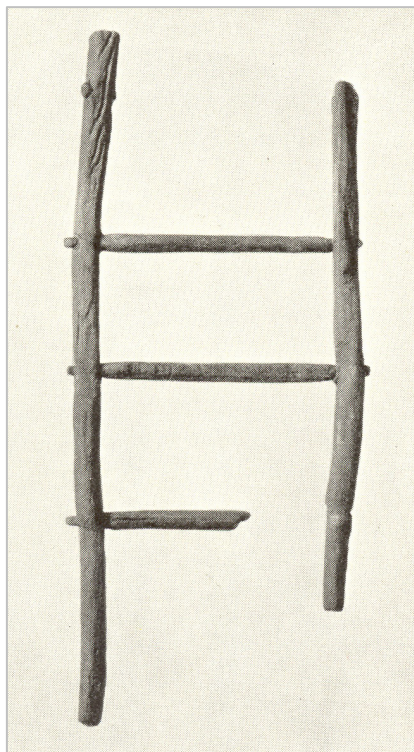


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Saving alum-treated archaeological wood

– report from a research project underway



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Saving alum-treated archaeological wood

– report from a research project underway

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Abstract

Alum, with glycerol and various surface coatings, was routinely used during the early 1900's as a conservation treatment for archaeological wood to prevent shrinkage and to impart strength. A history of the method and its associated problems is outlined. A survey assessing the condition of alum-treated wood according to the presence or degree of salt precipitation, surface flaking, cracking and pulverization was undertaken on the archaeological collection of the National Museum of Antiquities in Sweden. The majority of artefacts were found to exhibit on-going deterioration, loss of material, and a need for consolidation in the event of re-conservation. Alum-treated artefacts were generally found to be acidic with pH levels down to 1 and even 0. Of the artefacts surveyed, 5% were found to be destroyed or beyond re-conservation. A pilot study was initiated to test and evaluate potential re-conservation treatments. Artefacts were analyzed using SEM/EDS and FTIR for the presence of alum and other conservation materials such as glycerol and linseed oil. Desalination in warm and cold water using two different consolidants; Paraloid® B-72 and Parylene N, and two types of physical supports; polyester wadding with polyethylene netting and polyether foam with polyolefin film, was tested.

Introduction

For most of the former half of the 1900's in Sweden, the so called alum method was routinely applied to preserve waterlogged archaeological wood following excavation. Included in the alum-treated material from this time period are among others the wooden constructions and artefacts from the archaeologically significant sites of Bulverket, Käringsjön and Årby, the latter being material associated with one of few excavated Viking Age boats in Sweden to date.

In connection with the opening of a Viking Age exhibition at the Museum of National Antiquities in Stockholm in 2001, displaying among other things the Årby material, conservators of the National Heritage Board noticed signs of alarming deterioration on some of the objects. Although the problems associated with alum-treated wood had been well known, the artefacts themselves had never been thoroughly investigated. To build knowledge about the original treatment and the subsequent deterioration that alum evidently had caused, and to seek methods to prevent or control that deterioration, the research project; *Alum-treated archaeological wood - developing a rescue methodology for unique artefacts* was initiated as a joint project between the National Heritage Board and the Museum of National Antiquities in the autumn of 2002. The project is expected to run until the end of 2007 with a final report to be published in 2008.

The purpose of the project is to try to find solutions for the preservation of alum-treated archaeological wood in Sweden. Both active and preventive conservation measures will be investigated in order to develop recommendations for the treatment and preservation of these artefacts. By raising and sharing awareness and knowledge about alum-treated archaeological wood in Sweden, the project aims at preventing further loss of unique artefacts held in the national archaeological collections. Attention will in this way also be brought to wooden artefacts in general, an often overlooked and neglected part of the archaeological record.

This paper focuses on two of the project's major studies: a survey assessing the present condition of a collection of alum-treated wooden artefacts in view of possible re-conservation treatment, and a pilot study on the actual re-conservation treatment, testing and evaluating

different materials and techniques. Results of the former and preliminary results of the latter are presented. A review of the alum treatment and some of the historical background information concerning the conservation of archaeological wood is also presented.

Background

The nature of waterlogged archaeological wood and certain properties of alum

A waterlogged environment is one of few specific environments in which wooden artefacts might survive over time. In wet environments such as lake sediments, peat bogs and compact archaeological culture layers below the ground water table, supply of oxygen is limited. The microbiological activity from which wood will otherwise readily deteriorate is thus restricted. Under these conditions, the original shape and surface detail of waterlogged archaeological wooden artefacts may be preserved, seemingly unaltered.



Fig 1. The geologist Lennart von Post recovering house timbers during the excavations in 1923 of the prehistoric fortification, Bulverket, built on poles in the Tingstäde Träsk Lake on the island of Gotland, Sweden. The timbers were subsequently alum-conserved. (Copyright: Antiquarian Topographical Archive, National Heritage Board, Stockholm).

Wood degradation does however take place even in near anaerobic environments, and is in these cases most frequently caused by erosion bacteria.¹ Erosion bacteria mainly attack the cellulose rich part of the wood's cell walls, leaving behind a granular residue and a weakened lignin structure.² Artefact dimensions are intact only as long as water replaces lost wood substance and completely fills up the cell structure of the wood, i. e. as long as the wood is in

a waterlogged state. If dried, the wood suffers irreversible shrinkage with warping and cracking as a result, and subsequently irretrievable loss of archaeological information.³⁻⁴

In order to as far as possible retain the integrity of waterlogged wooden artefacts upon drying, the water must be replaced with another substance. When the alum method was developed, it was hoped and believed that alum had the properties required for such a substance.

Alum is a salt, a so called double salt referring to its two positive ions of potassium (K) and aluminum (Al). It has been used for centuries and was in ancient Greece and Rome used medically as an astringent (Latin *alumen*, bitter salt). It has also been used in later times for bating textiles and tanning leather.⁵⁻⁶ From 1861 and during the 90 following years, it was widely used for the conservation of excavated waterlogged archaeological wood in Scandinavia.

Its most common form is $KAl(SO_4)_2 \cdot 12H_2O$. It also exists without the 12 crystal waters: $KAl(SO_4)_2$. Some of their properties differ from one another. For example, whereas $KAl(SO_4)_2$ is a white hygroscopic powder, $KAl(SO_4)_2 \cdot 12H_2O$ consists of colourless crystals and decomposes slightly below 100 °C., i. e. the salt dissolves in its own crystal waters.⁷ This latter fact was important for its use in the conservation of waterlogged wood as it enables the use of very high concentrations in water solution. A saturated solution of alum is however fairly acidic with a pH of approximately 3. This fact and the potential problem it may pose to the integrity of the wood has not adequately been investigated.

The alum method - a retrospective

In the mid-19th century it became necessary to find a suitable conservation method for waterlogged wood in connection with the recovery of large quantities of archaeological wooden finds in bogs in Denmark. Without treatment, the degraded wooden objects tended to shrink heavily, crack and distort on drying. In 1861 the Danish archaeologist C.F. Herbst introduced the alum method. The original method involved immersing the cleaned wet objects in a hot supersaturated solution of alum where they were boiled for two hours, sometimes repeatedly for large objects. After the objects had been dried they were given a protective coating by saturation of the object's surface with boiled linseed oil. After a second drying a coating of clear thin varnish was applied.⁸⁻⁹ By substituting the water in the pores of the wood with alum, the method aimed at preventing shrinkage on drying and toward strengthening the material. The results were, at this time, found to be satisfactory. Objects treated in this way showed no change in shape or appearance according to Herbst.¹⁰

In order to improve the method so as to further prevent shrinkage while also retaining the fresh colour and shape of the wood, George Rosenberg, conservator at the National Museum in Denmark, modified the recipe in 1911 by including glycerol.¹¹ The pre-heated objects were placed in a solution of four parts by weight of alum, one part of glycerol and one of water, and were kept at a temperature of 92-94 °C for two to 30 hours depending on the size of the object. This was followed by one or several surface treatments, such as impregnation with melted beeswax, various types of oils, for example linseed oil, and coating with shellac or nitrocellulose varnishes.¹²⁻¹³ Furthermore, several and very liberal coatings of glycerol were at times applied to the surfaces of objects to counteract shrinkage and the formation of cracks on drying.¹⁴

During a long period, until the late 1950's, large quantities of waterlogged wood were routinely conserved with the alum method in Scandinavia and in the Baltic States. In Sweden the first evidence of the method is from brief notes on a so called boiling list dating from 1925.¹⁵ In the 1930's the method seems to have developed into a large-scale operation at the

Museum of National Antiquities in Stockholm. New laboratories were constructed and included two purpose-built alum-boilers, of which the longest was ten meters, built into the laboratory floor and heated by hot steam in copper pipes (see Fig 2).¹⁶ The magnitude of the operation is evident from an order of 700 kg of alum and 120 kg of glycerol in 1937, confirming that at this time Rosenberg's method with glycerol was favoured.¹⁷⁻¹⁹ However, the actual treatment records for the specific finds are rare and incomplete. There is essentially no information about the duration of the alum-boiling and scant information regarding surface treatments. From an article by the head of conservation at the time, Gillis Olsson, it is known that glycerol was in common use in combination with alum in Stockholm during the 1930's, however, the addition of glycerol is only mentioned in one case in the treatment records.²⁰

The alum method has been used in other countries, for example in England and the U.S., but to a lesser extent.²¹⁻²³



Fig 2. The alum-boiler at the Museum of National Antiquities in Stockholm around 1950. (Copyright: Antiquarian Topographical Archive, National Heritage Board, Stockholm).

