the metal (MacLeod et al., 1994, pp. 199-209).

As can been seen above, as well as in the experiments carried out by MacLeod et al. there is no method that is the clear winner, since none of them are "dominant" in the conservation of composite objects today. It appears that much effort was put into research and experiments on composite objects in the 1980s and early 90s, with very little work being carried out on the treatment of composite objects of iron and wood since then. Of course objects made of those material groups have been treated by conservators over the years. Information on how they have treated the objects is not easily accessible since the treatment is described often in the conservation reports.

2.3 Methods used today at SVK, Acta, and The National Museum of Denmark

The stabilisation of composite objects is still a problem today. An ultimate method of desalinating iron in composite objects without harming the other material, has yet to be developed. As part of this thesis, an inquiry was done at SVK, Acta, and The Nation Museum of Denmark on the current desalination processes in use for composite objects at these respective institutes.

SVK (Studio Västsvensk Konservering) in Gothenburg removes Cl^- from the iron (which is not enclosed by organic material) in a 0.1M aqueous solution of sodium hydroxide. During this process, the iron is

Figure 2: Iron dagger with wooden haldle during desalination with NaOH at SVK.

in the solution and the organic/iron part are kept separated with neoprene plastic (see Figure 2). If the object was wet before treatment the organic part is kept moist. This method means that the desalination process is isolated from the iron part of the object. In many cases, the desalination process is much shorter than usual since it is discontinued when the organic parts start to exhibit a degraded integrity. When the desalination process is discontinued, the object is impregnated with a PEG[5] solution with additive 1% Hostacor IT. Some of the objects are subsequently freeze-dried (Smits et al. 2008, p. 13).

At *Acta Konservering Centrum* in Stockholm an aqueous solution is used. The iron part of the object is placed in the solution, and the organic part is kept out and covered with polyester wadding to protect it from rust being deposited on the surface of the organic part. The aqueous solution is changed weekly and the chloride extraction process is monitored weekly with *Silver nitrate drop test*. When the results of the chloride test have been negative twice, the treatment is considered complete. Then the object is impregnated with a PEG solution to which 1.5% (v/v) Hostacor IT has been added as a corrosion inhibitor. Some of the objects are freeze-dried after treatment (Informant 1).

At the *National Museum of Denmark* they choose to separate the material groups whenever that is possible. If the nature of the object prohibits this, the iron is treated with an antioxidant (tannin). This treatment does not remove the Cl^- from the iron part of the composite object. Electrochemical methods are being used for the iron when the wood can be successfully separated from the iron in order to treat the materials separately.

In the answer from the National Museum of Denmark, Trina Wiinblad mentioned treatment that she and her college used on few unstable composite objects, at the Conservation Centre at Bevaringscenter Øst (BCØ) between 1998 and 2010. For the desalination process, 0.03 M sodium sesquicarbonate at pH 10 was used where the object were submerged in the solution. During the treatment, the pH and presence of chloride in the solution were monitored regularly by precipitating chloride ions with silver nitrate. The solution was replaced whenever chloride tests returned a positive result. When the test showed negative results (< 5 ppm), the treatment was deemed completed. The object is rinsed in demineralised water and dried under controlled conditions. After drying, the object is treated on the surface with an acid-free wax. This process can take from six months to several years, often 3-6 years. According to Wiinblad, the results have been satisfying. The bone material is perhaps a little bit more brittle after the treatment, but no warping or cracking has been observed (Informant 2).

What the above presentation of methods highlights is that the treatment of composite objects of organic and inorganic material is problematic and methods used are quite different between conservation institutions/laboratories.

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[5] The grade of the PEG solutions depends on the degree of detoration on the wood.

3. Deterioration mechanisms of iron, wood and bone

3.1 Introduction

Composite objects usually consist of both organic and inorganic materials, most commonly iron, wood and bone. The main structure of these different materials and the nature of their deterioration in a buried environment will be discussed.

3.2 Iron

The chemical symbol for iron is *Fe* which is derived from the Latin word *ferrum*. Iron is one of the most common elements in nature and occurs naturally in ores as a metal oxide. The properties of iron include hardness, plasticity, and flexibility, as well as the fact that it is possible to process it in an annealed state. Pure iron has a density of 7.87 g/cm3 and a rather high melting point, about 1538º C. In prehistoric times, iron was produced from bog iron or soils with a high content of iron oxides (Selwyn 2004a, p. 89 and Fjæstad 1999, p. 85). In Northern Europe, it was not until the end of the 15th century that man learned to build better ovens allowing higher temperatures to be reached during the refining process. Moreover, the process of iron extraction could be controlled by, among other things, adding or reducing the amount of carbon, thus learning how to produce cast iron (Thompson 1958, p. 349). All iron artefacts made of iron before the 15th century in Europe where made of wrought iron. Wrought iron contains less than 0.5% carbon and contains different quantities of slag, phosphorus, and sulphur (Fjæstad 1999, p. 85).

3.2.1 Deterioration of iron in buried soil

Iron is a relatively unstable metal and is usually covered with a thin oxide film when it is raw. For iron to corrode, the corrosion requires access to oxygen, water and an electrolyte, and even the pH matters. All of these factors are available in the soil and can lead to corrosion on the surface of the metal so that the volume changes and the corrosion migrates inward to the metal core and encasing in the process the surrounding sand and other particles in the burial environment (Selwyn et al., 1999, p. 217-218; Selwyn 2004a, p. 21).

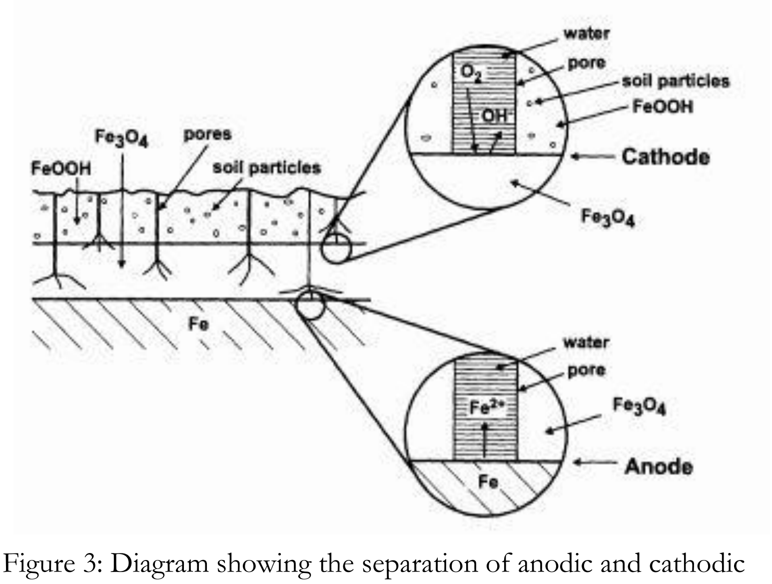


Figure 3: Diagram showing the separation of anodic and cathodic regions on buried iron.

In short, the corrosion process of iron is the oxidation of Fe to Fe^{2+} ions according to the reaction: $Fe \rightarrow Fe^{2+} + 2e^-$ (anode)

which takes place directly on the metal surface. As with all redox reactions, the loss of electrons must be counterbalanced by a corresponding reduction reaction where electrons are gained. The other reaction, on the metal surface, is:

$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$ (cathode) (Selwyn 2004b, p. 294).

The corrosion products that build up at the cathode are usually the electrically conducting corrosion products and form intermediate Fe^{2+}-Fe^{3+} compounds, e.g. magnetite (Fe_3O_4). Magnetite is a stable corrosion product that acts as a passivating layer on the surface of the iron (Selwyn 2004b, p. 295).

The soil water, with its dissolved ions, acts as the electrolyte in the process and contains dissolved salts, such as chloride ions (Cl^-) (Knight 1997, p. 36). Chloride ions tend to accumulate at the surface (the anode) on the metal. The cracks, pores and open spaces within the corrosion layer on archaeological iron become filled with an acidic iron (II) chloride solution (Selwyn 2004a, p. 105-6). The presence of Cl^- ions will accelerate corrosion after the object has been excavated (Selwyn et al., 1999, p. 217; Turgoose 1982a, p. 97; Rimmer et al., 2012, p. 29).

In the soil, iron (II) (Fe^{2+}) can be further oxidized to iron (III) (Fe^{3+}) ions according to the equation: $Fe^{2+} \leftrightarrows Fe^{3+} + le^-$.

The counter balancing reduction reaction at the cathode site that consumes the electrons generated most commonly involves the reduction of oxygen:

$O_2 + 2H_2O + 4e^- \leftrightarrows 4OH^-$ or $O_2 + 4H^+ + 4e^- \leftrightarrows 2H_2O$

(When the oxygen level is high and the local pH is less than 6)

$2H^+ + 2e^- \leftrightarrows H_2$ or $2H_20 + 2e^- \leftrightarrows H_2 + 20H^-$

(When the oxygen level is low and the local pH is less than 4)

Hydrogen evolution is less common than oxygen reduction, and only occurs when the pH is 4 or lower (Selwyn et al. 1999, p. 218; Scott & Eggert 2009, p. 99). Since both the anodic and cathodic reactions take place directly on the metal surface, Fe^{2+} ions can react directly with hydroxyl ions (OH^- ions) to form solid $Fe(OH)_2$ and passivation can occur. Over time, $Fe(OH)_2$ can transform to goethite through the loss of moisture, and over longer periods the corrosion will turn into stable magnetite. Magnetite formation occurs under lower oxygen levels than goethite. This corrosion process eventually slows down as the iron becomes covered with insoluble corrosion and soil particles that prevent further oxidation of the metal surface (Selwyn et al., 1999, p. 218; Turgoose 1982b, p. 2).

3.2.2 Post-excavation

After excavation, the changes in the object ambient environment are dramatic. The object goes from a high relative humidity (RH) and low oxygen content in the soil, to low RH and high oxygen content (Selwyn 2004b, p. 295; Mattson et al., 1996, p, 13). The corrosion process continues, especially if the iron still contains an iron core and is contaminated with salts or an acidic iron (II) chloride solution. Unless the iron is stored under 12% RH, or in an anoxic environment, it will continue to corrode. If RH levels exceed 12% and go over 15 % to 20%, there will be a phase change to mineral akaganéite (β-FeOOH), which will determine the corrosion rate of chloride-contaminated iron (Watkinson et al., 2005, p. 249). If excavated iron is allowed to dry out, the reduction in volume causes the corrosion layers to crack and oxygen becomes readily available. The acidic $FeCl_2$ solution will also become concentrated. The sudden supply of oxygen rapidly oxidizes the Fe^{2+} ions in solution (Selwyn et al., 1999, p. 219; Selwyn 2004b, p. 296). According to Turgoose (1982a, p. 98; 1982b, p. 6) the reaction is one of oxidation and hydrolysis, given by:

$$4Fe^{2+} + O_2 + 6H_2O \rightarrow 4FeOOH + 8H^+$$

Iron oxyhydroxides (FeOOH), precipitate, and the solution becomes more acidic. This enables further corrosion of any remaining iron through:

$$Fe + 1/2O_2 + 2H^+ \leftrightarrows Fe^{2+} + H_2O,$$

as all the original Fe^{2+} ions have been oxidized (Selwyn et al., 1999, p. 218-219).

Indication of active corrosion of an object is ‘weeping’ or ‘sweating’ iron and the formation of dry, hollow red spherical shells on the surface. The weeping is caused by the hygroscopic nature of iron chloride salts. The salts absorb water, dissolve, and form wet droplets of orange-coloured liquid. Iron oxyhydroxide forms the framework for the spherical shells (Selwyn 2004b, p. 296).

Akaganéite (β-FeOOH) is thought to be formed only after iron has been excavated (Mathias et al., 2004, p. 34), and only in the presence of Cl^- ions with RH levels higher than 20% where $FeCl_2$ will remain in solution (Watkinson et al., 2005, p. 249). When in contact with the iron core, the $FeCl_2$ will slowly oxidise to form akaganéite. It is the formation of akaganéite which causes artefacts to crack and flake when stored in ambient conditions. (Knight 1982, p. 51). Akaganéite also acts as a reservoir for chloride ions and can cause renewed corrosion (Knight 1997, p. 37).

Turgoose (1982b, p. 4) suggests that in most cases, if not all, the deterioration of excavated iron objects is induced by the presence of chloride, and corrosion products on archaeological artefacts contain chloride ions.

All these corrosion processes and changes in environment, cause both physical and chemical damage to the objects. The physical damage is caused by the formation of new iron oxyhydroide corrosion layers, β-FeOOH (akaganéite), which causes stress and cracks on the iron object which will change its shape. The chemical damage is caused by the formation of hydrochloric acid (HCl), which results in a cycle by which the Cl^- ions form a soluble salt with iron(II) ions (Selwyn 2004b, p. 296).

3.3 Wood

The biological structure of wood is a composite of many chemistries and cell types acting together. Wood is essentially composed of cellulose, hemicelluloses, lignin, and extractives (resin), consisting of organic polymeric compounds (Fjæstad 1999, p. 116). Although cellulose is a well-defined single polysaccharide, both hemicelluloses and lignin include a wide variety of individual polymer types. Together, cellulose, hemicelluloses, and lignin comprise 95 wt % or more of dry wood. Organic substances (such as fats, waxes, resins, and simple phenols), which can be extracted with nonpolar solvents, account for approximately l% of the remaining material (Hedges et al., 1990, pp. 112-113).

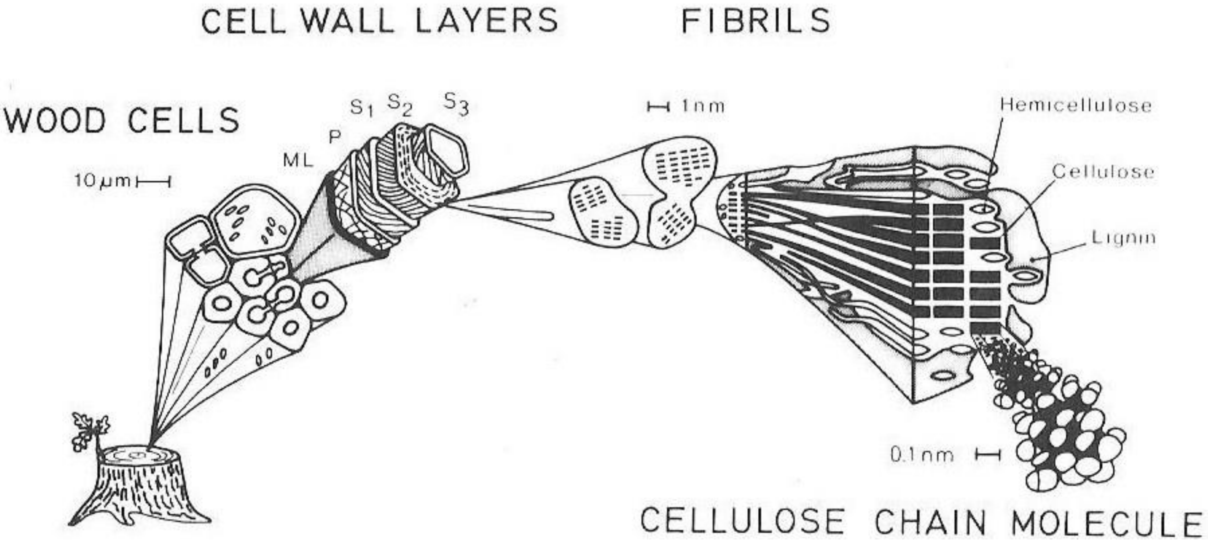


Figure 4: Structure of wood cells.

The taxonomy of archaeological wood is normally divided in to two groups, hardwoods (angiosperms) and softwoods (gymnosperms). Hardwoods have pores or vessel elements that occur among fibres and parenchyma cells. Cellulose content ranges from 40 to 50% with 15–25% lignin and 15–25% hemicellulose. Softwoods on the other hand are composed of overlapping tracheid (wood cells), connected by bordered pit apertures, and parenchyma cells as well as, in some cases, resin canals. Softwood has a similar amount of cellulose 40-50% as hardwood, but the lignin content is 5-10% larger in softwood than in hardwood, and therefore there is less hemicellulose in softwood (Blanchette 2000, pp. 189-190).

3.3.1 Deterioration of wood in buried soil

Archaeological wood in contact with soil decomposes rapidly due to bacterial and fungal attack. In aquatic environments the degradation of wood can take a much slower rate than in the atmosphere. Wood from waterlogged environments can look very "healthy" after excavation, with the wood retaining its physical integrity, colour, ornamentation, and tool marks (Björdal 2012, p 118). However, on the inside of the wood can be very decayed.

Fungal microbes can degrade wood by producing extracellular enzymes which break down the cell walls of the wood. For aerobic fungi to "survive" the environment needs to have oxygen and a wood\water content in excess of 18% (Cultural Heritage 2010, p. 27). The decay can be either physical or chemical, causing morphological changes in the wood (Blanchette 2000, p. 190). Three major groups of decay produced by fungi are: white rot fungi, brown rot fungi, and soft rot fungi. White rot fungi degrades all cell wall components, including lignin, discolouring the wood white or yellow and making it appear moist, soft, spongy, and stringy. With brown rot fungi, the enzymes from hyphae pull out holocellulosa from the cell wall and destroy and weaken the middle lamella (Björdal 2014, p. 13). The decay caused by brown rot fungi is characterised by extensive depolymerisation of cellulose (Blanchette 2000, p. 192). Soft rot fungi degrades wood in both terrestrial and aquatic environments and are considered to have lower requirements for oxygen (Björdal 2012, p. 120). Soft rot has two distinct types. Type 1 is characterized by longitudinal cavities formed within the secondary wall of wood cells, and Type 2 is used to describe an erosion of the entire secondary wall. The deterioration of the wood is characterised by a loss of cell wall material, the shrinking of the middle lamellae, discoloration as well as the formation of cracks on the wood (Blanchette 2000, p 191-193).

Bacteria usually attack wood that is in aquatic environments or saturated soil (Björdal et al., 1999, p. 63). There are three main groups of bacteria that are found on archaeological wood: erosion, cavitation, and tunnelling. Erosion bacteria are able to degrade wood under very low oxygen concentrations. They degrade secondary wall layers and deplete cellulose and hemicellulose from the wood (Blanchette 2000, p 193-194; Björdal 2012, p. 119). Tunneling bacteria characteristically produce tiny tunnels that occur within the secondary cell wall, but can also be found in lignin and the middle lammeallae. Cavitation bacteria from a small diamond shape or irregular cavities within the secondary wall (Blanchette 2000, p. 194).

3.4 Bone

Bone is a composite material consisting of both organic and mineral phases. In fresh dry bone, the organic part, which is mostly collagen, is about 22-23% by weight (Turner-Walker 2007, p. 660). The scaffolding of collagen fibrils is made rigid by the deposition around and in them, of crystalline inorganic material (apatite) hydroxyapatite

$Ca_{10}(PO_4)_6(OH)_2$. This makes up approximately 70% by weight (Turner-Walker 2007, p. 660; Cronyn 1990, p. 275). Collagen has a triple helical structure, making it very strong molecule. This triple helical structure prevents it from being broken down by most enzymes (Cunniffe et al., 2011, p. 67). There are two main types of structure in bone: cancellous tissue, which forms the internal part of the bone, and compact bone, which is a dense tissue that forms the outer part of the bone, as well as the shaft of long bones. Bone is perforated by a network of tiny canals, as well as a number of larger holes which allow blood vessel etc. to enter the bone (Cronyn 1990, p. 275).

3.4.1 Deterioration of bone in buried soil

Deterioration of bone in buried soils depends on the environment. The main factors are the local pH, soil hydrology, redox potential, microbial activity, temperature, and mechanical damage (Turner-Walker 2007, p 660; Borg et al., 1994, p. 92-94). Objects made of bone can be in a very good condition after excavation, especially when they have been in anaerobic, nonacidic conditions such as urban deposits or marine silts. In damp oxygenated deposits, such as coarser calcareous sand or loam, the surface is often rather rough and can easily be scratched. If a wet bone object dries too fast, the material can become chalky and warping/cracking/laminating of the surface can occur alongside the loss of weight (Cronyn 1990, p. 275-278).

4. Methods and Materials

4.1 Introduction

In order to compare the suitability and efficacy of different treatment solutions for the desalination of iron, a quantitative study was performed by means of three experiments. In first experiment, the amount of removed chloride ions and the amount of oxygen present in the solutions were measured periodically. The formation of rust was used as a visual indication on the efficacy of the different treatment methods.

Five different solutions were tested: 1) distilled water, 2) distilled water with paraffin oil, 3) distilled water degassed with nitrogen gas, 4) 1% (w/w) aqueous solution of disodium phosphate (Na_2HPO_4) and 5) 0.1M aqueous solution of sodium hydroxide (NaOH).

The second experiment was conducted in parallel with Experiment 1 where iron corrosion was evaluated on modern forged iron in the five solutions as mentioned in experiment 1.

A third experiment was performed to assess if vegetable oil could be used as a 'green' alternative to paraffin oil, by comparing the rates of oxygen ingress through the oil barrier films.

4.2 Materials

For the first experiment, the desalination of iron, the testing materials consisted of dry archaeological wrought iron from the 2013 excavations of Nya Lödöse, Gothenburg. Five large nails were cut into five sections (see Figure 5), to give a total of 25 fragments. One fragment from each nail was placed in each of the five solutions in Experiment 1 (see description of solutions below); thus, five samples for each treatment method to account for variability in the source material.

Before the nail fragments were placed in the solutions, they were cleaned of corrosion products with air abrasive using small glass beads.

Figure 5: Nail cut into five sections

To evaluate the rate of iron corrosion in the second experiment, five modern forged iron fragments were used in each solution. As archaeological iron always has some corrosion, modern forged iron was used as a surrogate material, as it is easier to estimate the extent of induced iron corrosion when the materials are initially in a pristine state.

Each nail fragment was placed in identical polypropylene (PP) boxes, for a total of 30 boxes. Each box had 400 ml of solution in it.

The solutions that were investigated were:

- distilled water with paraffin oil, with white spirit as a dispersion agent;
- distilled water degassed with nitrogen;
- distilled water;
- 1% disodium phosphate (Na_2HPO_4) in distilled water; and
- 0.1M Sodium hydroxide (NaOH)in distilled water

4.2.1 Nitrogen gas (N2)

Nitrogen gas is a colourless, odourless and tasteless gas. It is non-flammable and is slightly lighter than air and slightly soluble in water. With oxygen it forms nitric oxide and nitrogen dioxide (UGI 2015).

Nitrogen has been used in conservation to deoxygenate both in display cases (Maekawa 1989) and in experiments of desalination of archaeological iron (Rimmer et al., 2012 and Watkinson et al., 2014).

4.2.2 Disodium phosphate (Na_2HPO_4)

Aqueous disodium phosphate has been used in the Arkeologiska Forskningslaboratoriet, at the University of Stockholm for the desalination of iron (ca 0.5 mass-% Na_2HPO_4 in 50-60% distilled water). The method is easy to use; it only requires simple apparatus and has a low maintenance (Mattson et al., 1996, p. 18).

In my experiments a 1% disodium phosphate solution in distilled water with pH 6.5 was used as one of the solutions to desalination the iron. This solution was used as a positive control for the desalination experiment.

4.2.3 Sodium hydroxide (NaOH)

Aqueous sodium hydroxide is one of the most common treatment solutions for the desalination of archaeological iron. It is relatively inexpensive, readily available, and has a high pH. The common concentrations used are 0.1M to 0.5M with pH 13-14 (Selwyn 2004b, p. 299; North 1987, pp. 221-222). Due to the high pH of NaOH, it is very effective and it gives the opportunity to stop corrosion by passivating archaeological iron and reduces the corrosion rate, as an adherent layer of insoluble corrosion products form on the iron (Selwyn 2004b, p. 299)

In my experiments a 0.1M aqueous solution of sodium hydroxide with pH 13 was used as one of the solutions to desalinate the iron. This solution was used as a positive control for the desalination experiment.

4.2.4 Oils

Oil can be either animal, vegetable or mineral in origin, and have a high carbon and hydrogen content. Non-mineral oils belong to a group of biological substances called lipids. Lipids are biochemicals that are insoluble in water. Oil is chemically defined as a mixture of trihydric alcohol glycerol with a range of possible long chain fatty acids. Most fatty acids are linear carbon chains with 18 carbon atoms. The physical and chemical properties of individual fats are determine by the kinds and proportions of fatty acids that enter into the triglyceride composition (Mills et al. 1994, p. 31).

For the third experiment (see Chapter 5.3) both mineral and vegetable oil were used.

Vegetable oil

Most vegetable oils are very similar in structure, with varied amounts of fatty acids, but with different carbon chain lengths and saturation levels (Mills et al. 1994, p. 34). Vegetable oils are a blend of saturated, monounsaturated, and poly unsaturated fatty acids.

For the experiment a frying oil from Noury was used. In 100 gr of oil there are ca. 8.5 gr saturated, 45 gr monounsaturated and 38 gr polyunsaturated fatty acids.

