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THE FIELD MUSEUM ARCHAEOLOGICAL METALS PROJECT: DISTRIBUTED, IN SITU MICRO-ENVIRONMENTS FOR THE PRESERVATION OF UNSTABLE ARCHAEOLOGICAL METALS USING ESCAL BARRIER FILM

J.P. BROWN

ABSTRACT

Low-humidity microenvironments using polyethylene (PE) and, latterly, polypropylene (PP) boxes with ca. 80 kg/m^3 of desiccated silica gel are widely used in preventive conservation for the storage of unstable archaeological metals from terrestrial contexts (Cronyn 1990, Logan and Selwyn 2007, Scott and Eggert 2009). One notable disadvantage of this method is that, because PE and PP are poor moisture barriers, the silica gel must be regenerated or replaced annually. The Field Museum was found to have 4,500 unstable archaeological metal objects distributed through its stored collections, requiring an estimated 400 kg of silica gel if PE or PP boxes were used. Instead, we opted to use enclosures made of Escal, a transparent plastic laminate primarily used for anoxic storage because of its low oxygen transmission rate, but which also is also suitable for low-humidity applications by virtue of its extremely low water vapor transmission rate (comparable to aluminized plastic barrier films). Using Escal has allowed us to reduce the amount of silica gel used to 5 kg/m^3 with a predicted regeneration interval of at least ten years for iron objects and at least forty years for copper alloy objects. In other words, the use of Escal reduces the quantity of desiccant by a factor of sixteen and extends the interval between regenerations by a factor of ten. In this paper we discuss the theoretical considerations that led to our choice of Escal as a barrier film, the practical details of implementation, and report on the progress of the program after the first six years.

1. INTRODUCTION

A series of broad condition surveys performed at the Field Museum in the late 1990s noted that there were a significant number of archaeological metal objects which were actively corroding in the Anthropology Department's storage vaults. In 2002 funding became available specifically for metals conservation and we were able to address the problem.

We first performed an object-by-object condition survey broadly following Keene (1996) and entering data directing into a FileMaker database on a laptop. Objects with corrosion products that appeared to be unstable chloride-related compounds (copper trihydroxychlorides, β-ferric oxyhydroxide) were sampled and the corrosion product tested for chlorides using acidified silver nitrate on a blue spot-plate after Odegaard et al. (2000). Object condition was rated on a 1-4 scale with 1 representing good stability, and 4 representing active corrosion and/or delamination. Objects testing negative for chlorides were rated in categories 1 and 2. Additional information was collected to describe the number of objects and fragments associated with each catalog number, as well as the non-metallic components of composite objects (fig. 1).

By the end of the survey we had found 6,832 catalog numbers representing 10,237 archaeological objects made wholly or partially of metal with 9,838 associated fragments. 863 pieces (about 4% of the total) were classified as class 4 objects showing active corrosion; these objects were mostly archaeological iron but also included some copper alloys. A further 3,173 pieces (about 15% of the total) , mostly copper alloy showing evidence of bronze disease, were categorized as class 3 objects and judged likely to be unstable at high humidity. Several of the class 3 and 4 objects had associated, non-mineralized organic remains.

Treatment was not possible for this number of objects within the constraints of the available funding, so we looked for an environmental control method to prevent further deterioration.

