Article: Tips and tricks with epoxy and other casting and molding materials
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TIPS AND TRICKS WITH EPOXY AND OTHER CASTING & MOLDING MATERIALS

Stephen P. Koob

Introduction

Epoxy resins are used both as adhesives and as a fill material for loss compensation. Conservators should avoid them whenever possible. This class of materials is very difficult to use, and has some major limitations, which include incredible strength, a potential to yellow, and difficulty in reversibility. This paper introduces some suggestions as how to better use the epoxies, and how to better control them.

Considering the disadvantages, why are epoxies so popular? They bond well to glass (Fiorentino and Borrelli 1975) and are a major improvement over early joining methods, such as iron clamps; or early adhesives, such as shellac, and cellulose nitrate, which tends to separate from glass in less than 20 years. Acryloid B-72 is an excellent solvent adhesive for glass (Koob 1996, 1997), and is entirely satisfactory for the repair of Roman and Islamic glass, as it has moderate strength and excellent stability. The join line, however, will incorporate air bubbles, which can be slightly visible on the clearer glasses. For most clear modern glasses an epoxy adhesive will present a somewhat better appearance, as no solvent is necessary and the join line is bubble-free. The different types of epoxy resins also offer a broad range of refractive indices, from 1.51 (Hxtal NYL-1) to 1.56 (Epotek 301-2 and Fynebond), a useful property that allows the conservator to match the refractive index of the glass (Tennent and Townsend 1984). Epoxies are also excellent casting resins, useful primarily for loss compensation on repaired glass.

The tendency of epoxies to yellow (Down 1986), even in the dark (Down 1994), is perhaps the greatest drawback to their use, both as an adhesive and a fill material (Fig. 1). Their unbelievable strength is another issue (up to 5,400 psi tensile strength for Hxtal). The strength of the adhesive, combined with the small amount of shrinkage that occurs on setting (1- 2%), puts the glass surface at risk. When a join is stressed, the glass is more likely to break than the epoxy, and fills have been known to tear off edges of the surrounding glass.

Still, science and industry (and conservation) have all made some progress, and there is now a good supply of excellent epoxies available (see Table 1 and Suppliers). These are just some general properties, as the conditions of work and application can radically change many of these numbers.

Using Epoxies

All of these epoxy resins are supplied in two parts, the resin and the hardener. All are clear when supplied and the recommended shelf life is only one year. The resin has a higher viscosity than the hardener, and flows at about the consistency of thick syrup or thin honey. The hardener flows more like water. When mixed, the viscosity will drop dramatically, particularly during the first hour after mixing, and at this point the epoxies are very difficult to control without a closed space to contain them. The following suggestions are useful both for controlling the epoxies and for obtaining consistently reproducible results.

Table 1. Epoxies currently popular in the U.S.

Properties @ 22 ° C

Name	Working Time	Setting Time	R.I.	Viscosity	Tg (° C)
Hxtal NYL-1:	4-12 hours	12-24 hours	1.51-1.52	---	48 ° C
Epotek 301:	2-4 hours	4-8 hours	1.539	100-200 cPs	> 65 ° C
Epotek 301-2:	6-8 hours	8-16 hours	1.564	300-600 cPs	> 65 ° C
Fynebond:	8-10 hours	12-24 hours	1.565	"moderately low"	---

Laboratory Equipment

Recommended equipment includes a good binocular microscope with adjustable working distance; an analytical balance with precision to two decimal places; a fume hood, or fume extractor; and a small oven. Most suitable is an "incubator" oven (Fig. 2), that controls temperatures to moderate heat, in the range of 35° C at the lowest setting to 65 ° C at the highest, which is the highest level to which glass should be subjected.

The working area must be kept clean, which is extremely important for applying epoxies to delicate materials like glass.

Environment

A controlled environment is also critical to successful epoxy application, as problems in setting have been noted in high humidity situations (Haynes 1997). Work should be done in a moderately dry (40-55% RH) environment and at moderate temperature (20-25 ° C). For faster setting, a low temperature oven is useful to keep the temperature between 35 - 40 ° C. At these temperatures no adverse effects have been noted on the performance or ageing characteristics of any of the epoxies. One should not work with epoxies in a damp basement, or outdoors.