Early problems with the alum method

The disadvantages of the method were soon found to be several. The Danish conservator Christensen reported in 1950 that the objects became extremely heavy, since they contained more alum than wood, but gained no strength. He describes the objects as brittle and unnaturally hard.²⁴ The shallow depth of penetration of alum, described by Christensen as only a few millimetres, stabilized the surface of the object only. Thus, the original shape was preserved, while the interior of the wood shrunk heavily during drying, thereby causing substantial internal cracking.²⁵

When wood treated with alum in turn is subjected to fluctuations in relative humidity (RH) the results can be exceedingly destructive. The loss and re-gain of crystallisation-water of alum will cause physical disruption of the weakened cell structure of the wood. At high RH, alum may dissolve and migrate in the wood, and when the RH falls again, it will re-

crystallize. The result may be salt efflorescence and breaking-up of the wood surface, and eventually total collapse and pulverization of the artefact. Given that it is highly hygroscopic, the addition of glycerol seems to aggravate this process.^{26-27.}

The surface treatments, initially intended to give protection to the artefacts, have also turned out to be permeable to humidity.^{28.} This means that instead of providing protection, the treatment creates a micro environment inside the artefact and additionally serves to effectively mask the true condition of the object underneath. Consequently, degradation processes invisible to the eye may continue inside of objects seemingly well preserved, until a total rupture of the hard shell of the surface occurs. This happened with the Danish early Iron Age boat from Hjortspring where the hard shell broke open in areas, exposing an interior of pulverized wood.^{29.}

Another interesting point to note is that alum-conserved objects which have been stored under similar environmental conditions have often shown varying degrees of degradation. The explanation is assumed to be a balance between different factors such as the species of wood, the degree of degradation of the excavated wood and the amount of alum absorbed in the wood.^{30.}

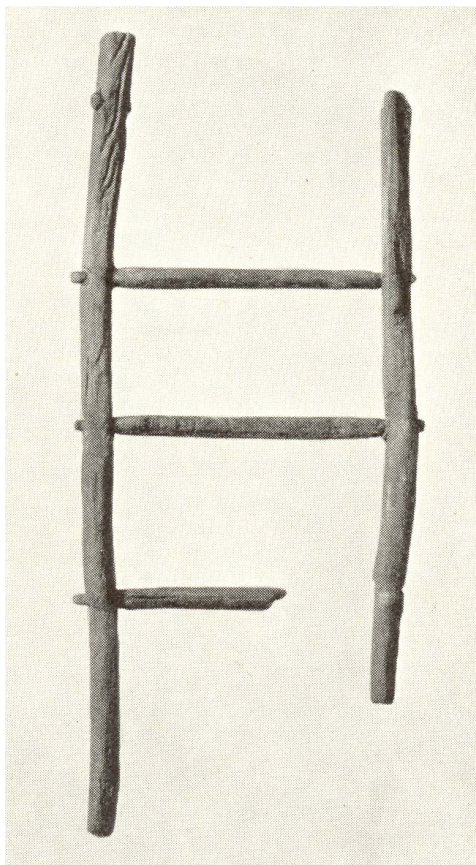


Plate 1. A wooden artefact from the Viking grave of Årby treated with alum and glycerol during the 1930's. The photo is taken shortly after the original alum-conservation. (Photo originally published by Arbman, 1940. Reproduced with the permission from Acta Archaeologica).



Plate 2. The same object severely degraded today. The hard shell of the surface has ruptured and the artefact has burst apart. (Copyright: National Heritage Board, Sweden).

Despite the drawbacks of the alum method, the lack of alternative satisfactory treatments meant that it remained in use until the end of the 1950's when new methods were developed and tried out.^{31.}

Previous research

Few investigations on alum-conserved wood deal with the actual deterioration, such as how the salt interacts physically and chemically with the wood and the effects stemming from the fact that alum is an acidic salt. Primarily the focus has been on the conservation of waterlogged archaeological wood with alum - its history and the general problems it causes.

A brief account of the existing research might begin with Gunhild Kopperud's studies of the distribution of the alum in the wood cell-structure. Her investigation suggests that the alum is either evenly distributed or concentrated into clusters, as in the case of hardwoods where alum clusters tend to be attracted to the large vessels within the wood.^{32.} Moreover, chemical analyses indicate that aluminum ions may be chemically bonded to hydroxyl groups in the cell walls of the wood.^{33.}

The physical properties of degraded alum-treated wood, such as the strength, have been investigated on Norwegian material. In anticipation of a possible move to a new museum of the alum-conserved finds from the Oseberg Viking ship, strength tests were carried out on the wood. Bending strength and impact bending value were measured and the wood was found to have lost 95 % or more of its strength as compared to fresh wood.^{34.}

The sensitivity of alum-treated wood to high and fluctuating RH is often discussed in the conservation literature; however, actual recommendations regarding storage conditions, such as RH and temperature, vary. When such recommendations are suggested, the theory or study upon which they are based is not always accounted for. Previous environmental recommendations for alum/glycerol-treated material include slowly adjusting to a dry and stable environment, starting at 30-45 % RH, at a temperature of 15-20 °C, and gradually lowering the RH until the moisture content of the wood reaches about 8-10 %.^{35.} Another suggestion has been to simply store the material at a stable RH of 30-40 %.^{36.}

Studies of crystallisation properties of alum in both alum- and alum/glycerol-treated wood in fluctuating and stable RH were carried out by Kopperud in 1992.^{37.} Among other things, it was found that growth of crystals happened over time, the concentration of crystals tended to increase and that the crystals were inclined to grow together on the surface of the wood samples. Even at a high and stable RH (100 %), growth of crystals occurred. In alum/glycerol-treated wood, splitting caused by alum crystallization was found to occur in the longitudinal vessels in the interior of hard-woods.^{38.} At low levels of RH (15 %), the flexibility and strength of the wood decreased, possibly making it more sensitive to external physical forces, such as alum crystallization. Kopperud concludes without giving any specific values that alum- and alum/glycerol-treated wood should be kept in a stable environment within a medium range of RH.^{39.}

Previous re-conservation attempts

Re-conservation of alum-treated archaeological wood has been attempted from time to time. The aim has then been to extract the alum and to replace it with an impregnant less susceptible to climate changes, and thus less destructive to the wood.

In 1964, attempts to re-conserve alum-treated wood were made at the Museum of National Antiquities in Stockholm. A collection of photographs was recently found at the museum

showing alum-treated objects before and after re-treatment. On the reverse of the photographs were very brief notes in pencil regarding the treatment procedure. According to the notes the alum in the objects was washed out; however, neither the medium for extraction (presumably water) nor the procedure is described. The objects were then dehydrated in acetone and impregnated with polyester resin followed by cold-curing.⁴⁰ In the condition survey these objects were examined, and the extensive salt depositions on the surface of these objects, together with SEM-EDS-analysis, demonstrate that the extraction of alum was unsuccessful. This is also demonstrated by the high initial conductivity values given by some of these objects in present salt extraction process. Furthermore, the condition survey showed the wood of these objects to be physically unstable with cracks and flaking surfaces in addition to a shiny plastic look.