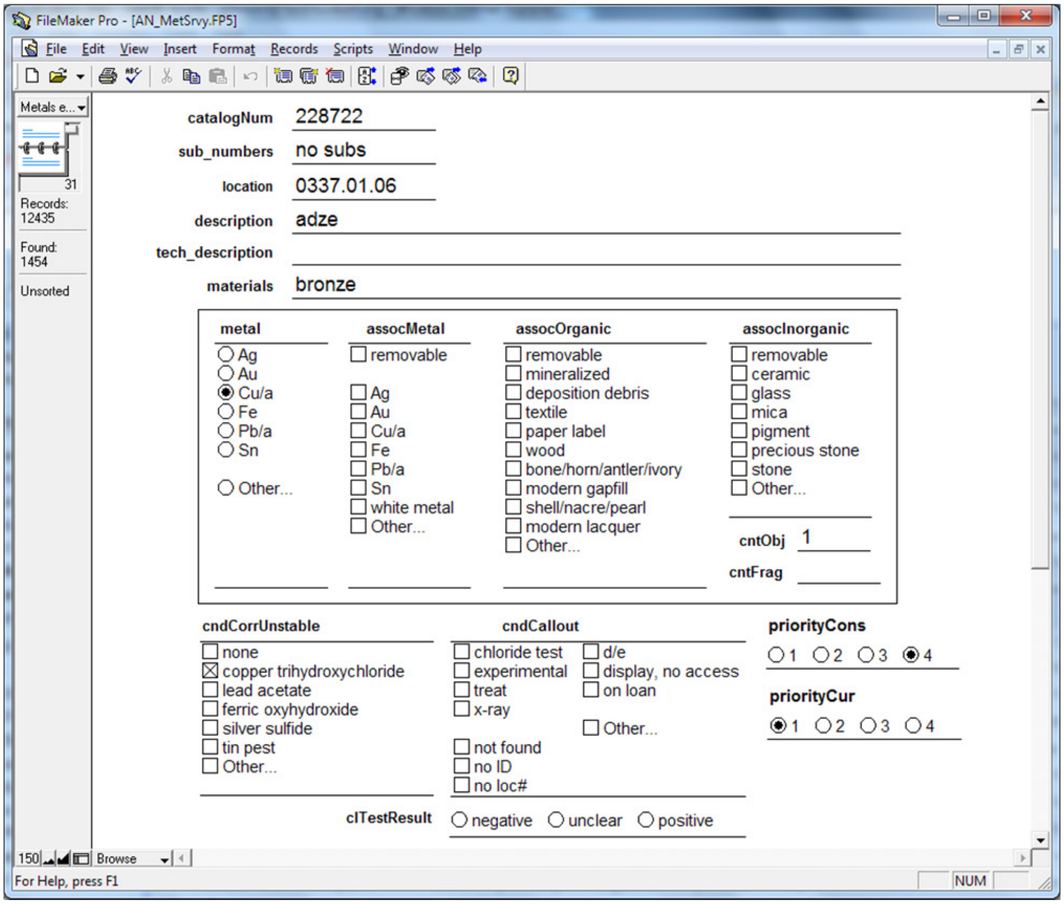

Fig. 1. Filemaker data entry screen for the survey phase of the project (Courtesy of JP Brown)

2. ENVIRONMENTAL CONTROL OF CORROSION

Corrosion of iron and copper alloy objects can be controlled environmentally either by removing oxygen from the atmosphere surrounding the objects, or by controlling the relative humidity to low levels.

Anthropology collections at the Field Museum are stored by continent, country and culture group rather than by period or material type; storage space, although not critically limited, is at a premium. Relative humidity in the archaeological storage rooms varies from a summer high of 66 %RH to a winter low of 20 %RH and median values are in the mid 40’s – even the winter levels are moist enough for unstable iron to corrode (Watkinson and Lewis 2005) and the median and summer levels are sufficient for bronze disease to progress (Scott 1990). Curatorial policy required that we maintain storage organized by culture group rather than material sensitivity (i.e., that we keep any unstable objects at or very near their original shelf locations). This requirement immediately ruled out a dedicated low-humidity room and meant that the selected control method(s) should not measurably increase the storage footprint of the objects.

We considered classical storage in polythene boxes with desiccated silica gel (Watkinson 1987:21), but the range of object sizes that we needed to accommodate would have made this very difficult to accomplish. In addition, polythene is not a particularly good moisture barrier and so 80 kg/m^3 of desiccated silica gel are recommended and the gel needs to be regenerated every year, sometimes biannually, to keep humidity below the corrosion point for unstable archaeological iron. From the survey we estimated that we required at least 5 or 6 m^3 of space for the corroding and at-risk collections giving a total of around half a metric tonne dry weight of silica gel to regenerate every year. The question then became whether there was a more effective material that we could substitute for polythene.

3. BARRIER FILMS AS AN ALTERNATIVE TO POLYTHENE BOXES

Metalized plastic films such as Marvelseal 360 have orders of magnitude lower oxygen and water vapor transmission rates than polyethylene (Burke 1992), the film material can be conformed close enough to object surfaces to allow the packaged object to be returned to its original shelf location, and enclosures for large objects can be pieced together from sections of the film.

