

828/DEA/GMB Batching Manual

Summer 2019 Update

McCoy Group NMT

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Introduction

Getting accurate, reliable data on epoxy starts with creating well-made epoxy samples. Epoxy has two primary components: a resin and a hardener. Other additives may be included in order to optimize certain material characteristics. This manual details the batching instructions to make an EPON 828 resin cured with Diethanolamine (DEA) epoxy mixture and using Glass Microballoons D-32/4500 (GMB) as filler. The high filler fraction makes batching difficult and the following procedure is based on Sandia National Labs best practice with New Mexico Tech modifications.

Safety Information

- Never batch with fewer than three people. Working unassisted and unmonitored can make responding to an event of injury or incapacitation much harder and take longer. Make sure that two 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. If possible, it should be handled in a hood. 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 GMB as inhalation can cause lung irritation and harmful exposure to heavy metals. Read the appropriate MSDS sheets before batching for more information.

Procedure

This manual contains the following sections:

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

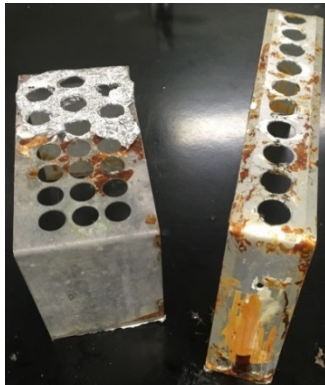
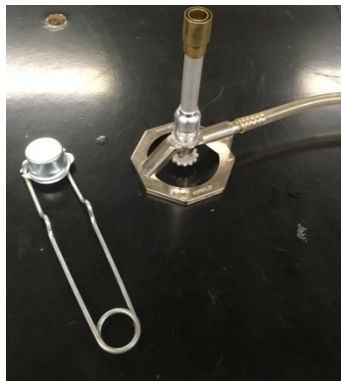
Materials

- Personal Protection Equipment: Safety glasses (stored in Jones 130B), a lab coat (also stored in 130B), and nitrile/latex gloves (stored in Jones 130 C).

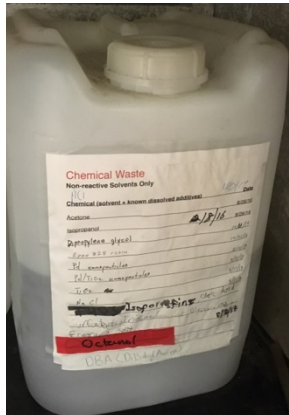


Personal protective equipment

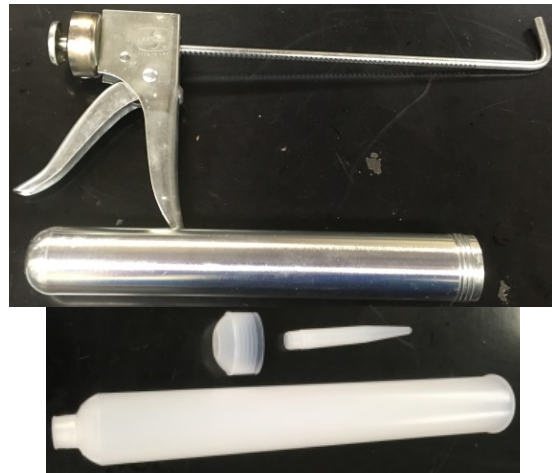
- Chemicals: Diethanolamine (DEA), 828 Epon Epoxy-Resin, and Glass Microballoons
- 15 to 17 10mL syringes
- Bunsen burner and lighter
- 2 racks to hold syringes during cure



- Top loading balance (accurate to 0.0g)
- Waste disposal container



- 4 Popsicle sticks
- Labeling Tape
- 3 Pipettes
- Semco Tube (PPG Aerospace Semco 220337 Cartridge 12oz Low Density)
- Semco plunger
- Semco gun
- Semco tip (PPG Aerospace Semco 220550 Model 440 Plastic 4" long* 1/8")
- Batching Sheet
- 2 32oz Cups
- Acetone for clean up
- GMB sifting screen
- Extra GMB bucket



Batching:

Before Starting:

1. Sift GMB in #100 150 micrometer (0.0059 inch) screen.
2. After sifting, place into ~107C equilibrated oven for a minimum of 6 hours.
3. Place 828/ DEA & Cups into ~70C equilibrated oven for a min of 1 hour.
4. Turn on and set vacuum oven to 75C and let equilibrate for 6 hours.
5. Fill out a batching sheet as shown below.
6. Using the different colored tape, mark one Popsicle stick and one pipette for 828, DEA and GMB separately.
7. Mark the cup with the mixing weights for the 828, DEA, and GMB in the order they are added.
8. The mixing cups, pipettes and Popsicle sticks should all be heated in the 70C oven for 1 hour.



