EXPERIENCES WITH FREEZING AS A METHOD OF INSECT ERADICATION IN MUSEUM COLLECTIONS WITH SPECIAL EMPHASIS ON WOODEN ARTIFACTS

ABSTRACT

The Pacific Regional Conservation Center at Bishop Museum, (PRCC), has been using a controlled cycled freeze method to treat museum collections with possible insect infestation for over four years. During this time, much has been learned about the cycled freeze process and the kinds of artifact materials and structures that are at risk during the procedure.

PRCC Senior Objects Conservator Dale Kronkright will clarify why the cycled freeze procedure is designed to provide high mortality rates in all insect life stages and review the artifact materials and structures which have been problematic as a result of the treatment. Special emphasis will be placed upon the problems of cycled freeze sterilization and wooden artifacts. A short bibliography and a reprint from PRCC's Spring '89 *Conservation Newsletter* discussing questions commonly asked about museum artifacts and the deep freeze will be available for photocopying.

Controlled cycle freezing is used at PRCC to treat both active infestations, where a live insect has been seen in association with the artifact and, as precautionary treatments where it is uncertain whether the insects are actively damaging the artifact. Freezing is also used as the principal routine treatment to insure that all collections returning to permanent storage within the museum have been treated for possible insect activity.

It is clear that cycled freeze sterilization cannot be considered safe for all materials and structures. Understanding the critical elements that make up an effective and successful cycled freeze treatment can help draw a few important guidelines for evaluating the appropriateness of cycled freezing as an acceptable method of insect eradication in collections. I will discuss some of these considerations later in this paper. But it seems wise to first discuss some of the salient issues regarding freezing as a method of insect eradication for museum collections. These include a practical overview of the evaluation and documentation process, a summary of the findings in the literature to help understand how cycled freezing kills insects and a survey of what the effects are of moisture on artifact materials and structures during cycled freeze treatments.

Documenting the collection item and the process.

PRCC considers freezing to be a conservation procedure and, as such, requires that some assessment be made of the condition of each object and the appropriateness of the procedure for the object in question. A check-off type condition report form is usually used to record the condition of the object, (or groups of objects in the case of documents and bound books), with space on the form provided for observations and Polaroid photographs or diagrams where necessary. The condition report form also indicates whether there is an active infestation, whether the insect was identified by an entomologist or whether the nature of the treatment is precautionary. The report form for cycled freeze treatment also includes a procedure check-list which assures that the freeze has been approved by a PRCC Conservator and that it has been scheduled with PRCC, that the condition has been documented and that the item was properly prepared for the cycle freeze treatment. The dates and the conditions of the freeze cycle are then filled in and signed off by the conservator with any remarks the conservator would like to make. Finally the form indicates who returned the item back into the collection and the date that return took place.

How is the cycle freeze procedure designed to kill insects?

Although scientists truly have no idea yet how many different species of insects there are, for museum cycled freeze eradication purposes, insects fall into two general categories. These two categories are of concern to us because they're based upon the insects' ability to resist or survive cold temperatures.

1. There are those insects that cannot survive extra-cellular ice formation and who must take advantage of avoidance mechanisms to survive freezing. These insects are referred to as **freeze-sensitive or freeze-susceptible** insects and make up the largest of our two groups, including most of the insects which damage or infest museum collections.

2. The second group includes all those insects which can survive extensive extra-cellular ice formation within their body tissues. There are very few of these insects and we call them **freeze-tolerant**.

Freeze-sensitive or freeze-susceptible insects

The first group, freeze-sensitive insects, avoid lethal ice formation principally by two mechanisms:

1. They take advantage of the supercooling properties of water in capillary spaces of approximately 30 microns and less. In these microcapillaries, water will not freeze until a temperature of approximately -18ºC has been reached. The capacity of water to supercool decreases as the volume of the capillaries increase and as the duration of exposure to the freezing temperatures increases.

2. Freeze-sensitive insects are also able to avoid lethal ice formation by chemically masking ice nucleating agents in extra-cellular spaces. Ice nucleating agents are those compounds around which ice could, under normal circumstances at freezing temperatures, begin to form.