In the 1970's, the Maritime Museum in Stockholm re-conserved a severely degraded and broken alum-treated pulley block of birch. The alum was extracted in water which was repeatedly changed for four months. The water was subsequently exchanged for acetone which was then in turn exchanged for white spirit. Finally the block was impregnated with 100 % paraffin wax and the pieces were joined together. The re-treatment was at the time considered successful.⁴¹

Extracting alum in water and replacing it with water soluble polyethylene glycol (PEG) is a method that has been used in Denmark and Latvia.⁴²⁻⁴⁴ The severely degraded and fragmented alum/glycerol-treated Hjortspring boat and its related objects were re-treated at the National Museum in Denmark starting in the 1960's and completed in the 1980's. Pulverized areas were first consolidated with soluble nylon. The pieces were then placed in crates with a supportive packing consisting of cotton wool and mineral wool wrapped in gauze. Previous surface treatments, lacquers and beeswax, were removed with solvents. The alum was extracted in water at 90 °C for several months. The water was continuously changed and the process of alum extraction monitored by measuring conductivity and the presence of sulphates in the extraction liquid. The wood was subsequently fully impregnated with PEG 4000 at 55-65 °C, with a gradual increase of the concentration to approximately 96 % over a period of about five months and then air-dried. Finally, the pieces of wood were taken out of their packages, excess PEG was removed, the fragments reassembled and mounted for display.⁴⁵⁻⁴⁶ Over the years, parts of the collection of alum-treated wood have been treated in a similar way at the National Museum in Denmark and at the National History Museum of Latvia.⁴⁷⁻⁴⁹

The National Museum in Denmark has also in recent years successfully used impregnation with PEG (2000 or 4000, up to 40 % in water) followed by vacuum freeze-drying for re-conservation of moderately deteriorated alum-treated wood. The alum has been extracted in water at 80 °C, and the process monitored with conductivity measurements and sulphate tests. The objects have been physically protected throughout the re-treatment by packing in polyether foam covered with a perforated heat-welded layer of polyolefin film. Surface treatments, for instance lacquers, have been found to loosen in the water and are then amenable to mechanical removal, at least to some extent, after the extraction and before PEG-impregnation. Finally, the excess water has been removed by vacuum freeze-drying.⁵⁰

At the Canadian Conservation Institute a para-xyllylene polymer, Parylene, has been applied to severely degraded alum-treated wood in order to consolidate its surface prior to salt extraction in water.⁵¹ The great advantage of Parylene is that it forms a very even, transparent and thin layer. It conforms to irregularities in the surface and thus the appearance of the object, such as its colour, remains all but unaltered.⁵²⁻⁵³

The condition survey

A condition survey was carried out in order to comprehend the extent and over-all condition of alum-treated wooden artefacts in the archaeological collection of the Museum of National Antiquities in Stockholm.

A classification model was developed, based on similar criteria as previous condition surveys on collections of alum-treated archaeological wood. The artefacts were condition assessed and, based on degree of degradation, categorized into different classes ranging from 1 to 5, representing a stable to a totally collapsed state.

Different sets of artefact data, such as pH and various signs of deterioration, were collected to be statistically evaluated and possible correlations between them investigated. Samples were taken from a number of artefacts to confirm presence of alum through SEM-EDS-analysis, and through FTIR-analysis to indicate presence of possible organic conservation substances, such as glycerol, linseed oil and beeswax. All artefacts were digitally photographed.

In connection with the condition survey work, test material for the re-conservation pilot study was chosen. A number of surveyed artefacts, representing different classes in need of active treatment, were selected.

Research material

In order to identify and locate whatever alum-treated archaeological wood was present in the collections of the Museum of National Antiquities, and if possible to specify its original treatments, the archive records on archaeological excavations and conservation treatments from the time period between 1900 and 1960 were gone through at the Antiquarian Topographical Archive at the National Heritage Board.

The earliest relevant documents are so called boiling lists, dating back to 1925. These show the weight changes of a number of wooden artefacts during drying and linseed oil impregnation following the treatment of alum-boiling. A coating of dilute crystal varnish is noted as the final treatment step.⁵⁴

The conservation files from 1931-1958 hold most of the relevant documents. During this time period, treatment with alum seems to have been the standard conservation method for waterlogged wood, sometimes done as a mass treatment with hundreds of artefacts treated at a time. Mentioned post-treatments are linseed oil impregnation, oil and turpentine coating, linseed oil saturation and wax impregnation. The notes are brief and the actual alum-treatment only described as alum-boiling. The last document mentioning alum-boiling as a treatment method dates to 1947, however other sources suggest that the alum method was still in use in Sweden up to 1950.⁵⁵⁻⁵⁶

Through further study of archive and museum records, most of the alum-treated artefacts found in the documents could be physically located to two of the Museum of National Antiquities' different storage facilities. All of these artefacts found were surveyed. In addition, all wooden artefacts excavated before 1966 present in the central storage facility were examined, and those showing salt precipitation were collected and included in the survey. This was also done in a third storage facility. According to current agreement, valid since January 1st 2001, the RH levels in the major storage facility should be controlled to 50 +/- 5 %; however measurements from July 2005 - July 2006 show RH and temperature variations between 31-54 % and 17-26 °C respectively.⁵⁷ RH and temperature measurements from the central storage facility during the same period varied between 43-50 % and 18-20

°C.⁵⁸ No information on the climate in the third storage facility or on the climate throughout the years is available.

In all, 1474 artefacts from 27 different archaeological sites were included in the survey, providing a good representative sample of artefacts from the Stone and Iron ages as well as the medieval period. Geographically the sites were spread from the southern to the very northern parts of the country.

Deterioration categorization and data collection

A classification model to categorize the state of deterioration of the artefacts was formulated (see Table 1). Condition criteria were based on similar concepts as in previously carried out condition surveys on collections of alum-treated archaeological wooden artefacts.

In a condition survey including 225 wooden artefacts in the archaeological collections of the Colonial Williamsburg Foundation, Virginia, USA, the concepts of physical integrity, cohesivity and surface interactions were used as criteria for condition assessment.⁵⁹ Physical integrity was used to refer to the physical structure of the wood. Signs of deterioration associated with physical integrity were identified as cracks and splits. Cohesivity was used to refer to the micro-structural strength of the wood, i. e. the ability of the wood to hold together. Deterioration signs associated with cohesivity were identified as flaking and splitting. Deterioration signs visible on the artefact surface, such as precipitation or distinct colour darkening, were classified as surface interactions.

Condition assessment of 74 alum-treated archaeological wooden artefacts was recently done at the Viking Ship Museum in Oslo, Norway.⁶⁰ As the condition assessment was done in view of a potential move of the artefacts, this survey had a different focus; however, it deals with the same type of material, and physical integrity and cohesivity served as criteria for condition evaluation here too. The presence of new cracks and the degree of pulverization were among the things examined for the categorization of artefacts into a five-grade condition scale.⁶¹ A similar condition assessment was carried out on another 78 artefacts from the same collection in 2005 by conservators from Denmark and Sweden, using the same evaluation system and five-grade condition scale.⁶²

The present survey focused similarly on condition assessment in view of potential action, in this case the possible need and potential for re-conservation treatment. The classification model (see Table 1), uses a five-grade scale, ranging from class 1, where no need for active measures is judged necessary, through classes 2, 3, and 4, where various degrees of consolidation or other physical support are suggested prior to re-conservation treatment, to class 5, representing artefacts that are totally collapsed and beyond possible conservation measures or rescue. Signs of deterioration are based on the concepts of physical integrity, cohesivity and surface interactions. Preventive measures include adjustment, stabilisation and control of climate, regular condition checks, and a continuous increase and improvement of knowledge. Possible active measures include removal of alum through salt extraction in water, PEG-impregnation, and vacuum freeze-drying.

class	state of deterioration	signs of deterioration	rescue measures
1	- stable - few signs of previous deterioration - no signs of active deterioration	- salt precipitation	- preventive measures
2	- signs of previous and active deterioration, such as surface interactions and occasional cracks (see Plate 3)	- salt precipitation - no or few (< 5) longitudinal and/or transversal cracks	- active and preventive measures
3	- signs of previous and active deterioration, such as surface interactions and cracking - loss of surface material during handling	- salt precipitation - several (≥ 5) longitudinal and/or transversal cracks - some (< 25 %) surface flaking or material loss	- active and preventive measures - surface consolidation and/or physical support prior to re-conservation treatment
4	- signs of previous and active deterioration, such as surface interactions and cracking - spontaneous loss of surface and/or bulk material	- salt precipitation - several (≥ 5) longitudinal and/or transversal cracks - extensive (≥ 25 %) surface flaking or material loss	- active and preventive measures - consolidation and/or physical support prior to re-conservation treatment
5	- total destruction (see Plate 4)	- all above - artefact collapse	- beyond rescue

Table 1. Classification model to categorize the state of deterioration of the artefacts.



