# WHAT GOES UP MUST COME DOWN. . . BUT IS IT REVERSIBLE?

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Reversibility is discussed starting with the principle of reversible processes in thermodynamics, and proceeding to reversibility of treatments for wood artifacts. Special reference is made to consolidation treatments for deteriorated wood with soluble resins. Preliminary experiments showed that Acryloid B-72 can largely be removed from wood after impregnation.

## Introduction

Reversibility is of concern to every conservator, and it may not be an exaggeration to say that it is the most basic of all of the concerns that conservators have. The reasons for this concern need not be discussed here, as they are well known in the profession. In fact, the general concept is one that can be readily explained to an intelligent lay person. What is perhaps less apparent is whether reversibility is in fact an absolute and practical reality, or whether it is an ideal that must be pursued, that can be approached, but that will never actually be attained.

If we turn to the AIC Code of Ethics (1) we find under the section "Obligations to Historic and Artistic Works" the I following three sentences under the heading "Principle of Reversibility:"

"The conservator is guided by and endeavors to apply the" 'principle of reversibility' in his treatments. He should avoid the use of materials which.may become so intractable that their future removal could endanger the physical safety of the object. He should also avoid the use of techniques, the results of which cannot be undone if that should become desirable."

This language indicates rather clearly that the drafters of the Code of Ethics did not conceive of reversibility as an absolute requirement but as a guiding principle that should be followed to the extent possible.

Similarly, from the Code of Ethics of the Institute for Conservation of Cultural Materials, Australia (2):

"The techniques and materials which adversely affect or modify the object the least, and which can most easily and completely be reversed, shall always be selected when applicable. Reversibility is a goal to strive for."

The Code of Ethics of the Canadian Association of Professional Conservators (3) does not use the term reversibility. It states that:

"Similarly, the conservator shall use materials which can be removed most easily and completely. "

Conversations with conservators, on the other hand, generally tend to reflect a much more restrictive attitude, suggesting that reversibility of treatments is conceived as an absolute mandate rather than the

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goal to strive for appearing in the codes cited.

The objective of the present treatise is to discuss the reversibility of treatments for wood artifacts, with particular reference to consolidation treatments of deteriorated wood with soluble resins.

**Reversible Processes in Thermodynamics**

If we examine something as seemingly simple and basic as work (such as the lifting of a weight) being done by an expanding gas, or work being expended in the compression of a gas, we find immediately that some idealized apparatus is required. A common example is a cylinder with a weightless, frictionless piston. Using such an apparatus to perform a reversible process as defined in thermodynamics requires that not only the system itself but also its surroundings return to their initial states when completing a reversible cycle. Furthermore, any changes must be done in infinitesimally small increments so that the system never departs from equilibrium by more than an infinitesimal amount.

Since the foregoing is clearly not possible--a process carried out in infinitesimal increments would take an infinite amount of time--a reversible process can never actually be done. To quote from a standard physical chemistry text (4): "Reversible processes are, therefore, not real processes, but <u>ideal ones</u>. <u>Real processes are always irreversible</u>."

It might well be argued that the thermodynamic definition of reversibility is too restrictive. In conservation it might be sufficient, for instance, if the system itself returns to its original state so that changes in the surroundings would not have to be considered. On the other hand, practical systems in conservation problems are usually \ much more complex than the example of an expanding gas lifting a weight given above, and we have to consider the possibility that absolute reversibility cannot be attained even with a more liberal interpretation of its definition.

**Changes in Wood Moisture Content**

In the course of conservation treatments wood may be exposed to moisture, either in the form of water vapor contained in the environment or by application of liquid water; it may be exposed to gases (fumigation) or other preservatives; and it may be glued, or coated, or consolidated, to mention some of the more common procedures.

Moisture in wood can exist either as bound water -that is adsorbed within the cell wall, or as free water in the cell lumen. Changes in the amount of free water will have relatively little effect, while changes in bound water content will have significant effects on virtually all wood properties.