Manufacturers' Recommendations

Manufacturers' recommendations on mixing proportions should always be followed precisely, hence the need for an analytical balance (Fig. 3). The balance should be kept clean, with the pan protected by a piece of filter paper (see Fig. 3). Drips onto any surface should be cleaned up as soon as possible. For ease of use and reproducible results, it is best to mix epoxies in small quantities. A little bit of epoxy goes a long way, and given the high expense, it is prudent not to waste it.

Mixing

For mixing, clean glassware (Fig. 4) is recommended and is both convenient and reusable. Different sizes of watch glasses accommodate small quantities of epoxy (1-5 g), and glass stirring rods should be used rather than metal spatulas, as epoxies are extremely sensitive to contamination and staining from metallic ions. If a larger batch is needed, a polystyrene cup or glass weighing bottle can be used. All glass should be carefully cleaned after every use.

Once the proportions are weighed out, the hardener should be thoroughly stirred into the resin for one to two minutes. The mix should then be set aside for 5 minutes, preferably in a warm area, such as in the incubator oven at the lowest setting. The epoxy should then be mixed again for at least a minute. This is one of the most important steps in obtaining a homogeneous mixture and consistent results.

At this point, the epoxy is usable, or it can be left to sit (in or out of the oven), depending on how soon it is needed. In general, for pouring into a mold it should be used within the first hour of mixing. Otherwise, it is used when the consistency is appropriate. For injection, it is best used soon after mixing (Fig. 5), but edges can be joined hours after the epoxy has started to thicken. A batch of Hxtal can be mixed at 9:00 AM, and after 8 hours in the oven at 37 ° C it will be about as tacky as a solvent adhesive. The setting times will vary, depending on the ambient temperature, either in the room or if the epoxy is warmed. With the exception of Epotek 301, which is rather fast setting and is unusable after about 4 hours, all the other epoxies allow for a much longer working time. No change has been noted in the strength, clarity or appearance of any of the epoxies after gentle heating or warming in an oven, and the use of an oven is preferable to heating on a hot plate, in a water bath, or under a light bulb.

Use as an adhesive

For use as an adhesive, the substrates to be joined need to be clean and dry. Glass should be washed with a dilute detergent, thoroughly rinsed in deionized water and dried. The edges of the breaks should be checked under a microscope, and dusted or cleaned again as needed. In the author's opinion, it is not necessary, nor recommended, to use any other pre-treatment or surface modification such as coating with a silane, or using an etching cream. As mentioned above, it is best to apply the epoxy in a relatively dry environment. Various methods can be used for joining, and have been published elsewhere (Davison 2003).

Loss Compensation

For casting and loss compensation, even on colored glasses, sometimes the epoxy can be used clear, immediately after mixing. For very small losses, no tinting or coloring is usually required, as the epoxy will pick up the color of the glass. Often a few drops will level beautifully, with no finishing required.

Other materials can also be added for special effects, as long as they do not interact with the epoxy. These include pigments, dyes, fumed silica, fumed titanium dioxide, to change the transparency, texture, color or surface appearance of the fill.

Adding dyes must be done with great care. A little dye goes a long way, and the dyes should be mixed individually with a small amount of the *epoxy resin only*, with *no hardener* added. The dye is stirred into the resin, put aside to sit for an hour, and then stirred again (Fig. 6, top and Fig. 6, center). The resin should not be warmed at this stage, and the mixture should simply be covered and left overnight, as the dye continues to bleed into the resin. Usually, after 12 hours the dye is completely dissolved (Fig. 6, bottom).

At this point, the dyed resin is ready for use and color matching. As much as is needed is poured out, or added to undyed resin which has been weighed, and the correct proportion of hardener then added. Not all the dyes retain their initial color. This is especially true of Orasol blue (GN), which is bright blue when mixed with the resin, but turns a rather different greenish-blue color with the hardener added. Sample testing with each dye should be carried out before attempting a final color match. If the dye is added to an epoxy that already has the hardener mixed in, it will continue to bleed and darken the mixture for hours, producing a much darker and more intense result, often accompanied by streaks and spots of undissolved dye.

Silicone Rubber

Casting and molding with silicone rubber requires considerable patience, care and attention to detail. Perhaps the simplest type of silicone mold is a one-piece or "enveloping" mold. For filling losses in glass, a detachable replacement fragment can easily be made by initially casting the loss with plaster, and then making a silicone rubber mold of the plaster fill (Koob 2000). For replication, the object itself will be used to make the mold. For either application, the object or fragment is placed in a plastic cup or walled container, and positioned so that the silicone rubber can be poured around it. Small toothpick pieces and a drop of B-72 adhesive (Fig. 7) are useful to position the fragment or object to be molded, and to keep it from moving during the silicone application. In addition, the securing points also serve as sites for the later injection or venting of the casting resin. Release agents (such as Krylon spray) may be used for plaster fragments, but silicone rubber does not naturally stick to solid glass.

