Saving archaeological iron using the Revolutionary Preservation System

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Abstract

Over the last few decades much research has been devoted to the removal of chlorides from and the stabilization of iron objects after excavation. Once this treatment is complete, iron artefacts are generally examined briefly by researchers and then passed to the collections manager. These materials represent structural hardware, tools, weaponry and, occasionally, decorative objects. They are sparsely represented in exhibit spaces and therefore usually occupy the "dead storage" areas of a museum or university laboratory. Often forgotten, iron objects generally turn to dust during their years of storage. For the past 12 years, researchers at Memorial University of Newfoundland have been uncovering the archaeological remains of one of Canada's earliest English settlements. George Calvert, later Lord Baltimore, established the Colony of Avalon in 1621. The site has been occupied almost continuously since then. More than one million artefacts have been excavated with approximately 20% of the assemblage made of iron. Realizing that treated iron in storage was re-corroding, fast intervention was needed to prevent further damage to the objects while preparing a re-treatment strategy. Our solution was to bag all re-corroding iron in RP/ESCAL enclosures. The Revolutionary Preservation (RP) System is an airtight packaging system. It was purchased to store items in an oxygen-free, low relative humidity (RH) environment thus preventing further corrosion until they could be re-treated. This paper outlines the authors' observations and experiences in bagging hundreds of fragile iron artefacts. In addition, preliminary results of an accompanying research project studying 150 building nails stored under a variety of conditions will be described.

Keywords: Archaeological iron, storage, corrosion prevention, Revolutionary Preservation/ESCAL

1. Introduction

The Ferryland archaeological site is located approximately 80km south of St. John's on the Avalon Peninsula of the Island of Newfoundland within Canada. Located on the Atlantic ocean this temperate zone provides a moist, acidic soil with a high salt content.

Historical documents indicate that Calvert's colonists constructed a mansion house, brew house, salt works, forge, hen house, kitchen, fishing stores and dwellings around the "Pool". Lord Baltimore visited his colony in the summer of 1627 and returned with his family in 1628. Sir David Kirke was granted the Island of Newfoundland in 1637 and took control of the colony in 1638. The Dutch attacked the area in 1673 inflicting significant damage on several plantations, especially Ferryland (Lovelace 1675). The colony recovered from this attack, but after the French captured and burned the settlement in 1696 activity at the site ceased until the 1700s.

The present Ferryland Project began excavations in 1992. The site itself is believed to cover an area of approximately 30,000 m^2. To date about 1,100 m^2 have been excavated and designated as Areas A through G. Briefly described, these areas represent the landward palisaded edge of the colony (Area A), the houses of middling planters (Areas B and D), a waterfront warehouse complex (Area C), defensive works (Area E), a high status house, possibly the Kirke residence (Area F), and another waterfront area with an early 18th century domestic structure (Area G). Artefact assemblages including textiles, glass, ceramics, ferrous and non-ferrous metals, brick and wood are concentrated in areas defined by structures. To date catalogued artefacts number about half a million with many numbers representing two or more objects. Of this group about 20% are made of iron. Each year of excavation uncovers about 7,000 iron nails and 1,000 to 2,000 iron objects. Though we are required by permit to document and retain the nails, conservation of these object types is not a requirement. We are required, however, to fully conserve and store all non-nail iron objects. The cost of conserving artefactual remains, for the Ferryland project, is equal to that of excavation. Table 1 presents the cost of conservation for a nine year period. The building facility is included here as this serves as the official repository for the collection.

Table 1. Total Cost for Conservation of Artifacts from 1991 to 1999

conservation supplies	$77,874
building facilities	$626,000
salaries	$1,289,840
analyses, treatment containers and collection storage containers	$323,000
TOTAL	$2,316,714

Table 2 shows the number of hours required to conserve artifacts. Note that objects made of iron require much more time to conserve because chloride removal is required to stabilize ferrous metal.