Freeze-tolerant insects

The second group, freeze-tolerant insects, survive extra-cellular ice formation by a number of more sophisticated chemical means.

1. One mechanism used by freeze-tolerant insects is to synthesize ice nucleating agents to control ice formation to non-lethal extra-cellular areas, only.

2. Freeze-tolerant insects also have a sophisticated array of chemical crytoprotectants. These chemicals fill what would otherwise be lethal areas of ice formation and thereby protect the insect.

3. Freeze-tolerant insects also use a variety of anti-freeze proteins and amino acids which they mix with body fluids to prevent extra-cellular ice formation within body tissue.

4. Freeze-tolerant insects also seem to utilize sophisticated biological clocks which measure patterns in light and temperature changes and allow the insect to build its chemical arsenal to fight off the affects of freezing temperatures.

These processes in both freeze-sensitive and freeze-tolerant insects, are called **cold hardening**. It takes some time for both groups of insects to prepare for cold hardening. The generation or the formation of ice nucleating masking agents in freeze-sensitive insects and the chemical battery of ice forming and resisting chemicals in freeze-tolerant insects require several weeks. This fact alone would seem to make a cycled freeze temperature highly lethal and effective. But when we receive an artifact for cycled freeze sterilization treatment, in many cases we have no way of knowing the precise conditions which the artifact has experienced over the past two weeks. The conditions of light and/or temperature may have contributed to the instinctive preparation by the insect for freezing conditions. Therefore, the freeze cycle must find ways of eradicating even those insects which have successfully prepared for sub-freezing temperatures through freeze-sensitive and freeze-tolerant mechanisms. There are five such ways:

1. **Using temperatures below the supercooling point of water**
   One obvious step is to use temperatures which are below the supercooling point of water within microcapillaries, therefore causing lethal formation of ice in extra-cellular space. We know this super cooling point to be -18ºC or lower and so this temperature is set as the threshold upper limit for lethal use of freezing for insect eradication.

2. **Holding temperatures below the supercooling point for an extended period**
   The lethal temperature needs to be held at or near the supercooling point for periods in excess of 24 hours. This, in effect, increases the formation of ice in microcapillaries in freeze-sensitive insects and also results in lethal gradations between ice lattices and extra-cellular solutions within freeze-tolerant insects which results in removal of water from living cells. These conditions result in lethal conditions for the insects during the extended freeze.

3. **Keeping infested materials at room temperatures and normal ambient humidities prior to cycled freezing.**
   Because long periods of preparation must occur before freeze-sensitive and freeze-tolerant insects can procure chemical crytoprotectants, and because acclimations to normal room temperatures frequently result in rapid loss' of crytoprotectants, keeping the infested artifact or specimen at normal room temperatures prior to freezing can increase the lethal affects of the cycled freeze treatments.

4. **Using the ability to cold-shock some insects**
   In many species cold shock or chilling over a 4-hour period to temperatures just above the super cooling point results in mortality in egg, larvae, pupae and adult.

5. **Prevent cold-hardening response in insects by avoiding rapid or blast freezing**
   There is a phenomenon known as rapid cold hardening response which can be problematic for cycled freeze eradication of insects in museum collections. Very rapid chilling, 10 minute chilling from room temperatures to 0ºC, can allow overall survival of insects at temperatures down to as much as -10ºC. Therefore, no pre-chilling or rapid chilling should occur. To take proper advantage of the cold shock tendency in many insect species and to avoid rapid cold hardening response in other insect species, temperatures should drop within roughly a four-hour period directly from room temperature to a -18ºC without stopping out at any plateau temperatures.

The behavior of moisture at sub-freezing temperatures.

One of the concerns about freezing artifacts as a method of pest control is the effect of moisture on artifact materials and structures during cycling temperatures. Generally we would like to know:

- what happens to water in artifact materials at freezing temperatures and

- what dimensional changes might take place in an artifact when frozen and subsequently warmed inside a sealed plastic bag?

