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AN ART CONSERVATION EQUIPMENT COMPANY

EQUIPMENT, SUPPLIES, AND SERVICES FOR INSTITUTIONS AND INDIVIDUALS

HXTAL NYL-1

MuseuM Services Corporation are pleased to be able to offer the first epoxy adhesive made specifically for conservation uses, not borrowed from another field. HXTAL NYL-1 is of exceptionally light color and yellows the least upon weathering of any epoxy known to us. The primary use for HXTAL is glass and china mending, but many other uses for such a low-colored epoxy resin have been found, for example HXTAL makes an excellent coating resin and can be air brushed on quite nicely from aromatic or ketone solvents. Used in the manner, HXTAL is a superb china glaze. This low viscosity, hard, colorless, glass-like epoxy resin also adheres strongly to metals, wood, ivory and marble.

This exciting epoxy cures in about 1 week at 75 degrees F or in 2 days at 95 degrees F. Cure times can be cut sharply by pre-heating the mix to 100 degrees F for 15 to 20 minutes before use, and for an even faster cure, immediately after mixing raise the temperature to 140 degrees F - 170 degrees F. for 1 to 2 minutes. After cooling, such pre-heated resin has a viscosity similar to the viscosity of the epoxies commonly sold in tubes, and the cure time is just a few minutes. The cure time can be shortened by heating the cemented object up to 140 degrees F.

HXTAL is a two-part epoxy resin system. The part must be weighed accurately to insure good results. The resin (part A) and the hardener (part B) are mixed A:B = 3:1 by weight. The unused mix can be kept for 4 to 5 days in the freezer. It is most important that the mix container be tightly stoppered. Cure at 96 degrees F, 95% RH produces the same highly glossy result as cure at 77 degrees F., 50% RH; showing that humidity does not seem to affect the cure at all. The cured resin does not embrittle with time and is very resistant to yellowing and surface degradation, even after prolonged exposure outdoors. HXTAL's initial yellowness index is 6 to 8, which is unchanged after 1600 hours of Weatheromoter exposure, rising slowly to 13 to 14 at 3,000 hours. The best competitive epoxy adhesive has an initial Yellowness of 9 which rises to over 40 after 800 hours in the Weatherometer. After 800 hours exposure, the coating began to erode away.

When colored with titania, the resin can be made to match the finest porcelain in color, translucency and surface appearance. With or without pigment, HXTAL NYL-1 can be used as a glaze that is highly resistant to water and most solvents. The cured resin can be removed from hard surfaces with our Epoxy Resin Dissolver, but prolonged soaking may be required to soften joints.

One general note from tests now underway: there is a marked increase in adhesion of glass when the surface is etched with ammonium fluoride (ceramic "etching cream").

Comments and Mixing Info

HXTAL Epoxy Adhesive is the only epoxy resin known to the field of conservation that does not yellow upon exposure to light. HXTAL owes its stability to its ultra purity – the traces of metal ions removed during the manufacture and purification of HXTAL, are responsible for the development of color in ordinary epoxy adhesives. Because of its ultra purity, HXTAL cannot be treated like an ordinary "hardware store" epoxy adhesive.

HXTAL comes in two liquid parts, both of low viscosity. Hxtal epoxy should be weighed out accurately, one part by weight of Part B plus three parts by weight of Part A. Transfer of the two parts from the original containers should be made by means of glass medicine droppers into a small, clean, glass, mixing jar. Clean baby food jars are ideal. After the two parts have been weighed into the jar; mix thoroughly with a glass, stirring rod. Do not rush – there is plenty of time before curing!