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 reevacuum 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.		

Preparing syringes to hold epoxy samples:

1. PPE for preparing the syringes is a lab coat, safety glasses, a well ventilated area, and a clean bench. This is the one part of the procedure where gloves are not worn.
2. Unwrap 10mL syringes.
3. Unplug all syringes. Save only one plunger, the rest may be disposed of.
4. Set up the syringe rack next to the workspace.
5. Turn on and light Bunsen burner.
6. 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. Take caution during this step: **you should not ignite the plastic**, just heat it enough to liquefy it.



Note: The open flame must be used away from the chemicals used for batching.

6. Place the freshly sealed syringe tip-down in the rack to cool.
7. Repeat steps 5 and 6 until all syringes have been sealed.
8. Allow each syringe to cool for several minutes.
9. 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 the syringe does not pass this test, repeat steps 5 and 6.
10. Turn off the Bunsen burner and put it away.

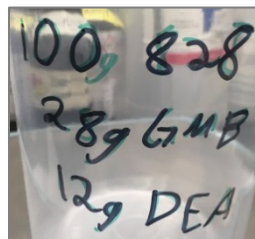
Set up mixing workspace:

1. Prepare workspace inside the fume hood by laying out paper towels around the balance and plugging the balance into the wall outlet. Check the bubble level to make sure the balance is level, and adjust as necessary. The rack holding the sealed syringes should be nearby and readily available.
2. Take out Popsicle sticks and pipettes. Label each for what chemical they will be handling with colored electrical tape, using a consistent labeling system (GMB: blue, DEA: green, 828: White).
3. Label a plastic container with the proportions of your batch: 100g 828; 12g DEA; 28g GMB.



Properly leveled balance

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)



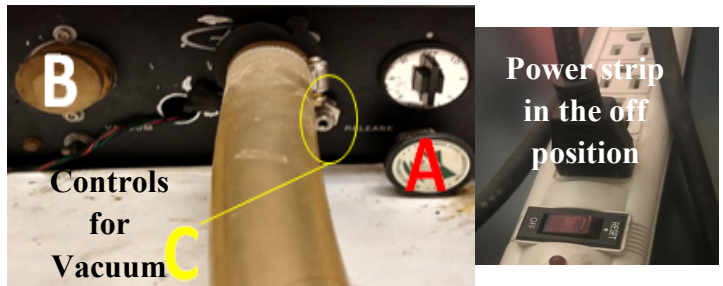
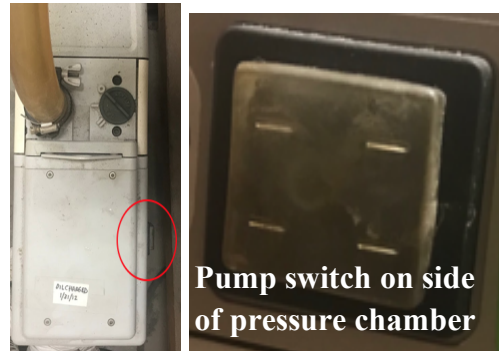
Plastic epoxy mixing container labeled with batch proportions.

Mixing Chemicals:

1. Begin by positioning your plastic container on the balance in the fume hood. Tare by pressing the “zero” button.
2. Take the 828 out of the oven and carefully pour it into the plastic container. 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 0.01g of the target mass. Put the 828 and the cup back in the oven when you are done with it.
3. Tare the scale again, and remove GMB from 107C oven. Remove 828 in cup from oven and place back on scale pour in required amount of GMB into 828 & mix w/ pre-heated stirrer. Again, try to get within 0.01g of the target mass.
4. Mix for ~ 2-3 minutes. After mixing place back into 70C oven ~ 2-3 minutes. Pull back out of 70C oven along w/ DEA pour in required amount of DEA & mix 2-3 minutes.