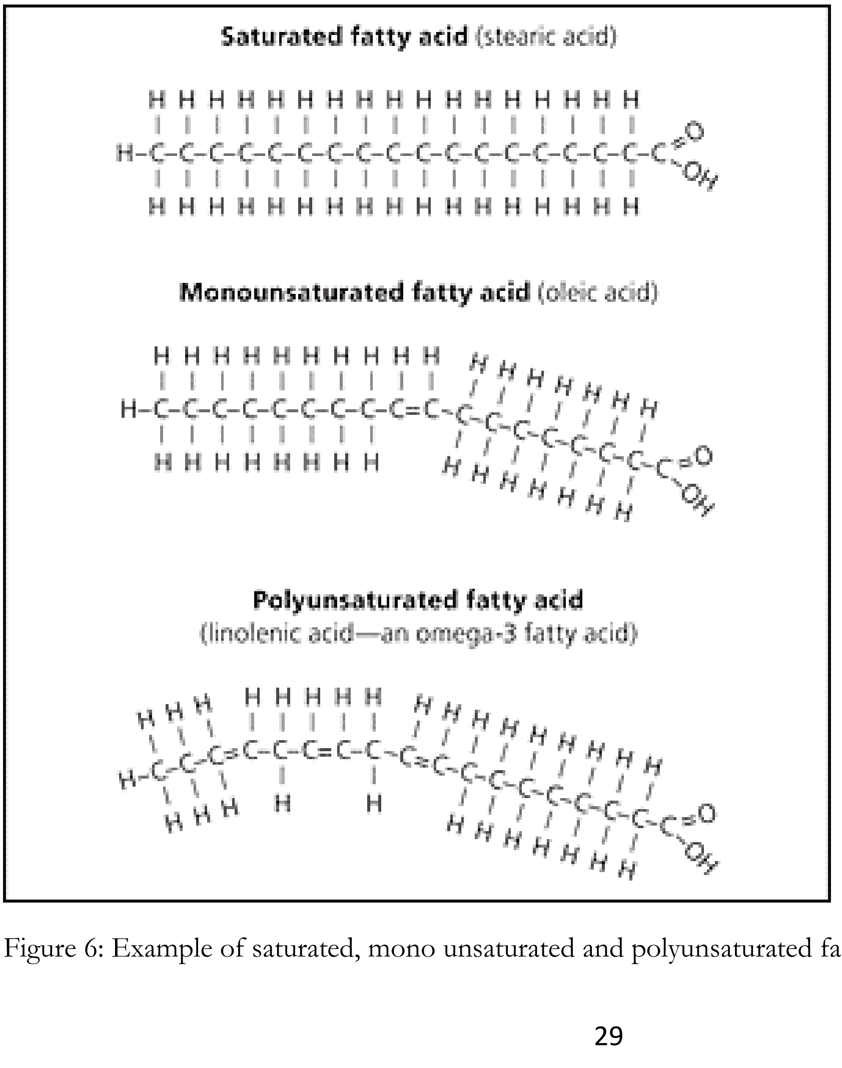


Figure 6: Example of saturated, mono unsaturated and polyunsaturated fatty acid structure.

Mineral oil

Paraffin oil is a clear, colourless transparent oil, also referred to as alkane. The molecular formula for paraffin oil is $CnH_{(2n+2)}$ where n=16~24 and has a density of 85 g/mL at 20°C. It is nearly tasteless and odourless, even when it is warmed. Paraffin oil is flammable and insoluble in water (Chemical book 2015).

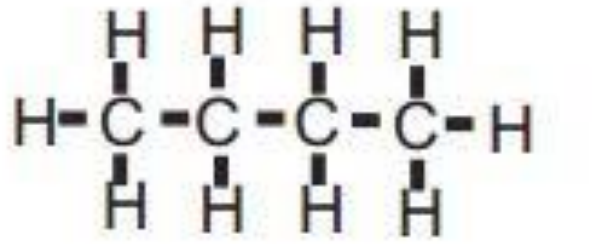
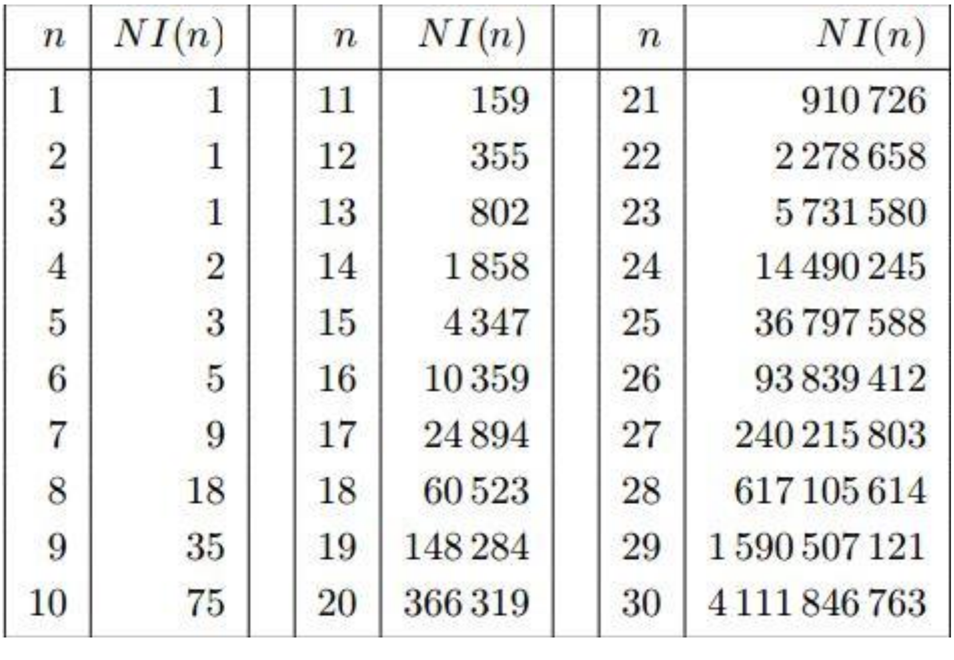


Figure 7: Example of n alkane, which are common in mineral oil.

The general chemistry formula for alkanes is $CnH_{(2n+2)}$ where n is a positive integer. Since paraffin oil is alkane, the structure can be different as can be seen in Figure 6. Paraffin oil has n=16~24; in Table 1 the number of n in paraffin oil can be seen.



n	$NI(n)$		n	$NI(n)$		n	$NI(n)$
1	1		11	159		21	910 726
2	1		12	355		22	2 278 658
3	1		13	802		23	5 731 580
4	2		14	1 858		24	14 490 245
5	3		15	4 347		25	36 797 588
6	5		16	10 359		26	93 839 412
7	9		17	24 894		27	240 215 803
8	18		18	60 523		28	617 105 614
9	35		19	148 284		29	1 590 507 121
10	75		20	366 319		30	4 111 846 763

Table 1: The number NI (n) of the $CnH_{(2n+2)}$ alkanes.

4.3 Methods

4.3.1 Measuring chloride, oxygen and soluble iron

The chloride ions in the solutions were measured using the *Silver nitrate test* with Sherwood MK II Chloride Analyzer 926S. The results with the machine should be within one standard deviation of the mean values of recognized QC schemes i.e. within ± 2.2 mmol/l at the 100 mmol/l level (Newton 2015)

The experiments were carried out between March 2nd and April 14th 2015. Chloride measurements were made once a week. Three measurements were performed for each sample each week. The average of those three measurements was taken and calculated to value mg/l of chloride in the solution with the formula:

$$\frac{Average + 35{,}453\ (atomic\ mass\ for\ chloride)}{10} = Value\ mg/l$$

The oxygen concentration of all solutions was measured with *Dissolved Oxygen test kit* from Hanna Instrument and was carried out prior to the start of the desalination. The oxygen was measured every week for five weeks, starting on March 2nd and ending on April 7th 2015. With the test kit, the oxygen concentration in water can be determined quickly and effectively using a modified Winkler method (see Appendix 1).

Soluble iron in the solutions was measured with *Iron Medium Range test kit* from Hanna instrument. This instrument was chosen since it is easy to use and with sufficient accuracy for this study; ± 0.04 ppm ± 2% of reading accuracy, 0.01 ppm (mg/L) resolution (500 points) (Hanna instrument). The first measurements were carried out at the beginning on March 2nd. The next measurement was on March 17th, and then once a week until April 14th 2015. Only three samples were measured from each solution, and were chosen by random using a normal six-sided die for selecting the sample.

At the end of the experiment, the sediments from all of the solutions were collected by filtration, dried, and weighed. The results are shown in Chapter 5.1.3.

The pH was measured for all of the samples at the start of the experiment using *pH indicator strips*. The next measurement was carried out on March 17th, and then once a week until the end of the experiment, April 14th 2015. The pH was measured for the same samples as were used in the soluble iron measurements.

4.3.2 Corrosion experiment

Corrosion rates (Experiment 2; see Chapter 5.2) on the five modern iron parts in the various test solutions were determined by simple weight loss measurements. Each modern iron sample was weighed wet before and after immersion in a test solution, and the corrosion rate was estimated by weight loss. To support the results, the sediment (from Experiment 1, part 3) from each solution was weighed.

4.3.3 Oil experiment

Separate experiments (Experiment 3; see Chapter 5.3) were performed to compare paraffin oil (mineral oil) and frying oil (vegetable oil) as a medium to limit the diffusion of oxygen in to a water bath. This was conducted to see if there was any difference between these two types of oils. The experiment was done by using distilled water and degassed distilled water where nitrogen gas (N_2) had been pumped through. Six boxes were prepared numbered 1-6. **1**) distilled water + paraffin oil, **2**) distilled water with N_2 + paraffin oil, **3**) distilled water + frying oil, **4**) distilled water with N_2 + frying oil, **5**) distilled water, and **6**) distilled water with N_2.

For Experiment 3 the same method was used as in the oxygen concentration from Experiment 1 (see Chapter 4.3.1). The experiment was carried out for one week and the measurements were done at the start and then after 2, 4, 6, 24, 48 and 72 hours. The last measurement was carried out 96 hours after the start of the experiment.

For Experiment 3 no objects were used in the solutions. The same polypropylene (PP) boxes as in Experiments 1 and 2 (a total of six boxes) were used.

5. Experimental

5.1 Experiment 1

Experiment 1 can be divided into three parts: part one includes measurement of chloride ions in five different solutions; part two consists of measurement of oxygen in the same five solutions; and, the third part measures the formation of rust in the same solutions (see description of different solutions in Chapter 4.2).

Removing Cl^- from archaeological iron is one of the most important treatments to slow down the corrosion process. As explained in Chapter 2.1, terrestrial archaeological iron can contain Cl^- and other impurities from the burial environment. When the chloride in iron artefacts comes in to contact with oxygen and high humidity, the iron will corrode (Mattsson 1996, pp. 29), and the corrosion will have both physical and chemical effects on the object. Therefore, the removal of chloride ions is important.

Oxygen is one of the key factors in the corrosion process of archaeological iron objects. There is a close correlation between the metal dissolution and the amount of oxygen reduced. The reduction of oxygen is the dominating cathodic reaction in the atmospheric corrosion (Matthisen 2013, p. 368 – 371). By measuring the oxygen consumption of an iron object it is possible to estimate the extent and rate of corrosion in each solution.

Figure 8: Week one. The fragments in the five different solutions.

It is possible to estimate the amount of corrosion from the fragments in each solution by measuring the rust. It can also support or give further results than Experiment 2 gives in which modern iron is used to see how much the corrosion develops in each solution as discussed in Chapter 5.2

5.1.1 Aim

The aim of Experiment 1 is to measure the amount (mg/l) of removed chloride ions in five different solutions investigated in the experiment (see Chapter 4.2), as well as measuring the amount of oxygen (ppm), and soluble iron (ppm) in each of the five solutions (see Chapter 4.2).

5.1.2 Materials and methods

As explained in Chapter 4.2, the material used for this experiment were 25 fragments from five archaeological nails divided over the different solutions. The Cl^-, O_2, and rust deposits were monitored as a means of anticipating the efficacy of the different methods.

As part of Experiment 1, the amount of removed chloride ions was measured. As is described in Chapter 4.3.1, the chloride measurement was performed with *Silver nitrate test* once a week for six weeks. The oxygen measurement (part 2) was performed simultaneously with the chloride measurements with using a modified Winker method for five weeks (see 4.3.1). The third part of Experiment 1 was to measure the soluble iron in the five different solutions with *Iron Medium Range* test kit (see Chapter 4.3.1).

5.1.3 Results

Chloride Concentration

The results from the measurement of chloride ions in Experiment 1 are shown in Figure 9. The results indicate that the distilled water with paraffin oil and sodium hydroxide solutions removed the largest amount of chloride ions, while both distilled water with nitrogen gas and distilled water alone remove the least. The disodium phosphate solutions is not comparable with the other solutions as a mistake was made during the preparation of solutions for the experiment. The mistake indicated that at the start of the experiment the pH of the solution was around 12, but it should have been about 6.5, so the solutions was changed in week 3 as can be seen in the yellow line in Figure 9. Due to this change, the disodium phosphate solutions cannot be used in comparison with the other solutions in the experiment. This is because the pH was wrong in the beginning, and by changing the pH of the solution from alkaline (pH 12) to more neutral (pH 6.5) the process gives erroneous results regarding the amount of removed chloride ions from the objects. However, it is kept in the graph to show what happens in the first 3 weeks of the desalination process on archaeological iron objects. There it exhibits the lowest level of removed chloride ions of the five solutions in the experiment, despite the fact that the pH is alkali.

In regard to the material used, it has to be stressed that the amount of chloride in each nail was unknown at the start of the experiment. Due to this, it is not possible to directly compare the amount of chloride removed in various solutions, since it is not known whether all the nails had the same amount of chloride ions at the start of the experiment, despite the fact that all the nails came from the same excavation site.

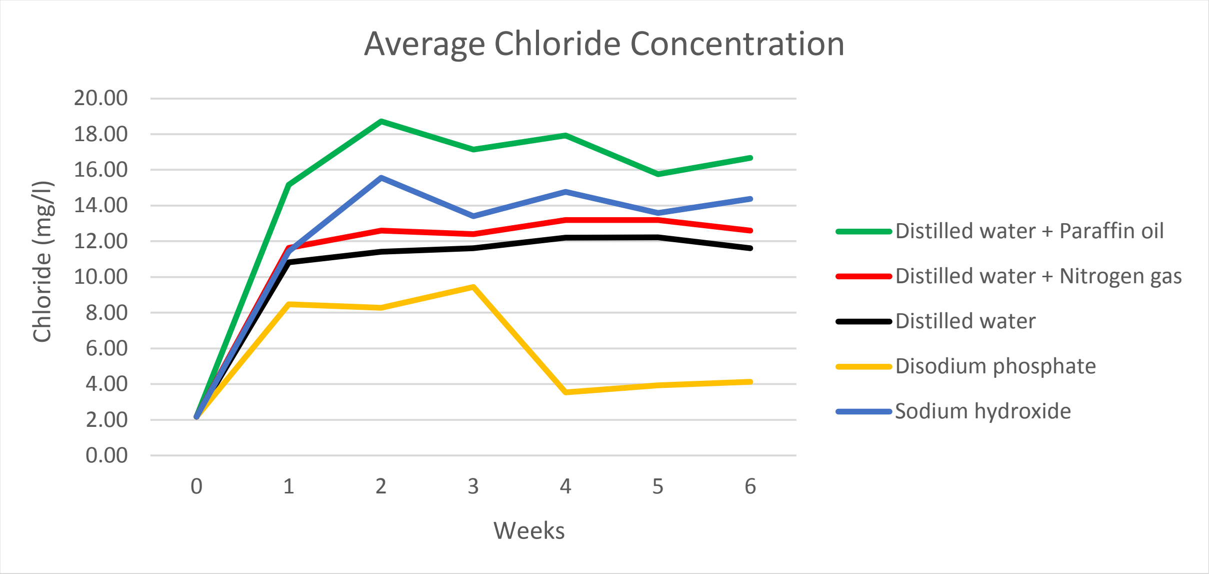


Figure 9: Average of concentration of chloride ions extracted into the solutions. The data for each sample is in Appendix 2.

Dissolved Oxygen Content

It was not possible to measure the amount of oxygen, or the formation of rust in the sodium hydroxide or the disodium phosphate solutions. These solutions were not compatible with the oxygen concentration test method. Therefore, for both the oxygen and the rust experiments only three solutions are presented in the graph below (see Figure 10).

The results from the measurement of oxygen in Experiment 1 are shown in Figure 10. From this it can be said that the dissolved oxygen in distilled water with paraffin oil solutions gives the best result, but only marginally so. There is no large difference between the solutions; although, the distilled water degased with nitrogen gas gave a good starting point, but then the next week the oxygen level was similar to that of the other solutions due to oxygen dissolving into the water. Distilled water with paraffin oil dropped down from 6.5 ppm at the starting point to 5 ppm in week 3, but then rises again and ends at 7.2 ppm, similar to that of distilled water. The trend in the first three weeks could be attributed to oxygen consumption by iron oxidation, and the later rising trend could be attributed to slow oxygen ingress. A possible explanation of the initial lowering and then later rising trends is that the rate of oxygen diffusion across the oil barrier is constant, but lower than the rate in the oil-free container throughout the experiment. Since the archaeological nails had been cleaned of corrosion before treatment, there was a latent potential corrosion at the cleaned iron surfaces. In this hypothesis, the first three weeks of the experiment marked a period in which the rate of oxygen consumption, due to rapid iron oxidation, exceeds the rate of oxygen diffusion across the oil barrier film. As the corrosion accumulates, at approximately week three, the chemistry of the system changed and the rate of oxygen consumption, again due to iron corrosion, fell to a level lower than the rate of oxygen ingress; thereby, resulting in the net increase in dissolved oxygen in the water. In the period after week three, the dissolved oxygen concentration steadily climbed to the equilibrium level of ca. 7.2 ppm comparable to distilled water without the oil barrier. It would appear that at ca. week three the rate of iron oxidation reaches a steady state, either due to passivation, or is perhaps dominated by the further oxidation of Fe^{2+} to $Fe3^{+}$. The latter seems to be supported by the formation of patches of green oxidation products, which could be vivianite, on the nails in the distilled water with paraffin oil solutions. It would have been interesting to have an additional solution of degassed distilled water with an oil barrier film to be able to see if the early, 'oxygen limited', rapid oxidation stage was proportionately extended by first degassing the water to a low oxygen level and then maintaining that low level with the barrier film and oxygen consumption by the iron.

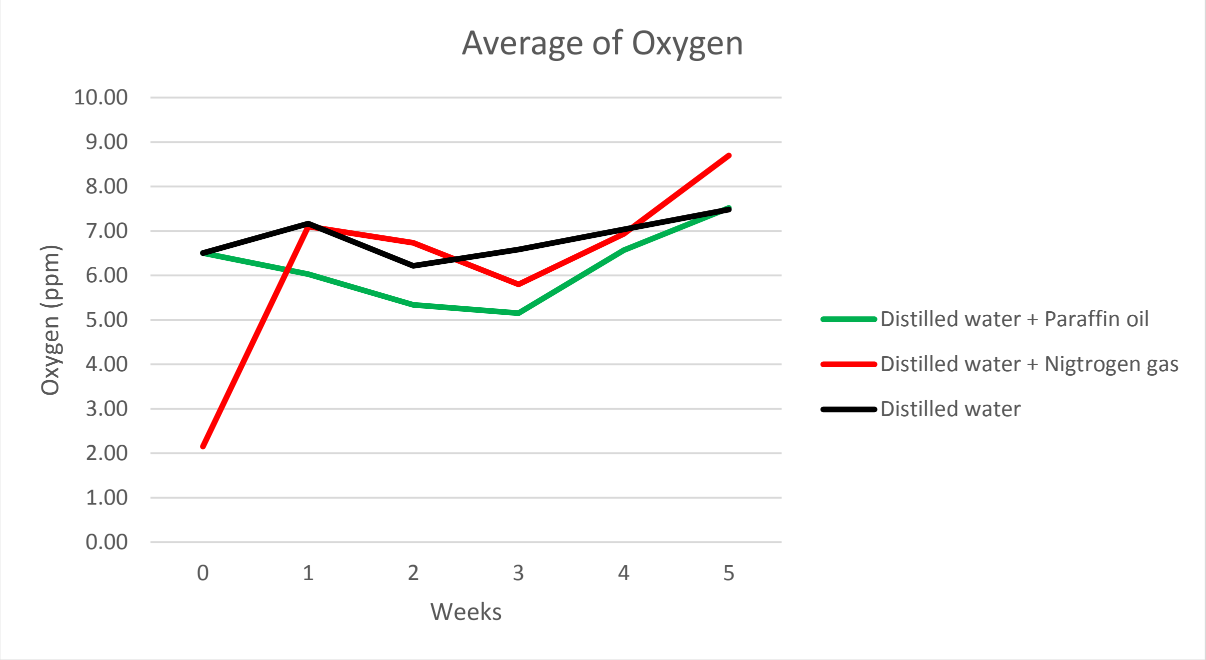


Figure 10: Average measurement of oxygen in three different solutions. The data for each sample is in Appendix 3.

Soluble iron in solutions

The result from the third part of Experiment 1 are shown in Figure 11. There is one clear trend in the data: the distilled water with paraffin oil had far more rust than distilled water degased with nitrogen gas, and distilled water alone.

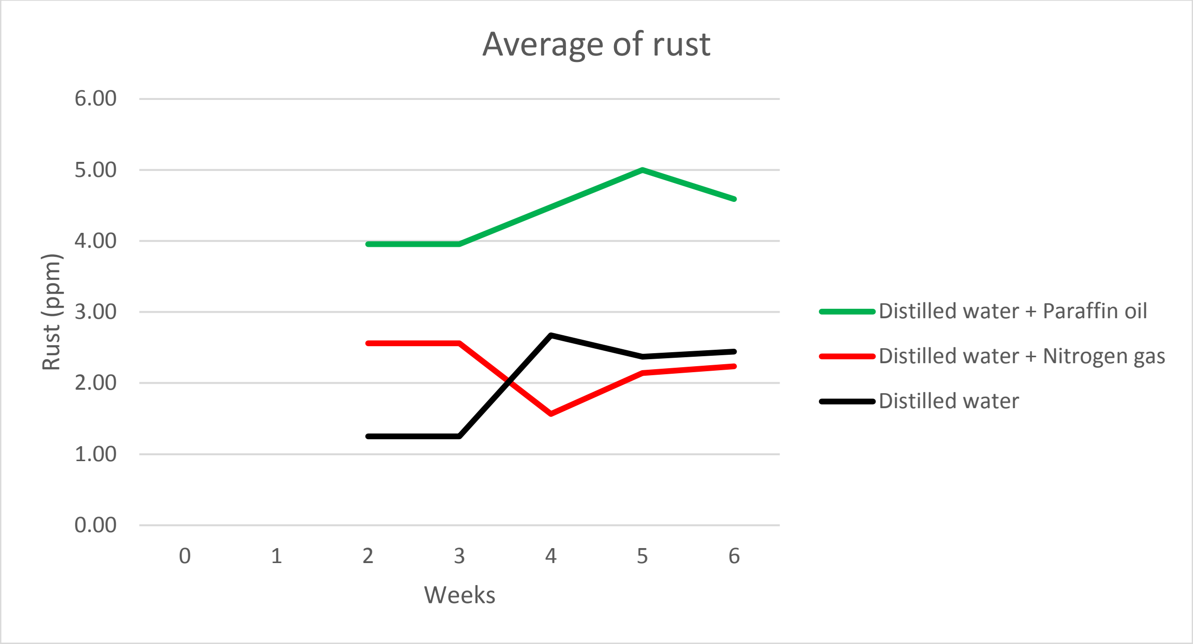


Figure 11: Average measurement of soluble iron in three solutions. The data for each sample is in Appendix 4.

To support the results from the measurement of rust in each solution, the sediment from the solutions was separated at the end of the experiment with filter paper and a Buchner funnel. The results are shown in Figure 12. It is interesting to compare those two results, Figure 11 and Figure 12. Since they correlate to each other, both show that there is most rust in the distilled water with paraffin oil. Also is it interesting that the sodium hydroxide and the disodium phosphate correlate.

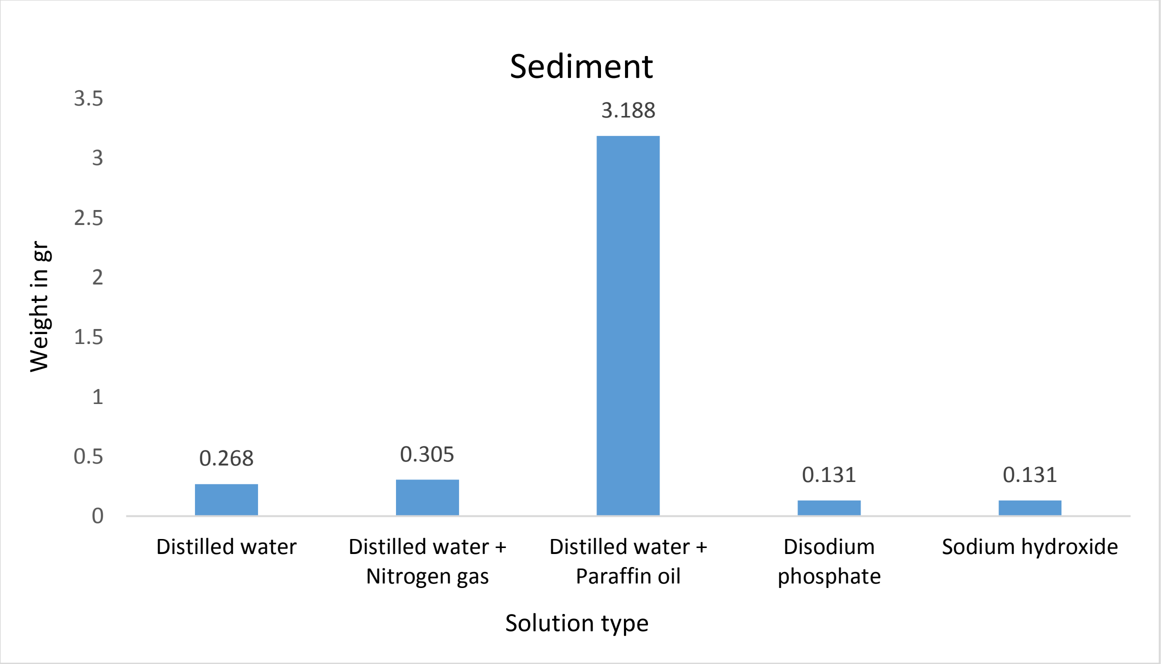


Figure 12: Sum of sediment in different solutions.

