Zeolite Molecular Traps and Their Use in Preventative Conservation

By Siegfried Rempel

INTRODUCTION:

Evolution Of Conservation Products

The evolution of archival quality paperboard housings is relatively recent. In the 1960's housing materials described as archival included acid pulped fibers with high residual lignin content and no alkaline reserve. We now know that these pulping methods and the presence of non-cellulosic residuals create an unstable environment for both the housing and the materials stored within them.

In 1979-80 the requirements for archival paperboard began to change, due in large part to specification changes required by the Library of Congress. An alkaline reserve of 3-5% was added throughout the pulped paperboard. This was added to neutralize the deterioration by-products being given off by the storage box and the materials stored in the box.

In 1980 the term "archival" was specified as being a material which was lignin-free, sulfur-free[1], alkaline pulped and containing an alkaline reserve. This functional, archival quality storage paperboard provided a high quality, safe storage environment for the materials contained within it. Because of further conservation concern for photographic materials, these products were also supplied in an unbuffered form[2].

In 1992 Conservation Resources International[3] began to market a new configuration of conservation quality product: paperboard (lignin-free, sulfur-free, alkaline pulped, alkaline reserve) with an additional element -- molecular traps, under the trade name MicroChamber® Products. The information associated with this product line is discussed in depth in the CRI catalogue and may already be familiar to many conservators.

In 1995 Bainbridge[4] introduced Artcare™, a matboard and mountboard housing system incorporating the same MicroChamber technology to provide preservation matting and mounting products for framing and museum professionals. This matboard product was also a traditional, quality conservation paperboard (high alpha cellulose, lignin-free, alkaline pulped, alkaline reserve) with molecular traps added to the fibers.

The need for molecular traps in paperboard to deal with both air-borne pollutant gases and the by-products of deterioration was reinforced by the investigative work completed by scientists working for the National Archives and Records Administration in Washington.

Acid Gases and the Alkaline Reserve in Paperboards

Guttman and Jewett (1993) examined the diffusion of an acid pollutant gas (sulfur dioxide) through paperboard as a part of a National Archives and Records Administration study and determined that significant amounts of the pollutant gas can enter the storage environment within the storage container. Guttman & Jewett's results and work published by Passaglia (1987), examining the need for protection within storage containers against pollutant gases in the environment, indicate that the alkaline buffer systems incorporated within the paperboard do not effectively neutralize acidic pollutant gases.

The presence of other pollutant gases, atmospheric, environmental, or the by-products of deterioration, in the collection storage environment must also be considered. Alkaline gases and oxidizing gases can also diffuse through the paperboard in a manner similar to the acid pollutant gas studies, and the presence of an alkaline reserve obviously would not contribute to the neutralization or elimination of these types of deterioration agent.

Pollutant Sources

Pollutant sources constitute the primary deterioration agents which are being targeted by the molecular trap technology. Pollutants are increasing in concentration in our environments, and therefore have become a preventative conservation priority.

Pollutant sources include external environments with automobile exhaust, primarily nitrogen oxides, and smoke stack emissions, primarily sulfur dioxide from burning fossil fuels. These pollutants are known to easily penetrate to a building's interior except in situations where the air handling system has been designed to remove pollutants. This type of mechanical system tends to be limited to larger galleries and museums.

Internal pollutant sources include those related to occupancy of internal spaces, for example cleaning solutions, construction, renovation and painting, etc. Added to this are the artifact's internal deterioration by-products as well as the deterioration by-products of the display case or storage cabinets (Grzywacs & Tennent 1994). These pollutants need to be considered since they tend to build-up within the storage environment, creating a micro-environment which may accelerate the rate of deterioration of the artifacts contained there.

In terms of renovation and construction work, the deterioration problems associated with oxidizing gases (peroxides) given off by oil-based paint has been documented by Feldman (1981) for photographic images. Wood materials used in construction emit aldehydes (formaldehyde) and acid gases including formic acid.