Plate 3. Example of a class 2 artefact. The only visible sign of active deterioration is the salt precipitation on the surface; the arrows show where samples for chemical analysis have been taken. The dowel is from the Roman Iron Age (0-500 A.D.) site of Käringsjön in Halland. The specific treatment is unknown, but the presence of alum has been shown in SEM-analysis. (Copyright: National Heritage Board, Sweden).



Plate 4. Example of a class 5 artefact, its original dimensions irreversibly lost. This small ladder from the Viking Age (800-1050 A.D.) burial site of Årby in Uppland, was alum-boiled, followed by a glycerol impregnation and an oil application.^{see 56} (Copyright: National Heritage Board, Sweden).

For documentation and for purposes of statistic analysis, an Access database was created. The data chosen to be recorded were:

- archaeological and museum data, such as site and location information, and artefact and accession numbers
- storage facility location
- number of parts or fragments (fragments or parts of artefacts clearly fitting together, were counted as one artefact; fragments or parts of artefacts lacking clear fitting were counted as separate artefacts, this to as far as possible avoid subjective interpretation)
- signs of deterioration:
 - longitudinal cracks: none, few (< 5), or several (≥ 5)
 - transversal cracks: none, few (< 5), or several (≥ 5)
 - splitting: yes or no
 - surface flaking in percentage of surface area: none, some (< 25 %), or extensive (≥ 25 %)
 - material pulverization: yes or no
 - salt precipitation: yes or no
 - class: 1 (stable) - 5 (totally collapsed), (see Table 1)
 - gluing: yes or no
 - general impression
- surface pH (indicator-strips, pH 0-14, Merck KGaA, 64271, Darmstadt, Germany; strips were moistened with water and pressed against the artefact surface)
- documented conservation treatment
- documented re-conservation treatment
- presence of alum and/or other compounds as shown in SEM-EDS-analysis
- indication of organic compounds present as shown in FTIR-analysis
- remarks

All artefacts were digitally photographed.

Possible correlations between the different sets of data were examined, and basic statistics calculated.

Results and discussion

A total of 1474 artefacts from the collections of the National Museum of Antiquities were identified for the condition survey on the basis of available treatment documentation and appearance as possibly having been treated with alum. Samples were taken from selected and questionable artefacts for later analysis.

The condition survey served as an introduction to alum treated archaeological wood as a material group. The surveyed artefacts were found to be made of different types of wood with different pre- and post burial conditions, a variety of variously modified treatments including in some cases various re-conservation treatments, as well as different storage conditions. Given the lack of documentation and the scope of this survey, these factors were not analysed and are therefore expected to account for some degree of variance within the results. Although the sample group was found to be very heterogeneous, certain characteristic deterioration signs were evident and quantifiable. Similar to the results of other recent surveys on alum treated wooden artefacts, these were salt precipitation, surface flaking, internal cracks and pulverization (see Plates 5, 6, 7 and 8).^{63-65.}



Plate 5. Example of salt precipitation and cracking. Detail of oar handle from Jukkasjärvi in Lappland, dated to Scandinavian Bronze Age (1800-500 B.C.). The specific treatment is unknown, but the presence of alum has been shown in SEM-analysis. (Copyright: National Heritage Board, Stockholm).

Plate 6. Example of surface flaking. Surface detail of a plank from Bulverket on Gotland, dated to Scandinavian Iron Age (500 B.C.-1050 A.D.). The specific treatment is unknown, but the presence of alum has been shown in SEM-analysis, and archival material strongly suggests glycerol has been added in the treatment.^{see 17-18.} (Copyright: National Heritage Board, Stockholm).

Plate 7. Cross-section of a trough in pieces, revealing internal cracks invisible on the surface. The trough is from the medieval site of Glimmingehus in Skåne, and treated with alum followed by an oil application.^{see 56.} (Copyright: National Heritage Board, Stockholm).

Plate 8. Example of pulverization. The sticks are from the Viking Age (800-1050 A.D.) burial site of Årby in Uppland. A treatment of alum-boiling followed by glycerol impregnation and oil application is known from archive records.^{see 56.} (Copyright: National Heritage Board, Stockholm).

Both internal cracks and pulverization may go undiscovered on visual examination as there may be no sign on the artefact's surface. If the artefact is in parts, internal cracks will naturally show on the cross-section (see Plate 7), as will pulverization in any crack or split. However, if the artefact is in one piece, X-raying may be needed to discover internal cracks. A more subtle way to detect these deterioration characteristics is by the hollow sound these artefacts typically give when tapped. Given the deceptive nature of this deterioration this means that in some cases, and for the purposes of this survey, artefacts may in fact be in a more deteriorated state than what has been visually determined.

The majority of the surveyed artefacts were on the basis of the observed physical deterioration signs found to be of class 3 and 4 (refer to Table 2), indicating on-going deterioration and loss of material, and the need for consolidation in the event of re-conservation. 5 % of the artefacts were considered be either destroyed or in a state of deterioration beyond which re-conservation could be considered possible (class 5). Less than 1 % were found to be stable.

class	number of artefacts	%
1	9	0,6
2	241	16,4
3	567	38,5
4	579	39,3
5	78	5,3
n	1474	100,0

Table 2. Percentage of artefacts in each class where 1 represents a stable condition and 5 an artefact that is considered destroyed or beyond re-conservation.

There was only a weak relationship found between pH and class to indicate that acidity and visible deterioration are related. Many of the artefacts were nevertheless found to be very acidic with pH levels down to 1 and even 0 in a few cases (refer to Table 3). As many as 80 % of the artefacts were in fact found to have a pH level of 3 or below, which can be considered lower than normal regardless of wood species. All green wood is slightly acidic. Wood will also in time, to varying degrees, release acetyl groups in the form of acetic acid, which in turn decrease the pH below that of green wood.

pH	number of artefacts (%)	average class	salt efflorescence	pulverization	flaking	cracking	
						longitudinal	perpendicular
0	1	3,6	93,8	68,8	31,3	81,3	56,3
1	18	3,9	100	83,9	64,6	90,6	60,2
2	28	3,4	98,8	45,4	47,2	85,9	47,6
3	33	3,2	97,5	26,4	47,6	77,1	53,2
4	9	2,9	95	20	37,1	75,4	63,2
5	3	3	92,9	7,1	27,4	89,3	58,3
6	3	3,6	84,4	57,8	50	95,6	85,6
7	5	2	5,8	1,4	0,7	50	23,9
total artefacts	n=1474		93,2	41	46,6	81,5	53,6

Table 3. Average class and percentage of observed characteristics associated with deterioration in relation to measured pH level.

Acid hydrolysis of wood is known to occur at low pH levels and can thus, in addition to the mechanical stress caused by alum crystallization, be a contributing factor to wood degradation. The chemical processes by which these very low pH levels arise, as seen in the wood in this study, and the chemical nature of any resulting deterioration within the wood, have not been fully investigated. Aluminium potassium sulphate will hydrolyze in water to give low pH levels. In this regard it is interesting to note that artefacts with a slightly moist or sticky surface, and presumably higher moisture content, have also been found to be very acidic.⁶⁶ Dissolution of the alum salt with fluctuating humidity and the presence of the

hygroscopic ingredient glycerol in the conservation treatment would in turn be expected to exasperate the problem.

Similar results of very low pH on degraded artefacts were also found on alum-treated wood from Oseberg in Norway. In this case, with perhaps a more homogenous sample, artefacts in very good condition were found to have a pH of 3,5 or above, whereas artefacts in very poor condition were found to have a pH of 3,5 or below. A fairly wide range of pH values between 1,5 to 4,5 and 6 were in turn observed for artefacts classified as poor, and acceptable or good respectively.⁶⁷

Higher levels of pH (6-7) were noted on 8 % of the artefacts. These higher levels, in excess of what one would expect in fresh wood of the same species and for alum-treated wood, may possibly be explained by *in situ* leaching associated with the archaeological context and/or by the fact that surface coatings of wax or other resins may in these cases have masked the true pH of the wood below. Artefacts with a surface pH of 7 were found to be in relatively good condition (an average class of 2) with little salt efflorescence, a negligible occurrence of pulverization and flaking, and a comparatively low occurrence of cracking. This is contrast, however, to artefacts with a surface pH of 6 which are in a very poor condition, more akin to artefacts with a very acidic surface. These artefacts also have much more salt efflorescence, often with a pulverized and flaking structure, and they are notably most prone to cracking. The relatively high degree of degradation seen in these artefacts may be due to the presence of a coating, acting to create a more humid micro-climate favouring degradation inside the artefact, and at the same time, as an inflexible shell making the artefact also more prone to cracking.