The sediment can indicate the type of corrosion on the fragments. In Figures 13 and 14, the colour from each solution is shown. The sediment from the sodium hydroxide and disodium phosphate solutions is almost colourless, and very little sediment was found in these solutions. On the other hand, the other three solutions contained much more sediment: black, orange, and yellow-brown in coloured. In the distilled water and distilled water degased with nitrogen gas solution, the sediment colour was the same for each nail, as can be seen in Figures 13 and 14. It can be seen that the sediment from solutions 3a, b, e and 2a, b, e, have black corrosion while nails 3c, d and 2c, d, have an orange corrosion. The modern iron fragments (marked as f) have yellow-brown corrosion. The fragments from the distilled water with paraffin oil solutions (nr 1) have a slightly more yellowish colour from the oil. When compared to Selwyn's (2004a) list of corrosion products, the black colour of the corrosion can be magnetite (Fe_3O_4) and orange corrosion can be lepidocrocite ($\gamma FeO(OH)$). Yellow-brown corrosion products can be akaganéite (Selwyn 2004a, p. 101)

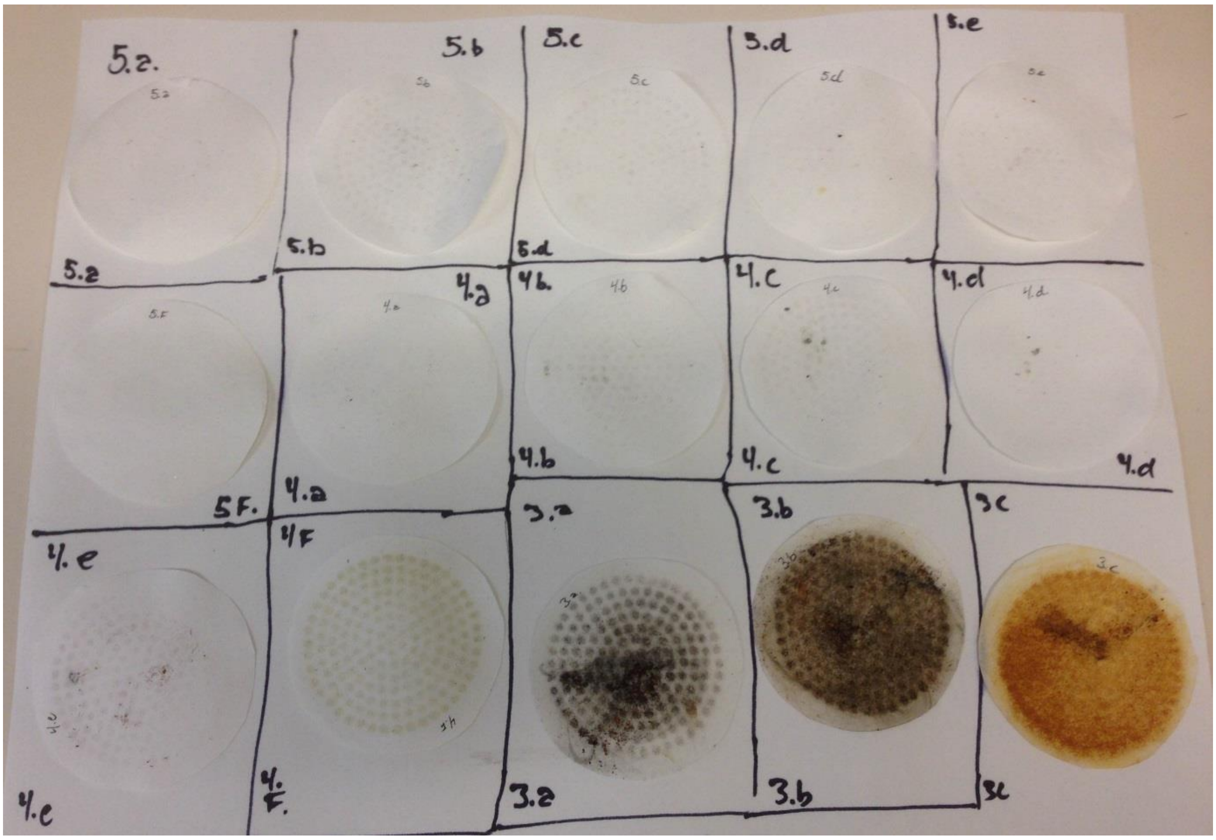


Figure 13: Sediment from NaOH (nr 5), Na_2HPO_4 (nr 4) and part of distilled water (nr 3).

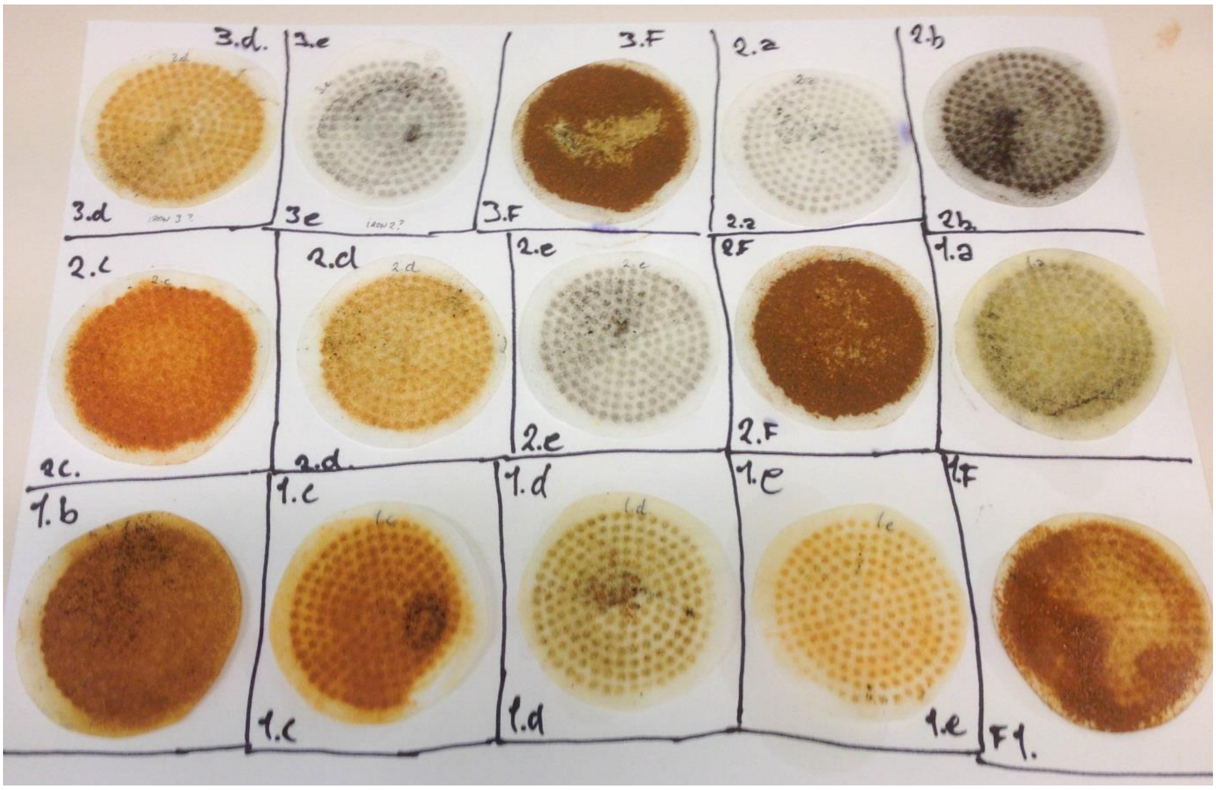


Figure 14: Sediment from distilled water (nr 3), distilled water + nitrogen gas (nr 2) and distilled water with paraffin oil (nr 1).

5.1.4 Experimental discussion

The results from Experiment 1 can be summarised as followed: distilled water with paraffin oil removed a higher amount of chloride than NaOH in the six week period during which this experiment was conducted; although, the removal efficiency of NaOH was only marginally less. Measurement of dissolved oxygen in the solutions demonstrates that distilled water with paraffin oil also gives the best results when the average is taken. In other words, the oil works as a seal. On the other hand, when the soluble iron in the solutions is examined, the distilled water with paraffin oil has the highest amount. The same goes for the sediment results in which distilled water with paraffin oil also has the highest amount of the solutions investigated.

From these results, the conclusion can be drawn that distilled water with paraffin oil is not a satisfactory option for the desalination of iron in composite objects. The desalination of Cl^- from iron is effective, but the corrosion of the iron also progresses, which can be harmful for the object. To support the conclusion from Experiment 1, it is worth reviewing the conclusion from Experiment 2.

5.2 Experiment 2

Factors like relative humidity and oxygen affect the corrosion process of iron (Selwyn 2004a, p. 21). Due to this, it is important to monitor whether or not iron was corroding in each of the five solutions which were used in the experiment. It is understood that the results from this experiment cannot be assumed to be directly applicable to actual archaeological objects, since modern iron fragments do not have any corrosion at the start of the experiment. However, if the modern iron corrodes in the solutions during the experiment, it will indicate that the solution is harmful to iron composite objects.

5.2.1 Aim

The aim for this part of the experiment was to judge the amount of corrosion on modern iron in each solution.

5.2.2 Material and methods

For this experiment, five modern forged iron fragments were used. All the fragments were cleaned by air abrasive using small glass beads and weighed before they were immersed in the solutions (see Chapter 4). Five identical polypropylene (PP) boxes with lids were used with 400 ml of the five different solutions (see Chapter 4.2). The modern iron fragments stayed in the solutions for six weeks, and the solutions were not changed during that time. At the end of the experiment, the fragments were cleaned of corrosion with water and a scalpel and weighted again.

5.2.3 Results

The weight loss on the modern iron fragments from Experiment 2 are shown in Figure 15 as the rate of corrosion.

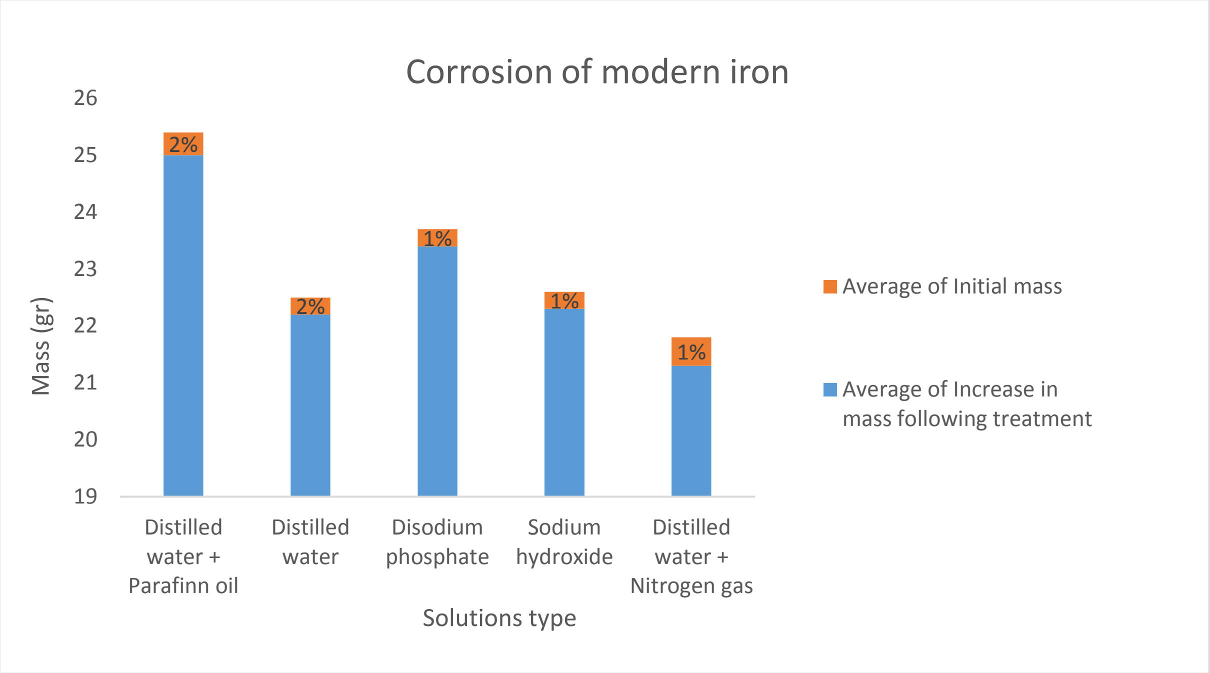


Figure 15. The initial mass of each fragment (orange column) is compared to the final mass (blue column) after all corrosion products have been removed. The difference in mass is expressed as a percentage.

As can be seen, there is no significant weight loss for the modern iron from the solutions. In addition, there is no discernible difference between solutions. All of them show a weight loss between 1-2 %.

5.2.4 Experimental discussion

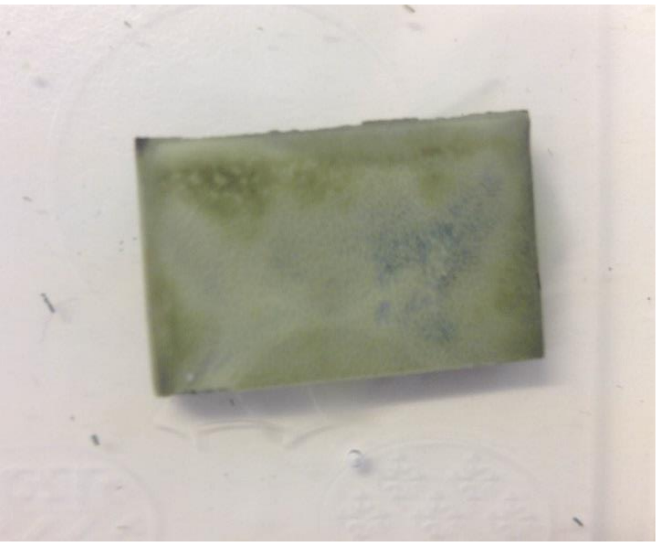


Figure 16: Modern iron in disodium phospahte solutions after 6 weeks.

It is interesting to compare Experiment 2 and part 3, measurement of rust in each solution in Experiment 1 (see 5.1.3). In this experiment, it is obvious that there are no significant results as to how much corrosion there is on each of the fragments in the solutions. However, in Experiment 1, part 3 the results are quite different from Experiment 2. In Experiment 2 it is clear that the objects are corroding more in the solutions with distilled water, distilled water with paraffin oil, and distilled water degased with nitrogen gas, compared to the sodium hydroxide and the disodium phosphate solutions.

The modern iron in the disodium phosphate solution did show a green coating when it was in the solution, as can be seen on Figure 16. This green coating could be the beginning of a corrosion, possible vivianite. Vivianite corrosion can develop when Fe^{2+} ions are oxidized to Fe^{3+} (Selwyn 2004a, p. 101). The same green colour was also found in other solutions, for example in one of the solutions which included distilled water with paraffin oil, as is shown in Figure 17.

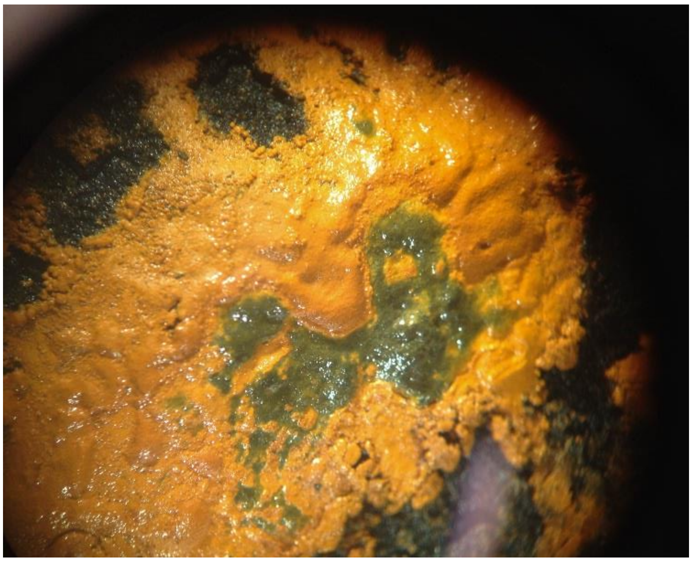


Figure 17: Archaeological iron in distilled water with paraffin oil after 6 weeks.

5.3 Experiment 3

The third experiment was performed to see if frying oil (vegetable oil) could be used as an oxygen barrier instead of paraffin oil (mineral oil) by comparing the rates of oxygen ingress through the oil barrier films. This experiment was also done to see if the vegetable oil could be used as a green and cheaper alternative to paraffin oil.

5.3.1 Aim

The aim for Experiment 3 was to compare the oxygen exclusion properties of frying oil (vegetable) and paraffin oil (mineral oil).

5.3.2 Material and methods

Two types of oils were used for Experiment 3: paraffin oil and frying oil (see Chapter 4.2.3).

The experiment was carried out with six identical polypropylene (PP) boxes with lids that contained about 400 ml of distilled water and distilled water degased with nitrogen gas (N_2). Six boxes prepared with numbers as are described in table 2.

Sample number	Solutions type
1	Distilled water + Paraffin oil
2	Distilled water + Nitrogen gas + Paraffin oil
3	Distilled water + Frying oil
4	Distilled water + Nitrogen gas + Frying oil
5	Distilled water
6	Distilled water + Nitrogen gas

Table 2: Solutions for Experiment 3.

The measurements were carried out at the start, then after 2, 4, 6, 24, 48, 72 and 96 hours.

5.3.3 Results

The results from Experiment 3 can been seen in Figure 18 and 19.

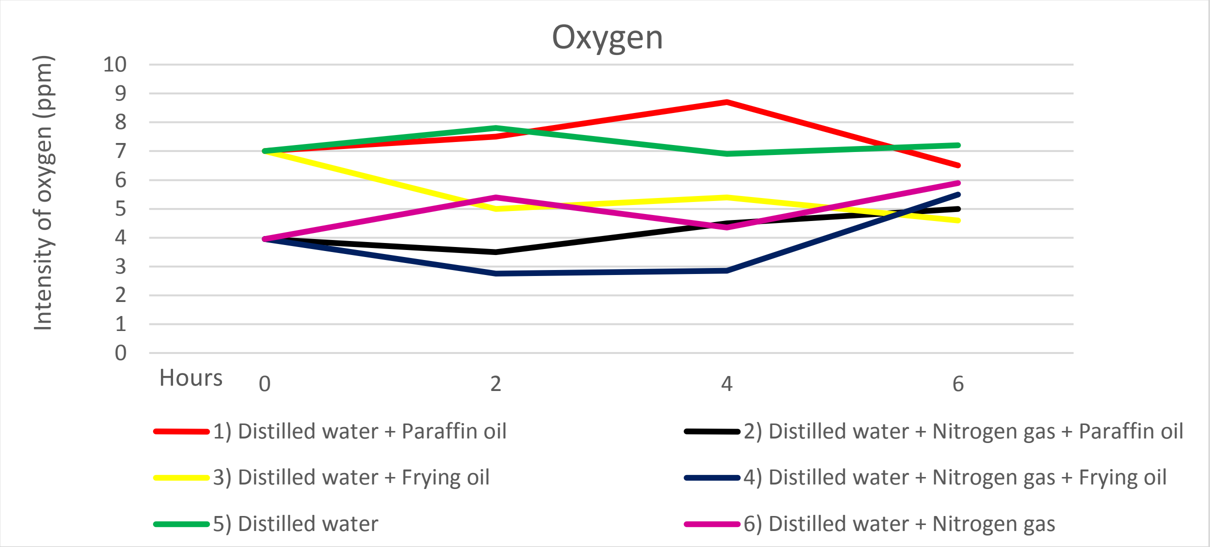


Figure 18: Average of oxygen in six different solutions with measurement of 2 hours between. Name of each sample are in Table 2.

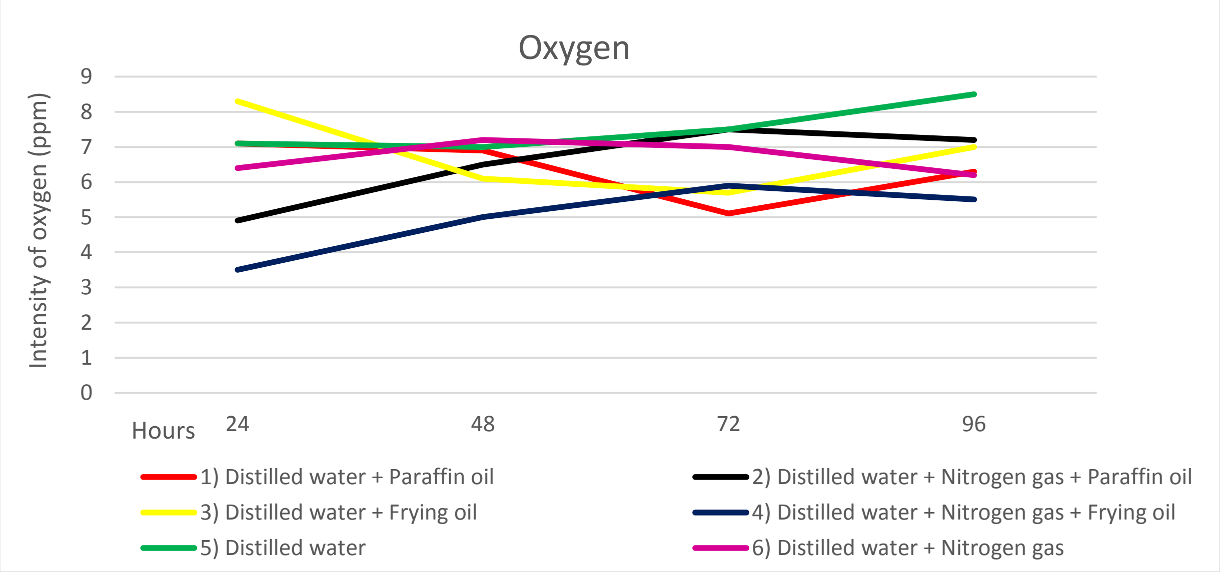


Figure 19: Average of oxygen in six different solutions with measurement of 24 hours between. Name of each sample are in Table 2.

From this one-week experiment it can be seen that in the beginning, the solutions that included nitrogen gas (sample 2, sample 6, and sample 4) are much lower in oxygen concentration than the solutions which only had distilled water (sample 1, sample 3, and sample 5).

The results from Experiment 3 show that the solution with water, N_2 and frying oil gave the best result. The solution with water, nitrogen gas and paraffin oil was rather promising the first 24 hours, but after that the oxygen level increased rapidly. What happens there is quite strange because the solution with water, nitrogen gas and frying oil decreased considerably at the same point. There was quite a difference between paraffin oil and frying oil where the frying oil maintained a lower oxygen level in the solution than the paraffin oil. The results of this experiment would therefore indicate that using frying oil instead of paraffin oil is definitely a better option as an oxygen barrier.

5.4 pH measurement

pH measurement was carried out at the start of the experiment and subsequently throughout the experiment, as explained in Chapter 4.3.1.

In Table 3 it can be seen that the pH was constant for each solution for the duration of the experiment (with the exception of the pH in the disodium phosphate solution due to a mistake with the pH in the initial solution.

Solutions Type	**Start**	**Week 2**	**Week 3**	**Week 4**	**Week 5**	**Week 6**
Distilled water	5	5	5	5	5	5
Distilled water + Nitrogen gas	5	5	5	5	5	5
Distilled water + Parafinn oil	5	5	5	5	5	5
Na2HPO4	12	12	6.5	6.5	6.5	6.5
NaOH	14	13	13	13	13	13

Table 3: Measurement of pH in each solution by week.

6. Discussion and conclusion

6.1 Interpretation of the results

The solution with distilled water and paraffin oil as a seal was effective in removing chloride ions from the object during the desalination time, and compared to the results in Chapter 5.2.3, it removes the soluble Cl^- better than NaOH. The distilled water with paraffin oil showed the lowest dissolved oxygen content of the three solutions tested. Theoretically, this should give minimal corrosion on the iron during the desalination period and prevent further oxidation of the existing corrosion on the object. However, the results showed otherwise. The iron in the solution as well as the sediments within it, indicate that corrosion of the object was the greatest in this solution. Compared to the other results, there is significantly greater corrosion of the object in the solution of distilled water with paraffin oil. This is a strong argument that this method should not be used for archaeological iron or composite objects.

The solution with distilled water was not effective for removing chloride ions from the object, and gave the lowest grade of results for the desalination. In terms of oxygen concentration content, the distilled water had a higher amount than the other solutions in the oxygen experiment. Still the distilled water is more "stable" than the two other distilled water solutions. The results from the rust in the solutions and the sediment for the distilled water solution gives the conclusion that the object does corrode in the solutions however less than in the distilled water with nitrogen gas or distilled water with paraffin oil. Therefore it can be concluded that the use of only distilled water is not a satisfactory choice for the treatment of composites archaeological objects.

The amount of chloride removed in the solution with distilled water degased with nitrogen gas was only slightly better than the solution with only distilled water. For the oxygen content, the results were promising in the first week but then got to the same level as distilled water, as can be seen in Figure 10. The iron in the distilled water degased with nitrogen gas solutions gave the best results of the three solutions which were measured in the experiment. But when the results from the sediments were compared, it could be seen that the distilled water degased with nitrogen gas gave the second worst result after the distilled water with paraffin oil.

The sodium hydroxide solution showed the second best results on removing chloride ions from the object. It has been demonstrated that sodium hydroxide solutions do give a good result in desalination for archaeological iron (Selwyn 2004b, p. 299), so it is no surprise that it did so in this current experiment. No measurements were carried out for the dissolved oxygen or iron in the solution since the measurement method was not compatible with this the solution as has been previously mentioned (see Chapter 5.3.1). The sediment results showed very little corrosion on the object from this solution. This is mostly since it has been shown that iron can be passivated in alkaline solutions (Hjelm-Hansen 1986, p. 11).

The disodium phosphate solution is not comparable to any of the other solutions, because of the mistake made at the start of experiment, as was explained in Chapter 5.2.3.