At this point, the HXTAL is ready for use – at least ready for some uses. Freshly mixed HXTAL is very thin. For many uses, as cementing porcelain, it is too thin. Let it stand, lightly covered, and over a period of several hours it will thicken. Bond strength is not lost by the use of thick HXTAL and it tends to stay in the joint line far better than the quite thin, freshly mixed HXTAL. However, freshly mixed, thin HXTAL does have one outstanding characteristic – it will penetrate cracks. It penetrates them quickly and makes them disappear from view. For the best results, warm the crack and its surroundings to about 120 degrees F – a hair dryer on low or a heat lamp at a two foot distance (do not warm the piece either too fast or too much) – and then apply one drop of freshly mixed HXTAL onto the crack. If all goes according to plan, the crack will simply vanish. Wipe off the excess HXTAL and then put the piece aside for a week. Freshly mixed HXTAL is also ideal as a coating for ground glass surfaces. For example, an abrasive cut pattern in glass coated with HXTAL appears to have been etched rather than abraded.

HXTAL sets slowly – at 75 degrees F, it require about 7 days to achieve 90% of the ultimate bond strength. However, ordinarily, HXTAL is set sufficiently after 24 – 30 hours to hold the two parts together if no serious stress is applied. This is an ideal time to clean up. A single edge razor blade or a razor knife is the best tool to scrape up the excess resin. At this time, HXTAL is rough, but removable. It is not recommended to use solvents to clean up – the solvents migrate into the joint line and can interfere with the bond to the artifact. Often, the bond weakening is not evident until much later and then in the form of little micro bubbles in the edge of the joint. Past 24 hours curing, HXTAL cannot be easily removed! A further word of caution – be certain that the two parts being joined are in their proper positions and that they cannot shift during the curing time. HXTAL is quite unforgiving and the joints are most difficult to part after 24-48 hours have passed. It's recommended to check position of bonded parts several times during the cure process to ensure no shifting has occurred.

Many glass artists use HXTAL epoxy adhesive to glue pieces of various glasses together to form art. Often, in the process, glued assemblies of blacks are cut or sliced with diamond or other abrasive saws and then more blocks are glued on the assembly. In cases where these abrasive cuts are made across HXTAL glued joints, we recommend treating all surfaces to be bonded with HXTAL epoxy with an A-1100 solution in reagent grade isopropanol. Glass artists have found that the pre-treatment with the A-1100 solution eliminates tiny micro bubbles that seem to form, often much later, in the glue line exactly along the sawn edge. Treatment with the A-1100 solution is simplicity itself. When the glass surfaces are totally clean and ready for gluing, simply apply the A-1100 solution over the entire surface to be glued – apply with lintless rag, brush, etc. – and allow the solvent to evaporate leaving an extremely thin film of A-1100 coating the surface of the glass. Immediately bond with HXTAL in the usual way. Note that the purity of the isopropanol is important, MuseuM Services Corporation offers an A-1100 Solution formulation in a reagent grade of isopropanol. Impure grades of isopropanol may interfere with the bonding of the A-1100 to the glass surface.

There is no difference in bond strengths between freshly mixed, very thin HXTAL opposed to thicker HXTAL that has been allowed to stand around somewhat. More viscous HXTAL does have a number of advantages. When gluing two very large, heavy pieces of glass together, the simple weight of the two pieces is often enough to squeeze out too much HXTAL, especially if the glue is thin. Such very THIN

joints are definitely weaker than standard thickness joint lines! Avoid excess squeeze-out of the glue. Using thicker HXTAL is one approach. In extreme cases, consider the use of tiny bits of glass shim at the corners, especially if these edges are to be ground or sawn off in later operations. Fragments of microscope cover glasses (available inexpensively at scientific supply companies) are ideal for use as shims.

Thicker HXTAL joint lines are also advantageous when joining materials with different coefficients of thermal expansion, such as glass to metal joints. Caution is urged in making theses types of joints! HXTAL adheres so strongly to glass that when these joints fail – as they often do when subjected to temperature differentials – almost always the HXTAL adheres to the glass and pulls a large, well-fractured piece of glass out of the joint area! These failures can be quite catastrophic to a piece of glass art.