Since research has shown that acid gases will not be neutralized by the alkaline reserve in the paperboard it seems reasonable to assume that other pollutant gases would also pass through the protective storage enclosures unaltered. The isolation and/or neutralization of these pollutants is required to reduce their deterioration effects on stored and displayed artifacts. The use of molecular traps incorporated within the storage container represents one solution to this problem.

MOLECULAR TRAPS:

Zeolites and Activated Carbon

Molecular trap is a general descriptive term for specific chemical entities such as zeolites or activated carbon. Both of these have been used to provide functions based on filtration, refining, and/or separation of chemical mixtures. The use of activated carbon, for example, to filter contaminants from gases and liquids is well established. Museums such as the Gene Autry Western Heritage Museum in Los Angeles[5], for example, use activated carbon in the mechanical air handling systems to remove atmospheric pollutants in make-up air and to remove pollutants in the recirculation air portion of the museum's general environment.

This ability of both activated carbon and zeolites to trap chemical species provides the basis for an application of molecular traps in preventative conservation. Activated carbon is an inert, porous graphite described as "graphite plates". The presence of imperfections on the plates' structure leads to the formation of a rigid skeletal structure which can comprise 75% or more of the total volume of the voids in the activated carbon. The voids are adsorption centers using primarily London dispersion forces (a Van der Waal's force) to hold the trapped entities in place (Hollinger, 1994).

Zeolite molecular traps are microporous crystalline aluminosilicate structures and provide selective molecular trapping based on size and polar properties (Dyer, 1988). They generally act as adsorbents for molecules small enough to pass into their internal cavities. The trapped molecules are held in the cavities by physical (physisorption) and chemical (chemisorption) bonding (Hollinger, 1994). The most significant point is that the zeolites' interior cavity can be modified during fabrication to target molecules of a particular size and polarity.

The crystalline nature of zeolite traps provides the inert, non-reactive character required for use in close proximity to artifacts, while providing a functional role in trapping various molecular species, in particular, undesirable gaseous chemical species, present in the collection environment.

The advantages of zeolites over activated carbon include the fact that they can be engineered to target specific sized chemical species and that they can be used in situations where the activated carbon cannot because of its color or handling problems[6].

Zeolite Characterization

Zeolites are naturally occurring aluminosilicate minerals with three-dimensional structures based on $[SiO_4]^{4-}$ and $[AlO_4]^{4-}$ polyhedra, (figure 1). These polyhedra are linked by their corners to produce an open structural form which has internal cavities in which molecules of various sizes can be trapped. These internal voids, engineered to have specific opening size ranges, trap and hold a variety of molecules which enter the structural matrix, (figure 2).

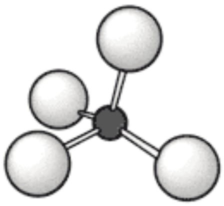

Figure 1.

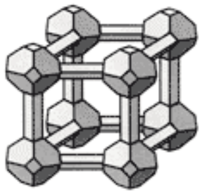

Figure 2.

Natural occurring mineral zeolites were first identified by Cronstedt, a Swedish mineralogist in 1756. In 1862 Deville produced a synthetic zeolite (levynite) from a mixture of potassium silicate mixed with sodium aluminate heated in a closed test tube. Studies on natural zeolites in 1925 established their molecular sieve attributes. In 1949 Union Carbide began the synthetic production of zeolites.

Synthetic zeolite synthesis is based on crystallization from a gel mixture of silica and alumina mixed in water under elevated pH conditions. The exact zeolite composition is determined by the ratio of $SiO_2:Al_2O_2$ in the gel mixture, the concentration of the components, the temperature and pressure under which crystallization occurs, and the time period over which the process takes place.

The attractiveness of engineering a zeolite lies in the ability to obtain specific properties in the zeolite including water repulsion, optimum internal cavity size as well as a number of other physical properties. Conservation Resources International presently markets a line of products utilizing molecular traps (both activated carbon and synthetic, acid resistant, hydrophobic zeolites) cast into a paper matrix containing alkaline buffers which is subsequently fabricated into MicroChamber conservation housing materials.

