

Methyl Methacrylate Casting & Embedding Kit

for making hard, clear castings

Catalog Number 23679

Introduction:

Hard clear castings or embedments of exceptional clarity can be prepared by polymerization of methyl methacrylate monomer. These castings have properties similar to the well known Plexiglas® and Lucite® plastics and are easily fabricated into finished articles by sawing, drilling or polishing. In the polymerization process the liquid methyl methacrylate monomer is converted into the hard poly(methyl methacrylate) which assumes the shape of the mold. This kit contains all of the materials, except for molds, required to make poly(methyl methacrylate) castings or embedments.

In preparing castings from methyl methacrylate it is essential that the following general principles be observed:

1. The monomer must be free of inhibitor if castings are to be free of color.
2. Polymerization must be done in the absence of oxygen. In the presence of oxygen the resulting casting may not attain the desired hardness or polymerization may not proceed at all.
3. Dissolved gases should be removed from the monomer to prevent the formation of bubbles in the casting.
4. Provision must be made for temperature control in thick castings. The polymerization reaction is exothermic; i.e., it generates heat which can heat the monomer to boiling unless it is cooled.
5. Shrinkage occurs during conversion of the liquid monomer to solid poly(methyl methacrylate). In most cases shrinkage can be accommodated by using a mold which is deeper than the height of the desired cast object.

Precautions to be Observed in Handling and Storing Monomer and Catalyst:

Methyl methacrylate monomer (Catalog #00834) must be handled with care. It has a low flash point and so is **very flammable**. **Never heat the monomer over an open flame**. Keep the monomer away from sources of ignition such as electrical sparks or static discharge. Observe the precautions described under "Selection of Polymerization Method".

Methyl methacrylate monomer (Catalog #00834) is toxic and must not be ingested. It is also a skin irritant and some individuals can become sensitized to it. Handle the monomer with impervious gloves and safety goggles.^{1,2} If spilled on the skin, the skin should be washed using water and mild soap. Washing should continue until the odor of the monomer can no longer be detected. If the monomer is spilled on garments, change immediately to fresh garments to avoid skin irritation or prolonged breathing of the vapors.

The catalyst 1,1'-Azobis(cyclohexanecarbonitrile) should be stored in the containers in which it is supplied with the lid tightly closed. Store in a cool

place. Avoid contact with skin and eyes and do not breathe the powder. Handle using goggles and gloves. In case of skin contact wash thoroughly with soap and water.

Specimen Selection and Preparation:

Specimens may be either organic or inorganic and must be dry to prevent clouding of the casting by moisture.³ Most inorganic specimens and some organic ones may be oven-dried without distortion, but most organic specimens will lose their shape and color if dried rapidly in an air oven.⁴ Vacuum freeze-drying helps to retain the original form and color of organic specimens. Organic specimens may also be dehydrated by successive immersion in a series of mixtures of decreasing water content of methyl alcohol or propylene oxide (Catalog #00236). Pure solvent is used in the final step. Some metals, notably copper, cause inhibition of polymerization and they should therefore be precoated with an inert protective film of poly(methyl methacrylate) before being embedded. For valuable specimens it is always best to run a trial using a closely related item to determine if the catalyzed monomer attacks the embedded object. Porous and colored specimens may change in appearance after casting since the polymer-specimen interface has a substantially different index of refraction than the air-specimen interface under which it is normally viewed.

Inhibitor Removal:

As supplied, methyl methacrylate monomer contains a small amount of inhibitor, either hydroquinone (HQ) or methyl ether of hydroquinone (MEHQ) to prevent polymerization during shipment and storage. Inhibited monomer remains liquid and free of polymers for long periods at room temperature and still longer times under refrigeration. Preparation of colorless castings requires freeing the monomer of this inhibitor. It is possible to polymerize monomer containing inhibitor by increasing catalyst concentration, but this can produce a slight yellowish hue in the finished casting especially if it is one half-inch or more in cross-section.

Removal of inhibitor can be accomplished in several ways which are equally effective but vary in the complexity of equipment required. Vacuum distillation of the inhibited monomer at 61°C/200mm pressure using a capillary air bleed is one method. Washing the monomer with portions of aqueous sodium hydroxide solution until a fresh wash solution is

colorless, then washing the monomer with water until portions of wash water are neutral and, finally, drying the uninhibited monomer over anhydrous magnesium sulfate is another method. Both of these methods require equipment which may not be available to many workers. A third, simpler method is passage of the inhibited monomer downward through a glass column containing alumina. Removal of inhibitor can be followed visually. Inhibitor adsorbed on the alumina column can be seen as a light yellow band which moves down the column as monomer is passed through. No further monomer should be added when the yellow band has reached the bottom of the column. (See *Catalog #'s 23678 and 19708 at the end of this data sheet.*)

Once the inhibitor is removed, the monomer must be used promptly or it may be stored in the dark for a few days at 4°C or below. Unless kept cold, uninhibited monomer will polymerize spontaneously upon standing. An air space should be maintained over the monomer at all times during storage to maintain stability, since oxygen acts as an inhibitor.