The survival chances of these dissimilar joints can be improved by using a thick joint line – as much as 1 millimeter thick. Fully cured HXTAL is not brittle, but rather quite rough. This lack of brittleness allows the thick joint line to better accommodate slight expansion and contraction during its lifetime.

Many customers may initially be hesitant to use HXTAL due to HXTAL's long set time. Attempts to speed up the set of HXTAL must be approached with great caution! NEVER< NEVER attempt to heat freshly mixed HXTAL with an open flame, a heat lamp, a hot plate, a hair dryer or similar heating device. In high temperatures, HXTAL will begin to cure unevenly at the surface of the container and could overheat, possibly even taking fire! Even if evidence of over heating is not visually evident, an unevenly heated mixture of HXTAL is more fully cured in some parts than others where the curing reaction may not have even started. If heating the mixed HXTAL to speed the thickening, use only a water bath at 120 degrees F. Heat initially, for 15 minutes, remove the glass container with its HXTAL contents and continue stirring for a few minutes. Observe the new, thicker viscosity at room temperature and decided if additional 5-minute periods in the water bath are needed to reach the viscosity suitable for the specific job. Once thickening has begun, it proceeds more rapidly with each subsequent heating period. Also, caution is advised when mixing larger batches of HXTAL. The behavior upon heating becomes more critical as batch size increases – large batches have a tendency to spontaneously exotherm from the internal heat of reaction. Keep a cold water bath handy when working with batch sizes over 100 grams.

It is also possible to reduce total curing time by placing the artifact in a warm 90 - 100 degree F area for a day or two. It is not recommended to a hot box or other elevated heating location during the first 36 hours of cure time. Too often, immediate use of the hot box will cause the HXTAL to thin out and run out of the joint. After approximately 36 hours, HXTAL is thick enough to not move at 90 - 100 degrees F. Thus, to reduce cure time to its practical minimum, thicken freshly mixed HXTAL in a water bath at 120 degrees F, apply the glue at room temperature (or a bit higher, if practical), wait 36 hours at room temperature, and then, if all is well and correctly aligned, place the piece in a 90 - 100 degree F hot box for 2 days.

Epoxy Hxtal NYL-1 (Sidney S. Williston, Pub.)

We have been most interested in a new epoxy formulation, HXTAL NYL-1, developed by Herbert V. Hillery with the assistance of Texaco and Shell Laboratories. We were given a sample in August 1962, then purchased some from the distributor, Conservation Materials, in September. We have just (January 1983) received a new lot – even more nearly colorless than the initial sample. The resin has a Munsell chroma of less than 0.5 for a 1" thick sample. This includes the contribution of the flint glass square. The hardener has a chroma of 2.0 (1" thick). As the mixture is approximately 4:1, the mixed product has very, very low color – negligible in glazing work. More important is color stability. Atlas Weatheromoter data from the manufacturer shows almost no change for 1200 hours and then only a very slow increase out to 2200 hours. Competing brands in this test show rapid increase in yellow index from the start. So far, we have experienced no yellowing problems. We hope to have specific test results in the fall.

Unlike some of the highly purified clear epoxies, NYL-1 does not crystallize at cool room temperatures. NYL-1 is a very slow-curing epoxy, we find to be easily manageable after adjusting our former procedures to allow for the longer cure time. At 75 degrees F., we find a reasonable (though not complete) cure takes place in three days, however, the cure does continue at lower temperatures. One can greatly accelerate this cure by pre-heating the mix to 150 degrees F. or higher for 15 – 20 minutes before application. At 95 degrees F., a good cure takes place in 1½ to 2 days. NYL-1 also will completely cure at lower temperatures but will require several days. We have constructed a four-foot square chamber with good insulation, which is maintained at about 10 degrees above ambient by one 75W lamp. So far our tests are inconclusive as to whether one gets a significant increase in bond strength at 95 degrees F cure compared to 75 degrees F cure. One manufacturer claims this to be true: we shall continue our tests.