Once the silicone has set (usually 4-8 hours, for most silicone rubbers, although faster setting silicones are available), the molded fragment is removed by cutting a slit around one side of the silicone rubber, and the object or fragment is removed. The mold should then be placed in the incubator oven at 40-50 ° C, to completely cure and off gas before it is used for casting (Fig. 8). This is an important step because it ensures a complete crosslinking of the silicone and prevents the epoxy from interacting with the mold or adhering to it.

At the Objects Session at the 2002 AIC Meeting in Miami, Jonathan Thornton suggested the use of clear silicone rubber as a molding material, as it allows one to see what is happening during the molding and casting procedure. There may be numerous types of clear silicone available (Fig. 9) and this is an area which requires more investigation. The author's initial attempts with a clear silicone rubber were somewhat disappointing, as the silicone rubber tested had a very high viscosity, and trapped numerous air bubbles on mixing. Even with the use of a vacuum pump, the resulting silicone mold was translucent at best. In this instance, the fault may have been the vacuum pump, which was old and was not able to draw a strong vacuum. After purchasing a small aspirator vacuum which attaches to the sink (Fig. 10), it was possible to get the clear silicone rubber bubble-free (Fig. 11). An aspirator vacuum costs less than $10.00, hooks easily onto the faucet of a sink, and draws an amazingly strong vacuum.

The above example was a reproduction molding of a "gold dollar". After the original coin was removed and the mold cured in the oven overnight at 45 ° C, the interior of the mold was dusted with bronze powder and a batch of murky brown Epotek 301 epoxy was dripped in by pipette

(Fig. 12) until it came out the vent hole (Fig. 13) where the toothpick had been. A small piece of clear tape was placed across the front vent, to prevent any further seepage of the epoxy. The top of the mold was overfilled, leaving a pool of epoxy on the top of the silicone rubber. Epoxies tend to shrink slightly (usually 1 - 2 %), and owing to their low viscosity they also leak easily, so it is always necessary to keep an eye on the mold and the level of the epoxy. Any leftover epoxy can be kept in the pipette and placed in the refrigerator or freezer in case more is needed later.

Lower viscosity silicone rubbers generally do not require the use of a vacuum to remove air bubbles, nor is it necessary to draw a vacuum on the epoxy before injecting it in a mold. All of the air bubbles should have left the epoxy within a few minutes of the second mixing, and slow and careful pipetting is usually all that is required. If the first casting is not entirely successful, the silicone mold can be reused numerous times. Most air bubbles can easily be filled using fresh epoxy and a sharpened bamboo skewer, but if there are too many air bubbles, it is simply easier to make a new casting.

Other useful materials

Harbutt's Plasticine is an excellent modeling clay (Fig. 14) and has good working properties for use as a backing material, or for molds. It can be warmed in the oven to soften it, as it is wax-based, and then rolled flat inside a plastic bag. If the plasticine is too oily or too waxy (as in this case), it can be "dried out" a bit by adding some baby powder (cornstarch and not talc, since talc is a silicate and should not be used in direct contact with glass or ceramics). Alternatively, the plasticine can simply be dipped in plaster and then kneaded again, to mix the plaster in.

An excellent dental plaster, with no additives, is French's Diamond "P". Dental waxes come in a variety of thicknesses and colors and are useful for interior and/or exterior molds (see Suppliers list). They do not require a separating agent as the wax can be removed using a petroleum distillate solvent that does not affect the epoxy; however, the surface will be matt in appearance. If a glossy surface is required, it is preferable to use a silicone rubber and to take the mold from a very shiny surface, such as the glass itself (Figs. 15 and 16). It is also possible to do a detachable fill (Koob 2000) using plaster, coated with Krylon spray, as an intermediate step.

A few more useful materials include Scotch 3M tapes, Transparent ™, or Masking ®, which do not interact adversely with any of the epoxies. Other tapes should always be tested, as the adhesive in the tape may produce an unwanted color change in the epoxy.