Polymerization Catalyst:

A number of catalysts, either thermal initiators or photoinitiators, can be used for polymerization of methyl methacrylate. The catalyst supplied with this kit, 1,1'-Azobis(cyclohexanecarbonitrile), can induce rapid polymerization of methyl methacrylate by either mechanism. Generally, catalyst concentrations in the range 0.02% to 0.10% on weight of uninhibited monomer are sufficient although the amount of catalyst needed depends on polymerization conditions and the thickness and size of the casting. The catalyst should be added to the monomer at room temperature and totally dissolved by stirring. The resulting solution is filtered to remove insoluble materials. The catalyzed monomer should be used immediately or stored in the dark at 4°C or below.

Variations on the thermal polymerization method are described in the following sections. Photopolymerization is produced by photoinitiators when exposed to ultraviolet light and has the advantage of occurring even at room temperature or below. This can be an advantage in embedding some sensitive biological specimens. Mold design and composition considerations limit applicability of photopolymerization. In photopolymerization a peak wavelength of a maximum 360nm is desirable.

Other thermal initiators such as benzoyl peroxide (*Catalog #21446*) or lauroyl peroxide or other photoinitiators such as benzoin ethers (*Catalog #00425 or #00932*) or other azo initiators can also be used as polymerization catalysts in place of 1,1'-Azobis(cyclohexanecarbonitrile).

Selection of Thermal Polymerization Method:

Two general methods of making cast acrylic embedments are:

1. Atmospheric Polymerization (at controlled, slightly elevated temperature).
2. Autoclave Polymerization (at controlled, elevated temperatures but with pressures up to 150 pounds per square inch). The autoclave method with its high pressures has the advantage of raising the boiling point of the monomer, thus minimizing bubbling due to vaporization of the monomer or degassing of air. It also permits faster curing times due to the higher operating temperatures.

Atmospheric Polymerization Method:

(Please see previous section on *Precautions to be Observed in Handling and Storing Monomer and Catalyst.*)

This method utilizes a clear monomer-polymer casting syrup (see Preparation of the Casting Mix section). The casting syrup is formed into a firm gel base to allow the accurate placement of embedment. The base is formed by pouring a layer of degassed casting syrup of the desired thickness into the mold. The mold is covered with a layer of barrier film and the gel base is formed by carefully heating the syrup so that it never exceeds 35-40°C under normal atmospheric pressure until it is firm enough to support an embedment. The film is then removed and a small amount of catalyzed, inhibitor-free monomer is poured over the gel base and the sides of the mold. Excess liquid monomer is drained, and casting syrup is poured so that it will cover the specimen to the desired depth. Bubbles which form can be removed by application of vacuum or by covering the mold with film, placing it in a refrigerator, and allowing bubbles to surface. The specimen, which is first wetted with liquid monomer, is gently placed in the syrup until it rests on the gel base. Care should be taken to avoid trapping air bubbles when placing the specimen on the gel base. Polymerization could be in an explosion-proof oven with a temperature range of 35-40°C, or could be done safely in a hot water bath. A block one inch thick will fully harden in approximately 7 days at this temperature. Successive layers of casting syrup can be poured on top of the original block until the requisite thickness is obtained. Care must be taken to remove the heat of polymerization by providing adequate cooling, otherwise the mass may undergo uncontrolled polymerization due to heat build up.

Autoclave Polymerization Method:

In the autoclave method, a casting syrup, or casting slurry, may be used. This method is best suited for massive castings. If the casting syrup is employed, use the same technique described for oven casting, by preparing a gel base for supporting the specimens in the mold.

If the casting slurry is used, the base is prepared by simply pouring the high polymer content slurry as soon as it is made, and letting it stand (covered with film) until a firm gel is formed. A slurry of two parts of polymer to one part of monomer may be used if it is rapidly cast. When the mold has been filled, a layer of film is placed in direct contact with the top surface of the casting mixture.

The mold is then placed in an autoclave which is evacuated of air and then pressurized with carbon dioxide or nitrogen at 35 to 150 psi. The autoclave temperature is then raised to 50-80°C depending on the pressure, size, shape and thickness of the specimen and the maximum temperature the specimen can withstand. The elevated temperature and pressure should be maintained until polymerization is complete. The casting should then be cooled slowly to room temperature while still under pressure in the autoclave. If massive castings are made precautions must be taken to allow for adequate heat dissipation from the casting, otherwise a runaway reaction may occur. The polymerization is strongly exothermic and it is necessary to remove heat at a rate sufficient to keep the mass below the boiling point of the monomer at the chosen operating pressure.