Nearly all artefacts (93 %), with the exception of artefacts having a pH of 7, showed some degree of efflorescence on the surface or within cracks. Pulverization of the wood structure was highest among artefacts with a pH of 0 to 1, and generally found to increase in relation to acidity. A similar trend was found for flaking, and as well for cracking, both longitudinally and perpendicularly to the wood grain. As mentioned, artefacts with a pH of 6 were anomalous in that they had a much higher than expected level of occurrence of pulverization, flaking and cracking. Artefacts at a pH of 0 on the other hand showed a somewhat lower occurrence for the same characteristics than what might be expected for a truly linear relationship. The reason for the latter in particular is unclear, but some degree of error might be expected due to the small sample size for this group.

Further analysis with more homogenous sample groups should provide clearer results. The condition survey work has nonetheless greatly increased the knowledge about alum-treated archaeological wood in general, and more specifically about its deterioration characteristics.

The re-conservation pilot study stage 1: salt extraction

Re-conservation of alum-treated wood has in recent years not been attempted in Sweden. The purpose of the re-conservation pilot study is therefore to test a number of different re-conservation methods for alum-treated artefacts representing different degrees of degradation (artefacts categorized into class 2, 3 and 4 in the condition survey).

The reasons for re-conservation can be several. Removal of alum considerably reduces the sensitivity of the artefacts to environmental factors, such as the level of RH and its degree of fluctuation. Furthermore, it is desirable to make artefacts which have already undergone severe degradation due to alum less vulnerable, so that they can be handled, studied, displayed and used as museum objects without the risk of further damage. In addition there may be aesthetical reasons.

The re-conservation pilot study is on-going and divided into three stages. Stage 1 deals with the extraction of alum in water, and is described in this chapter. In stage 2 the re-treatment will continue by substituting another impregnate for the alum and finally, in stage 3, the wood will be dried. In stage 2 polyethylene glycol (PEG) 4000 will be used as an impregnant, followed by water removal using vacuum freeze-drying in stage 3. These are both methods widely and successfully used in the conservation of freshly excavated archaeological wood. As previously described, they have also been used for re-treatment of alum-treated wood in Denmark.

In stage 1, extraction of alum from wood in water is studied. Furthermore, extraction rates in water at room temperature are compared to those in heated water.

When the cohesive properties of the alum-treated wood are reduced, such as for artefacts of classes 2-4 in the condition survey, the wood will not permit soaking in water or re-impregnation and freeze-drying without risk of material loss. Therefore, four different physical supports and consolidants have been applied to the artefacts. Their permeability to water and alum, as well as in stage 2 to PEG 4000, is being tested and evaluated. Their ability to consolidate and support the degraded wood during both steps will be assessed at the end of the re-treatment.

Preliminary results of stage 1 will be reported here. Stages 2 and 3 remain and will be reported when completed.

Materials and equipment

A set of 29 archaeological artefacts from the collection of the Museum of National Antiquities were chosen as sample material of alum-conserved wood. The selection was done in conjunction with the condition survey when objects were condition assessed and classified. The artefacts for the study were primarily chosen on the basis of their numerical classification. The artefacts that were selected were all from classes 2, 3 and 4, since these all have signs of previous and active deterioration and are judged to need and to be able to withstand active measures i.e. re-conservation. Size was also a criterion for selection. Small to medium sized objects were chosen for practical reasons and to give a more uniform sample group for purposes of comparison. Consideration was not taken to known factors, such as object type, date, archaeological site or previous conservation treatment such as type of surface treatment.

A thorough documentation was carried out on each artefact and is presented in Table 4. The artefacts were weighed and digitally photographed with high resolution from two to four different angles depending on the shape of the object. Data on surface pH and class were collected from the condition survey data base. The samples taken from the artefacts in connection with the condition survey were chemically analyzed with a Scanning Electron Microscope, SEM, LEO-1455VP equipped with an Energy-Dispersive X-Ray Spectrometer, LINK/EDS unit, Inca-400, for micro X-ray analyses and with a Fourier Transform Infrared Spectroscope, FTIR, PerkinElmer "Spectrum One" to confirm the presence of alum and possibly other conservation materials, such as glycerol or linseed oil.

	Artefact ID No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29		
ARCHAEOLOGICAL FINDS DATA	Fid No.	262855	262856	118012	262857	262858	262859	262861	263053	263054	263055	263056	263057	263058	263059	263060	263061	263062	263063	263064	263065	263066	263067	263068	362395	272166	263071	263072	271780	272172		
	SHM no.			22028			23159	21107	22028	22028	22028											22289	22289	22028	23159	23159	23159	23159	23159	23159		
	Province	Go	Go	Sk	Go	Go	Ha	La	Sk	Sk	Sk	Go	Go	Go	Go	Go	Go	Go	Go	Go	Go	Up	Up	Sk	Ha	Ha	Ha	Ha	Ha	Ha		
	Parish	Tingstade	Tingstade	Valby	Tingstade	Tingstade	Övraby	Jukkasjärvi	Valby	Valby	Valby	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Tingstade	Lagga	Lagga	Valby	Övraby	Övraby	Övraby	Övraby	Övraby	Övraby	
	Site	Bulverket	Bulverket	Glimmingehus	Bulverket	Bulverket	Käringsjön	Valkijärvi	Glimmingehus	Glimmingehus	Glimmingehus	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Bulverket	Mora Ang	Mora Ang	Glimmingehus	Käringsjön	Käringsjön	Käringsjön	Käringsjön	Käringsjön	Käringsjön
Object	dowel	board	trough	stick	log	handle	our handle	dowel	plank																							
ORIGINAL ALUM CONSERVATION	Original alum conservation from written records			alum-boiling					alum-boiling and application of oil	alum-boiling and application of oil	alum-boiling											alum-boiling and impregnation with beeswax	alum-boiling and impregnation with beeswax	alum-boiling and impregnation with oil								
	Type of written source/ date			Konservingsmissiven /1938					Konservingsmissiven /1938	Konservingsmissiven /1938	Konservingsmissiven /1938											Konservingsmissiven /1940	Konservingsmissiven /1940	Konservingsmissiven /1938								
EARLY RE-CONSERVATION	Re-conservation																					alum washed out; de-hydrated in acetone and impregnated with polyester resin and cold-cured	alum washed out; de-hydrated in acetone and impregnated with polyester resin and cold-cured			alum washed out; de-hydrated in acetone and impregnated with polyester resin and cold-cured	alum washed out; de-hydrated in acetone and impregnated with polyester resin and cold-cured	alum not washed out; impregnated with polyester resin and cold-cured				
	Source/ date re-conservation																					N. Lagergren, Tekn avd SHM/ 1964	N. Lagergren, Tekn avd SHM/ 1964			FK o N. Lagergren, Tekn avd SHM/ 1964	FK o N. Lagergren, Tekn avd SHM/ 1964	FK, Tekn avd SHM/ 1964				
ANALYSIS OF CONSERVATION MATERIAL	Results SEM analysis	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum	alum
	Results FTIR analysis	alum + glycerol	alum + glycerol	alum	alum + glycerol	alum + glycerol	alum + wax emulsion	alum + wax emulsion	alum	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum + glycerol	alum		wax	alum	alum + wax	wax	alum + wax	wax	alum + wax	alum + wax	wax	
	Analyzed elements from the first extraction bath				glycerol + sulphate + butanediol + fragment of carbohydrates					glycerol + sulphate + fatty acids + fragments of carbohydrates													glycerol + sulphate + low molecular acid + dimethylphthalate + fragments of carbohydrates			glycerol + sulphate + dimethylphthalate + fragments of carbohydrates						
SURFACE pH	pH	3	4	1	2	2	1	3	3	3	3	1	2	1	2	2	2	2	2	2	1	3	3	2	2	3	2	2	2	2	2	
PHOTO X-RADIOGRAPHY	Digital before re-cons. / date	20050503	20050509	20050503	20050503	20050509	20050503	20050503	20050509	20050509	20050509	20050502	20050502	20050502	20050503	20050503	20050503	20050503	20050503	20050503	20050503	20050502	20050502	20050502	20050502	20050825	20050525	20050502	20050502	20050510	20050525	
	X-radiograph yes/no	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes	yes
	Plane of artefact on x-raydiag. transverse (TS)/ longitudinal (LG)	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG and TS	LG	LG and TS	LG and TS	LG and TS	LG and TS	LG and TS	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG	LG
	Maximum thickness of artefact (mm)	34	66	42	75	82	44	51	16	45	28	35	31	26	59	69	57	57	48	48	80	10	10	15	24	19	60	67	15	23		
	Hidden internal cracks/ voids (number)	no	no	19 (of which 4 also show on the surface)	several diffuse	no	1	6 (which also show on the surface)	no	no	no	no	several diffuse	no	2	no	no	no	no	no	no	2	5 (which also show on the surface)	no	no	part 1: 2 part 2: 3 part 3: 7	no	2	4	1 (which also shows on the surface)	no	
Additional information from x-radiography	lighter towards the ends, uneven alum-impregnation	lighter towards the ends, uneven alum-impregnation, external cracks clearly visible	lighter in the cross-cut end, i. e. higher conc. of alum, uneven alum-impregnation, two wooden plugs and adhesive joint and large hidden internal cracks long the grain clearly visible	lighter in parts, uneven alum-impregnation	lighter in parts, uneven alum-impregnation	of alum, uneven alum-impregnation, hidden internal cracks along the grain of the wood	lighter in the cross-cut end, i. e. higher conc. of alum, uneven alum-impregnation	lighter in parts, uneven alum-impregnation	lighter along the edges and around the hole, i. e. higher conc. of alum, uneven alum-impregnation	lighter along the edges, uneven alum-impregnation	clearly lighter along the edges, uneven alum-impregnation	clearly lighter along the edges, uneven alum-impregnation	lighter in the center, uneven alum-impregnation	lighter in parts, uneven alum-impregnation	darker in the center, uneven alum-impregnation	darker in the center, uneven alum-impregnation	in TS x-ray diffuse lighter areas towards the surface and darker in the center, uneven alum-impregnation	darker in the center, uneven alum-impregnation	lighter in parts, uneven alum-impregnation	lighter in parts, uneven alum-impregnation	clear cracks across the grain of the wood	clear cracks across the grain of the wood clearly visible	lighter in parts, uneven alum-impregnation, external cracks across the grain of the wood clearly visible	indication of cracking across the grain of the wood on smaller piece	lighter in parts, uneven alum-impregnation	lighter in parts, uneven alum-impregnation, cracks and an adhesive joint across the grain of the wood clearly visible	lighter in parts, uneven alum-impregnation	lighter in the cross-cut end of the wood, uneven alum-impregnation	clearly lighter in the cross-cut end of the wood, uneven alum-impregnation	lighter in the cross-cut end of the wood, uneven alum-impregnation, cracks across the grain of the wood and one hidden crack along the grain of the wood clearly visible	lighter towards the ends, uneven alum-impregnation	
DRY WEIGHT LOSS (%)	Supports/consolidants excluded	60	2240	3155	662	1827	453	627	22,5	1213,9	296,2	86,9	23,4	169,9				181	100	87,1	893,8	25,6	76	141,8	part 1: 126,5 part 2: 7,2 part 3: 38,8	90,5	544,4	878,7	33,3	30		
	Supports/consolidants included	67,3	2315	3334	702	1882	456,7	645	25,2	1274		91,8	27,9	184,1	141,2	236,8	192,6	187,5	102,6		920,6	29,8	77,6	151,6	101,5	570,8	915	37,7				