This slow cure turns out to have some advantages. The day following cementing, tapes can be removed from the object and clean up of excess adhesive accomplished very easily – with light knife work and even acetone on a swab. We have been able to use local heat and make minor adjustments to joints, which have slipped slightly. Unused mixes can be kept in the freezer of a refrigerator (covered, of course) and used for at least four or five days. This is especially helpful when one has attained a good color match and needs to make additional fills or spray on more than one coat of NYL. (NYL air brushes well when thinned – the alcohols, ketones and aromatics work well.) We have found that the leveling of NYL is excellent. Gloss retention is claimed to be very good, even out-of-doors. No induction period is needed with NYL.

NYL-1 accepts both hydrophobic and hydrophilic silica – however, the thixotropic effect is attained with less silica when the NYL is only a few hours older rather than when it is two or three days old.

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<u>Tricks with Epoxy</u> Sidney S. Williston and Melba Myers, Mario's Conservation Services

Sidney S. Williston and Melba Myers spoke about "Tricks with Epoxy". They use epoxies for the repair of functional objects and for more rugged decorative finishes where heat resistant repairs are needed. In the case of glass, a preparation of 0.5% solution of a silane (Silane A-1100, manufactured by Union Carbide) in isopropyl alcohol will greatly increase the strength of a repair. Epoxies clean up with a variety of solvents before they set, but only swell with methylene chloride when cured. Items with epoxy repairs should never be placed directly into a bath of 100% methylene chloride: it could cause them to crack due to quick swelling. All epoxies yellow with time. At present, where color fastness is important, the best available in this country is HXTAL, (made by Herb Hillery, available from Conservation Materials Ltd.). Epoxies can be toned with pigments for opaque colors or dyes for transparent colors, but Hansa Yellow cannot be used because it will turn the epoxy brown. All additions should be tested before using. Epoxy hardeners will attack copper, copper alloys and zinc metals; therefore, the mixture should be thoroughly combined before use. Make sure that the mix ratio is carefully followed. IN certain mechanical situations, pins are necessary to resist deformation because even epoxies are thermoplastic. Five-minute epoxies cure quickly, but readily yellow, whereas HXTAL cures slowly and is very light fast. Elevating the curing temperature to around 110 degrees F. can accelerate the curing of HXTAL, although temperatures over 135 degrees F. will ruin epoxies.

They also discussed aids in applying epoxies, such a: thorough cleaning of a break with detergent, acetone, isopropyl alcohol and dry compressed air; the use of various tapes for holding during setting or for damming; the heating of one side to improve penetration; the use of additives such as microballoons and precipitated chalk for thickening; and fumed silica for thixotropic effects. Materials that contact the epoxies during curing must be tested for color reaction problems. Thinners should not be used for mending, but can be used when inpainting with epoxy. Thinned epoxies must be briefly heated to drive off the solvent before they are set aside to cure; otherwise the epoxy may remain spongy indefinitely. Saran wrap reacts with epoxies, but polyethylene wraps do not react. Lastly, several treatment slides were shown and the techniques were discussed.

Sidney S. Williston has published a note in the February 1983 AIC Newsletter concerning HXTAL and certain techniques he has developed using HXTAL.

Physical Properties of HXTAL

Tensile Strength 5,400 psi Tensile Modulus 316,000 psi

Elongation 3 %

Flexural Strength 10,100 psi Flexural Modulus 365,000 psi

Impact Strength 0.14 ft.lbs./in. Heat Distortion Temp. 264/66 psi 37.5 / 39.0 C Shore D. Hardness 0/10 sec. 78/73

Shore D. Hardness 0/10 sec. 78/73

Gel Time, 110g. mass 150 degrees F 25.8 min

100 degrees F 262.7 min

Cure of 0.2 mm Film at 25 degrees C

Set to touch 15.3 hrs Thorough dry 30.9 hrs

According to Herbert Hillery, developer of HXTAL, that the shelf life is 1 year. Anecdotally, many artists and conservators find that HXTAL continues to have good working quality/bonding even after 2 years.

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