Degassing:

1. Place the mixture in the vacuum oven, 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 the power strip to the left of the pressure chamber to start the vacuum gauge. It should be beneath the neighboring oven.
3. Turn the release valve (A) to the right as tight 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 toggle (C). Begin to turn the pressure valve (B) to the left. This will start dropping the pressure. Unlike the release valve, you generally do not want to turn this knob to the tightest possible level.
5. Watch as the material foams to be sure to open the release valve if the foam reaches the top and repeat pulling the vacuum and releasing until the foam no longer gets close to the top of the cup. This is known as breaking. Then vacuum the material for 2-3



minutes after material breaks [starting at 10 and decreasing to 7 torr].

- Equalize the pressure in the chamber. Simultaneously open the release valve and close the pressure valve, and wait until the chamber reaches equilibrium to open the vacuum door.

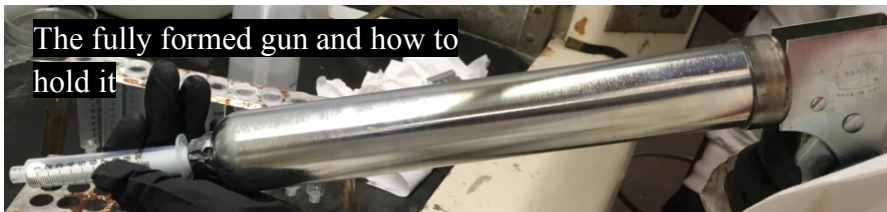
Pouring and Cure:

- Place back into 70C oven ~3-4 minutes until viscosity is thin. Place your degassed mixture along with your sealed syringes & syringe rack under the fume hood.
- Bend the lip of the mixture container into a spout shape.
- Hold the opening of the Semco Tube w/ tape on the nozzle end of Semco Tube & place upright in a container. Carefully bring the opening of the tube to the makeshift spout so that the sides of the two containers are near parallel. The quality of this configuration is what will determine how much air is incorporated back into the mixture during the pouring process, so line this up carefully.



Note: The goal of degassing is to remove air from the samples and decrease inconsistencies. Thus it is of utmost importance that while pouring the degassed epoxy into our molds, no air is re-incorporated into the mixture.

- Slowly pour the mixture down the side of the tube. Then place into heated vacuum chamber and pull vacuum for ~ 1-2 minutes.
 - After vacuuming place plunger in the end of the tube. Cut 2 small slits into tape to vent using a razor blade or scissors.
 - Put the tube inside the preheated metal sleeve and screw on the cocking end and screw on the tip through the tape.
 - Slowly cock the gun until the tip is starting to fill with epoxy.
- ****These steps must be done quickly. Within 1-2 min ****



- Fill the syringes by cocking the material into them making sure to put the tip as far in the syringe as possible and pulling out slowly as the syringe fills making sure that the syringe is filled up to the 10ml mark. Overfilling the syringe can make the epoxy removal process difficult, while under filling can lead to wasted epoxy when it comes time to cut samples for testing.



9. Carefully place the filled syringe back onto the rack.
10. Continue filling until you run out of epoxy; seal and cool extra syringes if needed; no epoxy should go to waste.
11. Place the rack of filled syringes inside the oven.
12. Cure for 24 hours

Note: The cure time should be handled with precision; as such, make sure that either you or another qualified lab assistant will be there to take out the epoxy cylinders exactly when specified.

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.
3. Put your lab coat and safety glasses back in 130B.
4. Wash your hands for at least a minute to ensure no chemicals linger on your skin.

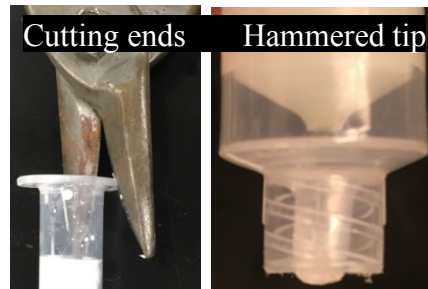
Recovering Cylinders:

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 bag, and sharpie. The first three can be found in the 130C toolbox, and the last two on the counter in 130B.
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. Repeat steps 4 and 5 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.