Table 4. Documentation data on the 29 artefacts included in the Re-conservation pilot study.

Finally all objects were X-rayed to reveal information, if possible, on depth of penetration of alum in the wood, internal condition of the artefacts and hidden features, such as joints. (The X-ray equipment: Philips Mobile Surgical X-ray Unit XG 4002, BV 21-S; film: Agfa Curix Ortho HT-G, Medical X-ray film, size 350 x 430 mm; exposures: 40 kV and between 1,2-20 seconds depending on the thickness of the artefact). Generally, with X-radiography it proved to be difficult to determine the depth of penetration and exact location of alum in the wood as X-radiographs are two-dimensional images of three-dimensional objects. However, it clearly showed that the alum was often in greater concentration in the cross-cut end of the wood where it would have had the easiest entrance into the wood structure. Furthermore, the X-radiography revealed details such as internal cracks and voids as well as invisible joints (see Plates 9 and 10).



Plate 9. Alum-treated wooden sticks from Kärringsjön. (Copyright: National Heritage Board, Sweden).

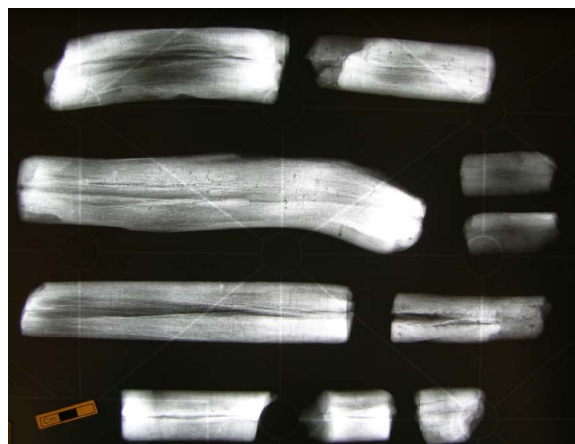


Plate 10. X-radiograph of the same artefacts showing alum in a greater concentration at the cross-cut ends of the wood (lighter areas) as well as hidden internal cracks along the grain of the wood. The white regular lines originate from the film cassette. (Copyright: National Heritage Board, Sweden).

Two types of physical supports were tested on artefacts of classes 2-4. These are intended to support the artefacts during the entire re-treatment and to be removed after the completion of the freeze-drying stage.

The first type consists of plain polyester wadding (Ohlssons Tyger & Stuvor AB, Sveav. 34, 11134, Stockholm, Sweden) covered by a tube-shaped polyethylene netting (R.W. Nissen AB, Box 124, 74623, Bålsta, Sweden). The artefacts were individually wrapped in the polyester and then covered by the polyethylene netting which was tied at the ends. This type of support made up loose packages (see Plate 11). The method is presently used with satisfaction for the support of freshly excavated archaeological material during conservation at the National Heritage Board in Sweden.

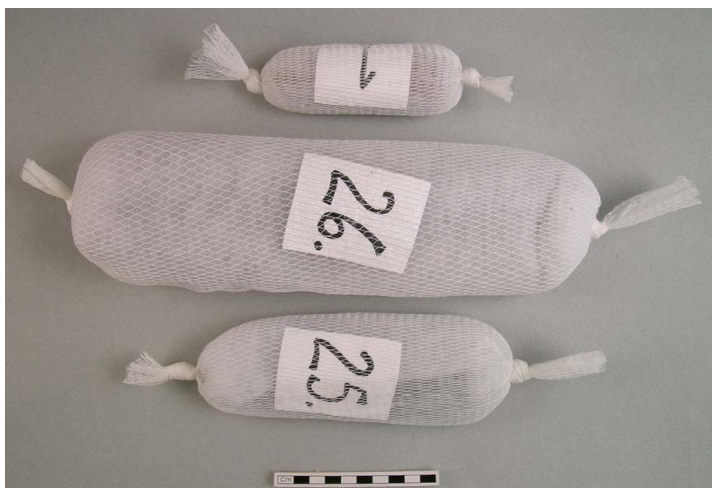


Plate 11. Artefacts supported by polyester wadding and polyethylene netting. (Copyright: National Heritage Board, Sweden).

The second type of physical support consists of polyether foam (thickness 5 mm, Specialplast Produktions AB, Gillinge, 18691, Vallentuna, Sweden) and perforated polyolefin film (Cryovac 570Y, with 28 holes per cm², diameter of holes 0,76 mm, Gemmer Emballage ApS, Bilstoftvej 1, 6800, Varde, Denmark). Each artefact was wrapped tightly in polyether foam and subsequently wrapped in the polyolefin film. The polyolefin film was heat-sealed with a soldering iron with a small flat tip (Weller® digital soldering station, WSD 81, working temperature set at 160 °C, Cooper Tools GmbH, Carl-Benz-Str. 2, 74345, Besigheim, Germany). The result was a tight support-package which followed the shape of the object closely. This method has successfully been used in the conservation of moderately deteriorated alum-treated wood at the National Museum in Denmark, and the application of the polyolefin film has been described by Lilja Jensen et al.⁶⁸⁻⁶⁹.



Plate 12. Making a supportive package of polyether foam and polyolefin film around an artefact. The polyolefin film is heat-sealed with a soldering iron. (Copyright: National Heritage Board, Sweden).



Plate 13. Artefacts supported by polyether foam and polyolefin film. (Copyright: National Heritage Board, Sweden).