The acid resistance of the zeolite is important since acid pollutant gases and acidic by-products are anticipated as component parts of the collection storage environment. The hydrophobic properties are important to prevent the internal cavity sites from filling up with water molecules present in the storage environment, reducing the number of sites available to trap pollutant molecules.

Structural Nature of the Molecular Trap Paperboard

The scanning electron microscope images (figures 3a and b) illustrate the physical relationship between the paper fibers, the alkaline reserve and the molecular traps (zeolites) at various magnifications.

Figure 3a. The scanning electron microscope shows the orderly array of paper fibres and crystals of buffers and zeolites.

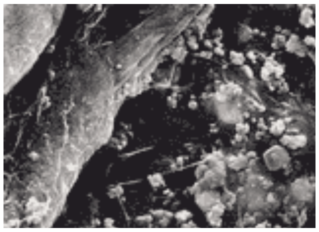

Figure 3b. Greater magnification reveals the larger cubic crystals (buffers) and the smaller zeolite molecular traps.

Research by Passaglia (1987) and Guttman & Jewett (1993) indicate that alkaline buffers are incapable of rendering acid gases inactive but they are required to neutralize the free acids generated within the paperboard, particularly as deterioration by-products. The alkaline buffer also provides a supporting role for the zeolites in the case when acid gases associated with the molecular traps react with water moisture in the paperboard to form an acid. This acid, should it become free, will be neutralized upon contact with the alkaline buffer present within the paperboard. The presence of both the alkaline buffer and the molecular traps provides a solution to the acid components which might be present within the paperboard.

The relative size of the zeolites in the paper matrix is small compared to the alkaline buffers, figure 3b. The large number of traps in the paperboard however, results in a very high efficiency in trapping molecules. Each zeolite crystal has hundreds or thousands of adsorption sites available along each crystal face. Each of these sites can trap a molecule thereby providing a very high adsorption capacity. Dyer (1988) provides very high magnification images of zeolite crystal faces which illustrate these adsorption sites.

Empirically this high level of adsorbency[7] is illustrated in figure 4, in which two bell jars have been filled with a standard quality paperboard (no molecular traps) and a MicroChamber storage paperboard. The photo on the left shows the two bell jars, MicroChamber on the left (gray color), standard paperboard in the right bell jar (white color) and a concentration of nitrogen oxides (gold yellow) in the bottom of the bell jars. The left photo represents the start of the experiment.

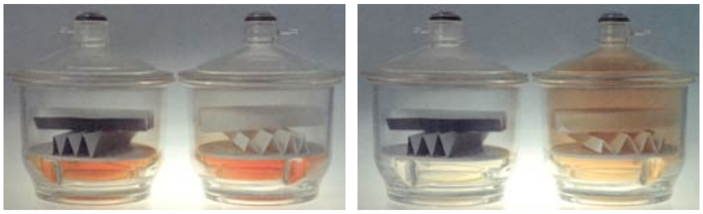

Figure 4. Left, before testing. Right, after one hour, no gas cloud formed in the MicroChamber jar.

The second photo, on the right, represents the same two bell jars after one hour. The bell jar on the left containing the MicroChamber (molecular traps) has eliminated the yellow cloud of pollutant nitrogen oxides while the bell jar on the right containing the standard paperboard product has not. The yellow pollutant gas is clearly visible within the bell jar and measured 3600ppm nitrogen oxides. When cold extraction pH measurements were made on the two board samples, the MicroChamber, after adsorbing the pollutant gas, tested at pH 6.0 while the traditional conservation quality paperboard read pH 2.4.

There is one other element in the paperboard's fabrication which has been modified: the porosity of the paperboard has been altered to produce a less porous material. The goal is to reduce the number of open pathways through which a pollutant gas molecule can traverse the board to reach the collection materials within the housing. Reduction of the porosity increases the probability of a pollutant being trapped in the paperboard. The reduced number of pathways also represents an increased tortuosity of the pathways, and a reduction in the number of pollutant molecules that can reach the artifacts being stored or displayed.