The answer to our first question is fairly straight-forward. At ambient museum relative humidities, water is not present in organic materials as a liquid. When we speak of moisture content of 6% to 12% in organic materials, what we are really talking about is adsorbed water - water which is hydrogen bonded to the amorphous molecules of the substrate material. In the case of wood, herbaceous materials or paper, we do not have water molecules next to cellulose molecules which can then freeze and form an ice lattice, therefore causing damage. We have a hydrated cellulose molecule, which does not freeze unless temperatures of less than -60ºC are achieved.

The answer to our second question is slightly more sophisticated, but every bit as elegant. Water molecules are continually making and breaking bonds with moisture adsorbent organic materials. At a stable relative humidity, the same number of bonds are being broken as are being made and the moisture content of the material remains constant. As a result, the object undergoes no dimensional changes. When thermal energy in the air around the object is removed, the capacity of the air in the bag to hold moisture decreases. Water molecules will begin to seek out other sites to bond onto. The organic material adsorbs these more abundant water molecules faster than it can release them and, subsequently, begins to undergo dimensional swelling. The rate at which these dimensional changes takes place depends upon the porosity of the material, the number of bonding sites available and the amount of water available.

Let's say, for instance, that we have a one cubic foot capacity polyethylene bag with about one pound of cotton wool sealed inside. In our example, if we drop the temperature continuously from 85ºF to -5ºF over an 8-hour period, there is an initial increase in the relative humidity within the bag of 6% in the first four hours. This begins to be adsorbed by the cotton wool and the RH is slowly brought to the ambient Hawaiian humidity of 62% within 6 hours of reaching -5ºF.

If we take our plastic bag with cotton wool and increase the temperature from -5ºF to 85ºF over a 24-hour period, this produces a 4% drop in the ambient relative humidity, mitigated by the liberation of moisture by the cotton wool. The humidity inside the bag slowly rises back to the initial relative humidity of 62% at the end of 24 to 36 hours.

What is this information telling us? It tells us that cotton wool is an effective moisture adsorbent with great porosity and many available bonding sites. Cotton within a sealed plastic bag can help mediate changes in relative humidity that result during a freezing treatment.

If the artifact material. has a slower moisture absorbancy response than cotton, because

1) it is a denser material,

2) because it has a finish or film which slows the rate of moisture regain or

3) because it has less surface area in relation to overall mass,

the rates of moisture regain and loss, (and therefore the resulting dimensional changes which the artifact will experience), can be substantially controlled.

Conditions which indicate possible problems during cycled freezing of wooden objects include:

What are some of the conditions which we should be on the look-out for when considering the suitability of a wooden object for cycled freezing?

Desiccated or partially failing glues.

These kinds of damages indicate that remaining glue-joints may be under additional stress and may fail at lower temperatures.

Craquelure or other evidence of finish deterioration or cleavage from the wood substrate.

If there is some evidence that the adhesion between the substrate and finish is failing, dimensional changes from lower temperatures, alone, may aggravate damage.

Inlays, veneers and marquetry showing mechanical damage, warpage or other distortion or tenting, cupping or other cleavage from the substrate surface.

Damage to these components may also indicate materials which are under stress from distortion or movement of adjacent materials and/or substrates. Lower temperatures may be enough to cause failure of the veneer adhesives, resulting in loss.

Tooth parts or inlays

Teeth hydrate without adverse effect during increases in relative humidity, but cannot undergo even slight dehydration during the warming part of the cycle without causing cracking and loosening

Drum heads under tension; strung instruments are generally slowly loosened.

Drum heads under tension are generally secured to materials which provide equal and opposite forces, thus holding the drum head in place. When the drum material that the head is secured to begins to move, the entire equilibrium, which may be centuries old, can be seriously altered, resulting in failure of one of the materials and damage.

Objects with checks or cracks which appear to be recent.

Where a check might be developing as a release of some internal stress which has not yet reached equilibrium with its present environment, dimensional changes due to temperature changes alone can result in damage. Old checks generally indicate the release of stresses and a state of equilibrium and tolerance on the part of the object to normal changes in temperature and relative humidity.

Warpage or other distortions which may indicate that the object is currently under odd stresses.