6.2 Conclusions

This experiment indicates that distilled water with mineral oil as an ‘air seal’ to exclude oxygen for the treatment of composite objects does not work. This conclusion is based on the amount of sediment and the iron in the solutions which showed a high level of corrosion.

6.3 Further research

Since this experiment did not give a positive result, it has to be considered what can be done instead? One of the questions raised during this experiment was whether using distilled water degased with nitrogen gas in combination with frying oil as a seal might be a solution. Since the frying oil sealed better than the paraffin oil, it might be a good option. Due to time limitations in this experiment, no work was put into inspecting the degradation of the oils and how this might affect the objects. It could also be very interesting to look into how oil residues affect the object. Working with oil is always very “greasy” and when the solutions need to be changed during desalination, the object will most likely get oil on it. These questions would be interesting to address in the future, and hopefully there will be further research carried out regarding treating composite archaeological objects.

Figures & Tables

Figures

Photos taken by Sigríður Þorgeirsdóttir if nothing else indicated.

Tables

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E-mail conversation 24. Mars 2015 and 7· May 2015.

Informant 2 Tina Wiinblad.

Conservator, National Museum of Denmark, Copenhagen, Denmark.

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Informant 3 Ian MacLeod and Jon Carpenter.

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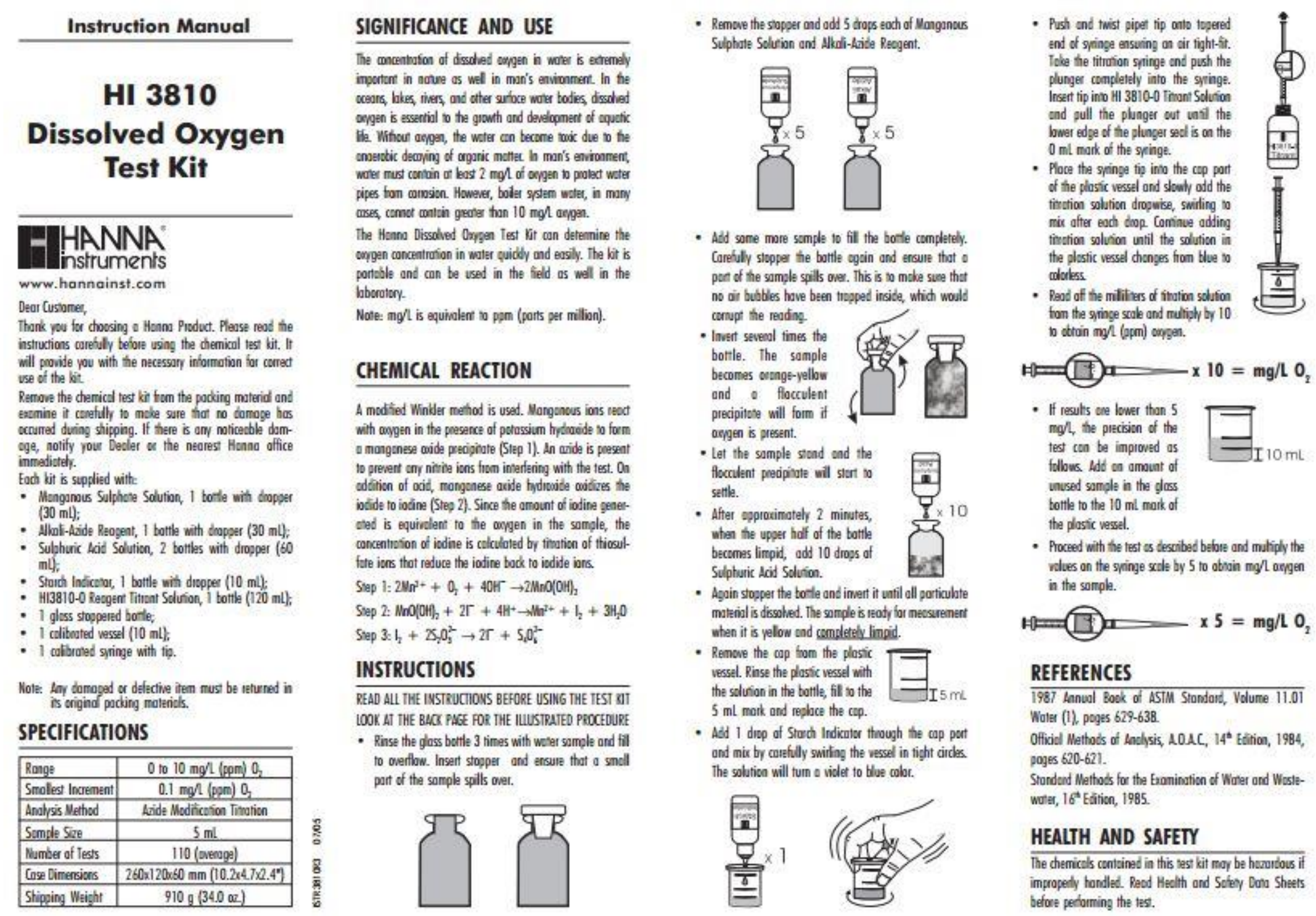
Appendix 1

Winkle method with instruction of the Hanna Instrument for dissolved oxygen test kit.



Instruction Manual

HI 3810
Dissolved Oxygen Test Kit

HANNA instruments

www.hannainst.com

Dear Customer,

Thank you for choosing a Hanna Product. Please read the instructions carefully before using the chemical test kit. It will provide you with the necessary information for correct use of the kit.

Remove the chemical test kit from the packing material and examine it carefully to make sure that no damage has occurred during shipping. If there is any noticeable damage, notify your Dealer or the nearest Hanna office immediately.

Each kit is supplied with:

- Manganous Sulphate Solution, 1 bottle with dropper (30 mL);
- Alkali-Azide Reagent, 1 bottle with dropper (30 mL);
- Sulphuric Acid Solution, 2 bottles with dropper (60 mL);
- Starch Indicator, 1 bottle with dropper (10 mL);
- HI3810-0 Reagent Titrant Solution, 1 bottle (120 mL);
- 1 glass stoppered bottle;
- 1 calibrated vessel (10 mL);
- 1 calibrated syringe with tip.

Note: Any damaged or defective item must be returned in its original packing materials.

SPECIFICATIONS

Range	0 to 10 mg/L (ppm) O_2
Smallest Increment	0.1 mg/L (ppm) O_2
Analysis Method	Azide Modification Titration
Sample Size	5 mL
Number of Tests	110 (average)
Case Dimensions	260x120x60 mm (10.2x4.7x2.4")
Shipping Weight	910 g (34.0 oz.)

ISTR3810R3 07/05

SIGNIFICANCE AND USE

The concentration of dissolved oxygen in water is extremely important in nature as well in man's environment. In the oceans, lakes, rivers, and other surface water bodies, dissolved oxygen is essential to the growth and development of aquatic life. Without oxygen, the water can become toxic due to the anaerobic decaying of organic matter. In man's environment, water must contain at least 2 mg/L of oxygen to protect water pipes from corrosion. However, boiler system water, in many cases, cannot contain greater than 10 mg/L oxygen.

The Hanna Dissolved Oxygen Test Kit can determine the oxygen concentration in water quickly and easily. The kit is portable and can be used in the field as well in the laboratory.

Note: mg/L is equivalent to ppm (parts per million).

CHEMICAL REACTION

A modified Winkler method is used. Manganous ions react with oxygen in the presence of potassium hydroxide to form a manganese oxide precipitate (Step 1). An azide is present to prevent any nitrite ions from interfering with the test. On addition of acid, manganese oxide hydroxide oxidizes the iodide to iodine (Step 2). Since the amount of iodine generated is equivalent to the oxygen in the sample, the concentration of iodine is calculated by titration of thiosulfate ions that reduce the iodine back to iodide ions.

Step 1: $2Mn^{2+} + O_2 + 4OH^- \rightarrow 2MnO(OH)_2$

Step 2: $MnO(OH)_2 + 2I^- + 4H^+ \rightarrow Mn^{2+} + I_2 + 3H_2O$

Step 3: $I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$

INSTRUCTIONS

READ ALL THE INSTRUCTIONS BEFORE USING THE TEST KIT
LOOK AT THE BACK PAGE FOR THE ILLUSTRATED PROCEDURE

- Rinse the glass bottle 3 times with water sample and fill to overflow. Insert stopper and ensure that a small part of the sample spills over.
- Remove the stopper and add 5 drops each of Manganous Sulphate Solution and Alkali-Azide Reagent.
- Add some more sample to fill the bottle completely. Carefully stopper the bottle again and ensure that a part of the sample spills over. This is to make sure that no air bubbles have been trapped inside, which would corrupt the reading.
- Invert several times the bottle. The sample becomes orange-yellow and a flocculent precipitate will form if oxygen is present.
- Let the sample stand and the flocculent precipitate will start to settle.
- After approximately 2 minutes, when the upper half of the bottle becomes limpid, add 10 drops of Sulphuric Acid Solution.
- Again stopper the bottle and invert it until all particulate material is dissolved. The sample is ready for measurement when it is yellow and <u>completely limpid</u>.
- Remove the cap from the plastic vessel. Rinse the plastic vessel with the solution in the bottle, fill to the 5 mL mark and replace the cap.
- Add 1 drop of Starch Indicator through the cap port and mix by carefully swirling the vessel in tight circles. The solution will turn a violet to blue color.
- Push and twist pipet tip onto tapered end of syringe ensuring an air tight-fit. Take the titration syringe and push the plunger completely into the syringe. Insert tip into HI 3810-0 Titrant Solution and pull the plunger out until the lower edge of the plunger seal is on the 0 mL mark of the syringe.
- Place the syringe tip into the cap port of the plastic vessel and slowly add the titration solution dropwise, swirling to mix after each drop. Continue adding titration solution until the solution in the plastic vessel changes from blue to colorless.
- Read off the milliliters of titration solution from the syringe scale and multiply by 10 to obtain mg/L (ppm) oxygen.

x 10 = mg/L O_2

- If results are lower than 5 mg/L, the precision of the test can be improved as follows. Add an amount of unused sample in the glass bottle to the 10 mL mark of the plastic vessel.
- Proceed with the test as described before and multiply the values on the syringe scale by 5 to obtain mg/L oxygen in the sample.

x 5 = mg/L O_2

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HEALTH AND SAFETY

The chemicals contained in this test kit may be hazardous if improperly handled. Read Health and Safety Data Sheets before performing the test.

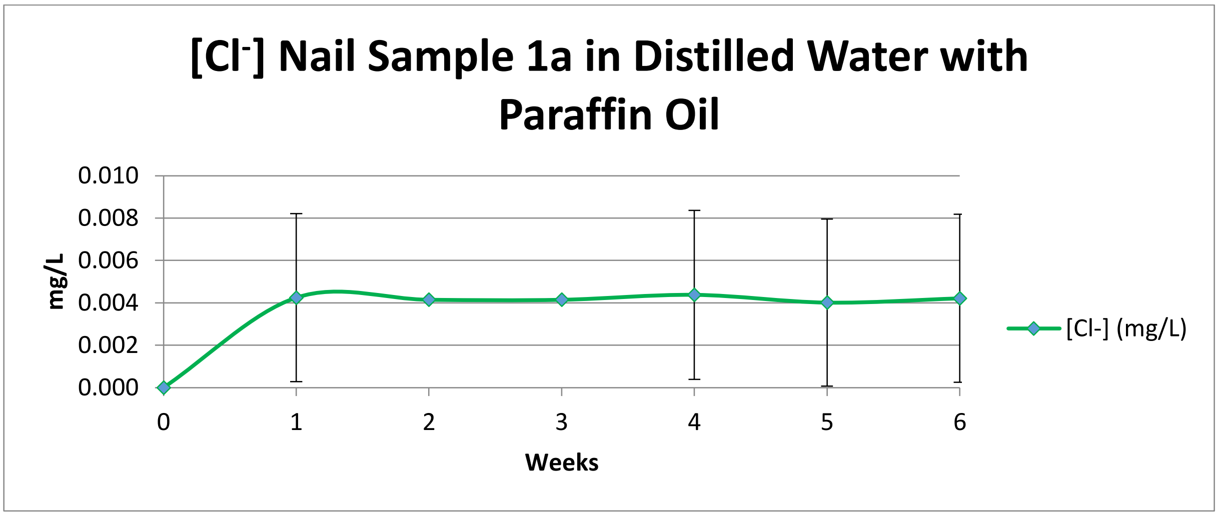
Appendix 2

Data for $[Cl^-]$ for each fragment in each solution.

$$[Cl^-] \text{ in mg/L} = \frac{\text{Average Chloride measurment} + 35{,}453 \text{ (atomic mass for chloride)}}{10}$$

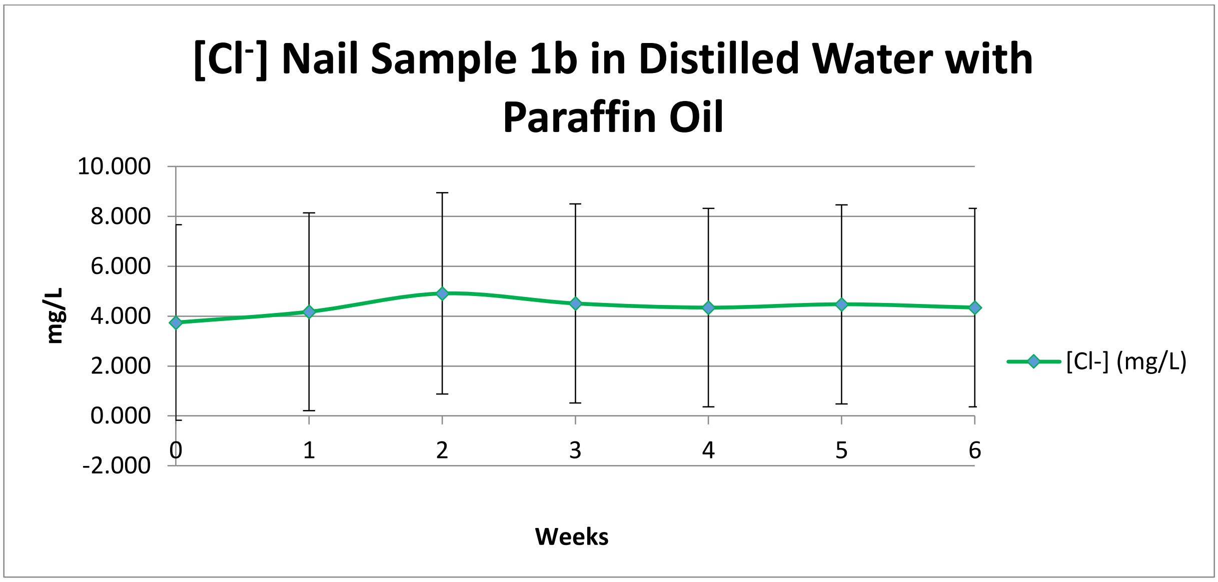
Distilled water with paraffin oil, sample 1.a:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
1.a	0	0	0	0	0.000	0.000	0.000
1.a	1	11	4	6	7.000	4.245	3.970
1.a	2	6	6	6	6.000	4.145	0.000
1.a	3	6	6	6	6.000	4.145	0.000
1.a	4	13	0	12	8.333	4.379	3.983
1.a	5	7	7	0	4.667	4.012	3.946
1.a	6	7	6	7	6.667	4.212	3.966



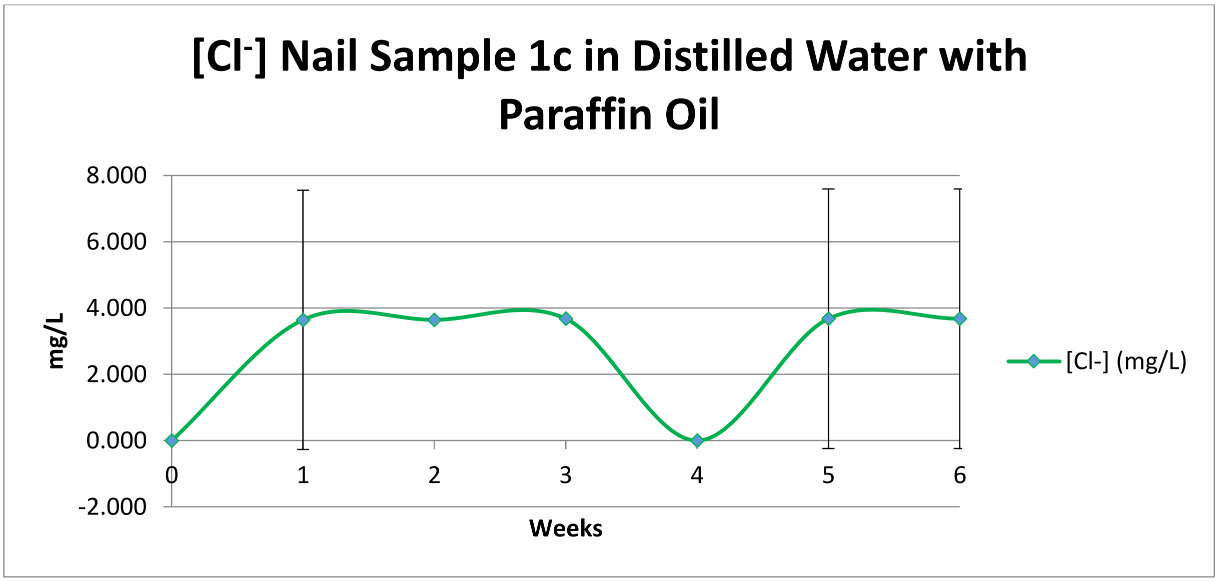
Distilled water with paraffin oil, sample 1.b:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
1.b	0	6	0	0	2.000	3.745	3.920
1.b	1	7	5	7	6.333	4.179	3.963
1.b	2	7	25	9	13.667	4.912	4.036
1.b	3	7	8	14	9.667	4.512	3.996
1.b	4	8	7	9	8.000	4.345	3.980
1.b	5	14	7	7	9.333	4.479	3.993
1.b	6	8	8	8	8.000	4.345	3.980



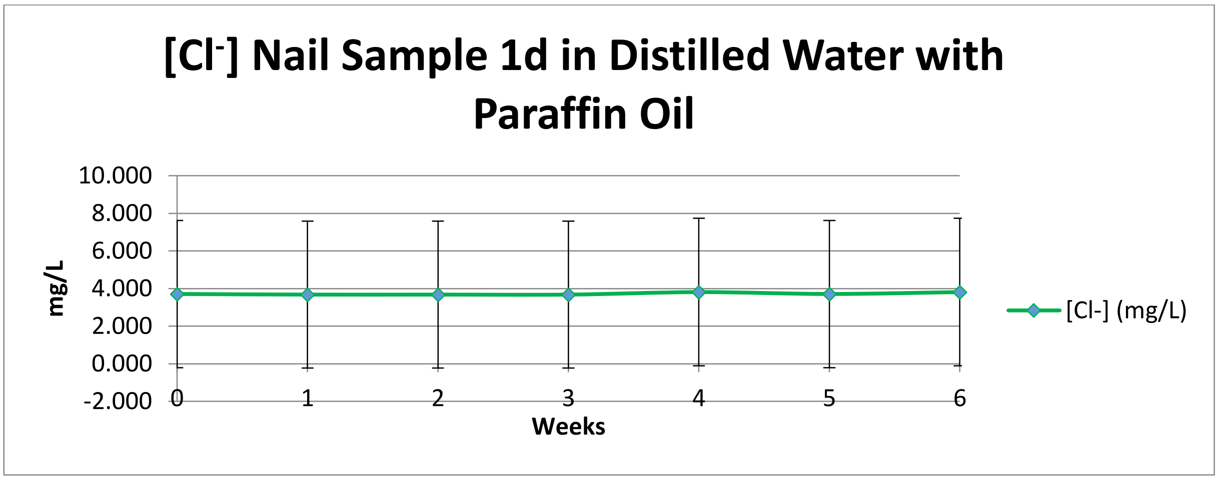
Distilled water with paraffin oil, sample 1.c:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
1.c	0	0	0	0	0.000	0.000	0.000
1.c	1	0	0	3	1.000	3.645	3.910
1.c	2	0	0	3	1.000	3.645	0.000
1.c	3	0	0	4	1.333	3.679	0.000
1.c	4	0	0	0	0.000	0.000	0.000
1.c	5	0	0	4	1.333	3.679	3.913
1.c	6	0	0	4	1.333	3.679	3.913



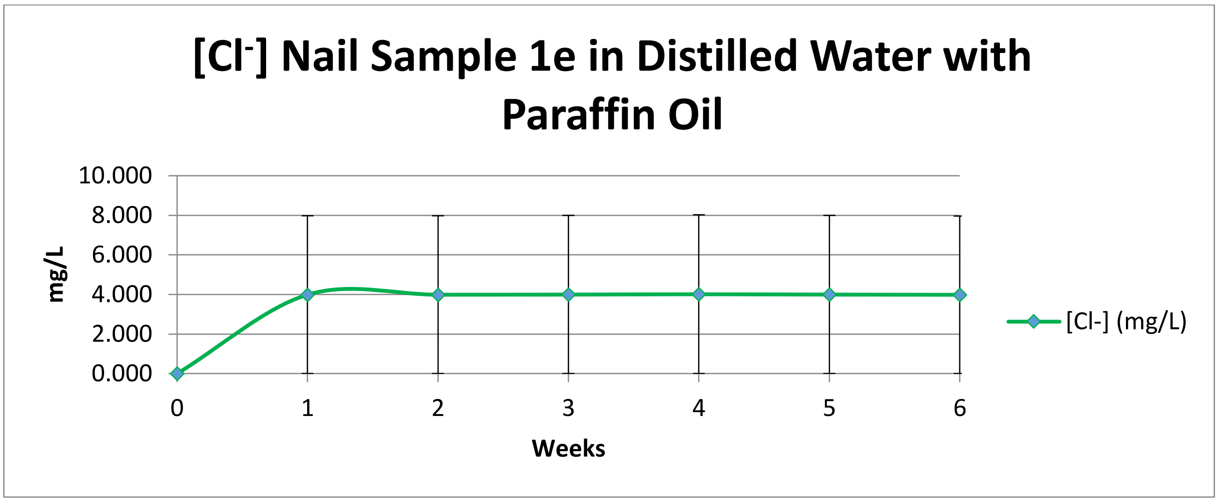
Distilled water with paraffin oil, sample 1.d:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
1.d	0	5	0	0	1.667	3.712	3.916
1.d	1	4	0	0	1.333	3.679	3.913
1.d	2	4	0	0	1.333	3.679	3.913
1.d	3	0	4	0	1.333	3.679	3.913
1.d	4	8	0	0	2.667	3.812	3.926
1.d	5	0	5	0	1.667	3.712	3.916
1.d	6	0	0	8	2.667	3.812	3.926



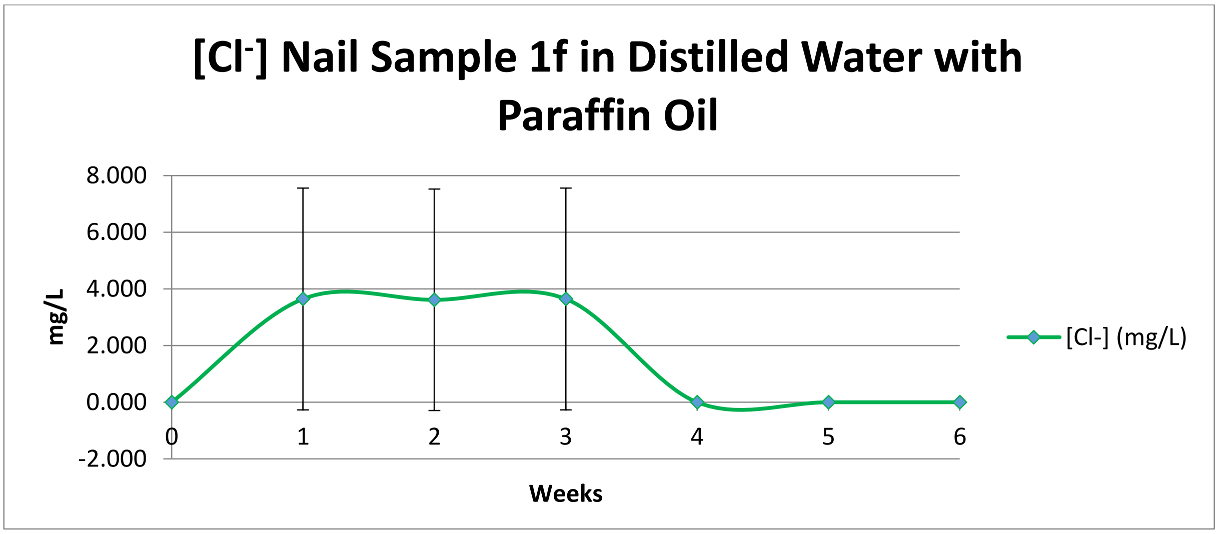
Distilled water with paraffin oil, sample 1.e:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
1.e	0	0	0	0	0.000	0.000	0.000
1.e	1	11	9	7	9.000	4.445	3.990
1.e	2	10	9	8	9.000	4.445	3.990
1.e	3	12	9	8	9.667	4.512	3.996
1.e	4	14	8	12	11.333	4.679	4.013
1.e	5	12	9	8	9.667	4.512	3.996
1.e	6	8	9	9	8.667	4.412	3.986