TESTING PROGRAMS

The tests that have been conducted to date have been completed by several different, independent testing facilities, and have been undertaken both by clients using the products and the manufacturers. As a result, the data being presented here lacks the consistency that would be present in work completed by a single researcher. It should also be noted that the testing configurations and procedures being presented include empirical work which is presently being re-examined in a more rigorous program.

The test configurations include the following groups: tests on molecular traps, activated carbon and selected zeolites, and calcium carbonate; tests with paper-based materials; tests with photographic materials, and tests with animation cells.

Tests With Molecular Traps/Alkaline Buffer

The laboratory test set-up consists of a gas generation station with a rubber tube delivery to a Drager tube, which in turn is attached to a pump device that assists in drawing the test gas through the Drager tube. Drager tube gas detectors are available to provide concentration readings of a number of different chemical gases. The molecular traps and alkaline buffer to be tested were packed into a glass tube which was then inserted in front of and connected to the Drager tube.

The first test sequence tested five molecular traps (activated carbon and four zeolites) and calcium carbonate against three acid gases (sulfur dioxide, nitrogen oxides, and acetic acid). The test gas was passed over each of the molecular traps or the calcium carbonate packed into the glass tube connected to a Drager tube. Table 1 contains the results of this preliminary test sequence and is given as parts per million of the acid test gas which passed through the Drager tube (i.e. was not adsorbed by the molecular trap or alkali being tested).

Table 1	**Test Gas Passed in Parts Per Million (ppm)**		
Absorbent	Sulfur Dioxide	Nitrogen Oxides	Acetic Acid
Calcium Carbonate	200	250	4000
Molecular Trap #1 (Activated Charcoal)	0	0	0
Molecular Trap #2 (Zeolite)	70	100	15
Molecular Trap #2 (Zeolite)	10	0	20
Molecular Trap #2 (Zeolite)	30	0	0
Molecular Trap #5 (Zeolite SPZ)	0	0	0

The test results indicate that the molecular traps adsorb varying amounts of the acid test gases based on the trap's specificity for these gas molecules. Of the five molecular traps tested, two of the traps are very efficient at isolating all three acid gases tested. The calcium carbonate, representing the alkaline reserve in paperboard, allowed significant concentrations of all three acid gases to reach the Drager tubes, without effectively neutralizing them. The data reinforces the observation cited in the literature that acid gases are not effectively neutralized by alkaline buffers used as the alkaline reserve in archival paperboards.

The second test sequence used the most adsorbent zeolite (identified as SPZ) from the previous test and calcium carbonate against six pollutant gases which could be found in collection environments. These six gases again included the sulfur dioxide, nitrogen dioxide, and acetic acid tested in the initial sequence as well as ammonia, carbon disulfide, and formaldehyde. Again the test results (table 2) indicate that the zeolite adsorbs significantly more of these six pollutants gases than the calcium carbonate.

Table 2	**Test Gas Passed in Parts Per Million (ppm)**					
Absorbent	Sulfur Dioxide	Nitrogen Oxides	Acetic Acid	Ammonia	Carbon Disulfide	Formaldehyde
Calcium Carbonate	200	250	4000	80	95	50
Zeolite SPZ	0	0	0	0	0	0

For the balance of the testing program the molecular traps were tested as paperboard samples with the molecular traps incorporated into the paper matrix, thus emulating the storage environment configuration in which the product is used.

Tests With Paper-Based Materials

The tests conducted with paper-based materials included a number of different materials tested in several different test regimes. In one test a comic book (newsprint) and an alkaline paper were dry oven aged, enclosed in conservation quality paper storage enclosures or storage enclosures with molecular traps. The second test involved taking a lithographic print, one half matted and framed in matboard made with the molecular trap (zeolite and alkaline reserve) and the other half in a museum (conservation) quality matboard (alkaline reserve but no molecular trap), and placing them both into a concentrated pollutant environment.