Storing Unused Epoxies

Unused batches of epoxy can be kept in the refrigerator in film canisters or glass weighing jars for short periods in case of the need for touching up flaws and/or air bubbles in the initial casting. With the exception of Epotek 301, they should be useable for 24-36 hours, even if the hardener has been added. Hxtal and Epotek 301-2 can be kept in the freezer, and should be useable for at least 10 days, even after mixing. If a precisely mixed color is needed for multiple castings, it is recommended that the resin color mix, without hardener, be kept in the refrigerator until the project is completed. Some of the epoxy resins will crystallize over time, but can be gently re-warmed back to a liquid phase, without any apparent problems.

Finishing

Finishing can be done with a variety of materials, including solvents (in the first 24 hours), mechanical carving or abrasives (Fig. 17). Touch-ups can also easily be done using fresh epoxy, either to fill air bubbles or to apply a final overall coating to produce a shiny surface. Air bubbles can usually be filled using a sharpened toothpick and applying a small amount of epoxy to the edge of the air bubble; the use of a magnifier is recommended. The epoxy is allowed to flow down the side of the air bubble until it fills it to the top. Polishing compounds such as Solvol Autosol are useful for achieving a smooth, polished surface on a fill, but should not be used directly on the glass object.

Final Suggestions

- Avoid fills where possible
- Work in a CLEAN environment, with CLEAN tools & equipment
- Work in a climate-controlled environment, preferably 40-55 % RH; 20-25 °C
- Follow all manufacturers' advice, especially mixing ratios
- Enjoy your work

Acknowledgments

The author wishes to expresses his deep appreciation to Sidney Williston, a good friend and colleague who passed away in December, 2000. Sidney did a considerable amount of work with epoxies, and personally carried out numerous critical tests on the "quality control" of Hxtal NYL-1. We often discussed these and various applications, both on the phone and in his shop. Sidney both wrote and lectured about the use of Hxtal, including a note in the AIC *Newsletter* in March 1983 (Williston 1983). It was titled "Tricks with Epoxy", and is certainly a little out-of-date (e.g., the mixing ratio of Hxtal is no longer 4:1). This update is dedicated to Sidney.

Thanks are also offered to Stanley Robertson, who died in May, 2003. The author has many fond memories of Stanley, with whom he shared many ideas on adhesives and coatings when they worked together at the Freer Gallery of Art.

Sources of Materials (in order of appearance in the paper)

Hxtal NYL-1: Conservation Support Systems, 924 West Pedregosa Street, Santa Barbara, CA 93101.

Epotek 301 and Epotek 301-2: Epoxy Technology, Inc.,14 Fortune Drive, Billerica, MA 01821-3972, (800)227-2201.

Fynebond: Fyne Conservation Services, Airds Cottage, St. Catherine's, Loch Fyne, Argyl PA25 8BA, Scotland, UK

Orasol Dyes: Conservators Emporium, 100 Standing Rock Circle, Reno, NV 89511, (702) 852-0404.

Aspirator vacuum: Fisher Scientific, (800) 766-7000, (www.fishersci.com)

Harbutt's Plasticine: Conservation Resources, 8000-H Forbes Place, Springfield, VA 22151, (800) 634-6932 (www.conservationresources.com).

"Diamond P" Lab Plaster: Samuel H. French & Co., 4446-50 Cresson St., Philadelphia, PA 19127, (215) 482-6770.

Krylon Acrylic Crystal Clear (Spray): Hardware Stores or Hobby Shops

P-44 Silicone Rubber: Silocones Inc., P.O. Box 363, 211 Woodbine, High Point, NC 27261, (910) 886-5018.

Dental waxes: Paul H.Gesswein & Co., Inc., 255 Hancock Avenue, Bridgeport, CN 06605, (800) 243-4466, (gessweinco@aol.com).

Solvol Autosol: Conservation Resources, 8000-H Forbes Place, Springfield, VA 22151 (800) 634-6932, (www.conservationresources.com).

Scotch ™ Transparent Tape, Scotch ® Masking Tape: Hardware Stores or Hobby Shops

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Tennent, N.H., and J. H. Townsend. 1984. The significance of the refractive index of adhesives for glass repair, in *Adhesives and Consolidants*, Preprints of the IIC Paris Congress, eds., A. Roy and P. Smith. London:IIC. 205-206.

Williston, S., Feb. 1983. Epoxy Hxtal NYL-1. *AIC Newsletter.* Washington, D.C.: AIC 8 (2) 14.

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Figure 1. Epoxy fill, yellowed after over 30 years.

Figure 2. Incubator oven.

Figure 3. Analytical balance for precise weighing.