Suppose we place a large drop of water on the planed and sanded, but otherwise unfinished surface of dry wood. If this is done in a controlled atmosphere, and the piece of wood had been in equilibrium with these conditions, the drop of water will wet the wood at first but in due course all of the added moisture will evaporate, reestablishing equilibrium. Thus the "treatment" appears to have been reversed, but has it really?

If we look carefully, there might be a slight stain. If so, it would be because the liquid water had dissolved some of the water soluble extractives in wood and, in evaporation, brought them .to the surface and deposited them there. The extractives, also referred to as extraneous substances because they are not an integral part of wood structure, cover a wide range of chemical compounds. Their nature varies greatly from one species to the next (5). Extractives are responsible for the characteristic color of different species. Local changes in concentration of extractives will consequently also mean a change in color. The water drop "treatment" therefore may have been irreversible to the extent of any staining that has taken place.

In addition to the stain, if any, we might also notice that the area where the drop had been applied had become rough and slightly raised. This represents a recovery of "permanent" deformations, i. e., deformations that would not be recovered without the swelling that accompanied the application of liquid water, that were incurred when the surface was worked or machined. This recovery of deformation that had been frozen into the material is also an irreversible change. A cabinet maker would dampen the entire surface and then sand it lightly, but sanding is about the only practical way to remove the rough spot.

There is yet one more change that could have possibly taken place in that portion of the wood that had been wetted by the drop of water. Wood exposed to constant conditions of temperature and relative humidity will eventually reach a moisture content in equilibrium with those conditions by either adsorbing water vapor from the atmosphere or by losing moisture to it. The exact moisture content reached will be higher if equilibrium is approached from above, i.e., a high moisture content, than when it is approached from below. This phenomenon is known as sorption hysteresis (6). In our example of the water drop, we would expect no change if equilibrium of the entire piece had previously been approached from below. In contrast to the other two effects, the raised grain and the strain, any hysteresis effect would not be noticeable nor even measurable, but it is another aspect of not being able to return to precisely the original state after a treatment.

The shrinking and swelling that accompanies changes in moisture content of wood can be considered as cyclic variations about a stable average dimension, as long as there are no restraints placed in the way of this dimensional movement. With restraints, wood can be made to undergo permanent deformations (7). The classic example is the axe handle that is soaked in water to make it tight. Each time this is done, there is some compression set (permanent deformation) and subsequent shrinkage takes place from a smaller base, until no amount of soaking can make the handle tight anymore. Warping of panel paintings where the restraints arise from', unequal shrinking and swelling of face and back because of differences in coating, or exposure, or both, is a closely related phenomenon.

**Consolidation of Wood with Soluble Resins**

The gluing, coating, and consolidation of wood are treatments with distinctly different purposes, but all have in common that the material used must adhere to wood to be effective, and will penetrate into the wood structure to varying degrees. In most cases these materials are applied in the form of solutions, so that a discussion of consolidation of deteriorated wood with soluble resins can in many ways be representative of other types of treatments as well.

Preliminary experiments were initiated to investigate the reversibility of consolidation treatments of deteriorated wood with soluble resins. Douglas-fir "archaeological wood of the same type used in previous studies (8,9) was chosen for the experimental material. This wood had been in the ground for approximately 70 years in log form, and the outer layers had been found deteriorated by bacteria when the material was unearthed in 1979. The material chosen for the present experiments was taken from full diameter sections, 3 ft. long, that were left over from the same technical study (10) that had provided specimens for the studies referred to above. The sections had been stored outdoors from 1980 to the end of 1986, so that the wood had been dried and exposed to weathering for that period. From a total of over 60 sections, eight were chosen for study. Of these, four were chosen to represent pieces of relatively sound appearance and the other four represented the more severely deteriorated material of the lot.

Specimens 2 inches long, 1 inch wide, and approximately 1/4 inch thick were sawn from the log sections in such a way that the undisturbed surface of the latter became one of the wide faces of the specimen. The specimens were placed into an environmental room controlled to 66% relative humidity and 70°F, which is expected to produce a nominal equilibrium moisture content in wood of 12%.