Comic Book and Alkaline Papers

This test sequence examined the protection provided by having a conservation quality enclosure with molecular traps versus a traditional, conservation quality enclosure. Both papers were also tested without the use of enclosures. The three groups of samples were dry oven aged under TAPPI test conditions and the degree of deterioration determined using fold endurance testing also under TAPPI conditions.

The data is presented as the Degree of Deterioration (table 3) based on a statistical treatment of the fold endurance data. There is an anomaly in the data. The buffered alkaline paper aged in the molecular trap (MicroChamber) enclosure had an apparent reduction in the Degree of Deterioration. This situation is unlikely, as the paper would not be expected to become stronger after being subjected to a deteriorative test such as dry oven aging. The value for this entry is considered to be zero, no change.

Table 3	Degree of Deterioration	
Dry Oven Aging	Buffered Alkaline Paper	Comic on Newsprint
Aged Without Enclosure	7.6	23.3
Aged With Conservation (Buffered) Enclosure	6.0	20.0
Aged With Molecular Trap (MicroChamber) Enclosure	-1.5	1.9

The results of testing these two papers, a poor quality newsprint comic and a high quality alkaline copy paper, show that the protection afforded by the molecular traps is significantly greater when the enclosures include molecular traps. The newsprint samples showed greater overall deterioration than the alkaline paper due in large part to the large number of non-cellulosic materials in the paper. These non-cellulosics increase the volume of deterioration by-products during the aging test. The ability of molecular traps to reduce the degree of deterioration is apparent when comparisons are made with the paper samples aged in the conservation quality enclosures (no molecular traps). In fact, the protection provided by the conservation quality enclosure shows only a very small degree of protection when compared to the control where no enclosures were used.

Tests With A Lithographic Print

This test sequence examined the protection afforded by the use of Alphamat/Alphamount Artcare matboard (zeolite incorporated paperboard) when a lithographic print, matted and framed (metal frame) in a typical frame assembly, was subjected to an elevated pollution gas test environment. The print was cut into two sections, one matted and framed with Artcare (zeolite molecular traps) and the other with a conservation quality rag matboard (no molecular traps). The glazing was glass. The test environment was made up of nitrogen oxides, a mixture of both nitrogen dioxide and nitrogen oxide, at 1000ppm.

Figure 5

The test chamber was charged each day, the total test period was 286 hours and both samples were tested together at the same time, in the same chamber.[8] The analysis was limited to a visual assessment of the changes in the image's appearance; the print is shown reassembled in figure 5. The half on the right, matted and framed with the Artcare (zeolite molecular traps) shows virtually no image deterioration. The half on the left, matted and framed with the traditional conservation quality rag matboard shows considerable image degradation. The ability of the zeolite based matboard to protect the matted object was significantly greater than that which could be obtained from a conservation quality matboard which did not have molecular traps included within the paperboard.

TESTS WITH PHOTOGRAPHIC MATERIAL

Black and White Film

Black and white photographic film was tested in an oxidizing test environment of hydrogen peroxide. The test was an incubation test based on ANSI IT9.15, 2000ppm hydrogen peroxide for 18 hours at 50oC and 80%RH. Three paperboard products were tested in this oxidizing environment using a black and white photographic film as the test monitor. The test chamber was a bell jar in which the samples were placed. The samples were made up of a piece of glass and the photographic negative in contact with the test paperboard, with the entire assembly taped together along the outside edges.

The three test paperboards were a lignin-free, sulfur-free, neutral pH, non-buffered conservation paperboard typically used with gelatin photographic materials; a lignin-free, sulfur-free, alkaline buffered conservation paperboard; a lignin-free, sulfur-free alkaline buffered conservation paperboard with molecular traps (zeolites and activated carbon).