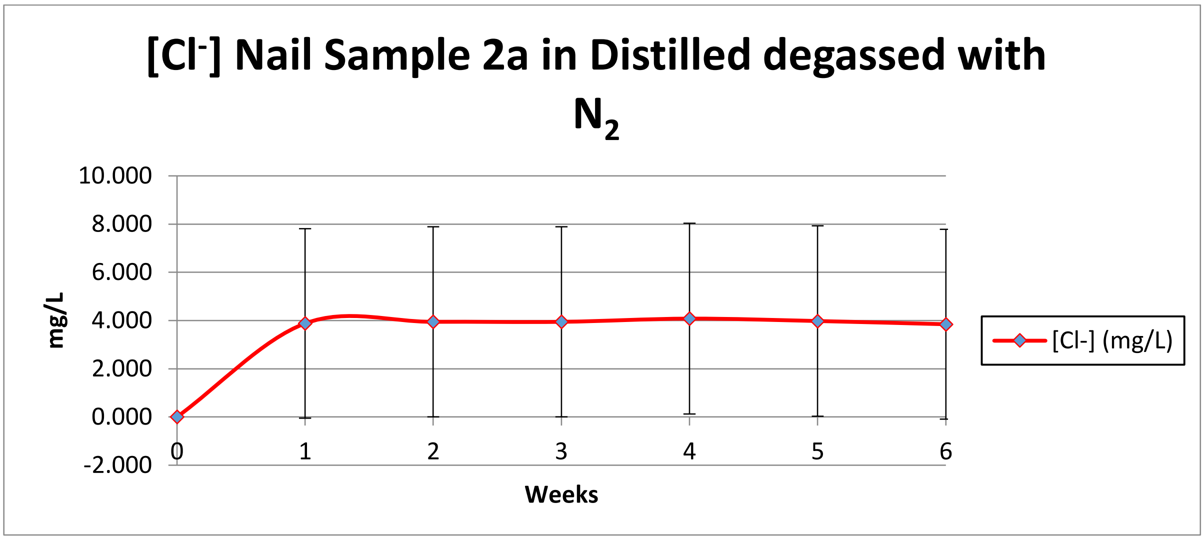
Distilled water with paraffin oil, sample 1.f:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
1.f	0	0	0	0	0.000	0.000	0.000
1.f	1	0	0	3	1.000	3.645	3.910
1.f	2	0	0	2	0.667	3.612	3.906
1.f	3	0	0	3	1.000	3.645	3.910
1.f	4	0	0	0	0.000	0.000	0.000
1.f	5	0	0	0	0.000	0.000	0.000
1.f	6	0	0	0	0.000	0.000	0.000



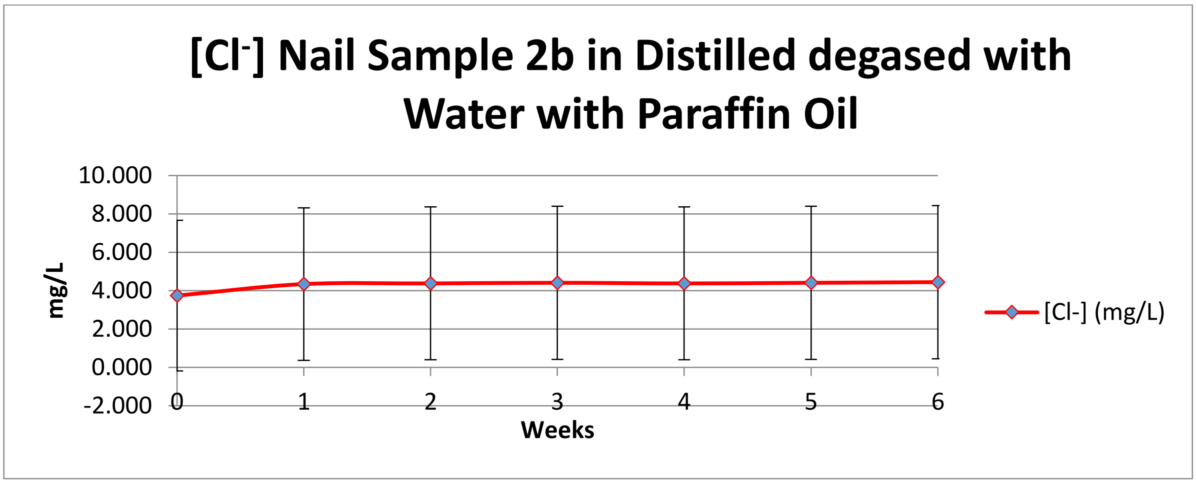
Distilled water degassed with N_2, sample 2.a:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
2.a	0	0	0	0	0.000	0.000	0.000
2.a	1	5	1	4	3.333	3.879	3.933
2.a	2	5	3	4	4.000	3.945	3.940
2.a	3	4	0	8	4.000	3.945	3.940
2.a	4	8	0	8	5.333	4.079	3.953
2.a	5	0	8	5	4.333	3.979	3.943
2.a	6	5	0	4	3.000	3.845	3.930



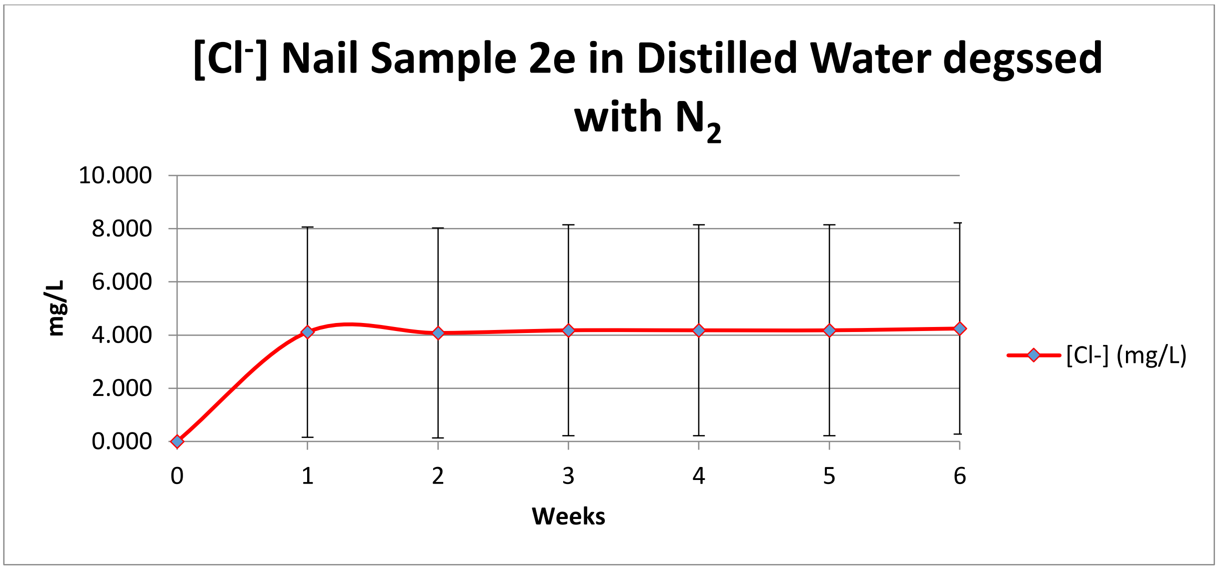
Distilled water degassed with N_2, sample 2.b:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
2.b	0	6	0	0	2.000	3.745	3.920
2.b	1	8	8	8	8.000	4.345	3.980
2.b	2	9	8	8	8.333	4.379	3.983
2.b	3	8	9	9	8.667	4.412	3.986
2.b	4	9	7	9	8.333	4.379	3.983
2.b	5	9	9	8	8.667	4.412	3.986
2.b	6	9	9	9	9.000	4.445	3.990



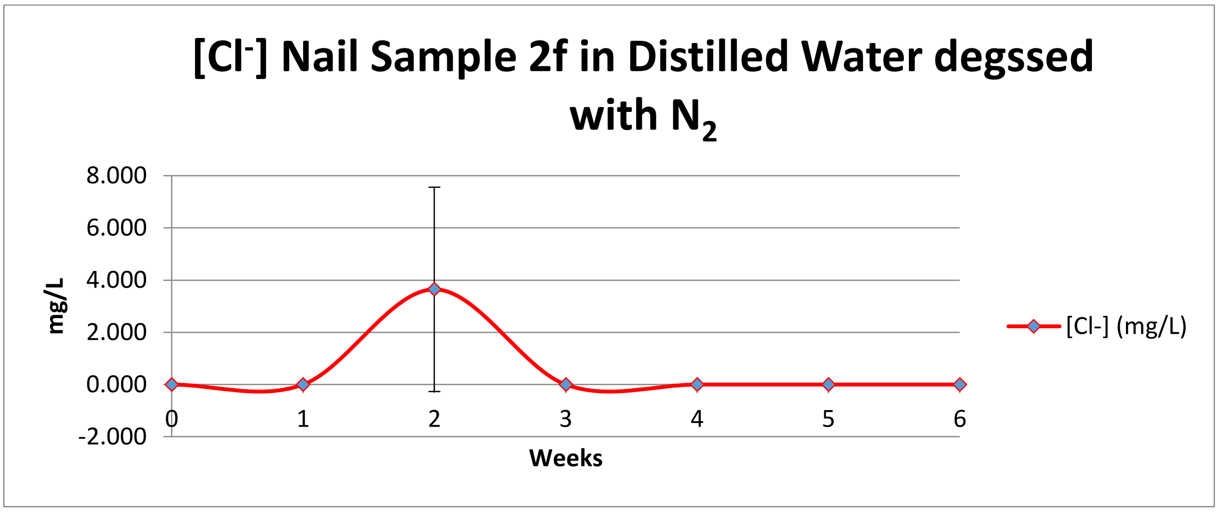
Distilled water degassed with N_2, sample 2.e:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
2.e	0	0	0	0	0.000	0.000	0.000
2.e	1	7	4	6	5.667	4.112	3.956
2.e	2	7	4	5	5.333	4.079	3.953
2.e	3	9	5	5	6.333	4.179	3.963
2.e	4	9	0	10	6.333	4.179	3.963
2.e	5	8	5	6	6.333	4.179	3.963
2.e	6	11	0	10	7.000	4.245	3.970



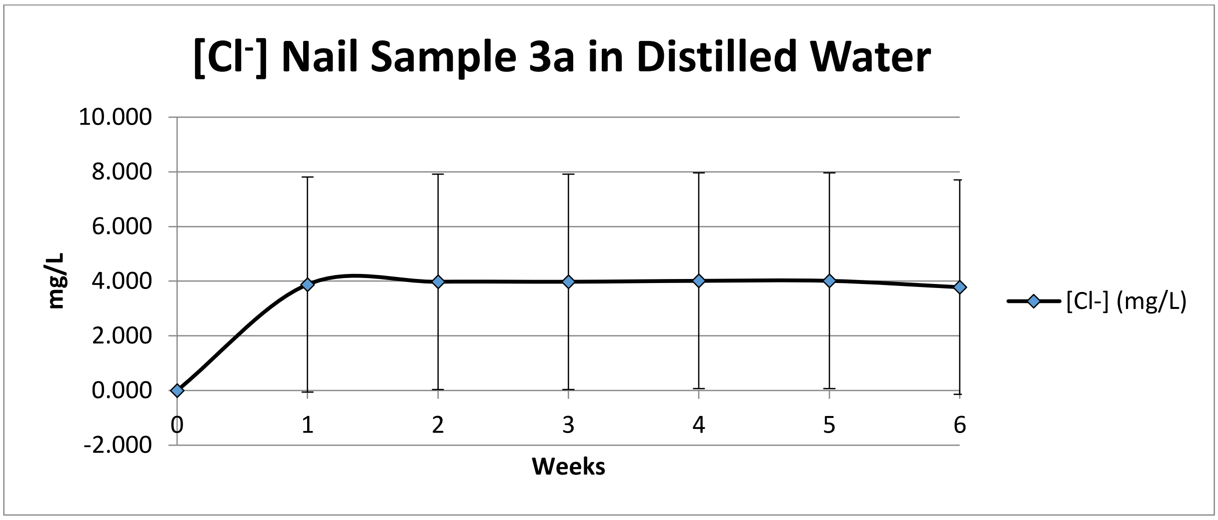
Distilled water degassed with N_2, sample 2.f:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
2.f	0	0	0	0	0.000	0.000	0.000
2.f	1	0	0	0	0.000	0.000	0.000
2.f	2	0	3	0	1.000	3.645	3.910
2.f	3	0	0	0	0.000	0.000	0.000
2.f	4	0	0	0	0.000	0.000	0.000
2.f	5	0	0	0	0.000	0.000	0.000
2.f	6	0	0	0	0.000	0.000	0.000



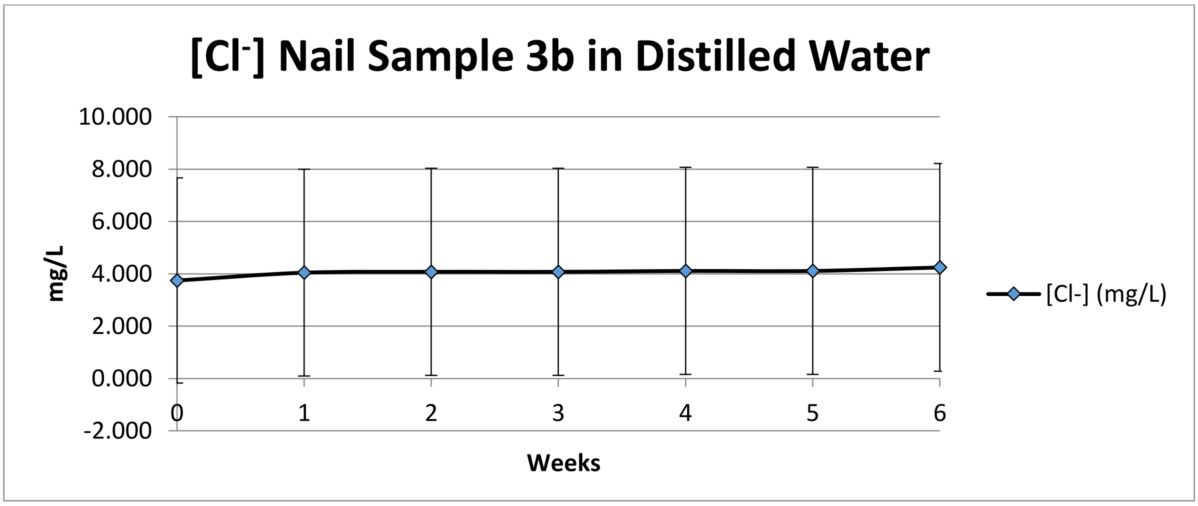
Distilled water, sample 3.a:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
3.a	0	0	0	0	0.000	0.000	0.000
3.a	1	5	0	5	3.333	3.879	3.933
3.a	2	7	0	6	4.333	3.979	0.000
3.a	3	7	0	6	4.333	3.979	0.000
3.a	4	6	0	8	4.667	4.012	3.946
3.a	5	6	0	8	4.667	4.012	3.946
3.a	6	0	7	0	2.333	3.779	3.923



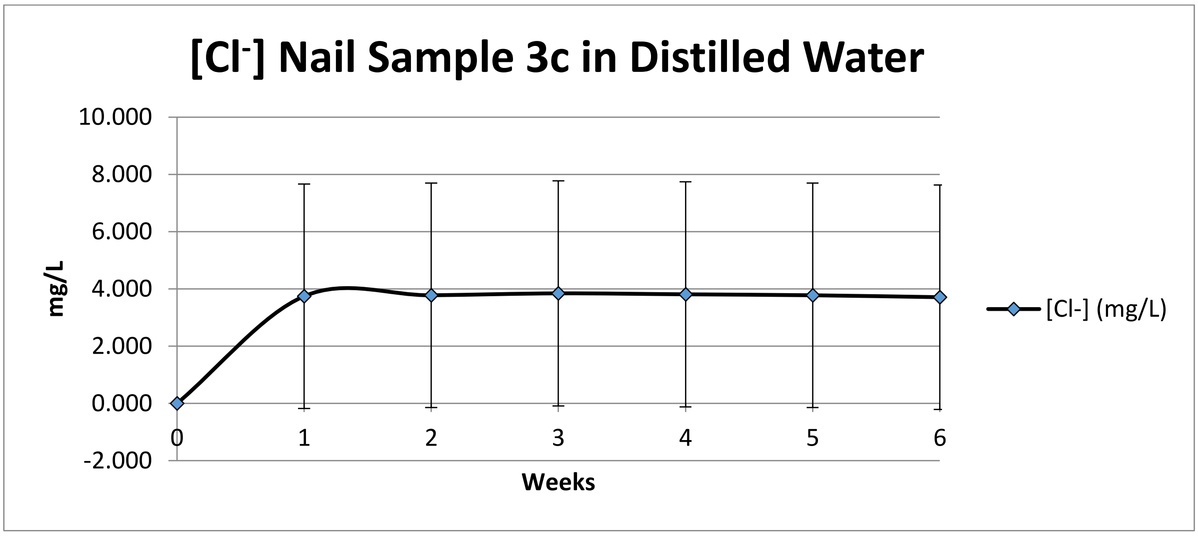
Distilled water, sample 3.b:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻]meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
3.b	0	6	0	0	2.000	3.745	3.920
3.b	1	6	5	4	5.000	4.045	3.950
3.b	2	6	5	5	5.333	4.079	3.953
3.b	3	5	5	6	5.333	4.079	3.953
3.b	4	5	6	6	5.667	4.112	3.956
3.b	5	5	6	6	5.667	4.112	3.956
3.b	6	9	6	6	7.000	4.245	3.970



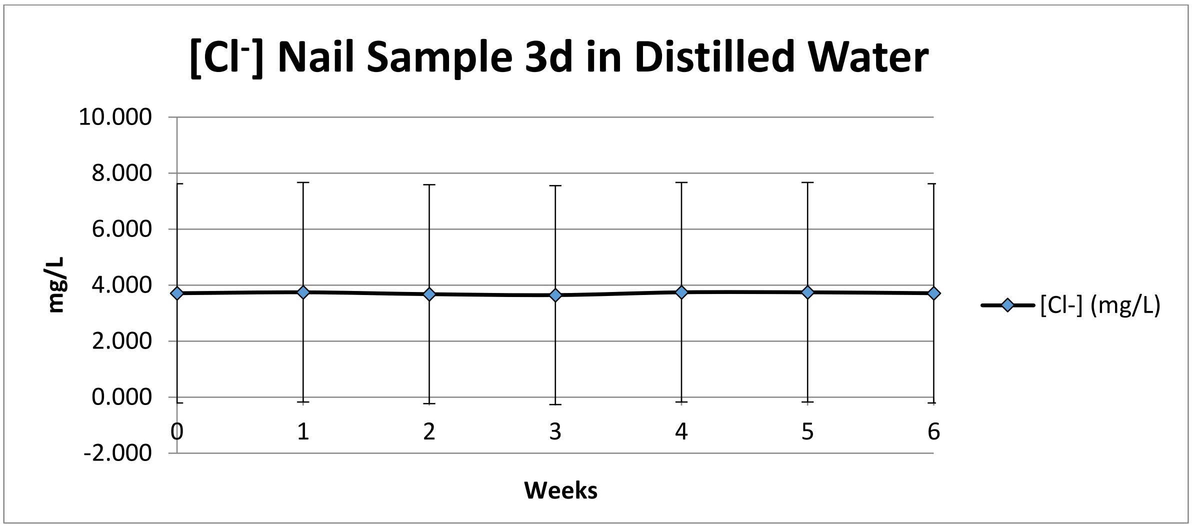
Distilled water, sample 3.c:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
3.c	0	0	0	0	0.000	0.000	0.000
3.c	1	0	5	1	2.000	3.745	3.920
3.c	2	4	3	0	2.333	3.779	3.923
3.c	3	0	5	4	3.000	3.845	3.930
3.c	4	0	8	0	2.667	3.812	3.926
3.c	5	0	7	0	2.333	3.779	3.923
3.c	6	0	0	5	1.667	3.712	3.916



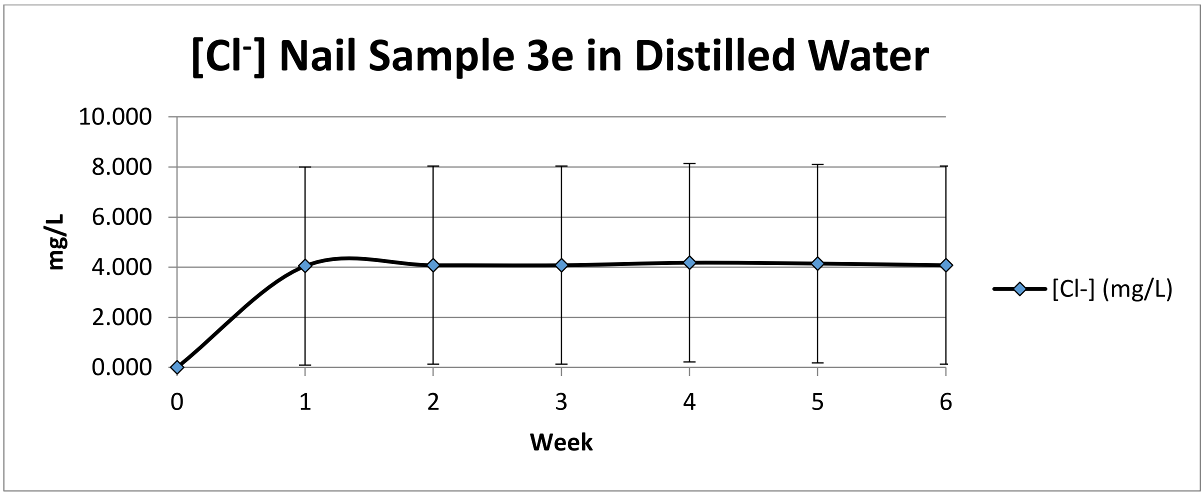
Distilled water, sample 3.d:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
3.d	0	5	0	0	1.667	3.712	3.916
3.d	1	6	0	0	2.000	3.745	3.920
3.d	2	4	0	0	1.333	3.679	3.913
3.d	3	0	0	3	1.000	3.645	3.910
3.d	4	0	6	0	2.000	3.745	3.920
3.d	5	0	6	0	2.000	3.745	3.920
3.d	6	0	0	5	1.667	3.712	3.916



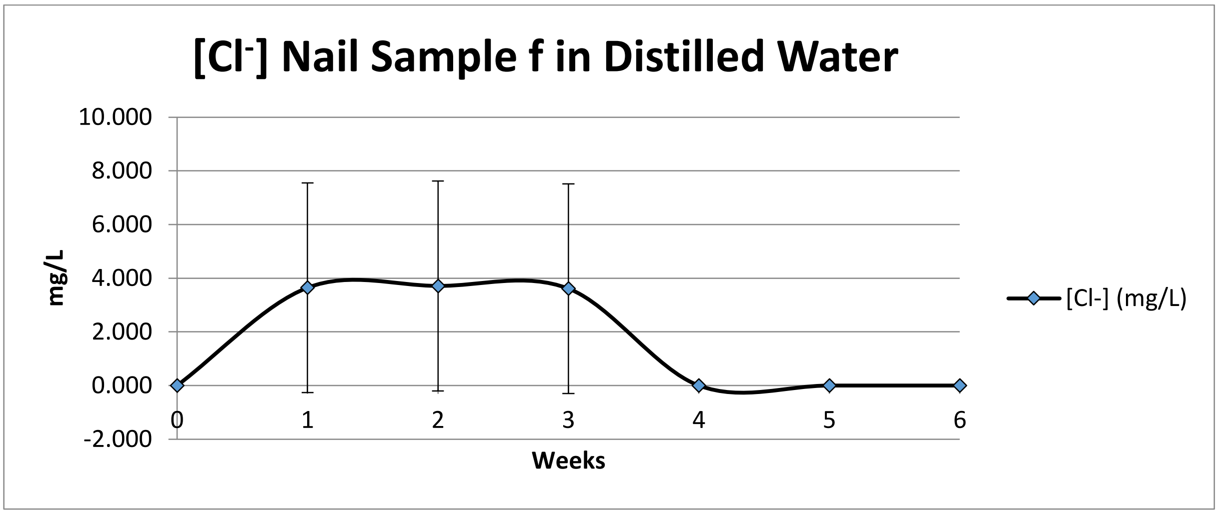
Distilled water, sample 3.e:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
3.e	0	0	0	0	0.000	0.000	0.000
3.e	1	6	4	5	5.000	4.045	3.950
3.e	2	6	5	5	5.333	4.079	3.953
3.e	3	6	5	5	5.333	4.079	3.953
3.e	4	7	0	12	6.333	4.179	3.963
3.e	5	7	5	6	6.000	4.145	3.960
3.e	6	5	5	6	5.333	4.079	3.953



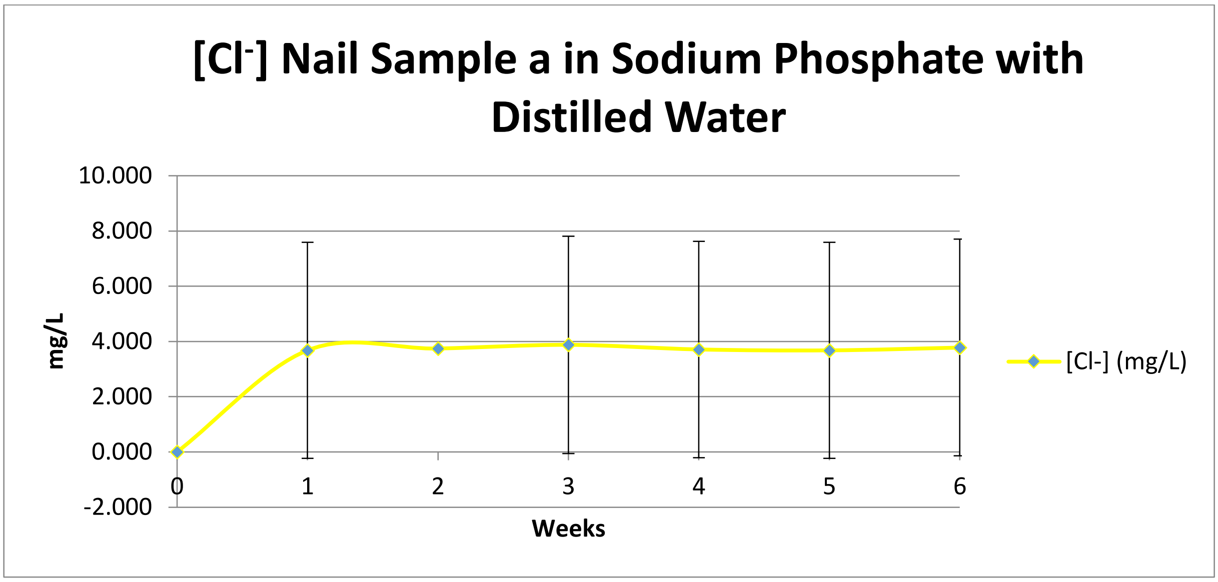
Distilled water, sample 3.f:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
3.f	0	0	0	0	0.000	0.000	0.000
3.f	1	3	0	0	1.000	3.645	3.910
3.f	2	2	0	3	1.667	3.712	3.916
3.f	3	0	2	0	0.667	3.612	3.906
3.f	4	0	0	0	0.000	0.000	0.000
3.f	5	0	0	0	0.000	0.000	0.000
3.f	6	0	0	0	0.000	0.000	0.000