For the initial set of experiments, four groups of 8 specimens each were chosen. Each group contained one specimen from each of the original 8 log sections. A set of specimens similar to those used is shown in Fig. 1. Two sets were used for determining moisture content, one was used to determine extractive content, and the fourth was used for impregnation with consolidant which was subsequently extracted again with solvent.

There are two components in wood, namely the adsorbed \ water and the extractives, that make it impossible to carry out a simple determination of the amount of consolidant retained after initial treatment and any residual consolidant remaining after an attempt at removal. This is particularly true when the solvent used in the consolidant is miscible with water, as it will tend to extract not only the extractives soluble in it but also the adsorbed water. Such extraction is likely to occur to only a limited extent during consolidation treatment, but will be a much more significant factor when the consolidant is being extracted. Determination of moisture content and extractive content are therefore best done with duplicate specimens. These also have to be done separately from each other, because the only method for complete removal of adsorbed water is by oven-drying, which however might drive off volatile extractives or might cause chemical alteration, such as polymerization, of the extractives that remain.

Specimens for moisture content determination were weighed, dried for 24 hrs. in an oven at 103°C, cooled in c-weighing bottles placed inside a desiccator containing silica gel, and weighed again. They were then coated with hot wax and the oven-dry volume was determined by the immersion method, so that the specific gravity could be calculated.

Specimens for extractive content determination were weighed first, and then extracted with acetone in a Soxhlet apparatus. In this apparatus the extractor body in which the specimens are located is filled dropwise with freshly distilled solvent until it is full, and the solvent is then siphoned back into the solvent reservoir. One cycle of filling and siphoning required 75 minutes. The extraction was continued for 4 days, or approximately 77 cycles. Following the extraction, the specimens were dried and reconditioned in the same environmental room. The reconditioning was continued for 16 days but equilibrium appeared to have been reached after about 5 to 7 days. Specimens were then weighed and subsequently their moisture content and specific gravity were determined as described above.