Table 2 . Hours Required to Care for an Historic Archaeological Collection

Hours required to process artifacts from 10 weeks of excavation				
Material	Number of Artifacts	Hours		
		Cataloguing	Stabilization	Conservation
glass ceramic pipe	18,000	1,500	1,500	1,000
bone	1,100	100		175
iron objects	544	200	500	5,000
iron nails	12,000	10,000	2,000	1,000
misc. inorganic + organic	78	48	30	800
copper	47	10		37
lead objects	105	15		38
lead shot	276	30	40	
wood	300	25		25

By the early modern period iron was used for a variety of objects from structural hardware, to blacksmith's tools, to snuff boxes (Figure 1). For this reason iron artifacts are numerous on any historic site and scattered through all areas of excavation. For the thousands of iron objects excavated each year at Ferryland condition varies greatly by object and area of excavation. Some are better preserved than others. Because the site's soils are coarse in texture and allow easy movement of water and dissolved salts objects appear to always be exposed to the corrosive environment of a maritime climate.

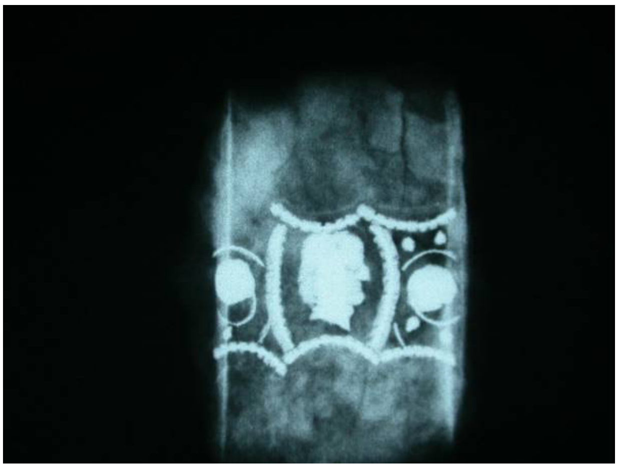

Figure 1 X-radiograph of iron snuff box with silver decoration

Geochemical analysis of the soil solution indicates a range of chloride concentrations from 635 ppb to 33,109 ppb. Soil pH ranges from 3.81 (H_20) to 5.74 (H_20). Corrosion rate measurements range from 0.02-0.22 millimetres per year. After some 350 years of burial this means that an object's corrosion layer could vary from 7 mm to 77 mm. Table 3 shows the optimal and mean values for the soil composition.

Table 3 Optimal Burial Environment

	Particle size distribution (%)			pH			SiO_2 wt%	Cl (ppb) for soil solution	Cl (ppm) for soil
				H_2O					
Optimal	43	53	4	5	30	0.09	55	3,858	417
Occupation/destruction events									
mean	45	50	4	5.03	22	0.08	55	3,169	399
Fill/building events									
mean	48	48	4	4.61	33	0.09	56	6,487	358

Table 4 provides a summary of soil texture for the site. The burial environment has been described by event to distinguish between areas of occupation and building construction. Fortunately most iron objects were excavated from soils with a lower rate of corrosion. The higher rate of corrosion corresponds with areas of high soil chloride concentrations where the seawater is in constant contact with soils. These are areas along the colony's waterfront where recovered metals were found to be in poor condition. Thick corrosion layers can be expected in areas with high chloride concentrations.

Table 4 Soil texture of the Ferryland Archaeological Site Matrix

	pH	Organic wt%	Colour	Mineral Composition
Range	3.81 to 5.74	2 to 30	dark yellow brown - dark grey	subsoil- quartz, albite, phlogopite
Occupation events				
mean	5.03	11.3	dark yellow brown - dark brown	quartz, albite, sepiolite, phlogopite, illite, muscovite
Fill/building events				
mean	4.61	10.8	Pale brown - brown	quartz, albite, phlogopite

We know that soaking iron in sodium hydroxide solutions removes most of the chlorides.