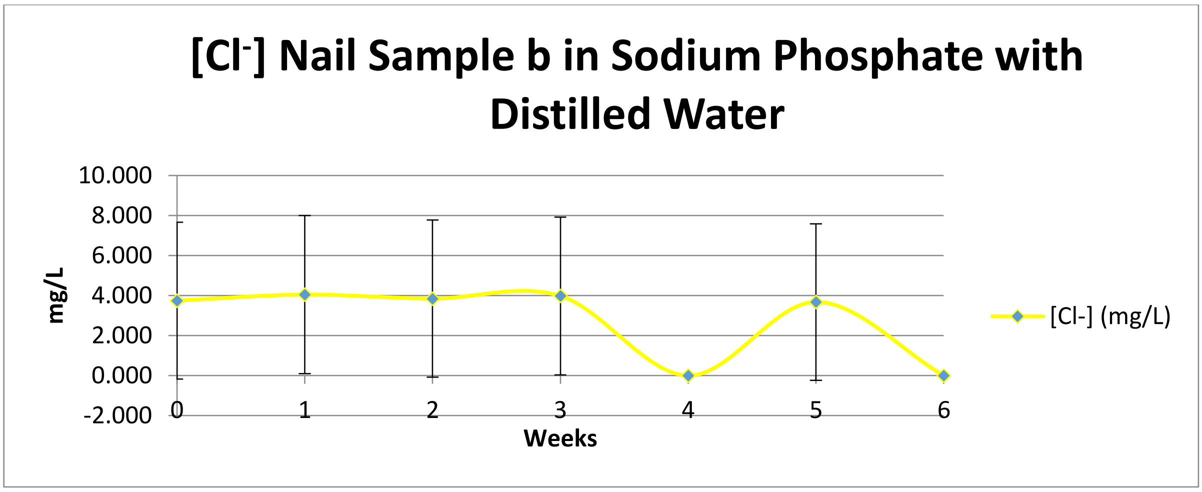
Sodium Phosphate in Distilled water, sample 4.a:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.a	0	0	0	0	0.000	0.000	0.000
4.a	1	4	0	0	1.333	3.679	3.913
4.a	2	2	2	2	2.000	3.745	0.000
4.a	3	6	0	4	3.333	3.879	3.933
4.a	4	0	5	0	1.667	3.712	3.916
4.a	5	4	0	0	1.333	3.679	3.913
4.a	6	7	0	0	2.333	3.779	3.923



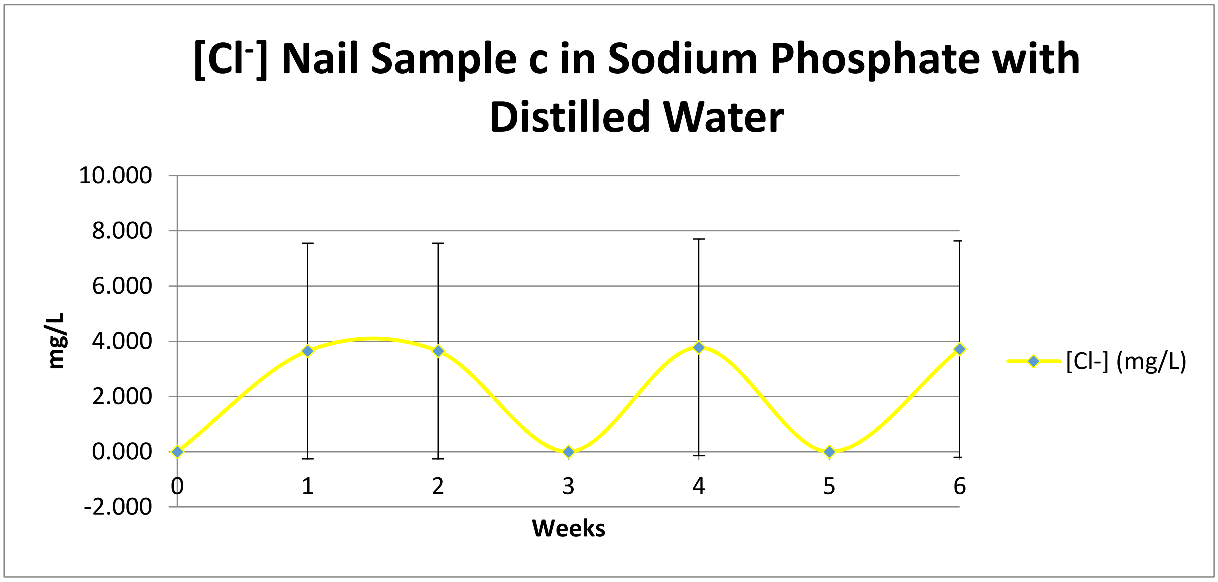
Sodium Phosphate in Distilled water, sample 4.b:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.b	0	6	0	0	2.000	3.745	3.920
4.b	1	8	0	7	5.000	4.045	3.950
4.b	2	5	4	0	3.000	3.845	3.930
4.b	3	0	8	5	4.333	3.979	3.943
4.b	4	0	0	0	0.000	0.000	0.000
4.b	5	0	4	0	1.333	3.679	3.913
4.b	6	0	0	0	0.000	0.000	0.000



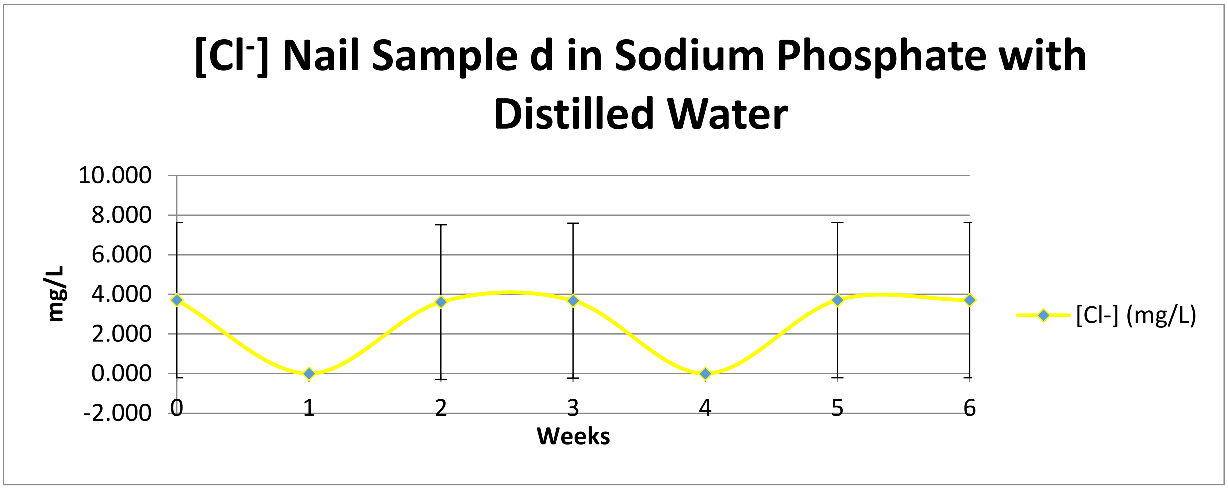
Sodium Phosphate in Distilled water, sample 4.c:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.c	0	0	0	0	0.000	0.000	0.000
4.c	1	0	1	2	1.000	3.645	3.910
4.c	2	0	0	3	1.000	3.645	3.910
4.c	3	0	0	0	0.000	0.000	0.000
4.c	4	0	7	0	2.333	3.779	0.000
4.c	5	0	0	0	0.000	0.000	0.000
4.c	6	0	5	0	1.667	3.712	3.916



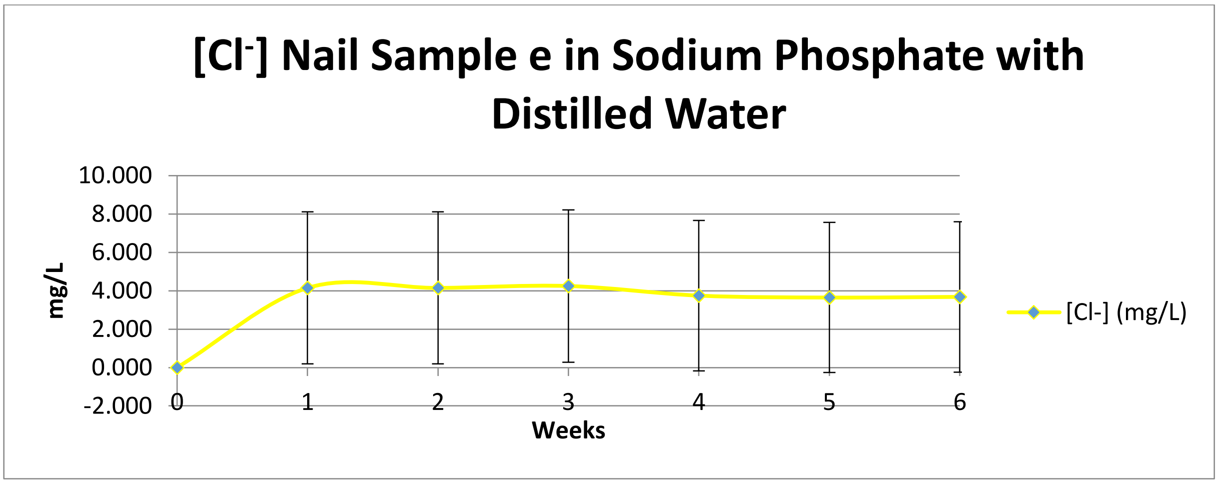
Sodium Phosphate in Distilled water, sample 4.d:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.d	0	5	0	0	1.667	3.712	3.916
4.d	1	0	0	0	0.000	0.000	0.000
4.d	2	0	2	0	0.667	3.612	3.906
4.d	3	4	0	0	1.333	3.679	3.913
4.d	4	0	0	0	0.000	0.000	0.000
4.d	5	5	0	0	1.667	3.712	3.916
4.d	6	0	0	5	1.667	3.712	3.916



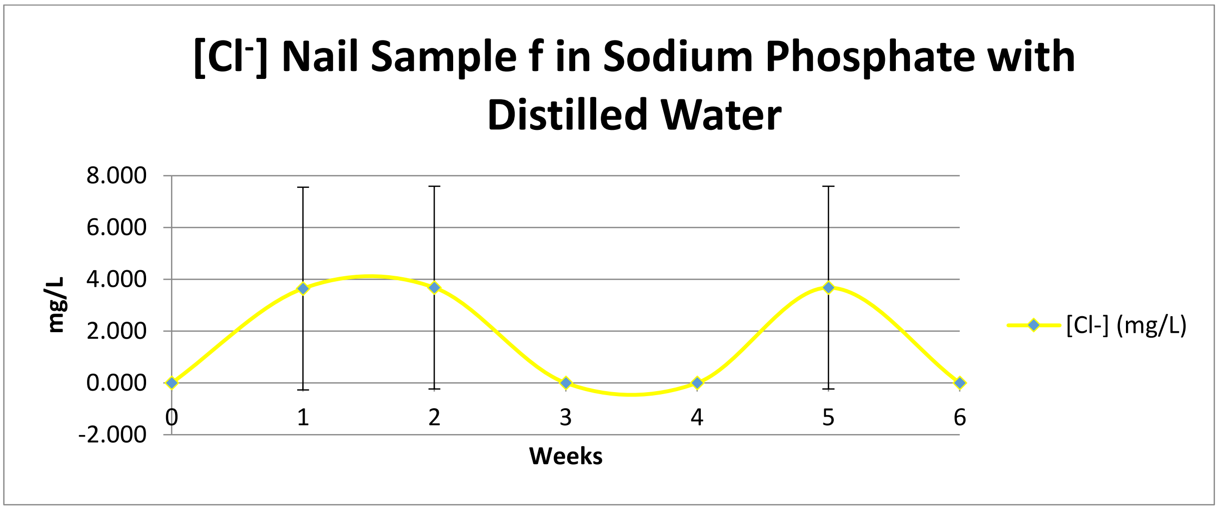
Sodium Phosphate in Distilled water, sample 4.e:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.e	0	0	0	0	0.000	0.000	0.000
4.e	1	8	4	6	6.000	4.145	3.960
4.e	2	7	5	6	6.000	4.145	3.960
4.e	3	9	6	6	7.000	4.245	3.970
4.e	4	0	6	0	2.000	3.745	3.920
4.e	5	3	0	0	1.000	3.645	3.910
4.e	6	0	0	4	1.333	3.679	3.913



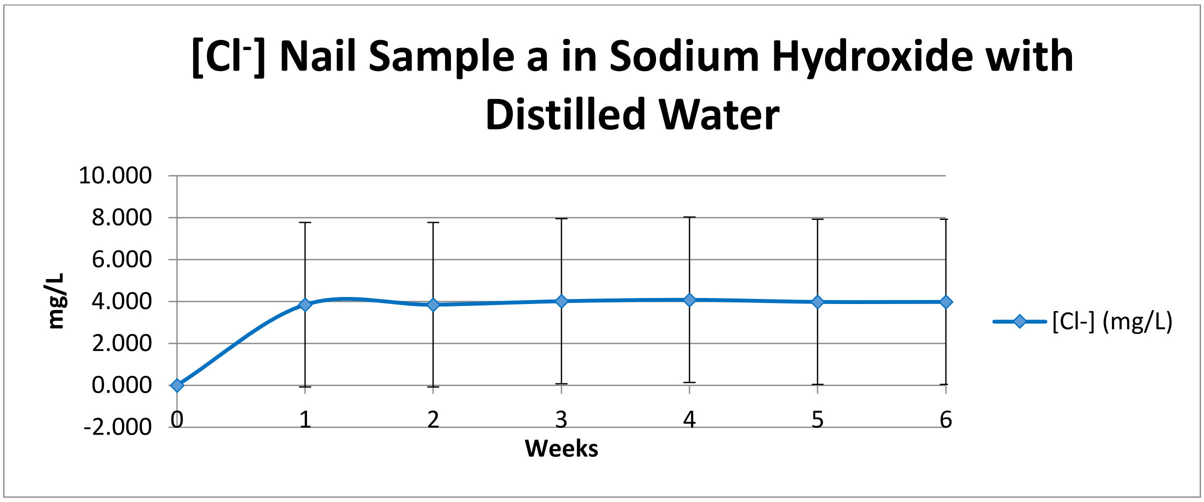
Sodium Phosphate in Distilled water, sample 4.f:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
4.f	0	0	0	0	0.000	0.000	0.000
4.f	1	0	3	0	1.000	3.645	3.910
4.f	2	0	2	2	1.333	3.679	3.913
4.f	3	0	0	0	0.000	0.000	0.000
4.f	4	0	0	0	0.000	0.000	0.000
4.f	5	4	0	0	1.333	3.679	3.913
4.f	6	0	0	0	0.000	0.000	0.000



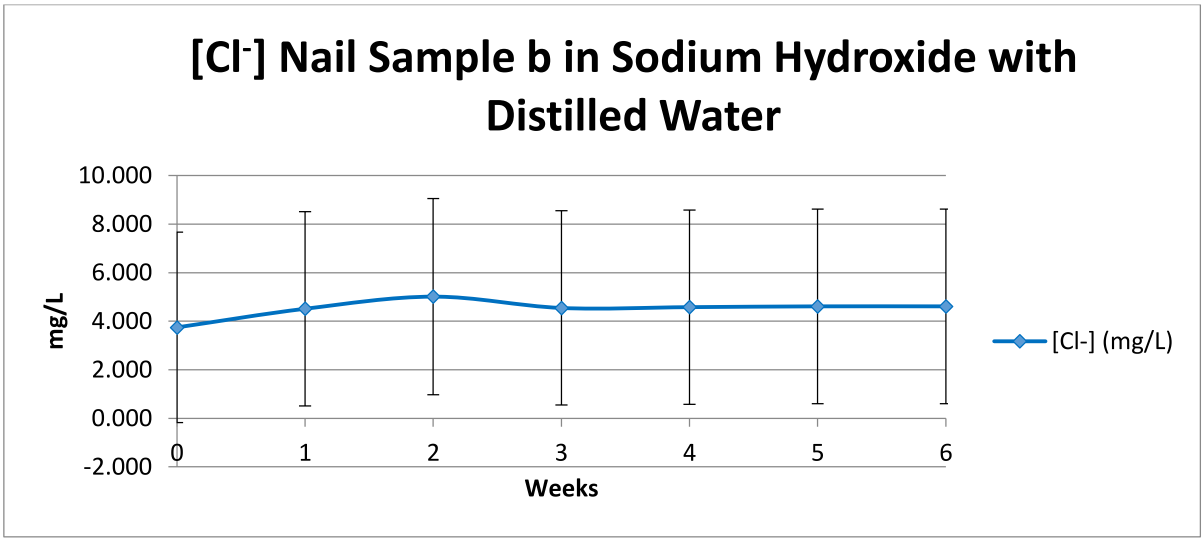
Sodium Hydroxide in Distilled water, sample 5.a:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
5.a	0	0	0	0	0.000	0.000	0.000
5.a	1	6	0	3	3.000	3.845	3.930
5.a	2	3	3	3	3.000	3.845	3.930
5.a	3	7	0	7	4.667	4.012	3.946
5.a	4	9	0	7	5.333	4.079	3.953
5.a	5	7	0	6	4.333	3.979	3.943
5.a	6	6	0	7	4.333	3.979	3.943



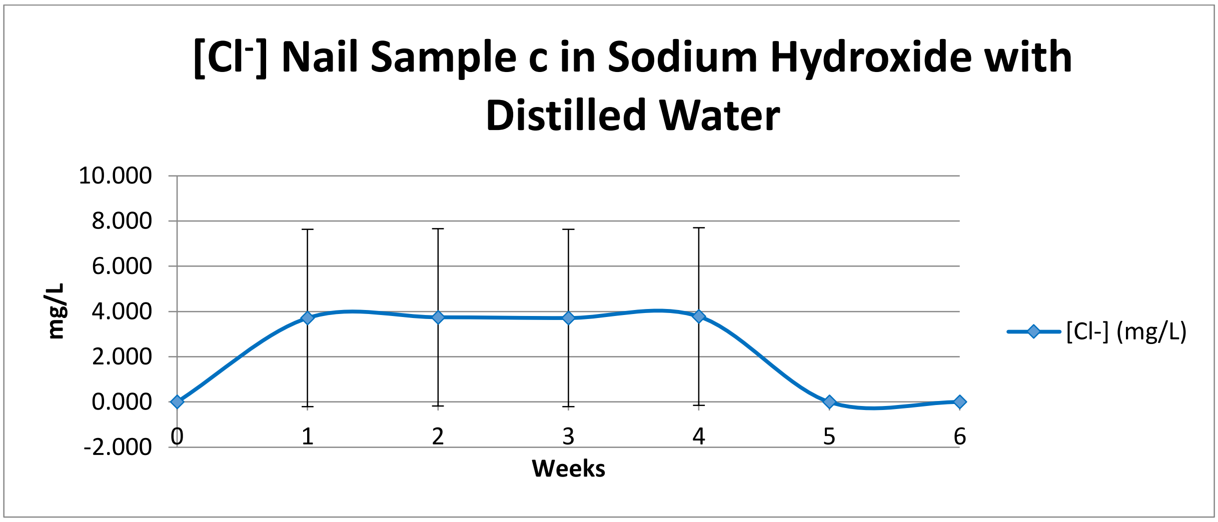
Sodium Hydroxide in Distilled water, sample 5.b:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
5.b	0	6	0	0	2.000	3.745	3.920
5.b	1	11	9	9	9.667	4.512	3.996
5.b	2	24	9	11	14.667	5.012	4.046
5.b	3	10	9	11	10.000	4.545	4.000
5.b	4	11	10	10	10.333	4.579	4.003
5.b	5	11	10	11	10.667	4.612	4.006
5.b	6	11	10	11	10.667	4.612	4.006



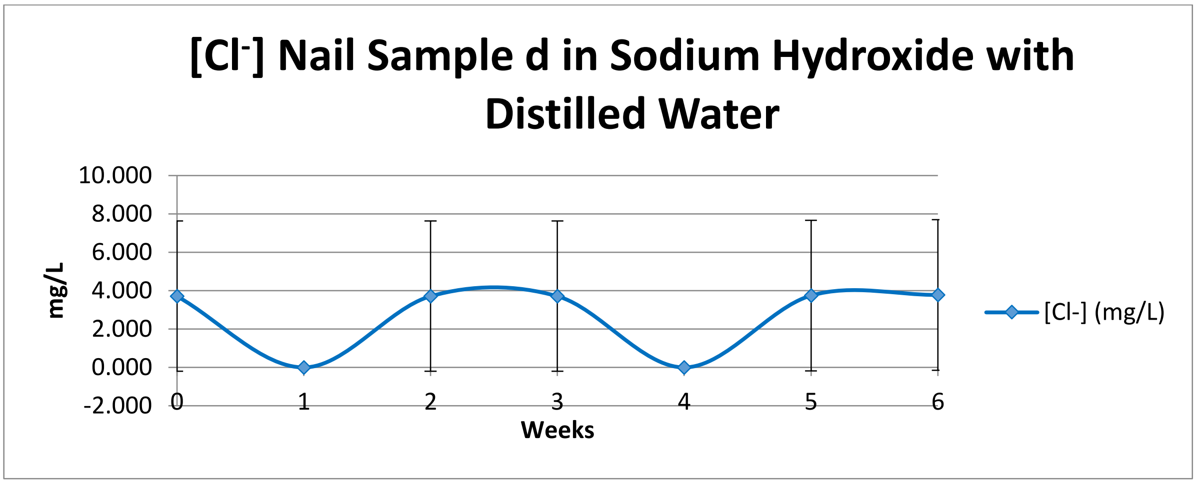
Sodium Hydroxide in Distilled water, sample 5.c:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
5.c	0	0	0	0	0.000	0.000	0.000
5.c	1	0	5	0	1.667	3.712	3.916
5.c	2	2	2	2	2.000	3.745	3.920
5.c	3	0	0	5	1.667	3.712	3.916
5.c	4	0	0	7	2.333	3.779	3.923
5.c	5	0	0	0	0.000	0.000	0.000
5.c	6	0	0	0	0.000	0.000	0.000



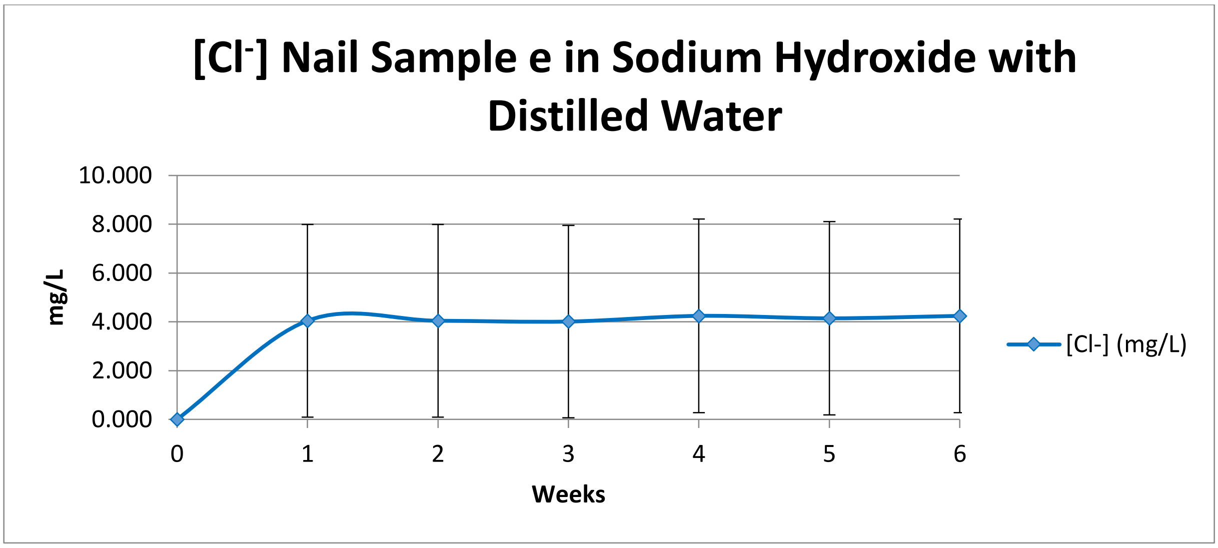
Sodium Hydroxide in Distilled water, sample 5.d:

Sample	Week	[Cl⁻] meas 1	[Cl⁻] meas 2	[Cl⁻] meas 3	avg meas	[Cl⁻] (mg/L)	std dev in (mg/L)
5.d	0	5	0	0	1.667	3.712	3.916
5.d	1	0	0	0	0.000	0.000	0.000
5.d	2	0	5	0	1.667	3.712	3.916
5.d	3	0	0	5	1.667	3.712	3.916
5.d	4	0	0	0	0.000	0.000	0.000
5.d	5	6	0	0	2.000	3.745	3.920
5.d	6	7	0	0	2.333	3.779	3.923