These conditions sometimes place materials and structures under stresses for which they were never intended. Failure of materials and structures from temperature changes alone may aggravate and compound the problem and result in damage.

Completely finished wooden objects.

Good finishes on wood prevent absorption of moisture during the rapid drop in bag air temperature, sometimes resulting in condensation of moisture within the bag once the bag reaches dew point. This condition can be fully mediated by the inclusion of cotton wool or silica gel within the bag.

Non-moisture adsorbent materials.

Many artifacts such as metal and stone do not provide a host food source for insects but can be highly desirable as a nesting and breeding sites and therefore should undergo sterilization prior to re-introducing an artifact into a permanent collection storage. It is important for these materials to have a moisture adsorbent material such as cotton wool or silica gel included within the bag airspace so that due point cannot be reached and thereby prevent moisture from forming on the inside of the bag.

What are some of the kinds of objects which we would never freeze?

Common sense suggests that the following objects and materials should never undergo cycled freezing as a method of insect eradication. These include materials and/or structures which would be damaged as a result of freezing temperatures alone.

- paintings on canvas
- Ivory
- ancient and deteriorating glass and glass components
- high fire ceramics
- waterlogged specimens and artifacts
- thick powdery and/or mat paints with relatively little binder which have been painted on wood substrates
- paintings on joined wooden panels
- objects with wax components or large wax fills. Many wax objects undergoing cycled changes in temperature can produce a polymorphism, resulting in an opaque, powdery wax formation on the surface. Wax components are often brittle and can withstand no dimensional change if they are built up upon a wood or other organic substrate

Critical factors for using cycled freezing as an insect eradication method for museum collections

We feel that there are three critical elements which are necessary for successful insect eradication using cycle freeze sterilization. They are:

1. proper preparation of the artifact
2. temperature during the freeze
3. timing of the freeze cycles

These are all crucial for successful eradication of all phases of insect life including eggs, larvae, pupae and adult.

As a result of the information we have just reviewed about the lethal effects of freezing on insects and using what we know about the behavior of water vapor and moisture adsorbent materials, the following represent the critical factors, in my experiences, with cycled freeze sterilization:

A) Keep the artifact close to a warm room temperature prior to treating.

B) Bag in a strong, airtight polyethylene bag, 3mil to 4 mil is recommended; double bagging whenever possible in 2mil plastic seems to be extra reliable.

C) Extra moisture adsorbent material, such as silica gel modules, cotton wool, cotton mattress pads, towels or cotton diapers should be added to bags, when artifacts have complex structures, finished surfaces or fragile materials, to mediate the effects of the increase and decrease in relative humidity within the bag upon freezing and subsequent warming.

D) As much excess air as possible should be evacuated from the bag. This will keep the total amount of moisture within the bag to an absolute minimum and therefore, further mediate the amount of moisture which must find moisture adsorbent bonding sites.

E) Lower the temperature of the artifact to -5ºF which is approximately -20ºC in 4 to 6 hours continuously, whenever possible.

F) Hold this temperature constant during the first and second freezing periods of 48 hours.

G) Massive or dense materials such as books and paper may not reach the lethal temperature for nearly 20 hours. These materials should be separated in smaller containers, if possible. Use of a thermo couple will also help determine how quickly materials achieve lethal temperatures.

H) Slowly allow frozen items to reach room temperature, ideally over a 24-hour period, both after the first 48-hour freeze increment and after the second 48-hour freeze increment. This can be done by turning the freezer off and allowing it to slowly come back up to room temperature or by removing the bagged specimen from a freezer into a refrigerated space before going into a room temperature space. Once the object has achieved room temperature it should be held at room temperature for a 24-hour period. Between the first and second freeze periods, the bag must remain sealed at all times.

I would like to thank the staff of the PRCC Objects Lab and administrative staff, as well as the Bishop Museum, whose committed joint effort over the past four and one-half years, this presentation represents.

Literature reviews for:
adhesives: acrylic, PYA, hide glue, nitrocellulose, wood, ivory, shell, varnishes, shellac, lacquer, musical strings.

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