2. Treatment

At Memorial, iron artefacts have been treated with aqueous sodium hydroxide solutions of either 0.5 or 1.0% (w/v) through at least six changes of solution. Most treatment times average two years. This treatment method has been developed based on work by Scott and Seeley (1987),Costain (2000) and Mathias (1994). Mathias worked with Costain on the holding solution experiments while interning at the Canadian Conservation Institute (CCI). Realizing the ability of sodium hydroxide to remove chlorides, after monitoring solutions for chloride concentrations, Mathias felt this would be a suitable treatment for the bulk treatment of iron at Memorial University. This decision was made in consultation with Judy Logan, archaeological conservator working at CCI. After treatment some mechanical cleaning is conducted, but no such cleaning is performed during treatment because of the limited availability of personnel. In many instances it appears that chlorides remain within the pores of the artefacts and will not move into solution if a thick silica-rich corrosion layer is still present during and after chemical treatment. It is these artefacts for which the RP/ESCAL system is used. Because chlorides are potentially still trapped in the iron they will begin to re-corrode during the summer months when the relative humidity can exceed 65% in June, July or August and akaganeite will form. Figure 2 presents the change in RH for the collections storage area from 1993 to 2003.

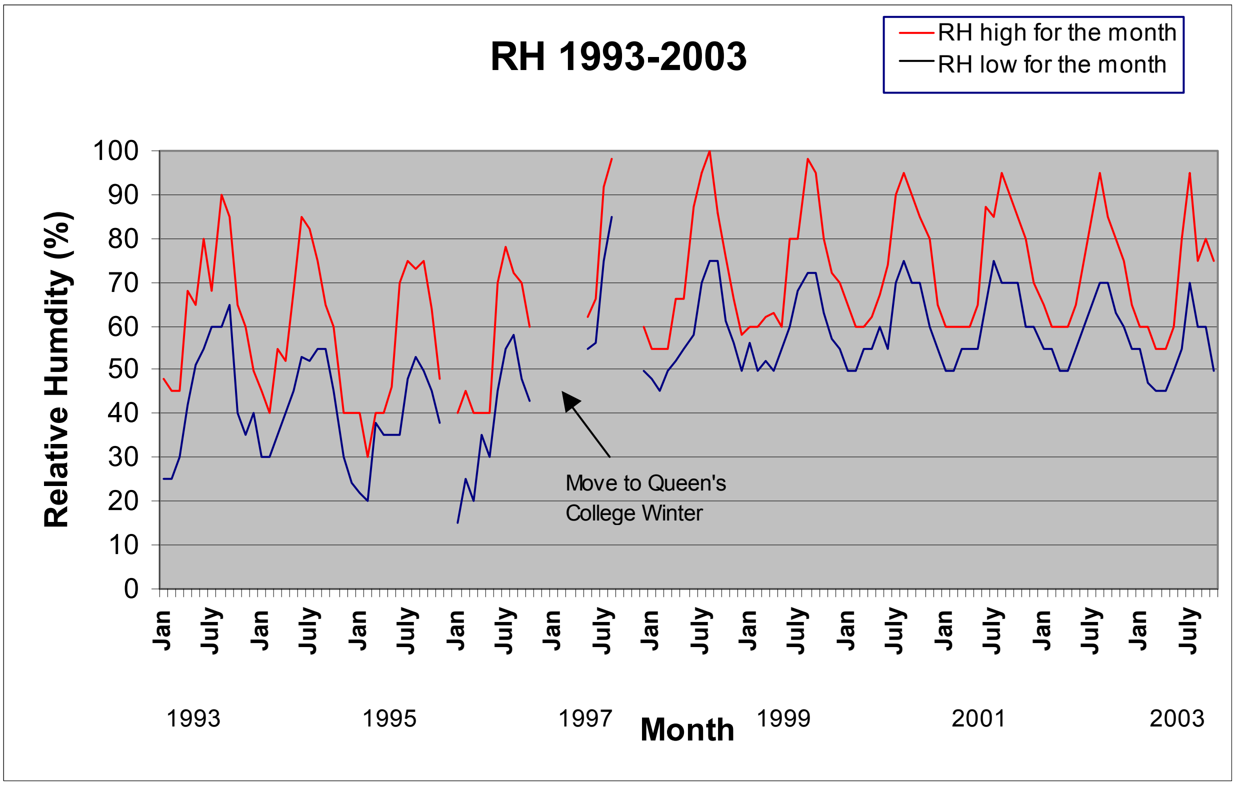

Figure 2 RH for collections storage area from 1993 to 2003.

