

828/DEA/GMB Batching Manual

Spring 2022 Update

McCoy Group NMT

Safety Information

- Make sure that one or more qualified lab workers are available to help you batch before you begin.
- Personal protection equipment is required. Please consult the “Materials” section for specifics.
- Long pants and closed-toed shoes should be worn. Long hair should be worn up. Gloves should be changed in the event of an epoxy spill.
- Uncured epoxy resins will cause skin sensitivities to develop if contact is experienced on a regular basis. After the epoxy mixture has cured, the material is non-reactive and safe to be handled without gloves or safety glasses.
- Particular care must be taken with additives such as DBA, which is both corrosive and flammable. Carefully read all additives’ MSDS sheets before batching.
- **In the case of epoxy (EPON 828) or hardener (DEA) exposure to skin:** Rinse thoroughly with soap and water. Monitor site of exposure for adverse reactions, and seek medical attention if necessary.
- **In the case of epoxy, 828, or DEA exposure to eyes:** Remove contacts if wearing. Go to the nearest eye-wash station and rinse eyes for fifteen minutes. Monitor eyes for adverse reactions, and seek medical attention if necessary.

Procedure

This manual contains the following sections:

- A. Before Starting
- B. Syringe Preparation
- C. Mixing Chemicals
- D. Degassing
- E. Pouring and Cure
- F. Cleanup
- G. Recovering Cylinders

Materials

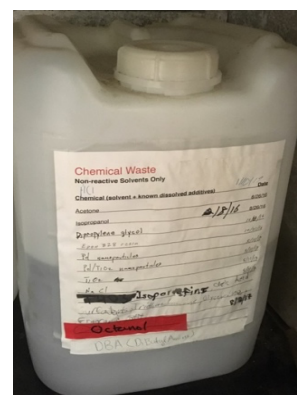
- Personal Protection Equipment: Safety glasses, a lab coat, and nitrile/latex gloves. All of these can be found in 130C.
- Chemicals: Diethanolamine (DEA), 828 Epon Epoxy-Resin
- Glass Micro Balloons – contained in a high-temperature-stable bucket fitted with a sieve.
- 15 10mL syringes
- Bunsen burner and lighter
- 2 syringe racks
- Top loading balance (accurate to 0.0g)
- Batching Sheet
- 2 mixing cups
- 3 Popsicle sticks
- 3 Pipettes
- Semco Tube (PPG Aerospace Semco 220337 Cartridge 12oz Low Density) and tip (PPG Aerospace Semco 220550 Model 440 Plastic 4" long* 1/8")
- Semco gun and plunger
- Acetone (to clean up spilled epoxy)
- Waste disposal container



GMB Bucket and Sieve



Semco tube, tip, plunger, and gun



Waste disposal container

Batching:

A. Before Starting:

1. Sift GMB using the #100 sieve (150 micrometer mesh)
2. After sifting, place the GMB into an oven set to 110C for at least 6 hours.
3. Place the 828, DEA, and Semco gun into a 70C oven for at least 1 hour.
4. Turn on the vacuum oven, turn the heating knob to between levels 4 and 5 (70-80C), and let equilibrate at least 1 hour.
5. Fill out a batching sheet (pictured below).
6. Mark the cup with the mixing weights for the 828, DEA, and GMB in the order they are added.



The vacuum oven heating knob is on the left.

828/DEA/GMB Batching Sheet		Date:
Batch No. : B		Made By:
Materials Used:	Quantity:	
EPON 828		
DEA		
GMB		
Mix ratio: 100:12:28 (828:DEA:GMB)		
Samples Made:	Quantity:	
Syringes		
Process:		
Pre-heat Semco metal sleeve, cups, resin and hardener at 70C for at least 1 hr prior to use. Sift GMB and reheat at 110C.		
Weigh out the appropriate mass of resin, heat, then the GMB. Mix until thoroughly combined and the consistency of toothpaste.		
Mix in hardener and stir until well combined.		
Place mixture in the 70C vacuum oven and pull a vacuum until the mixture breaks using the vacuum knob and release valve to ensure the mixture doesn't foam over the top of the cup. After the mixture breaks continue to vacuum for 2-3 min.		
Carefully pour mixture into tape sealed Semco tube and revacuum for 2-3 min. Insert plunger into the open end of the tube and cut the tape and replace with the tip. Cover with metal sleeve and screw into the cocking gun.		
Use gun to fill sealed syringes. When all epoxy is in syringes, place filled syringe racks in 70C oven for 24 hours.		

828DEAGMB Batching Sheet

B. Syringe Preparation

1. Unwrap 10mL syringes and unplug all (save one plunger)
2. Set up the syringe rack next to the workspace.
3. Turn on and light Bunsen burner.



4. Carefully melt the needle adapter of the syringe (the tip where liquid would be pushed out) by holding it over the flame. Let gravity help you seal the opening by rotating the syringe on its side.
Note: The open flame must be used away from the chemicals used for batching.
5. Place the freshly sealed syringe tip-down in the rack to cool.
6. Allow the syringes to cool for a couple of minutes.
7. Using the plunger you saved earlier, attempt to push air through the cooled syringes. If properly sealed, the plunger should resist the motion, and should slide backwards once released. If not, reheat the tip of the syringe.
8. Turn off the Bunsen burner and put it away.