We chose Escal as our barrier in preference to Marvelseal 360. Escal is a three layer composite film: polypropylene on the outside, tiny ceramic platelets in a polyvinyl alcohol binder at the center providing a tortuous path to control gas exchange, and polyethylene on the inside to allow heat-sealing (Keepsafe Microclimate Systems, nd). Escal's oxygen and water vapor transmission rates are very low – comparable to Marvelseal – and, whereas Marvelseal is opaque, Escal is transparent and thus allows inspection and inventory of the objects in the enclosures.

Escal is available as rolls of sheet stock or as lay-flat tubing in three exterior widths: 16, 24, and 48 cm. We used the lay-flat tubing because the material can be cut to length and only requires two heat-seals (one at each cut end) to form a sealed enclosure. Each of the two long sides of the lay-flat tubing has a 1 cm seam and so the interior dimensions of the tubes are 2 cm less than those given above. The bulk Escal film has a water vapor transmission rate of 0.01 g/m^2.day at 60 %RH and 25 °C (Mitsubishi Gas Chemical Company, nd). The polythene-to-polyethylene heat-sealed seam travels right around the edges of a finished enclosure and, given that the water vapor transmission rate of polythene is much higher than an equivalent thickness of Escal, the thickness of this seam is the limiting factor in the oxygen and water vapor transmission rates of the enclosure. If the polythene-to-polythene heat-seal seam is 10 mm wide, the seam's resistance to the passage of water vapor is only a little less than the resistance of bulk Escal. A white data stripe is printed on one side of the tubing which allows labeling of the enclosures with permanent marker to help with subsequent inventory and survey.

For control of corrosion we generally used desiccated silica gel to reduce the relative humidity in the Escal enclosure below the threshold for corrosion. However, where unmineralized organics were attached to an unstable metal object we chose an oxygen scavenging as the primary corrosion control mechanism with buffered silica gel to maintain mid-range humidity conditions inside the enclosure and prevent damage to the organic components.

3.1 CONSTRUCTION OF BARRIER FILM ENCLOSURES

To make an enclosure we cut an appropriate length of tube, and sealed one end. A unique identifier was assigned to each enclosure and written on the data stripe. We added an appropriate amount of environmental control agent and an environmental performance indicator so that we could tell when the action of the control agent was exhausted. Early experimentation showed that it was better to bundle the control agent and the performance indicator together to stop all the components sliding around independently inside the enclosure. The bundling material needs to be fairly transparent to water vapor and oxygen (and light so that the performance indicator can be read) and so we used 2 mil polythene bags with press-to-close seal at the top for bundling the control agent.

For objects we cut a shaped support for the object so that it did not slide around in the Escal enclosure. 1.5 cm thick, Plastazote LD45 (medium density, nitrogen-blown, closed cell polyethylene foam) was used as the support material. The Plastazote sheets were cut down to strips of standard width to fit the three standard Escal tubing sizes.

Objects were placed on the Plastazote strip and their outlines scored into the foam with a wooden modeling tool (fig. 2). The objects were then removed and a cavity to receive the object was carved in the foam using the hot knife with a flat sled attachment (fig. 3). The flat sled attachment on the hot knife ensured that, when multiple cuts were required to form the cavity, all cuts were at a consistent depth (fig. 4). The catalog number for each object was then written next to the corresponding cavity on the Plastazote with a black permanent marker. The objects placed in the support, the appropriate environmental control agent and indicator were added to the enclosure, and the enclosure sealed (fig. 5).

Plastazote is available in a range of colors; we used the white-colored product in this project to make catalog numbers clearly visible. In retrospect, a higher density (more rigid) grade of foam would have been a better choice—the LD45 is too flexible to provide adequate support for larger enclosures and we have subsequently had to place the larger enclosures in acid-free cardboard boxes to provide adequate support.

Generally we had more than one unstable object from a particular shelf or drawer. Where objects were small we put several objects in one enclosure as far as the geometry of the storage location and the objects' properties permitted. For multiple small fragments we first bundled the fragments together in one or more 2 mil press-to-close polythene bags and then enclosed them in Escal with a separate 2-mil polythene bag containing the appropriate environmental control agent and indicator.