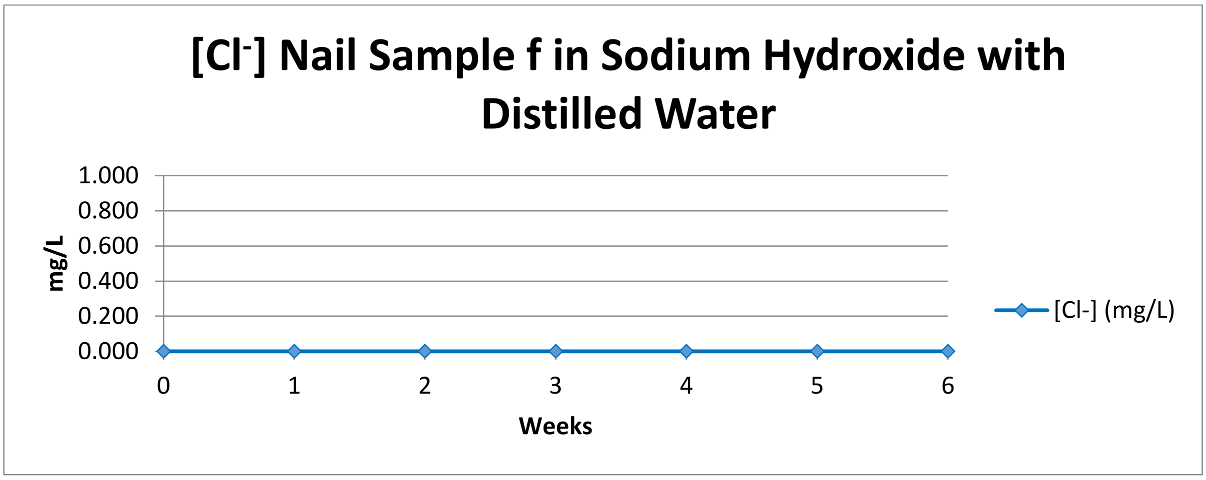
Sodium Hydroxide in Distilled water, sample 5.e:

Sample	Week	$[Cl^-]$ meas 1	$[Cl^-]$ meas 2	$[Cl^-]$ meas 3	avg meas	$[Cl^-]$ (mg/L)	std dev in (mg/L)
5.e	0	o	0	0	0.000	0.000	0.000
5.e	1	7	0	8	5.000	4.045	3.950
5.e	2	6	3	6	5.000	4.045	3.950
5.e	3	0	9	5	4.667	4.012	3.946
5.e	4	10	0	11	7.000	4.245	3.970
5.e	5	8	0	10	6.000	4.145	3.960
5.e	6	8	8	5	7.000	4.245	3.970



Sodium Hydroxide in Distilled water, sample 5.f:

Sample	Week	[Cl$^-$] meas 1	[Cl$^-$] meas 2	[Cl$^-$] meas 3	avg meas	[Cl$^-$] (mg/L)	std dev in (mg/L)
5.f	0	0	0	0	0.000	0.000	0.000
5.f	1	0	0	0	0.000	0.000	0.000
5.f	2	0	0	0	0.000	0.000	0.000
5.f	3	0	0	0	0.000	0.000	0.000
5.f	4	0	0	0	0.000	0.000	0.000
5.f	5	0	0	0	0.000	0.000	0.000
5.f	6	0	0	0	0.000	0.000	0.000

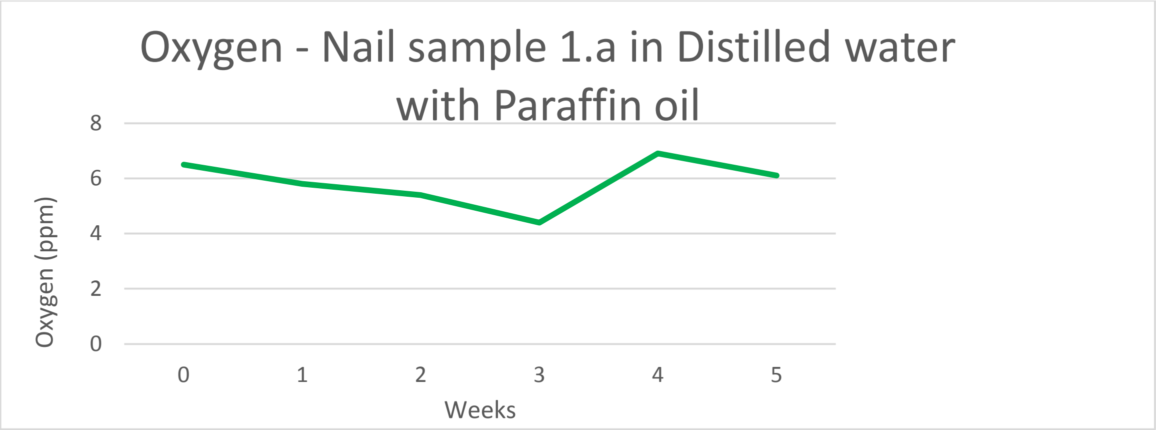


Appendix 3

Data for Dissolved oxygen content for each fragment in each solutions

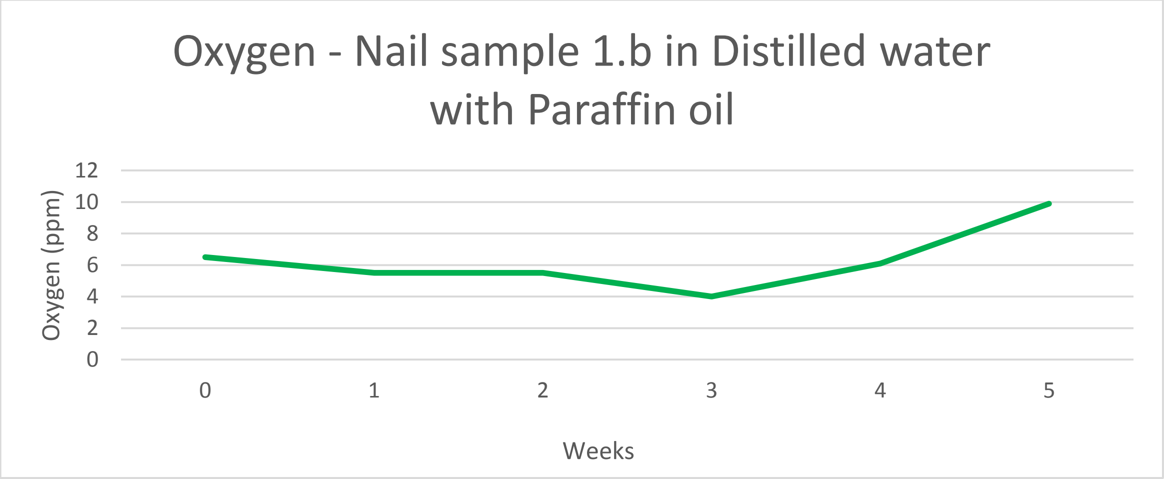
Distilled water with paraffin oil, sample 1.a:

Sampel	Week	oxygen (O_2) ppm	Date
1.a	0	6.50	2.3.2015
1.a	1	5.8	10.3.2015
1.a	2	5.4	17.3.2015
1.a	3	4.4	24.3.2015
1.a	4	6.9	31.3.2015
1.a	5	6.1	7.4.2015



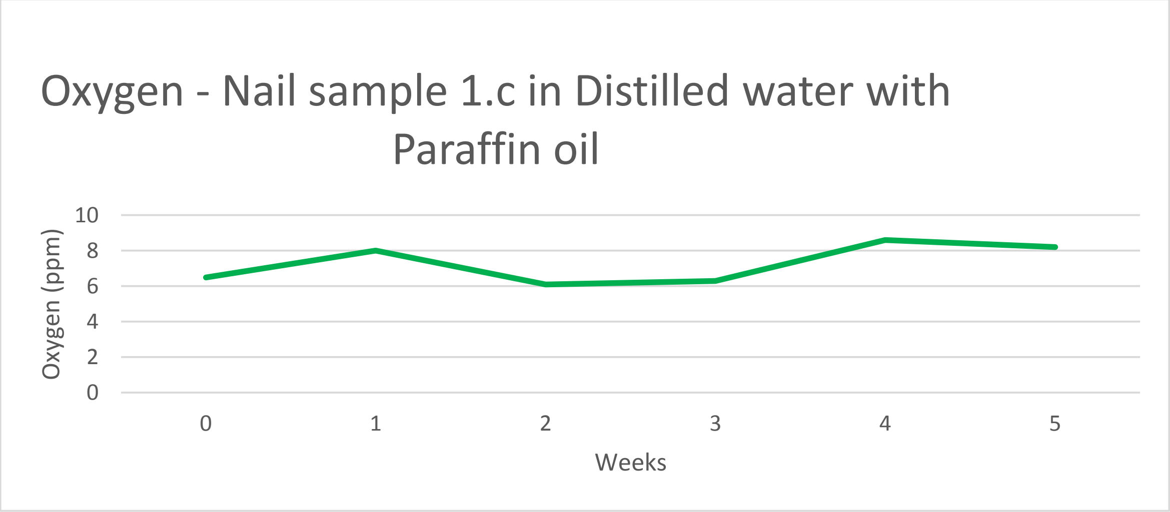
Distilled water with paraffin oil, sample 1.b:

Sampel	Week	oxygen (O_2) ppm	Date
1.b	0	6.5	2.3.2015
1.b	1	5.5	10.3.2015
1.b	2	5.5	17.3.2015
1.b	3	4	24.3.2015
1.b	4	6.1	31.3.2015
1.b	5	9.9	7.4.2015



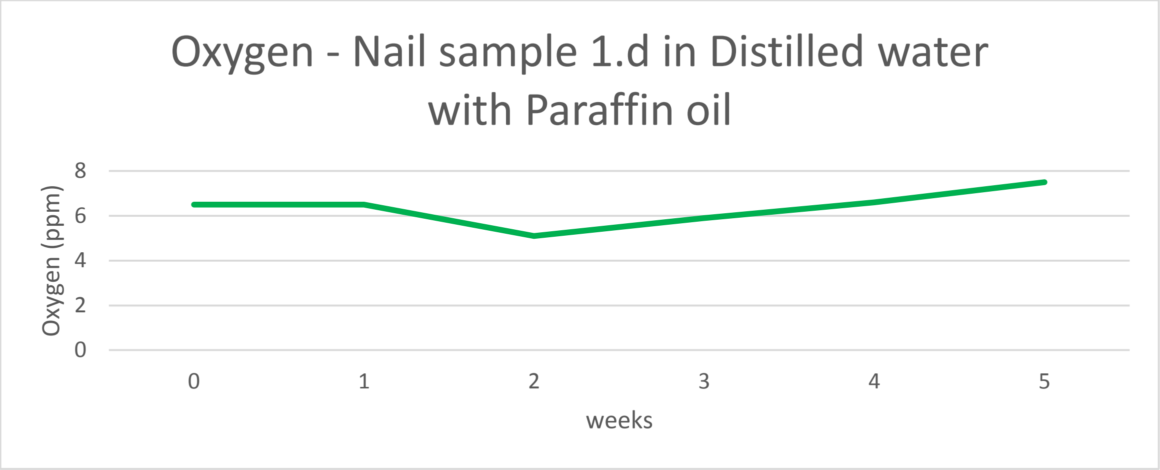
Distilled water with paraffin oil, sample 1.c:

Sampel	Week	oxygen (ppm(	Date
1.c	0	6.5	2.3.2015
1.c	1	8	10.3.2015
1.c	2	6.1	17.3.2015
1.c	3	6.3	24.3.2015
1.c	4	8.6	31.3.2015
1.c	5	8.2	7.4.2015



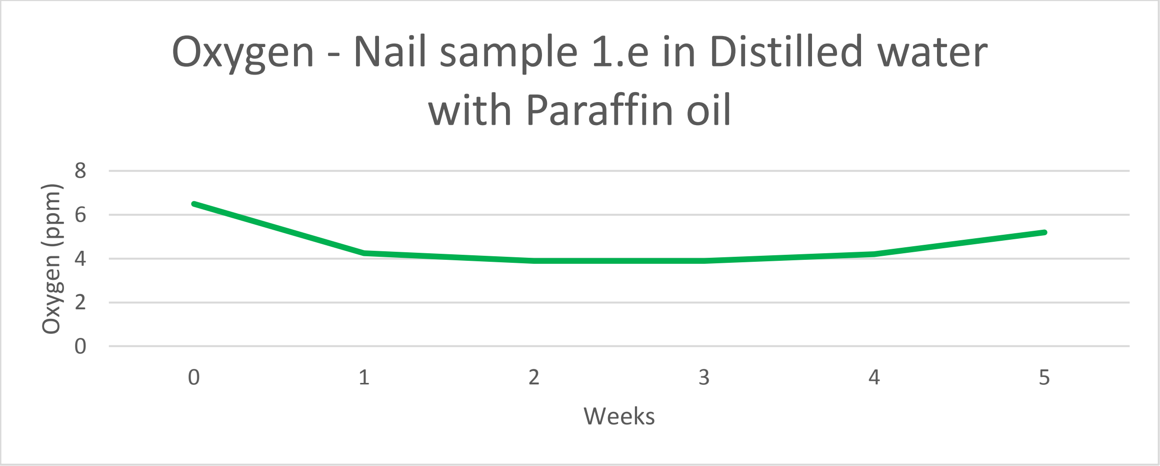
Distilled water with paraffin oil, sample 1.d:

Sampel	Week	oxygen (ppm)	Date
1.d	0	6.5	2.3.2015
1.d	1	6.5	10.3.2015
1.d	2	5.1	17.3.2015
1.d	3	5.9	24.3.2015
1.d	4	6.6	31.3.2015
1.d	5	7.5	7.4.2015



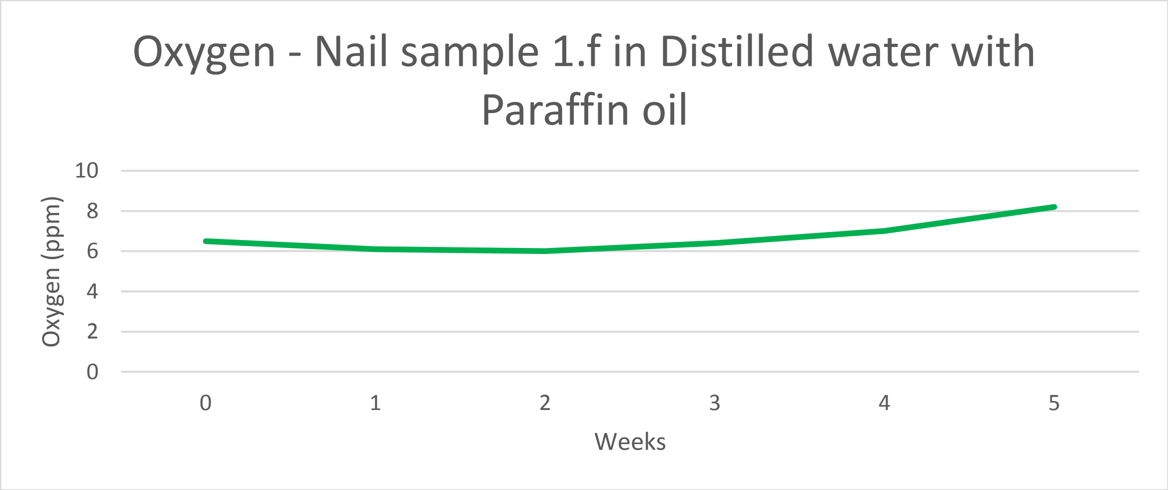
Distilled water with paraffin oil, sample 1.e:

Sampel	Week	oxygen (ppm)	Date
1.e	0	6.5	2.3.2015
1.e	1	4.25	10.3.2015
1.e	2	3.9	17.3.2015
1.e	3	3.9	24.3.2015
1.e	4	4.2	31.3.2015
1.e	5	5.2	7.4.2015



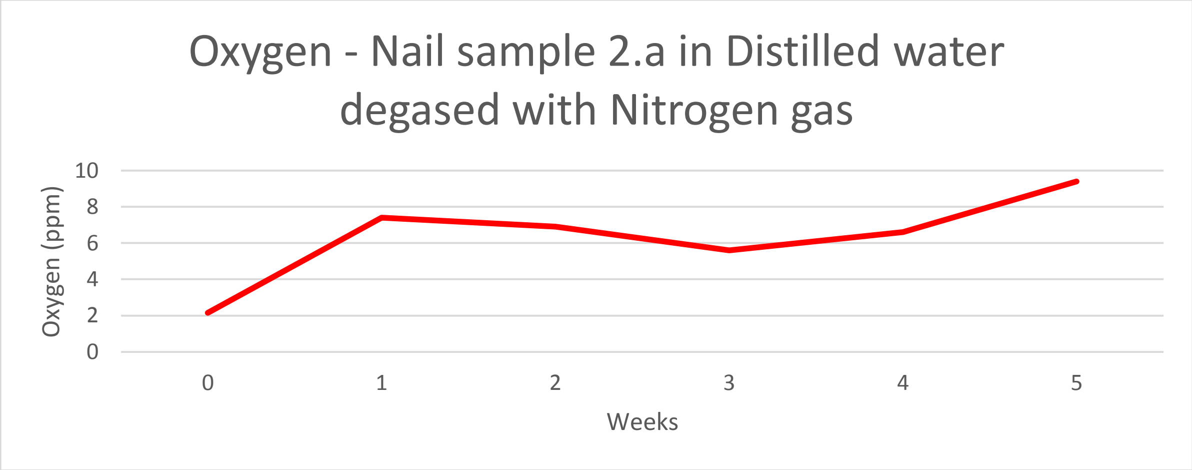
Distilled water with paraffin oil, sample 1.f:

Sampel	Week	oxygen (ppm)	Date
1.f	0	6.5	2.3.2015
1.f	1	6.1	10.3.2015
1.f	2	6	17.3.2015
1.f	3	6.4	24.3.2015
1.f	4	7	31.3.2015
1.f	5	8.2	7.4.2015



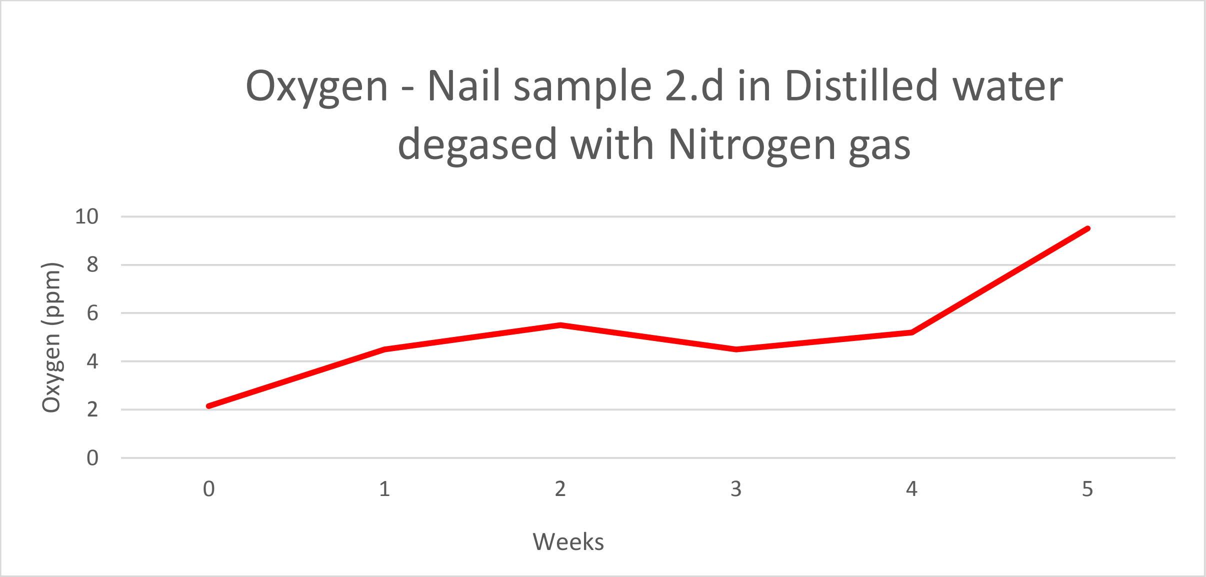
Distilled water degassed with N_2, sample 2.a:

Sampel	Week	oxygen (ppm)	Date
2.a	0	2.15	2.3.2015
2.a	1	7.4	10.3.2015
2.a	2	6.9	17.3.2015
2.a	3	5.6	24.3.2015
2.a	4	6.6	31.3.2015
2.a	5	9.4	7.4.2015



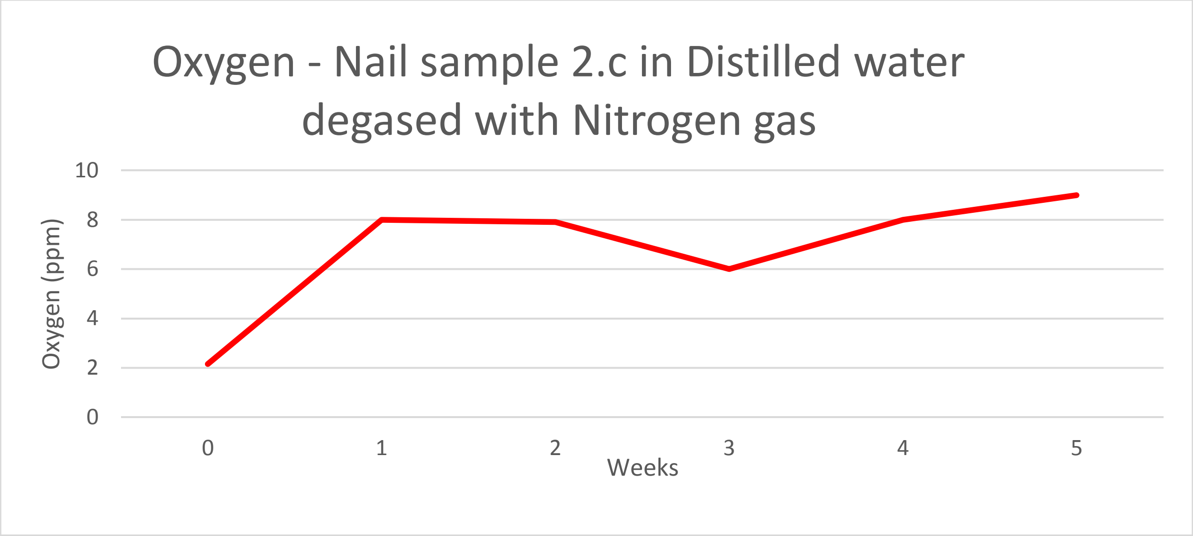
Distilled water degassed with N_2, sample 2.b:

Sampel	Week	oxygen (ppm)	Date
2.b	0	2.15	2.3.2015
2.b	1	4.5	10.3.2015
2.b	2	5.5	17.3.2015
2.b	3	4.5	24.3.2015
2.b	4	5.2	31.3.2015
2.b	5	9.5	7.4.2015



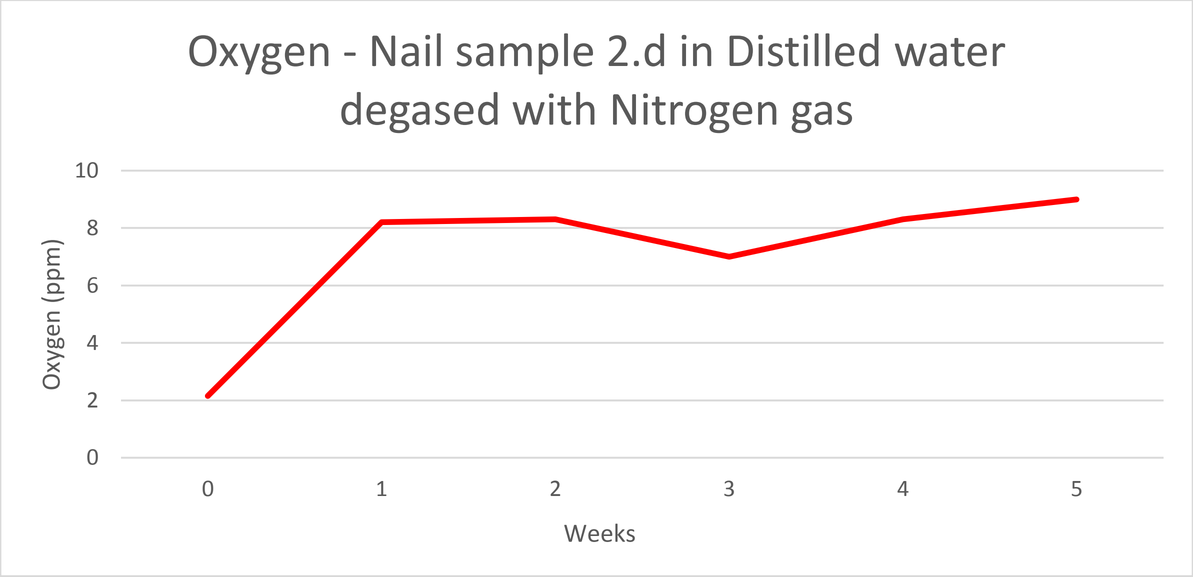
Distilled water degassed with N_2, sample 2.c:

Sampel	Week	oxygen (ppm)	Date
2.c	0	2.15	2.3.2015
2.c	1	8	10.3.2015
2.c	2	7.9	17.3.2015
2.c	3	6	24.3.2015
2.c	4	8	31.3.2015
2.c	5	9	7.4.2015