The test results included densitometric readings of the density changes in the different negatives tested with the three paperboards (Hollinger and Vine, 1993). In the tests the unbuffered and buffered paperboards failed to protect the silver-based image and the characteristic yellow color of colloidal silver was observed after reprocessing the film. This form of damage is documented in the literature as redox blemishes and has been investigated by researchers examining silver microfilm deterioration (Henn & Wiest, 1963; McCamy & Pope, 1970).

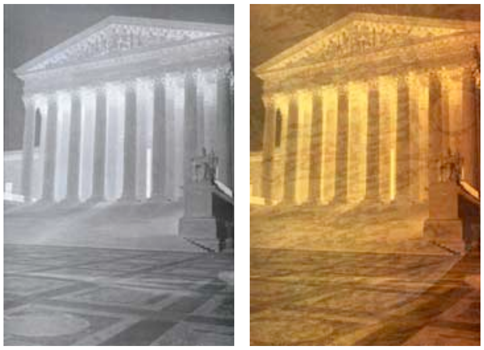

Figure 6

The visual differences in the deterioration of the test negatives is clearly evident in figure 6. The negative on the left was tested with the molecular trap paperboard (MicroChamber) and shows virtually no visual or measurable deterioration. The negative on the right was tested with the non-buffered conservation paperboard typically used with silver gelatin photographic materials. The degree of deterioration of this sample is extensive, both visually and densitometricly. The traditional quality conservation paperboards recommended for housing photographic materials failed to provide protection against peroxide oxidizing environments while paperboard with molecular traps prevented image degradation by the hydrogen peroxide.

Color Prints

Color photographic images are composed of dyes rather than pigments or metallic silver, and as such they display a greater sensitivity to chemical deterioration than lithographic prints or silver gelatin images. The pollutant gas tests conducted on conventional color chromogenic prints included a mixture of acetic acid and nitrogen oxides. Again the color prints were matted and framed (framing tape) using either Artcare matboard (zeolite incorporated paperboard), Alphamat matboard (no molecular traps) or a conservation quality rag matboard (no molecular traps).

Figure 7

All three paperboards were subjected to a pollutant gas mixture of acetic acid and nitrogen oxides at 1000ppm. The test chamber was charged each day, the total test period was 253 hours and all three samples were tested together in the same chamber. Again, the test was limited to a visual assessment of the changes in the image's stability and the print is shown re-assembled in figure 7. The middle section, matted and framed with the Artcare (zeolite molecular traps) shows virtually no image deterioration. The top section, matted and framed with the traditional conservation quality rag matboard shows considerable image degradation as does the lower section which was the Alphamat matboard (no molecular traps). The ability of the zeolite based matboard to protect the matted color photographic print was significantly greater than that which could be obtained from either of the two conservation quality matboards which did not have molecular traps in the paperboard.

Tests With Animation Cels

Animation cels have become collectible materials and they can often be seen as matted and framed display art. The relative stabilities of the colors in the cells and their safe display in pollutant environments is presently being resolved by conservators and collectors. The cels themselves represent a transitory material in the creation of an animated film; the colors used were chosen for vibrancy rather than stability. The fact that these materials are now being collected and placed on display prompted their inclusion in the testing sequences.

This test examined the protection afforded by the use of Artcare matboard/mountboard (zeolite incorporated paperboard) with an animation cel cut in two, matted and framed (metal frame), and subjected to an elevated level pollution gas test environment. One half of the Aladdin cel was matted and framed with Artcare (zeolite molecular traps) and the other half with a conservation quality rag matboard without molecular traps. The glazing was glass. The test environment was made up of nitrogen oxides, both nitrogen dioxide and nitrogen oxide, at 1000pm. The test chamber was charged each day, the total test period was 200 hours and both samples were tested together in the same chamber at the same time.