During the winter of 1996/97, the conservation lab and collections storage were moved from the Ingstad building to the lower level of Queen's College, several city blocks away. Queen's College is located next to a large pond, which seems to have had an adverse effect on the ambient environment inside the building. Data collected over a ten year period at both buildings shows an average increase in relative humidity of over 10% at Queen's

College as compared to the Ingstad Building.

The archaeological iron collection is currently stored either in baked enamel, gasket sealed, metal cabinets, if a non-nail iron object, or in large plastic tubs, for nails. The cabinets are conditioned with silica gel sachets but given the limited availability of staff these are not well maintained. Individual items are wrapped in acid-free tissue and placed in hard plastic boxes or in polyethylene bags. Ethafoam supports are made for delicate items. Humidity is controlled only through the use of portable dehumidifiers. Figure 3 shows an area of "fuzzy" akaganeite which developed along a crack on an object stored under these conditions.

Figure 3 Akaganeite growing on iron substrate

A scanning electron microscopy image, (220 X magnification and 20 KV), of akaganeite growing perpendicular to the iron substrate, is shown as Figure 4.

Figure 4 SEM of akaganeite growing perpendicular to iron substrate

Overall it was found that the interesting iron objects stored in the larger collections storage area in the storage cabinets were at greater risk of physical breakage because of the appearance of akaganeite. To date we have not observed the akaganeite phase on freshly excavated iron (Mathias 1999 and Turgoose 1985). Akaganeite appears only after an object has been treated, dried and in storage for a period of at least one year. This observation may be specific to the burial environment from which the archaeological iron was excavated and the storage environment, in terms of both relative humidity and air pollutants (Turgoose 1982b and Jones 1992). As stated above, accelerated deterioration because of corrosive processes, occurs with an increase in relative humidity within the storage environment. Humidity is necessary for this type of atmospheric corrosion. Condensed water forming with an increase in relative humidity on the surface of the metal will provide the electrolyte needed for electrochemical corrosion (Jones 1992). Contaminants near the surface layer, such as chlorides, enhance the effectiveness of the electrolyte (Jones 1992). Thus uncontaminated iron would not corrode as the humidity increased, however, this is not the case for the Ferryland iron (Mathias 1999). The ESCAL/RP will slow the deteriorating effects of the environment by reducing humidity and oxygen until these objects can be re-treated. Therefore, to prevent corrosion from occurring, archaeological iron should be stored in an oxygen free environment with an RH of less than 15%

2.1 Revolutionary Preservation (RP) System

The RP agent is available in two different forms - RP-A, for use with metal artefacts, and RP-K, to be used with non-metal items. The use of RP-A will result in a microenvironment with an RH of less than 10% and an oxygen concentration of below 0.1%. RP-A is available in three sizes - 3A, 5A, and 20A. The numbers correspond to the volume of air each packet can condition - 3 = 300 ml, 5 = 500 ml, 20 = 2000 ml.
The RP-A packets contain mordenite, calcium oxide, polyethylene and activated carbon, along with unsaturated organic compounds, the makeup of which is proprietary information.

The heat-sealing layer of ESCAL film is linear low density polyethylene (LLDPE). In rolls of ESCAL that are pre-sealed on two sides, the LLDPE layers are already oriented to the inside, where they will melt together when heat of an appropriate temperature is applied. In ESCAL film, the plastic must be folded correctly or the heat will not seal the bag. Although heat-sealers are recommended for use with the ESCAL film and RP system, they can be quite expensive. Heat sealing using a tacking iron has also been suggested as a low-cost option to the more expensive heat sealers. As an alternative, the conservation lab at Memorial has used a conventional iron for heat-sealing the ESCAL bags. The strength of the seal created by the iron was tested by pulling on the film to try and separate the newly joined layers. It was impossible to tear the seal by hand, indicating that the iron creates a sufficiently strong seal.

The microenvironment created using ESCAL and the RP agent can last from four to six years assuming: a good seal has been obtained, that the film remains undamaged, and the proper amount of RP agent has been added. After this time, the RH will begin to rise, although the oxygen-free environment can last much longer (Conservation by Design 2003).