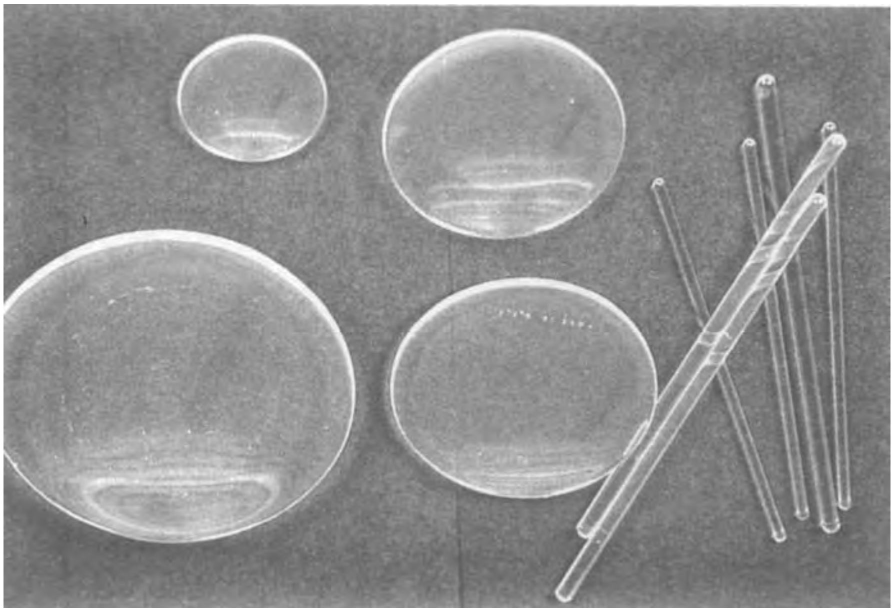

Figure 4. Glassware for mixing epoxies.

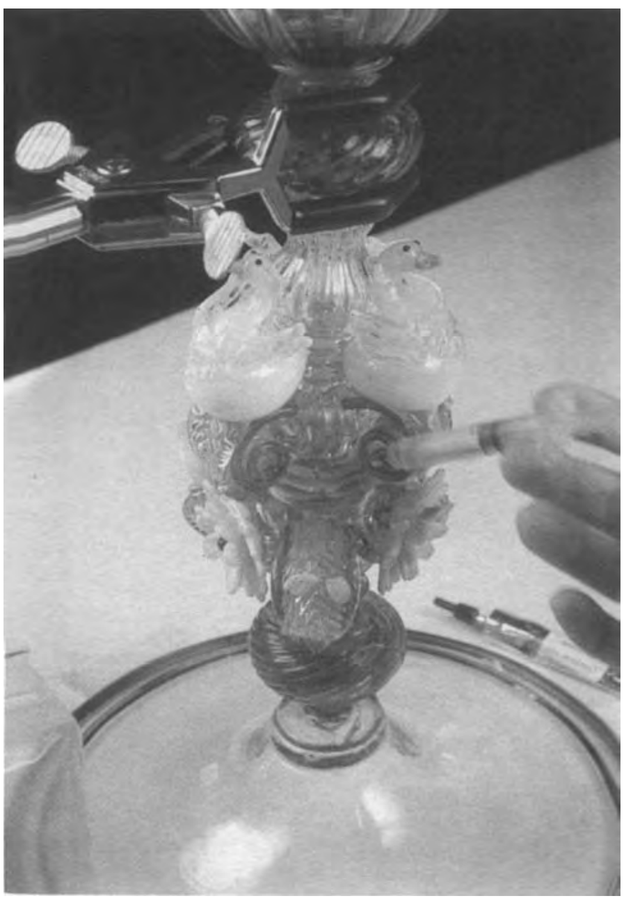

Figure 5. Injecting epoxy into a mold.