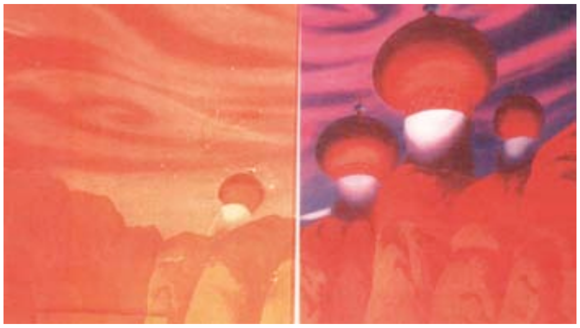

Figure 8

Again the test was limited to a visual assessment of the changes in the image's color stability and the print is shown re-assembled in figure 8. The half on the right, matted and framed with the Artcare (zeolite molecular traps) shows virtually no image deterioration. The half on the left, matted and framed with the traditional conservation quality rag matboard shows extensive image color degradation. Again the ability of the zeolite based Artcare to protect the matted object was significantly greater than that which could be obtained from a conservation quality matboard which did not include molecular traps.

SUMMARY

The use of molecular traps in storage and display environments can increase the preservation profile of the collections. At the present time two paperboard configurations are on the market in the form of storage containers and papers under the MicroChamber trade name and in the form of matboard and mountboard under the Artcare trade name.

The tests and data presented here represent information taken from a number of different sources. At the present time a rigorous testing program is being conducted to provide data (and visual examples) of the functionality of using molecular traps in collection environments to improve a collection's preservation profile. This data will be presented once the testing program has been completed.

CONCLUSIONS

The addition of molecular traps to paperboards used in the storage and display of collection materials provides an improved level of protection from environmental pollutants. The effects of these environmental pollutants, the by-products of deterioration, the results of industrial/automobile emissions or the solvents, paints or other materials which release pollutants, can be moderated by using storage and display paperboards with molecular traps.

The ability of these molecular trap product configurations, generally in a paper matrix but not limited to paper, to trap pollutant gases provides pro-active protection for collections[9]. The traps can moderate the effects of pollutant gases whatever their source, whether they result from industrial/automotive emissions, the use of solvents or paints, or are the by-products of deterioration. In the case of objects or materials which display self-destructive elements associated with inherent vice, these housing materials can provide an improved preservation profile. By effectively sequestering the artifacts' destructive deterioration by-products, the traps reduce the rate of deterioration of the artifacts.

The preventative conservation profile of collections can now be enhanced by using preservation housings which continue to function in the traditional manner, providing physical security for the contained collection materials while also providing an environment in which pollutant gases and by-products of deterioration are trapped, preventing their interaction with the collection.

Notes

1 Sulfur-free is specified for silver artifacts and for silver-based photographic materials due to possible sulfiding of the image.

2 The issue of alkaline materials in contact with gelatin photographic materials, or other photographic images which are alkaline sensitive, was resolved by requesting producers of conservation paperboards to provide an unbuffered product. The issue of alkaline buffers and silver gelatin papers is now be examined by conservation scientists to resolve compatibility issues.

3 Conservation Resources International is located at 8000H Forbes Place, Springfield, Virginia, 22151 and can be reached at (800) 634-6932, fax (703) 321-0629.

4 Information on Artcare can be obtained from Nielsen & Bainbridge, Marketing Department, 40 Eisenhower Dr., Paramus, N.J. 07652, (800) 927-8227.

5 Contact at the museum is Oren L. Gray, Facilities Manager, (213) 667-2000 x207.

6 The use of activated carbon in a storage box is not a problem since it is embedded into the paper core but it could not be used in a matboard since the bevel edge would show a gray line in the ply laminates and could also smear if touched during handling.

7 Tests presently underway are designed to determine the saturation point for these paperboard products containing molecular traps and this information will be released once the testing is completed.

8 While the test chamber was set up to provide 1000ppm of the pollutant gases, it required recharging every day to maintain the 1000ppm concentration. The actual pollutant gas levels were considered to be somewhat less than 1000ppm.

9 The Bainbridge Artcare products are also available from local suppliers in sheet form.

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