C. Mixing Chemicals:

1. Begin by positioning your plastic container on the balance in the fume hood. Tare (with the plastic cup on the balance) by pressing the “zero” button.
2. Measure out the required amount of 828. As you get closer to your desired figure, use the designated 828 pipette to add the resin at a more controlled rate. Try your best to get within 0.1g of the target mass.
3. Tare the scale again, and **carefully** pour in required amount of GMB. Again, try to get within 0.1g of the target mass. Remove excess with a popsicle stick. Mix for 2 minutes, and place into the 70C oven for 3 minutes.
4. Add the required amount of DEA. Use the designated DEA pipette to minimize risk of over-pouring. Mix for 3 minutes. Proceed directly to the first degassing.

Note: All materials that come in contact with the epoxy/hardener mixture must be cured in the oven for 24 hours before going into the trash (after this period, the resin is fully cured and is no longer hazardous)

D. Degassing:

1. Place the mixture in the pressure chamber, and close the chamber door securely. Turn on the pump using the switch located on the lower right side of the pressure chamber.
2. Next, turn on pressure gauge using the switch labelled below.



Vacuum Pump Switch



Vacuum Controls

3. Turn the release knob to the right as far as possible. If the valve is not sealed, air will enter the chamber and you will hear a whistling sound.
4. Switch on the vacuum. Begin to turn the vacuum knob to the left. This will start reducing the pressure.
5. Maintain a pressure of about 10 Torr. The 828DEAGMB mixture should expand and then eventually “break”, collapsing back to its original volume.



Pressure gage on top of vacuum chamber

6. If after five minutes the mixture seems adequately degassed (that is, if the bubbling has significantly slowed), you may equalize the pressure in the chamber. Simultaneously twist *out* the release knob (to open the valve) and twist *in* the vacuum knob (to stop vacuuming), and wait until the chamber reaches equilibrium to open the vacuum door.

1. Pouring and Cure:

1. Place your degassed mixture back into the 70°C oven for a few minutes if it seems viscous.
2. Put a square of duct-tape over the narrow end of the plastic Semco tube.
3. Carefully pour the 828DEAGMB into the Semco tube. Minimize the incorporation of air by making the sides of the two containers near-parallel.
4. Degas the Semco tube. A “break” may or may not occur. You may stop the degas after 3 minutes under vacuum.



Perform steps 5-9 as rapidly as possible:

5. Carefully puncture the duct tape on the plastic tube with a razor blade. Make sure the threads on the inside of the tube are exposed.
6. Put the plastic tube inside the heated metal sleeve. Take care to not smear epoxy inside the metal tube. Screw on the plunger.
7. Screw on the plastic tip.
8. **Slowly** cock the gun until the tip is filled with epoxy.
9. Fill the syringes with epoxy, and take care to avoid incorporating any air. Start with the Semco tip at the far end of the syringe, and pull the tip out as the syringe fills. Don't fill beyond the 10mL mark. Place filled syringes in a rack.
 - a. Have one person operate the Semco gun while one person switches out the syringes. Take turns with the gun- it's hard on the arms!
 - b. If the epoxy gets too viscous, place in the 70C oven for a few minutes.
10. Place the rack of filled syringes inside the 70C oven. Cure for 24 hours

**F. Cleanup**

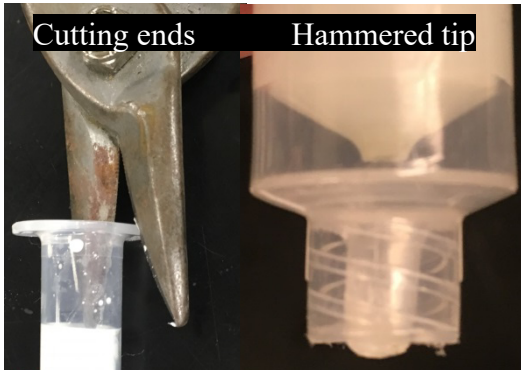
1. Any trash that has come in contact with any of the chemicals should be stored in the mixing container and cured in the 70°C oven for twenty-four hours. It is only after this curing period that the trash can be disposed of.
2. Wipe your work surfaces with acetone and paper towels. Place these wipes in a zip-loc bag before disposal.
3. Put your lab coat and safety glasses back in 130C.
4. Wash your hands thoroughly.

G. Recovering Cylinders:

1. Suit up with proper PPE. Safety glasses, thick heat gloves (located in the drawers of 130C), and closed-toe shoes are a must for this step. Wearing a lab coat is recommended.
2. Lay out all necessary tools for this process: wire cutters, hammer, pliers, Ziploc baggies, and sharpie. All of these are found in 130C.
3. Using the heat gloves, take the rack of cured epoxy cylinders out of the oven.
4. While still wearing heat gloves for all steps, make two cuts using the wire cutters from the end of the syringe up the length to the end of the epoxy. Begin hammering at the sealed tip of a syringe with firm but controlled taps. The objective is not to crush the syringe tip, but rather to knock the epoxy



cylinder down the length of the syringe. Aim to get the cylinder down at least a centimeter before proceeding with the pliers.



Note: Hammering the syringes can send sharp plastic debris into the air. Be sure to wear your safety glasses and the heat gloves to prevent injury.

5. Using the pliers, pinch the newly empty tip of the syringe in order to push the epoxy cylinder further out. Continue with this pinching action down the length of the syringe until a good portion of the epoxy protrudes from the syringe, at which point you can simply use your hands to completely pull the cylinder out.



6. Continue until all epoxy cylinders have been extracted.
7. Store the cylinders in baggies labeled with the material and the appropriate batching number (B and the date; for example, if it was batched on July 24, 2019 the batching number is B072419)
8. Place new sample baggie in the freezer in 130C.
9. Throw away the syringe husks and sweep up the plastic debris from the hammering step. Put your tools and PPE away.