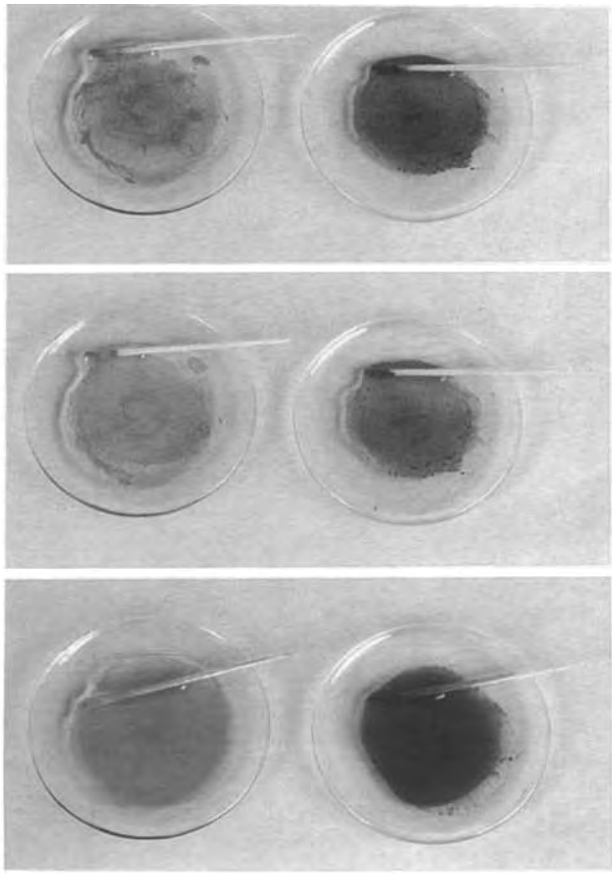

Figure 6. Dissolving dyes.
Top: initial mixing
Center: after one hour
Bottom: after 12 hours

Figure 7. Coin attached with toothpick fragments before pouring of silicone rubber.

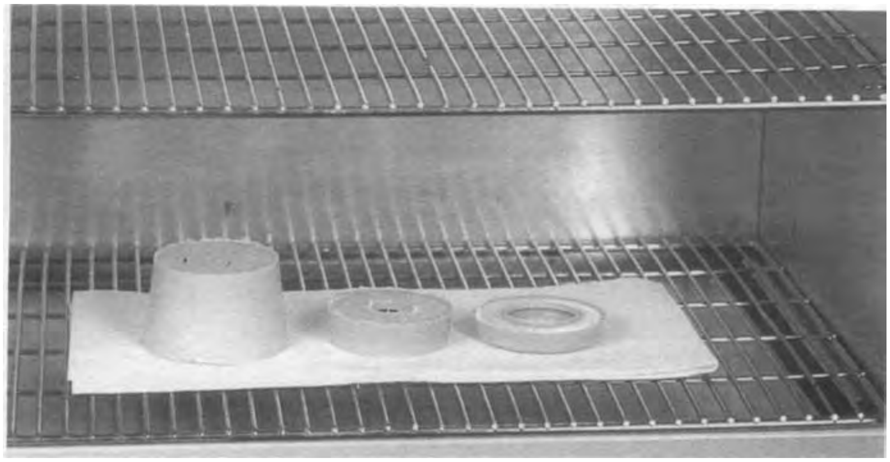

Figure 8. Silicone molds de-gassing in oven.

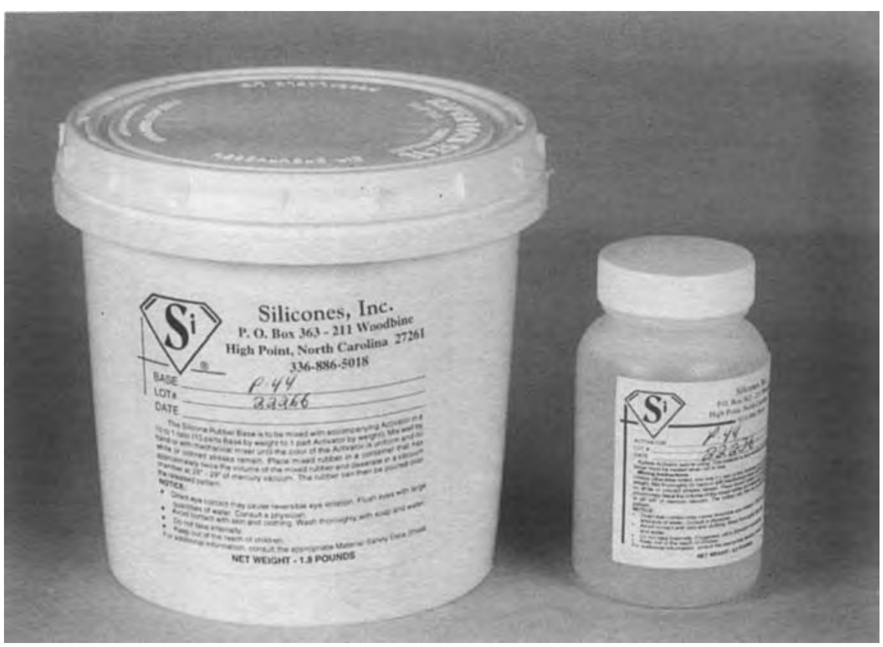

Figure 9. "P-44" clear silicone rubber.