To determine how much and how long the RH drop takes in the microenvironment using the RP system, a datalogger was enclosed to chart the decline of the RH. A HOBO LCD Temperature/Relative Humidity datalogger was used, set at a sampling interval of two minutes. The logger has an operating range of 0-95% RH, but a measurement range of only 15-95%. The ambient RH at the time of placement in the enclosure was 42%. In just over two hours, the RH within the package dropped to 15% (lowest range for the logger).

Although the temperature in the enclosure is not directly affected by the RP agent, there was a temperature spike noted on the logger graph. It is possible that the oxygen-scavenging reaction caused by the RP agent is exothermic, resulting in small rise of 1ºC in temperature during the first two hours of enclosure.

2.2 Working with the RP System

The following points illustrate the method used in packaging the artefacts:

- The appropriate size ESCAL film is chosen (either 160 mm or 480 mm wide)
- The ESCAL is cut (with ordinary scissors) to fit the object, making sure to leave enough room for the RP packets and sufficient plastic to seal the enclosure on both ends
- One end of the bag is sealed with the iron set to the ‘nylon’ setting
- The object is protected if needed - delicate objects may be placed in small, hard plastic boxes with microfoam padding, and objects with sharp projections may be padded with microfoam to prevent the possibility of puncturing the bag
- The object is then placed inside the bag, along with its catalogue tag. If the objects are small, more than one may be placed in each enclosure. They can be placed in polyethylene bags within the ESCAL to separate them.
- The appropriate number of RP packets are placed in with object, then the end is sealed with the iron. This is determined by calculating the volume of the bag and subtracting the volume of the object. It is much easier to measure only the volume of the enclosure and disregard the volume of the object - this may result in adding slightly more RP agent than is necessary. This will not harm the object, and the manufacturer has suggested adding 25-50% more RP agent to each enclosure. Through trial and error we have found that for wrought iron one bag of the RP- 5A is needed per 100 gram of weight.
- The artefact’s catalogue number can be recorded on the white strip on the ESCAL film.

Previous studies of the RP system using archaeological metal indicate that there should be no change in the appearance of the objects. Research has been conducted in which excavated iron nails were used as samples to show that objects stored using the RP system do not corrode while in storage, and in fact have a slight decrease in weight, due likely to a loss of moisture (Lampel 2003). Nails stored only in polyethylene bags suffered an average weight gain of 7.4% after three years, due to the formation of corrosion products.

After working with the system, it was possible to determine easier methods of working, and several problems were also discovered. The bag of RP agent contains 25 packets, and these should be exposed to air for only 30 minutes before being resealed in their new enclosures. Because most artefacts are small and use only one packet per enclosure it is most efficient to design a working session where 20 artefacts will be bagged. In this way, the entire bag of RP agent could be used.

Heat sealing the ESCAL film with an iron is a fairly straightforward task, though several problems were encountered. If the object is large a bag formed from the large tube (480 mm in width) of ESCAL is difficult to seal. Because several passes have to be made with the iron to seal the end, it is difficult to seal without incorporating any air channels. Also the corrosion 'dust' created by the iron artefacts can actually prevent a complete seal.

When the oxygen is removed from the enclosure, the bag does suck down about 20%. In most cases, this is a visible change, and it indicates that the seal was successful and the correct number of RP packets was enclosed.

2.3 Why is the use of ESCAL/RP system advantageous to archaeologists?

From the perspective of field archaeology the use of the RP/ESCAL system is advantageous for a number of reasons including: its relatively low cost; its ease of use in the field; the accessibility of the artefacts following their enclosure; and the opportunity to stabilize artefacts while alternate conservation strategies are considered. Figure 5 shows stored iron objects in RP/ESCAL bags. These enclosures make access easy as they support the object and allow for close examination of the object while still providing a stable environment free from the contaminants of handling.