The cut ends of the Escal tubing proved more difficult to heat-seal than we had expected: in particular, if the sealing iron is a little too hot, then the PVOH interlayer bubbles, compromising the tortuous path; if the sealing iron is too cold then the inner polythene layers do not weld together to form a seal. The product literature for Escal suggests using an impulse sealer which heats both the top and bottom jaws, has at least a 10mm seal width, and has a timer cycle; we have found that such a unit gives satisfactory results. The model we used (AIE AIE610FDA) can be remotely operated by a foot switch, leaving the operator free to pull the mouth of the enclosure taut prior to sealing. Even with this unit, we sometimes had to make two overlapping seals to get a satisfactory seal.

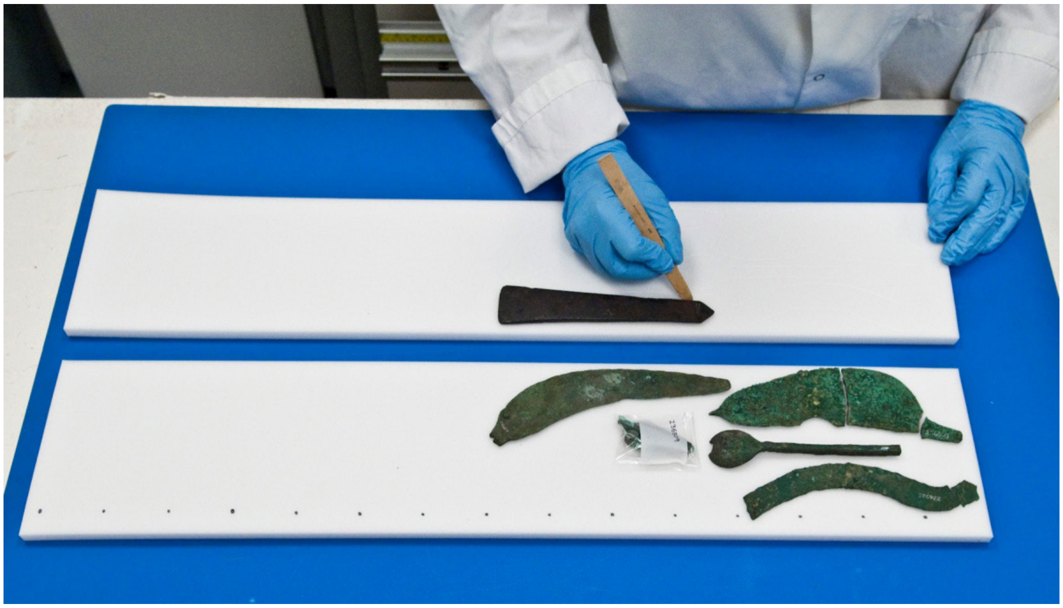

Fig. 2. Tracing shape of objects onto Plastazote foam with wooden modeling tool (Courtesy of JP Brown)

Fig. 3. Carving cavities to receive objects with a hot knife and sled (Courtesy of JP Brown)