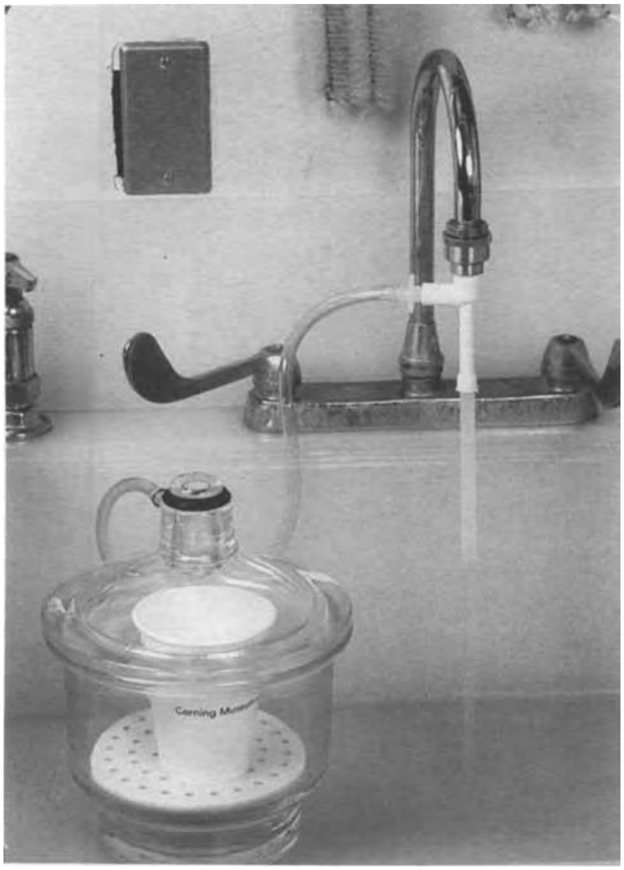

Figure 10. Aspirator vacuum and desiccator.

Figure 11. Clear, bubble-free silicone rubber casting.

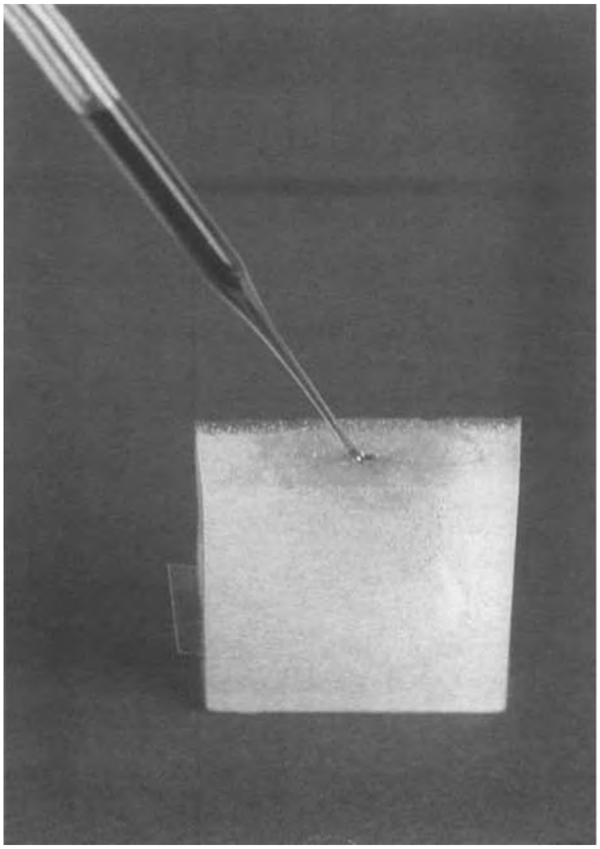

Figure 12. Injecting tinted epoxy into a mold.