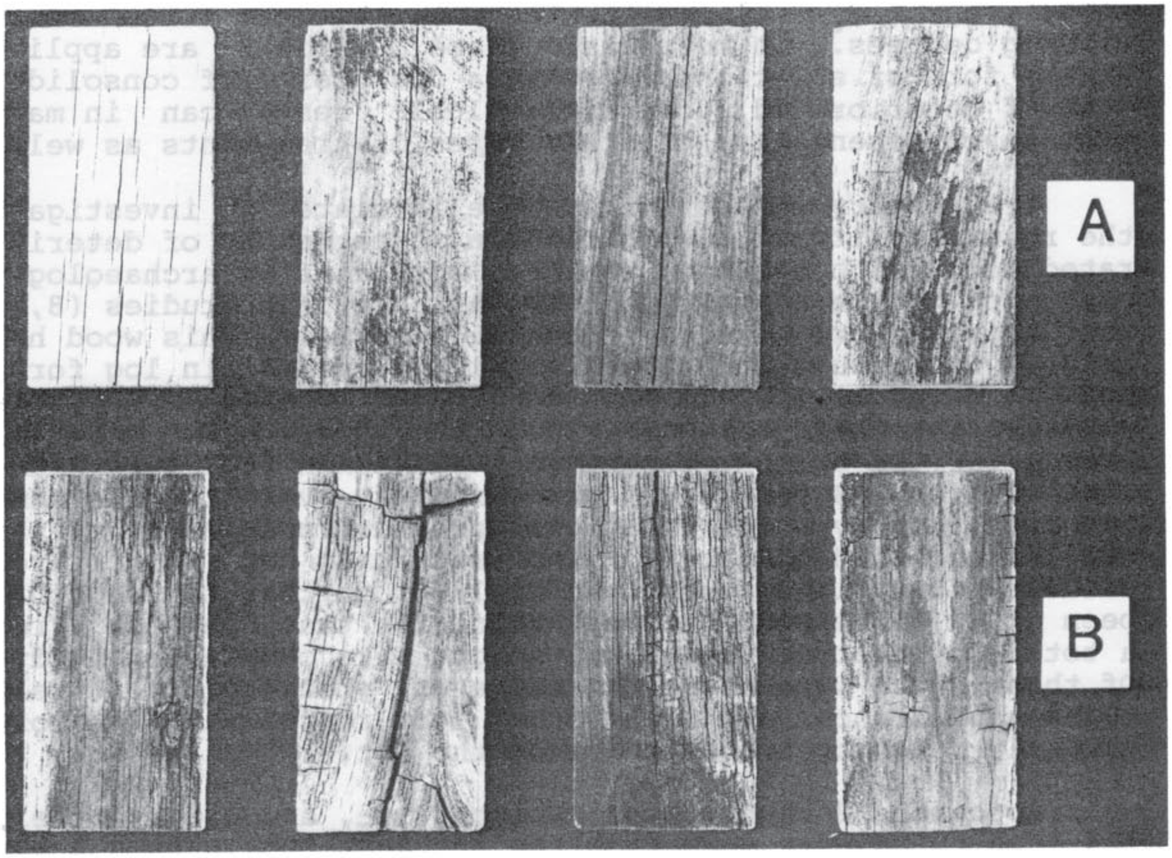

**Fig. 1. Typical set of specimens: deterioration less than average (A), and more than average (B).**

Specimens treated with consolidant were weighed first and then soaked for 2 hours in a 15% (weight basis) solution of Acryloid B72 in acetone. The dish with consolidant solution and the submerged specimens was then placed into a desiccator and a vacuum was drawn for 4 minutes, reaching a level of 66.5 cm Hg. The system was then closed and left for another 15 minutes. Vacuum was then released and the specimens were allowed to soak in the solution for another 2 hrs. The specimens were then blotted, allowed to dry, and reconditioned in the environmental room described above. After 11 days, when periodic weighings had indicated that the samples had reached equilibrium, they were weighed again and then placed into the Soxhlet apparatus for extraction. Acetone was used as the solvent, and the extraction was carried out for 4 days using the same procedure already described. The specimens were then reconditioned once again, followed by f determination of final moisture content and specific gravity.

**Results of Preliminary Experiments**

In presenting the results, moisture content is expressed as percentage of moisture based on oven-dry weight, as is customary in wood science. Extractive content is also a percentage based on oven-dry weight. Retention of consolidant after treatment and residual consolidant after extraction, however, are based on the weight of air-dry wood, i.e., wood at a nominal equilibrium moisture content of 12%.

The average moisture content of two sets of specimens (16 samples) was found to be 13.7%, and the specific gravity was 0.40. The moisture content was higher than the nominal equilibrium moisture content of 12%, but some variability is to be expected in wood as a biological material. Coast type Douglas-fir is expected to have an average specific gravity based on oven-dry weight and volume of 0.51 (11), which indicates that there has been a weight loss of better than 20% due to deterioration in the sample material. The average specific gravity from both sets calculated separately for the samples classified as having moderate deterioration was 0.43, while those with more severe damage had a value of only 0.36, which supports the visual observations.

The content of acetone extractives is shown in Table 1. Acetone is not one of the standard solvents in extractive determinations, which makes comparisons difficult. The average value of 0.61% is not large, considering Fengel and Grosser in their extensive compilation of extractive data (12) list the extractive content of Douglas-fir sapwood at 5.0% in hot water, 1.6% in alcohol/benzene, and 0.4% in ether. It is quite possible, however, that some of the extractives were leached out by groundwater during the underground exposure. The moisture content after extraction, at an average of 12.5%, is lower than the original moisture content of 13.7%. There are two possible explanations, one being the effect of sorption hysteresis, since acetone will remove not only extractives but also water from wood, and thus equilibrium was approached from below after extraction. Another factor that needs to be taken into account is that wood can adsorb small amounts of polar organic solvents, that are capable of swelling wood, so tenaciously that they cannot even be removed by drying at 105°C (13). Acetone falls into that group, and attachment of acetone molecules to sorption sites otherwise available to water would thus lower the measured moisture content.