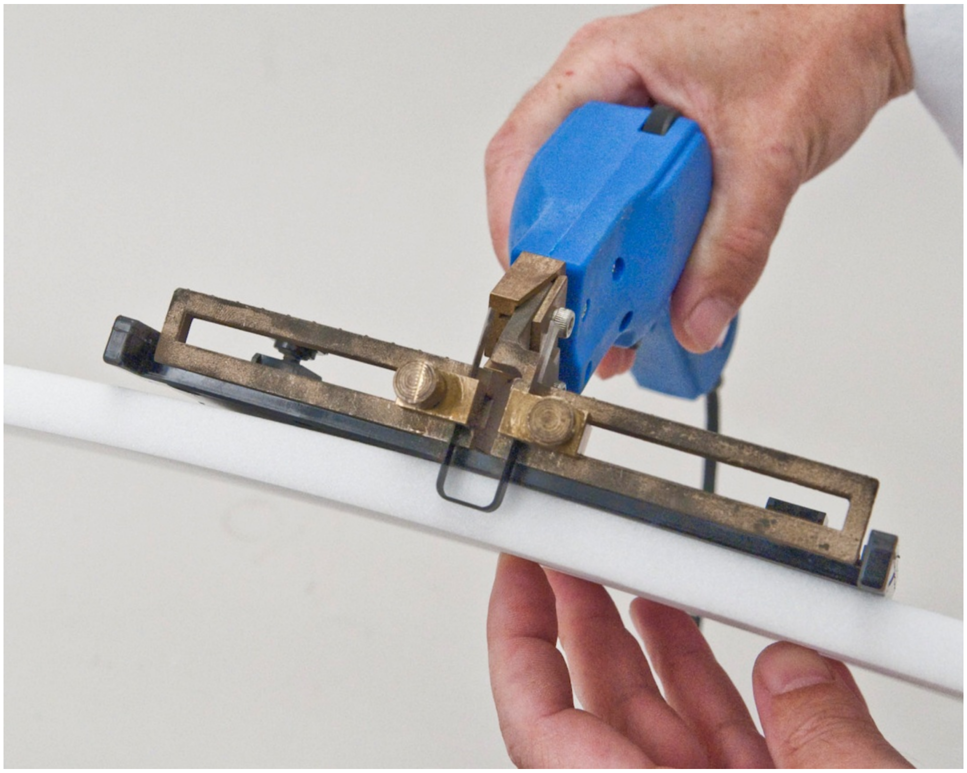

Fig. 4. The hot knife and sled, showing how the sled attachment gives a consistent depth for repeated cuts in the Plastazote foam (Courtesy of JP Brown)

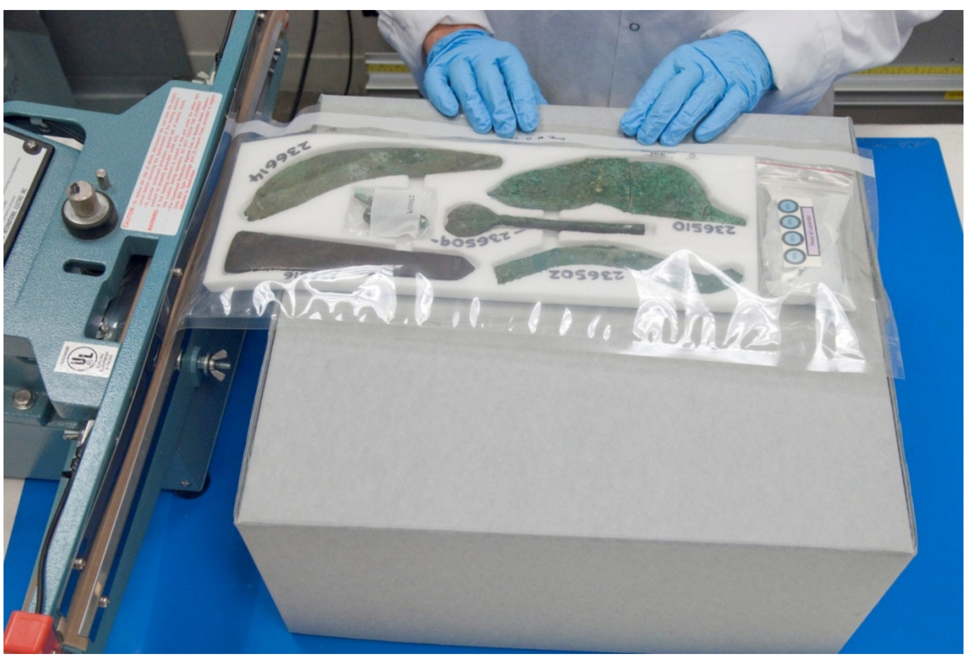

Fig. 5. Heat sealing the open end of an Escal enclosure (Courtesy of JP Brown)

3.1.1 Low-Humidity Enclosures

We determined during the design phase for the project that 5 kg/m^3 dry silica gel would provide adequate desiccating capacity for a well-sealed Escal enclosure, the volume being calculated on the basis that the tube is formed as a cylinder of circular cross-section. Design parameters were that the interior of the enclosures would not reach 10 %RH (just below the threshold for iron corrosion) until after ten years and not reach 40 %RH until after 40 years. Calculations were conservative and assumed the presence of pin-holing, minor imperfections in the heat seal, and a constant exterior humidity of 60 %RH.