Two types of consolidants were tested, Paraloid® B-72 and Parylene N. On artefacts of class 4, with salt precipitation, several (≥ 5) longitudinal and/or transversal cracks and extensive (≥ 25 %) surface flaking or material loss, surface consolidation was applied. The consolidants are intended to be kept on the surface after completion of the freeze-drying stage and thus their chemical long-term stability has been regarded as being of great significance. Reversibility has been considered of less importance since the artefacts are deteriorated to a degree that removal of the consolidant after PEG-impregnation and freeze-drying has been judged impossible without the risk of further damage. The aim has been to consolidate the surface of the artefact only in order to retain the surface morphology and shape of the object during the entire re-treatment.

Paraloid® B-72 is a co-polymer of ethyl methacrylate and methylacrylate with a glass transition temperature (T_g) of 40 °C.⁷⁰ It was chosen because it is well known and widely used by conservators and because it has a long-term chemical stability (classified as a Feller Class A material - a standard of stability).⁷¹ Furthermore, Paraloid® B-72 has been tested and proven to be successful as a consolidant during desalination of salt-laden ceramics by Paterakis. Her study showed that Paraloid® B-72 as a consolidant had little effect on the rate of desalination.⁷²

A 10 % (w/v) solution of Paraloid® B-72 (Preben Munch-Nielsen, Jernbane Allé 55, 3060, Espergaerde, Denmark) in ethanol:acetone (4:1) was applied to the artefacts. The artefacts were immersed in the solution for approximately five seconds which was judged to be the necessary time for the consolidant to cover the walls of the pores, the open cracks and the loose fragments within the surface-layer of the degraded wood. The depth of penetration of the consolidant was not measured. After consolidation the artefacts were air-dried.

Parylene is the generic name for members of a polymer series. Parylene N is poly-paraxylylene. Parylene C is modified by the substitution of one chlorine atom, Parylene D by the substitution of two chlorine atoms. All three of them are extremely stable polymers, e.g. insoluble in all organic solvents up to 150 °C; the substitution of chlorine enhances the stability and decreases the pore size. When it comes to reversibility Parylene C can be dissolved in chloronaphtalene at 175 °C and Parylene N at the boiling point of chloronaphtalene, 225 °C, i. e. they are in practice non-reversible.⁷³ Since the consolidant needs to be permeable to dissolved alum and PEG Parylene N, the type with the largest pore sizes, was chosen.

The application of Parylene involves special equipment including oven heating and a vacuum chamber. In short the process starts with a vaporization of the solid dimer at ca 150 °C. This dimer is then cleaved into very reactive monomers at ca 680 °C. The monomers are transported in gas phase to the vacuum chamber where the object is situated at room temperature. There they form a very thin polymer layer on the object. The thickness is controllable from 1 μm up.⁷⁴ Three different thicknesses, 1, 10 and 17 μm , were studied in order to evaluate the possible difference in permeability.

Experimental

As the first step of the re-treatment process, the destructive alum must be removed from the artefacts.

The artefacts were put through a salt extraction process in water purified by reversed osmosis (RO-water). In the salt extraction process, the permeability of the different supports and consolidants was demonstrated and, by dividing the selected artefacts into two groups - one to be desalinated in RO-water at room temperature (15 artefacts) and one to be desalinated in RO-water heated to 50 °C (13 artefacts) - the effect of water temperature on the

salt extraction rate investigated. The different classes and combinations of physical support or consolidation were all represented in both groups (see Table 5). Of the 29 artefacts selected, one was set aside to serve as a reference sample (artefact ID no. 19).

<i>artefact data</i>		<i>physical support/consolidant</i>					<i>salt extraction in RO-water</i>	
artefact identification number	class	none	polyester wadding and polyethylene netting	polyether foam and perforated polyolefin	surface consolidation: Paraloid® B-72 10% (w/v) in ethanol:acetone (4:1)	surface consolidation: Parylene N different thicknesses	room temperature	48 °C
10	2	x					x	
29	2	x						x
2	2		x				x	
1	2		x					x
23	2			x			x	
28	2			x				x
9	3		x				x	
25	3		x					x
24	3			x			x	
21	3			x			x	
3	3			x			x	
8	3			x				x
27	4		x				x	
4	4		x				x	
12	4		x					x
26	4		x					x
5	4			x			x	
17	4			x			x	
13	4			x			x	
11	4			x				x
7	4			x				x
20	4			x				x
18	4				x		x	
22	4				x		x	
6	4				x			x
16	4					2 x 0,5 µ	x	
14	4					10 µ		x
15	4					17 µ		x

Table 5. Artefact combinations of physical supports or consolidants and water temperatures during salt extraction.

The artefacts were put into individual salt extraction baths, using beakers and containers of glass, stainless steel and polypropylene. Beaker and container sizes were chosen so as to fit the artefact sizes as closely as possible. In order to keep the artefacts immersed and waterlogged they were covered with polypropylene lids, at the water surface weighed down by sand bags.

The salt extraction in heated water was similarly carried out in individual water-baths. The artefact beakers were placed in a larger water tank provided with a heat-controlling pump, Circulator HAAKE DC30/DL30 (Thermo Electron GmbH, D-76227 Karlsruhe, Germany), which circulated the heated water around all of the individual beakers containing the artefacts. The temperature of the water in the beakers was 1-2 °C below the temperature of the circulating water, which was set to 50 °C.

To monitor the salt extraction process, the conductivity of the extraction water was measured (see Graph 1). A 644 Conductometer (Metrohm Ltd., CH-9100, Switzerland) conductivity meter was used, and measurements were taken in solution samples before water-changes. These were at average done every second week. The pH in each bath was also measured in connection with the water-changes. The same kind of pH-strips as in the condition survey were used, i. e. indicator-strips, pH 0-14 (Merck KGaA, 64271, Darmstadt, Germany).

As a reference, the pH and conductivity of the RO-water from the tap were measured at each water-change. The pH was at all times 5, while the conductivity varied between 4,9-7,9 µS/cm, giving a mean value of 6,1 µS/cm.

In cases of salt extraction of ceramics, the figure ≤ 150 µS/cm has been given as an acceptable concentration of soluble salts in the final extraction bath, and used as an indication for the completion of the extraction process.⁷⁵ This is considered to be a lower limit for ceramics given that over-washing may weaken the structure as soluble compounds leach out.⁷⁶ In the present case, where there was no such risk, the level was set substantially lower. The artefacts were judged to be desalinated when conductivity measurements fell below 50 µS/cm and when the pH was 5 on three successive occasions.

In order to identify what was possibly being extracted other than alum, four samples of the residues from the evaporated first extraction bath were sent to STFI-Packforsk AB for analyses. The evaporated residues chosen were from the baths of artefacts with ID nos. 4, 9, 21 and 24. At STFI-Packforsk AB the residues were analyzed using pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS).

Preliminary results and discussion

Since the re-conservation pilot study is on-going and its first stage of salt extraction not yet finished, only preliminary results can be presented here. Firstly however, results from the various chemical analyses are presented (see also Table 4).

Given the lack of any written documentation regarding conservation with alum for many of the objects chosen for the re-conservation study, material samples from each artefact were analyzed in the SEM-EDS to confirm that these artefacts had been treated with alum. Presence of K, Al and S was used only as an indication of alum. K, Al and S were found in all wooden and/or salt samples for all of the 29 objects. For one object in the study, artefact ID no. 28, the only evidence to indicate that it has been conserved with alum is represented by the SEM-EDS results. This is due to the small amount of sample collected. Since there was not enough for both FTIR and SEM-EDS, the latter was given priority.

The FTIR analysis was used to confirm the presence of alum and other conservation and surface treatment materials, such as e.g. glycerol, linseed oil or beeswax. The presence of

alum was confirmed for most objects, 24 out of 29. Glycerol was found in 13 out of 14 objects from the archaeological site of Bulverket. Glycerol was also found in one case from the archaeological site of Glimmingehus, a collection of artefacts of which no prior knowledge of glycerol being used in the conservation treatment existed. This gives further evidence for the large-scale use of alum and glycerol first indicated by archival material.^{see 17-18.} Other materials found were wax and wax emulsion.