**Table 1. Extractive content, and moisture content and specific gravity after extraction**

| Section | Extractive | M.C. | Specific | |
|---|---|---|---|---|
| Deterioration | No. | Content. | (%) | Gravity |
| Moderate | 16E | -0.11* | 2.8 | 0.61 |
| Moderate | 17 | 0.53 | 12.5 | 0.40 |
| Moderate | 18 | 0.36 | 12.2 | 0.63 |
| Moderate | 19 | 0.86 | 12.4 | 0.44 |
| Severe | 3A | 1.02 | 11.5 | 0.35 |
| Severe | 20 | 0.65 | 13.0 | 0.28 |
| Severe | 21 | 0.96 | 12.8 | 0.37 |
| Severe | 22 | 0.65 | 12.7 | 0.37 |
| | Average | 0.61 | 12.5 | 0.43 |

*Specimen showed some weight gain.

Specimens retained an average of 23.6% of Acryloid B72 upon consolidation, as shown in Table 2. Amounts were larger for the more severely degraded specimens, and the specimen with the lowest specific gravity (0.29) also retained the largest amount of consolidant (40.4%). Values of residual consolidant content after extraction are positive for only two of the eight specimens, which were also the specimens with the lowest amount of original consolidant retention. This suggests that these specimens were less permeable than the others, and that a longer period of extraction might have removed more of the consolidant. Moisture content after extraction was less than 13% for this group of specimens also, and the same comments made above regarding hysteresis and adsorption of solvent apply. Adsorption of residual consolidant is not likely, as its molecules are too large to be able to penetrate into the cell wall.

**Table 2. Consolidant retention, and residual consolidant, moisture content and specific gravity after extraction.**

| Section Deterioration | Consolidant No. | Ret. (%) | Residual Cons.(%) | M.C. (%) | Spec. Grav. |
|---|---|---|---|---|---|
| Moderate | 16E | 18.2 | 0.29 | 13.0 | 0.50 |
| Moderate | 17 | 22.2 | -0.07 | 12.9 | 0.44 |
| Moderate | 18 | 18.3 | -0.06 | 13.2 | 0.49 |
| Moderate | 19 | 14.2 | 1.03 | 13.2 | 0.47 |
| Severe | 3A | 25.8 | -1.26 | 11.9 | 0.38 |
| Severe | 20 | 40.4 | -0.11 | 12.7 | 0.28 |
| Severe | 21 | 23.8 | -0.33 | 13.3 | 0.38 |
| Severe | 22 | 25.8 | -0.32 | 12.8 | 0.38 |
| | Average | 23.6 | -0.10 | 12.9 | 0.42 |

Although there is an average weight loss of 0.1% after extraction of consolidant, we would have expected a loss of 0.61% due to loss of extractives (Table 1). The difference may be presumed to be residual consolidant retained inside wood, in the amount of 0.5%. A similar amount of residual was reported by Nakhla; (14) for the same resin but different solvent and different methods. However, when examining the data for residual consolidant it must be considered that they are based on taking small differences of large measurement values that are subject to a large degree of natural variability.

## Conclusion

In examining some of the factors that influence reversibility of conservation treatments applied to wood, we find good reason for the language in the AIC Code of Ethics describing reversibility as something to strive for to the extent possible. Speaking of soluble synthetic resins as consolidants, there is evidence that they can be largely removed again, but unless extreme measures are taken some consolidant may remain. Small residues may be considered unacceptable (15), and then again their effects may not be noticeable. As has been pointed out, wood undergoing any type of treatment is likely to undergo many subtle changes that are not strictly reversible, but may have little or no practical effect.

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