It would have been possible to weigh out the dry silica gel for each enclosure, but the weighing of gel into enclosures would have been time-consuming and messy. We purchased dry WR Grace ProTek Sorb silica gel, prepackaged in 0.5, 1 and 2 "unit" nonwoven polyolefin sachets. A "unit" of silica gel is a performance-based measurement defined in US Military Specification MIL-D-3464E (1987) as the amount of desiccant required to absorb 2.85 grams of water vapor at 20 %RH and 5.7 grams of water at 40 %RH. The dry weight of a unit of this silica gel is 23.5 grams.

We developed a nomogram for the number of units of dry gel required for a particular length of the different widths of Escal tubing (fig. 6). Clearly, the internal volume per unit length of a given width of tubing will vary depending on the contents and the degree of inflation (the profile of the cross-section of the enclosure). However, the surface area of a given width of tubing is proportional to its length, and since control of the internal humidity of a diffusion-controlled enclosure is dependent primarily on its surface area and the absorption abilities of the control agent, the mass and efficiency of the control agent (expressed here in MIL-D-3464E units) and the length of the tubing are the appropriate measures.

The silica gel sachets together with a humidity-indicating card reading 10-40 %RH were enclosed in a 2 mil polythene bag with press-to-close seal. We cut away the excess card at top and bottom of the indicator strip to make it easier to pack.

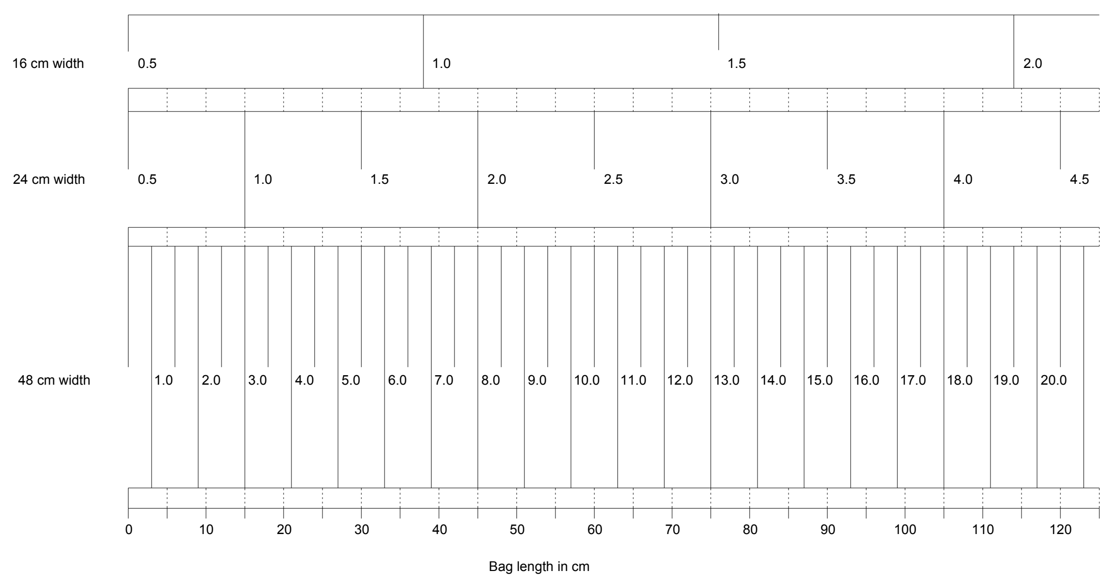

Fig. 6. Nomogram showing the number of “units” of silica gel required
for different lengths of the three widths of Escal lay flat tube (Courtesy of JP Brown)

3.1.2. Low-Oxygen Enclosures

Where there were unstable metals and associated organic materials which could be separated (e.g., wood stand for Chinese mirror) we enclosed the metal separately and then boxed the metal and organic components together (fig. 7).

Where there were non-mineralized organics which could not be removed from corroding metals (e.g., bone handle on unstable iron blade) we used humidified low-oxygen enclosures to control corrosion without desiccating the organics (fig. 8).