Figure 5 Storage of iron in RP/ESCAL bags

The relatively low cost of the RP/ESCAL enclosure system derives, on the one hand, from the low cost of the enclosing materials themselves, and on the other, from savings in terms of labour in a field laboratory. In our experience with the purchase of some $3,000 (Canadian dollars) worth of materials, which may at first glance seem overly expensive, we have managed to enclose more than 200 iron objects of various sizes and still have more than half of the original materials left for additional enclosures. Although other barrier films may be used with the RP agent, ESCAL is recommended for long-term storage as it has very low rates of migration for both water vapour and oxygen - it is 2,000 times more effective as a barrier than polypropylene bags, and it’s transparent, so that objects can be viewed without opening the package (MGC 2003). A carefully planned strategy for the initial processing of the artefacts themselves (for example, a light cleaning followed by the documentation of the object and its condition post-excavation) combined with the careful and judicious use of the enclosing materials, makes a little go a long way. The labour savings in a field lab derive from the relatively “light” handling an artefact receives prior to its being enclosed in a relatively sturdy environmentally-appropriate enclosure. The light handling of the objects not only protects them, but reduces the number of laboratory staff required to process the objects themselves.

The ease with which enclosures can be created in the field is likewise advantageous in terms of time, effort, and materials. In our experience, it is more efficient to enclose a number of objects at once where attention can be focussed specifically on cutting ESCAL enclosing tubes to size, estimating the number of RP sachets to be included, placing the artefacts within the enclosure and then heat-sealing it. It doesn’t take long to get the feel for estimating the size of tube to be cut and the number of sachets to be added. Once the iron is up to heat, sealing the enclosures is quick. A team of two can easily enclose some 20 objects an hour. Laboratory requirements for creating the enclosures are meagre: a clean work area; a flat surface upon which to iron the ends of the enclosures; and an electrical outlet for the iron.

Once enclosed in the RP/ESCAL system the artifacts can be easily visually examined not only for purposes of additional identification, but also to monitor their condition. For the purposes of transportation and storage, the semi-rigid nature of the enclosure allows the objects to be packed more readily, albeit carefully, for travel, while in storage the enclosure provides ongoing environmental protection for the object. If an artefact requires closer examination or additional stabilization, it is as easy as cutting the end of the ESCAL tube and removing the object. In this sense, the RP/ESCAL system is readily reversible, unlike most other chemical treatment strategies. Following examination or additional stabilization, a new enclosure can be made and the artefact returned to a stable storage environment.

Perhaps the most important advantage of the RP/ESCAL system for field archaeologists is that it provides us the opportunity to consider alternate stabilization strategies -in a real sense it buys us time. It is abundantly clear that a variety of conservation strategies have been undertaken in various circumstances and that some have been more successful than others. Depending on the degree and nature of the corrosion experienced by ferrous objects, different conservation strategies are appropriate. This system allows archaeologist to take the necessary first steps, in the field, to stabilize objects until the best long-term strategy is selected. Because the objects themselves have not been otherwise "treated", this system reduces the likelihood that inappropriate and irreversible treatments will be performed by archeologists who lack the specific knowledge that is the realm of the conservator.

3. Methods

A small research project was begun in December 2003 in an effort to determine the efficiency of the RP/ESCAL system in reducing the re-corrosion of iron nails recovered from the Ferryland archaeology site. The total sample included 150 nails, half of which had received 2.5 years of treatment in sodium hydroxide to remove chlorides, the other half had received no post-excavation conservation treatment. Each group of 75 nails was subdivided into three groups of 25 nails each. These three groups of treated and untreated nails were then stored differently. The first group of 50 nails (25 treated and 25 untreated) was stored in individual RP/ESCAL enclosures, the second group of 50 nails (25 treated and 25 untreated) in individual polypropylene bags, and the third group of 50 nails (25 treated and 25 untreated) stored without enclosure. The total sample of 150 nails has been stored in an ambient environment. Our intention is to visually monitor the condition of the three groups three times each year for the next five years.

Additionally it was decided to select a group of objects(not nails) from four areas of excavation, and make observations about the RP/ESCAL packaging technique over the course of the experiment. A total of 55 objects were sealed in the RP/ESCAL system, some with microfoam for support and protection against film puncture. Once bagged the initial weight was recorded. The quality of the bag seal was visually assessed as good, fair or poor. The initial bagging occurred March, 2004. Objects were monitored in April and June of 2004.