The Py-GC/MS analysis of residues from the evaporated extraction baths done at STFI-Packforsk AB found sulphates, most probably from alum, and glycerol in all four cases. The fact that Py-GC/MS is more sensitive to small amounts of materials, together with the fact that the samples used here were more concentrated, explain why glycerol was found in three cases where it was not detected by FTIR. Of note is that glycerol was found in objects from the archaeological sites of Mora Äng and Käringsjön, materials not previously known to have been treated with glycerol, again adding to the picture of a large-scale use of alum with the addition of glycerol. The fatty acids found in the sample from artefact ID no. 9 might come from e.g. linseed oil or from the wood. The fragments of carbohydrates found in all samples most likely come from degraded wood. Dimethylphthalate, which was found in samples from artefact ID nos. 21 and 24, is a well known substance often used as a plasticizer in polymeric materials. It most likely derives from the physical support material polyether foam used as a physical support material on these artefacts. If polyether foam is found to be satisfactory as a physical support in the re-conservation pilot study, consideration might in the future be taken to exchanging it for another more stable polymeric foam material with similar properties to polyether foam e.g. polyethylene foam. This would eliminate the risk of getting dimethylphthalate into the wood.

Regarding the consolidants, one result was obvious already two weeks into the salt extraction process; Paraloid® B-72 failed as a consolidant in the heated bath at a temperature of approximately 48 °C. The artefacts consolidated with Paraloid® B-72, artefact ID no. 6, started to fall apart in the second week, and after 44 weeks in the heated bath the artefacts were completely destroyed and beyond rescue.

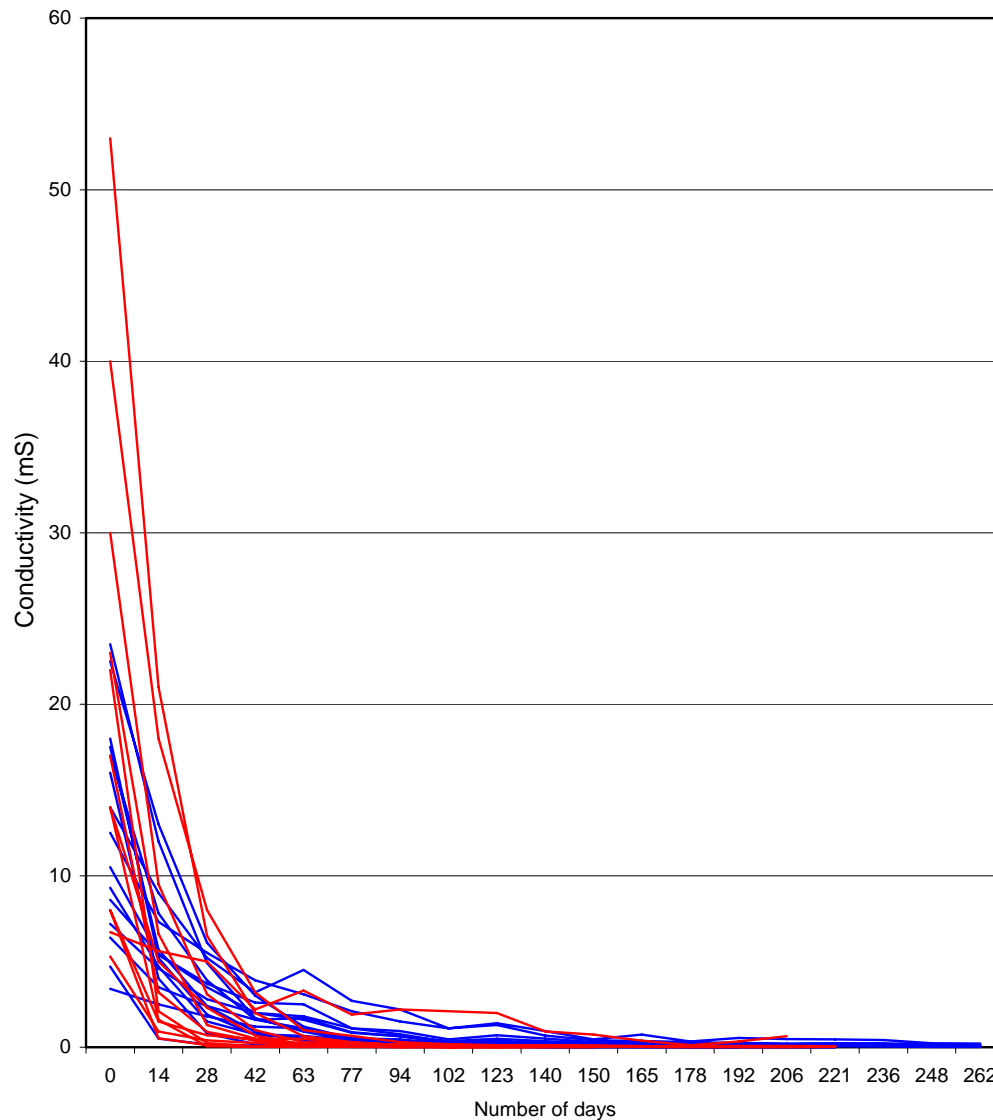
The failure of the consolidant Paraloid® B-72 is most likely explained by its glass transition temperature (T_g) which is 40 °C, i.e. lower than the temperature of the heated extraction water. Therefore, if Paraloid® B-72 is to be used as a consolidant during salt-extraction at elevated temperatures, it is obviously important to set the temperature below 40 °C. The artefacts consolidated with Paraloid® B-72 in water at room temperature, artefacts with ID nos. 18 and 22, are so far intact despite the loss of some minute fragments. An overall evaluation of the consolidating and supporting abilities of the materials tested here will be done after the final step of the re-conservation study.

With regards to the salt extraction process, some tendencies can be noted. Up until the end of May 2006, the extraction baths at room temperature had been running for 271 days and a total of 19 water-changes had been carried out. Out of the 15 samples, desalination was completed for nine according to the set criteria (see above). The desalination process had on average required 216 days and 16 water-changes to reach completion.

The heated baths, initiated nearly seven weeks later, had been running 224 days. A total of 15 water-changes had been carried out during this time. Out of the 13 samples, desalination was completed for nine. On average, these artefacts needed 146 days and 11 water-changes to complete the process, i. e. more than 30 % less in terms of time and work as compared to the un-heated process.

The graphs representing the decrease in conductivity over time clearly illustrate the difference between the un-heated and the heated baths (see Graph 1). However, heated water can not be recommended until its possible negative effects on the material have been examined, which can be done systematically only when all artefacts have completed the salt

extraction process. The damage risks are likely to be higher as increased heat generally causes increased chemical and mechanical action.



Graph 1. Comparison of desalination curves for artefacts in the re-conservation pilot study; blue curves represent artefacts desalinated in cold water (room temperature) and red curves represent artefacts desalinated in warm water (approx. 48 °C).

Naturally, many factors other than the extraction water temperature influence the salt extraction process and its rate. The purity of the extraction water, how often water-changes are carried out, the thickness and volume of the artefact, the amount of alum in the wood, the porosity of the wood, which in turn is dependent on the wood species and the state of deterioration of the wood, and the permeability of possible surface treatments and physical supports or consolidants, are all further factors which influence the rate and success of the process.

To what degree the permeability of the various supports and consolidants may differ will not be studied here; it can simply be stated that all physical supports and consolidants tested are clearly permeable to the ions of the alum salt in water solution. In stages 2 and 3 of the re-

conservation pilot study it will be shown how well the supports and consolidants allow for PEG-impregnation and freeze-drying.

Yet another obvious aspect of the process is the influence of micro-biological activity. Although this aspect will not be studied here, it should be noted that micro-biological growth was evident and extensive in baths at room-temperature, whereas no activity visible to the eye showed in the heated baths.

Concluding remarks

The methods and results presented in the re-conservation pilot study should be regarded as general guidelines given the small number of artefacts used in the study in conjunction with the extremely heterogeneous nature of archaeological objects, and including the large number of unknown factors involved. It is hoped that the final result of this practically oriented study will serve as a general direction-finder and it would be of great advantage if this study could be followed by a larger scale study, both concerning quantity of artefacts and number of physical supports and consolidants tested.

Existing research on alum points to the necessity of further study into the actual behaviour of the alum salt at various RH levels. A number of important questions need to be answered including at what levels of RH alum gains or loses water? How do the crystals grow? What is the effect of fluctuating RH levels on the crystals? How does the addition of glycerol affect these processes? Determining the critical RH-range and deciding what levels of RH fluctuation the wood will tolerate will allow us to give better environmental recommendations for storage and display. It is also essential that we gain better understanding of the chemical and physical processes governing the deterioration in the wood caused by the alum method.

When considering re-treatment of objects of cultural and historical value the ethical aspects of minimum intervention must also be taken into consideration. An active re-conservation measure is always a potential threat to the original state of and the historical record represented by the artefact. The possible risk of damage during re-conservation has to be weighed against the positive effects it may yield i.e. increasing the physical stability of the artefact and making the object less sensitive to environmental factors such as RH.

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