We added 5 kg/m^3 of silica gel conditioned to 40 %RH to stabilize relative humidity in the enclosure (primarily as a precaution against humidity changes caused by abrupt temperature fluctuations) and RP-K oxygen scavenger to maintain low oxygen levels. Some oxygen scavengers are activated by atmospheric moisture and consume the water vapor in the enclosure (thus reducing the relative humidity in a sealed environment close to 0 %RH). RP-K, however, is moisture-neutral (i.e., does not consume water vapor), and was ideal for our low-oxygen, mid-range humidity application. For each enclosure, three Ageless eye oxygen indicating tablets were bundled together with the RP-K sachets and a silica gel sachet in a 2 mil press-to-close polythene bag. The Ageless eye tablets are blue in color at >0.5 % oxygen, but change to pink at ca. 0.1 % oxygen and below. Manufacturer-recommended quantities of RP-K were multiplied by three to allow for imperfections in enclosure sealing, pin-holing, and extended life. After the objects, support, and environmental control and indicator bundle had been placed in the Escal enclosure, the open end of the enclosure was partially sealed leaving enough space for a gas tube to be introduced. The enclosure was then flushed with dry nitrogen prior to final sealing to maximize the life of the scavenger and prevent the enclosure contracting.

Fig. 7. Unstable Chinese mirror placed in low humidity enclosure and boxed together with associated wooden stand (Courtesy of JP Brown)

Fig. 8. Unstable iron blade with unmineralized bone handle in low-oxygen enclosure with RP-K oxygen scavenger and silica gel conditioned to mid-range as a humidity buffer (Courtesy of JP Brown)

3.1.3 Enclosures for Large Objects

Although Escal is too stiff to drape easily, the film can conform fairly closely to the surface of the objects. Where the Escal tubing was not large enough for objects, we opened up the 48 cm size tubes to make 92 cm wide sheets and supplemented the missing areas with Marvelseal 360 (fig. 9). A layer of Plastazote was cut and shaped to prevent direct contact between the bottom of the object and the Escal film. We found that Escal seals much more easily to Marvelseal (and to aluminized polythene films in general) than to itself. We speculate that this is because one can safely use a higher temperature to get a good weld since the aluminum foil in Marvelseal dissipates some of the excess heat.

3.2 PROCEDURE FOR OPENING ENCLOSURES

A significant feature of gas barrier enclosures for long-term storage of objects is that, compared to storage in polythene boxes, the opening and resealing of the enclosures is a relatively complex process. In early experiments we had tried leaving excess length of the enclosure to allow for cutting and resealing, folding the extra length under the enclosure, and securing it with pressure-sensitive tape. Folding the extra length proved difficult to implement for the more three-dimensional objects, and was abandoned in favor of re-sealing with gas-tight removable clips after enclosures are opened.

It is important that as much is done as possible to preserve the proper functioning of the environmental control agent while the objects it contains are being examined. The end of the enclosure should be cut open as close to the original heat-seal as possible and the objects withdrawn on their support. The environmental control agent and the performance indicator are left inside the enclosure and a reusable gas-tight clip (supplied by Mitsubishi Gas Chemical Corp, the manufacturer of Escal and RP-K), is applied to the open end. When examination is complete, the clip is removed, the objects are slid back into the enclosure on their support, and then the enclosure is resealed with the clip (fig. 10). The enclosure is then left overnight and the environmental control agent is checked for correct performance (as shown by the environmental indicator in the enclosure) before returning the enclosure to storage. This procedure works well for silica gel controlled enclosures, but the low-oxygen enclosures require re-flushing with nitrogen prior to re-sealing. The clips are available in lengths corresponding to the three different widths of Escal lay flat tubing and are relatively expensive. We bought only the size corresponding to the 24 cm width and cut them down as necessary for the narrower enclosures. The clips tend to splinter when cut by hand with box-cutter-type knives, but a ratchet-action cutter designed for cutting 2" PVC tubing works well.

Fig. 9. Large enclosure made by opening up 48 cm Escal tube supplemented with Marvelseal (Courtesy of JP Brown)

Fig. 10. An opened enclosure re-sealed with Mitsubishi gas-tight clip (Courtesy of JP Brown)

4. PERFORMANCE OF ESCAL ENCLOSURES AFTER SIX YEARS

We surveyed all the enclosures at the end of the housing phase of the project in Dec 2004. Only two out of 848 enclosures showed a change in indicated environmental properties: these were both medium-sized humidity-controlled enclosures and the failures were due to visible defects in the heat seals.