4. Results

The first monitoring interval for the nail experiment has revealed the following. All nails treated with sodium hydroxide appear stable regardless of method of enclosure. Some untreated nails show signs of continued deterioration and this varies by method of enclosure. After being bagged for 5 months:

12% of those nails in RP/ESCAL enclosures are visibly cracking;
60% of those nails in polypropylene bags are visibly cracking; and
56% of those nails with no enclosure are visibly cracking.

The nails were monitored again in June. Though the time interval between examinations, in this case, was short, we were entering the first period of increased humidity and felt we may see changes. The treated nails remained stable. However within the untreated nail group an additional nail in each of the polypropylene and RP/ESCAL groups appeared cracked. The results are presented in Table 5. The second group of objects being monitored showed that for three samples the protective microfoam had moved off the intended area of protection. This will probably result in bag damage. Overall, however, the seals were good.

Table 5 Total Nails Showing Signs of Deterioration

	Untreated Nails			Treated Nails		
Date	no enclosure	polypropylene	RP/ESCAL	no enclosure	polypropylene	RP/ESCAL
Dec. 15/03	0	0	0	0	0	0
May 20/04	14	15	3	0	0	0
June 11/04	14	16	4	0	0	0

*Samples for untreated nails were excavated in 2003

5. Discussion

It is too early to know yet how effective the bagging of corroding iron in the RP/ESCAL system will be but at this point it does look promising. Experiments, to date, do reveal that it is important to have a good seal and that for archaeological objects with sharp edges and irregular three dimensional shapes maintaining the seal will be the greatest challenge. I know that over the past 24 years that I have been involved in the preservation of our heritage that there has been a move away from bench conservation treatment to preventive conservation. Institutions can no longer afford a conservator who may spend one year on one object. To justify the conservation position they must have one conservator stabilizing hundreds or thousands of objects per year. This preservation system, if it proves successful, will help archaeological conservators meet their goal of stabilizing thousands of objects. However, I must emphasize that archaeological materials will require some form of chemical stabilization prior to storage and the use of the RP/ESCAL system. The ESCAL packaging system will, however, continue to protect these fragile objects once removed from chemical treatment and make them easily accessible to researchers.

Additionally the RP/ESCAL system can be used for fragile artefacts being excavated and as an emergency intervention. In the former case, objects excavated from a wet saline environment can be bagged and held in a stable state until aqueous treatment for chloride removal can begin. The Ferryland cross made of iron, brass and gold, provides an example for the latter. This 17th century artifact began re-corroding while on exhibit in 2003. Figures 6 and 7 show the extent of damage.

Figure 6 Ferryland iron cross with physical signs of damage

One of the orbs cracked off as a result of this damage. Figure 7 shows the cross-section image of the orb. Note that the corrosion has progressed from the metal surface inward to the core. This object is currently bagged in RP-5A/ESCAL awaiting further treatment.

Figure 7 Cross-section view of Ferryland cross orb

6. Conclusions

Collections storage issues are becoming more challenging for institutions worldwide. Within academic units, issues such as budget cuts and increasing numbers of students are forcing administrators to carefully monitor space usage. Archaeology departments with on-going research projects will require significant space for collections storage. Because the RP-5A/ESCAL system provides a constantly low RH and oxygen free environment it may be ideally suited for the metal artefacts in these collections. It is a relatively low cost solution compared to air-conditioning the larger collections storage space. Additionally these enclosures provide support to archaeological objects which may be moved many times in their post-excavation period.

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Suppliers

The RP Agent and Escal Barrier Film are manufactured by
Mitsubishi Gas Chemical Company
5-2, Marunouchi 2-chome, Chiyoda-ku, Tokyo 100-8324, Japan
www.mgc-a.com

The RP System is supplied to Canada and the USA by
Keepsafe Systems
570 King Street West
Suite 400
Toronto, Ontario
M5V 1M3
www.keepsafe.ca

and in the UK by
Conservation by Design Limited
Timecare Works, 5 Singer Way
Woburn Rd. Ind. Estate
Kempston, Bedford
MK42 7AW
www.conservation-by-design.co.uk

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