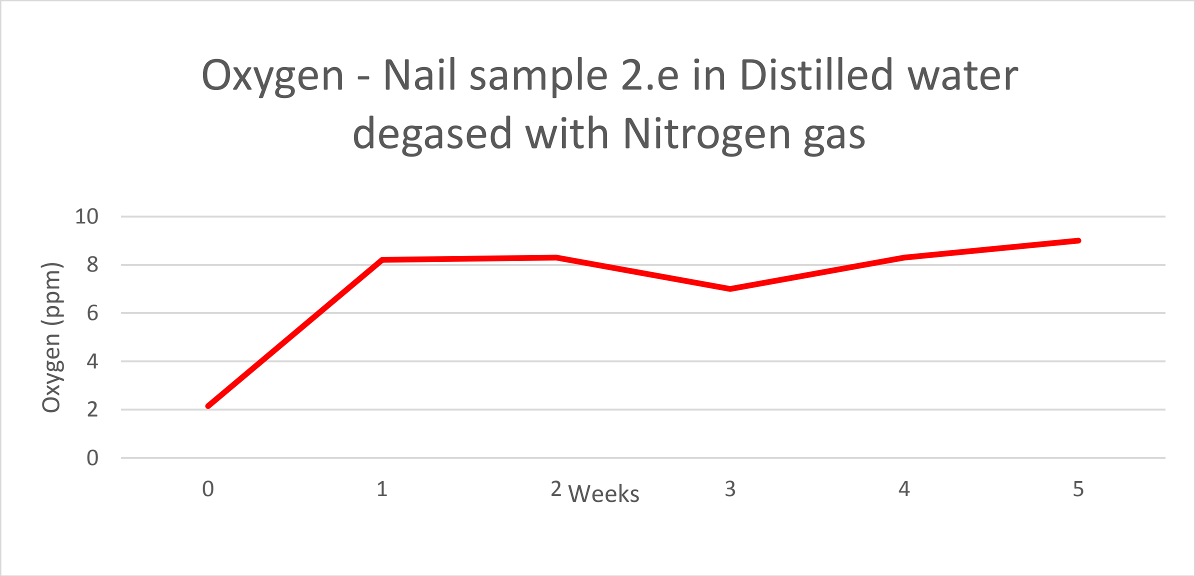
Distilled water degassed with N_2, sample 2.d:

Sampel	Week	oxygen (ppm)	Date
2.d	0	2.15	2.3.2015
2.d	1	8.2	10.3.2015
2.d	2	8.3	17.3.2015
2.d	3	7	24.3.2015
2.d	4	8.3	31.3.2015
2.d	5	9	7.4.2015



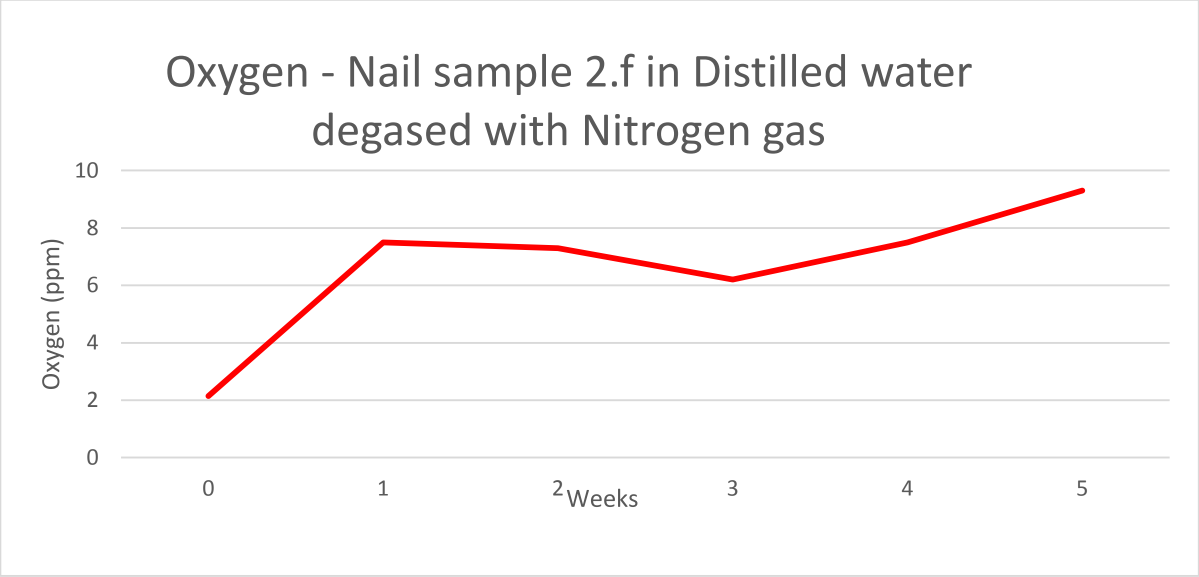
Distilled water degassed with N_2, sample 2.e:

Sampel	Week	oxygen (ppm)	Date
2.e	0	2.15	2.3.2015
2.e	1	8.2	10.3.2015
2.e	2	8.3	17.3.2015
2.e	3	7	24.3.2015
2.e	4	8.3	31.3.2015
2.e	5	9	7.4.2015



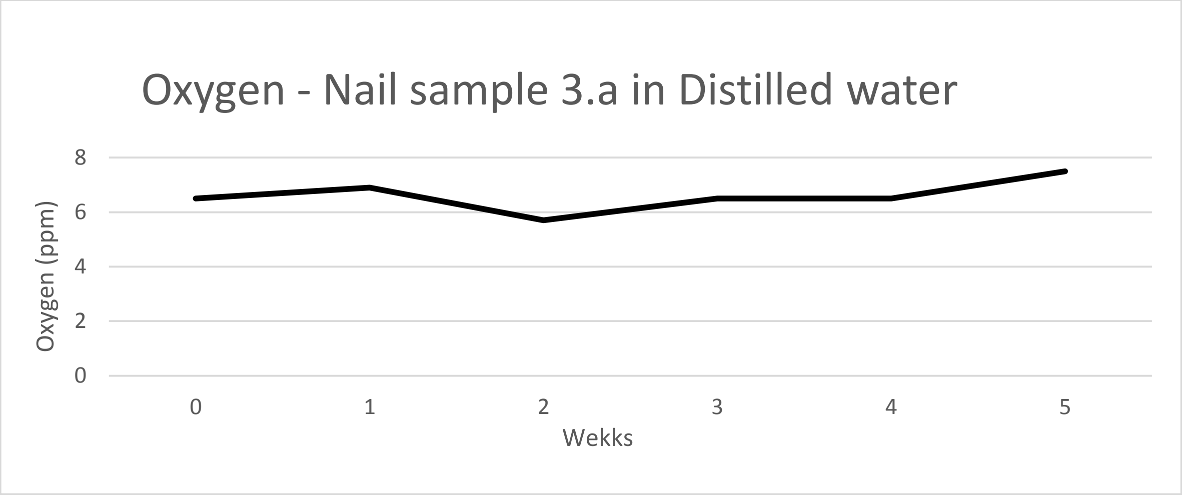
Distilled water degassed with N_2, sample 2.f:

Sampel	Week	oxygen (ppm)	Date
2.f	0	2.15	2.3.2015
2.f	1	7.5	10.3.2015
2.f	2	7.3	17.3.2015
2.f	3	6.2	24.3.2015
2.f	4	7.5	31.3.2015
2.f	5	9.3	7.4.2015



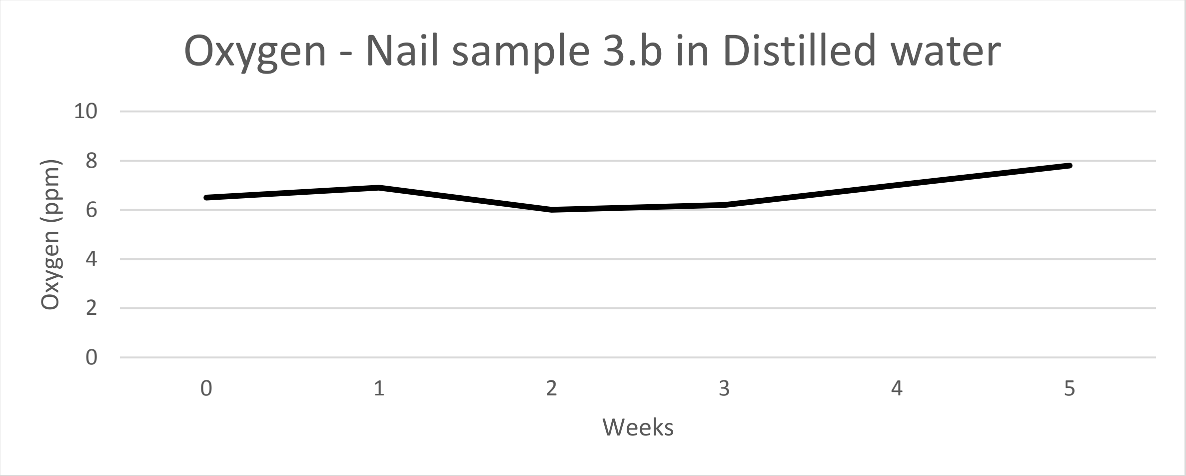
Distilled water, sample 3.a:

Sampel	Week	oxygen (ppm)	Date
3.a	0	6.50	2.3.2015
3.a	1	6.9	10.3.2015
3.a	2	5.7	17.3.2015
3.a	3	6.5	24.3.2015
3.a	4	6.5	31.3.2015
3.a	5	7.5	7.4.2015



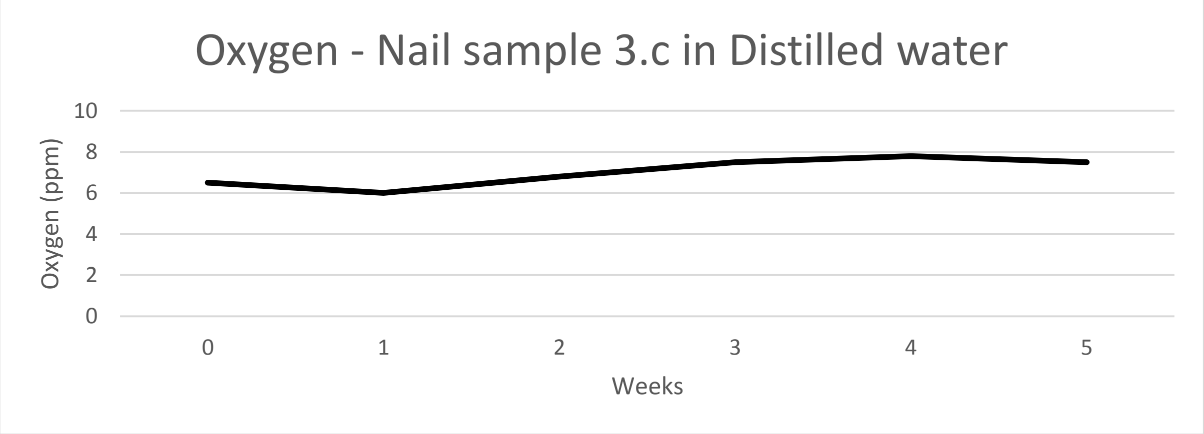
Distilled water, sample 3.b:

Sampel	Week	oxygen (ppm)	Date
3.b	0	6.50	2.3.2015
3.b	1	6.9	10.3.2015
3.b	2	6	17.3.2015
3.b	3	6.2	24.3.2015
3.b	4	7	31.3.2015
3.b	5	7.8	7.4.2015



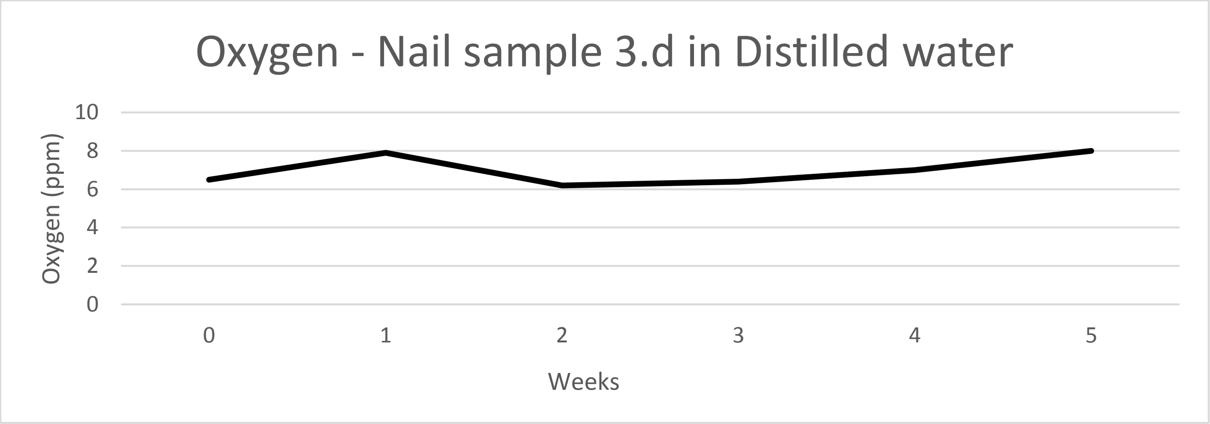
Distilled water, sample 3.c:

Sampel	Week	oxygen (ppm)	Date
3.c	0	6.50	2.3.2015
3.c	1	6	10.3.2015
3.c	2	6.8	17.3.2015
3.c	3	7.5	24.3.2015
3.c	4	7.8	31.3.2015
3.c	5	7.5	7.4.2015



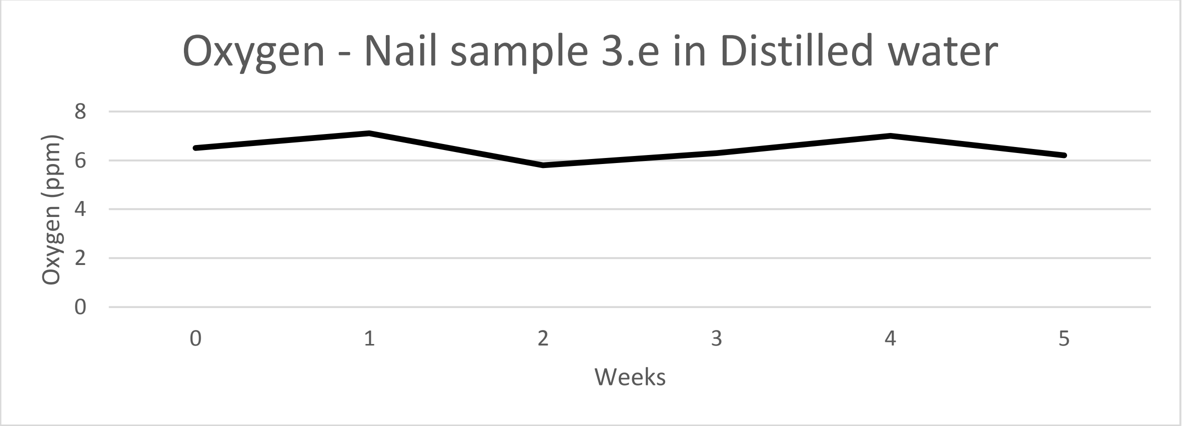
Distilled water, sample 3.d:

Sampel	Week	oxygen (ppm)	Date
3.d	0	6.50	2.3.2015
3.d	1	7.9	10.3.2015
3.d	2	6.2	17.3.2015
3.d	3	6.4	24.3.2015
3.d	4	7	31.3.2015
3.d	5	8	7.4.2015



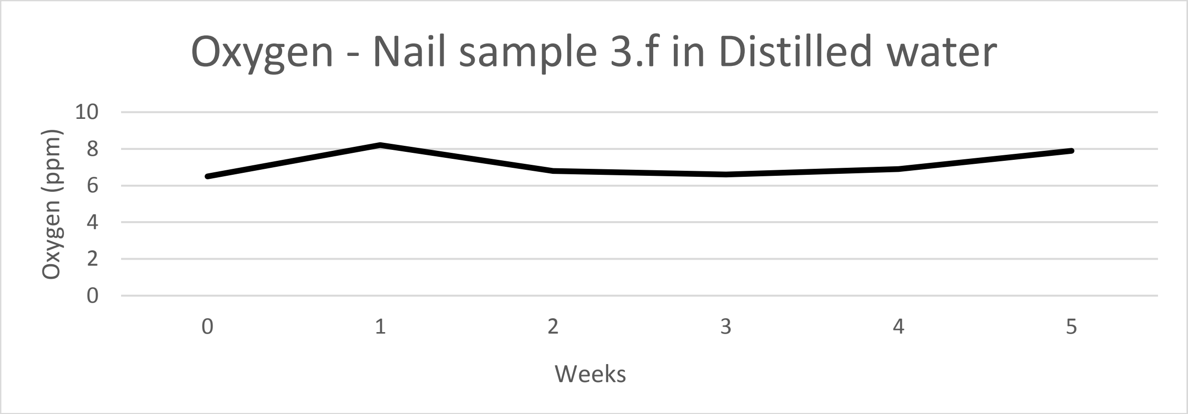
Distilled water, sample 3.e:

Sampel	Week	oxygen (ppm)	Date
3.e	0	6.50	2.3.2015
3.e	1	7.1	10.3.2015
3.e	2	5.8	17.3.2015
3.e	3	6.3	24.3.2015
3.e	4	7	31.3.2015
3.e	5	6.2	7.4.2015



Distilled water, sample 3.f:

Sampel	Week	oxygen (ppm)	Date
3.f	0	6.50	2.3.2015
3.f	1	8.2	10.3.2015
3.f	2	6.8	17.3.2015
3.f	3	6.6	24.3.2015
3.f	4	6.9	31.3.2015
3.f	5	7.9	7.4.2015



Appendix 4

Data for Soluble Iron in solution for each fragment in each solution. Since the measurement was only done on three samples randomly each week for the soluble iron in the solution there will be no graphs for this data. The gaps in the tables means that there were no measurement done for this sample that week.

Distilled water with paraffin oil, sample 1.a:

Sampel	Week	iron /ppm	Solution type	Date
1.a	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.a	2		Distilled Water + Parafinn oil	17.3.2015
1.a	3		Distilled Water + Parafinn oil	24.3.2015
1.a	4	3.44	Distilled Water + Parafinn oil	31.3.2015
1.a	5	5.00	Distilled Water + Parafinn oil	7.4.2015
1.a	6	3.77	Distilled Water + Parafinn oil	14.4.2015

Distilled water with paraffin oil, sample 1.b:

Sampel	Week	iron /ppm	Solution type	Date
1.b	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.b	2	4.20	Distilled Water + Parafinn oil	17.3.2015
1.b	3	4.20	Distilled Water + Parafinn oil	24.3.2015
1.b	4		Distilled Water + Parafinn oil	31.3.2015
1.b	5	5.00	Distilled Water + Parafinn oil	7.4.2015
1.b	6		Distilled Water + Parafinn oil	14.4.2015

Distilled water with paraffin oil, sample 1.c:

Sampel	Week	iron /ppm	Solution type	Date
1.c	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.c	2		Distilled Water + Parafinn oil	17.3.2015
1.c	3		Distilled Water + Parafinn oil	24.3.2015
1.c	4	5.00	Distilled Water + Parafinn oil	31.3.2015
1.c	5	5.00	Distilled Water + Parafinn oil	7.4.2015
1.c	6		Distilled Water + Parafinn oil	14.4.2015

Distilled water with paraffin oil, sample 1.d:

Sampel	Week	iron /ppm	Solution type	Date
1.d	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.d	2	2.67	Distilled Water + Parafinn oil	17.3.2015
1.d	3	2.67	Distilled Water + Parafinn oil	24.3.2015
1.d	4		Distilled Water + Parafinn oil	31.3.2015
1.d	5		Distilled Water + Parafinn oil	7.4.2015
1.d	6		Distilled Water + Parafinn oil	14.4.2015

Distilled water with paraffin oil, sample 1.e:

Sampel	Week	iron /ppm	Solution type	Date
1.e	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.e	2	5.00	Distilled Water + Parafinn oil	17.3.2015
1.e	3	5.00	Distilled Water + Parafinn oil	24.3.2015
1.e	4	5.00	Distilled Water + Parafinn oil	31.3.2015
1.e	5		Distilled Water + Parafinn oil	7.4.2015
1.e	6	5.00	Distilled Water + Parafinn oil	14.4.2015

Distilled water with paraffin oil, sample 1.f:

Sampel	Week	iron /ppm	Solution type	Date
1.f	0	0.00	Distilled Water + Parafinn oil	2.3.2015
1.f	2		Distilled Water + Parafinn oil	17.3.2015
1.f	3		Distilled Water + Parafinn oil	24.3.2015
1.f	4		Distilled Water + Parafinn oil	31.3.2015
1.f	5		Distilled Water + Parafinn oil	7.4.2015
1.f	6	5.00	Distilled Water + Parafinn oil	14.4.2015

Distilled water degassed with N_2, sample 2.a:

Sampel	Week	iron /ppm	Solution type	Date
2.a	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.a	2	2.20	Distilled Water + Nitrogen gas	17.3.2015
2.a	3	2.20	Distilled Water + Nitrogen gas	24.3.2015
2.a	4	2,34	Distilled Water + Nitrogen gas	31.3.2015
2.a	5		Distilled Water + Nitrogen gas	7.4.2015
2.a	6	5.00	Distilled Water + Nitrogen gas	14.4.2015

Distilled water degassed with N_2, sample 2.b:

Sampel	Week	iron /ppm	Solution type	Date
2.b	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.b	2	2.69	Distilled Water + Nitrogen gas	17.3.2015
2.b	3	2.69	Distilled Water + Nitrogen gas	24.3.2015
2.b	4		Distilled Water + Nitrogen gas	31.3.2015
2.b	5	3.22	Distilled Water + Nitrogen gas	7.4.2015
2.b	6		Distilled Water + Nitrogen gas	14.4.2015

Distilled water degassed with N_2, sample 2.c:

Sampel	Week	iron /ppm	Solution type	Date
2.c	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.c	2	2.79	Distilled Water + Nitrogen gas	17.3.2015
2.c	3	2.79	Distilled Water + Nitrogen gas	24.3.2015
2.c	4		Distilled Water + Nitrogen gas	31.3.2015
2.c	5		Distilled Water + Nitrogen gas	7.4.2015
2.c	6	2.23	Distilled Water + Nitrogen gas	14.4.2015

Distilled water degassed with N_2, sample 2.d:

Sampel	Week	iron /ppm	Solution type	Date
2.d	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.d	2		Distilled Water + Nitrogen gas	17.3.2015
2.d	3		Distilled Water + Nitrogen gas	24.3.2015
2.d	4	1.61	Distilled Water + Nitrogen gas	31.3.2015
2.d	5		Distilled Water + Nitrogen gas	7.4.2015
2.d	6		Distilled Water + Nitrogen gas	14.4.2015

Distilled water degassed with N_2, sample 2.e:

Sampel	Week	iron /ppm	Solution type	Date
2.e	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.e	2		Distilled Water + Nitrogen gas	17.3.2015
2.e	3		Distilled Water + Nitrogen gas	24.3.2015
2.e	4		Distilled Water + Nitrogen gas	31.3.2015
2.e	5	2.64	Distilled Water + Nitrogen gas	7.4.2015
2.e	6	1.60	Distilled Water + Nitrogen gas	14.4.2015

Distilled water degassed with N_2, sample 2.f:

Sampel	Week	iron /ppm	Solution type	Date
2.f	0	0.12	Distilled Water + Nitrogen gas	2.3.2015
2.f	2		Distilled Water + Nitrogen gas	17.3.2015
2.f	3		Distilled Water + Nitrogen gas	24.3.2015
2.f	4	1.52	Distilled Water + Nitrogen gas	31.3.2015
2.f	5	0.56	Distilled Water + Nitrogen gas	7.4.2015
2.f	6		Distilled Water + Nitrogen gas	14.4.2015

Distilled water, sample 3.a:

Sampel	Week	iron /ppm	Solution type	Date
3.a	0	0.00	Distilled Water	2.3.2015
3.a	2		Distilled Water	17.3.2015
3.a	3		Distilled Water	24.3.2015
3.a	4	3.24	Distilled Water	31.3.2015
3.a	5	2.29	Distilled Water	7.4.2015
3.a	6	3.18	Distilled Water	14.4.2015

Distilled water, sample 3.b:

Sampel	Week	iron /ppm	Solution type	Date
3.b	0	0.00	Distilled Water	2.3.2015
3.b	2		Distilled Water	17.3.2015
3.b	3		Distilled Water	24.3.2015
3.b	4		Distilled Water	31.3.2015
3.b	5		Distilled Water	7.4.2015
3.b	6	2.85	Distilled Water	14.4.2015

Distilled water, sample 3.c:

Sampel	Week	iron /ppm	Solution type	Date
3.c	0	0.00	Distilled Water	2.3.2015
3.c	2		Distilled Water	17.3.2015
3.c	3		Distilled Water	24.3.2015
3.c	4	3.02	Distilled Water	31.3.2015
3.c	5		Distilled Water	7.4.2015
3.c	6		Distilled Water	14.4.2015

Distilled water, sample 3.d:

Sampel	Week	iron /ppm	Solution type	Date
3.d	0	0.00	Distilled Water	2.3.2015
3.d	2	1.40	Distilled Water	17.3.2015
3.d	3	1.40	Distilled Water	24.3.2015
3.d	4		Distilled Water	31.3.2015
3.d	5	1.53	Distilled Water	7.4.2015
3.d	6	1.30	Distilled Water	14.4.2015

Distilled water, sample 3.e:

Sampel	Week	iron /ppm	Solution type	Date
3.e	0	0.00	Distilled Water	2.3.2015
3.e	2	2.17	Distilled Water	17.3.2015
3.e	3	2.17	Distilled Water	24.3.2015
3.e	4	1.76	Distilled Water	31.3.2015
3.e	5	3.29	Distilled Water	7.4.2015
3.e	6		Distilled Water	14.4.2015

Distilled water, sample 3.f:

Sampel	Week	iron /ppm	Solution type	Date
3.f	0	0.00	Distilled Water	2.3.2015
3.f	2	0.18	Distilled Water	17.3.2015
3.f	3	0.18	Distilled Water	24.3.2015
3.f	4		Distilled Water	31.3.2015
3.f	5		Distilled Water	7.4.2015
3.f	6		Distilled Water	14.4.2015