In March 2010 we completed a new survey of all the enclosures, six years after all enclosures were completed (Table 1). The majority of the 836 humidity-controlled enclosures are maintaining internal relative humidity at or below 10 %RH, but 39 of them (about 5% of the total) are now at 20 %RH or above. The ends of ten of 39 "failing" enclosures were cut open and, for unknown reasons, left open and not resealed. Another ten that have been opened and re-sealed with clips are above 20 %RH -- the high humidity may be the result of procedural error (failure to keep the bag clipped shut after the objects were withdrawn). However, the final 19 enclosures (about 2% of the total) are still heat-sealed and their internal environments are nonetheless above 20 %RH. The heat-seals on these enclosures do not appear defective to the naked eye. It is possible that these enclosures were opened, left unsealed, and then resealed. It has also been suggested (Shiner 2010) that if the two surfaces of the cut end of the Escal tubing are slightly twisted during sealing, then a small, unsealed aperture can form along the edge of the tubing seam – more work is needed to confirm whether this is the cause of the problem.

Of the 22 anoxic enclosures, six have been opened and then clipped, and, based on the color of the Ageless Eye oxygen indicating tablets, three of these were showing oxygen levels above 0.5%. It is not clear whether this is due to the clip seal, procedural factors, or some other cause. Procedural factors seem likely. However, it is now six or seven years since these bags were sealed and it is possible that the active life of RP-K oxygen scavenger is reaching its endpoint.

Table 1. Performance of environmental enclosures as surveyed March 2010, six years after initial survey

RH controlled bags: 836

Indicated %RH	**slit**	**clip**	**seal**	**total**
<10	-	80	690	**770**
10	-	7	20	**27**
20	-	7	14	**21**
30	-	1	5	**6**
40	10	2	-	**12**

Oxygen controlled bags: 22

Indicated %O_2	**slit**	**clip**	**seal**	**clip**
< 0.1	-	3	16	**19**
> 0.5	-	3	-	**3**

5. CONCLUSION

Escal barrier film enclosures with 10mm wide heat-seals and appropriate control agents can be used to created humidity-controlled micro-environments for storage where active control of environment is unavailable or inappropriate. The use of 5 kg/m^3 of silica gel in these enclosures will generally maintain internal humidity below 10 %RH for at least six years, probably ten years. A small percentage of enclosures can be expected to perform more poorly than anticipated for reasons which are presently unclear but may relate to imperfections in the heat-seals that are small enough to become significant only after several years. Close control of procedures for opening and resealing the enclosures is required during access to objects if the environmental control agent is not to be exhausted by the enclosure being left open. In view of the small number of enclosures that failed to maintain expected the expected humidity and oxygen parameters it is apparent that a regular program of inspection of enclosures (once every two or three years) is required to spot faulty enclosures. One cautionary note is that the long-term stability of Escal in this application is not known. We have not observed any deterioration of its properties, but the long-term success of this storage method may well be determined by this factor.

ACKNOWLEDGMENTS

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SOURCES OF SUPPLIES

AIE610FDA Dual 24" Impulse Heat Sealer, 10mm seal width
American International Electric
2835 Pellissier Place
Whittier, CA 90601

Escal lay-flat tubing, RP-K oxygen absorber, Ageless Eye oxygen indicator tablets
Keepsafe Microclimate Systems
9 Oneida Avenue
Toronto, ON CANADA M5V 1M3

SMD-Humector HM-04 humidity indicator cards
AGM Container Controls Inc.
3526 East Fort Lowell Road
Tucson, AZ 85716

ProTek Sorb Prepackaged Silica Gel
WR Grace
7500 Grace Drive
Columbia, MD 21044

Plastazote LD45 Foam, 1x2m sheet, 1.5 cm thickness
UFP Technologies
1235 National Avenue
Addison, IL 60101

Quick Cut hot knife, sled attachment, and blades
University Products
P.O. Box 101, 517 Main St.
Holyoke, MA. 01041

Press-to-seal polythene bags, ratchet-action tube cutter for 2" PVC pipe
McMaster-Carr
600 N County Line Rd.
Elmhurst, IL 60126-2081

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