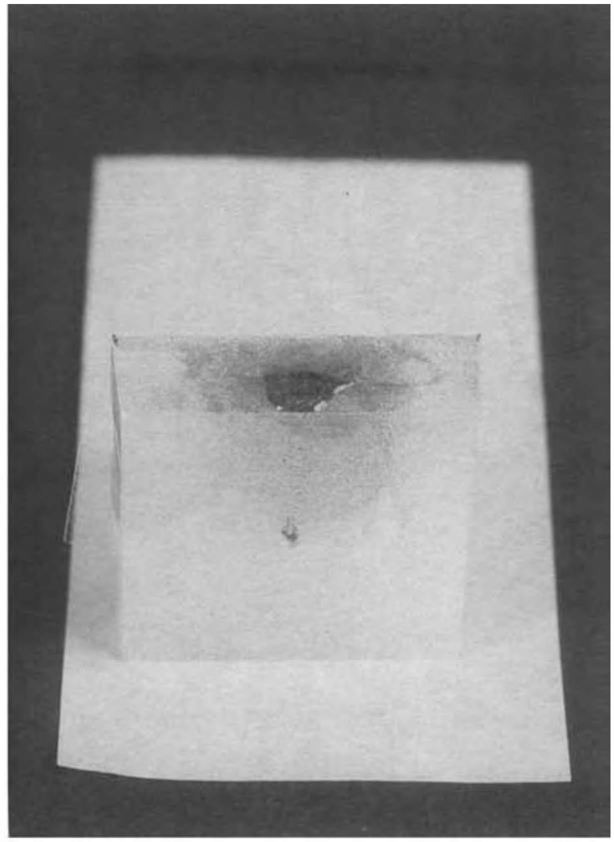

Figure 13. Over-filling to ensure complete penetration.

Figure 14. Harbutt's grey plasticine.

Figure 15. Venetian goblet with loss.

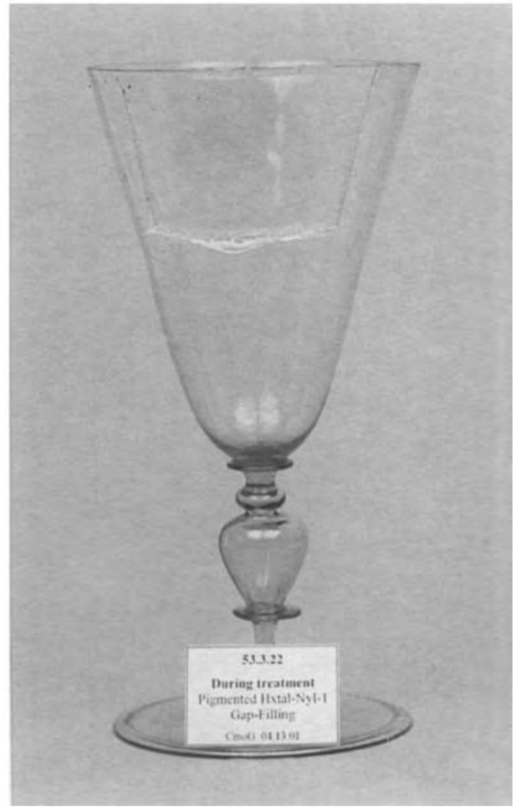

Figure 16. Venetian goblet with cast epoxy fill, matching refractive index and reflectance (before finishing).

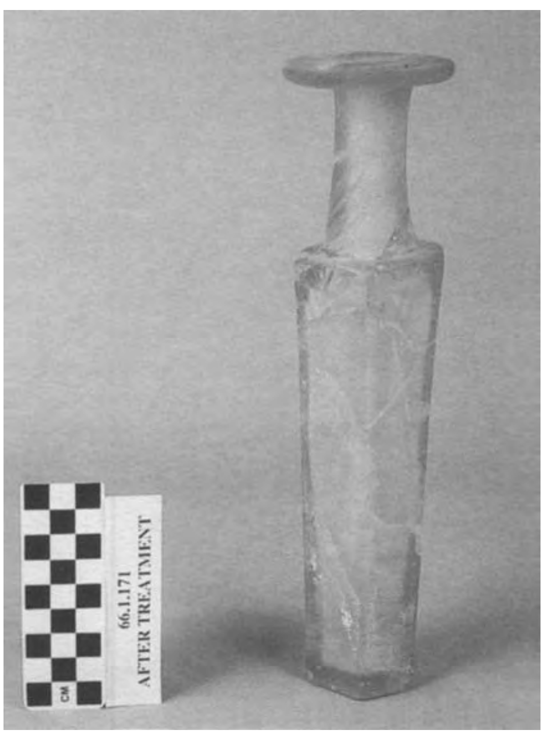

Figure 17. Finished epoxy fills on a Roman bottle (same as Fig.1, with new fills).