Preparation of the Casting Mix:

Two principal types of casting mix are generally used for cast acrylic embedments:

1. **Syrup:** A clear liquid casting syrup which is a monomer- polymer solution is prepared by heating small amounts (usually less than 100 grams) of the inhibitor-free monomer at 70°C on a steam or hot water bath with continuous stirring (Please see previous section on Precautions to be Observed in Handling and Storing Monomer and Catalyst). Catalyst up to 0.02% may be used. The polymerization reaction is highly exothermic and once started may cause very rapid boiling unless the reaction heat is removed by external cooling and stirring. The viscosity of the mix will increase rapidly as polymerization proceeds. When the hot syrup reaches the consistency of glycerine, it should be cooled quickly to room temperature and stored at 4°C (or below) until ready for use. A casting syrup may also be made by completely dissolving at room temperature about 15% of the finely divided methacrylate polymer provided with the kit. Such casting syrups can be polymerized either by the atmospheric oven or autoclave methods.
2. **Slurry:** A casting slurry is prepared by mixing a high proportion (50-65% by weight) of fine-mesh poly(methyl methacrylate) beads (*Catalog #04553*) in the catalyzed, inhibitor-free monomer. The slurry should be cast around the embedment within minutes of mixing; otherwise it will set to a non-pourable gel. Casting slurries are best polymerized by the autoclave method. The slurry is usually viscous and air bubbles that are not removed before polymerization must be forced into solution under autoclave pressures. Momentary application of vacuum will help to degas the fluid slurry when it is first mixed.

Molds and Mold Release Agents:

The container selected for use as a mold should have an opening large enough to permit easy placement and manipulation of the specimen, yet should be capable of being sealed by a layer of film to exclude oxygen. Silicone rubber molds are particularly useful because their flexibility allows for removal of complex castings, including those with undercuts, from the molds. Unlike other mold compositions, silicone molds can be made by the person making the casting by using RTV silicone resins which are generally available in hobby shops. Polyethylene containers can also be used as molds, but as with glass molds, are generally limited to available shapes. Polymethacrylates tend to stick to metal molds unless they are highly polished. Mold release agents, such as lauric acid, stearic acid, or a 10% solution of cellulose acetate in methylene chloride can be wiped over the surface of the mold to prevent sticking. Glass makes a good mold surface due to its smoothness and transparency.

Annealing Cured Blocks:

After the embedment is removed from the mold, it is necessary to anneal it to reduce stresses due to shrinkage during polymerization. Annealing time is determined by allowing two hours of heating at 107°C for each 1/2" of thickness. Cooling rates differ for various thicknesses. For example, embedments having large cross-sections must not be cooled quickly to room temperature because severe internal stress may be produced due to uneven cooling.

Finishing:

The finished casting of poly(methyl methacrylate) may be cut and sanded with tools similar to those used in working with soft metals.⁷ The final polish is achieved by sanding the faces of the plastic, first using coarse grit and gradually working down to very fine 600 grit emery paper. After this last sanding, the sample may be buffed on a cloth wheel using ordinary rouge or fine alumina buffing compound.

References:

1. *Ind. Med.*, **14**, 292 (1945)
2. *J. Ind. Hyg. Toxicol.*, **23**, 343 (1941)
3. *J. Soc. Plast. Eng.*, **6** (4), 3 (1950)
4. *J. Lab Clin. Med.*, **28**, 1140 (1944)
5. *Stain Technology*, **50** (1975)
6. *Stain Technology*, **38**, 213 (1963)
7. *Bulletins on working with poly(methyl methacrylate)*, PL-2, PL-3, PL-9, Rohm and Haas Co., Philadelphia, PA 19105.

Ordering Information:

Cat. #	Description	Size
23679	Methyl Methacrylate Casting & Embedding Kit Contains: methyl methacrylate monomer (MMA) poly(methyl methacrylate) powder 1,1'-Azobis(cyclohexanecarbonitrile) catalyst alumina, activated powder	1 kit 3kg 1kg 30g 250g
00834	MMA monomer	1 liter 4 x 1 liter 3.8 litre 5 gallons
04553	PMMA beads	500g
23678	Alumina, activated powder (for inhibitor removal)	250g
00236	Propylene oxide (dehydrating agent)	1 pint
21446	Benzoyl peroxide, 70% (thermal initiator)	100g
00425	Benzoin methyl ether (photoinitiator)	10g
00932	Benzoin iso-propyl ether (photoinitiator)	100g
19708	Inhibitor Removal Column	1ea

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